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EMB Report 91-ASP-11
September 1991

Air

ASPHALT
Emission Test Report

Mathy Construction Company
[redacted], Wisconsin

New Richmond

EMISSION TESTING FOR ASPHALT CONCRETE INDUSTRY

EMISSION TEST REPORT

Mathy Construction Company
Plant 26

EMB File No. 91-ASP-10
Work Assignment 1.44
Contract No. 68-D-90054

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1. INTRODUCTION

1.1 BACKGROUND

The Emission Inventory Branch (EIB) of the Environmental Protection Agency's (EPA's) Office of Air Quality Planning and Standards (OAQPS) is responsible for developing and maintaining air pollution emission factors for a variety of industrial processes. The EPA publishes these data in Compilation of Air Pollutant Emission Factors, commonly known as AP-42.

In October of 1986, the part of AP-42 that deals with asphaltic concrete plants was revised by dividing the plants into two major types based on the procedure used to mix the asphaltic concrete: continuous-mix plants and batch-mix plants. Following these revisions, and on the recommendation of the National Asphalt Paving Association (NAPA), EPA instructed Radian Corporation to direct emission testing efforts at these two types of plants.

Mathy Construction Company owns and operates asphaltic concrete plants that have been cited by NAPA as being representative of both continuous-mix and batch-mix plants currently operating in the United States. Radian has already conducted emissions tests at Mathy Construction's Plant No. 6, a batch-mix plant.

This report discusses the results of the testing program conducted by Radian, under contract to the EPA's Emission Management Branch (EMB), at Mathy Construction's Plant No. 26, a continuous drum-mix plant located near New Richmond, Wisconsin, and one of two asphaltic concrete plants studied for the revision of AP-42. The testing program quantified emissions of criteria and other air pollutants from the facility. The test results will be used by EIB to update the asphaltic concrete plant section of AP-42.

1.2 OVERVIEW OF TESTING PROGRAM

The specific pollutants of interest in the testing program were particulate matter (PM); PM less than 10 μm (PM_{10}); condensible PM (CPM); sulfur dioxide (SO_2); nitrogen oxides (NO_x); carbon monoxide (CO); total hydrocarbons (THC); polynuclear aromatic hydrocarbons (PAH), excluding aldehydes and ketones; and trace metals, excluding mercury (Hg).

Testing was performed from September 23 through 25, 1991. The principal objectives of the testing were to:

- Determine levels of CO, SO_2, NO_x, and THC emitted from the plant stack.
- Determine the levels of toxic metals being emitted from the stack, including lead (Pb), chromium (Cr), cadmium (Cd), beryllium (Be), thallium (Tl), arsenic (As), nickel (Ni), antimony (Sb), barium (Ba), silver (Ag), zinc (Zn), phosphorus (P), copper (Cu), manganese (Mn), and selenium (Se). The Hg levels were not analyzed because Hg was not expected to be present in the process stream.
- Determine the filterable PM_{10} and CPM fractions emitted from the stack.
- Determine the levels of PAHs emitted from the stack.
- Determine the quantities of benzene, toluene, xylene, and methane present in the stack exhaust gas.
- Monitor the process operating conditions, including aggregate flow rate, moisture, and ambient moisture.

In order to ensure repeatability of results, the above measurements were repeated in triplicate at near-design operating conditions while the plant was operating on waste fuel oil.

Flue gas concentrations of CO_2, O_2, NO_x, SO_2, and CO were determined using continuous emissions monitoring (CEM) systems designed in accordance with EPA Methods 3A, 7E, 6C, and 10. Emissions of THC were determined by CEM following EPA Method 25A. The EPA's Method 18 was followed in the gas chromatography (GC) analysis for flue gas concentrations of benzene, toluene, xylene, and methane. Samples of PM and metals were collected during three sampling train runs, performed according to EPA Method 5/Combined Train SW 846 Test Method 0031. The EPA SW 846 Test Method 0010 and 0011 were used in the collection of PAHs and speciated aldehydes and ketones, respectively. PM_{10} and CPM were sampled by means of three test runs, a combination of protocols outlined in EPA Methods 201A and 202, respectively. All of the manual method flue gas samples were analyzed in Radian's Perimeter Park laboratory facility in Morrisville, North Carolina.

1.3 BRIEF PROCESS AND SITE DESCRIPTION

Continuous mix plants operate in the following manner. The cold feed material known as aggregate is hauled from storage piles and placed in the appropriate hoppers. The aggregate is transferred to a set of vibrating screens and classified into as many as four different grades (sizes). The classified material then enters the drum mix operation.

The drum mix process uses proportioning feed controls in place of the hot aggregate storage bins, the vibrating screens, and a mixer, which are used in a batch mix plant. Aggregate is introduced near the burner end of a revolving drum mixer, and the asphalt is injected midway along the drum. A variable flow asphalt pump is linked electronically to the aggregate belt scales to control mix specifications. The hot mix is discharged from the revolving drum mixer into surge bins or storage silos.

Drum mix plants usually use parallel flow design for hot burner gases and aggregate flow. Parallel flow designs have the advantage of giving the mixture a longer time to coat the particles with asphalt and to collect dust in the mix, thereby reducing particulate emissions. The amount of particulate generated within the dryer in this process is usually lower than that generated within conventional dryers, but because asphalt is heated to high temperatures for a long period of time, organic emissions (gaseous and liquid aerosol) are greater than in conventional plants.

In recent years, old asphalt is removed from the road base and is broken up at a job site. This material is then transported to the plant, crushed and screened to the appropriate size for further processing. This recycled paving material is then heated and mixed with new aggregate, to which the proper amount of new asphaltic cement is added to produce a grade of hot asphalt paving suitable for laying.

Direct flame heating is typically performed with a drum mixer, wherein all materials are simultaneously mixed in the revolving drum. Split feed drum mixers were first used for recycling in 1976 and are now the most popular design. At about the midpoint of the drum, the recycled bituminous material is introduced by a split feed arrangement and is heated by both the hot gases and heat transfer from the superheated virgin aggregate.

The unit tested is a mobile 330-ton-per-hour-rated unit that is fired on waste oil. The rotary kiln dryer/mixer is a co-current design similar to that shown in Figure 1-1. The oil-fired burner fires into the upper end of the kiln and induces air flow around the annular area between the kiln and the burner. This arrangement provided approximately 30 percent of the air through the burner, with about 70 percent induced through the annular area.

The rest of this report is structured as follows. Section 2 contains a summary of the test results. The process is discussed in Section 3, and the sample locations and sampling and analytical procedures are presented in Sections 4 and 5, respectively. Section 6 presents results of implementing the quality assurance/quality control (QA/QC) procedures followed in the test program. Appendices to this report include detailed methods and procedures, field and laboratory data, and complete calculations used in deriving the results presented here.

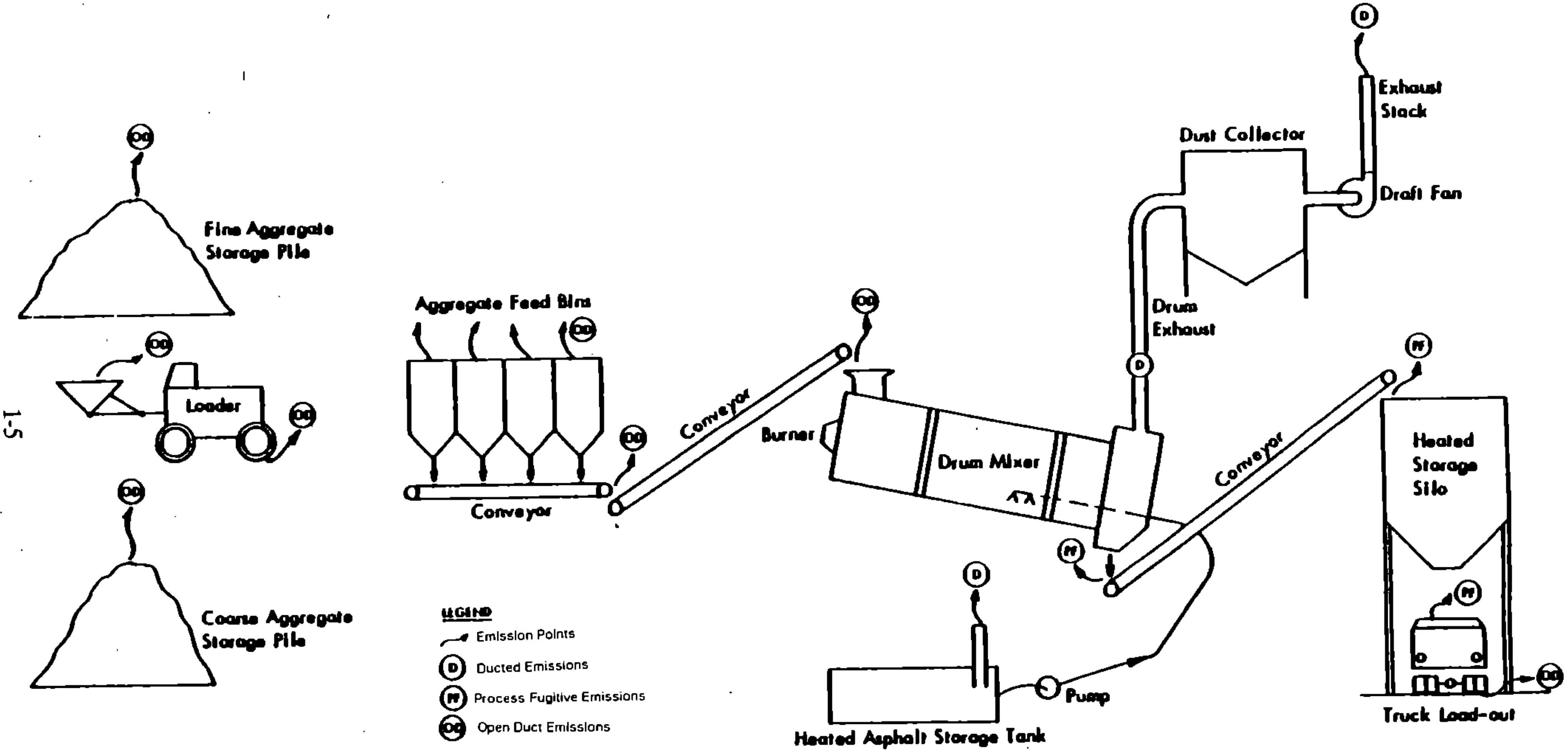


Figure 1-1. General Process Flow Diagram for Drum Mix Asphalt Paving Plants

2. SUMMARY OF TEST RESULTS

2.1 SUMMARY OF RESULTS

This section provides the results of the emission test program conducted at Mathy Construction Company's Plant 26. Included in this section are results of manual tests conducted for trace metals (excluding Hg), PM, PM_{10}, CPM, aldehydes and ketones, and PAHs. This section also contains the results of the continuous emissions monitoring (CEM) for CO_2/O_2, CO, SO_2, NO_x, and THC gases, as well as gas chromatography (GC) results for benzene, toluene, xylene, and methane.

The following list summarizes the significant emissions from Mathy Construction's Plant No. 26:

- Of the 15 metals analyzed, 11 were found in detectable quantities. Their average emission rates (lb/ton of product) were:

Arsenic	1.80×10^{-6};
Beryllium	4.81×10^{-6};
Cadmium	0.66×10^{-6};
Chromium	11.8×10^{-6};
Copper	6.05×10^{-6};
Lead	6.02×10^{-6};
Manganese	11.3×10^{-6};
Nickel	15.1×10^{-6};
Phosphorus	54.9×10^{-6};
Silver	1.36×10^{-6};
Zinc	52.6×10^{-6}.

- Of the 22 PAHs analyzed, 1 was found in detectable levels. Its average emission rate (lb/ton of product) was:

  Naphthalene 0.690×10^{-4}.

- For PM and PM_{10} the average emission rates (lb/ton of product) were:

PM	0.014;
PM_{10}	0.005;
PM_{10} + condensibles	0.040.

- Of the 18 aldehydes analyzed, 13 were found in detectable quantities. Their average emission rates (lb/ton of product) were:

Acetaldehyde	134×10^{-5};
Acetone	82.7×10^{-5};
Acrolein	4.83×10^{-5};
Benzaldehyde	11.2×10^{-5};
Butyraldehyde/Isobutyraldehyde	16.1×10^{-5};
Crotonaldehyde	8.62×10^{-5};
Formaldehyde	203×10^{-5};
Hexanal	10.9×10^{-5};
Isovaleraldehyde	4.10×10^{-5};
Methyl ethyl ketone	2.52×10^{-5};
Propionaldehyde	13.3×10^{-5};
Quinone	15.6×10^{-5};
Valeraldehyde	6.70×10^{-5}.

The following sections present more detailed discussions of the results of this test program.

2.2 EMISSIONS TEST LOG

Emissions testing was conducted over a three-day period, from September 23 to September 25, 1991. Table 2-1 shows the emissions test log, which includes the test date, sample location, run number, test type, run times, and average asphalt production rate during testing. Testing was performed using EPA manual test methods for six different analytes. Testing was conducted in triplicate for each type of analyte.

Particulate matter and metals were sampled in the same sampling train by employing a combination of EPA Method 5 and EPA SW 846 Test Method 0031. Particulate matter was determined gravimetrically from the front-half filter catch, and then was combined with the back half for total metals analysis.

The PAHs were sampled concurrently with PM and metals by EPA SW 846 Test Method 0010 using a dual probe arrangement, which allowed both trains to operate side-by-side with their nozzles in approximately the same sample location.

Sampling for aldehydes was conducted in a separate train using EPA SW 846 Test Method 0011.

Testing for PM_{10} and CPM was performed in a single train employing a combination of EPA Method 201A and EPA Method 202. An in-stack cyclone with a

Table 2-1. Emissions Test Log

Date	Location	Run Number	Test Type	Run Time	Average Production Rate (tph)
09/23/91	Stack[a]	1	PAH	08:30-10:39	193
09/23/91	Stack	2		13:55-16:06	184
09/24/91	Stack	3		12:36-15:03	147
09/23/91	Stack	1	PM	08:30-10:39	193
09/23/91	Stack	2		13:55-16:06	184
09/24/91	Stack	3		12:36-15:03	147
09/23/91	Stack	1	Metals	08:30-10:39	193
09/23/91	Stack	2		13:55-16:06	184
09/24/91	Stack	3		12:36-15:03	147
09/24/91	Stack	1	PM_{10}/CPM	10:05-11:20	212
09/25/91	Stack	2		10:25-11:35	257
09/25/91	Stack	3		13:15-14:30	223
09/23/91	Stack	1	Aldehydes	11:05-12:14	195
09/23/91	Stack	2		15:46-16:41	181
09/24/91	Stack	3		08:45-09:58	140
09/25/91	Stack	4		11:44-12:54	181
09/23/91	Stack	M-1	O_2/CO_2, SO_2, NO_x, THC	08:30-10:39	193
09/23/91	Stack	M-2		13:55-16:06	184
09/24/91	Stack	M-3		12:36-15:03	147
09/23/91	Stack	P-1		10:05-11:20	212
09/23/91	Stack	P-2		10:25-11:38	257
09/24/91	Stack	P-3		13:15-14:09	195

Table 2-1, continued

Date	Location	Run Number	Test Type	Run Time	Average Production Rate (tph)
09/23/91	Stack	A-1	O_2/CO_2, SO_2, NO_x, THC	11:15-12:14	195
09/23/91	Stack	A-2		15:51-16:41	181
09/24/91	Stack	A-3		08:45-09:58	140
09/25/91	Stack	A-4		11:44-12:54	181
09/23/91	Stack	1	Benzene, Toluene, Ethylbenzene, Xylene, Methane	09:20[b]	287
09/23/91	Stack	2		09:51	287
09/23/91	Stack	3		10:41	287
09/23/91	Stack	4		11:48	290
09/23/91	Stack	5		14:07	279
09/23/91	Stack	6		15:25	270
09/23/91	Stack	7		15:34	270
09/23/91	Stack			16:24	267
09/24/91	Stack	9		10:05	212
09/24/91	Stack	10		10:44	212
09/24/91	Stack	11		11:21	212
09/24/91	Stack	12		13:16	217
09/24/91	Stack	13		13:54	217
09/24/91	Stack	14		14:35	217
09/25/91	Stack	15		10:37	257
09/25/91	Stack	16		11:15	257
09/25/91	Stack	17		11:49	241
09/25/91	Stack	18		12:28	241
09/25/91	Stack	19		13:42	223

[a] After baghouse exit and ID fan.

[b] Exact times GC samples were shot. Individual results were averaged over the time period during which manual sampling was conducted.

backup filter composed the front half of this train. The cyclone captured PM greater than 10 microns. The backup filter caught PM of 10 microns or less. The CPM was caught in the back-half impingers.

A combination of CEM and GC instruments was used to sample for 11 other analytes. To the extent possible, these tests were conducted concurrently with the manual method tests. Continuous emissions monitoring was operated continuously and results were averaged over the manual test period in which they were performed. Gas chromatography measurements were taken on a semicontinuous basis and multiple readings were averaged over the manual test period in which they were performed.

2.3 METALS AND POLYNUCLEAR AROMATIC HYDROCARBON RESULTS

2.3.1 Overview

The PM/metals and PAH manual sampling trains shared a dual-probe arrangement, which allowed testing to be conducted simultaneously in separate trains at the same port location. The PM/metals sampling train was used to determine emissions of 15 metals (Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, P, Se, Ag, Tl, and Zn) and PM. The PAH sampling train was used to quantify emission rates of 19 PAHs (acenaphthylene, ancenaphthene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perlyene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, dibenzofuran, 7.12-dimethylbenz(a)anthracene, fluoranthene, florene, indeno(1,2,3-cd)pyrene, 2-methylnaphthalene, naphthalene, phenanthrene, and pyrene).

Three manual sampling runs for PM/metals and PAHs were performed to ensure representative test results. The back-half sample bottle from Run 2 of the PM/metals train was broken during shipment, so only the front half was analyzed for PM and metal content.

The remainder of this section discusses process operations, average emission rates, metals-to-PM ratios, and metals amounts in the flue gas-by-sample fractions.

2.3.2 Process Operation

Table 2-2 summarizes the metals and PAH emission factor results along with the process operating data. Sampling was performed in two separate trains simultaneously using a unique dual-probe arrangement. Total asphaltic concrete production varied between 217 tons/hr and 287 tons/hr (72 to 96 percent of capacity). Waste oil fuel use was not determined because no accurate method to evaluate fuel consumption was available.

The PAH emissions from asphaltic concrete plants may originate from fuel combustion; the volatile fraction of the asphalt cement, if any; and organic residues that are commonly found in recycled asphalt (i.e., gasoline, engine oils). Because there is no fuel use information for this facility, the emission factors presented are expressed in lb/ton of product rather than lb/ft^3 of fuel oil. During the emission tests, the plant was operating at near normal capacity, with Run 1 at 96 percent, Run 2 at 90 percent, and Run 3 at slightly lower than 72 percent capacity. Although Run 3 of the PAHs was at the low end of normal production capacity, it was still within acceptable limits (within ±20 percent of each other) and was used with Runs 1 and 2 in averaging. The production rates for Runs 1, 2, and 3 were 287 tons/hr, 270 ton/hr, and 217 tons/hr, respectively.

Only the metals and PAHs detected are given in Table 2-2. The other metals and PAHs were analyzed, but they were not collected in detectable amounts. Eleven of the 15 analyzed were detected (As, Ba, Cd, Cr, Cu, Pb, Mn, Ni, P, Ag, and Zn). Of the 22 PAHs analyzed, only naphthalene was detected. The metals and PAHs detected were blank-corrected and reported as shown.

2.3.3 Emissions

Metals

Table 2-3 presents the metals emissions results for the three tests. The date, metered volume (in dscm), O_2 concentration, and flue gas flow rate for each run are also shown. Flue gas concentrations are given in terms of μg/dscm and μg/dscm corrected to 7 percent O_2. Oxygen concentrations were determined from CEM data (see Section 2.7).

Table 2-2
SUMMARY OF METALS/PM AND PAHS EMISSION FACTORS AND PROCESS OPERATING DATA
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	Average
Date		09/23/91	09/23/91	09/24/91	
Production Rate	(tons/hr)	287	270	217	258
Virgin Aggregate Rate	(tons/hr)	193	183	147	174
Recycled Asphalt Rate	(tons/hr)	83	76	61	73
Asphalt Cement Rate	(tons/hr)	11.4	10.8	9.4	10.5
Percent of Rated Capacity	(%)	96	90	72	86
Aggregate Moisture	(%)	3.8	3.6	4.2	3.9
Burner Setting - Flame Meter	(%)	96	77	51	75
Ambient Temperature	(degree F)	51.1	60.2	56.0	55.8
Ambient Humidity	(%)	86	43	59	63
Kiln Exit Temperature	(degree F)	NA c	NA c	347	347
Stack Flow Rate	(dscfm)	18000	17900	17900	17900
Stack Flow Rate	(dscf/ton of product)	3765	3981	4952	4233
Stack Temperature	(degree F)	326	326	328	327
Stack Moisture	(% volume)	31.1	31.1	28.8	30.3
Stack CO2	(volume % dry)	9.2	7.6	7.2	8.0
Stack O2	(volume % dry)	10.3	10.0	11.8	10.7
Stack CO	(ppmV)	42.9	46.9	129	72.9
Control Device		Baghouse	Baghouse	Baghouse	
Arsenic	(lbs x 10(-6)/ton of product) a	1.49	0.915	2.99	1.80
Barium	(lbs x 10(-6)/ton of product) a	4.14	0.370	9.91	4.81
Cadmium	(lbs x 10(-6)/ton of product) a	0.266	0.588	0.965	0.606
Chromium	(lbs x 10(-6)/ton of product) a	15.6	18.6	1.26	11.8
Copper	(lbs x 10(-6)/ton of product) a	4.31	4.39	9.43	6.05
Lead	(lbs x 10(-6)/ton of product) a	4.66	5.28	8.13	6.02
Manganese	(lbs x 10(-6)/ton of product) a	5.64	14.1	14.1	11.3
Nickel	(lbs x 10(-6)/ton of product) a	25.1	19.8	0.458	15.1
Phosphorus	(lbs x 10(-6)/ton of product) a	43.7	48.4	72.8	54.9
Silver	(lbs x 10(-6)/ton of product) a	1.04	1.66	1.38	1.36
Zinc	(lbs x 10(-6)/ton of product) a	38.9	49.7	69.2	52.6
Particulate Matter	(lbs/ton of product)	0.0169	0.00968	0.0167	0.0144
Naphthalene	(lbs x 10(-4)/ton of product) b	0.540	0.935	0.594	0.690

a = 1 x 10(-6) or (0.000001)
b = 1 x 10(-4) or (0.0001)
c = Not available due to faulty plant instrumentation.

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run.
See Table 3-1 and 3-2 for the individual readings.
Metals and PAH compounds analyzed, but not detected, are not included in this table.
Metals and PAH concentrations have been blank corrected.

Table 2-3
METAL GAS CONCENTRATIONS EMISSION RATES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/23/91	09/23/91	09/24/91	
Metered Volume (dscm)		1.172	1.116	1.129	
O2 Concentration (%V)		10.3	10.0	11.8	
Flow Rate (dscmm)		510	508	508	Average
Arsenic	(ug/dscm)	6.31	3.67	9.65	6.55
	(ug/dscm @ 7% O2)	8.30	4.69	14.7	9.23
	(g/hr)	0.193	0.112	0.294	0.200
Barium	(ug/dscm)	17.6	1.49	32.0	17.0
	(ug/dscm @ 7% O2)	23.1	1.90	48.7	24.6
	(g/hr)	0.539	0.0453	0.975	0.520
Cadmium	(ug/dscm)	1.13	2.36	3.12	2.20
	(ug/dscm @ 7% O2)	1.49	3.01	4.75	3.08
	(g/hr)	0.0346	0.0720	0.0950	0.0672
Chromium	(ug/dscm)	66.3	74.9	4.07	48.4
	(ug/dscm @ 7% O2)	87.3	95.6	6.20	63.0
	(g/hr)	2.03	2.28	0.124	1.48
Copper	(ug/dscm)	18.3	17.6	30.5	22.1
	(ug/dscm @ 7% O2)	24.1	22.5	46.4	31.0
	(g/hr)	0.561	0.538	0.928	0.675

Table 2-3 (Continued)
METAL GAS CONCENTRATIONS EMISSION RATES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/23/91	09/23/91	09/24/91	
Metered Volume (dscm)		1.172	1.116	1.129	
O2 Concentration (%V)		10.3	10.0	11.8	
Flow Rate (dscmm)		510	508	508	Average
Lead	(ug/dscm)	19.8	21.2	26.3	22.4
	(ug/dscm @ 7% O2)	26.1	27.1	40.0	31.0
	(g/hr)	0.607	0.646	0.800	0.684
Manganese	(ug/dscm)	24.0	56.5	45.4	42.0
	(ug/dscm @ 7% O2)	31.5	72.1	69.1	57.6
	(g/hr)	0.734	1.72	1.38	1.28
Nickel	(ug/dscm)	107	79.6	1.48	62.6
	(ug/dscm @ 7% O2)	140	102	2.25	81.3
	(g/hr)	3.26	2.43	0.0451	1.91
Phosphorus	(ug/dscm)	186	194	235	205
	(ug/dscm @ 7% O2)	244	248	358	283
	(g/hr)	5.69	5.92	7.16	6.26
Silver	(ug/dscm)	4.42	6.68	4.47	5.19
	(ug/dscm @ 7% O2)	5.81	8.52	6.81	7.05
	(g/hr)	0.135	0.203	0.136	0.158
Zinc	(ug/dscm)	165	200	224	196
	(ug/dscm @ 7% O2)	218	255	340	271
	(g/hr)	5.06	6.08	6.81	5.99

During the emission tests, P had the highest average mass rate, with 6.26 g/hr, followed by Zn, with 5.99 g/hr. These emission rates correspond to an average emission factor of 54.9 x 10^{-6} lb/ton of product for P and 52.6 x 10^{-6} lb/ton of product for Zn. After blank correction, Sb, Be, Se, and Tl were not collected in detectable amounts for any of the runs during these emission tests. Metal values ranged from 0.0346 g/hr of CD in Run 1 to 7.16 g/hr of P in Run 3.

The metals values for the emission tests are not significantly different between runs except for As, Ba, and Mn. Arsenic varied from 0.112 to 0.294 g/hr, Ba varied from 0.0453 g/hr to 0.975 g/hr, and Mn varied from 0.734 to 1.72 g/hr.

Polynuclear Aromatic Hydrocarbons

Table 2-4 presents the PAH emission results for three test runs. The date, metered volume, O_2 concentration, and flow rate for each run are also shown. Flue gas concentrations are given in terms of g/dscm and g/dscm corrected to 7 percent O_2.

During the emission tests, only naphthalene was collected in a detectable amount. The average mass rate was 54.8 g/hr. These emission rates correspond to the average emission factors of 0.690 x 10^{-4} lb/ton of product. It should be noted that naphthalene may be a degradation by-product of the XAD absorbent used in the sample train. However, these results were blank-corrected and are reported as shown. The naphthalene values ranged from 39.6 g/hr in Run 3 to 77.6 g/hr in Run 2.

2.3.4 Ratios of Flue Gas Metals to Particulate Matter

A summary of the ratios of metals to PM for the emission tests is presented in Table 2-5. Metals-to-PM ratios are given in units of milligrams of metals per gram of PM collected by the sampling train. The values ranged from 0.0157 mg of Cd per gram of PM during Run 1 to 5.12 mg of Zn per gram of PM during Run 2. Phosphorous had the highest ratio for Runs 1 and 3, with 2.58 mg metal/gram PM and 4.34 mg metal/gram of PM, respectively.

2.3.5 Metals in Flue Gas By Sample Fraction and Metals in Flue Gas Sample Parameters

Table 2-6 presents the metal amounts in the flue gas samples by fraction for the emission tests. All metals detected were collected in the highest proportions in the front

Table 2-4
PAH GAS CONCENTRATIONS EMISSION RATES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/23/91	09/23/91	09/24/91	
Metered Volume (dscm)		1.173	1.186	1.102	
O2 Concentration (%V)		14.2	12.5	14.3	
Flow Rate (dscmm)		511	509	515	Average
Naphthalene	(ug/dscm)	1540	2540	1280	1790
	(ug/dscm @ 7% O2)	3170	4220	2680	3360
	(g/hr)	47.3	77.6	39.6	54.8

NOTE: Concentrations given have been blank corrected. PAH compounds analyzed, but not detected, are not included in this table.

Table 2-5
RATIO OF METALS TO PARTICULATE MATTER MATHY CONTRUCTION COMPANY PLANT 26 (1991)

Metal	METALS/PARTICULATE RATIO (mg metal per gram of particulate)			
	Run 1 (mg/g)	Run 2 (mg/g)	Run 3 (mg/g)	Average (mg/g)
Arsenic	0.0878	0.0943	0.178	0.120
Barium	0.245	0.0382	0.591	0.291
Cadmium	0.0157	0.0606	0.0576	0.0446
Chromium	0.923	1.92	0.0752	0.973
Copper	0.255	0.453	0.563	0.423
Lead	0.276	0.544	0.485	0.435
Manganese	0.333	1.45	0.838	0.874
Nickel	1.48	2.04	0.0273	1.18
Phosphorous	2.58	4.99	4.34	3.97
Silver	0.0615	0.171	0.0826	0.105
Zinc	2.30	5.12	4.13	3.85

NOTES: Metals analyzed but not detected are not included in this table.

Metals have been blank corrected.

Table 2-6
METAL AMOUNTS IN FLUE GAS SAMPLES BY SAMPLE FRACTION MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

METALS	Run 1			Run 2			Run 3		
	Front Half (total ug)	Back Half (total ug)	Total (ug)	Front Half (total ug)	Back Half (total ug)	Total (ug)	Front Half (total ug)	Back Half (total ug)	Total (ug)
Antimony	ND	ND	ND	ND	ND	ND	ND	ND	ND
Arsenic	7.40	ND	7.40	4.10	ND	4.10	10.9	ND	10.9
Barium	19.3	1.32	20.6	ND	1.66	1.66	34.8	1.33	36.1
Beryllium	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cadmium	1.07	0.254	1.32	1.92	0.716	2.64	3.52	ND	3.52
Chromium	76.5	1.26	77.8	80.5	3.07	83.6	3.30	1.30	4.60
Copper	20.4	1.07	21.5	16.8	2.89	19.7	34.4	ND	34.4
Lead	22.9	0.355	23.2	20.2	3.50	23.7	28.7	0.969	29.6
Manganese	26.7	1.39	28.1	38.7	24.4	63.1	49.7	1.56	51.3
Nickel	123	2.22	125	84.5	4.32	88.8	ND	1.67	1.67
Phosphorus	203	14.7	218	194	22.9	217	248	17.3	265
Selenium	ND	ND	ND	ND	ND	ND	ND	ND	ND
Silver	5.18	ND	5.18	7.45	ND	7.45	5.05	ND	5.05
Thallium	ND	ND	ND	ND	ND	ND	ND	ND	ND
Zinc	180	14.4	194	199	24.3	223	247	5.84	252

ND = Not detected

NOTE: Metals have been blank corrected.

collected in the highest proportions in the back half fraction. Laboratory analytical results for each sample fraction are presented in detail in Appendix E.1.

Sampling and flue gas parameters for the PM/metals runs are shown in Table 2-7. Total sampling times, sample volume, and isokinetic results for each sampling run are presented. Appendix C.1 contains a complete list of these and additional sampling and flue gas parameters for each run. The field data sheets are contained in Appendix A.1.

Polynuclear Aromatic Hydrocarbons

Table 2-8 presents the PAH amounts in the flue gas sample for the emission tests in total μg for each run. Laboratory analytical results for each sample are presented in detail in Appendix E.4.

Sampling and flue gas parameters for the PAH runs are shown in Table 2-9. Total sampling times, sample volume, and isokinetic results for each sampling run are presented. Appendix C.4 contains a complete list of these and additional sampling and flue gas parameters for each run, along with the field data sheets.

2.4 PARTICULATE MATTER

2.4.1 Overview

Particulate matter emissions were measured using the front half particulate catch collected in the combined PM/metals train. Before performing the metals speciation analysis, the filter and front half acetone rinse (e.g., rinsate from nozzle, probe, and filter holder) were analyzed gravimetrically as described in Section 5. The sampling and flue gas parameters have been presented previously in Table 2-7. Detailed sampling parameters are provided in Appendix C.1 and analytical results are given in Appendix E.1.

2.4.2 Particulate Matter Results

The results of the gravimetric analyses are summarized in Table 2-10. Exhaust grain loadings, corrected to 7 percent O_2, ranged from 0.0217 gr/dscf to 0.0413 gr/dscf, with an average of 0.0330 gr/dscf. Emission rates ranged from 2.61 lb/hr to 4.84 lb/hr, with an average of 3.69 lb/hr.

Table 2-2 summarizes emission factors for total PM, which varied from 0.00968 lb/ton of product to 0.0169 lb/ton of product with an average of 0.0144 lb/ton of

Table 2-7
METALS/PM EMISSIONS SAMPLING AND FLUE GAS PARAMETERS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run No. Date	Run 1 09/23/91	Run 2 09/23/91	Run 3 09/24/91	Average
Total Sampling Time (min)	125	125	124	NA
Average Sampling Rate (dscfm)	0.330	0.320	0.320	0.323
Metered Volume (dscf)	41.4	39.4	39.9	40.2
Metered Volume (dscm)	1.172	1.116	1.129	1.139
Average Stack Temperature (deg. F)	326	323	329	326
O2 Concentration (%V)	10.3	10.0	11.8	10.7
CO2 Concentration (%V)	9.2	7.6	7.2	8.0
Stack Gas Moisture (%V)	31.1	30.8	28.8	30.2
Volumetric Flow Rate (dscfm)	18000	17900	17900	17900
Volumetric Flow Rate (dscmm)	510	508	508	509
Percent Isokinetic	102	97.9	100	NA
Particulate Catch (grams)	0.0843	0.0435	0.0611	0.0630

NA = Not Applicable

Table 2-8
PAH AMOUNTS IN FLUE GAS SAMPLE - BLANK CORRECTED MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Field Blank (total ug)
Acenaphthylene	ND	ND	ND	ND
Acenaphthlene	ND	ND	ND	ND
Anthracene	ND	ND	ND	ND
Benzo(a)anthracene	ND	ND	ND	ND
Benzo(a)pyrene	ND	ND	ND	ND
Benzo(b)fluoranthene	ND	ND	ND	ND
Benzo(e)pyrene	ND	ND	ND	ND
Benzo(g,h,i)perylene	ND	ND	ND	ND
Benzo(k)fluoranthene	ND	ND	ND	ND
2-Chloronapthalene	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND
Dibenz(a,h)anthracene	ND	ND	ND	ND
Dibenzofuran	ND	ND	ND	ND
7,12-Dimethylbenz(a)anthracene	ND	ND	ND	ND
Fluoranthene	ND	ND	ND	ND
Fluorene	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	ND	ND	ND	ND
2-Methylnaphthalene	ND	ND	ND	ND
Naphthalene	1810	3010	1410	87.4 a
Perylene	ND	ND	ND	ND
Phenanthrene	ND	ND	ND	ND
Pyrene	ND	ND	ND	ND

a Estimated Maximum Possible Concentration

ND = Not Detected

NOTE: PAH values have been blank corrected.

Table 2-9
PAH EMISSIONS SAMPLING AND FLUE GAS PARAMETERS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run No. Date	Run 1 09/23/91	Run 2 09/23/91	Run 3 09/24/91	Average
Total Sampling Time (min)	125	125	125	NA
Average Sampling Rate (dscfm)	0.330	0.340	0.310	0.327
Metered Volume (dscf)	41.4	41.9	38.9	40.7
Metered Volume (dscm)	1.173	1.186	1.102	1.154
Average Stack Temperature (F)	326	323	327	325
O2 Concentration (%V)	14.2	12.5	14.3	13.7
CO2 Concentration (%V)	5.6	5.8	5.4	5.6
Stack Gas Moisture (%V)	31.4	31.0	28.3	30.2
Volumetric Flow Rate (dscfm)	18100	18000	18200	18100
Volumetric Flow Rate (dscmm)	511	509	515	512
Percent Isokinetic	102	104	95.4	NA

NA = Not Applicable

Table 2-10

PARTICULATE MATTER CONCENTRATIONS AND EMISSIONS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

				Flue Gas Concentration				Flue Gas Emission Rate	
Date	Location	Run #	Time (min)	(grains/dscf)	(grains/dscf @ 7% O_2)	(grams/dscm)	(grams/dscm @ 7% O_2)	(lb/hr)	(kg/hr)
09/07/91	Outlet	1	125	0.0314	0.0413	0.0719	0.0946	4.84	2.20
09/09/91	Outlet	2	125	0.0170	0.0217	0.0390	0.0497	2.61	1.19
09/10/91	Outlet	3	125	0.0237	0.0360	0.0541	0.0824	3.63	1.65
Averages				0.0240	0.0330	0.0550	0.0756	3.69	1.68

product. The emission factors generated from sampling Runs 1 and 3 are in relatively close agreement, whereas the second run emission factor represents the low endpoint of the data set.

These emission factors are slightly greater than those currently published in AP-42 for similar facilities (0.098 lb/ton after baghouse control, from data from circa 1973-74). This difference may be attributable to a variety of factors, including product specifications or differences in baghouse design, operation, and maintenance between facilities tested.

2.5 PM_{10}/CPM RESULTS

2.5.1 Overview

Three test runs were conducted to determine the concentration and emission rate of PM_{10}. The testing procedures followed EPA Method 201A for the determination of PM_{10} emissions using the constant sampling rate (CSR) procedure coupled with EPA Method 202 for determining condensible emissions from the sampling train's back half. The CSR employs normal isokinetic sampling procedures except that the sample duration at each sampling point is proportional to the gas velocity at that point.

Sampling cutpoints ranged from 9.24 microns to 10.6 microns, with an average cut size of 9.79 microns. These particle size separations were achieved with isokinetic rates measured from 81 to 91 percent which are acceptable by the method specifications.

A full description of the method procedures can be found in Section 5, as well as in the EPA Reference Method located in Appendix I.2. The final results from this test procedure are presented in terms of the phase of PM caught as well as the cut size. The following PM weight fractions were determined:

- Noncondensible PM > 10 microns (cyclone fraction);
- Noncondensible PM < 10 microns (filter fraction);
- Inorganic CPM associated with the water fraction (< 10 microns); and
- Organic CPM associated with the methylene chloride fraction (< 10 microns).

2.5.2 PM_{10} Emissions

The average emission rate and emission factor for PM_{10} including condensibles were 8.24 lb/hr and 0.0352 lb/ton of product, although the condensible and PM_{10} filter catches for Run 2 totaled almost twice those collected in Runs 1 and 3. The sizable deviation in condensible emissions detected in Run 2 may be attributable to several factors, including abnormalities in fuel characteristics or an increase in VOC residues in the recycled asphalt concrete.

The emission factors calculated for PM greater than 10 microns averaged 0.017 lb/ton, and all sampling runs were in relatively close agreement. Also, these data are similar to the emission factors for PM calculated from the PM/metals tests discussed earlier.

The emission factors developed from both fractions of these tests are higher than those currently found in AP-42. The factors for PM_{10} are three to six times greater than current AP-42 data, although AP-42 literature does not discuss specific fuels used or residual VOC contents of recycled asphalt cement for the emission factors published (e.g., natural gas or oil).

It should be noted that EPA Method 201A recommends adding the condensible fractions to the PM_{10} filter catch when compiling data for emission inventory purposes. As seen in Tables 2-11 and 2-12, this procedure results in substantially higher PM_{10} emission factors due to the large quantities of condensible PM found in the back half water fraction. The quantities of PM_{10} alone (from the backup filter catch only) are shown for comparison. The average emission rate and emission factor for PM_{10} alone were 1.19 lb/hr and 0.0052 lb/ton of product, which is substantially lower than the values with the condensible fractions added, namely 8.24 lb/hr and 0.0352 lb/ton of product.

The front half instack filter catch (i.e., cyclone catch plus backup filter catch) does not compare well with the PM filter catch performed by EPA Method 5. One possible reason for this discrepancy is the fact that testing by these methods was performed at different times. One would expect the Method 5 PM results to be slightly larger since the filter was operated at lower temperatures that then EPA Method 201A instack filter

Table 2-11

SUMMARY OF PM10/CPM EMISSION FACTORS AND PROCESS OPERATING DATA MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	Average
Date		09/24/91	09/25/91	09/25/91	
Production Rate	(tons/hr)	212	257	223	231
Virgin Aggregate Rate	(tons/hr)	143	175	152	157
Recycled Asphalt Rate	(tons/hr)	58	72	62	64
Asphalt Cement Rate	(tons/hr)	10.9	10.1	9.1	10.0
Percent of Rated Capacity	(%)	71	86	74	77
Aggregate Moisture	(%)	4.2	4.4	4.2	4.3
Burner Setting - Flame Meter	(%)	51	88	70	70
Ambient Temperature	(degree F)	51.7	60.3	58.2	56.7
Ambient Humidity	(%)	66	NT	NT	66
Kiln Exit Temperature	(degree F)	347	334	322	334
Stack Flow Rate	(dscfm)	20000	16000	17900	17967
Stack Flow Rate	(dscf/ton of product)	5600	5270	6710	5860
Stack Temperature	(degree F)	328	317	305	317
Stack Moisture	(% volume)	28.8	37.7	36.3	34.3
Stack CO2	(volume % dry)	7.1	7.6	4.7 a	7.4 a
Stack O2	(volume % dry)	12.0	10.4	10.8	11.1
Stack CO	(ppmV)	130	120	55.0	102
Control Device		Baghouse	Baghouse	Baghouse	
PM10 Emission	(lbs/ton) b	0.0050	0.0046	0.0060	0.0052
Particulate Emission < Cut Size	(lbs/ton of product) c	0.0336	0.0447	0.0274	0.0352
Particulate Emission > Cut Size	(lbs/ton of product) d	0.0146	0.0168	0.0198	0.0171
Particulate Emission Total	(lbs/ton of product) e	0.0482	0.0615	0.0472	0.0523

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run. See Table 3-2 and 3-3 for the individual readings.

a CO2 value suspiciously low. Not used in average.
b Includes PM10 filter catch only.
c Includes PM10 filter catch and back half condensibles.
d Includes cyclone catch only.
e Includes the sum of all fractions.

Table 2-12
PM10 EMISSION TEST RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Stack Conditions	Run 1	Run 2	Run 3	Average
Corrected Barometric Pressure (in. Hg)	29.38	20.98	20.98	23.78
Stack Static Pressure (in. H2O)	-0.80	-0.80	-0.80	-0.80
Average Stack Temperature (deg. F)	328	317	305	317
Carbon Dioxide Concentration (%V)	7.06	7.60	4.74 a	7.33 a
Oxygen Concentration (%V)	12.01	10.40	10.83	11.08
Nitrogen Concentration (%V)	80.93	80.93	84.43	82.10
Stack Moisture (%V)	28.8	37.7	36.3	34.3
Stack Gas Velocity,Vs (fps)	64.5	81.6	87.5	77.9
Volumetric Flow Rate (acfm)	43000	54300	58200	51800
Volumetric Flow Rate (dscfm)	20000	16000	17900	18000
Stack Viscosity (micropoise)	219	209	207	212
Sampling Parameters				
Total sampling time (min)	64.3	65.1	70.2	66.5
Average Meter Temperature (deg. F)	59	69	71	66
Average Meter Pressure (in.H2O)	0.24	0.30	0.30	0.28
Average Sampling Rate (dscfm)	0.27	0.22	0.22	0.24
Average Sampling Rate (acfm - cyclone cond)	0.57	0.74	0.71	0.67
Standard Metered Volume,Vm(std) (dscf)	17	14	15	16
Percent Isokinetic	89.1	91.4	80.8	87.1
PM10 Results				
Cut Size (um)	10.62	9.22	9.49	9.78
Cyclone Particulate Catch (g)	0.0202	0.0294	0.0286	0.0261
PM10 Filter Particulate Catch (g)	0.0070	0.0080	0.0086	0.0079
H2O Back Half Particulate Catch (g)	0.0354	0.0619	0.0279	0.0417
MeCl Back Half Particulate Catch (g)	0.0040	0.0080	0.0031	0.0050
Total Particulate Catch (g)	0.0666	0.1073	0.0682	0.0807
PM10 Concentration (grains/dscf) b	0.0062	0.0086	0.0087	0.0078
PM10 Concentration (grains/dscf @7% O2) b	0.0098	0.0113	0.0120	0.0110
Particulate Conc. < Cut Size (grains/dscf) c	0.0414	0.0835	0.0400	0.0549
Particulate Conc. < Cut Size (grains/dscf @7% O2) c	0.0647	0.1105	0.0552	0.0768
Particulate Conc. > Cut Size (grains/dscf) d	0.0180	0.0315	0.0289	0.0261
Particulate Conc. > Cut Size (grains/dscf @7% O2) d	0.0282	0.0417	0.0398	0.0366
Particulate Conc. Total (grains/dscf) e	0.0594	0.1149	0.0688	0.0810
Particulate Conc. Total (grains/dscf @7% O2) e	0.0929	0.1521	0.0950	0.1133
PM10 Emissions (lbs/hr) b	1.07	1.18	1.33	1.19
Particulate Emissions < Cut Size (lbs/hr) c	7.11	11.48	6.12	8.24
Particulate Emissions > Cut Size (lbs/hr) d	3.10	4.33	4.42	3.95
Particulate Emissions Total (lbs/hr) e	10.21	15.81	10.54	12.19

a CO2 value suspiciously low. Not used in average.
b Includes PM10 filter catch only.
c Includes PM10 filter catch an back half condensibles.
d Includes cyclone catch only.
e Includes the sum of all fractions.

(248°F versus 317°F). Therefore, the EPA Method 5 filter would be expected to catch more of the condensible matter than the instack cyclone filter combination. However, such was not the case. It is not known why this discrepancy occurred.

The PM_{10} emission factors and process operating data are summarized in Table 2-11. The analytical results are shown in Table 2-12. Figure 2-1 illustrates the particle size contributions to mass emission rates. Figure 2-2 shows the particle size contributions to flue gas PM concentrations under actual conditions and conditions corrected to 7 percent O_2. Figure 2-3 illustrates the relative contribution of each sample fraction to total PM catch for each sample run.

2.6 ALDEHYDE RESULTS

2.6.1 Overview

A single sampling train was used to collect samples for analysis of 18 aldehydes (acetaldehyde, acetone, acetophenone/o-tolualdehyde, acrolein, benzaldehyde, butyraldehyde/isobutyraldehyde, crotonaldehyde, 2,5-dimethylbenzaldehyde, formaldehyde, hexanal, isophorone, isovaleraldehyde, MIBK/p-tolualdehyde, methyl ethyl ketone, propionaldehyde, quinone, m-tolualdehyde, and valeraldehyde). Four sampling runs were performed in order to ensure representative test results.

2.6.2 Process Operation

Table 2-13 presents the aldehyde emission factors with a summary of process operating data for the four test runs. During the emission tests, the plant was operating at maximum production load of 97, 89, and 80 percent for Runs 1, 2, and 4, respectively, but at a reduced load of 67 percent for Run 3. However, all runs were considered within typical operating ranges and, therefore, all four runs were used in averaging. The production rates were 290 tons/hr, 267 tons/hr, 202 tons/hour, and 241 tons/hr for Runs 1, 2, 3, and 4, respectively.

Only the aldehydes detected are presented. The other aldehydes were analyzed, but were not collected in detectable amounts. Of the 18 aldehydes analyzed, the following 13 were detected: acetaldehyde, acetone, acrolein, benzaldehyde, butyraldehyde /isobutyraldehyde, crotonaldehyde, formaldehyde, hexanal, isovaleraldehyde, methyl ethyl ketone, propionaldehyde, quinone, and valeraldehyde.

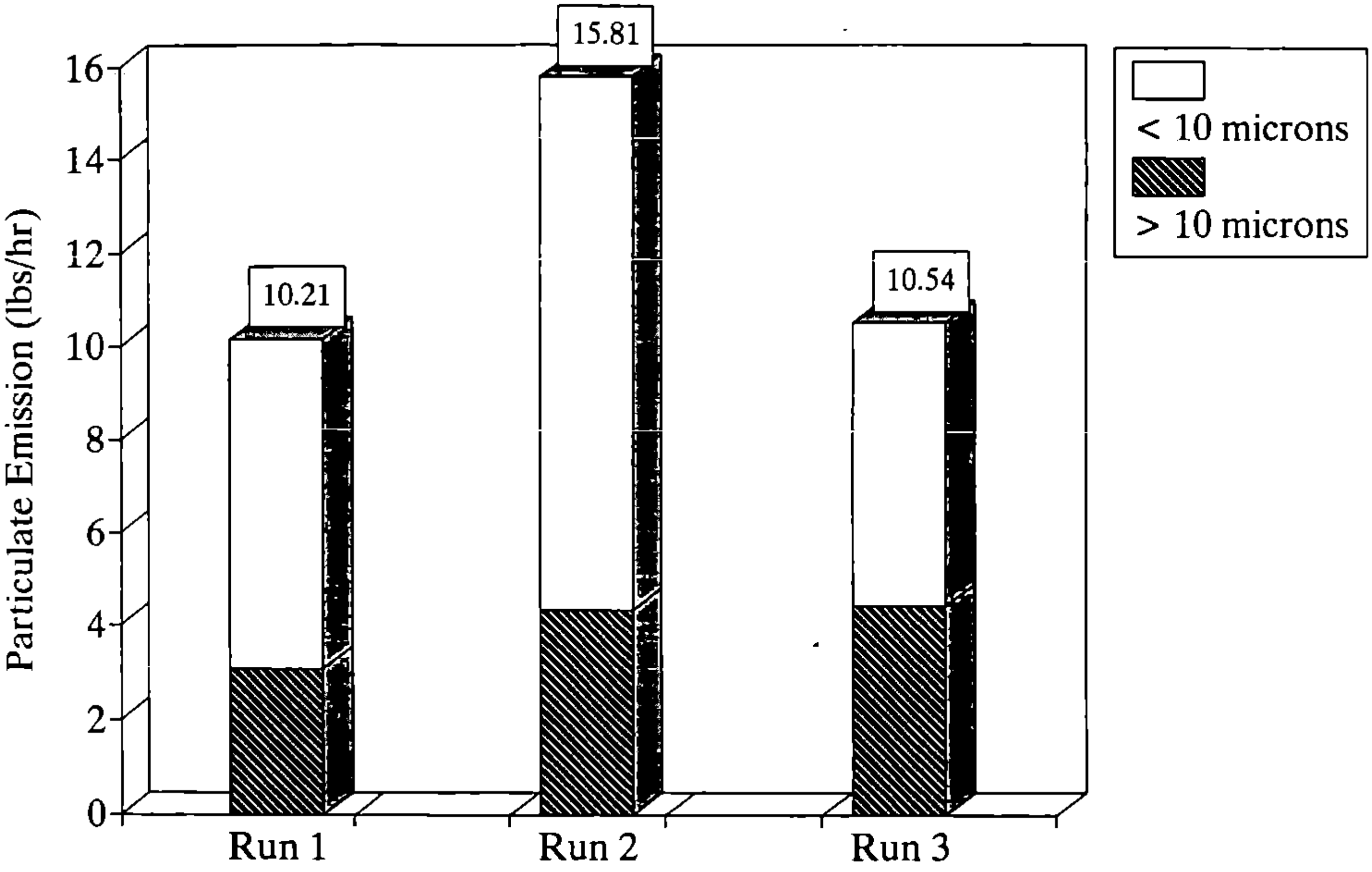
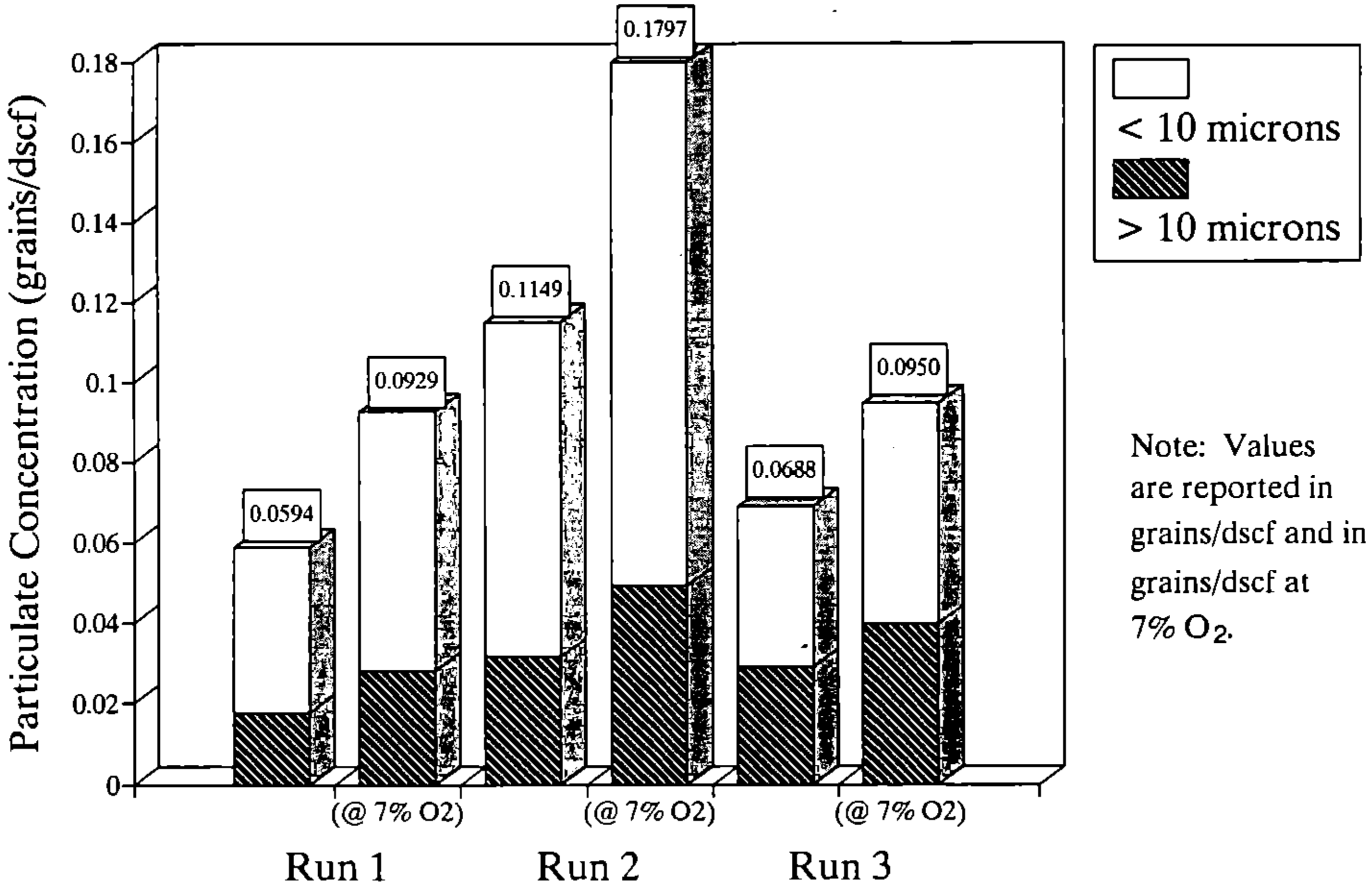


Fig 2-1 PM_{10} PARTICULATE EMISSIONS (lbs/hr)

Fig 2-2 PM_{10} PARTICULATE CONCENTRATION (grains/dscf)



Note: Values are reported in grains/dscf and in grains/dscf at 7% O_2.

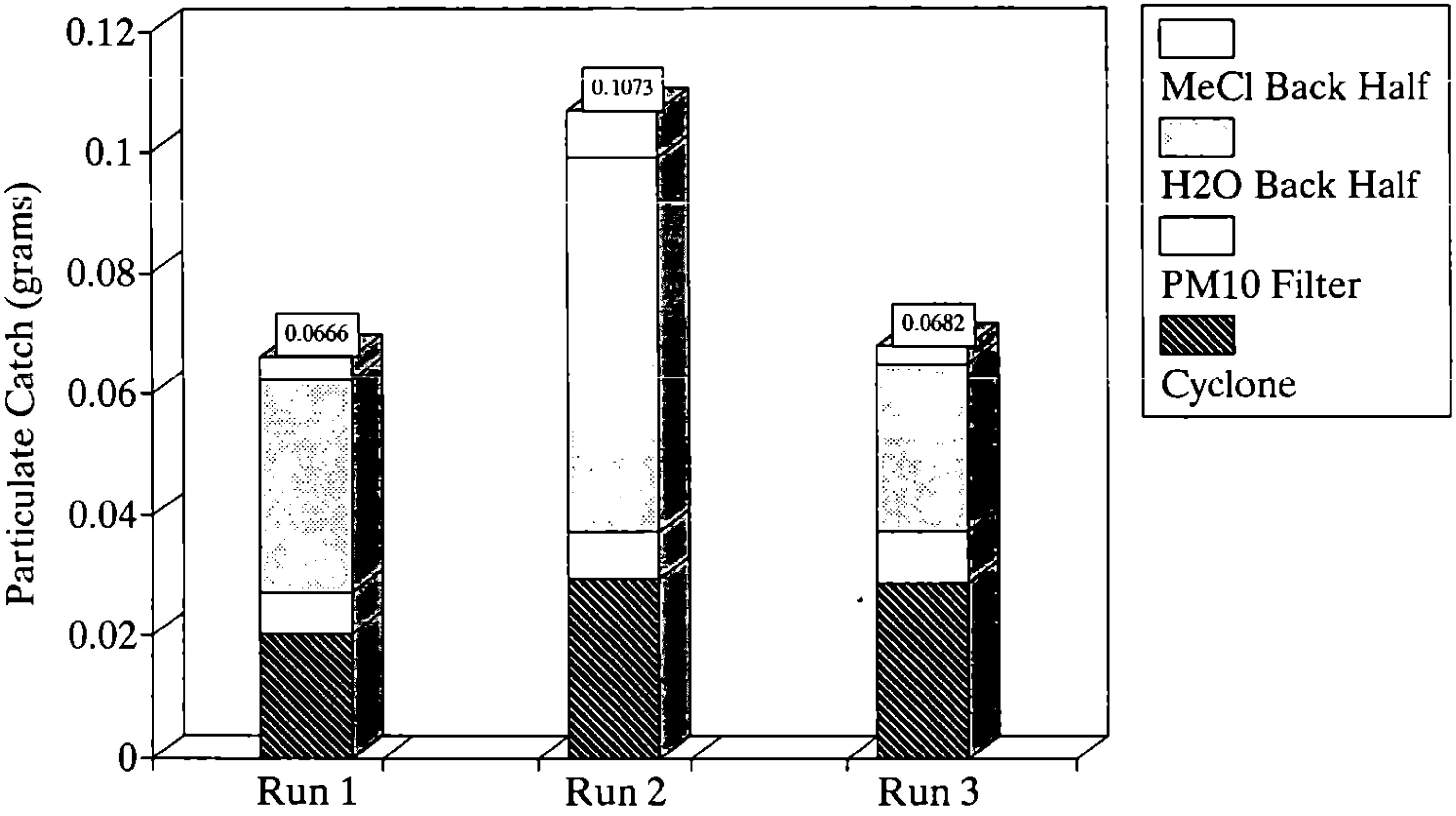


Fig 2-3 PM_{10} PARTICULATE CATCH (grams)

Table 2-13
SUMMARY OF ALDEHYDE EMISSION FACTORS AND PROCESS OPERATING DATA
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	4	Average
Date		09/23/91	09/23/91	09/24/91	09/25/91	
Production Rate	(tons/hr)	290	267	202	241	250
Virgin Aggregate Rate	(tons/hr)	192	181	138	165	169
Recycled Asphalt Rate	(tons/hr)	87	75	55	66	71
Asphalt Cement Rate	(tons/hr)	11.4	10.8	9.4	9.8	10.4
Percent of Rated Capacity	(%)	97	89	67	80	83
Aggregate Moisture	(%)	3.8	3.6	4.2	4.4	4.0
Burner Setting - Flame Meter	(%)	100	74	37	79	73
Ambient Temperature	(degree F)	57.9	58.6	48.4	58.7	55.9
Ambient Humidity	(%)	37	46	86	NT	56
Kiln Exit Temperature	(degree F)	NA b	NA b	333	341	337
Stack Flow Rate	(dscfm)	19400	18800	19500	18300	19000
Stack Flow Rate	(dscf/ton of product)	4008	4228	5781	4560	4644
Stack Temperature	(degree F)	311	320	315	317	316
Stack Moisture	(% volume)	30.6	39.5	24.4	28.9	30.9
Stack CO2	(volume % dry)	6.8	8.2	6.2	6.4	6.9
Stack O2	(volume % dry)	13.0	10.3	12.9	11.1	11.8
Stack CO	(ppmV)	31.2	47.6	374	86.5	134.8
Control Device		Baghouse	Baghouse	Baghouse	Baghouse	
Acetaldehyde	(lbs x 10(-5)/tons of product) a	103	55.3	253	125	134
Acetone	(lbs x 10(-5)/tons of product) a	51.7	85.1	80.8	113	82.7
Acrolein	(lbs x 10(-5)/tons of product) a	3.11	ND	ND	6.55	4.83
Benzaldehyde	(lbs x 10(-5)/tons of product) a	2.51	2.96	32.6	6.60	11.2
Butyraldehyde/Isobutyraldehyde	(lbs x 10(-5)/tons of product) a	14.1	11.5	27.2	11.6	16.1
Crotonaldehyde	(lbs x 10(-5)/tons of product) a	3.17	2.18	24.1	4.99	8.62
Formaldehyde	(lbs x 10(-5)/tons of product) a	59.9	94.0	509	149	203
Hexanal	(lbs x 10(-5)/tons of product) a	5.52	6.16	22.3	9.78	10.9
Isovaleraldehyde	(lbs x 10(-5)/tons of product) a	2.18	ND	5.94	4.17	4.10
Methyl Ethyl Ketone	(lbs x 10(-5)/tons of product) a	1.07	ND	5.60	0.880	2.52
Propionaldehyde	(lbs x 10(-5)/tons of product) a	7.00	4.75	32.7	8.85	13.3
Quinone	(lbs x 10(-5)/tons of product) a	3.55	7.02	35.2	16.8	15.6
Valeraldehyde	(lbs x 10(-5)/tons of product) a	2.59	2.85	14.8	6.55	6.70

a = 1 x 10(-5) or (0.00001)
b = Not available due to faulty plant instrumentation.
NT = Not Taken
ND = Not Detected

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run.
See Table 3-1 and 3-2 for the individual readings.
Concentrations given have been blank corrected.
Aldehyde compounds analyzed, but not detected, are not included in this table.

Aldehyde emissions from this plant are most likely a function of fuel consumption and any volatile fraction of the asphalt cement. Because a fuel flow rate could not be measured at this facility, the emission factors presented are expressed in lb/ton of product rather than lb/ft^3 of fuel oil.

2.6.3 Emissions

Table 2-14 presents the aldehyde emissions results for the four test runs. The date, metered volume (in dscm), O_2 concentration, and flow rate for each run are also shown. Flue gas concentrations are given in terms of μg/dscm, and μg/dscm corrected to 7 percent O_2 and g/hr. Oxygen concentrations were collected from CEM data.

During the emission tests, formaldehyde had the highest average mass rate, with 206 g/hr, followed by acetaldehyde, with 143 g/hr. After blank correction, acetophenone/o-tolualdehyde, 2,5-dimethylbenzaldehyde, isophorone, isovaleraldehyde, MIBK/p-tolualdehyde, and m-tolualdehyde yielded insignificant amounts for any of the runs during the emission tests. Aldehyde values ranged from 2.88 g/hr of isovaleraldehyde in Run 1 to 467 g/hr of formaldehyde in Run 3.

The aldehyde values for the emission tests did not change significantly from Runs 1, 2, and 4. Run 3 shows markedly higher values because of the low production rate and poor combustion characteristics at the lower end of unit design specifications.

2.6.4 Flue Gas Aldehydes by Sample and Sample Parameters

Table 2-15 presents the aldehyde amounts in the flue gas sample for the emission tests in total μg for each run. Laboratory analytical results for each sample are presented in detail in Appendix E.3.

Sampling and flue gas parameters for the aldehyde runs are shown in Table 2-16. Total sampling times, sample volume, and isokinetic results for each sampling run are presented. Appendix A.3 contains a complete list of these parameters for each test run, along with the field data sheets.

Table 2-14

ALDEHYDE CONCENTRATIONS EMISSION RATES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run		Run 1	Run 2	Run 3	Run 4	
Date		09/23/91	09/23/91	09/24/91	09/25/91	
Metered Volume (dscm)		0.863	0.582	0.838	0.581	
O2 Concentration (%V)		13.0	10.3	12.9	11.1	
Flow Rate (dscmm)		551	531	552	517	Average
Acetaldehyde	(ug/dscm)	4090	2100	7020	4410	4400
	(ug/dscm @ 7% O2)	7180	2750	12200	6250	7100
	(g/hr)	135	66.9	232	137	143
Acetone	(ug/dscm)	2060	3230	2240	3980	2880
	(ug/dscm @ 7% O2)	3620	4240	3910	5640	4350
	(g/hr)	68.1	103	74.2	123	92.2
Acrolein	(ug/dscm)	124	ND	ND	231	177
	(ug/dscm @ 7% O2)	218	ND	ND	327	272
	(g/hr)	4.10	ND	ND	7.16	5.63
Benzaldehyde	(ug/dscm)	100	112	903	232	337
	(ug/dscm @ 7% O2)	176	147	1580	329	558
	(g/hr)	3.31	3.58	29.9	7.21	11.0
Butyraldehyde/Isobutyraldehyde	(ug/dscm)	561	435	755	408	540
	(ug/dscm @ 7% O2)	984	570	1320	578	863
	(g/hr)	18.5	13.9	25.0	12.7	17.5
Crotonaldehyde	(ug/dscm)	126	82.6	669	176	263
	(ug/dscm @ 7% O2)	222	108	1170	249	437
	(g/hr)	4.17	2.63	22.2	5.45	8.61
Formaldehyde	(ug/dscm)	2390	3570	14100	5230	6320
	(ug/dscm @ 7% O2)	4200	4680	24600	7410	10200
	(g/hr)	79.0	114	467	162	206

ND = Not Detected

NOTES: Concentrations given have been blank corrected.

Aldehyde compounds analyzed, but not detected, are not included in this table.

Table 2-14 (Continued)
ALDEHYDE CONCENTRATIONS EMISSION RATES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run		**Run 1**	**Run 2**	**Run 3**	**Run 4**	
Date		**09/23/91**	**09/23/91**	**09/24/91**	**09/25/91**	
Metered Volume (dscm)		**0.863**	**0.582**	**0.838**	**0.581**	
O2 Concentration (%V)		**13.0**	**10.3**	**12.9**	**11.1**	
Flow Rate (dscmm)		**551**	**531**	**552**	**517**	**Average**
Hexanal	(ug/dscm)	220	234	619	344	354
	(ug/dscm @ 7% O2)	386	306	1080	488	565
	(g/hr)	7.27	7.45	20.5	10.7	11.5
Isovaleraldehyde	(ug/dscm)	87.0	ND	165	147	133
	(ug/dscm @ 7% O2)	153	ND	287	208	216
	(g/hr)	2.88	ND	5.45	4.56	4.29
Methyl Ethyl Ketone	(ug/dscm)	42.8	ND	155	31.0	76.3
	(ug/dscm @ 7% O2)	75.0	ND	271	43.9	130
	(g/hr)	1.41	ND	5.14	0.961	2.50
Propionaldehyde	(ug/dscm)	279	180	906	312	419
	(ug/dscm @ 7% O2)	490	237	1580	441	687
	(g/hr)	9.23	5.75	30.0	9.67	13.7
Quinone	(ug/dscm)	141	266	976	592	494
	(ug/dscm @ 7% O2)	248	349	1700	839	784
	(g/hr)	4.67	8.49	32.3	18.4	16.0
Valeraldehyde	(ug/dscm)	103	108	411	231	213
	(ug/dscm @ 7% O2)	181	142	716	327	341
	(g/hr)	3.41	3.45	13.6	7.16	6.90

ND = Not Detected

NOTES: Concentrations given have been blank corrected.

Aldehyde compounds analyzed, but not detected, are not included in this table.

Table 2-15
ALDEHYDE AMOUNTS IN FLUE GAS SAMPLES - BLANK CORRECTED MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Run 4 (total ug)	Field Blank (total ug)
Acetaldehyde	3530	1220	5880	2560	ND
Acetone	1780	1880	1880	2310	396
Acetophenone/o-Tolualdehyde	ND	ND	ND	ND	ND
Acrolein	107	ND	ND	134	ND
Benzaldehyde	86.4	65.3	757	135	ND
Butyraldehyde/Isobutyraldehyde	484	253	633	237	ND
Crotonaldehyde	109	48.1	561	102	ND
2,5-Dimethylbenzaldehyde	ND	ND	ND	ND	ND
Formaldehyde	2060	2080	11800	3040	14.5
Hexanal	190	136	519	200	ND
Isophorone	ND	ND	ND	ND	ND
Isovaleraldehyde	75.1	ND	138	85.3	ND
MIBK/p-Tolualdehyde	ND	ND	ND	ND	ND
Methyl Ethyl Ketone	36.9	ND	130	18.0	ND
Propionaldehyde	241	105	759	181	ND
Quinone	122	155	818	344	ND
m-Tolualdehyde	ND	ND	ND	ND	ND
Valeraldehyde	89.1	62.9	344	134	ND

ND = Not Detected
NOTE: Aldehyde values have been blank corrected.

Table 2-16
ALDEHYDES EMISSIONS SAMPLING AND FLUE GAS PARAMETERS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run No. Date	Run 1 09/23/91	Run 2 09/23/91	Run 3 09/24/91	Run 4 09/25/91	Average
Total Sampling Time (min)	62.5	38.5	62.5	62.5	NA
Average Sampling Rate (dscfm)	0.490	0.530	0.470	0.330	0.455
Metered Volume (dscf)	30.5	20.6	29.6	20.5	25.3
Metered Volume (dscm)	0.863	0.582	0.838	0.581	0.716
Average Stack Temperature (F)	311	320	315	317	316
O2 Concentration (%V)	13.0	10.3	12.9	11.1	11.8
CO2 Concentration (%V)	6.8	8.2	6.2	6.4	6.9
Stack Gas Moisture (%V)	30.6	39.5	24.4	28.9	30.8
Volumetric Flow Rate (dscfm)	19400	18800	19500	18300	19000
Volumetric Flow Rate (dscmm)	551	531	552	517	538
Percent Isokinetic	98.8	112	95.8	96.0	NA

NA = Not Applicable

2.7 CONTINUOUS EMISSIONS MONITORING RESULTS

2.7.1 Overview

Continuous emissions monitoring was conducted at the outlet to the air pollution control device (APCD) during the three days of testing. Concentrations of O_2, CO_2, CO, NO_x, and SO_2 were determined on a dry basis by extracting the gas from the flue, transferring it to the CEM trailer through heated Teflon tubing (heat trace), passing it through gas conditioners to remove moisture, and directing it to each respective analyzer. A full description of Radian's CEM system and methods is given in Section 5. Concentrations of THC were also monitored, with gas concentrations determined on a wet basis by allowing a slipstream from the heated sample line to bypass the sample conditioners so that the wet flue gas was directed to the analyzer as it exited in the flue.

All CEM data were recorded as 30-second averages from multiple-readings-per-second input by Radian's CEM data acquisition system (DAS). The resulting CEM data files were averaged over the duration of each test run. The averages are presented in Section 2.7.2. The 30-second data are included in Appendix D.

2.7.2 Continuous Emission Monitoring Results

The CEM averages are presented in Table 2-17. The average O_2 concentrations ranged from 10.1 to 13.0 percent by volume. Average CO values ranged from 4.7 to 9.2 percent by volume. Average NO_x concentrations ranged from 94.2 to 145.7 ppmv. Concentrations of THC were also monitored, with the resulting average concentrations ranging from 488.4 to 601.8 ppmv (wet). Sulfur dioxide values were approximately 100 to 150 ppmv, while average CO concentrations ranged from 31.3 to 374.0 ppmv.

All of the QA/QC procedures were followed as specified in the test plan. The final results are valid, as all of the CEM performance specifications were attained and 9 of the 10 runs had 100 percent completeness in relationship to the manual run times. The QA/QC results are discussed further in Section 6.

2.7.3 Nonmethane Hydrocarbon Emission Test Results

EPA Method 18 analysis of the flue gas was performed using GC to separate the hydrocarbon species (C_1-C_6) present in the flue gas stream. Several samples were injected into the GC during each test day. Methane, benzene, toluene, ethylbenzene,

Table 2-17
CONTINUOUS EMISSIONS MONITORING DAILY TEST AVERAGES
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Test	Run Number	Date	Manual Run Time	CEM Run Time	O2 (%V)	THC (ppmv)	SO2 (ppmv)	CO (ppmv)	CO2 (%V)	NOx (ppmv)
PAH/Metals	1	09/23/91	0830-1039	0830-1039	10.3	554.2	150.9	42.9	9.2	142.9
	2	09/23/91	1355-1606	1355-1606	10.1	582.9	144.0	41.8	7.6	145.7
	3 a	09/24/91	1236-1503	1236-1503	11.8	515.1	123.0	128.4	7.2	112.7
Aldehyde	1	09/23/91	1105-1214	1115-1214	13.0	564.7	102.0	31.3	6.8	104.5
	2	09/23/91	1546-1641	1551-1641	10.1	601.8	145.1	42.0	8.4	145.1
	3	09/24/91	0845-0958	0845-0958	12.9	531.0	112.8	374.0	6.2	94.2
	4	09/25/91	1144-1254	1144-1254	11.0	538.3	138.0	79.3	6.5	132.7
PM10/CPM	1	09/24/91	1005-1120	1005-1120	12.0	483.4	118.2	129.9	7.1	109.9
	2	09/25/91	1025-1138	1025-1138	10.4 b	561.1	144.5	118.7	7.6 b	139.4
	3	09/25/91	1315-1430	1315-1409	10.8 b	497.1	135.9	55.3	4.7 b	139.8

a PAH/Metals Run 3 interrupted 1311-1334 because of down time.

b Suspicious data could be due to instrument problems.

and xylene were determined by this method. Total hydrocarbon emissions as methane were determined by EPA Method 25A.

The hydrocarbon concentrations and emission rates in ppmv and lb/ton of asphalt produced are presented in Table 2-18. The nonmethane hydrocarbon emissions as methane were calculated by subtracting the methane concentration measured by the GC from the total hydrocarbons as methane measured by the total hydrocarbon analyzer (CEM) at the time of sample injection into the GC. The average nonmethane hydrocarbon emissions were 0.0913, 0.121, and 0.01 lb/ton for the three test days. The average emission factors of benzene, toluene, ethylbenzene, and xylene for the first test day were 0.00021, 0.0011, 0.00027, and 0.000098 lb/ton, respectively. The average emission factors for the second test day were 0.00072, 0.00068, 0.00078, and 0.0 lb/ton, respectively. The average emission factors for the third test day were 0.00041, 0.00028, 0.00019, and 0.00014 lb/ton, respectively.

2.8 ASTM METHODS

This section presents results of laboratory analysis of waste oil fuel collected September 26, 1991. Standard ASTM methods were used to assess heat of combustion, ultimate analysis (ash, O_2, carbon, hydrogen, sulfur, and nitrogen), and chlorine content of the sample.

The results are given in Table 2-19. The waste fuel oil sample was anlayzed by McCoy & McCoy Laboratories.

Table 2-18. Hydrocarbon Emission Rates and Concentrations

Date	Time	Stack Flow Rate (dscfm)	Asphalt Production Rate (tons/hr)	Methane By GC (ppmv)	Total Hydrocarbons By THC (ppmv)	Nonmethane Hydrocarbons		Benzene		Toluene		Ethylbenzene		Xylene	
						(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)
09/23/91	0920	18061	287	5.128	481.1	476.0	7.47E-02	0.417	3.19E-04	1.31	1.18E-03	ND	0	ND	0
	0951	18061	287	ND	554.2	554.2	8.69E-02	0.161	1.23E-04	1.137	1.03E-03	ND	0	ND	0
	1041	18061	287	ND	559.4	559.4	8.78E-02	ND	0	1.075	9.70E-04	ND	0	ND	0
	1148	19443	290	ND	567.6	567.6	9.49E-02	0.316	2.57E-04	1.546	1.49E-03	ND	0	0.101	1.12E-04
	1407	17975	279	ND	557.5	557.5	8.95E-02	0.301	2.36E-04	0.577	5.33E-04	0.332	3.54E-04	0.158	1.68E-04
	1525	17975	270	0.796	566.5	565.7	9.39E-02	0.283	2.29E-04	ND	0	0.266	2.93E-04	0.007	7.71E-06
	1534	17975	270	ND	593.2	593.2	9.84E-02	0.347	2.81E-04	1.927	1.84E-03	1.157	1.27E-03	0.454	5.00E-04
	1624	18760	267	ND	596.7	596.7	1.05E-01	0.309	2.64E-04	1.759	1.77E-03	0.22	2.56E-04	ND	0
AVERAGE		18289	280	0.74	559.5	558.8	9.13E-02	0.27	2.14E-04	1.17	1.10E-03	0.25	2.72E-04	0.09	9.85E-05
STANDARD DEVIATION		517	8	0.347	15.3	15.4	5.3E-3	0.11	8.89E-5	0.63	6.2E-4	0.36	4.0E-4	0.15	1.64E-4
09/24/91	1005	20000	212	0.04	508.1	508.1	1.19E-01	0.581	6.66E-04	ND	0	1.502	2.34E-03	ND	0
	1044	20000	212	0.606	489.7	489.1	1.15E-01	0.554	6.35E-04	ND	0	ND	0	ND	0
	1121	20000	212	0.362	492.2	491.8	1.16E-01	0.664	7.61E-05 ?	0.748	1.01E-03	1.179	1.84E-03	ND	0
	1316	18185	217	0.223	738.6	738.4	1.54E-01	0.797	8.12E-04	0.86	1.03E-03	0.349	4.84E-04	ND	0
	1354	18185	217	0.125	547.9	547.8	1.14E-01	0.721	7.34E-04	0.911	1.09E-03	ND	0	ND	0
	1435	18185	217	0.561	508.9	508.3	1.06E-01	0.726	7.39E-04 ?	0.811	9.74E-04	ND	0	ND	0
AVERAGE		19093	215	0.32	547.6	547.2	1.21E-01	0.67	7.25E-04	0.56	6.86E-04	0.51	7.78E-04	0	0
STANDARD DEVIATION		1252	11	0.195	85.3	85.3	1.54E-2	0.08	6.29E-5	0.37	4.5E-4	0.57	8.85E-4	0.01	8.2E-6
09/25/91	1037	16000	257	2.028	609.2	607.2	9.42E-02	0.99	7.49E-04	1.002	8.94E-04	0.261	2.69E-04	0.441	4.54E-04
	1115	16000	257	0.128	545.8	545.7	8.47E-02	0.517	3.19E-04	0.132	1.18E-04	0.068	7.00E-05	0.036	3.71E-05

Table 2-18. Continued

Date	Time	Stack Flow Rate (dscfm)	Asphalt Production Rate (tons/hr)	Methane By GC (ppmv)	Total Hydrocarbons By THC (ppmv)	Nonmethane Hydrocarbons		Benzene		Toluene		Ethylbenzene		Xylene	
						(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)
	1149	18260	241	0.155	520.3	520.1	9.82E-02	0.378	3.48E-04	ND	0	ND	0	ND	0
	1228	18260	241	0.162	535.4	535.2	1.01E-01	ND	0	ND	0	ND	0	0.259	3.25E-04
	1342	17900	223	0.136	505.2	505.1	1.01E-01	0.224	2.19E-04	ND	0	0.004	5.31E-06	0.005	6.64E-06
AVERAGE		17585	239	0.49	543.9	543.4	1.00E-01	0.46	4.05E-04	0.28	2.83E-04	0.14	1.87E-04	0.12	1.37E-04
STANDARD DEVIATION		1318	19	0.688	43.1	42.6	6.78E-3	0.3	2.24E-4	0.36	3.21E-4	0.1	1.23E-4	0.16	1.73E-4

ND = Not Detected

? ? ?

Avg. computed differently from rest.

Table 2-19

Summary of Waste Oil Analysis
Mathy Construction Company Plant 26 (1991)

Analyte	Amount Found (%)
Ash	2.91
Carbon	83.43
Hydrogen	11.03
Nitrogen	0.22
Sulfur	0.65
Oxygen	1.72
Chlorine	0.04
Heat of Combustion	18364 Btu/lb

3. FACILITY DESCRIPTION

This section provides a description of the Mathy Construction Company's Plant No. 26 asphaltic concrete plant located near New Richmond, Wisconsin. The process equipment and production materials used and the process parameters recorded during the emissions test are discussed.

3.1 PROCESS DESCRIPTION

Mathy Construction Plant No. 26 is a typical continuous drum asphaltic concrete plant with a rated production capacity of 300 tons per hour. The plant consists of the following components:

- Aggregate storage piles;
- Recycled asphalt concrete storage piles;
- A rotary kiln/drum mixer for aggregate drying and asphalt cement mixing; and
- A heated asphalt cement storage tank.

Virgin aggregate and recycled asphalt concrete must be combined and dried to product specifications in the oil-fired rotary kiln/drum mixer. Both materials are introduced at the burner end of the drum. Asphalt cement, an amorphous solid that must be heated to a liquid state for injection into the drum mixer, is then mixed with the dried aggregate and asphaltic concrete. The final product leaving the drum mixer is transferred to a hot mix storage bin and then loaded into trucks.

Aggregate fines become entrained in the combustion exhaust leaving the rotary kiln/drum mixer. The fines are then transported by the flue gas to a fabric filter, where they are collected. Process fugitive PM and VOCs from the drum mixer are also routed to the fabric filter inlet. Emissions of VOCs from fuel combustion, organic contaminants in recycled asphalt, and the asphalt cement are not controlled by the fabric filter, nor are any VOC emission control devices used. The exhaust flow is induced by a clean-air-side fan.

The data collected only reflect emissions from vented process equipment as described above and do not include emissions from process and area fugitive sources such as:

- Aggregate and recycled asphalt concrete storage piles and cold transport of these materials;
- Cold asphalt cement and hot mix storage tanks; or
- Plant vehicular traffic.

Point source PM, PM_{10}, and metal emissions are attributable primarily to aggregate-drying and hot transport mechanisms. Condensible PM, PAH, and aldehyde emissions are generally associated with fuel combustion products and the volatile fraction of the liquid asphalt, although relative contributions of each are not found in the literature. The aggregate/asphalt throughputs are functions of the desired product specifications, whereas fuel consumption is determined by the drying requirements, aggregate moisture, and throughput of the aggregate. For the emission factors developed, the final product composition is 41 to 43 percent recycled asphalt and 5.8 percent asphalt cement by weight, and the drum fuel is a specification waste oil. The emission factors developed in this study reflect these constraints, and the effects of changes in these parameters on emission factors have not been evaluated.

3.2 PROCESS CONDITIONS DURING TESTING

Production monitoring data for all tests are presented in Tables 3-1 through 3-3.

3.2.1 Process Conditions During Metals/PAH Testing

Table 3-4 summarizes the production and operating conditions associated with the metals and PAH test data. Production rates for three runs varied from 217 tons per hour to 287 tons per hour (72 to 96 percent of rated capacity). Data regarding fuel consumption were not readily available because no functioning fuel flow rate measuring device was available. Sampling Run 3 was interrupted by a brief process shutdown, but sampling activity was suspended during this time.

3.2.2 Process Conditions During Aldehyde Testing

Table 3-5 summarizes the production and operating data associated with aldehyde test data. Production rates varied from 241 tons per hour to 290 tons per hour (80 to

Table 3-1
SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING EMISSION TESTING MATHY CONSTRUCTION COMPANY PLANT 26 - SEPTEMBER 23, 1991

Time (24hr)	Burner Setting (%)	Production Rate			Mix Temp. (degree F)	Kiln Exit Gas Temp. (degree F)	Baghouse Pressure Drop (In w.g.)	Ambient Temp. (degree F)	Relative Humidity (%)	Exhaust Damper Position
		Virgin Aggregate (tons/hr)	RAP (tons/hr)	Asphalt Cement (tons/hr)						
8:30	100	195	81	11.3	285	NA a	10.5	47.0	---	Fully Open
9:13	100	187	79	11.1	270	NA a	10.0	49.8	86	Fully Open
9:56	86	190	82	11.1	279	NA a	10.5	52.6	---	Fully Open
10:39	96	201	88	12.0	275	NA a	10.5	55.0	---	Fully Open
11:28	100	198	87	11.6	275	NA a	10.5	57.8	---	Fully Open
12:00	100	193	83	11.4	287	NA a	10.5	58.0	37	Fully Open
14:05	84	187	88	11.2	275	NA a	10.7	58.8	---	Fully Open
14:29	84	186	69	10.8	285	NA a	10.5	60.8	43	Fully Open
15:00	64	182	79	10.7	264	NA a	10.5	61.2	---	Fully Open
15:30	76	182	80	10.7	295	NA a	10.5	60.0	---	Fully Open
16:30	72	180	72	10.8	285	NA a	10.0	58.2	46	Fully Open
16:47	Shutdown									

a Not available due to faulty plant instrumentation.

Table 3-2
SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING EMISSION TESTING
MATHY CONSTRUCTION COMPANY PLANT 26 - SEPTEMBER 24, 1991

		Production Rate								
Time (24hr)	Burner Setting (%)	Virgin Aggregate (tons/hr)	RAP (tons/hr)	Asphalt Cement (tons/hr)	Mix Temp. (degree F)	Kiln Exit Gas Temp. (degree F)	Baghouse Pressure Drop (In w.g.)	Ambient Temp. (degree F)	Relative Humidity %	Exhaust Damper Position
8:30	34	148	65	8.8 c	269	331	11	48.2	---	Fully Open
9:14	30	134	55	6.8 c	265	324	10.8	48.5	86	Fully Open
9:36	48	138	49	10.0 c	275	345	10.7	---	---	Fully Open
10:31	48	143	57	11.2 c	282	355	10.5	51.4	---	Fully Open
11:08	50	145	63	11.9 c	278	339	10.6	51.8	---	Fully Open
11:30	56	143	58	9.5 c	282	348	10.5	52.0	---	Fully Open
12:15	---	---	---	---	---	---	---	55.6	47	Fully Open
12:21	53	144	65	8.3	275	334	10.5	---	---	Fully Open
12:35	53	146	62	8.6	275	341	10.3	55.8	---	Fully Open
13:18 a	54	147	62	10.7	275	366	10.5	---	---	Fully Open
13:36 b	52	145	56	10.7	265	342	11.2	55.2	---	Fully Open
14:06	51	144	56	8.9	282	357	10.0	---	---	Fully Open
14:31	48	152	67	8.6	275	344	10.0	57.0	59	Fully Open
15:06	48	147	60	8.7	272	334	10.0	---	---	Fully Open

a Plant Down
b Restarted Plant
c Value Varies

Table 3-3
SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING EMISSION TESTING MATHY CONSTRUCTION COMPANY PLANT 26 - SEPTEMBER 25, 1991

Time (24hr)	Burner Setting (%)	Production Rate			Mix Temp. (degree F)	Kiln Exit Gas Temp. (degree F)	Baghouse Pressure Drop (in w.g.)	Ambient Temp. (degree F)	Relative Humidity (%)	Exhaust Damper Position
		Virgin Aggregate (tons/hr)	RAP (tons/hr)	Asphalt Cement (tons/hr)						
8:54	---	---	---	---	---	---	---	56.0	71	Fully Open
9:25	72	146	65	8.9	288	358	11.2	---	---	Fully Open
10:00	62	151	68	8.8	299	349	9.7	---	---	Fully Open
10:33	97	182	77	10.6	280	340	10.0	60.4	---	Fully Open
11:10	90	175	69	10.3	285	330	10.0	60.2	---	Fully Open
11:49	76	189	76	10.8	275	333	9.5	---	---	Fully Open
12:07	82	173	76	10.2	295	349	10.0	---	---	Fully Open
13:24	63	146	58	8.9	255	320	10.6	58.2	---	Fully Open
13:36	76	173	74	9.4	270	324	9.9	---	---	Fully Open
14:15 a	---	---	---	---	---	---	---	---	---	Fully Open
14:30 b	---	---	---	---	---	---	---	62.0	---	Fully Open
14:47	84	167	72	9.8	295	338	10.6	---	---	Fully Open
15:00	90	172	87	10.4	270	---	10.6	60.0	---	Fully Open

a Plant Down
b Restarted Plant

Table 3-4
SUMMARY OF METALS/PM AND PAHS PROCESS OPERATING CONDITIONS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	Average
Date		09/23/91	09/23/91	09/24/91	
Production Rate	(tons/hr)	287	270	217	258
Virgin Aggregate Rate	(tons/hr)	193	183	147	174
Recycled Asphalt Rate	(tons/hr)	83	76	61	73
Asphalt Cement Rate	(tons/hr)	11.4	10.8	9.4	10.5
Percent of Rated Capacity	(%)	96	90	72	86
Aggregate Moisture	(%)	3.8	3.6	4.2	3.9
Burner Setting - Flame Meter	(%)	96	77	51	75
Ambient Temperature	(degree F)	51.1	60.2	56.0	55.8
Ambient Humidity	(%)	86	43	59	63
Kiln Exit Temperature	(degree F)	266	269	347	294
Stack Flow Rate	(dscfm)	18000	17900	17900	17933
Stack Flow Rate	(dscf/ton of product)	3765	3981	4952	4233
Stack Temperature	(degree F)	326	326	328	327
Stack Moisture	(% volume)	31.1	31.1	28.8	30.3
Stack CO2	(volume % dry)	9.2	7.6	7.2	8.0
Stack O2	(volume % dry)	10.3	10.0	11.8	10.7
Stack CO	(ppmV)	42.9	46.9	129	72.9
Control Device		Baghouse	Baghouse	Baghouse	

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run. See Table 3-1 and 3-2 for the individual readings.

Table 3-5
SUMMARY OF ALDEHYDE PROCESS OPERATING CONDITIONS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	4	Average
Date		09/23/91	09/23/91	09/24/91	09/25/91	
Production Rate	(tons/hr)	290	267	202	241	250
Virgin Aggregate Rate	(tons/hr)	192	181	138	165	169
Recycled Asphalt Rate	(tons/hr)	87	75	55	66	71
Asphalt Cement Rate	(tons/hr)	11.4	10.8	9.4	9.8	10.4
Percent of Rated Capacity	(%)	97	89	67	80	83
Aggregate Moisture	(%)	3.8	3.6	4.2	4.4	4.0
Burner Setting - Flame Meter	(%)	100	74	37	79	73
Ambient Temperature	(degree F)	57.9	58.6	48.4	58.7	55.9
Ambient Humidity	(%)	37	46	86	NT	56
Kiln Exit Temperature	(degree F)	264	278	333	341	304
Stack Flow Rate	(dscfm)	19400	18800	19500	18300	19000
Stack Flow Rate	(dscf/ton of product)	4008	4228	5781	4560	4644
Stack Temperature	(degree F)	311	320	315	317	316
Stack Moisture	(% volume)	30.6	39.5	24.4	28.9	30.9
Stack CO2	(volume % dry)	6.8	8.2	6.2	6.4	6.9
Stack O2	(volume % dry)	13.0	10.3	12.9	11.1	11.8
Stack CO	(ppmV)	31.2	47.6	374	86.5	135
Control Device		Baghouse	Baghouse	Baghouse	Baghouse	

NT = Not Taken

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run. See Table 3-1 and 3-2 for the individual readings.

97 percent of capacity). No process shutdowns or upsets occurred during the other sampling runs.

3.2.3 Process Conditions During PM_{10}/CPM Testing

The production and operating data that correspond to the PM_{10}/CPM test results are summarized in Table 3-6. For three sampling runs, total asphaltic concrete production varied between 212 tons per hour and 257 tons per hour (71 to 86 percent of capacity). Sampling Run 3 was briefly interrupted by a process shutdown. The sampling train was removed from the stack and capped during this interruption.

Table 3-6
SUMMARY OF PM10/CPM PROCESS OPERATING CONDITIONS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Run Number		1	2	3	Average
Date		09/24/91	09/25/91	09/25/91	
Production Rate	(tons/hr)	212	257	223	231
Virgin Aggregate Rate	(tons/hr)	143	175	152	157
Recycled Asphalt Rate	(tons/hr)	58	72	62	64
Asphalt Cement Rate	(tons/hr)	11.0	10.0	9.0	10.0
Percent of Rated Capacity	(%)	71	86	74	77
Aggregate Moisture	(%)	4.2	4.4	4.2	4.3
Burner Setting - Flame Meter	(%)	51	88	70	70
Ambient Temperature	(degree F)	51.7	60.3	58.2	56.7
Ambient Humidity	(%)	66	NT	NT	66
Kiln Exit Temperature	(degree F)	347	334	322	334
Stack Flow Rate	(dscfm)	20000	16000	17900	17967
Stack Flow Rate	(dscf/ton of product)	5600	5270	6710	5860
Stack Temperature	(degree F)	328	317	305	317
Stack Moisture	(% volume)	28.8	37.7	36.3	34.3
Stack CO2	(volume % dry)	7.1	7.6	4.7	6.5
Stack O2	(volume % dry)	12.0	10.4	10.8	11.1
Stack CO	(ppmV)	130	120	55.0	102
Control Device		Baghouse	Baghouse	Baghouse	

NOTES: Run averages were calculated from readings taken periodically throughout the duration of the emission test run. See Table 3-1 and 3-2 for the individual readings.

4. SAMPLING LOCATIONS

This section describes the locations where flue gas samples were taken during the emission testing program at Mathy Construction Company Plant No. 26. All samples collected by manual methods, including PM_{10} samples, were collected from sampling ports at equal heights in the exhaust stack. Samples for CEMS were collected from a single point near the manual sampling ports. The sampling location arrangement is shown in Figure 4-1.

The test ports were located according to EPA Method 1. The nearest upstream disturbance was 3.7 equivalent diameters away and the nearest downstream disturbance was 1.2 equivalent diameters away from the test ports.

The minimum number of traverse points required for manual sampling was 24. Five points at each of the five ports were used, as shown in Figure 4-2.

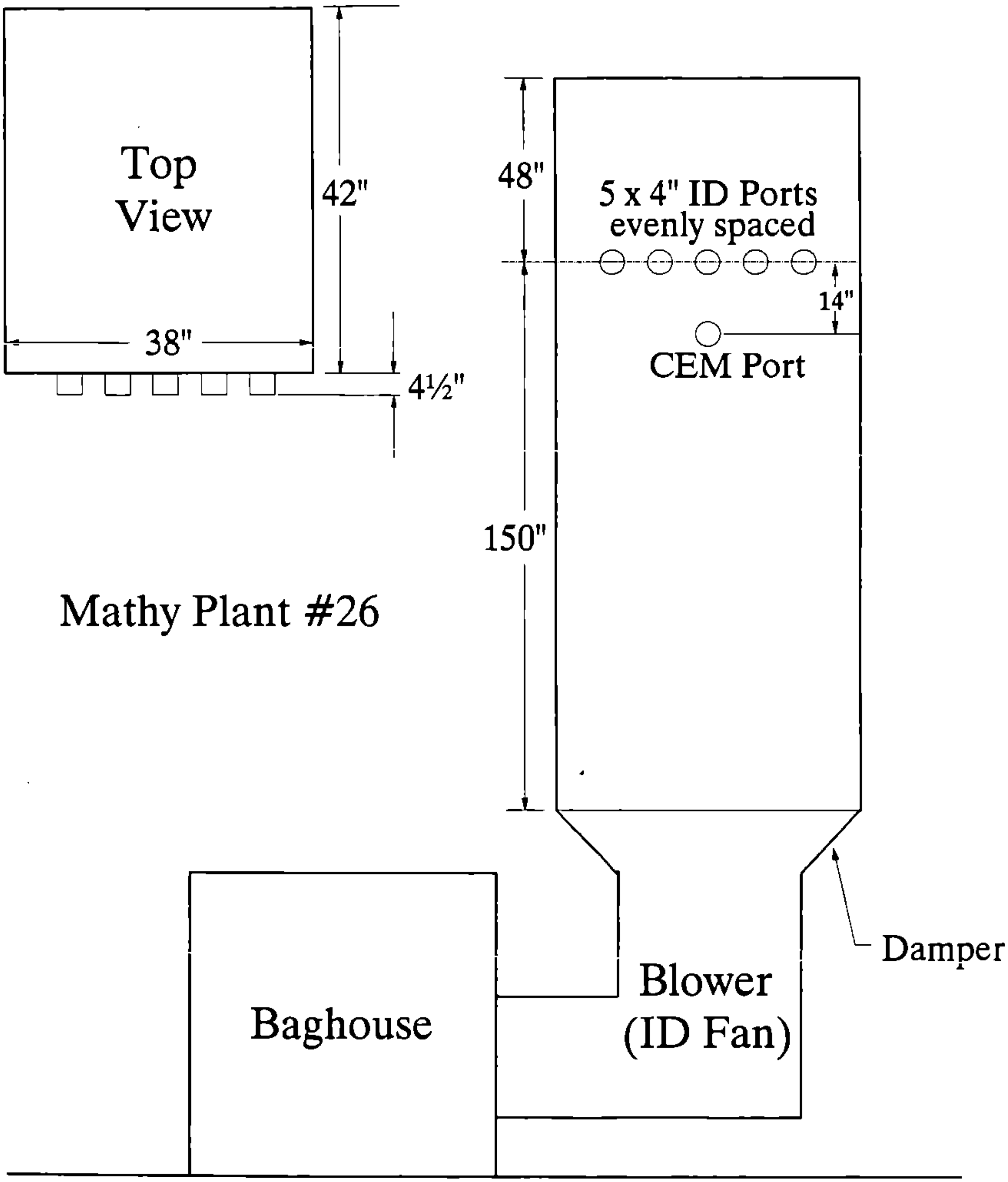
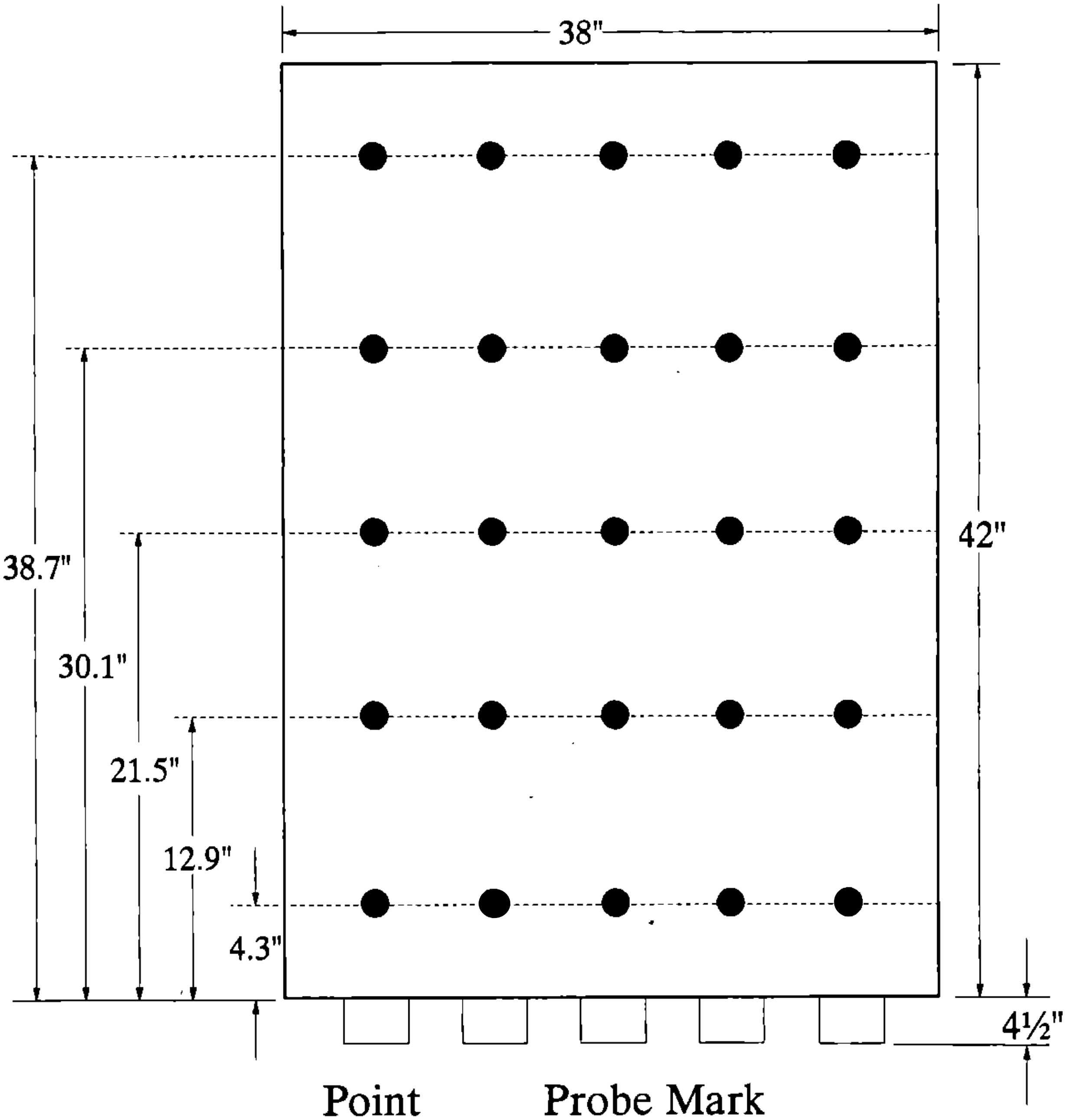


Figure 4-1. Sampling Location Arrangement.



Point	Probe Mark
1	8.8"
2	17.4"
3	26.0"
4	34.6"
5	43.2"

Figure 4-2. Traverse Point Layout at Stack.

5. SAMPLING AND ANALYTICAL PROCEDURES

The sampling and analytical procedures used for the asphalt plant test program were the most recent revisions of the published EPA methods or proposed EPA methods. In either case, state-of-the-art sampling and analytical methods were used. This section describes the sampling and analytical method used for each compound analyzed.

5.1 PARTICULATE MATTER AND METALS EMISSIONS TESTING METHOD

Sampling for particulate matter (PM) and metals was performed according to an EPA EMB draft protocol entitled "Methodology for the Determination of Metals Emissions in Exhaust Gases from Incineration Processes." The protocol is presented in Appendix J.1. This method is applicable for the determination of PM emissions and Pb, Ni, Zn, P, Cr, Cu, Mn, Se, Be, Tl, Ag, Sb, Ba, Cd, As, and Hg emissions from various types of processes. The test samples were not analyzed for Hg because Hg was not expected in the process stream. Particulate emissions were based on the weight gain of the filter and the front half acetone rinses of the probe, nozzle, and filter holder. After the gravimetric analyses were completed, the sample fractions were analyzed for the target metals as discussed in Section 5.2.5.

5.1.1 Sampling Equipment for Particulate Matter and Metals

This methodology used the sampling train shown in Figure 5-1. The sampling train consisted of a quartz nozzle/probe liner followed by a heated filter assembly with a Teflon® filter support, a series of five impingers, and the standard EPA Method 5 meterbox and vacuum pump. The sample was not exposed to any metal surfaces in this train. Two of the sequential impingers contained a 5 percent nitric acid (HNO_3)/10 percent hydrogen peroxide (H_2O_2) solution and one contained silica gel. The first and fourth impingers were empty knockout impingers not required by the method, but added because of the high moisture content of the flue gas. The second impinger containing HNO_3/H_2O_2 was of the Greenburg-Smith design; the other impingers had straight tubes. The impingers were connected together with clean glass

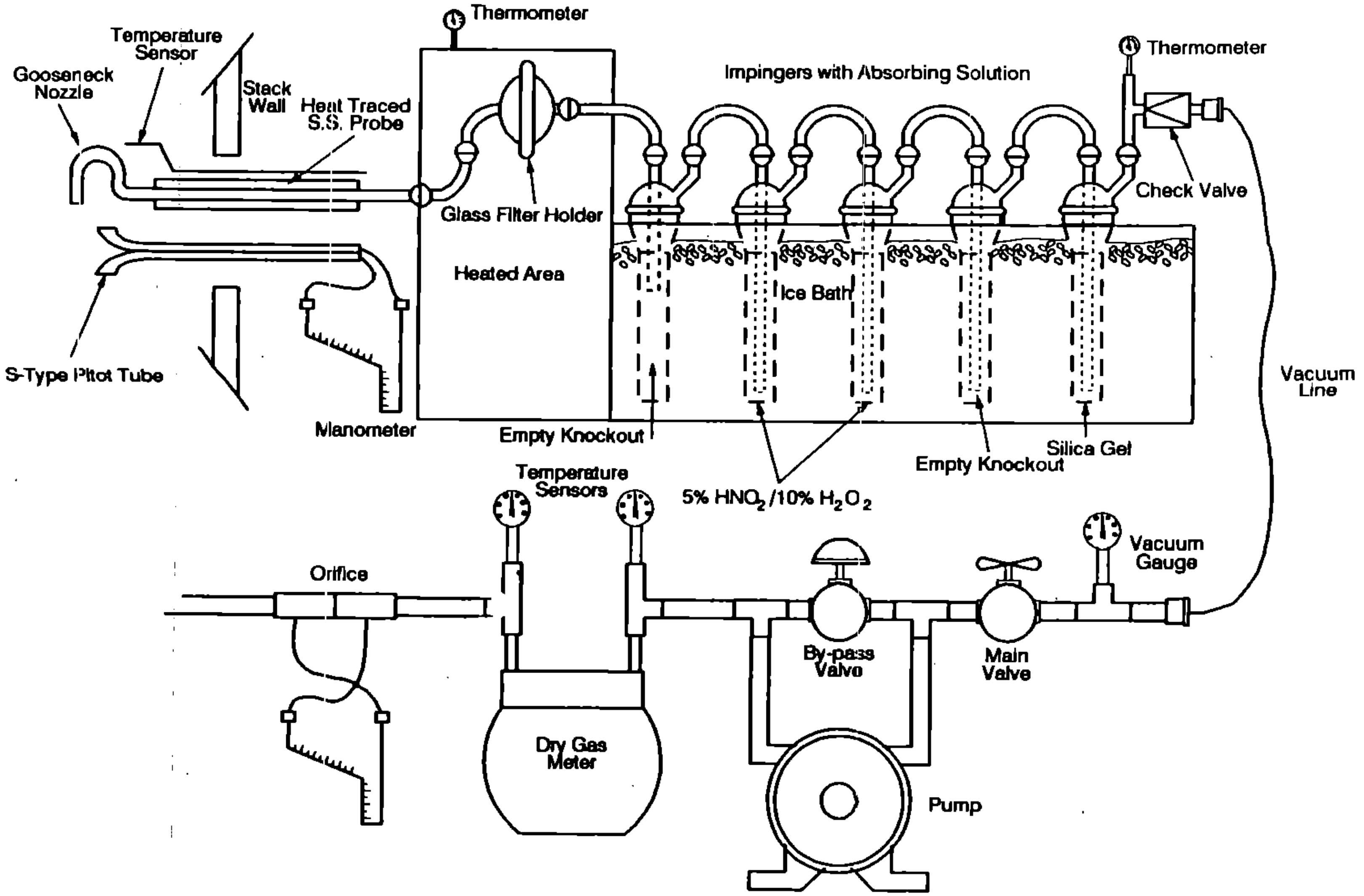


Figure 5-1. Schematic of Multiple Metals Sampling Train

U-tube connectors and were arranged in an impinger bucket. Sampling train components were recovered and analyzed in separate front and back half fractions according to the described method.

5.1.2 Equipment Preparation for Particulate Matter and Metals Sampling

5.1.2.1 Glassware Preparation. Glassware was washed in hot, soapy water, rinsed three times with tap water and then rinsed three times with deionized distilled water. The glassware was then subjected to the following series of soaks and rinses:

- Soaked in a 10 percent HNO_3 solution for a minimum of 4 hours;
- Rinsed three times with deionized distilled water rinse; and
- Rinsed with acetone rinse.

The cleaned glassware was allowed to air dry in a contamination-free environment. The ends were then covered with parafilm. All glass components of the sampling train plus any other sample bottles, pipes, Erlenmeyer flasks, petri dishes, graduated cylinders, and other laboratory glassware used during sample preparation, recovery, and analysis were cleaned according to this procedure.

5.1.2.2 Reagent Preparation. The sample train filters were Pallflex Tissuequartz 2500QAS filters. The acids and H_2O_2 were Baker "Instra-analyzed" grade or equivalent. The H_2O_2 was purchased specifically for this test site.

The reagent water was Baker "Analyzed HPLC" grade or equivalent. The lot number, manufacturer, and grade of each reagent that was used were recorded in the laboratory notebook.

The HNO_3/H_2O_2 absorbing solution was prepared fresh daily according to Section 4.2.1 of the reference method. The analyst wore both safety glasses and protective gloves when the reagents were mixed and handled. Each reagent had its own designated transfer and dilution glassware. To avoid contamination, this glassware was marked for identification with a felt tip glass-marking pen and used only for the reagent for which it was designated.

5.1.2.3 Equipment Preparation. The remaining preparation included calibration and leak checking of all the train equipment, which included meterboxes, thermocouples, nozzles, pitot tubes, and umbilicals. Referenced calibration procedures were followed when available, and the results were properly documented and retained. A discussion of the techniques used to calibrate this equipment is presented below.

Type-S Pitot Tube Calibration. The EPA has specified guidelines concerning the construction and geometry of an acceptable Type-S pitot tube. A pitot tube coefficient of 0.84 is used if the specified design and construction guidelines are met. Information pertaining to the design and construction of the Type-S pitot tube is presented in detail in Section 3.1.1 of EPA Document 600/4-77-027b. Only Type-S pitot tubes meeting the required EPA specifications were used. Pitot tubes were inspected and documented as meeting EPA specifications prior to field sampling.

Sampling Nozzle Calibration. Glass nozzles were used for isokinetic sampling. Calculation of the isokinetic sampling rate required that the cross sectional area of the sampling nozzle be accurately and precisely known. All nozzles were thoroughly cleaned, visually inspected, and calibrated according to the procedure outlined in Section 3.4.2 of EPA Document 600/4-77-027b.

Temperature Measuring Device Calibration. Accurate temperature measurements were required during source sampling. Thermocouple temperature sensors were calibrated using the procedure described in Section 3.4.2 of EPA document 600/4-77-027b. Each temperature sensor was calibrated at a minimum of two points over the anticipated range of use against an NBS-traceable mercury-in-glass thermometer. All sensors were calibrated prior to field sampling.

Dry Gas Meter Calibration. Dry gas meters (DGMs) were used in the sample trains to monitor the sampling rate and to measure the sample volume. All DGMs were calibrated to document the volume correction factor just before the equipment was shipped to the field. Post-test calibration checks were performed as soon as possible after the equipment has been returned to Research Triangle Park, North Carolina (RTP). Pre- and post-test calibrations agreed to within 5 percent. Prior to calibration, a positive pressure leak check of the system was performed using the procedure outlined in Section 3.3.2 of EPA document 600/4-77-237b. The system was placed under approximately 10 inches of water pressure and a gauge oil manometer was used to determine if a pressure decrease was detected over a 1-minute period. If leaks were detected, they were eliminated before actual calibrations were performed.

After the sampling console was assembled and leak checked, the pump was allowed to run for 15 minutes. This allowed the pump and DGM to warm up. The valve was then adjusted to obtain the desired flow rate. For the pretest calibrations, data were collected at orifice manometer settings (ΔH) of 0.5, 1.0, 1.5, 2.0, 3.0 and 4.0 inches of H_2O. Gas volumes of 5 ft^3 were used for the two lower orifice settings, and volumes of 10 ft^3 were used for the higher settings. The individual gas meter correction factors were calculated for each orifice setting and averaged. The method required that each of the individual correction factors fall within ±2 percent of the average correction factor or the meter was cleaned, adjusted, and recalibrated. In addition, Radian required that the average correction factor be within 1.00 ±1 percent. For the post-test calibration, the meter was calibrated three times at the average orifice setting and vacuum used during the actual test.

Rockwell Model 175 DGMs in Research Appliance Company (RAC) enclosures were used for measuring gas sampling rates. The DGM calibrations were performed at Radian's RTP laboratory using an American wet test meter as an intermediate standard. The intermediate standard is calibrated every six months against the EPA spirometer at EPA's Emissions Measurement Laboratory in RTP.

5.1.3 Particulate Matter/Metals Sampling Operations

5.1.3.1 Preliminary Measurements. Before sampling began, preliminary measurements were required to ensure isokinetic sampling. These included determining the traverse point locations and performing a preliminary velocity traverse, cyclonic flow check, and moisture determination. These measurements were used to calculate a "K factor." The K factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements made during the pretest site survey were then checked for accuracy. Measurements were made of the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances. These measurements were used to verify sampling point locations by following EPA Reference

Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

5.1.3.2 Assembling the Train. Assembling the PM/metals sampling train components was initiated in the recovery trailer and final train assembly was completed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground-glass joint was carefully inspected for hairline cracks. The first impinger was a knockout impinger with a short tip. The purpose of this impinger was to collect condensate. The next two impingers were modified tip impingers, which each contained 100 ml of 5 percent HNO_2 and 10 percent H_2O_2. The fourth impinger was empty, and the fifth impinger contained 200 to 300 grams of blue-indicating silica gel. After the impingers were loaded, each impinger was weighed, and the initial weight and contents of each impinger were recorded on a recovery data sheet. The impingers were connected together by clean glass U-tube connectors and arranged in the impinger bucket. The height of all the impingers was approximately the same to obtain a leak free seal. The open ends of the train were sealed with parafilm or teflon tape.

The second step was to load the filter into the filter holder in the recovery trailer. The filter holder was then capped off and placed into the impinger bucket. A supply of parafilm and socket joints was also placed in the bucket in a clean plastic bag for use by the samplers. To avoid contamination of the sample, sealing greases were not used. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-1.

5.1.3.3 Sampling Procedures. After the train was assembled, the heaters for the probe liner and heated filter box were turned on. When the system reached the appropriate temperatures, the sampling train was ready for pretest leak checking. The filter skin temperature was maintained at 120 ±14°F (248 ±25°F). The probe temperature was maintained above 100°C (212°F).

The sampling trains were leak checked at the start and finish of sampling. (EPA Method 5 protocol required post-test leak checks and recommended pretest leak checks.) Radian protocol also incorporated leak checks before and after every port change. An acceptable pretest leak rate was less than 0.02 acfm (ft^3/min) at approximately 15 inches of mercury (in. Hg). If, during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed, and after the train was reassembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

After a successful pretest leak check had been conducted, all train components were at their specified temperatures, and initial data were recorded (DGM reading), the test was initiated. Sampling train data were recorded periodically on standard data forms. A checklist for sampling is included in Table 5-1.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occurred during sampling were recorded on the task log such as pitot cleaning, thermocouple malfunctions, heater malfunctions, or any other unusual occurrences.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. (The post-test leak check procedure is identical to the pretest procedure; however, the vacuum should be at least 1 in. Hg higher than the highest vacuum attained during sampling.) An acceptable leak rate was less than 4 percent of the average sample rate, or 0.02 acfm (whichever was lower). If a final leak rate did not meet the acceptable criterion, the test run could still have been accepted upon approval of the test administrator.

5.1.4 Particulate Matter/Metals Sample Recovery

Recovery procedures began as soon as the probe was removed from the stack and the post-test leak check was completed.

Table 5-1

Sampling Checklist

Before Test Starts:

1. Check impinger set for correct order and number. Verify probe markings, and re-mark if necessary.
2. Verify that you have all the correct pieces of glassware.
3. Get data sheets and read barometric pressure.
4. Bag sampling equipment needs to be ready (with bags labeled and ready to go) if applicable.
5. Examine meter box; level it and confirm that the pump is operational.
6. Assemble train to the filter and leak check at 15 in Hg. Attach probe to train and do final leak check; record leak rate and pressure on sampling log.
7. Check out thermocouples; make sure they are reading correctly.
8. Turn on all heaters and check to see that they are increasing.
9. Leak check pitots.
10. Check that cooling water is flowing and on. Add ice to impinger buckets.
11. Check isokinetic K-factor; make sure it is correct. (Refer to previous results to confirm assumptions). (Two people should calculate this independently to double check it.)
12. Have a spare probe liner, probe sheath, meter box and filter ready to go at location.

Table 5-1

Continued

During Test:

1. Notify crew chief of any sampling problems immediately. Note problem on sampling log.
2. Perform simultaneous/concurrent testing with other locations (if applicable). Maintain filter temperature between 248°F ±25°F. Keep temperature as steady as possible. Maintain XAD trap and impinger temperatures below 68°F. Maintain probe temperature above 212°F.
3. Leak check between ports and record on sampling log.
4. Record sampling rate times and location for the fixed gas (CO, CO_2, O_2) sample (if applicable).
5. Blow back pitot tubes at inlet location every 15 minutes.
6. Change filter if pressure drop exceeds 15 in. Hg.
7. Check impinger solutions every 1/2 hr; if bubbling into impinger prior to silica gel, empty out first impinger into pre-weighed bottle and replace.
8. Check impinger silica gel every 1/2 hr; if indicator disappears request a pre-filled impinger from van lab and replace.
9. Check manometer fluid levels and zero every hour.

After Test Is Completed:

1. Record final meter reading.
2. Check completeness of data sheet.
3. Do final leak check of sampling train at 1 in Hg greater than maximum vacuum during test.

Table 5-1

Continued

4. Leak check each leg of pitot tubes.

5. Disassemble train. Cap sections. Take sections to recovery trailer.

6. Probe recovery (use 950 ml bottles)
    a) Bring probes into recovery trailer (or other enclosed area).
    b) For acetone rinses (all trains)
        - Attach flask to end of probe
        - Add about 50 mls of acetone
        - Put in brush down probe, and brush back and forth
        - Rinse back and forth in probe
        - Empty out acetone in sample jar
        - Do this 3 times
    c) For $MeCl_2$ rinses
        Rinse 3 times with flask attached (no brushing)

7. Reattach nozzle and cap for next day, store in dry safe place.

8. Make sure data sheets are completely filled out and give to location leader.

To facilitate its transfer from the sampling location to the recovery trailer, the sampling train was disassembled into three sections: the nozzle/probe liner, filter holder, and impingers in their bucket. Each of these sections was capped with Teflon® tape or parafilm before being transported to the recovery trailer.

Once in the trailers, the sampling train was recovered as separate front and back half fractions. Figure 5-2 is a diagram illustrating front half and back half sample recovery procedures. No equipment with exposed metal surfaces was used in the sample recovery procedures. The weight gain in each of the impingers was recorded to determine the moisture content in the flue gas. Following weighing of the impingers, the front half of the train was recovered, which included the filter and all sample-exposed surfaces forward of the filter. The probe liner was rinsed with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces were wetted. The acetone was quantitatively collected into the appropriate sample bottle. This rinse was followed by additional brush/rinse procedures using a nonmetallic brush; the probe was held in an inclined position and acetone was squirted into the upper end as the brush was pushed through with a twisting action. All of the acetone and particulate was caught in the sample container. This procedure was repeated until no visible particulate remained and was finished with a final acetone rinse of the probe and brush. The front half of the filter was also rinsed with acetone until all visible particulate was removed. After all front half acetone washes were collected, the cap was tightened, the liquid level marked and the bottle weighed to determine the acetone rinse volume. The method specifies that a total of 100 ml of acetone must be used for rinsing these components. However, a thorough rinse usually requires more reagent. For blank correction purposes, the exact weight or volume of acetone used was measured. An acetone reagent blank of approximately the same volume as the acetone rinses was analyzed with the samples.

The nozzle/probe liner, and front half of the filter holder was rinsed three times with 0.1N HNO_3 and the rinse was placed into a separate amber bottle. The container wascapped tightly, the weight of the combined rinse was recorded, and the liquid level

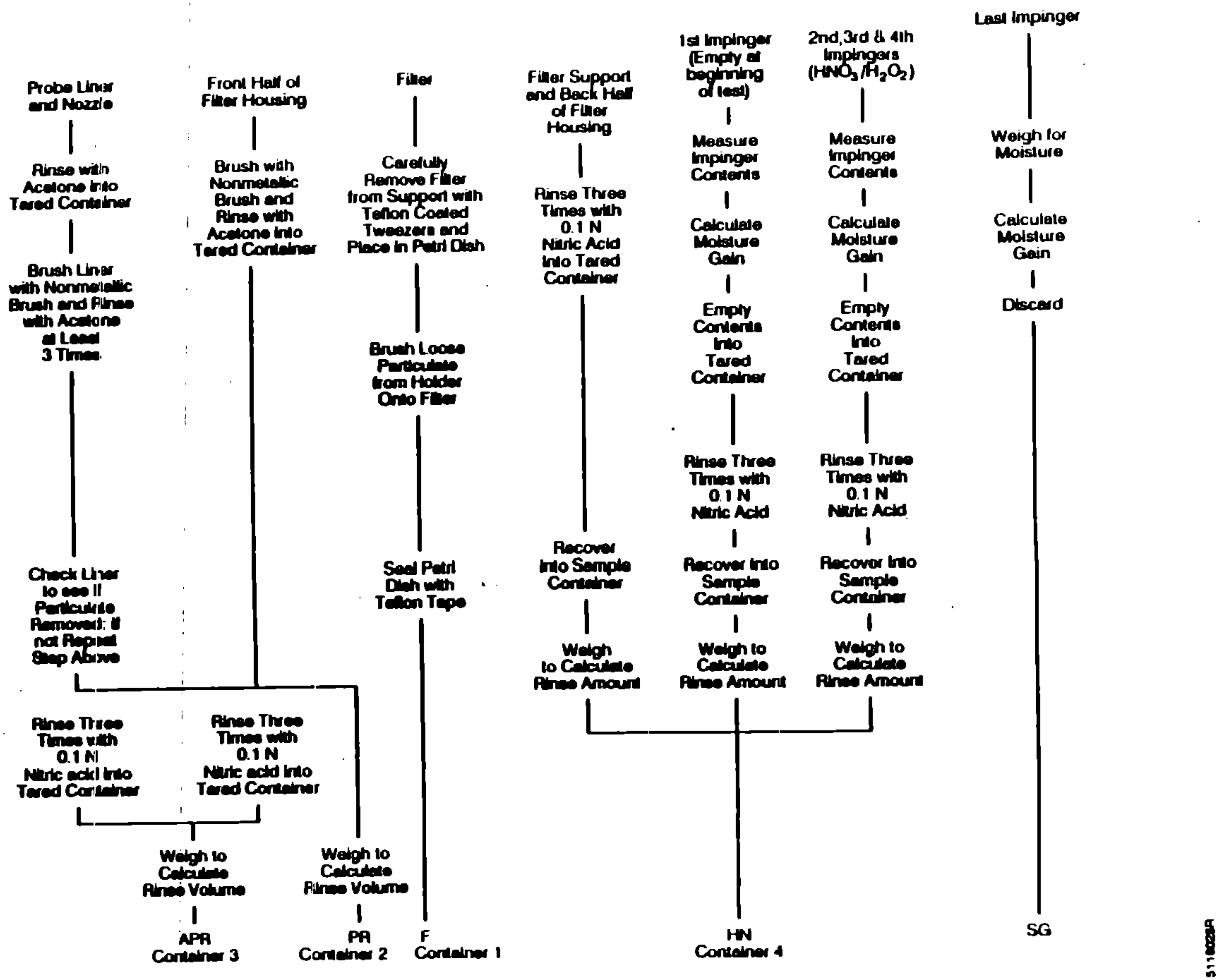


Figure 5-2. Metals Sample Recovery Scheme

was marked on the bottle. The filter was placed in a clean, well-marked glass petri dish and sealed with Teflon® tape.

Prior to recovering the back half impingers, the contents were weighed for moisture content. Any unusual appearance of the filter or impinger contents was noted in the logbook.

The contents in the knockout impinger was recovered into a preweighed, prelabeled bottle with the contents from the HNO_3/H_2O_2 impingers. These impingers and connecting glassware were rinsed thoroughly with 0.1N HNO_3, the rinse was captured in the impinger contents bottle, and a final weight was taken. Again, the method specifies a total of 100 ml of 0.1N HNO_3 be used to rinse these components. The weight of reagent used for rinsing was determined by weighing the impinger contents bottle before and after rinsing the glassware. A nitric acid reagent blank of approximately the same volume as the rinse volume was analyzed with the samples.

After final weighing, the silica gel from the train was saved for regeneration. The ground-glass fittings on the silica gel impinger were wiped off after sample recovery to ensure a leak tight fit for the next test.

A reagent blank was recovered in the field for each of the following reagents:

- Acetone blank--100-ml sample size;
- 0.1N HNO_3 blank--1000-ml sample size;
- 5 percent HNO_3/10 percent H_2O_2 blank--200-ml sample size;
- Dilution water--100-ml sample size; and
- Filter blank--one each.

Each reagent blank was from the same lot used during the sampling program. Each lot number and reagent grade was recorded on the field blank label and in the logbook.

The liquid level of each sample container was marked on the bottle in order to determine if any sample loss occurred during shipment. If sample loss had occurred, the sample might have been voided or a method could have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

Approximate detection limits for the various metals of interest are summarized in Table 5-2.

5.1.5 Particulate Analysis

The general gravimetric procedure described in EPA Method 5, Section 4.3, was followed. Both filters and precleaned beakers were weighed to a constant weight before use in the field. The same balance used for taring was used for weighing the samples.

The acetone rinses were evaporated under a clean hood at 70°F to dryness in a tared beaker. The residue was desiccated for 24 hours in a desiccator containing fresh room temperature silica gel. The filter was also desiccated to a constant weight under the same conditions. Weight gain was reported to the nearest 0.1 mg. Each replicate weighing agreed to within 0.5 mg or 1 percent of total weight less tare weight, whichever was greater, between two consecutive weighings, and was at least 6 hours apart.

5.1.6 Metals Analytical Procedures

A diagram illustrating the sample preparation and analytical procedure for the target metals is shown in Figure 5-3.

The front half acetone and filter fractions were digested with concentrated HNO_3 and hydrofluoric acid (HF) in a microwave pressure vessel. The microwave digestion took place over a period of approximately 10 to 12 minutes in intervals of 1 to 2 minutes at 600 watts. The nitric probe rinse was digested by EPA SW 846 Method 3020. The digested filters and the digested probe rinses were combined to yield the front half sample fraction. The fraction was diluted to a specific volume with DI water and analyzed by applicable instrumentation.

The absorbing solutions from the HNO_3/H_2O_2 impingers were combined, acidified, and reduced to near dryness. The sample was then digested by conventional digestion, with 5 percent HNO_3. After the fraction has cooled, it was filtered and diluted to a specified volume with DI water.

Each sample fraction was analyzed by inductively coupled argon plasma spectroscopy (ICAP) using EPA Method 200.7. Interelement corrections were applied to

TABLE 5-2. APPROXIMATE DETECTION LIMITS

Metal	Method[a]	Analytical Detection Limits (μg/ml)	Instack Method Detection Limits[b] Front Half (300 ml sample size) (μg/m³)	Back Half (150 ml sample size) (μg/m³)
Chromium	ICAP	0.006	1.4	0.7
Cadmium	ICAP	0.002	0.5	0.2
Arsenic[c]	GFAAS	0.004	1.0	0.5
Lead[c]	GFAAS	0.003	0.7	0.4
Nickel	ICAP	0.003	0.7	0.4
Barium	ICAP	0.001	0.2	0.1
Beryllium	ICAP	0.0001	0.2	0.1
Silver	ICAP	0.006	1.4	0.7
Antimony	ICAP	0.015	3.6	1.8
Thallium	ICAP	0.100	24	12
Zinc	ICAP	0.015	3.6	1.8
Copper	ICAP	0.004	1.0	0.5
Manganese	ICAP	0.003	0.7	0.4
Phosphorus	ICAP	0.300	72	36
Selenium	ICAP	0.005	1.2	0.6

[a] ICAP = Inductively Coupled Argon Plasma
GFAAS = Graphite Furnace Atomic Absorption Spectroscopy
CVAAS = Cold Vapor Atomic Absorption Spectroscopy

[b] These detection limits are based on a stack gas sample volume of 1.25 m³. If 5 m³ are collected, the instack detection method detection limits are 1/4 of the values indicated.

[c] If Fe and Al are present, samples will be diluted which may raise analytical detection limits.

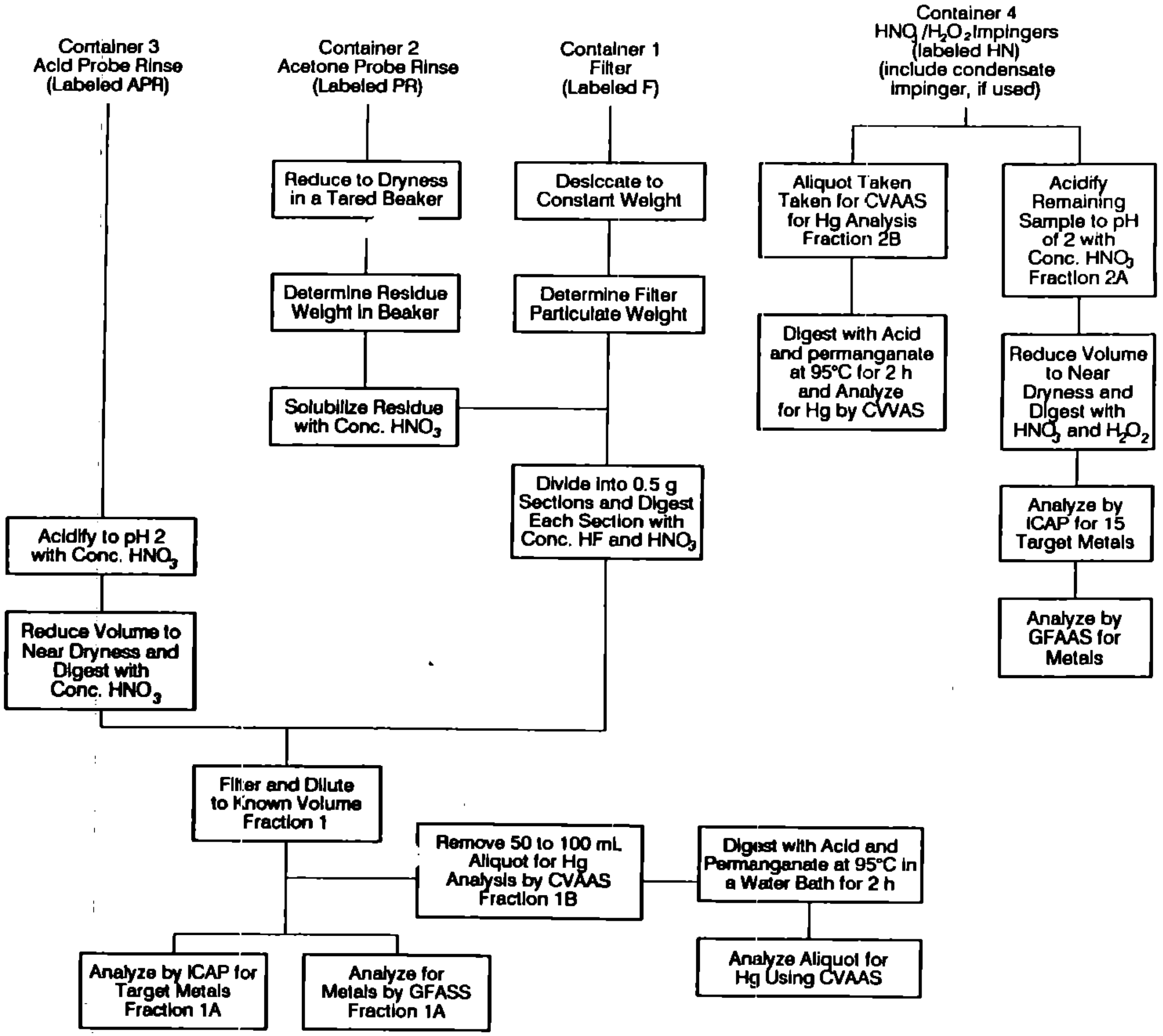


Figure 5-3. Metals Sample Preparation and Analysis Scheme

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the analytes to remove the effects of unwanted emissions. If arsenic or lead levels were less than 2 ppm, graphite furnace atomic absorption spectroscopy (GFAAS) was used to analyze for these elements by EPA Methods 7060 and 7421. Matrix modifiers such as specific buffering agents were added to these aliquots to make the matrix more volatile and/or stabilize the analyte element. The total volumes of the absorbing solutions and rinses for the various fractions were measured and recorded in the laboratory notebook.

5.1.7 Quality Control for Metals Analytical Procedures

All quality control (QC) procedures specified in the test method were followed. All field reagent blanks were processed, digested and analyzed as specified in the test method. To ensure optimum sensitivity in measurements, the concentrations of target metals in the solutions were at least 10 times the analytical detection limits.

5.1.7.1 Inductively Coupled Argon Plasma Spectroscopy Standards and Quality Control Samples. The QC procedures used for ICAP analysis include running two QC standards, and a calibration blank after every 10 samples. One interference check standard was analyzed at the beginning and the end of the analytical run. One duplicate analysis and one analytical spike were analyzed to check for precision and matrix effects.

Standards less than 1 μg/ml of a metal were prepared daily; those with concentrations greater than this were made monthly.

5.1.7.2 Graphite Furnace Standards and Quality Control Samples. Standards used for GFAAS analysis were matrix matched with the samples analyzed and the matrix modifiers added. Standards with less than 1 μg/ml of a metal were prepared daily; those with concentrations greater than this were made monthly. A minimum of five standards composed the standard curve. Quality control samples were prepared from a separate 10 μg/ml standard by diluting it into the range of the samples.

One analytical spike was analyzed for every 10 samples. If recoveries were below 80 percent of 100, the samples were analyzed by method of additions as explained in EPA SW 846 Method 7000. One QC sample was analyzed to verify the standard curve used to quanitate the samples.

5.2 EMISSIONS TESTING FOR PARTICULATE MATTER LESS THAN 10 MICRONS/CONDENSIBLE PARTICULATE MATTER

The sampling method for fine particulate matter/condensible particulate matter (PM_{10}/CPM) was a combination of the protocols outlined in EPA Method 201A [entitled "Determination of PM_{10} Emissions (Constant Sampling Rate Procedure)"] and EPA Method 202 (entitled "Determination of Condensible Emissions from Stationary Sources"). These methods are presented in Appendix J.2, and are summarized below. Method 201A is applicable to the measurement of PM emissions with aerodynamic diameters less than or equal to 10 microns (PM_{10}). Method 202 applies to the determination of CPM from various types of combustion devices. Condensible PM emissions are gaseous matter and aerosols that condense after passing through a filter that captures liquid and solid particulates. Analyses of the test samples were performed for total PM (including PM greater than 10 μm), PM_{10}, and CPM. Total PM emission rates were determined from the PM/metals train.

Particulate matter emissions larger than 10 microns were determined by measuring the weight of the catch of an in-stack PM_{10} cyclone. The PM_{10} emissions were determined from the weight gain of an in-stack backup filter that was downstream of the cyclone. The CPM emissions were determined from the evaporated residue of the impinger solution, as outlined in Section 5.2.5.2

5.2.1 Sampling Equipment for PM_{10}/CPM

Figure 5-4 shows the sampling train for the PM_{10}/CPM method, which combined the in-stack cyclone, filter assembly, and probe from EPA Method 201A with the impinger assembly from EPA Method 202. The sample train consisted of a tapered stainless steel inlet nozzle, an in-stack PM_{10} cyclone, a backup filter holder and filter behind the cyclone, a heated glass probe liner, a series of 4 impingers, and the standard EPA Method 17 meterbox and vacuum pump.

The instrument used in PM_{10} determination was a Sierra Instruments Series 280 Cyclade™ cyclone. This device collected particulates larger than 10 microns and allowed particulates smaller than 10 microns to pass through to a backup filter. The cyclone

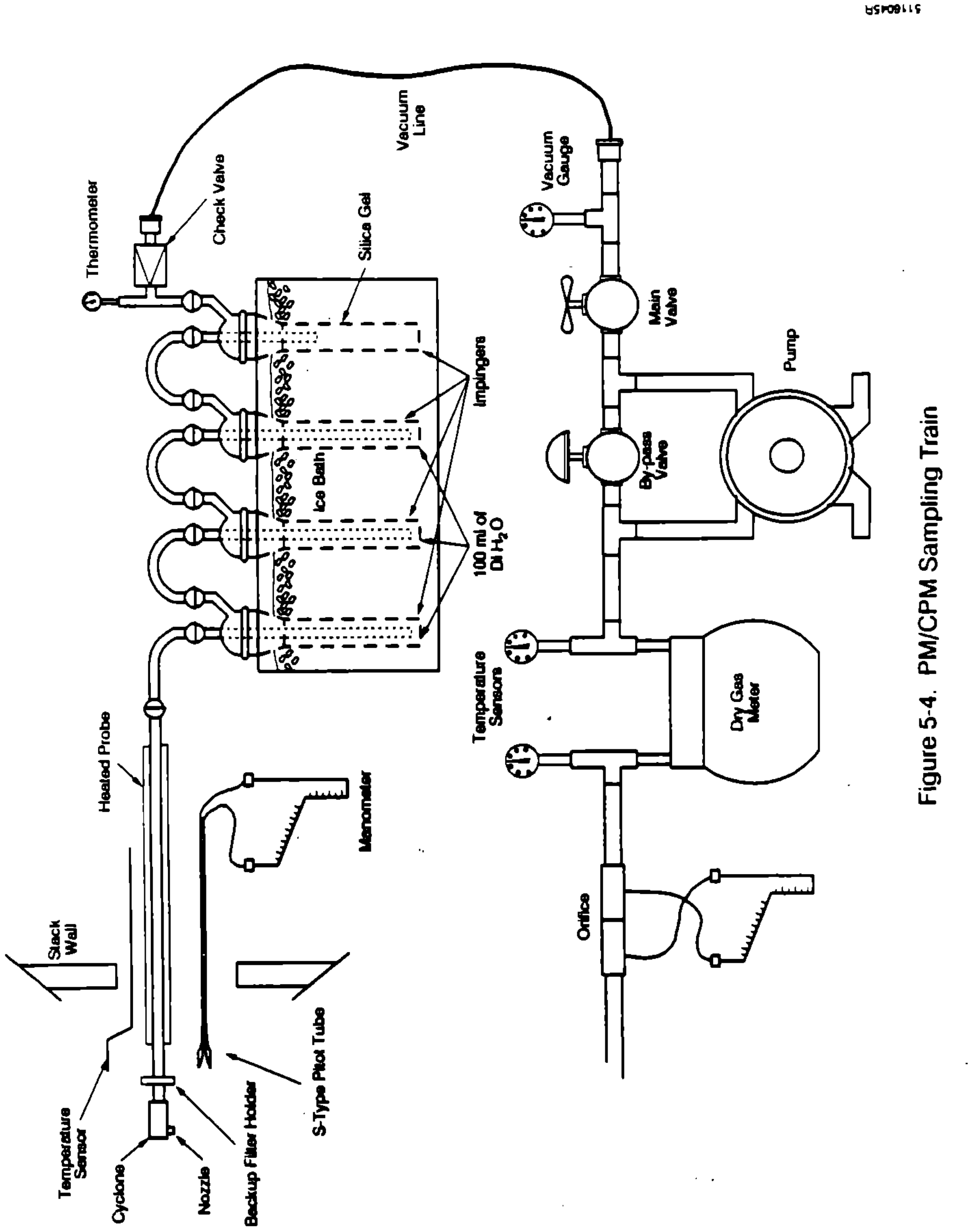

Figure 5-4. PM/CPM Sampling Train

caused the gas stream to swirl in a vortex; larger particulates contacted the cyclone wall and fell into a collection cup.

The in-stack backup filter used after the cyclone had a demonstrated collection efficiency of greater than 99.95 percent on diocytylphepthalate (DOP) smoke particles, as required by ASTM Standard Method D 2986.

As outlined in EPA Method 202, the first two impingers each contained 100 ml of deionized distilled H_20, the third impinger was empty, and the fourth contained silica gel. The first two impingers were of the Greenburg-Smith design with standard tips; the other impingers had straight tubes. The impingers were connected together with clean glass U-tube connectors.

5.2.2 PM_{10}/CPM Sampling Equipment Preparation

5.2.2.1 Glassware Preparation. Glassware was washed as follows:

- Washed in hot soapy water;
- Rinsed with tap water;
- Rinsed with deionized distilled water;
- Rinsed with acetone; and
- Rinsed with methylene chloride ($MeCl_2$).

The cleaned glassware was allowed to air dry in a contamination-free environment. After drying, the ends were covered with parafilm. All glass components of the sampling train plus any sample bottles, pipets, Erlenmeyer flasks, petri dishes, graduated cylinders, and other laboratory glassware used during sample preparation, recovery, and analysis were cleaned according to this procedure.

The cyclone housing, nozzle, and interior surfaces were cleaned with hot, soapy water, rinsed with hot tap water, rinsed with distilled deionized water, and finally rinsed with acetone and dried.

5.2.2.2 Reagent Preparation. The deionized distilled reagent water used conformed to the American Society for Testing and Materials Specification D 1193-74, Type II.

5.2.2.3 Equipment Preparation. All measuring devices used during sampling were calibrated prior to use, as specified in EPA Method 17. This equipment included top loading scales, probe nozzles, pitot tubes, metering system, probe heater, temperature gauges, dry gas metering system, and barometer. A laboratory field notebook was maintained to record these calibration values.

Before they were used, all filters were desiccated and tared on a five-place balance. Replicate weighings at least 6 hours apart must agree to within 0.5 mg to yield an acceptable weight. Each filter was then stored in an individual petri dish with an identification number, and all data were recorded in the logbook.

5.2.3 Sampling Operations for PM_{10}/CPM

The sampling procedure for the PM_{10}/CPM method is similar to the procedure for EPA Method 5, except that the PM_{10}/CPM method includes a post-test nitrogen (N_2) purge to purge SO_2 from the sample, if considerable amounts of SO_2 are present in the flue gas. Also, a different method was used for nozzle size selection and sampling time, and no silicone grease was used in assembling the sample train in order to avoid contamination.

Prior to sampling for PM_{10}, a preliminary velocity traverse was performed. Moisture content, flue gas molecular weight, and temperature were determined using EPA Methods 1 through 4. These data were used to determine the appropriate sampling rate (as outlined in EPA Method 201A) through the cyclone and to select an appropriate sampling nozzle or nozzles. Since a constant sampling rate was required throughout a given run, more than one nozzle was required to maintain approximate isokinetic sampling conditions. In preparation for sampling, the tester calculated an appropriate nozzle size for each anticipated range of pitot readings (delta P), such that isokinetics could be maintained within ±20 percent of the constant sampling rate.

In addition to the above mentioned preliminary data, particulate loading was also known in order to calculate the required run duration to achieve a representative sample in the cyclone. Because of the complexity of the PM_{10} method, only experienced samplers performed the sample tests.

The impinger train was prepared according to EPA Method 5. Teflon tape was used to provide leak-free connections between glassware. The impingers and impinger contents were weighed and the weights recorded. The sample train components were carefully assembled in the recovery trailer except for attachment of the cyclone, backup filter, and probe, which was performed at the stack sampling location.

The train was assembled at the stack location by connecting the cyclone, filter, and probe liner to the impinger train, which was connected to the meterbox. After the probe and filter heaters were turned on, the train was leak checked at 15 in. Hg. The leak rate must be below 0.02 cfm.

The samples were withdrawn at a constant flow rate from the stack at the traverse points determined by EPA Method 1. The sampling time at each point was based on the relative gas velocity at that point. A leak check was performed before and after each sample test. Parafilm or Teflon tape was used to seal the train components at the end of each test. As soon as possible after the post-test leak check, the probe was disconnected from the impinger train.

5.2.4 Sample Recovery for PM_{10}/CPM

Recovery procedures began as soon as the probe was removed from the stack at the end of the sampling period. The recovery scheme is shown in Figure 5-5. To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into four sections: the cyclone, the filter holder, the nozzle/probe liner, and the impingers in their bucket. Each of these sections was capped with parafilm or Teflon tape before being transported to the recovery trailer.

5.2.4.1 Cyclone Recovery. The cyclone was disassembled and the nozzle removed. The PM was quantitatively recovered from the interior surfaces of the nozzle, cyclone, and collection cup (excluding the exit tube) by brushing with a nylon bristle brush and rinsing with acetone until the rinse showed no visible particles. After this procedure, a final rinse of the cyclone surfaces and brush was performed. All particulate and acetone rinse was collected in a sample jar and sealed. The liquid level was marked,

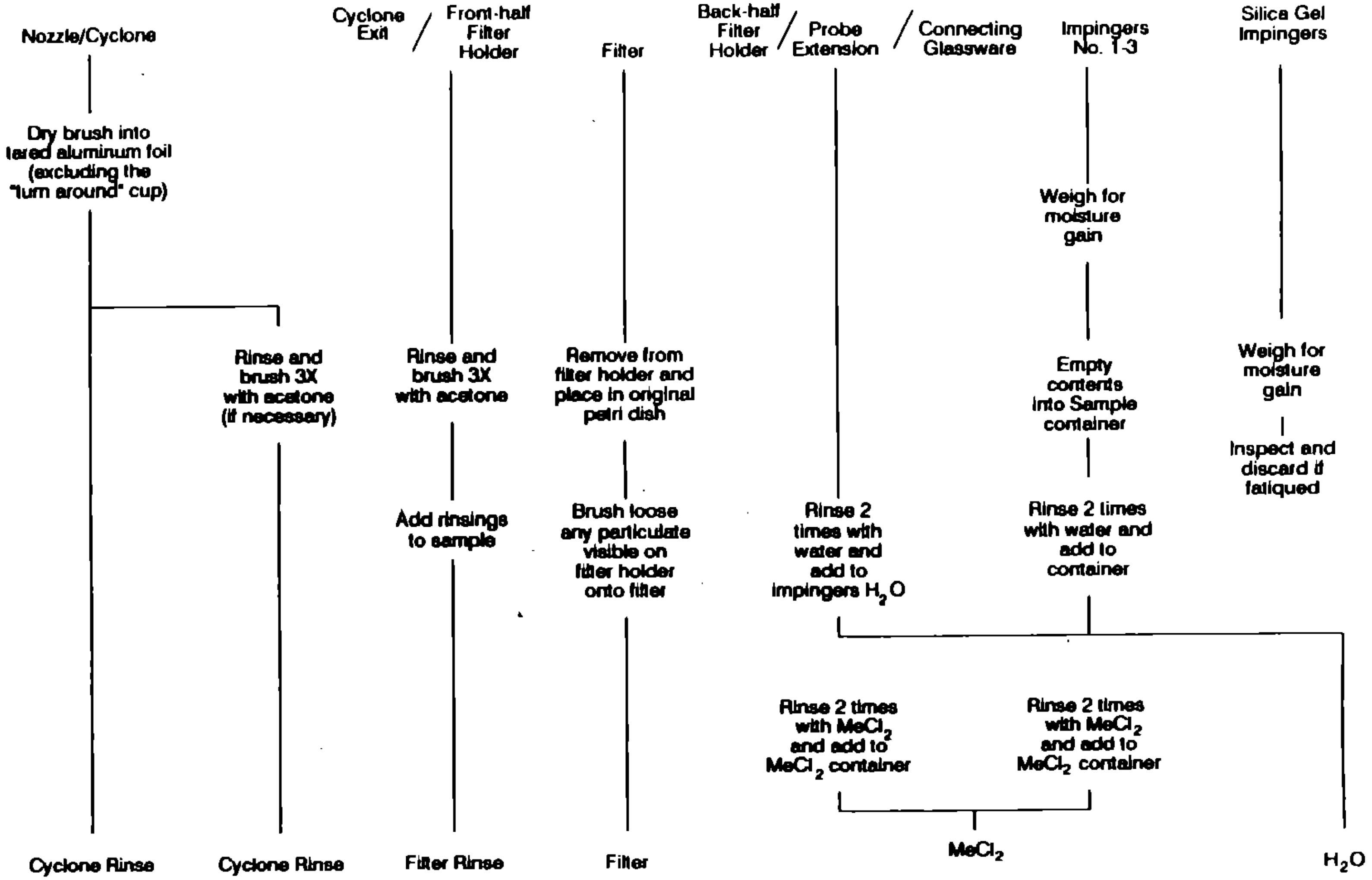

Figure 5-5. PM_{10}/CPM Sample Recovery Scheme

and the jar was identified. This information was logged into the field notebook.

The above procedure was repeated for all interior surfaces from the exit tube to the front half of the in-stack filter. The acetone rinse was collected in a separate sample jar, sealed, identified, the liquid level was marked, and the sample information was logged into the field notebook.

5.2.4.2 -In-stack Filter Recovery. The in-stack filter holder was opened and the filter was removed with tweezers or rubber gloves. The filter was placed in a marked petri dish sealed with Teflon tape, and the filter number was logged into the field notebook.

5.2.4.3 Probe and Impingers Recovery. The weight or volume gain in each of the impingers was recorded and used to determine the moisture content in the flue gas. The liquid from the three impingers was transferred into a clean glass sample jar. The impinger bottles, back half of the filter holders, and probe liner were rinsed twice with water, the rinse water was added to the same sample bottle, and the liquid level was marked on the bottle.

Following the water rinses, the impingers, filter holder, and probe were rinsed twice with $MeCl_2$. The $MeCl_2$ rinse was saved in a clean glass sample jar and the liquid level was marked. The sample information was logged into the field notebook.

All sample jars were fully identified and sealed. Pertinent information was logged into the field notebook.

5.2.4.4 Field Blanks. Field blanks of water (500 ml), $MeCl_2$ (a volume approximately equal to the volume used for the $MeCl_2$ rinses), and acetone (200 ml) were taken. Each reagent blank was of the same lot as was used during the sampling program. Each lot number and reagent grade were recorded on the field blank label and recorded into the field notebook.

5.2.5 Analysis for PM_{10}/CPM

The PM_{10}/CPM gravimetric analyses were completed as shown in Figure 5-6. Sample jars were checked to ascertain if leakage during shipment had occurred. If sample loss occurred during shipment, the sample may have been voided or a method may have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

5.2.5.1 Cyclone Catch Analysis. The dry cyclone catch, stored in foil; the cyclone rinse; and the front half filter rinses were analyzed gravimetrically according to EPA Method 5. Each rinse was evaporated at 70°F in a tared beaker to dryness. The residue was then desiccated at room temperature for 24 hours to a constant weight in a desiccator containing anhydrous calcium sulfate. To be considered constant weight, each replicate weighing must agree to within 0.5 mg and must be at least 6 hours apart. Weight gain for each fraction was reported to the nearest 0.1 mg. This weight gain constituted the PM greater than 10 microns in size.

5.2.5.2 Filter Catch Analysis. The in-stack filter catch was analyzed gravimetrically according to EPA Method 5 requirements.

For each filter, the filter and loose particulates were transferred to a tared glass weighing dish and dried for 24 hours in a desiccator containing silica gel. The sample was weighed to a constant weight, with results reported to the nearest 0.1 mg. The resulting weight gain from the filter and exit tube acetone rinses constituted the noncondensible PM_{10} portion of the sample.

5.2.5.3 Impinger and Probe Sample Rinse Analysis. Data were recorded on the data sheet shown in Figure 5-7. The water sample was measured volumetrically.

The $MeCl_2$ sample was combined with the water sample in a 1000-ml separatory funnel. After mixing, the aqueous and organic phases were allowed to separate; most of the organic/$MeCl_2$ phase was drained off and collected in a tared 350-ml weighing tin (approximately 100 ml). Then 75 ml of $MeCl_2$ was added and mixed; again most of the organic $MeCl_2$ was drained into the weighing tin. This procedure was repeated with another 75 ml of $MeCl_2$. A total of approximately 250 ml of organic extract was drained into the weighing tin. No water was drained during this procedure.

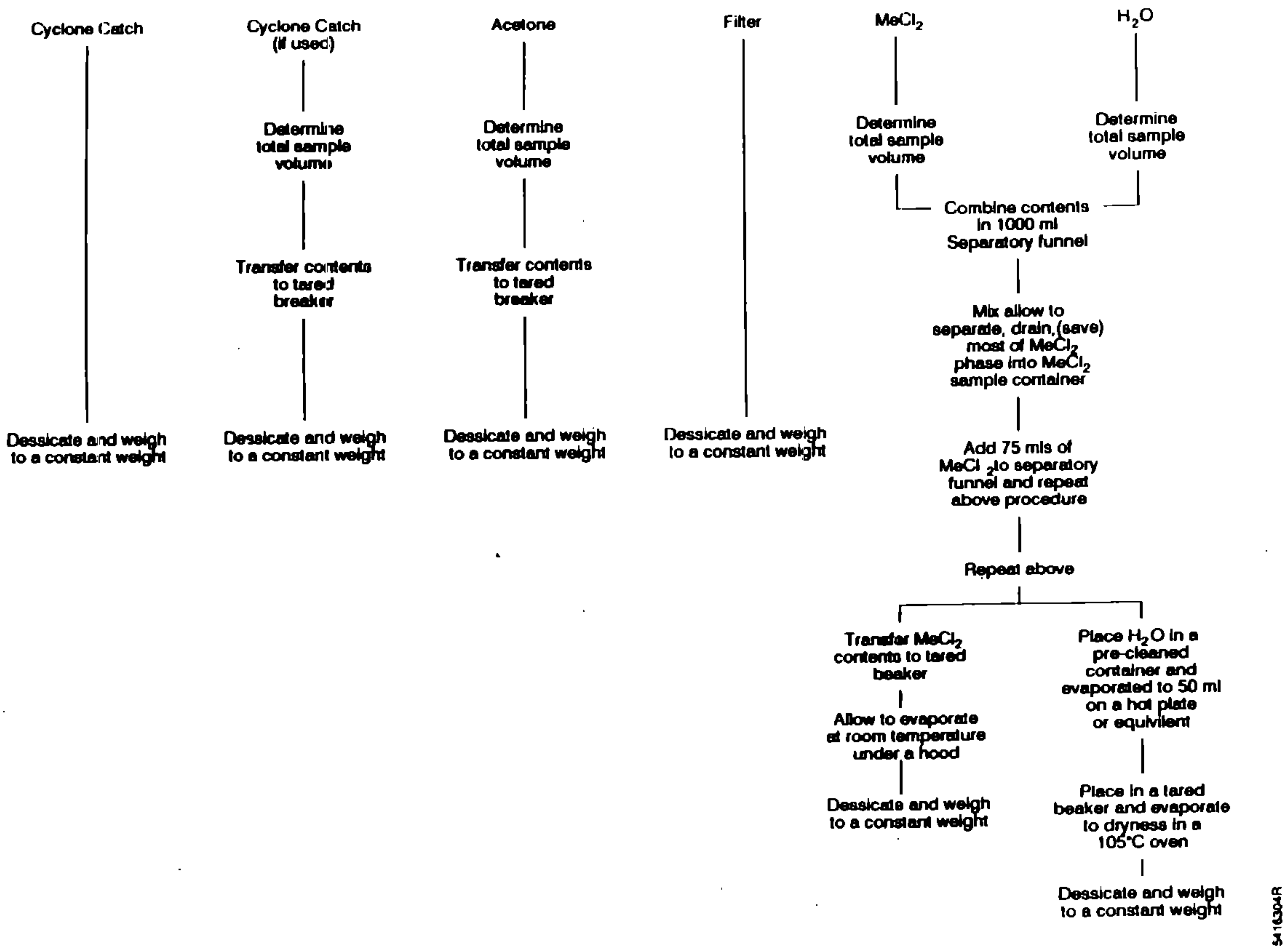

Figure 5-6. PM_{10}/CPM Analytical Scheme

Moisture Determination

Volume or weight of liquid in impingers ________________ ml or g
Weight of moisture in silica gel ________________ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport ________________ ml
Final volume ________________ ml
pH of sample prior to analysis ________________
Addition of NH_4OH required? ________________
Sample extracted 2X with 75 ml $MeCl_2$? ________________

For Titration of Sulfate

Normality of NH_4OH ________________ N
Volume of sample titrated ________________ ml
Volume of titrant ________________ ml

Sample Analysis

Container number	Weight of Condensible Particulate, mg		
	Final Weight	Tare Weight	Weight Gain
4 (Inorganic) 4 & 5 (Organic)			

Total ________________
Less Blank ________________
Weight of Condensible Particulate ________________

Figure 5-7. Analytical Data Sheet

Organic Fraction Weight Determination

The organic extract was evaporated under a laboratory hood. Following evaporation, it was dried for 24 hours in a desiccator containing silica gel. The resulting sample was weighed to the nearest 0.1 mg.

Inorganic Fraction Weight Determination

The water sample was evaporated on a hot plate to approximately 50 ml, then evaporated to dryness in a 105°C oven. Because no N_2 recovery purge was used, the sample was then desiccated and weighed to a constant weight.

5.2.5.3 Field Blank Analysis. The acetone field blank was measured gravimetrically and transferred to a 250-ml beaker. The sample was evaporated to dryness, desiccated for 24 hours, and weighed to a constant weight.

The $MeCl_2$ and water field blanks were analyzed as described in Sections 1.2.5.2.1 and 1.2.5.2.2 of the test method, respectively. Blank correction was not required, because the sum of the values for the water blank and the $MeCl_2$ blank was less than 2 mg or 5 percent of the mass of the CPM, whichever is greater.

5.3 ALDEHYDES EMISSIONS TESTING

Sampling for aldehydes was performed according to EPA SW-846 Test Method 0011, "Sampling for Aldehyde and Ketone Emissions from Stationary Sources."

5.3.1 Sampling Equipment for Aldehydes

This methodology used the sampling train shown in Figure 5-8. The four-impinger train consisted of a quartz nozzle/probe liner followed by a series of impingers and the standard EPA Method 5 meterbox and vacuum pump. The contents of the sequential impingers were: the first two impingers with 2,4-dinitrophenylhydrazine (DNPH), the third impinger empty, and the fourth impinger with silica gel. The first, third, and fourth impingers were of the Greenburg-Smith design; the second impinger had a straight tube. The impingers were connected together with clean glass U-tube connectors. Sampling train components were recovered and analyzed in several fractions in accordance with the described method.

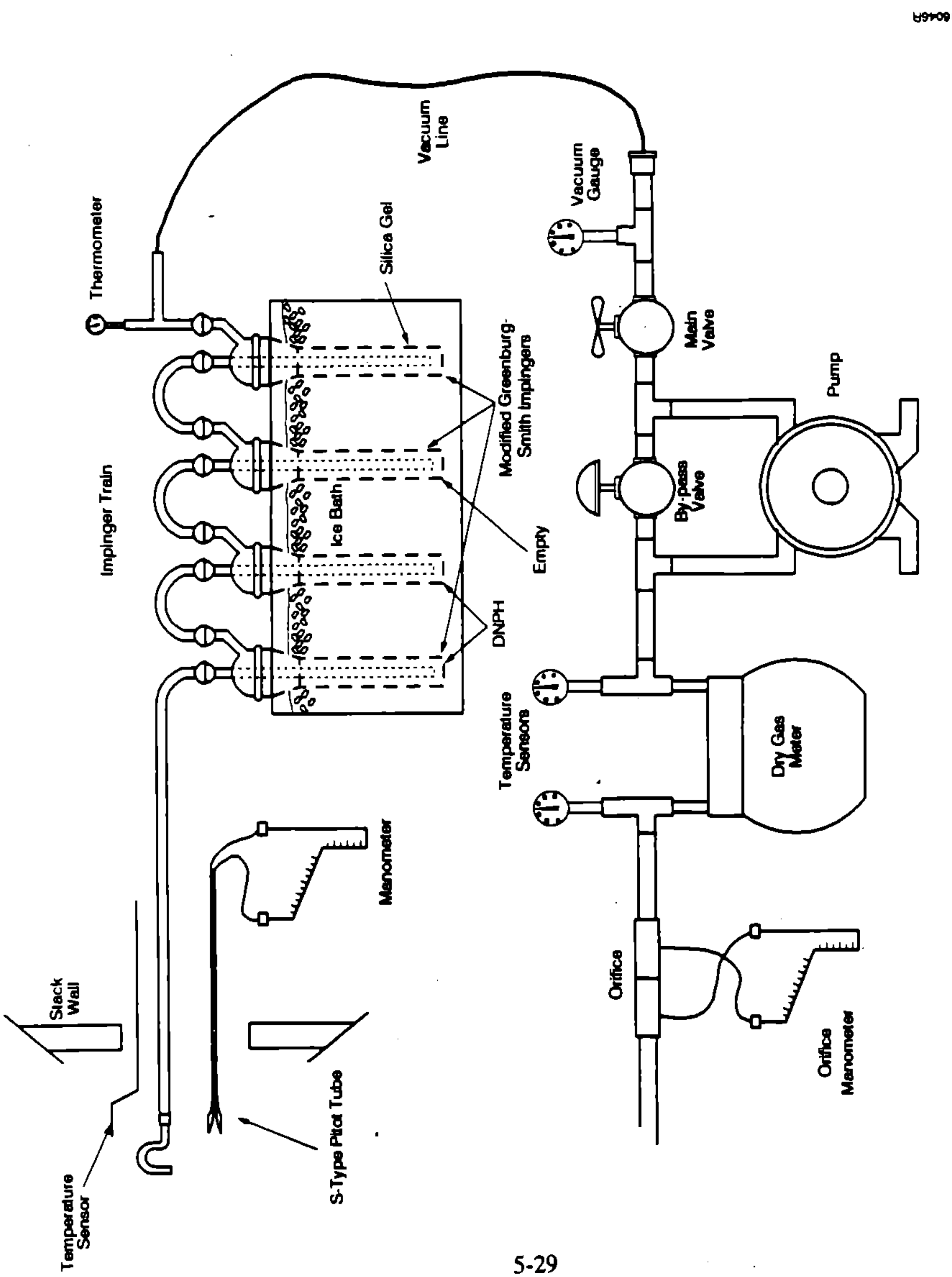

Figure 5-8. Aldehyde Sampling Train

5.3.2 Sampling Equipment Preparation for Aldehydes

5.3.2.1 Glassware Preparation. Glassware was washed in hot, soapy water, rinsed with tap water three times, and then rinsed with deionized distilled water three times. The glassware was then rinsed with methylene chloride, drained, dried, and heated in a laboratory oven at 130°C for several hours. Solvent rinses with methanol were substituted for the oven heating. After drying and cooling, glassware was stored in a clean environment to prevent any accumulation of dust or other contaminants.

5.3.2.2 Reagent Preparation. Reagent grade chemicals were used in all tests and conformed to the specifications of the Committee on Analytical Reagents of the American Chemical Society.

The reagent water was organic-free reagent water. The lot number, manufacturer, and grade of each reagent that was used were recorded in the laboratory notebook.

The DNPH absorbing solution was prepared according to Section 3.5.5.4.2 of the reference method. The analyst wore plastic gloves and safety glasses when handling DNPH crystals or solutions. Reagent bottles for storage of cleaned DNPH derivatizing solution were rinsed with acetonitrile and dried before use.

5.3.2.3 Equipment Preparation. The remaining preparation included calibration and leak checking of all train equipment as specified in EPA Method 5. This equipment included probe nozzles, pitot tubes, metering system, probe heater, temperature gauges, leak check metering system, and barometer. A field laboratory notebook was maintained to record these calibration values.

5.3.3 Aldehydes Sampling Operations

5.3.3.1 Preliminary Measurements. Prior to sampling, preliminary measurements were required to ensure isokinetic sampling. These included determining the traverse point locations, performing a preliminary velocity traverse, cyclonic flow check, and moisture determination. These measurements were used to calculate a K factor. The K-factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements were then made to verify the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances.

These measurements were then used to determine sampling point locations by following EPA Reference Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

5.3.3.2 Assembling the Train. Initial assembly of the aldehyde sampling train components was completed at the recovery trailer. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground glass joint was carefully inspected for hairline cracks. The first impinger was of the Greenburg-Smith design and contained DNPH. The second impinger was a straight tube and also contained DNPH. The third impinger, of the Greenburg-Smith design, was empty, and served as a knockout to collect condensate. The fourth impinger contained 200 to 300 grams of blue indicating silica gel.

After the impingers were loaded, each impinger was weighed, and the initial weight and contents of each impinger was recorded on a recovery data sheet. Final assembly of the sampling train components was completed at the stack location. The impingers were connected using clean, glass U-tube connectors. The height of all impingers was approximately the same to obtain a leak-free seal. The open ends of the train were sealed with ground-glass caps.

5.3.3.3 Sampling Procedures. After the train was assembled, the heaters for the probe liner were turned on. When the system reached the appropriate temperature, the sampling train was ready for pretest leak checking. The probe temperature was maintained above 100°C (212°F). The sampling trains were leak checked at the start and finish of sampling. An acceptable pretest leak rate was less than 0.02 acfm (ft^3/min) at approximately 15 in. Hg. If, during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed and after the train was reassembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled through the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the train was systematically checked by first capping the train at the first impinger, the second impinger, etc., until the leak was located and corrected.

After a successful pretest leak check had been conducted, all train components were at their specified temperatures, and initial data were recorded (DGM reading), the test was initiated. Sampling train data were recorded periodically on standard data forms.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other occurences during sampling were recorded on the task log, such as pitot cleaning, thermocouple malfunctions, heater malfunctions, or any other unusual occurrence.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. The procedure was identical to the pretest procedure, but the vacuum should have been at least one in. Hg higher than the highest vacuum attained during sampling. An acceptable leak rate was less than 4 percent of the average sample rate or 0.02 acfm (whichever is lower). If a final leak rate did not meet the acceptable criterion, the test run may still have been accepted upon approval of the test administrator.

5.3.4 Aldehydes Sample Recovery

Recovery procedures began as soon as the probe was removed from the stack and the post-test leak check was completed.

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into two sections: the nozzle/probe liner, and the impingers in their bucket. Each of these sections was capped before being removed to the recovery trailer.

Once in the trailer, the entire sampling train was recovered into one sample container. The weight gain in each of the impingers was recorded to determine the moisture content in the flue gas. Following weighing of the impingers, the nozzle/probe was recovered. The probe liner was rinsed with methylene chloride by tilting and rotating the probe while squirting methylene chloride into its upper end so that all inside surfaces were wetted. The methylene chloride was quantitatively collected into the sample container. This rinse was followed by additional brush/rinse procedures; the probe was held in an inclined position and methylene chloride was squirted into the upper end as the brush was pushed through with a twisting action. The procedure was performed three times. The brush was also rinsed with methylene chloride and the washing liquid was quantitatively collected in the sample container.

The first three impingers were then rinsed three times with methylene chloride and the washing was collected in the same sample container that was used for the probe. There were at least two liquid phases in the impingers. This two-phase mixture did not pour well and a significant amount of the impinger catch was left on the walls after the methylene chloride rinse. The use of water as a final rinse helped make the recovery quantitative.

After all methylene chloride and water washing and particulate matter had been collected in the sample container, the lid was tightened so solvent, water, and DNPH reagent did not leak out.

A sample blank was prepared by using an amber flint glass container and adding a volume of DNPH reagent and methylene chloride equal to the total volume in the first container.

The silica gel from the train was saved in a bag for regeneration after the job was completed. The ground-glass fittings on the silica gel impinger were wiped off after sample recovery to ensure a leak-tight fit for the next test.

The liquid level of each sample container was marked on the bottle in order to determine if any sample loss occurred during shipment. If sample loss had occurred, the sample would be voided or a method would have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

5.3.5 Aldehydes Analysis

The methylene chloride extract was solvent exchanged into acetonitrile prior to HPLC analysis. Liquid chromatographic conditions are described which permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

5.3.6 Quality Assurance for Aldehydes

The quality assurance (QA) program required for this method included the analysis of the field and method blanks, procedure validations, and analysis of field spikes. The assessment of combustion data and positive identification and quantitation of formaldehyde were dependent on the integrity of the samples received and the precision and accuracy of the analytical methodology. The QA procedures for this method were designed to monitor the performance of the analytical methodology and to provide the required information to take corrective action if problems were observed in laboratory operations or in field sampling activities.

Field blanks were submitted with the samples collected at each sampling site. The field blanks included the sample bottles containing aliquots of sample recovery solvents, methylene chloride and water, and unused DNPH reagent. At a minimum, one complete sampling train was assembled in the field staging area, taken to the sampling area, and leak checked at the beginning and end of testing. The probe of the blank train was heated during the sample test. The train was recovered as if it were an actual test sample. No gaseous sample was passed through the blank sampling train.

To evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory, a method blank was prepared for each set of analytical operations.

A field spike was performed by introducing 200 μl of the field spike standard into an impinger containing 200 ml of DNPH solution. Standard impinger recovery procedures were followed and the spike was used as a check on field handling and recovery procedures. An aliquot of the field spike standard was retained in the laboratory for derivatization and comparative analysis.

5.4 NONMETHANE HYDROCARBON ANALYSIS BY METHOD 25A AND C1-C6 BY METHOD 18

Benzene, toluene, and xylene concentrations were determined according to EPA Method 18. Total gaseous hydrocarbon (THC) concentrations were determined according to EPA Method 25A. Methane concentrations were determined by subtracting the results of EPA Method 18 from EPA Method 25A.

The instrument used to determine THC utilized a flame ionization detector (FID). For FIDs, the flue gas entered the detector and hydrocarbons were combusted in a hydrogen flame. The ions and electrons formed in the flame entered an electrode gap, decreased the gas resistance, and permitted a current flow in an external circuit. The resulting current was proportional to the instantaneous concentration of the total hydrocarbons.

The flue gas was analyzed by a Ratfisch Model 55 analyzer. The analyzer utilized a FID. The results are reported on a methane basis. Methane was used as the calibration gas.

EPA Method 18 analysis was performed using gas chromatography (GC) to separate the hydrocarbon (C_1 - C_6) species present in the gas stream. Prior to sampling of the source gas, the GC/FID system was calibrated with standard gas mixtures containing each hydrocarbon (CH_4, C_2H_6, C_3H_6, C_4H_{10}, C_5H_{12}, and C_6H_{14}) to establish calibration curves and retention times. The calibration curves and retention times were used to quantify the concentrations in the source samples.

A heat-traced slipstream from the stack was transferred to the Shimadzu Mini2 GC/FID to prevent any condensation of the sample gas. The gas sampling loop of the GC/FID was also heated and was purged each time a sample was analyzed. A schematic of the CEM and GC system is shown in Figure 5-9. Each analysis was approximately 10 to 15 minutes in duration. Thus, this analysis was semicontinuous with a result being generated approximately every 5 to 10 minutes during the sampling period.

The source sample was drawn into the GC sampling loop under conditions that prevented any condensation of the sample gas. The sample was injected into the GC and the hydrocarbon compounds were separated by absorbing them onto the column and

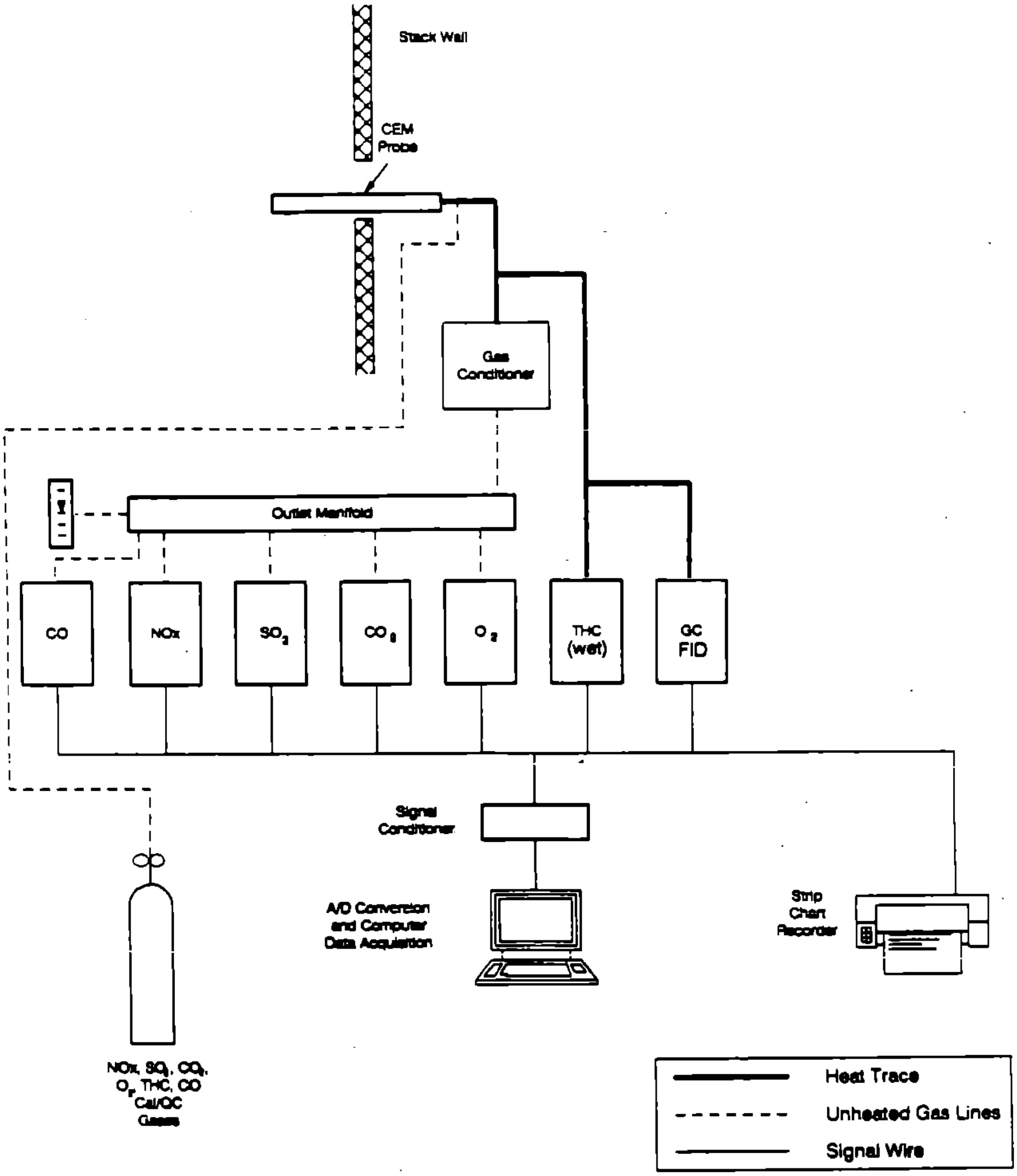

Figure 5-9. Schematic of CEM System

desorbing them at different times. As each hydrocarbon compound was eluted, it was combusted in the hydrogen flame of the FID. The ions and electrons formed in the flame entered an electrode gas, decreased the gas resistance, and permitted a current flow in an external circuit. The resultant current was proportional to the instantaneous concentration of the hydrocarbon.

The response and retention times of the individual hydrocarbons were recorded on a strip chart recorder. A built-in integrator measured the peak areas and printed out the retention times and counts. The peaks were identified from the established retention times and the concentration of each hydrocarbon was determined by referring to the calibration curve.

The nonmethane hydrocarbon concentration was calculated by subtracting the average methane concentration as measured by GC/FID (EPA Method 18) from the average total hydrocarbon concentrations by EPA Method 25A.

5.5 EPA METHODS 1-4

5.5.1 Traverse Point Location By EPA Method 1

The number and location of sampling traverse points necessary for isokinetic and flow sampling was dictated by EPA Method 1 protocol. These parameters were based upon how much duct distance separated the sampling ports from the closest downstream and upstream flow disturbances. The minimum number of traverse points for a square duct of this size was 28. A set of perpendicular sampling ports was established in the stack.

5.5.2 Volumetric Flow Rate Determination by EPA Method 2

Volumetric flow rate was measured according to EPA Method 2. A Type K thermocouple and S-type pitot tube were used to measure flue gas temperature and velocity, respectively. All of the isokinetically sampled methods that were used incorporate EPA Method 2.

5.5.2.1 Sampling and Equipment Preparation. For EPA Method 2, the pitot tubes were calibrated before use following the directions in the method. Also, the pitots were leak checked before and after each run.

5.5.2.2 Sampling Operations. The parameters that were measured included the pressure drop across the pitots, stack temperature, stack static and ambient pressure. These parameters were measured at each traverse point, as applicable. A computer program was used to calculate the average velocity during the sampling period.

5.5.3 O_2 and CO_2 Concentrations by EPA Method 3A

The O_2 and CO_2 concentrations were determined by CEMs following EPA Method 3A. Flue gas was extracted from the duct and delivered to the CEM system through heated Teflon® tubing. The sample stream was then conditioned (particulate and moisture removed) and was directed to the analyzers. The O_2 and CO_2 concentrations were, therefore, determined on a dry basis. Average concentrations were calculated to coincide with each respective time period of interest. More information on the CEM system will be given in Section 5.6.

5.5.4 Average Moisture Determination by EPA Method 4

The average flue gas moisture content was determined according to EPA Method 4. Before sampling, the initial weight of the impingers was recorded. When sampling was completed, the final weights of the impingers were recorded, and the weight gain was calculated. The weight gain and the volume of gas sampled were used to calculate the average moisture content (percent) of the flue gas. The calculations were performed by computer. EPA Method 4 was incorporated in the techniques used for all of the manual sampling methods that were used during the test.

5.6 CONTINUOUS EMISSIONS MONITORING METHODS

EPA Methods 3A, 7E, 6C, and 10 were the continuous monitoring methods used for measuring CO_2/O_2, NO_x, SO_2, and CO concentrations, respectively. Total hydrocarbons were analyzed by EPA Method 25A. A diagram of the CEM system is shown in Figure 5-9.

One extractive system was used to obtain flue gas samples for the CEM systems. For the main CEM system, samples were withdrawn continuously at a single point from the outlet duct and transferred to the CEM trailer through heat-traced Teflon® line. The flue gas was conditioned (temperature lowered and moisture removed) before the flue gas stream was split through a manifold to the various analyzers. Total hydrocarbon measurements were made on an unconditioned, hot basis. Therefore, this sample stream bypassed the gas conditioner.

5.6.1 CEM Sampling Equipment

5.6.1.1 Sample Probes. The main CEM probe consisted of a black iron pipe mounted to a Swagelok® reducing union which was attached directly to the heat trace tubing. The probe was placed approximately at a point of average velocity in the stack determined by a prior velocity traverse by EPA Method 2.

5.6.1.2 Heated Lines. Heated sample lines were used to transfer the flue gas samples to the instrument trailer for O_2, CO_2, NO_x, SO_2, CO, and THC analyses. These lines were heated in order to prevent condensation. Condensate could clog sample lines or provide a medium for the flue gas sample to react and change composition.

All heat trace lines contained three 3/8-inch Teflon® tubes. One tube carried the sample, one tube was used for calibration and QC gases, and the other was available as a backup. Calibration and QC gases were directed to the sampling probe through the transfer tube and then back through the entire sampling/conditioning system.

5.6.1.3 Gas Conditioning. Special gas conditioners were used to reduce the moisture content of the flue gas. A Radian designed gas conditioning system utilized a chiller system to cool a series of glass cyclones. An antifreeze liquid system was used to chill the glass cyclones. The hot flue gas was chilled by heat conduction through the glass wall causing the moisture to condense into droplets. The droplets and any PM were flung outward toward the glass walls by the centrifugal force. Particles impacted the glass walls and fell to the bottom of the cyclone where they were drained from the system. In this manner, both moisture and PM were effectively removed from the flue gas sample stream. This system operated under positive pressure eliminating the possibility of leakage which would dilute the gas samples. The gas conditioner was located in the CEM trailer.

5.6.2 CEM Principles of Operation

5.6.2.1 Sulfur Dioxide Analysis. The Western 721A SO_2 analyzer was essentially a continuous spectrophotometer in the ultraviolet range. Sodium dioxide selectively absorbed ultraviolet (UV) light at a wavelength of 202.5 nm. To take advantage of this property of SO_2, the analyzer emitted UV light at 202.5 nm and measured the absorbance (A) of the radiation through the sample cell by the decrease in intensity. Beer's law, A = abc, was used to convert the absorbance into SO_2 concentration (A = absorbance, a = absorbitivity, b = path length, c = concentration). Sulfur dioxide measurements were performed using EPA Method 6C.

5.6.2.2 Nitrogen Oxide Analysis. The principle of operation of the TECO Model 10AR was a chemiluminescent reaction in which ozone (O_3) reacted with nitric oxide (NO) to form O_2 and nitrogen dioxide (NO_2). During this reaction, a photon was emitted which was detected by a photomultiplier tube. The instrument was capable of analyzing total oxides of nitrogen (NO + NO_2) by thermally converting NO_2 to NO in a separate reaction chamber prior to the photomultiplier tube. Nitrogen oxide measurements were performed using EPA Method 7E.

5.6.2.3 Oxygen Analysis. The Thermox WDG IV measured O_2 using an electrochemical cell. Porous platinum electrodes were attached to the inside and outside of the cell, which provided the instrument voltage response. Zirconium oxide contained in the cell conducted electrons when it was hot from the mobility of O_2 ions in its crystal structure. A difference in O_2 concentration between the sample side of the cell and the reference (outside) side of the cell produced a voltage. This response voltage was proportional to the logarithm of the O_2 concentration ratio. A linearizer circuit board was used to make the response linear. The reference gas was ambient air at 20.9 percent O_2 by volume.

5.6.2.4 Carbon Dioxide Analysis. Non-dispersive infrared (NDIR) CO_2 analyzers emitted a specific wavelength of infrared radiation which was selectively absorbed by CO_2 molecules through the sample cell. The intensity of radiation which reached the end of the sample cell was compared to the intensity of radiation through a CO_2-free reference cell. A reference cell was used to determine background absorbance which was subtracted from the sample absorbance. The detector used two chambers filled with CO_2 and connected by a deflective metallic diaphragm. One side received radiation from the sample cell and the other side received radiation from the reference cell. Since more radiation was absorbed in the sample cell than in the reference cell, less radiation reached the sample side of the detector. This caused a deflection of the diaphragm due to increased heat from radiation absorption on the reference side. Deflection of the diaphragm created an electrical potential which was proportional to absorbance. Absorbance was directly proportional to CO_2 concentration in the gas. Carbon dioxide measurements were performed according to EPA Method 3A using a Beckman Model 880 NDIR anlayzer.

5.6.2.5 Carbon Monoxide Analysis. A TECO Model 48 analyzer was used to monitor CO emissions. The TECO analyzer measured CO using the same principle of operation as CO_2 analysis. A wave length of 5 nm is selective for CO. Carbon monoxide measurements were performed using EPA Method 10.

5.6.2.6 Total Hydrocarbon Analysis. A Ratfisch Model 55 was used to monitor THC emissions. By allowing the THC sample stream to bypass the gas conditioner, concentrations were determined on a wet basis. The analyzer employed an FID. As the flue gas entered the detector, the hydrocarbons were combusted in a hydrogen flame. The ions and electrons formed in the flame entered an electrode gap, decreased the gas resistance, and permitted a current flow in an external circuit. The resulting current was proportional to the instantaneous concentration of the total hydrocarbons. This method was not selective between species. EPA Method 25A applies to the continuous measurement of total gaseous organic concentrations of primarily alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The results were reported on a methane basis and methane was used as the calibration gas.

5.6.3 CEM Calibration

All the CEM instruments were calibrated once during the test program (and linearized, if necessary) using a minimum of three certified calibration gases (zero and two upscale points). Radian performed the multipoint calibrations with four general categories of certified gases: zero gas (generally N_2), a low scale gas concentration, a midrange concentration, and a high scale concentration (span gas). The criterion for acceptable linearity was a correlation coefficient (R^2) of greater than or equal to 0.998, where the independent variable was cylinder gas concentration and the dependent variable was instrument response. If an instrument did not meet these requirements, it was linearized by adjusting potentiometers on the linearity card within the instrument or by other adjustments, if necessary.

The CEM analyzers were calibrated before and after each test run (test day) on a two point basis: zero gas (generally N_2), and a high-range span gas. These calibrations were used to calculate response factors used for sample gas concentration determinations. Instrument drift as a percent of span was also determined using these calibrations for each test run.

After each initial calibration, midrange gases for all instruments were analyzed, with no adjustment permitted, as a quality control (QC) check. If the QC midrange gas concentration observed was within ±2 percent of full scale, the calibration was accepted and the operator began sampling. If the QC check did not fulfill this requirement, another calibration was performed and linearization was performed if deemed necessary. Calibration procedures are further detailed in the daily operating procedure (Section 5.6.5).

Table 5-3 lists the concentration of all calibration and QC gases used on this test program.

5.6.4 Data Acquisition

The data acquisition system consisted of a Dianachart PC Acquisitor data logger, a signal conditioner, and a 386 desktop computer. All instrument outputs were connected in parallel to stripchart recorders and the data acquisition system. The stripchart recorders were a back-up system to the data logger. The PC Acquisitor scanned the instrument output and logged digitized voltages. A Radian computer program translated the digitized voltages into relevant concentrations in engineering units (ppmv, %V, etc.). The computer program had several modes of operation: calibration, data acquisition, data reduction, data view, data edit, and data import. The import function was used to combine other data files for comparison and correlation.

5.6.5 Daily Operating Procedure

The following is a detailed standard operating procedure for calibrating and operating the CEM system:

Table 5-3

CEM Operating Ranges And Calibration Gases

Analyte	Gas Concentration
CO_2	
Instrument	Beckman 880
Range	0 - 20%
Span Gas Value	18%
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	10%
Low Range QC Gas Value	5%
CO - dry	
Instrument	TECO 48H
Range	0 - 100 ppmvd
Span Gas Value	98 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	60 ppm
Low Range QC Gas Value	20 ppm
O_2	
Instrument	Thermox WDG III
Range	0 - 25%
Span Gas Value	20%
Zero Gas	0.2% O_2
Midrange QC Gas Value	10%
Low Range QC Gas Value	5%
SO_2	
Instrument	Western 721A
Range	0 - 200 ppmvd
Span Gas Value	180 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	100 ppm
Low Range QC Gas Value	30 ppm

Table 5-3

Continued

Analyte	Gas Concentration
NO_x	
Instrument	TECO 10AR
Range	0 - 250 ppmvd
Span Gas Value	200 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	100 ppm
Low Range QC Gas Value	50 ppm
THC (EPA Method 25A)	
Instrument	Ratfisch RS-55
Range	0 - 100 ppmvd
Span Gas Value	90% as methane
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	45 ppm as methane
Low Range QC Gas Value	25 ppm as methane

1. Turn on computer and printer, put printer on-line, and load the CEM.EXE program. Be sure that the CEM instruments have been on for at least 20 hours.

2. Synchronize DAS clock with sample location leaders and the test leader.

3. Turn on strip chart recorders (SCR) and make appropriate notes on charts and in logbook (write down all procedures and observations in logbook and on SCRs as the day progresses).

4. Turn on the gas conditioners and blow back compressor. Blow back the system.

5. Open all calibration gas cylinders so that they may be introduced to the instruments via control panel valves.

6. Perform daily pretest leak check on CEMs by introducing ultra high purity nitrogen to the system. Zero all instruments except the Thermox O_2 analyzers. Make adjustments to the zero potentiometers as required to zero the instruments. Be sure to check and maintain all flows throughout calibration and operation.

7. Record the zero values in the computer calibration routine.

8. Introduce 2.0 percent O_2 to set the low scale response for the Thermox O_2 analyzers and repeat Step 7 for these instruments.

9. Introduce the mixed span gases for O_2, CO_2, and CO. Make adjustments as required to these instruments.

10. Enter these values in the computer calibration routine.

11. Introduce the NO_x span gas.

12. Make adjustments to the NO_x instruments as required and enter the value into the computer calibration routine.

13. Introduce the SO_2 span gas for the SO_2 analyzer, repeat Step 12 for the SO_2 analyzer. (Note that all calibration gases are passed through the entire sampling system.)

14. Switch the Western SO_2 analyzer range to 0-500 ppm introduce the span gas for this range and repeat Step 12 for this instrument.

15. Check the calibration table on the computer, and make a hardcopy. Put the computer in the standby mode.

16. Introduce QC gases to instruments in the same sequence as the calibration gases. Record three minutes of data for each, once the responses have stabilized. If the QC gas response is not within ±2 percent of the instrument range the operator should recalibrate the instrument, or perform other corrective actions.

17. Begin sampling routine, with the computer on stand by.

18. Start the data acquisition system when signaled by radio that system is in stack.

19. Carefully check all flows and pressures during the operation of the instruments and watch for apparent problems in any of the instruments, such as unusual readings or unreasonable fluctuations. Check the gas conditioning system periodically and drain the traps.

20. Stop the data acquisition system at the end of the test when signaled.

21. Perform final leak check of system.

22. Perform the final calibration (Repeat steps 6-16) except make no adjustments to the system.

23. Check for drift on each channel.

5.7 POLYNUCLEAR AROMATIC HYDROCARBON EMISSIONS TESTING

The polynuclear aromatic hydrocarbon (PAH) sampling and analytical method is a combination of EPA SW-846 Test Method 0010 and EPA SW-846 Test Method 8270.

5.7.1 Sampling Equipment

The PAH sampling method used the sampling train shown in Figure 5-10. Radian modified the protocol configuration to include a horizontal condenser rather than a vertical condenser. The horizontal condenser lowered the profile of the train and reduced breakage. The XAD trap following the condenser was maintained in a vertical position.

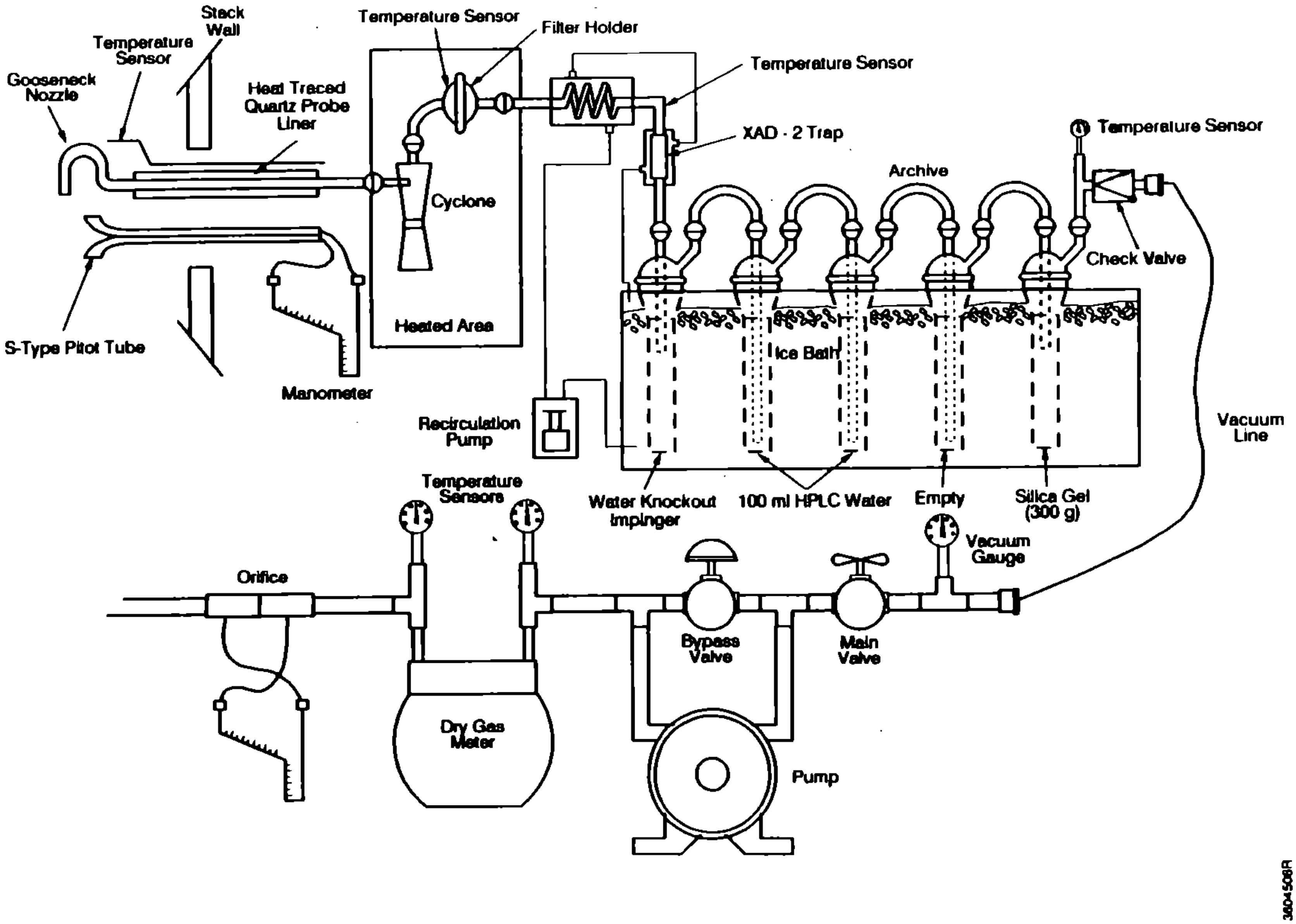

Figure 5-10. PAH Sampling Train Configuration

5.7.2 Sampling Equipment Preparation

In addition to the standard EPA Method 5 requirements, the PAH sampling method included several unique preparation steps which ensured that the sampling train components were not contaminated with organics that may interfere with analysis. The glassware, glass fiber filters, and XAD resins were cleaned and checked for residuals before being packed.

5.7.2.1 Glassware Preparation. Glassware was washed in soapy water, rinsed with distilled water, baked, and then rinsed with acetone followed by methylene chloride. This included all the glass components of the sampling train including the glass nozzles plus any sample bottles, Erlenmeyer flasks, petri dishes, graduated cylinders or stirring rods that were used during recovery. Nonglass components (such as the teflon-coated filter screens and seals, tweezers, teflon squeeze bottles, nylon probe brushes and nylon nozzle brushes) were cleaned following the same procedure except that no baking was performed. The specifics of the cleaning procedure are presented in Table 5-4.

5.7.2.2 XAD II and Filters Preparation. XAD resin and glass fiber filters were placed together in a soxhlet and extracted in HPLC-grade water, methyl alcohol, methylene chloride and hexane, sequentially. At the conclusion of the soxhlet extractions, one filter and 30 grams of XAD resin were analyzed for background contamination following the same procedure followed for the flue gas samples. The XAD and filter blank were analyzed for PAH compounds. The pressure drop for the XAD traps was checked before and after the resin was loaded to ensure that the pressure drop across the XAD traps was less than seven inches of mercury.

5.7.2.3 Method 5 Equipment Preparation. The EPA Method 5 equipment was prepared according to the protocol discussed in Section 5.1.2.3.

Table 5-4

Glassware Cleaning Procedure
(Train Components and Sample Containers)

NOTE: USE DISPOSABLE GLOVES AND ADEQUATE VENTILATION

1. Soak all glassware in hot soapy water (Alconox®).
2. Tap water rinse to remove soap.
3. Distilled/deionized H_2O rinse (X3).[a]
4. Bake at 450°F for 2 hours.[b]
5. Acetone rinse (X3), (pesticide grade).
6. Methylene Chloride (X3).
7. Cap glassware with clean glass plugs or methylene chloride rinsed aluminum foil.
8. Mark cleaned glassware with color-coded identification sticker.

[a] (X3) = Three times.

[b] Step (4) is not used for probe liners and non-glass components of the train that cannot withstand 450°F (i.e., Teflon-coated filter screen and seals, tweezers, Teflon squeeze bottles, nylon probe and nozzle brushes). The probe liners are too large for the baking ovens.

5.7.3 Sampling Operations

5.7.3.1 Preliminary Measurements. Prior to sampling, preliminary measurements were made as described in Section 5.1.3.1.

5.7.3.2 Assembling the Train. Initial assembly of the PAH sampling train components was performed in the recovery trailer. Final assembly of the train with the probe, nozzle, and filter was performed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order. The first impinger was a knockout impinger which had a short tip. The purpose of this impinger was to collect condensate which formed in the coil and XAD trap. However, the gas was not bubbled through the condensate to prevent carry-over to the next impinger. The next two impingers were modified tip impingers that contained 100 ml of HPLC grade water each. The fourth impinger was empty, and the fifth impinger contained 200 to 300 grams of silica gel. When the impingers were loaded, they were wrapped with teflon tape to secure the two sections of the impinger. Then each impinger was weighed and the weight recorded along with information on the contents of the impingers. The impingers were connected together using cleaned glass U-tube connectors and arranged in the impinger bucket. The height of all the impingers should be approximately the same to obtain a leak-free seal. The open ends of the train were sealed with methylene chloride-rinsed aluminum foil.

The second step was to load the filter into the filter holder. The filter holder was then capped off and placed with the XAD trap and condenser coil (capped) into the impinger bucket. A supply of precleaned foil and socket joints were also placed in the bucket in a clean plastic bag. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-10. Sealing greases were not used to avoid contamination or adsorption of the sample.

5.7.3.3 Sampling Procedures. After the train was assembled, the heaters were turned on for the probe liner and heated filter box. When the system reached the appropriate temperatures, the sampling train was ready for leak checking.

The PAH train was leak checked at the start and finish of sampling as required in EPA Method 5 as well as before and after each port change. If a piece of glassware needed to be emptied or replaced, a final leak check was performed before the glassware piece was removed. After the train was reassembled, an initial leak check was performed.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. The leak rate is required to be less than 0.02 acfm (ft^3/min). After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off:

If the leak-rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

In the event that a final leak rate was found to be above the minimum acceptable rate (0.02 acfm) upon removal from a port, acceptance is subject to the approved of the EPA administrator. Otherwise, the run was voided and repeated.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occurred during sampling were recorded on the task log such as pitot cleaning, thermocouple malfunctions, heater malfunctions and any unusual occurrences.

Sampling train data were recorded every five minutes on standard data forms. A checklist for sampling was given previously in Table 5-2. The purpose of the checklist is to remind samplers of the critical steps during sampling.

A sampling operation that was unique to PAH sampling was maintaining the gas temperature entering the XAD trap below 68°F. The gas was cooled by the condenser and the XAD trap, which both have a water jacket in which ice water was circulated.

5.7.4 Sample Recovery

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into four sections: the probe liner, the XAD trap and condenser, filter holder, and the impingers in their bucket. Each of these sections was capped with methylene chloride-rinsed aluminum foil before removal to the recovery trailer. Once in the trailer, field recovery personnel followed the scheme shown in Figure 5-11. The samples were placed in cleaned amber glass bottles to prevent light degradation.

The solvents used for train recovery were acetone (pesticide grade) followed by methylene chloride. The use of the highest grade acetone for train recovery was essential to prevent the introduction of chemical impurities which interfere with the quantitative analytical determinations.

Field recovery resulted in the sample components listed in Table 5-5. The samples were shipped to the analytical laboratory as expediently and carefully as possible.

5.7.5 Analytical Procedures

The analytical procedure used to determine PAH concentrations from the Modified Method 5 sample followed EPA SW-846 Test Method 8270 protocol. The detection limit for PAH was about 1 μg per train. The compounds/isomers of interest in the analysis are shown in Table 5-6.

5.7.5.1 Preparation of Samples for Extraction. Upon receiving the sample shipment, the samples were checked against the chain-of-custody forms and then assigned an analytical laboratory sample number. Each sample component was reweighed to determine if leakage occurred during travel. Color, appearance, and other particulars of the samples were noted. Samples were extracted within 21 days of collection.

Glassware used in the analytical procedures (including soxhlet apparatus and disposable bottles) was cleaned by washing twice with detergent, rinsing with distilled water, and then rinsing with acetone, methanol, and methylene chloride. The glassware was allowed to air dry.

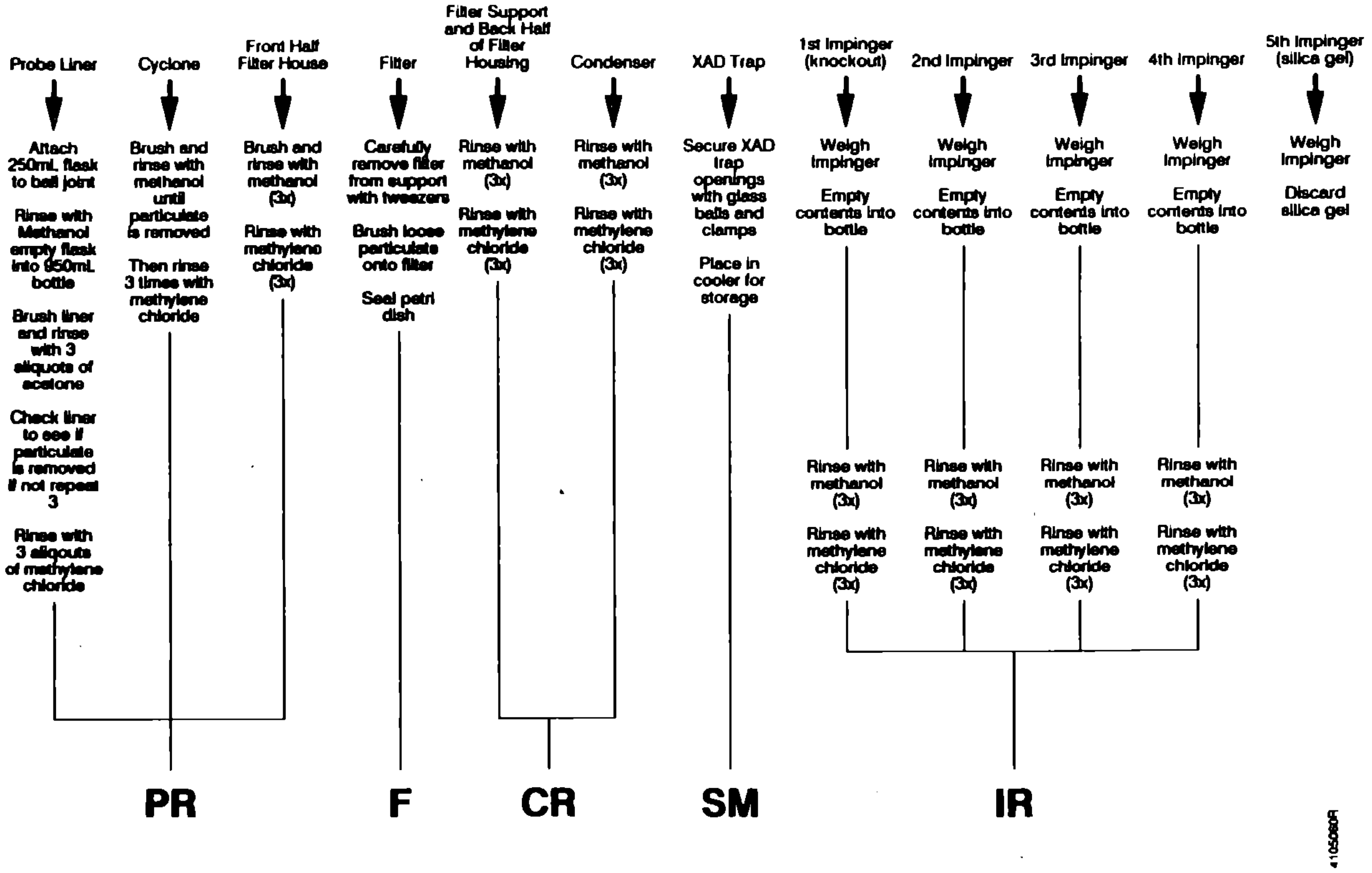

Figure 5-11. PAH Field Recovery Scheme

Table 5-5

Polynuclear Aromatic Hydrocarbon Sample Components Shipped to Analytical Laboratory

Container/Component	Code	Glassware
1	F	Filter(s)
2	PR	Rinses[a] of nozzle, probe, and front half of filter holder
3	CR	Rinses[a] of back half of filter holder, filter support, and condenser
4	IR	First, second, third, and fourth impinger contents and rinses[a]
5	SM	XAD-2 resin

[a] Rinses include acetone and methylene chloride recovered into the same sample bottle.

Table 5-6

Polynuclear Aromatic Hydrocarbon Compounds To Be Analyzed

Naphthalene
Acenapthylene
Acenapthene
Fluorene
Phenathrene
Anthracene
Fluoranthene
Pyrene
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(a)pyrene
2-Methylnapthalene
2-Chloronapthalene
Benzo(k)fluoranthene
Benzo(e)pyrene
Perylene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Dibenzofuran
7,12-Dimethylbenz(a)anthracene
Benzo(g,h,i)perylene

5.7.5.2 Calibration of GC/MS System. An initial calibration of the GC/MS system was performed to demonstrate instrument linearity over the concentration range of interest. Analyses for PAH was performed using low resolution mass spectrometry. A typical calibration range consisted of points at 4:100, 40:100, and 400:100 for the ratio of analytes to isotopically labeled internal standards. Relative response factors were calculated for each compound of interest. The response factors were verified on a daily basis using a continuing calibration standard consisting of mid-level standard (typically the 40:100 standard).

5.7.5.3 Sample Extraction. For PAH analyses, isotopically labeled surrogate compounds were added to the samples before the extraction process was initiated. These surrogates were used to monitor the efficiency of the extraction/clean-up. The internal standards used in the quantitative analysis of these analytes were added to the samples immediately prior to analysis, and used to perform the quantitative calculations.

5.7.5.4 Analysis by GC/MS. The PAH analyses were performed by high-resolution GC followed by low resolution mass spectrometry.

Data from the MS were recorded and stored on a computer file as well as printed on paper. A duplicate analysis was performed on every tenth sample in the sample batch. A method blank which was carried through the complete extraction procedure was also analyzed. Results such as amount detected, detection limit, retention time, and internal standard and surrogate standard recoveries were calculated by computer. The chromatograms were retained by the analytical laboratory and also included in the analytical report.

5.7.6 Analytical QA/QC Procedures

This section discusses the general quality control procedures that were followed for the analytical methods. Method-specific analytical QA/QC procedures are also presented.

5.7.6.1 Quality Control. This section presents the PAH quality control requirements.

Blanks. Two different blanks were collected for the PAH analyses: a laboratory proof blank and a field blank. Proof blanks were obtained from a complete set of

Modified Method 5 sample train glassware that had been cleaned according to the procedure presented in Section 5.1.2. The precleaned glassware, which consisted of a probe liner, filter holder, condenser coil, and impinger set, was loaded and then recovered by rinsing with acetone and methylene chloride three times each. All sets of glassware were blanked.

A field blank was collected from a set of PAH glassware that was used to collect at least one sample and had been recovered. The train was reloaded and left at the sampling location during a test run. The train was then recovered. The field blank was used to measure the level of contamination that occurred from handling, loading, recovering, and transporting the sample train. The field blank was analyzed concurrently with the flue gas samples. If the field blank results were acceptable, the laboratory proof blank and reagent blanks were archived but not analyzed.

Analytical method (reagent) blanks were also analyzed as part of the QC program. The QC criteria for method blanks was concentrations less than or equal to the detection limit (in the noise range).

<u>Standards Duplicates</u>. Isotopically labelled internal standards and surrogate compounds were added to the sample before the extraction process began. Once added to the samples, the internal standards went through the entire extraction process and were measured on the GC/MS. The recoveries of the internal standards were determined and the results of the native species were adjusted according to the internal standard recoveries. The results contained in the analytical report were adjusted for internal standard recoveries. The surrogate compounds were added in a similar manner, but the surrogate recoveries were not used to adjust the results of the native species. Surrogate recoveries provided additional data on the efficiency of the extraction procedure and the performance of the instruments. The QC objective for internal standards and surrogate recoveries was 100 ±50% recovery.

The purpose of duplicate analyses was to evaluate the reproductibility (precision) of the combined sample preparation and analytical methodology. The QC criterion for analysis of duplicates was agreement to within ±50% (for each PAH species). Analytical duplicates (two injections of the same sample) were also analyzed to assess the precision of the analytical methodology. For PAH flue gas samples, only analytical duplicates were performed. For every 10 samples, one duplicate analysis was performed.

5.8 ASTM METHODS

Standard ASTM methods were used to assess heat of combustion, ultimate analysis (ash, O_2, carbon, hydrogen, sulfur, and nitrogen), and chlorine content of the waste oil fuel. Aggregate moisture and ambient humidity were also analyzed. Descriptions of applicable ASTM methods follow.

5.8.1 Relative Humidity

Sampling for relative humidity was performed using ASTM Method E337-62, "Standard Test Method for Relative Humidity by Wet- and Dry-Bulb Psychrometer." This method covers the determination of the relative humidity of atmospheric air by means of wet- and dry-bulb temperature readings.

5.8.1.1 Sampling Equipment and Method. A sling psychrometer was used for measuring relative humidity. Two thermometers, one with a wet-bulb covering were mounted on the psychrometer. The wet-bulb covering was moistened and the psychrometer slung through the air for several minutes. The thermometers were read and the psychrometric chart was used to calculate the relative humidity.

5.8.2 Heat of Combustion Test Method

Heat of combustion of the fuel sample was determined according to ASTM Method D240-87, "Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter." This test method covered the determination of the heat of combustion of liquid hydrocarbon fuels ranging in volatility from that of light distillates to that of residual fuels.

5.8.2.1 Sampling Equipment and Method. Heat of combustion was determined using an O_2 bomb, calorimeter, stirred water jacket, and a thermometer. A weighed sample was burned in an O_2 bomb calorimeter under controlled conditions. The heat of combustion was computed from temperature observations before, during, and after combustion with proper allowance for thermochemical and heat transfer corrections. Adiabatic calorimeter jackets were used.

5.8.3 Total Moisture Content Test Method

Total moisture content of the aggregate sample was determined according to ASTM Method C566-89, "Standard Test Method for Total Moisture Content of Aggregate by Drying." This test method covered the determination of the percentage of evaporative moisture in a sample. The plant routinely performed this test at least several times per day. This information was provided by the plant personnel and is included in the process data section of the test report.

5.8.3.1 Sampling Equipment and Method. Total moisture content was determined using a balance or scale accurately readable and sensitive to within 0.1 percent of the test load, a source of heat such as a ventilated oven capable of maintaining the temperature surrounding the sample at 110 ±5°C (230 ±9°F); and a sample container not affected by the heat and of sufficient volume to contain the sample without danger of spilling.

The sample was weighed to the nearest 0.1 percent and then dried in the sample container. The temperature was controlled when excessive heat may alter the character of the aggregate or where more precise measurement was needed. The dried sample was weighed to the nearest 0.1 percent after it had cooled sufficiently to prevent damage to the balance. Total moisture was calculated using the formulas presented in Section 7.1 of the reference method.

5.8.4 Sulfur Test Method

Sulfur concentrations in the sample were determined according to ASTM Method D1552-90, "Standard Test Method for Sulfur in Petroleum Products (High Temperature Method)." This test method covered the procedures for the determination of total sulfur in petroleum products including lubricating oils containing additives and in additive concentrates.

5.8.4.1 Sampling Equipment and Method. Sulfur content of a sample was determined using a furnace, an absorber, a buret, and other miscellaneous apparatus.

The IR detection system was used for the determination of sulfur. The sample was weighed into a special ceramic boat which was then placed into a combustion furnace in an O_2 atmosphere. Any sulfur was combusted to SO_2 which was then measured with an IR detector after moisture and dust were removed by traps. A microprocessor calculated the mass percent sulfur from the sample weight, the integrated detector signal and a predetermined calibration factor. Both the sample identification number and mass percent sulfur were then printed out. The calibration factor was determined using standards approximating the material to be analyzed.

5.8.5 Nitrogen Test Method

Nitrogen concentration in the sample was determined according to ASTM Method D3179-84, "Standard Test Methods for Nitrogen in the Analysis of Coal and Coke." This test method covered the determination of total nitrogen in samples of coal and coke.

5.8.5.1 Sampling Equipment and Method. Total nitrogen was determined using a digestion unit, digestion flasks, distillate unit, buret, Erlenmeyer flasks, rubber tubing, and pipets. Reagents included an alkali solution, ethyl alcohol, and sulfuric acid. Nitrogen in the sample was converted into ammonium salts by destructive digestion of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts were subsequently decomposed in a hot alkaline solution from which the ammonia was recovered by distillation and finally determined by alkalimetric or acidimetric titration.

5.8.6 Carbon and Hydrogen Test Method

Carbon and hydrogen concentrations in the sample were determined according to ASTM Method D3178-84, "Standard Test Method for Carbon and Hydrogen in the Analysis Sample of Coal and Coke."

5.8.6.1 Sampling Equipment and Method. Carbon and hydrogen content were determined using an O_2 purifying train that consisted of two water absorbers and a CO_2 absorber, a flow meter, a combustion unit, and reagents. A quantity of the sample was burned in a closed system. The products of combustion were fixed in an absorption train after complete oxidation and purification from interfering substances. This test method gave the total percentages of carbon and hydrogen and included the carbon in carbonates and the hydrogen in the moisture and in the water of hydration of silicates.

6. QUALITY ASSURANCE AND QUALITY CONTROL

To ensure the production of useful and valid data, specific QA/QC procedures were strictly adhered to during this test program. Detailed QC procedures for all manual flue gas sampling, process sample collection, GC operations, and CEM operations can be found in the site-specific test plan prepared by Radian Corporation[1]. This section presents the test program QA parameters and results so that the degree of data quality can be shown.

6.1 SUMMARY

Tests were conducted over a three-day period at Mathy Construction Company, Plant 26. Three sets of runs were completed successfully at normal operating conditions while the plant was operating on waste fuel oil. No sampling-related problems that would affect data quality were encountered during testing.

In summary, the data quality was maintained throughout the project and this data can be used as described. Post-test leak checks for all sampling trains were within acceptable limits and all post-test calibration checks for the dry gas meters were within acceptable limits. All PM/metals, aldehydes, and PAH manual sampling trains met the isokinetic criterion of ±10 percent out of 100 percent, with the exception of one aldehyde run. This run was accepted because it exceeded the limits only slightly. The PM_{10}/CPM manual sampling trains met the isokinetic criterion of ±20 percent out of 100 percent, which is acceptable for this test method.

Method blank and field blank results for the manual sampling trains showed some contamination. Also, a few method spike recovery values for the metals, aldehydes, and PAH analyses were not within the QA allowance.

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[1] "Emission Testing for Asphalt Concrete Industry, Site-Specific Test Plan and Quality Assurance Project Plan, Mathy Construction Company Plant 26," Radian Corporation, September 1991.

The CEM results showed acceptable calibration drift values and QC gas responses. All CEM QC procedures and objectives described in the site-specific test plan were followed.

The GC used for EPA Method 18 analysis was calibrated each test day before and after flue gas sampling. Quality assurance/quality control results showed allowable response factor drift values for most of the runs conducted.

The remainder of this section is organized as follows: Section 6.2 presents the QA/QC definitions and data quality objectives; Section 6.3 presents manual flue gas sampling and recovery parameters, and a further discussion of method blank, field blank, and method spike results; Section 6.4 presents method-specific analytical QA parameters; Section 6.5 discusses the CEM QA parameters; and Section 6.6 presents the GC QA parameters.

6.2 QUALITY ASSURANCE/QUALITY CONTROL DEFINITIONS AND OBJECTIVES

The overall QA/QC objective in this test program was to ensure precision, accuracy, completeness, comparability, and representativeness for each major measurement parameter. The following definitions were used:

- Quality Control: The overall system of activities whose purpose is to provide a quality product or service. Quality control procedures are routinely followed to ensure high data quality.

- Quality Assurance: A system of activities whose purpose is to ensure that overall QC is being carried out effectively. The degree of data quality achieved can be assessed from QA parameters.

- Data Quality: The characteristics of a product (measurement data) that bear on its ability to satisfy a given purpose. These characteristics are:

    - Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation, and in this report is expressed as the relative standard deviation or coefficient of variation.

- Accuracy - The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, which can be expressed as the difference between two values, X-T, the ratio X/T, or the difference as a percentage of the reference or true value, 100 (X-T)/T.

- Completeness - A measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under prescribed test conditions.

- Comparability - A measure of the confidence with which one data set can be compared with another.

- Representativeness - The degree to which data accurately and precisely represent a characteristic of a population, variations of a parameter at a sampling point, or an environmental condition.

A summary of the estimated precision, accuracy, and completeness objectives is presented in Table 6-1.

6.3 MANUAL FLUE GAS SAMPLING QUALITY ASSURANCE

The following section reports manual sampling QA parameters in order to provide insight into the quality of the emissions test data produced from manual tests during the test program.

6.3.1 Particulate Matter/Metals Sampling Quality Assurance

Table 6-2 presents post-test check results for all of the manual sample trains. The acceptance criterion was that all post-test leak checks be less than 0.02 cfm. All PM/metals post-test leak checks met the acceptance criterion.

The isokinetic sampling rates for all of the manual sampling runs are presented in Table 6-3. The acceptance criterion for the PM/metals, aldehydes, and PAH sampling runs is that the average sampling rate must be within 10 percent of 100 percent isokinetic. All PM/metals runs deviated by no more than 3 percent of 100 percent, thereby meeting the isokinetic criterion.

All dry gas meters used for manual sampling were fully calibrated within the last six months against an EPA-approved intermediate standard. The full calibration factor, or meter Y, was used to correct actual metered volume to true sample volume. To

Table 6-1

Summary of Precision, Accuracy, and Completeness Objectives

Parameter	Precision[a] (%)	Accuracy[a] (%)	Completeness[b] (%)
Flue Gas Formaldehyde	±15	±20	100
Flue Gas Metals	±15[d]	±30[c]	100
Polynuclear Aromatic Hydrocarbons	±15	±20	100
Flue Gas Total Particulate Matter	±11	±10	100
Continuous Monitoring System	±2[e]	±2[d]	95
Velocity/Volumetric Flow Rate	±6	±10	95
Fixed Gases/Molecular Weight	±0.3%V	±0.5%V	100
Moisture	±20	±10	95
Flue Gas Temperature	±2°F	±5°F	100

[a] Precision and accuracy estimated based on results of EPA collaborative tests. All values stated represent worst case values. All values are absolute percentages unless otherwise stated.

[b] Minimum valid data as a percentage of total tests conducted.

[c] Relative error (%) derived from audit analyses, where:

$$\text{Percent Error} = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100$$

[d] Percent difference for duplicate analyses, where:

$$\text{Percent Difference} = \frac{\text{First Value} - \text{Second Value}}{0.5(\text{First} + \text{Second Values})} \times 100$$

[e] Minimum requirements of EPA method 6C, based on percent of full scale.

Table 6-2
LEAK CHECK RESULTS FOR MANUAL SAMPLE TRAINS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Sample Train	Date	Run Number	Maximum Vacuum	Avg. Sample Rate (acfm)	Measured Leak Rate	Vacuum (in. Hg)	Acceptable
PM/Metals	09/23/91	1	1	0.33	0.012	5	Yes
PM/Metals	09/23/91	2	1	0.33	0.014	5	Yes
PM/Metals	09/24/91	3	1	0.33	0.012	8	Yes
Aldehydes	09/23/91	1	2	0.50	0.010	7	Yes
Aldehydes	09/23/91	2	2	0.56	0.006	5	Yes
Aldehydes	09/25/91	3	1	0.48	0.014	6	Yes
Aldehydes	09/24/91	4	1	0.34	0.010	6	Yes
PAH	09/23/91	1	4	0.34	0.008	7	Yes
PAH	09/23/91	2	3	0.35	0.010	5	Yes
PAH	09/24/91	3	4	0.32	0.008	7	Yes
PM10	09/24/91	1	1	0.25	0.010	10	Yes
PM10	09/25/92	2	1	0.28	0.012	10	Yes
PM10	09/25/91	3	1	0.31	0.016	5	Yes

Table 6-3
ISOKINETIC SAMPLING RATES FOR MANUAL SAMPLING TEST RUN MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Sample Type	Date	Run Number	Isokinetic Sampling Rates	Acceptable a
PM/Metals	09/23/91	1	102	Yes
PM/Metals	09/23/91	2	97.9	Yes
PM/Metals	09/24/91	3	100	Yes
PM10/CPM	09/24/91	1	89.1	Yes
PM10/CPM	09/25/91	2	91.4	Yes
PM10/CPM	09/25/91	3	80.8	Yes
Aldehydes	09/23/91	1	98.8	Yes
Aldehydes	09/23/91	2	112	Yes b
Aldehydes	09/24/91	3	95.8	Yes
Aldehydes	09/25/91	4	96.0	Yes
PAH	09/19/91	1	107	Yes
PAH	09/19/91	2	106	Yes
PAH	09/20/91	3	105	Yes

a PM/Metals, Aldehydes and PAH test metals specify isokinetic sampling rates must be within 10 percent of 100 percent isokinetic. The PM10 test method allows ioskinetic sampling rates to be within 20 percent of 100 percent isokinetic.

b Marginally accepted because it did not exceed prescribed tolerances by a large amount.

verify the full calibration, a post-test calibration was performed. The full- and post-test calibration coefficients must be within 5 percent to meet Radian's internal QA/QC acceptance criterion. The results of the full- and post-test calibration check of the meter boxes used for manual sampling are presented in Table 6-4. The post-test calibration factor for the meter box used for PM/metals test runs was within the 5 percent criterion of the full calibration factor.

6.3.2 PM_{10}/CPM Sampling Quality Assurance

Post-test leak checks, isokinetic rates, and dry gas meter post-test calibrations for the PM_{10}/CPM test runs were within QA allowances and are presented in Tables 6-2, Table 6-3, and 6-4, respectively. Note that the isokinetic acceptance criterion for PM_{10}/CPM runs is more lenient than for other methods, allowing the average sampling rate to be within 20 percent of 100 percent isokinetic.

6.3.3 Aldehydes Sampling Quality Assurance

The post-test leak checks for the aldehydes sample trains met the QA acceptance criterion. The isokinetic rates for the aldehydes test runs deviated by no more than 5 percent of 100 percent for Runs 1, 3, and 4. Run 2 was slightly out of QA acceptance with an isokinetic rate of 112 percent. However, the run was accepted because the deviation from acceptable limits was so slight. Post-test leak check results, isokinetic values, and dry gas meter calibration results for the aldehydes runs are presented in Tables 6-2, 6-3, and 6-4, respectively.

6.3.4 Polynuclear Aromatic Hydrocarbon Sampling Quality Assurance

The post-test leak check results for the PAH trains are presented in Table 6-2. All the trains met the QA acceptance criterion. Isokinetic rates, presented in Table 6-3, varied no more than 6 percent of 100 percent, meeting the acceptance criterion. The post-test calibration results of the dry gas meter used for PAH sampling are presented in Table 6-4, and show that the calibration factor is within the QA allowance.

6.4 ANALYTICAL QUALITY ASSURANCE

The following sections briefly report QA parameters for the metals, PM_{10}/CPM, aldehydes, and PAH analytical results. Field blanks were collected for the PM/metals, PM_{10}/CPM, aldehydes, and PAH sampling trains. A train of each sample type was fully

Table 6-4
DRY GAS METER POST-TEST CALIBRATION RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Meter Box I.D.	Sample Trains	Full Calibration Factor	Post-Test Calibration Factor	Post-Test Deviation (%) a	Acceptable
N-30	PAH	0.9998	1.0218	2.20	Yes
N-32	Aldehydes	1.0006	1.0051	0.45	Yes
N-33	PM/Metals - PM10/CPM	0.9875	0.9788	-0.88	Yes

a [(Post-Test)-(Full)]/(Full)*100

prepared, taken to the sample location, leak checked, and then recovered. The analytical methods used for the flue gas samples are discussed fully in Section 5.

6.4.1 Metals Analytical Quality Assurance

Table 6-5 presents the results of the metals field blank analysis compared to the test run results. There was a noticeable contamination of certain metals in the blank. The front-half fraction was contaminated with all of the metals except Se, Ag, and Tl. The back-half fraction was contaminated with Mn, Ni, and P. The flue gas samples were blank corrected based on field blank results.

Table 6-6 presents the metals method blank results for the flue gas samples. Lead, Mn, and Ni were detected in the flue gas method blank at low levels. Because the flue gas samples were blank corrected for the field blank, no corrections were needed for the method blank.

Table 6-7 presents the method spike results for the metals analysis. All spiked recoveries for the front-half fraction, except for Ag, were within the QA allowance of ±20 percent of 100 percent. Barium, Cu, and P were slightly below the 20 percent acceptance in the back-half fraction, with 78.4 percent, 79.3 percent, and 78.2 percent, respectively. Poor Ag method blank recoveries may be due to the fact that silver nitrate solutions are light-sensitive and have a tendency to plate out on container walls. No spike corrections were applied.

6.4.2 PM_{10}/CPM Analytical Quality Assurance

Table 6-8 presents the results of the PM_{10}/CPM field blank analysis compared to the test run results. Amounts of PM_{10} were detected in the field blank within all of the separate analytical fractions. The total amount detected was 3.2 percent of the average total for the three runs. The PM_{10}/CPM samples were gravimetrically analyzed according to EPA Method 5 requirements. Sample jars were checked to determine if leakage occurred during shipment. The residue for the cyclone catch, filter catch, organic fraction, and inorganic fraction were weighed to within 0.5 mg. The weight determinations were conducted at least six hours apart. Weight gain for each fraction was reported to the nearest 0.1 mg. Water and methylene chloride blanks were analyzed with the samples. Blank correction was not required because the sum of the values for

Table 6-5

METALS FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

METALS	Run 1			Run 2			Run 3			Field Blank		
	Front Half (total ug)	Back Half (total ug)	Total (ug)	Front Half (total ug)	Back Half (total ug)	Total (ug)	Front Half (total ug)	Back Half (total ug)	Total (ug)	Front Half (total ug)	Back Half (total ug)	Total (ug)
Antimony	15.1	[1.69]	15.1	11.8	[1.58]	11.8	9.58	[1.58]	9.58	9.38	[1.68]	9.38
Arsenic	23.5	[0.423]	23.5	20.2	[0.421]	20.2	27.0	[0.421]	27.0	16.1	[0.447]	16.1
Barium	64.3	1.32	65.6	40.0	1.66	41.7	79.8	1.33	81.1	45.0	[0.112]	45.0
Beryllium	0.550	[0.106]	0.550	0.425	[0.105]	0.425	0.275	[0.105]	0.275	0.400	[0.112]	0.400
Cadmium	2.25	0.254	2.50	3.10	0.716	3.82	4.70	[0.210]	4.70	1.18	[0.224]	1.18
Chromium	101	1.26	102	105	3.07	108	27.8	1.30	29.1	24.5	[0.671]	24.5
Copper	23.0	1.07	24.1	19.4	2.89	22.3	37.0	[0.421]	37.0	2.60	[0.447]	2.60
Lead	31.0	0.355	31.4	28.3	3.50	31.8	36.8	0.969	37.8	8.13	[0.335]	8.13
Manganese	82.0	1.84	83.8	94.0	24.8	119	105	2.01	107	55.3	0.447	55.7
Nickel	138	2.59	141	99.8	4.69	104	13.3	2.04	15.3	15.3	0.369	15.7
Phosphorus	315	64.9	380	306	73.1	379	360	67.5	428	112	50.2	162
Selenium	[4.00]	[1.69]	[569]	[4.00]	[1.68]	[5.68]	[4.00]	[1.68]	[5.68]	[4.00]	[1.79]	[5.79]
Silver	5.18	[0.634]	5.18	7.45	[0.631]	7.45	5.05	[0.631]	5.05	[1.50]	[0.671]	[2.17]
Thallium	[25.0]	[10.6]	[35.6]	[25.0]	[10.5]	[35.5]	[25.0]	[10.5]	[35.5]	[25.0]	[11.2]	[36.2]
Zinc	249	14.4	263	268	24.3	292	316	5.84	322	69.5	[1.68]	69.5

[ ] = Minimum Detection Limit

Table 6-6
METALS FLUE GAS METHOD BLANK RESULTS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Metal	Flue Gas Method Blank	
	Front Half (total ug)	Impingers 1,2,3 (total ug)
Antimony	[1.50]	[1.59]
Arsenic	[0.400]	[0.424]
Barium	[0.100]	[0.106]
Beryllium	[0.100]	[0.106]
Cadmium	[0.200]	[0.212]
Chromium	[0.600]	[0.636]
Copper	[0.400]	[0.424]
Lead	[0.300]	0.744
Manganese	0.330	2.37
Nickel	[0.300]	0.572
Phosphorous	[30.0]	[31.8]
Selenium	[1.60]	[1.70]
Silver	[0.600]	[0.636]
Thallium	[10.0]	[10.6]
Zinc	(2.41)	[1.59]

[ ] = Minimum Detection Limit.
( ) = Estimated Value.

Table 6-7
METALS METHOD SPIKE RESULTS
MATHY CONSTRUTION COMPANY PLANT 26 (1991)

Metal	Method Spike (% rec)		Method Spike Duplicate (% rec)	
	Front Half	Impingers 1,2,3	Front Half	Impingers 1,2,3
Antimony	100%	86.8%	100%	96.8%
Arsenic	87.4%	86.0%	95.4%	97.8%
Barium	91.4%	88.2%	91.0%	78.4%
Beryllium	95.0%	83.4%	95.2%	93.4%
Cadmium	100%	98.5%	99.9%	88.5%
Chromium	99.6%	97.0%	99.4%	86.5%
Copper	93.5%	90.1%	93.9%	79.3%
Lead	97.8%	88.6%	102%	98.8%
Manganese	96.1%	94.0%	96.2%	84.9%
Nickel	99.9%	88.1%	100%	98.5%
Phosphorous	89.2%	78.2%	92.1%	86.5%
Selenium	99.0%	85.3%	99.6%	95.8%
Silver	74.5%	41.0%	24.7%	30.4%
Thallium	103%	94.7%	100%	97.5%
Zinc	103%	90.7%	104%	101%

Table 6–8

PM10/CPM FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Analysis	Run 1 (grams)	Run 2 (grams)	Run 3 (grams)	Field Blank (grams)
Filter	0.0070	0.0080	0.0086	0.0004
MeCl2	0.0048	0.0087	0.0035	0.0006
H2O	0.0036	0.0628	0.0287	0.0008
Cyclone	0.0207	0.0296	0.0288	0.0005
Total	0.0361	0.1091	0.0696	0.0023

the water blank and the methylene chloride blank was less than 3 mg, as specified in the test method.

6.4.3 Aldehydes Analytical Quality Assurance

Aldehydes field blank results are compared to the test run results in Table 6-9. Acetone was detected in the field blank at a noticeable level and formaldehyde was detected at a low level. The flue gas samples were blank-corrected.

The aldehydes method blank results for the flue gas samples are presented in Table 6-10. Acetone and formaldehyde were detected in the flue gas method blank at low levels. Because the samples were blank corrected for the field blank, they were not blank corrected for the method blank.

Table 6-11 presents the method spike results for the aldehyde analysis. All spike recoveries were within the ±20 percent of the 100 percent criterion except for acetophenone/o-tolualdehyde at 70 percent recovery, acrolein at 23 percent recovery, crotonaldehyde at 25 percent recovery, and quinone at 64 percent recovery. No spike corrections were applied.

6.4.4 Polynuclear Aromatic Hydrocarbon Results

Table 6-12 presents the PAH field blank results compared to the test run results. Naphthalene was detected in the field blank at an estimated level. The flue gas samples were blank-corrected.

The PAH method blank results for the flue gas samples are presented in Table 6-13. Naphthalene was detected in the flue gas method blank at estimated levels.

Table 6-14 presents the method spike results for the PAH analysis. All spike recoveries were within the QA criterion of ±20 percent of 100 percent except for nitrophenol, pentachlorophenol, and di-n-butylphthalate. No spike corrections were applied. Table 6-15 presents the PAH surrogate recovery results. 2-Fluorobiphenyl was out of the laboratory control limits for Run 1, Run 3, and the Method Spike, but nitrobenzene-d5 and terphenyl-d14 were within control limits for all sample runs.

6.5 CONTINUOUS EMISSION MONITORING QUALITY ASSURANCES

Flue gas was analyzed continuously for O_2/CO_2, CO, SO_2, NO_x, and THC using EPA Reference Methods 3A, 10, 6C, 7E, and 25A. Daily QA/QC procedures were

TABLE 6-9
ALDEHYDE FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Run 4 (total ug)	Field Blank (total ug)
Acetaldehyde	3530	1220	5880	2560	[11.0]
Acetone	2180	2280	2280	2710	396
Acetophenone/o-Tolualdehyde	[23.0]	[23.0]	[23.0]	[23.0]	[23.0]
Acrolein	107	[13.0]	[13.0]	134	[13.0]
Benzaldehyde	86.4	65.3	757	135	[21.0]
Butyraldehyde/Isobutyraldehyde	484	253	633	237	[18.0]
Crotonaldehyde	109	48.1	561	102	[18.0]
2,5-Dimethylbenzaldehyde	[24.0]	[24.0]	[24.0]	[24.0]	[24.0]
Formaldehyde	2070	2100	11800	3050	14.5
Hexanal	190	136	519	200	[20.0]
Isophorone	[18.0]	[18.0]	[18.0]	[18.0]	[18.0]
Isovaleraldehyde	75.1	[18.0]	138	85.3	[18.0]
MIBK/p-Tolualdehyde	[23.0]	[23.0]	[23.0]	[23.0]	[23.0]
Valeraldehyde	89.1	62.9	344	134	[18.0]

[ ] = Minimum Detection Limit

Table 6-10
ALDEHYDE FLUE GAS METHOD BLANK RESULTS MATHY CONTRUCTION COMPANY PLANT 26 (1991)

Compound	Flue Gas Method Blank (total ug)
Acetaldehyde	[0.580]
Acetone	2.74
Acetophenone/o-Tolualdehyde	[1.20]
Acrolein	[0.710]
Benzaldehyde	[1.10]
Butyraldehyde/Isobutyraldehyde	[0.940]
Crotonaldehyde	[0.940]
2,5-Dimethylbenzaldehyde	[1.30]
Formaldehyde	1.24
Hexanal	[1.10]
Isophorone	[0.940]
Isovaleraldehyde	[0.980]
MIBK/p-Tolualdehyde	[[1.20]
Methyl Ethyl Ketone	[0.940]
Propionaldehyde	[0.730]
Quinone	[0.940]
m-Tolualdehyde	[1.20]
Valeraldehyde	[0.980]

[ ] = Minimum Detection Limit.

Table 6-11
ALDEHYDE METHOD SPIKE RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Method Spike (% rec)
Acetaldehyde	90.0%
Acetone	83.0%
Acetophenone/o-Tolualdehyde	70.0%
Acrolein	23.0%
Benzaldehyde	119%
Butyraldehyde/Isobutyraldehyde	NA
Crotonaldehyde	25.0%
2,5-Dimethylbenzaldehyde	85.0%
Formaldehyde	87.0%
Hexanal	117%
Isophorone	91.0%
Isovaleraldehyde	NS
MIBK/p-Tolualdehyde	106%
Methyl Ethyl Ketone	NA
Propionaldehyde	107%
Quinone	64.0%
m-Tolualdehyde	93.0%
Valeraldehyde	82.0%

NS = Not Spiked
NA = Not Analyzed

Table 6-12
PAH FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ng)	Field Blank (total ug)
Acenaphthylene	[10000]	[10000]	[10000]	[50.0]
Acenaphthlene	[10000]	[10000]	[10000]	[50.0]
Anthracene	[10000]	[10000]	[10000]	[50.0]
Benzo(a)anthracene	[10000]	[10000]	[10000]	[50.0]
Benzo(a)pyrene	[10000]	[10000]	[10000]	[50.0]
Benzo(b)fluoranthene	[10000]	[10000]	[10000]	[50.0]
Benzo(e)pyrene	[10000]	[10000]	[10000]	[50.0]
Benzo(g,h,i)perylene	[10000]	[10000]	[10000]	[50.0]
Benzo(k)fluoranthene	[10000]	[10000]	[10000]	[50.0]
2-Chloronapthalene	[10000]	[10000]	[10000]	[50.0]
Chrysene	[10000]	[10000]	[10000]	[50.0]
Dibenz(a,h)anthracene	[10000]	[10000]	[10000]	[50.0]
Dibenzofuran	[10000]	[10000]	[10000]	[50.0]
7,12-Dimethylbenz(a)anthracene	[20000]	[20000]	[20000]	[100]
Fluoranthene	[10000]	[10000]	[10000]	[50.0]
Fluorene	[10000]	[10000]	[10000]	[50.0]
Indeno(1,2,3-cd)pyrene	[10000]	[10000]	[10000]	[50.0]
2-Methylnaphthalene	[10000]	[10000]	[10000]	[50.0]
Naphthalene	1810	3010	1410	(87.4)
Perylene	[10000]	[10000]	[10000]	[50.0]
Phenanthrene	[10000]	[10000]	[10000]	[50.0]
Pyrene	[10000]	[10000]	[10000]	[50.0]

( ) = Estimated Values

NOTE: PAH values have been blank corrected.

Table 6-13
PAH FLUE GAS METHOD BLANK RESULTS
MATHY CONSRTUCTION COMPANY PLANT 26 (1991)

Compound	Flue Gas Method Blank (total ug)
Acenaphthylene	[50.0]
Acenaphthlene	[50.0]
Anthracene	[50.0]
Benzo(a)anthracene	[50.0]
Benzo(a)pyrene	[50.0]
Benzo(b)fluoranthene	[50.0]
Benzo(e)pyrene	[50.0]
Benzo(g,h,i)perylene	[50.0]
Benzo(k)fluoranthene	[50.0]
2-Chloronapthalene	[50.0]
Chrysene	[50.0]
Dibenz(a,h)anthracene	[50.0]
Dibenzofuran	[50.0]
7,12-Dimethylbenz(a)anthracene	[100]
Fluoranthene	[50.0]
Fluorene	[50.0]
Indeno(1,2,3-cd)pyrene	[50.0]
2-Methylnaphthalene	[50.0]
Naphthalene	(0.370)
Perylene	[50.0]
Phenanthrene	[50.0]
Pyrene	[50.0]

[ ] = Minimum Detection Limit.

( ) = Estimated Values

Table 6-14
PAH METHOD SPIKE RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Compound	Method Spike (% rec)
Phenol	84.8%
2-Chlorophenol	94.2%
1,4-Dichlorobenzene	95.6%
N-Nitroso-di-n-propylamine	104%
1,2,4-Trichlorobenzene	109%
4-Chloro-3-methylphenol	99.9%
2,4-Dinitrotoluene	98.4%
Pentachlorophenol	26.6%
Di-n-butylphthalate	2.15%
4-Nitrophenol	53.0%

Table 6-15
PAH SURROGATE RECOVERY RESULTS
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

COMPOUND	Run 1 (% rec)	Run 2 (% rec)	Run 3 (% rec)	Field Blank (% rec)	Method Blank (% rec)	Method Spike (% rec)	Control Limits
2-Fluorobiphenyl	132%	107%	135%	59.4%	99.2%	143%	30% to 115%
Nitrobenzene-d5	102%	102%	122%	39.4%	81.8%	105%	23% to 120%
Terphenyl-d14	85.1%	90.3%	85.8%	44.8%	101%	127%	18% to 137%

followed in accordance with the QA/QC guidelines in the reference methods and Radian standard operating procedures. These procedures are fully detailed in the site-specific test plan. A summary of the QA/QC parameters and results is provided in this section. Deviations from the test plan and/or problems encountered during the test program are also discussed.

6.5.1 Calibration and Drift Assessments

Continuous monitoring instruments were calibrated at the beginning of the test period on a two-point basis using a zero gas (N_2) and a high-range span gas. A mid-range gas was analyzed with no adjustment permitted as a QC check at least once on site. The observed mid-range QC gas concentration had to be within ±2 percent of full scale for the linearity check to be considered acceptable. The results of this check for the different instruments are presented in Tables 6-16 through 6-21.

In addition to conducting the linearity check, instrument drift was also determined for each analyzer on a daily basis. Typically, the mid-range gas was analyzed at mid-day and/or at the end of each test day to determine an "inter-run" drift; however, the span gas was used in some cases in order to conserve gases that were available in limited quantities, or if the span gas was closer to the observed concentrations. Because production runs were limited, drift check was not determined between every manual run in order to allow for completion of as many manual method tests as possible while the plant was actually on-line.

The inter-run instrument drift value was calculated as a difference on a percent scale basis by comparing the current observed response to the previous response. The instrument drift over the entire test program was calculated similarly, except that the final span-gas observed response was compared to the initial span-gas observed response. These inter-run and overall drift values are also provided in Tables 6-16 through 6-21. The allowable drift of ±3 percent of full scale was met in all cases except for one inter-run check of the NO_x analyzer; however, the overall drift for this analyzer over the entire test period was determined as -0.4 percent, which is within ±3 percent limit.

Table 6–16

METHOD 3A OXYGEN ANALYZER AND DRIFT SUMMARY

MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: Thermox WDGIII				Serial Number: 35983–1		
Full Scale Concentration: 25 %V						
Date	Time	QC Gas	Certified Gas Conc. (%V)	Observed Gas Conc. (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.0
9/23/91	08:12	Span:	18.0	18.0	0.0	0.0
Inter–run Drift Summary b						
9/23/91	11:06	Mid:	7.99	7.7	–0.3	–1.2
9/23/91	12:40	Mid:	7.99	7.7	0.0	0.0
9/23/91	14:45	Mid:	7.99	8.1	0.4	1.6
9/23/91	16:46	Mid:	7.99	7.5	–0.6	–2.4
9/24/91	15:12	Mid:	7.99	7.8	0.3	1.0
9/25/91	15:55	Mid:	7.99	7.4	–0.3	–1.4
Test Period Drift Summary c						
9/25/91	15:55	Span:	18.0	18.4	0.4	1.6

a Multipoint Linearity - Difference Percent Scale = (observed conc - certified conc)/full scale * 100%.

b Inter–run Drift - Difference Percent Scale = (current conc - previous observed conc)/full scale * 100%.

c Drift Summary - Difference Percent Scale = (final observed conc - initial observed conc)/full scale * 100%.

Table 6-17
METHOD 3A CARBON DIOXIDE ANALYZER AND DRIFT SUMMARY
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: Beckman 865 Serial Number: 0103778

Full Scale Concentration: 20 %V

Date	Time	QC Gas	Certified Gas Conc. (%V)	Observed Gas Conc. (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.0
9/23/91	08:12	Span:	17.0	17.0	0.0	0.0
Inter-run Drift Summary b						
9/23/91	11:06	Mid:	9.91	10.3	0.4	2.0
9/23/91	12:40	Mid:	9.91	10.3	0.0	0.0
9/23/91	14:45	Mid:	9.91	10.4	0.1	0.5
9/23/91	16:46	Mid:	9.91	9.8	-0.6	-3.0
9/24/91	15:12	Mid:	9.91	10.2	0.4	2.0
9/25/91	15:55	Mid:	9.91	10.5	0.3	1.5
Test Period Drift Summary c						
9/25/91	15:55	Span:	17.0	17.2	0.2	1.0

a Multipoint Linearity - Difference Percent Scale = (observed conc - certified conc)/full scale * 100%.

b Inter-run Drift - Difference Percent Scale = (current conc - previous observed conc)/full scale * 100%.

c Drift Summary - Difference Percent Scale = (final observed conc - initial observed conc)/full scale * 100%.

Table 6–18

METHOD 6C SULFER DIOXIDE ANALYZER AND DRIFT SUMMARY

MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: Western Research 721AT2			Serial Number: 90–721AT2–7654–1			
Full Scale Concentration: 500 ppmV						
Date	Time	QC Gas	Certified Gas Conc. (ppmV)	Observed Gas Conc. (ppmV)	Difference (ppmV)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.0
9/23/91	08:12	Span:	295.0	295.0	0.0	0.0
Inter–run Drift Summary b						
9/23/91	11:06	Span:	295.0	295.0	0.0	0.0
9/23/91	12:40	Span:	295.0	302.0	7.0	1.4
9/23/91	16:46	Mid:	98.0	101.0	-3.0	–0.6
9/24/91	15:12	Mid:	98.0	105.0	4.0	0.8
9/25/91	15:55	Mid:	98.0	106.4	1.4	0.3
Test Period Drift Summary c						
9/25/91	15:55	Span:	295.0	293.0	-2.0	–0.4

a Multipoint Linearity - Difference Percent Scale = (observed conc – certified conc)/full scale • 100%.

b Inter-run Drift - Difference Percent Scale = (current conc – previous observed conc)/full scale * 100%.

c Drift Summary - Difference Percent Scale = (final observed conc – initial observed conc)/full scale * 100%.

Table 6–19

METHOD 7E NITROGEN OXIDES ANALYZER AND DRIFT SUMMARY
MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: TECO 10AR				Serial Number: 25558–221		
Full Scale Concentration: 250 ppmV						
Date	Time	QC Gas	Certified Gas Conc. (ppmV)	Observed Gas Conc. (ppmV)	Difference (ppmV)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.0
9/23/91	08:12	Span:	201.0	201.0	0.0	0.0
Inter–run Drift Summary b						
9/23/91	11:06	Span:	201.0	192.0	-9.0	-3.6
9/23/91	12:40	Span:	201.0	201.0	9.0	3.6
9/23/91	16:46	Mid:	97.0	99.0	2.0	0.8
9/24/91	15:12	Mid:	97.0	96.0	-3.0	-1.2
9/25/91	15:55	Mid:	97.0	104.0	8.0	3.2
Test Period Drift Summary c						
9/25/91	15:55	Span:	201.0	200.0	-1.0	-0.4

a Multipoint Linearity – Difference Percent Scale = (observed conc – certified conc)/full scale * 100%.

b Inter–run Drift – Difference Percent Scale = (current conc – previous observed conc)/full scale • 100%.

c Drift Summary – Difference Percent Scale = (final observed conc – initial observed conc)/full scale • 100%.

Table 6–20

METHOD 10 CARBON MONOXIDE ANALYZER AND DRIFT SUMMARY MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: TECO 48				Serial Number: 4829758–236		
Full Scale Concentration: 500 ppmV						
Date	Time	QC Gas	Certified Gas Conc. (ppmV)	Observed Gas Conc. (ppmV)	Difference (ppmV)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.0
9/23/91	08:12	Span:	474.0	474.0	0.0	0.0
Inter–run Drift Summary b						
9/23/91	12:40	Span:	474.0	482.0	8.0	1.6
9/23/91	14:45	Mid:	92.1	92.5	0.4	0.1
9/23/91	16:46	Mid:	92.1	93.7	1.2	0.2
9/24/91	15:12	Mid:	92.1	92.1	-1.6	-0.3
9/25/91	15:55	Mid:	92.1	90.0	-2.1	-0.4
Test Period Drift Summary c						
9/25/91	15:55	Span:	474.0	466.0	-8.0	-1.6

a Multipoint Linearity - Difference Percent Scale = (observed conc - certified conc)/full scale * 100%.

b Inter-run Drift - Difference Percent Scale = (current conc - previous observed conc)/full scale * 100%.

c Drift Summary - Difference Percent Scale = (final observed conc - initial observed conc)/full scale * 100%.

Table 6–21

METHOD 25A TOTAL HYDROCARBON ANALYZER AND DRIFT SUMMARY MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Instrument: Ratfisch 55				Serial Number: 212291		
Full Scale Concentration: 1000 ppmV						
Date	Time	QC Gas	Certified Gas Conc. (ppmV)	Observed Gas Conc. (ppmV)	Difference (ppmV)	Difference Percent Scale (%)
Multipoint Linearity a						
9/23/91	08:12	Zero:	0.0	0.0	0.0	0.00
9/23/91	08:12	Mid:	95.3	95.0	0.3	0.03
9/24/91	11:58	Span:	810	810	0.0	0.00
Inter-run Drift Summary b						
9/23/91	12:40	Mid:	95.3	94.5	-0.5	-0.05
9/23/91	16:46	Mid:	95.3	102.5	8.0	0.80
9/24/91	15:12	Span:	810	811.0	1.0	0.10
9/25/91	15:55	Span:	810	817.0	6.0	0.60
Test Period Drift Summary c						
9/25/91	15:55	Mid:	95.3	97.6	2.6	0.26

a Multipoint Linearity - Difference Percent Scale = (observed conc - certified conc)/full scale • 100%.

b Inter-run Drift - Difference Percent Scale = (current conc - previous observed conc)/full scale * 100%.

c Drift Summary - Difference Percent Scale = (final observed conc - initial observed conc)/full scale • 100%.

6.5.2 Line Bias Checks

Radian performed all multi-point and QC calibrations through the entire sampling system. A three-way valve was located between the reference method CEM probe and the heat-traced line. This valve was shut during calibration and QC drift checks, and the standard gases were directed from the gas cylinder through the heat-traced line to the analyzer probe and back before the gas was directed to the CEM analyzers. This procedure eliminated the need for performing the line bias checks described in Methods 3A, 7E, and the test plan.

6.5.3 Leak Checks

Because Radian performed all calibrations through the entire sampling system, leak checks were incorporated in each calibration. The criterion used for this test was an O_2 response to a zero gas of less than 0.5 percent O_2. All leak checks performed at this test site met this criterion.

6.6 GAS CHROMATOGRAPHY QUALITY ASSURANCE

EPA Method 18 analysis of the flue gas was performed using a GC to separate the hydrocarbon (C_1-C_6) species in the gas stream. At the beginning of each test day, and prior to sampling the flue gas, the GC was calibrated with standard gas mixtures containing each hydrocarbon (methane, ethane, propane, butane, pentane, hexane) and an instrument response factor for each hydrocarbon was determined. Response factors for each hydrocarbon were determined again at the end of each test day after sampling was completed. In this way, a daily calibration drift was determined for these compounds. Additional calibrations were completed for benzene, toluene, and xylene (BTX). The calibration drift values are shown in Table 6-22. A Post-test Response was not conducted on the second day of testing. Most of the drift values were within the QA allowance criterion of 10 percent. The compounds that did not meet this criteria were benzene on the first day of testing, and pentane, hexane, ethylbenzene, and xylene on the third day of testing.

Table 6-22

GC RESPONSE FACTOR DRIFT VALUES

MATHY CONSTRUCTION COMPANY PLANT 26 (1991)

Date	9/23/91			9/24/91			9/25/91		
Compound	Pre-Test Response Factor a	Post-Test Response Factor a	Drift (%) b	Pre-Test Response Factor a	Post-Test Response Factor a	Drift (%) b	Pre-Test Response Factor a	Post-Test Response Factor a	Drift (%) b
Methane	3.7905E-05	4.0398E-05	-6.58	3.8790E-05	ND	ND	3.2275E-05	2.9360E-05	9.03
Ethane	1.8466E-05	1.8494E-05	-0.15	2.0130E-05	ND	ND	1.5665E-05	1.4370E-05	8.27
Propane	1.2269E-05	1.2237E-05	0.26	1.2860E-05	ND	ND	1.0435E-05	9.5230E-06	8.74
Butane	8.9445E-06	9.1091E-06	-1.34	9.1560E-06	ND	ND	7.7740E-06	7.0230E-06	9.66
Pentane	7.2191E-06	7.2154E-06	0.05	7.3835E-06	ND	ND	6.3380E-06	5.6400E-06	11.01
Hexane	6.2334E-06	6.3088E-06	-1.21	6.4075E-06	ND	ND	5.4855E-06	4.8820E-06	11.00
Benzene	6.2892E-06	5.5769E-06	11.33	6.1030E-06	ND	ND	5.1640E-06	5.3010E-06	-2.65
Toluene	5.1461E-06	ND	ND	2.9780E-06	ND	ND	5.9850E-06	6.4060E-06	-7.03
Ethylbenzene	4.3137E-06	3.9281E-06	8.94	4.8500E-06	ND	ND	4.0110E-06	4.4210E-06	-10.22
Xylene	3.9692E-06	3.7633E-06	5.19	4.9210E-06	ND	ND	3.8220E-06	4.4090E-06	-15.36

ND = Not Determined

a Response Factor = Calibration Gas Concentration (ppm)/Peak Area Count

b Drift Percent = ((Post-Test Response Factor - Pre-Test Response Factor)/Post-Test Response Factor) x 100

APPENDIX A

EMISSIONS TESTING FIELD DATA SHEETS

A.1	PM/Metals
A.2	PM_{10}/CPM
A.3	Aldehydes
A.4	PAH

APPENDIX A.1

PM/METALS

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page 1 of

SAMPLING LOCATION Stack RUN NO. Metals-1
DATE 9-23-91 TIME START 0830 TIME FINISH TEST DURATION 120 min.
DUCT DIMENSIONS 43 x 38 DIAMETER ft INITIAL LEAK RATE .010 at 10" cfm
y PTCF 10006 DGMCF 1.75 NOZZLE DIA. .191 inches FINAL LEAK RATE .012 at 5" cfm
BAR PRESS " Hg
STATIC PRESS -.17 " H2O OPERATOR JBP
~~Meter no.~~

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	
	0830	80.891								2		
A1	0835	82.3	.48	.21	324	51	50	274	I	46	1	
2	840	83.5	.44	.19	322	51	50	258		44	1	
3	845	84.9	.38	.16	322	53	51	247		43	1	
4	850	85.9	.25	.12	318	55	52	240		43	1	
5	855	86.886	.25	.12	318	55	52	227				
STOP	~~STOP~~ 856	"										
B1	901	88.2	.64	.30	326	59	55	273		45	1	
2	906	89.6	.53	.25	320	58	54	248		42	1	
3	911	91.3	.68	.32	326	62	57	258		47	1	
4	916	93.2	.61	.29	326	63	57	259		44	1	
5	921	94.752	.63	.30	321	65	60	266		45	1	
STOP	922	"										
C1	927	96.5	.76	.37	333	68	62	228		45	1	
2	932	98.4	.80	.40	333	68	62	228		45	1	
3	937	100.3	.87	.42	337	69	64	260		47	1	
4	942	102.1	.84	.40	332	71	65	245		44	1	
5	947	103.999	.90	.43	324	71	65	241		47	1	
STOP	948	"										
D1	~~1003~~ 953	106.0	.84	.41	331	72	67	239		45	1	
2	~~1008~~ 958	107.8	.81	~~.44~~ .39	330	72	68	228		45	1	
3	1003	109.7	.93	.45	332	74	69	249		47	1	
4	1008	111.7	.92	.45	330	75	70	257		48	1	
5	1013	113.497	.82	.40	327	75	70	252		47	1	
STOP	1014	"										
E1	1019	115.0	.75	.36	326	75	70	245		48	1	
2	1024	116.9	.66	.31	327	76	72	268		49	1	
3	1029	118.7	.79	.38	325	79	73	235		46	1	
4	1034	120.6	.85	.42	321	78	72	226		47	1	
5	1039	122.718	.87	.43	320	77	73	233		42	1	

4.6

-.476

CONSOLE # N-32
FILTER # 5 (0.6406)
AMBIENT TEMP. 60

PROBE LENGTH 6 glass
LINER MATERIAL

WEATHER Clear

REMARKS V_{ACT} = 41.83 $\sqrt{\Delta P}_{AVG}$ = 0.822
T_M = 65°
T_S = 326°F V_{STD} = 41.162 ft³

.74
.66

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	MT	—	MOD G-S	Final Initial	859.3 ~~847.6~~ JBC VLL ~~452.9~~ 495.6	Weight Gain	363.7 ~~847.6~~ JBC
2	HNO_3/H_2O_2	200	MOD G-S	Final Initial	638.7 635.0	Weight Gain	3.7
3	HNO_3/H_2O_2	200	MOD G-S	Final Initial	637.8 620.8	Weight Gain	17
4	MT	—	MOD G-S	Final Initial	490.4 490.1	Weight Gain	0.3
5	Silica Gel	200(g)	MOD G-S	Final Initial	742.3 731.4	Weight Gain	10.9
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 395.6 VLL grms

Analyst ________

Vf = Final Meter Volume = ________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = ________ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ ft3})(\_\_\_\_\text{ "Hg})}{(\_\_\_\_\text{ R})} = \_\_\_\_\text{ ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____g) = ______ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std)} + \text{Vm(std}} = \frac{\_\_\_\_\text{ ft3}}{\_\_\_\_\text{ ft3} + \_\_\_\_\text{ ft3}} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. Metals-2

DATE 9-23-91 TIME START ________ TIME FINISH ________ TEST DURATION ________ min.

DUCT DIMENSIONS 43 x 35 DIAMETER ________ ft INITIAL LEAK RATE 0.016 at 9"Hg cfm

✓ PTCF 1.0006 DGMCF 1.75 NOZZLE DIA. .191 inches FINAL LEAK RATE 0.014 at 5"Hg cfm

BAR PRESS ________ " Hg

STATIC PRESS -0.17 " H2O OPERATOR JBP

~~47~~ .47

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	______
						Inlet	Outlet					
	1355	26.6952										
E1	1400	28.6	.69	.34	315	69	71	271	8	51	1	
2	1405	30.3	.72	.36	319	71	73	307		50	1	
3	1410	32.1	.80	.38	322	71	74	323		48	1	
4	1415	33.8	.73	.36	318	72	75	252		50	1	
5	1420	35.553	.84	.40	325	72	76	230		47	1	
STOP	1421	"										
D1	1426	37.3	.76	.37	336	72	75	255		46	1	
2	1431	38.9	.82	.39	331	73	76	302		46	1	
3	1436	40.8	.91	.44	333	73	77	290		46	1	
4	1441	42.6	.89	.43	328	73	76	278		46	1	
5	1446	44.540	.97	.46	328	73	77	280		46	1	
STOP	1447	"										
C1	1452	46.3	.74	.37	324	73	75	286		47	1	
2	1457	48.0	.73	.36	325	75	78	289		48	1	
3	1502	49.9	.84	.40	324	75	79	262		48	1	
4	1507	51.6	.80	.38	328	75	81	280		48	1	
5	1512	53.269	.82	.39	322	75	81	275		48	1	
STOP	1514	"										
B1	1519	54.8	.59	.29	319	75	77	275		51	1	
2	1524	56.4	.60	.29	320	76	79	264		49	1	
3	1529	58.0	.63	.31	323	76	81	262		48	1	
4	1534	59.4	.52	.26	325	76	80	271		48	1	
5	1539	61.064	.62	.31	323	77	81	277		49	1	
STOP	1541	"										
A1	1546	62.5	.48	.22	324	77	80	295		56	1	
2	1551	63.9	.46	.22	323	76	80	280		50	1	
3	1556	65.2	.38	.18	317	76	80	283		47	1	
4	1601	66.2	.25	.12	319	76	79	283		48	1	
5	1606	67.373	.26	.12	310	76	79	289		51	1	

CONSOLE # N-32 PROBE LENGTH 8

FILTER # 14 (0.6142) LINER MATERIAL Glass

AMBIENT TEMP. 60

WEATHER ________

REMARKS ________

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams	
1	MT	—	M006-S	Final 774.9 Initial 475.4	Weight Gain 299.5
2	HNO_3/H_2O_2	200	M006-S	Final 707.1 Initial 664.7	Weight Gain 42.4
3	HNO_3/H_2O_2	200	M006-S	Final 665.2 Initial 643.8	Weight Gain 21.4
4	MT	—	M006-S	Final 453.3 Initial 451.8	Weight Gain 1.5
5	Silica Gel	~200g	M006-S	Final 675.6 Initial 667.8	Weight Gain 7.8
6	______	______	______	Final ______ Initial ______	Weight Gain ______

Total Impinger Weight Gain (Wc) 372.6 grms

Analyst__________

Vf = Final Meter Volume = ____________ft3

Vi = Initial Meter Volume= _________ ft3

DGMCF = Dry Gas Meter Correction Factor = _________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = _________ ft3

Tm = Average Meter Temp. = F+460 = _______ R

Pm = Meter Pressure (Barometric Pressure) = _______ in. Hg

%CO ________ %N2 ________
%CO2 ________ %H2 ________
%O2 ________ %CH ________

CONDENSED WATER __________gms
FILTER WT. GAIN __________gms
PROBE WASH WT. GAIN _________gms

$$Vm\,(std) = \frac{(17.64)(Vm)(Pm)}{Tm} = \frac{(17.64)(\_\_\_\_\,ft3)(\_\_\_\_\,"Hg)}{(\_\_\_\_\,R)} = \_\_\_\_\,ft3$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(_____g) = _______ft3

$$Bws = Moisture\ Fraction = \frac{Vw(std)}{Vw(std) + Vm(std} = \frac{\_\_\_\_\,ft3}{\_\_\_\_\,ft3 + \_\_\_\_\,ft3} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. Metals - 3
DATE 9-24-91 TIME START 1236 TIME FINISH ________ TEST DURATION 120 min.
DUCT DIMENSIONS 43" x 38" DIAMETER ________ ft INITIAL LEAK RATE 0.009 at 10" cfm
1.0006 PTCF ~~.9875~~ DGMCF 2001.75 NOZZLE DIA. .191 inches FINAL LEAK RATE 0.012 at 6" cfm
BAR PRESS 29.4 " Hg
STATIC PRESS -0.58 " H2O OPERATOR JB,P

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	______
	1236	25.730										
E1	1241	27.4	.72	.39	323	68	64	289		47	1	
2	1246	29.0	.73	.39	306	69	67	301		48	1	
3	1251	30.9	.72	.39	328	71	69	298		49	1	
4	1256	32.8	.90	.50	326	71	68	299		52	1	
5	1301	34.512	.83	.45	324	70	70	289		52	1	
STOP	1302	"										
D1	1307	36.4	.70	.38	320	71	67	296		54	1	
2	1312	38.1	.75	.41	334	72	68	297		53	1	
3	1317	39.8	.82	.45	336	72	68	297		55	1	
~~4~~	~~1322~~	40.492	.80	.44	338	72	70	298		55	1	
~~5~~	~~1327~~ 1336.5	"										
4	1340	41.9	.94	.54	298	73	72	293		57	1	
5	1345	43.697	.94	.41	302	73	72	300		56	1	
STOP	1346	"										
C1	1351	45.4	.72	.39	329	73	73	298		56	1	
2	1356	47.1	.66	.36	334	74	72	302		57	1	
3	1401	48.9	.75	.40	338	73	71	297		58	1	
4	1406	50.8	.83	.46	336	72	69	300		53	1	
5	1411	52.33	.68	.37	334	75	71	299		52	1	
STOP	1412	"										
B1	1417	53.9	.65	.34	337	76	71	300		53	1	
2	1422	55.5	.60	.32	334	77	72	300		52	1	
3	1427	56.9	.56	.30	330	74	71	297		50	1	
4	1432	58.6	.57	.31	329	75	71	299		51	1	
5	1437	60.23	.60	.33	324	70	72	295		49	1	
STOP	1438	"										
A1	1443	61.8	.48	.27	325	69	72	296		52	1	
2	1448	63.0	.44	.25	327	69	72	299		55	1	
3	1453	64.0	.39	.22	328	74	70	300		54	1	
4	1458	65.3	.26	.15	327	77	73	300		55	1	
5	1503	66.510	.26	.15	324	77	74	302		55	1	

.54

1318.54

CONSOLE # N-30 PROBE LENGTH 8
FILTER # 18 (0.6183) LINER MATERIAL glass
AMBIENT TEMP. 65

WEATHER P. Cloudy

REMARKS

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	MT		MOD G-S	Final Initial	753.1 469.8	Weight Gain	283.3
2	HNO_3/H_2O_2	200	MOD G-S	Final Initial	708.9 662.0	Weight Gain	46.9
3	HNO_3/H_2O_2	200	MOD G-S	Final Initial	699.2 695.2	Weight Gain	4.0
4	MT	—	MOD G-S	Final Initial	485.1 484.8	Weight Gain	0.3
5	Silica Gel	200(g)	MOD G-S	Final Initial	659.5 652.6	Weight Gain	6.9
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 341.4 grms

Analyst__________

Vf = Final Meter Volume = __________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = _______ R

Pm = Meter Pressure (Barometric Pressure) = ______ in. Hg

%CO ________ %N2 ________

%CO2 ________ %H2 ________

%O2 ________ %CH ________

CONDENSED WATER _________gms

FILTER WT. GAIN _________gms

PROBE WASH WT. GAIN _________gms

$$Vm\ (std) = \frac{(17.64)(Vm)(Pm)}{Tm} = \frac{(17.64)(\_\_\_\_ ft3)(\_\_\_\_ "Hg)}{(\_\_\_\_ R)} = \_\_\_\_\_ ft3$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____g) = ______ft3

$$Bws = Moisture\ Fraction = \frac{Vw(std)}{Vw(std) + Vm(std} = \frac{\_\_\_\_ ft3}{\_\_\_\_ ft3 + \_\_\_\_ ft3} = \_\_\_\_\_$$

Revision Date 2/91

APPENDIX A.2

PM_{10}/CPM

PLANT Mattay #20
DATE 9-24
SAMPLING LOCATION STACK
SAMPLE TYPE PM-10
RUN NUMBER 1
OPERATOR JBC
AMBIENT TEMPERATURE -56
BAROMETRIC PRESSURE 29.24
STATIC PRESSURE, (P_s) ~~-0.45~~ -0.45
FILTER NUMBER (s)

PROBE LENGTH AND TYPE 8' Glass
NOZZLE I.D. 0.175
ASSUMED MOISTURE, % 31
SAMPLE BOX NUMBER
METER BOX NUMBER N-33
METER $\Delta H_@$ 2.0
C FACTOR 0.9575
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE Δp

$\Delta H = 0.28$

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY ______ MINUTES

Leak √ 0.01 @ 10'

TRAVERSE POINT NUMBER	SAMPLING TIME, ~~min~~ Sec / CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET $(T_{m\ in})$, °F	DRY GAS METER TEMPERATURE OUTLET $(T_{m\ out})$, °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F	On
	~1005	2.490										
E 1	147	7.9	.73	.28		316	54	52	1	282	48	0.175
2	154	8.4	.8	"		320	54	52	1	291	48	'
3	151	9.3	.77	"		319	55	52	1	286	49	"
4	165	10.0	.92	"		312	54	52	1	282	47	"
5	167	10.89	.44	"		315	57	54	1	278	49	"
D 1	167	11.70	.94	.26		320	56	54	1	265	49	"
2	169	12.55	.97	"		324	57	54	1	285	48	"
3	174	13.75	1.02	"		324	58	54	1	280	47	"
4	176	14.40	1.05	"		330	60	55	1	260	49	"
5	163	15.13	.90	"		329	59	55	1	282	51	"
C 1	163	15.9	.9	"		334	57	55	1	261	52	"
2	174	16.67	1.02	"		336	61	56	1	292	52	"
3	180	17.43	1.1	.21		338	63	58	1	286	52	"
4	168	18.1	.96	.21		335	64	58	1	282	49	"
~~5~~	~~missed this point will catch on end of run.~~					~~337~~				~~277~~		
B 1	149	18.97	.75	.21		335	63	59	1	266	50	"
2	148	19.55	.74	"		337	65	60	1	291	47	"
3	149	20.25	.75	"		335	65	60	1	283	48	"
4	154	21.0	.8	"		335	66	61	1	285	47	"
5	157	21.675	.83	"		336	67	61	1	284	47	"

COMMENTS: Limited by length of Nozzle. if ΔPs > 0.95 need 0.158 nozzle but it won't fit in port. will use 0.175 anyway.

V_m

EPA (Dur) 235
4/72

Diameter
NO2
0.175

TRAVERSE POINT NUMBER	SAMPLING TIME ~~min sec~~ / CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET $(T_{m_{in}})$, °F	DRY GAS METER TEMPERATURE OUTLET $(T_{m_{out}})$, °F	PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE °F
	~1104	21.675									
A 1	140	22.217	.66	.21		328	65	62	1	273	48
2	123	22.66	.91	"		330	66	61		287	48
3	~~119~~ ~~20~~ 15sec	22.764	.48			326	66	62	1	287	49
~~4~~	Stop process upset										
~~5~~											
~~C 4 5~~											
restart	~1115 hrs										
		22.764	.57								
A 3	115	23.3	.57	.21		325	65	62	1	283	53
4	115	23.81	.45	"		325	67	62	1	248	49
5	104	24.275	.37	"		323	68	63	1	290	49
C 5	168	25.020	.95	"		326	68	63	1	270	54
stop											

ΔH avg = 0.238

Vmet = 17.53
Ts = 328
Tm = 60°
Vstd = 17.181
Y.Mfw = 0.897

Vel = 65.6 fps
Time = 64.25

Water = 148.6 g

Bwo = 28.9%

%I = 88.14

$Q_{s_{sf}}$ = 0.267

Q_s = 0.568

D_{50} = 10.68 uM

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy # 26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. PM10 - 1

DATE ________ TIME START ________ TIME FINISH ________ TEST DURATION ________ min.

DUCT DIMENSIONS ________ X ________ DIAMETER ________ ft INITIAL LEAK RATE ________ cfm

PTCF ________ DGMCF ________ NOZZLE DIA. ________ inches FINAL LEAK RATE ________ cfm

BAR PRESS ________ " Hg

STATIC PRESS ________ " H2O OPERATOR ________

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	____

CONSOLE # ________ PROBE LENGTH ________

FILTER # PM10-19 (0.2152) LINER MATERIAL ________

AMBIENT TEMP. ________

WEATHER ________

REMARKS ________

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DI H_2O	100	MOD G-S	Final	739.2	Weight Gain	131.7
				Initial	607.5		
2	DI H_2O	100	MOD G-S	Final	645.7	Weight Gain	11.9
				Initial	633.8		
3	MT	—	MOD G-S	Final	496.6	Weight Gain	0.90
				Initial	495.7		
4	Silica Gel	200g	MOD G-S	Final	656.0	Weight Gain	4.1
				Initial	651.9		
5				Final		Weight Gain	
				Initial			
6				Final		Weight Gain	
				Initial			

Total Impinger Weight Gain (Wc) 148.6 grms

Analyst VLL

Vf = Final Meter Volume = ____ ft3

Vi = Initial Meter Volume= ____ ft3

%CO ____

%CO2 ____

%O2 ____

%N2 ____

%H2 ____

%CH ____

DGMCF = Dry Gas Meter Correction Factor = ____

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ____ ft3

CONDENSED WATER ____ gms

FILTER WT. GAIN ____ gms

PROBE WASH WT. GAIN ____ gms

Tm = Average Meter Temp. = F+460 = ____ R

Pm = Meter Pressure (Barometric Pressure) = ____ in. Hg

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\text{ft3})(\_\_\_\text{"Hg})}{(\_\_\_\ \text{R})} = \_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____g) = ____ ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std) + Vm(std}} = \frac{\_\_\_\ \text{ft3}}{\_\_\_\ \text{ft3} + \_\_\_\ \text{ft3}} = \_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page 1 of 1

SAMPLING LOCATION Stack RUN NO. PM10-2

DATE 9-25-91 TIME START ~1025 TIME FINISH 1138 TEST DURATION ______ min.

DUCT DIMENSIONS ______ X ______ DIAMETER ______ ft INITIAL LEAK RATE 0.012 at 10" Hg cfm

7 PTCF 1.0037 DGMCF 1.92 NOZZLE DIA. 0.175 inches FINAL LEAK RATE —— cfm

BAR PRESS 29.0 " Hg

STATIC PRESS -0.8 " H2O OPERATOR JBR

Travers Point	SAMPLE ~~Clock~~ Time SEC	Dry gas meter reading ft3	^P In H2O	^H In H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	~~Probe Temp~~ NOZ.	Last Impinger	Vacuum In. Hg	______
E 1	-0-	64.4[illegible]										
1 ~~2~~	171	65.3	1.05	.30	316	65	63	300	.175	48	1	
2 ~~3~~	163	66.2	.95	"	319	65	63	289	"	48	1	
3 ~~4~~	161	67.1	.93	"	318	66	63	288	"	48	1	
4	183	68.05	1.2	"	318	67	64	292	"	49	1	
5	171	68.90	1.05	"	317	68	65	294	"	50	1	
D 1	175	69.80	1.1	"	325	68	65	292	"	50	1	
2	167	70.67	1.0	"	323	70	66	298	"	50	1	
3	175	71.55	1.1	"	322	70	66	300	"	51	1	
4	183	72.50	1.2	"	325	70	66	300	"	51	1	
5	175	73.41	1.1	"	322	71	66	310	"	52	1	
C 1	161	74.25	.93	"	320	71	67	308	"	55	1	
2	165	75.1	.98	"	321	72	67	310	"	54	1	
3	175	76.03	1.1	"	320	73	67	312	"	59	1	
4	171	76.92	1.05	"	319	73	66	310	"	59	1	
5	161	77.75	.98	"	318	73	67	311	"	58	1	
B 1	150	78.53	.81	"	310	72	67	314	"	58	1	
2	149	79.33	.79	"	312	74	67	311	"	58	1	
3	151	80.12	.82	"	313	74	67	309	"	57	1	
4	145	80.92	.75	"	314	74	68	311	"	57	1	
5	148	81.690	.78	"	316	75	68	314	"	58	1	
A 1	151	82.50	.82	"	307	75	69	309	"	59	1	
2	138	83.25	.68	"	306	78	70	311	"	59	1	
3	128	83.94	.59	"	307	79	70	310	"	60	1	
4	96	84.45	.33	"	308	80	70	308	"	60	1	
5	93	84.932	.31	"	308	81	71	304	"	60	1	

CONSOLE # N-34

FILTER # PM10-14 (0.2136)

AMBIENT TEMP. 60

PROBE LENGTH 8'

LINER MATERIAL Glass

V_M = 20.485

$T_{M avg}$ = 69.[illegible]

ΔH_{avg} = 0.30

WEATHER Clear

REMARKS 0.158 NOZ will not fit in 4" port. am on edge of acceptability with 0.175" NOZ but only choice.

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DI H2O	100	MOD G-S	Final Initial	797.5 628.9	Weight Gain	~~56.6~~ 16[illegible]
2	DI H2O	100	MOD GS	Final Initial	611.5 605.1	Weight Gain	6.4
3	MT	—	MOD G-S	Final Initial	458.3 458.0	Weight Gain	0.3
4	Silica Gel	200 g	MOD G-S	Final Initial	728.8 719.1	Weight Gain	~~7.7~~ 9.7
5				Final Initial		Weight Gain	~~71.0~~ 1[illegible]
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc)________grms

Analyst__________

Vf = Final Meter Volume = ____________ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = _______ R

Pm = Meter Pressure (Barometric Pressure) = ______ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER __________gms

FILTER WT. GAIN __________gms

PROBE WASH WT. GAIN __________gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ft3})(\_\_\_\_\text{"Hg})}{(\_\_\_\_\text{ R})} = \_\_\_\_\_\text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(_____g) = ______ft3

$$\text{Bws = Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std) + Vm(std}} = \frac{\_\_\_\_\text{ft3}}{\_\_\_\_\text{ft3} + \_\_\_\_\text{ft3}} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page 1 of 1

SAMPLING LOCATION Stack RUN NO. PM10-3

DATE 9-25 TIME START 2/1315 TIME FINISH ______ TEST DURATION ______ min.

DUCT DIMENSIONS ______ X ______ DIAMETER ~~1.75~~ ft INITIAL LEAK RATE 0.016 @ 5" cfm

PTCF 1.44 DGMCF 1.0032 NOZZLE DIA. 0.175 inches FINAL LEAK RATE — cfm

BAR PRESS ______ " Hg

STATIC PRESS -0.80 " H2O OPERATOR BL

ΔH@ = 1.92

Travers Point	SAMPLE TIME ~~Clock Time~~ SEC	Dry gas meter reading ft3	ΔP In H2O	ΔH In H2O	Stack Temp.. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp ^02	Last Impinger	Vacuum in. Hg	______
—	—	86.510	~									
E 1	175	87.4	1.1	.30	303	70	69	280	.175	61	1	
2	163	88.2	.96	"	301	71	69	285	"	61	1	
3	171	89.1	1.05	"	300	72	69	287	"	61	1	
4	179	90.05	1.15	"	301	73	69	291	"	60	1	
5	171	90.93	1.05	"	300	73	69	287	"	59	1	
D 1	171	91.83	1.05	"	304	73	69	291	"	59	1	
2	175	92.73	1.1	"	308	73	69	295	"	60	1	
3	191	93.71	1.3	"	312	74	69	299	"	60	1	
4	198	94.75	1.4	"	310	74	69	288	"	59	1	
5	171	95.645	1.05	"	307	75	70	289	"	60	1	
C 1	~~156~~ 183	96.55	~~1.1~~ 1.2	"	308	74	70	295	"	60	1	
2	175	97.58	1.1	"	316	74	69	290	"	60	1	
3	175	98.49	1.1	"	315	74	69	295	"	60	1	
4	175	99.29	1.1	"	314	74	69	290	"	60	1	
5	175	100.218	1.1	"	308	75	70	291	"	59	1	
B 1	~~164~~ 159	101.035	.96	"	308	75	71	293	"	61	1	
~~2~~	stop	process upset			(stopped kiln for cleaning)							
start B ~~1~~ 2	167	101.91	1.0	"	303	70	68	280	"	58	1	
~~4~~ 3	161	~~16~~ 102.70	.93	"	300	71	68	289	"	58	1	
~~5~~ 4	154	103.53	.85	"	300	72	69	293	"	57	1	
5	154	104.330	.85	"	302	72	68	295	"	56	1	
A 1	151	105.11	.81	"	299	71	69	287	"	55	1	
2	155	105.89	.86	"	301	72	69	291	"	56	1	
3	158	106.70	.89	"	299	72	69	294	"	59	1	
4	152	107.50	.83	"	298	73	69	290	"	57	1	
5	154	108.304	.85	"	299	74	70	288	"	58	1	
stop - end												

CONSOLE # N-34

FILTER # PM10-12 (0.2123)

AMBIENT TEMP. 62

PROBE LENGTH 8'

LINER MATERIAL GLASS

$V_m = 21.794$

$T_{m\,avg} = 70.98$

$\Delta H_{avg} = 0.30$

WEATHER ______

REMARKS ______

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DI H_2O	100	MOD G-S	Final Initial	749.7 578.8	Weight Gain	170.9
2	DI H_2O	100	MOD G-S	Final Initial	591.4 582.9	Weight Gain	8.5
3	MT	—	MOD GS	Final Initial	496.1 494.5	Weight Gain	1.6
4	Silica Gel	200(g)	MOD G-S	Final Initial	657.6 653.6	Weight Gain	4.0
5				Final Initial		Weight Gain	185 0
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 185.00 grms

Analyst ______

Vf = Final Meter Volume = ______ ft3

Vi = Initial Meter Volume= ______ ft3

DGMCF = Dry Gas Meter Correction Factor = ______

Vm = Metered Gas Volume = (Vf-Vi)(DGMCF) = ______ ft3

Tm = Average Meter Temp. = F+460 = ______ R

Pm = Meter Pressure (Barometric Pressure) = ______ in. Hg

%CO	______	%N2	______
%CO2	______	%H2	______
%O2	______	%CH	______

CONDENSED WATER ______ gms

FILTER WT. GAIN ______ gms

PROBE WASH WT. GAIN ______ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ft3})(\_\_\_\_\text{"Hg})}{(\_\_\_\_\ \text{R})} = \_\_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(______ g) = ______ ft3

$$\text{Bws = Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std) + Vm(std}} = \frac{\_\_\_\_\ \text{ft3}}{\_\_\_\_\ \text{ft3} + \_\_\_\_\ \text{ft3} = \_\_\_\_}$$

Revision Date 2/91

APPENDIX A.3

ALDEHYDES

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. Aldehydes-1

DATE 9-23-91 TIME START ____ TIME FINISH ____ TEST DURATION 62.5 min.

DUCT DIMENSIONS 43 X 38 DIAMETER ____ ft INITIAL LEAK RATE .012 at 10" cfm

? PTCF 1.0032 DGMCF 1.92 NOZZLE DIA. .191 inches FINAL LEAK RATE .010 at 7" cfm

0.227 ??

BAR PRESS ____ " Hg

STATIC PRESS -0.17 " H2O OPERATOR JBP

K = 1.06

Travers Point	Clock Time	Dry gas meter reading ft3	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	____
						Inlet	Outlet					
	1105	78.387										
A1	1107.5	79.5	.69	.70	310	65	64	278		48	1	
2	1110	80.5	.54	.55	311	65	64	270		46	1	
3	1112.5	81.4	.41	.42	309	65	64	271		46	1	
4	1115	82.1	.32	.33	308	66	65	282		46	1	
5	1117.5	83.035	.26	.27	308	68	66	285		46	1	
STOP	1119.5	"										
B1	1122	84.2	.79	.77	309	68	66	276		49	1	
2	1124.5	85.3	.62	.64	308	69	66	267		46	1	
3	1127	86.3	.55	.57	307	70	66	260		44	1	
4	1129.5	87.4	.56	.58	307	71	67	263		47	1	
5	1132	88.482	.66	.68	304	72	67	281		47	1	
STOP	1133	"										
C1	1135.5	90.3	.88	.92	308	72	68	293		51	1	
2	1138	91.4	.75	.78	308	74	68	298		47	1	
3	1140.5	92.7	.86	.89	307	75	69	303		49	1	
4	1143	94.1	.97	1.0	313	76	69	303		48	1	
5	1145.5	95.596	.87	.90	309	77	70	303		48	1	
STOP	1147	"										
D1	1149.5	96.9	.91	.95	314	76	70	305		50	1	
2	1152	98.1	.85	.88	314	78	71	303		49	1	
3	1154.5	99.5	.94	.98	313	78	71	303		48	1	
4	1157	101.0	1.2	1.3	315	79	71	305		49	1	
5	1159.5	102.508	1.0	1.1	312	79	71	304		49	1	
STOP	1201	"										
E1	1203.5	103.8	.87	.90	314	78	72	308		52	2	
2	1206	105.0	.88	.91	314	78	72	305		49	2	
3	1208.5	106.4	.94	.98	315	80	73	307		50	2	
4	1211	107.9	1.2	1.3	319	81	73	306		51	2	
5	1213.5	109.434	.97	1.0	312	82	74	304		52	2	

CONSOLE # N-34 PROBE LENGTH 8

FILTER # ____ LINER MATERIAL Glass

AMBIENT TEMP. 55

WEATHER Clear

REMARKS ____

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DNPH	200	mod G-S	Final Initial	837.5 667.6	Weight Gain	169.9
2	DNPH	200	G-S	Final Initial	738.2 632.0	Weight Gain	106.2
3	MT	—	mod G-S	Final Initial	473.9 472.7	Weight Gain	1.2
4	Silica Gel	-200g	mod G-S	Final Initial	687.9 680.7	Weight Gain	7.2
5				Final Initial		Weight Gain	
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 284.5 grms

Analyst________

Vf = Final Meter Volume = ________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf–Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = ________ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

$$Vm\ (std) = \frac{(17.64)(Vm)(Pm)}{Tm} = \frac{(17.64)(\_\_\_\_\ ft3)(\_\_\_\_\ "Hg)}{(\_\_\_\_\ R)} = \_\_\_\_\ ft3$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____ g) = ______ ft3

$$Bws = Moisture\ Fraction = \frac{Vw(std)}{Vw(std) + Vm(std} = \frac{\_\_\_\_\ ft3}{\_\_\_\_\ ft3 + \_\_\_\_\ ft3} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page 1 of 1

SAMPLING LOCATION Stack RUN NO. Aldehydes-2

DATE 9-23 TIME START ______ TIME FINISH ______ TEST DURATION ______ min.

DUCT DIMENSIONS 43" X 38" DIAMETER ______ ft INITIAL LEAK RATE 0.012 at 10" Hg cfm

Yd ~~PTCF~~ 1.0032 ~~DGMCF~~ ΔH@ 1.92 NOZZLE DIA. .227 inches FINAL LEAK RATE 0.006 @5" cfm

BAR PRESS 29.24 " Hg

STATIC PRESS ______ " H2O OPERATOR

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	______
						Inlet	Outlet					
—	1546	10.687	—									
E 1	48.5	12.0	.9	1.0	323	76	75	228		62	2	
2	51	13.4	.95	1.1	316	78	75	249		51	2	
3	53.5	14.8	.92	1.0	315	81	76	263		50	2	
4	56	16.2	1.0	1.1	314	83	76	274		49	2	
5	58.5	17.5	.8	.91	312	84	76	280		51	2	
—	59	17.5	—	—								
D 1	1601.5	18.9	1.0	1.0	320	84	76	281		49	2	
2	04	20.3	.95	1.1	321	85	76	290		50	2	
3	06.5	21.7	.95	1.1	324	84	77	280		50	2	
4	09	23.1	.99	1.1	325	86	77	278		51	2	
5	1611.5	24.7	~~.86~~	~~.98~~ 1.1	319	86	77	281		53	2	
—	1627	"	—									
C 1	29.5	26.0	.8	.91	322	80	78	290		56	2	
2	32	27.4	.83	.95	322	83	78	290		49	2	
3	34.5	28.7	.89	1.0	323	84	78	285		50	2	
4	37	30.1	.85	.97	320	84	78	287		50	2	
5	39.5	31.5	.94	1.1	318	85	78	289		50	2	
—	1640	31.5	—									
B 1	1641 ~~42.5~~	32.090	.70	.80	318	86	79	287		54	2	
~~2~~	stop	process	shut down.									
~~3~~												
~~4~~												
~~5~~												

CONSOLE # N-34

FILTER # ______

AMBIENT TEMP. 60

PROBE LENGTH 9'

LINER MATERIAL GLASS

WEATHER ______

REMARKS ______

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DNPH	200	mob G-S	Final Initial	766.8 642.2	Weight Gain	124.6
2	DNPH	200	G-S	Final Initial	660.9 608.5	Weight Gain	52.4
3	MT	—	moo G-S	Final Initial	481.7 475.5	Weight Gain	6.2
4	Silica Gel	~200g	mob G-S	Final Initial	763.5 750.2	Weight Gain	13.3
5				Final Initial		Weight Gain	
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 196.5 grms

Analyst ______

Vf = Final Meter Volume = ______ ft3

Vi = Initial Meter Volume= ______ ft3

DGMCF = Dry Gas Meter Correction Factor = ______

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ______ ft3

Tm = Average Meter Temp. = F+460 = ______ R

Pm = Meter Pressure (Barometric Pressure) = ______ in. Hg

%CO ______

%CO2 ______

%O2 ______

%N2 ______

%H2 ______

%CH ______

CONDENSED WATER ______ gms

FILTER WT. GAIN ______ gms

PROBE WASH WT. GAIN ______ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ft3})(\_\_\_\_\text{"Hg})}{(\_\_\_\_\ \text{R})} = \_\_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____g) = ______ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std)} + \text{Vm(std}} = \frac{\_\_\_\_\text{ft3}}{\_\_\_\_\text{ft3} + \_\_\_\_\text{ft3}} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26

Page ____ of ____

SAMPLING LOCATION Stuck

RUN NO. Aldehydes-3

DATE 1-24-91 TIME START 0845

TIME FINISH ______

TEST DURATION 62.5 min

DUCT DIMENSIONS 43" x 38"

DIAMETER ______ ft

INITIAL LEAK RATE 0.016 at 10" Hg cfm

7 PTCF 1.0032 DGMCF 1.92

NOZZLE DIA. .264 Inches

FINAL LEAK RATE 0.014 at 6" Hg cfm

BAR PRESS 29.4 " Hg

STATIC PRESS -0.58 " H2O

OPERATOR JBP

4 3

2

Nozzle fell off on [illegible]H exit → new nozzle .227 K=1.06

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	______
	0845	33.175										
E1	0847.5	34.8	.77	1.5	310	56	53	304		46	1	
2	850	36.1	.58	1.2	305	58	53	304		46	1	
3	852.5	37.6	.59	1.2	302	58	53	303		45	1	
4	855	39.1	.75	1.5	310	60	54	301		46	1	
5	857.5	40.776	.80	1.6	308	62	55	304		46	1	
STOP	~~859~~ 903	"										
D1	905.5	42.0	.87	.87	316	61	57	300		50	1	
2	908	43.2	.80	.80	318	63	56	307		48	1	
3	910.5	44.4	.76	.76	320	64	57	296		49	1	
4	913	45.7	.83	.83	318	65	57	294		48	1	
5	915.5	46.863	.79	.79	315	66	58	296		48	1	
STOP	917	"										
C1	919.5	48.1	.85	.85	316	65	58	295		51	1	
2	922	49.4	.87	.87	320	67	59	295		51	1	
3	924.5	50.6	.87	.87	321	69	60	295		52	1	
4	927	51.9	.90	90	318	69	60	296		50	1	
5	929.5	53.038	.70	.70	316	71	61	295		51	1	
STOP	931	"										
B1	933.5	54.2	.64	.64	308	68	62	294		53	1	
2	936	55.2	.60	60	315	71	62	293		51	1	
3	938.5	56.3	.66	.66	318	72	63	293		51	1	
4	941	57.4	.61	.61	319	72	63	295		53	1	
5	943.5	58.445	.58	.58	319	72	64	294		54	1	
STOP	945	"										
A1	947.5	59.5	.57	.57	318	70	64	293		56	1	
2	950	60.4	.44	.44	319	72	65	294		52	1	
3	952.5	61.3	.43	.43	320	72	65	294		50	1	
4	955	62.1	.29	.29	319	72	65	304		49	1	
5	957.5	62.882	.31	.31	316	72	66	303		51	1	

CONSOLE # N-34

PROBE LENGTH 8

FILTER # ______

LINER MATERIAL glass

AMBIENT TEMP. 50

WEATHER Overcast / Drizzle

REMARKS ______

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	DNPH	200	MOD G-S	Final Initial	847.4 690.4	Weight Gain	157.0
2	DNPH	200	MOD G-S	Final Initial	711.7 676.0	Weight Gain	35.7
3	MT	—	MOD G-S	Final Initial	477.7 474.8	Weight Gain	2.90
4	Silica Gel	200g	MOD G-S	Final Initial	770.7 763.4	Weight Gain	7.30
5				Final Initial		Weight Gain	
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 202.9 grms

Analyst________

Vf = Final Meter Volume = ________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = ________ in. Hg

%CO		%N2	
%CO2		%H2	
%O2		%CH	

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\ \text{ft3})(\_\_\_\_\ \text{"Hg})}{(\_\_\_\_\ \text{R})} = \_\_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____ g) = ____ ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std)} + \text{Vm(std}} = \frac{\_\_\_\_\ \text{ft3}}{\_\_\_\_\ \text{ft3} + \_\_\_\_\ \text{ft3}} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. Aldehydes-4

DATE 9-25-91 TIME START ________ TIME FINISH ________ TEST DURATION ________ min.

DUCT DIMENSIONS 38" X 43" DIAMETER .195 ft INITIAL LEAK RATE 0.008 at 9"Hg cfm

PTCF 1.0006 DGMCF 1.75 NOZZLE DIA. . ________ Inches FINAL LEAK RATE 0.010 at 6"Hg cfm

BAR PRESS 29.0 " Hg

STATIC PRESS ________ " H2O OPERATOR JBP

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	______
	1144	14.155										
E 1	1146.5	15.2	.82	.44	325	70	68	220		62	1	
2	1149	16.1	.80	.43	318	72	69	222		62	1	
3	1151.5	17.0	.83	.44	314	72	70	230		60	1	
4	1154	18.1	.94	.49	314	74	70	246		58	1	
5	1156.5	19.008	.89	.47	313	73	70	273		56	1	
STOP	1200	"										
D 1	1202.5	20.0	.84	.45	323	71	71	280		57	1	
2	1205	20.9	.79	.43	326	73	71	281		53	1	
3	1207.5	21.8	.81	.44	325	72	71	290		52	1	
4	1210	22.9	.97	.52	328	73	71	296		52	1	
5	1212.5	23.67	.92	.48	325	73	71	287		51	1	
STOP	1214	"										
C 1	1216.5	24.7	.76	.41	325	73	72	279		54	1	
2	1219	25.6	.70	.38	324	74	72	272		53	1	
3	1221.5	26.6	.77	.42	320	72	72	274		52	1	
4	1224	27.5	.82	.44	320	72	71	277		51	1	
5	1226.5	28.43	.80	.43	319	71	71	278		51	1	
STOP	1228	"										
B 1	1230.5	29.2	.55	.30	317	71	71	279		55	1	
2	1233	29.9	.51	.28	317	72	71	288		55	1	
3	1235.5	30.8	.52	.28	317	71	70	273		55	1	
4	1238	31.5	.54	.29	311	71	71	292		55	1	
5	1240.5	32.23	.48	.26	316	70	70	292		55	1	
STOP	1241.5	"										
A 1	1244	33.0	.41	.25	308	72	71	283		58	1	
2	1246.5	33.7	.40	.22	308	73	72	279		58	1	
3	1249	34.3	.36	.20	304	74	72	287		58	1	
4	1251.5	34.9	.22	.13	303	73	71	295		58	1	
5	1254	35.427	.20	.12	303	74	72	296		58		

CONSOLE # N-32 PROBE LENGTH 8'

FILTER # ________ LINER MATERIAL glass

AMBIENT TEMP. 60

WEATHER ________

REMARKS ________

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight In grams			Weight Gain
1	DNPH	200	mod G-S	Final Initial	822.7 696.0	Weight Gain	126.7
2	DNPH	200	G-S	Final Initial	673.5 632.0	Weight Gain	41.5
3	MT	—	mod G-S	Final Initial	494.3 492.8	Weight Gain	1.5
4	Silica Gel	~200g	mod G-S	Final Initial	748.5 741.6	Weight Gain	6.9
5				Final Initial		Weight Gain	
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 176.6 grms

Analyst ________

Vf = Final Meter Volume = ________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = ________ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ft3})(\_\_\_\_\text{"Hg})}{(\_\_\_\_\ \text{R})} = \_\_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____g) = ______ ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std)} + \text{Vm(std}} = \frac{\_\_\_\_\text{ft3}}{\_\_\_\_\text{ft3} + \_\_\_\_\text{ft3}} = \_\_\_\_$$

Revision Date 2/91

APPENDIX A.4

PAH

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page 1 of

SAMPLING LOCATION Stack RUN NO. PAH-1

DATE 9-23-91 TIME START 0830 TIME FINISH TEST DURATION 120 min.

DUCT DIMENSIONS 42 x 38 DIAMETER ft INITIAL LEAK RATE .02 at 10" cfm

Y PTCF .9875 DGMCF 2.00 NOZZLE DIA. .191 inches FINAL LEAK RATE .008 at 7" cfm

BAR PRESS " Hg

STATIC PRESS -.17 " H2O OPERATOR JBP

Meter N Vd. Orifice 3 4 5

49
.53

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp. Filter	Probe Temp	Last Impinger	Vacuum in. Hg	XAD
	0830	20.437										
A1	0835	21.6	.48	.24	324	51	50	274	~~22~~	43	1	42
2	840	23.0	.44	.22	322	53	50	258		42	1	43
3	845	24.2	.38	.16	322	53	51	247		42	1	43
4	850	35.2	.25	.13	318	55	52	240		42	1	43
5	855	26.241	.25	.13	318	54	52	227		42	1	41
STOP	856	"										
B1	901	27.6	.64	.34	326	56	54	273		43	3	45
2	906	29.1	.53	.28	320	56	54	246		43	2	42
3	911	30.8	.64	.36	326	58	55	258		43	4	44
4	916	32.5	.61	.32	326	59	56	259		44	3	45
5	921	34.120	.63	.33	321	61	57	266		44	2	44
STOP	922	"										
C1	927	36.0	.78	.42	333	62	58	228		44	2	44
2	932	37.8	.80	.43	333	63	60	228		44	2	46
3	937	39.6	.87	.47	337	64	61	260		45	3	45
4	942	41.5	84	.45	332	64	62	245		46	4	46
5	947	43.511	.90	.49	324	65	62	241		45	4	45
STOP	948	"	—									
D1	953	45.5	.84	.46	331	64	63	239		45	3	47
2	958	47.3	.91	.39	330	65	63	228		45	3	47
3	1003	49.3	.93	.50	332	65	64	249		48	3	48
4	1008	51.2	.92	.50	330	65	64	257		48	3	49
5	1013	53.051	.92	.45	327	66	64	252		49	3	50
STOP	1014	"	-									
E1	1019	54.9	.75	.40	326	67	66	245		51	3	52
2	1024	56.7	.66	.35	327	69	67	268		51	3	52
3	1029	58.6	.79	.43	325	68	68	235		51	3	50
4	1034	60.4	.85	.47	321	70	68	226		51	3	49
5	1039	62.514	.87	.49	320	70	69	233		49	3	48

CONSOLE # N-33 PROBE LENGTH 8

FILTER # LINER MATERIAL Glass

AMBIENT TEMP. 60

WEATHER Clear

REMARKS $V_m = 42.077$ $V_{std} = 41.296$ $V_s = 59.94$ fps

$T_m = 59.5$ $\sqrt{\Delta P}$ avg $= .921$

$T_s = 326.$ $B_{wo} = 31\%$ I% = 102.5

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	MT	—	MOD G-S	Final Initial	847.6 457.4	Weight Gain	390.2
2	HPLC H_2O	100	MOD G-S	Final Initial	580.0 VLL ~~604.8~~ 579.7	Weight Gain	0.3
3	HPLC H_2O	100	MOD G-S	Final Initial	594.4 VLL ~~592.9~~ 594.3	Weight Gain	0.1
4	MT	—	MOD G-S	Final Initial	474.1 VLL ~~472.5~~ 474.1	Weight Gain	0.0
5	Silica Gel	~200(g)	MOD G-S	Final Initial	6569 VLL ~~646.1~~ 647.8	Weight Gain	9.1
6				Final Initial		Weight Gain	399.7

Total Impinger Weight Gain (Wc) 399.7 grms

Analyst ________

Vf = Final Meter Volume = ____________ ft3

Vi = Initial Meter Volume= ________ ft3

%CO ________

%N2 ________

%CO2 ________

%H2 ________

%O2 ________

%CH ________

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf–Vi)(DGMCF) = ________ ft3

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

Tm = Average Meter Temp. = F+460 = _______ R

Pm = Meter Pressure (Barometric Pressure) = _______ in. Hg

$$Vm\ (std) = \frac{(17.64)(Vm)(Pm)}{Tm} = \frac{(17.64)(\_\_\_\_ ft3)(\_\_\_\_ "Hg)}{(\_\_\_\_ R)} = \_\_\_\_ ft3$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(_____g) = ______ft3

$$Bws = Moisture\ Fraction = \frac{Vw(std)}{Vw(std) + Vm(std} = \frac{\_\_\_\_ ft3}{\_\_\_\_ ft3 + \_\_\_\_ ft3} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. PAH-2

DATE 9-23-91 TIME START ________ TIME FINISH ________ TEST DURATION ________ min.

DUCT DIMENSIONS 43" X 38" DIAMETER ________ ft INITIAL LEAK RATE 0.010 at 10" Hg cfm

4 PTCF .9875 DGMCF 2.00 NOZZLE DIA. .191 inches FINAL LEAK RATE 0.010 at 5" Hg cfm

BAR PRESS ________ " Hg

STATIC PRESS -0.17 " H2O OPERATOR JBP

A B C D E 4 3 2 1
0 0 0 0 0

Travers Point	Clock Time	Dry gas meter reading ft3	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	XAD
	1355	63.125										
EA1	1400	64.8	.69	.36	315	67	68	271		51	2	50
2	1405	66.6	.72	.38	319	69	68	307		51	2	52
3	1410	68.6	.80	.42	322	69	68	323		52	2	53
4	1415	70.5	.73	.38	318	69	70	252		52	2	52
5	1420	72.338	.84	.44	325	70	70	230		49	2	53
STOP	1421	"										
D1	1426	74.1	.76	.40	336	71	71	258		51	2	55
2	1431	75.8	.82	.43	331	71	71	302		49	2	51
3	1436	79.9	.91	.48	333	71	71	290		50	2	50
4	1441	81.1	.89	.47	328	71	72	278		49	3	51
5	1446	82.290	.97	.52	328	72	72	280		50	3	53
STOP	1447	"										
C1	~~1502~~ 1452	84.1	.74	.39	324	71	72	286		51	2	52
2	~~1507~~ 1457	86.0	.73	.38	325	71	72	289		50	2	51
3	1502	88.0	.84	.44	324	73	73	262		50	3	50
4	1507	90.0	.80	.42	328	74	73	280		49	2	50
5	1512	91.827	.82	.43	322	74	73	275		51	2	51
STOP	1514	"										
B1	1519	93.5	.59	.32	319	74	72	275		52	2	54
2	1524	95.3	.60	.32	320	74	73	264		53	2	51
3	1529	96.9	.63	.34	323	74	74	262		53	2.5	54
4	1534	98.5	.52	.28	325	74	73	271		54	2	52
5	1539	100.254	.62	.34	324	74	75	277		53	2	55
STOP	1541	"										
A1	1546	101.8	.48	.25	324	75	75	295		58	2	58
2	1551	103.1	.46	.24	323	76	75	280		54	2	56
3	1556	104.6	.38	.20	317	75	75	283		54	2	53
4	1601	105.7	.25	.13	319	74	75	283		53	1	55
5	1606	106.660	.25	.13	310	75	76	289		54	1	56

CONSOLE # N-33 PROBE LENGTH 8'

FILTER # ________ LINER MATERIAL Glass

AMBIENT TEMP. 60

WEATHER ________

REMARKS ________

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	MT	—	MOD G-S	Final Initial	844.3 457.4	Weight Gain	386.9
2	HPLC H_2O	100	MOD G-S	Final Initial	588.9 590.0	Weight Gain	-1.1
3	HPLC H_2O	100	MOD G-S	Final Initial	565.9 566.2	Weight Gain	-0.3
4	MT	—	MOD G-S	Final Initial	474.5 474.0	Weight Gain	0.5
5	Silica Gel	~200g	MOD G-S	Final Initial	700.9 688.8	Weight Gain	12.1
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 398.~~3~~1 VLL grms

Analyst VLL

Vf = Final Meter Volume = ____________ ft3

Vi = Initial Meter Volume= _________ ft3

DGMCF = Dry Gas Meter Correction Factor = _________

Vm = Metered Gas Volume = (Vf–Vi)(DGMCF) = _________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = _______ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER __________ gms

FILTER WT. GAIN __________ gms

PROBE WASH WT. GAIN __________ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\_\text{ ft3})(\_\_\_\_\text{ "Hg})}{(\_\_\_\_\text{ R})} = \_\_\_\_\_\text{ ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(_____ g) = _______ ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std) + Vm(std}} = \frac{\_\_\_\_\text{ ft3}}{\_\_\_\_\text{ ft3} + \_\_\_\_\text{ ft3}} = \_\_\_\_$$

Revision Date 2/91

SOURCE SAMPLING FIELD DATA

PLANT NAME Mathy #26 Page ____ of ____

SAMPLING LOCATION Stack RUN NO. PAH-3

DATE 9 24 91 TIME START 1236 TIME FINISH ________ TEST DURATION 120 min.

DUCT DIMENSIONS 43" X 35" DIAMETER ________ ft INITIAL LEAK RATE 0.008 at 19" Hg cfm

PTCF ________ DGMCF ________ NOZZLE DIA. .191 inches FINAL LEAK RATE 0.008 at 1" cfm

BAR PRESS 29.4 " Hg

STATIC PRESS -0.58 " H2O OPERATOR JSP

	Travers Point	Clock Time	Dry gas meter reading ft3	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp Inlet	Dry gas meter temp Outlet	Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	YAD
		1236	69.006										
.5	E1	1241	70.6	.72	.36	323	73	68	289		55	2	55
	2	1246	72.2	.73	.36	306	73	69	301		50	3	51
	3	1251	74.1	.72	.36	328	75	70	298		50	3	58
	4	1256	75.9	.70	.45	326	76	71	299		52	3	59
	5	1301	77.738	.83	.42	324	77	72	289		53	3	57
	STOP	1302	"	—									
	D1	1307	79.4	.70	.35	320	76	72	296		53	3	55
	2	1312	81.2	.74	.38	334	76	73	297		54	3	58
	3	1317	83.0	.82	.41	336	75	73	297		51	3	55
1318.5	4 ~~4~~	~~1322~~	83.571	.80	.40	336	75	73	298		51	3	56
	~~5~~	~~1327~~ 1335	"										
	4	1340	85.0	.94	.57	298	75	74	293		53	3	57
	5	1345	86.777	.74	.37	302	76	73	300		52	4	57
	STOP	1346	"	~~.72~~	~~.36~~								
	C1	~~1401~~ 1351	88.5	.72	.36	329	76	74	298		54	3	58
	2	1356	89.1	.66	.33	334	74	73	302		54	3	58
	3	1401	91.9	.75	.37	338	77	73	297		55	3	58
	4	1406	93.8	.83	.42	336	76	72	300		55	3	62
	5	1411	95.49	.68	.34	334	77	72	299		53	3	58
	STOP	1412	"	—									
	B1	1417	97.1	.65	.32	337	77	72	300		55	3	58
	2	1422	98.6	.60	.30	334	78	72	300		54	3	54
	3	1427	100.2	.56	.28	330	77	72	297		53	3	54
	4	1432	101.8	.57	.29	329	75	71	299		52	3	52
	5	1437	103.38	.60	.30	324	75	71	295		52	3	54
STOP	A1	1438 1443	" 104.7	.48	.24	325	72	69	296		53	3	53
	2	1448	106.1	.44	.22	327	73	70	299		52	3	57
	3	1453	107.3	.39	.19	328	73	69	300		50	3	52
	4	1458	108.3	.26	.13	327	74	70	300		51	2	55
	5	1503	109.523	.26	.13	324	[illegible]	[illegible]	302		54	2	55

CONSOLE # ________ PROBE LENGTH 8

FILTER # ________ LINER MATERIAL Glass

AMBIENT TEMP. 65

WEATHER

REMARKS

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Approx. ml	Configuration	Weight in grams			
1	MT	—	MOD G-S	Final Initial	773.7 457.5	Weight Gain	316,2
2	HPLC H_2O	100	MOD G-S	Final Initial	583.3 583.4	Weight Gain	-0,1
3	HPLC H_2O	100	MOD G-S	Final Initial	565.9 566.1	Weight Gain	-0, 2
4	MT	—	MOD G-S	Final Initial	474,5 474.2	Weight Gain	0, 3
5	Silica Gel	200 g	MOD G-S	Final Initial	709.7 700.9	Weight Gain	8,8
6				Final Initial		Weight Gain	

Total Impinger Weight Gain (Wc) 325 grms

Analyst ________

Vf = Final Meter Volume = ________ ft3

Vi = Initial Meter Volume= ________ ft3

DGMCF = Dry Gas Meter Correction Factor = ________

Vm = Metered Gas Volume = (Vf−Vi)(DGMCF) = ________ ft3

Tm = Average Meter Temp. = F+460 = ________ R

Pm = Meter Pressure (Barometric Pressure) = ________ in. Hg

%CO ________

%CO2 ________

%O2 ________

%N2 ________

%H2 ________

%CH ________

CONDENSED WATER ________ gms

FILTER WT. GAIN ________ gms

PROBE WASH WT. GAIN ________ gms

$$\text{Vm (std)} = \frac{(17.64)(\text{Vm})(\text{Pm})}{\text{Tm}} = \frac{(17.64)(\_\_\_\text{ft3})(\_\_\_\text{"Hg})}{(\_\_\_\ \text{R})} = \_\_\_\ \text{ft3}$$

Vw(std) = Volume of Water Vapor = .0472(Wc) = .0472(____ g) = ______ ft3

$$\text{Bws} = \text{Moisture Fraction} = \frac{\text{Vw(std)}}{\text{Vw(std) + Vm(std}} = \frac{\_\_\_\ \text{ft3}}{\_\_\_\ \text{ft3} + \_\_\_\ \text{ft3}} = \_\_\_$$

Revision Date 2/91

APPENDIX B

PROCESS DATA SHEETS

Recycle - 3.3 additive - 0
asphalt - 5.8 Filler - 0

9/25/91
9/~~23/91~~ Plant # 26 Mathy

PAGE 1 OF __

1	2	3	4	5		6	7	8	9		10	11	12
Time (24 HR) OTE: check all box in lumn when mple is ken	Fuel Use ~~Fuel~~ Oil ✓ Nat. Gas __ Propane __ Coal __ other	Burner Setting %	~~Blower Pressure~~ Total Mix	Production Rate		Asphalt Cement %	Mix Temp. °F	Kiln Exit Exhaust Gas Temp. °F	Venturi Scrubber __ Baghouse		Ambient Temp. °F	Relative Humidity %	Exhaust Damper Position
				✓ Mix Aggregate TPH	✓ RAP TPH				Pressure Drop in w.g.	~~Water Pressure~~ ~~and~~ BH(F) Outlet Temp			
0854											56.0	60/55	open
0925	~~0935~~	72	67.3	146	65	8.9	288	358/355.5	11.2	—	—	—	"
1000	~~10:00~~	62	198.9	151	68	8.8	299	349/355.5	9.7	—	—	—	"
1033	~~10:35~~ ~~8340gr~~	97	328.4	182	77	10.6	280	340/355.5	10.0	—	60.4	—	"
11:10		90	476	175	69	10.3	285	310/355.5	10.0	—	60.2	—	"
11:49		76	660.9	189	76	10.8	275	333/355.5	9.5	—	—	—	"
12:07		82	740.5	173	76	10.2	295	349/355.5	16.0	—	—	—	"
1336		76	1093.4	173	74[illegible]	9.4	270	324/355.5	9.9	—		—	"
Shutdown 1415-1430			1262.8	0							62.0	—	"
1447		84	1340	167	72	9.8	295	338/355.5	10.6	—	—	—	"
1500		90	1391.8	172	87	10.4	270	—	10.6	—	60.0	—	"
1324	6707	63	1493	146	58	8.9	255	330/355	10.6	—	58.2	—	—

Agg. Moist. - 3.0
Recycle Moist - 3.3
Asphalt - 5.8

Add' - 0
Fill - 0

35 psig on oil pressure

246-2468 Fax

PAGE 1 OF 1

9/23/91 Plant # 26 Mathy

9:30 filled 6400 gallons oil

1	2	3	4	5		6	7	8	9		10	11	12
Time (24 HR) NOTE: check small box in column when moisture sample is taken	Fuel Use Fuel Oil ✓ Nat. Gas __ Propane __ Coal __ other	Burner Setting %	~~Baghouse Pressure~~ Total Mix Tons	Production Rate ✓ Mix ~~Aggregate~~ TPH	✓ RAP TPH	Asphalt Cement TPH	Mix Temp. °F	Kiln Exit Exhaust Gas Temp. °F 277st	Venturi Scrubber / ✓ Baghouse Pressure Drop in w.g.	Water Pressure ~~psi~~ BH (F) Outlet Temp	Ambient Temp. °F	Relative Humidity %	Exhaust Damper Position
0830	8:26 .43" 3951	100	9:25 45" 349	195[illegible]	81	11.3	285	263	10.5	—	47.0	—	wide open
0913	—	100	647	187	79	11.1	270	275 264	10.0	—	49.8	45 47	"
0956	—	86	850	190	82	11.1	279	275.5 268	10.5	—	52.6		"
1039	—	96	1043	201	88	12.0	275	277.5 268	10.5	—	~~52.6~~ 55.0		"
1128	—	100	1279	198	87	11.6	275	272.5 259	10.5	—	57.8		"
1200	—	100	1431	193	83	11.4	287	272.5 268	10.5	—	58.0	43 54	"
14:05	[illegible] 33" 7665	84	1728	187	88	11.2	275	263.5 263	10.7	—	58.8		"
14:29	—	84	1952	186	69	10.8	285	263.5 272	10.5	—	60.8	49 60	"
15:00	—	64	2139	182	79	10.9	264	262.5 263	10.5	—	61.2	—	"
15:30	—	76	2232	182	80	10.7	295	263.5 278	10.5	—	60	—	"
16:30	—	72	2499	180	72	10.8	285	263.5 276	10.0	—	58.2	48 54	"
16:47	Shut down												

Agg. Mast. - 3.7 add - 0
Recycle Moist - 3.3 Fill - 0
Asphalt - 5.8

715-246-6468 Fax

PAGE 1 OF ___

* varies quite a bit

9/~~23~~ 24/91 Plant # 26 Mathy

1 Time (24 HR) NOTE check small box in column when moisture sample is taken	2 Fuel Use Fuel Oil ✓ Nat. Gas ___ Propane ___ Coal ___ other	3 Burner Setting %	4 ~~Blower Pressure~~ Total Mix Tons	5 Production Rate ~~Mix~~ Aggregate TPH	 ✓ RAP TPH	6 Asphalt Cement %	7 Mix Temp. °F	8 Kiln Exit Exhaust Gas Temp. °F	9 Venturi Scrubber ✓ Baghouse Pressure Drop in w.g.	 ~~Water Pressure~~ BH(F) Outlet Temp	10 Ambient Temp. °F	11 Relative Humidity %	12 Exhaust Damper Position
0830		34	85.6	148	65	8.8*	269	331/3365	11.0	—	48.2	—	open
914		30	217.8	134	55	6.8*	265	324/3335	10.8	—	48.5	44/46	"
936		48	307.9	138	49	10.0*	275	345/3335	10.7	—	—	—	"
1031	4342~~8~~ +	48	485.4	143	57	11.2*	282	355/3335	10.5	—	51.4	—	"
1108	4224/2	50	621.7	145	63	11.9*	278	339/3335	10.6	—	51.8	—	"
1130	4283	56	708.7	143	58	9.5*	282	348/3335	10.5	—	52.0	—	"
1138	61.5"		727•								55.6	44/53	"
1215			~~727~~									—	"
1221		53	884.3	144	65	8.3	275	334/3335	10.5	—		—	"
1235		53	938.2	146	62	8.6	275	341/3335	10.3	—	55.8	—	"
1318	Plant Down	54	1094.4	147	62	10.7	275	366/3335	10.5	—	—	—	"
1336	Restarted	52	1101.7	145	56	10.7	265	342/3335	11.2	—	55.2	—	"
1406		51	1212.8	144	56	8.9	282	337/3335	10.0	—			
1411	69" 3466		1233										"
1431		48	1302.1	152	67	8.6	275	344/3333	10.0	—	57.0	48/50	"
1506		48	1425.4	147	60	8.7	272	334/333.5	10.0	—			

APPENDIX C

SAMPLE PARAMETER CALCULATION SHEETS

C.1 PM/Metals
C.2 PM_{10}/CPM
C.3 Aldehydes
C.4 PAH

APPENDIX C.1

PM/METALS

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 1.00

SAMPLING PARAMETER	TM
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.37
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	326.04
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	41.83
Average Meter Pressure (in.H2O)	0.33
Average Meter Temperature (deg. F)	64.68
Moisture Collected (g)	395.60
Carbon Dioxide Concentration (%V)	9.22
Oxygen Concentration (%V)	10.33
Nitrogen Concentration (%V)	80.45
Dry Gas Meter Factor	1.00060
Pitot Constant	0.84
Particulate Catch (g)	0.08430

Average Sampling Rate (dscfm)	0.33
Standard Metered Volume,Vm(std) (dscf)	41.40
Standard Metered Volume,Vm(std) (dscm)	1.172
Standard Volume Water Vapor,Vw (scf)	18.65
Standard Volume Water Vapor,Vw (scm)	0.528
Stack Moisture (%V)	31.06
Mole Fraction Dry Stack Gas	0.689
Dry Molecular Weight	29.89
Wet Molecular Weight	26.20
Stack Gas Velocity,Vs (fpm)	3577.88
Stack Gas Velocity,Vs (mpm)	1090.54
Volumetric Flow Rate (acfm)	39654.84
Volumetric Flow Rate (acmm)	1123.025
Volumetric Flow Rate (dscfm)	18016.18
Volumetric Flow Rate (dscmm)	510.218
Percent Isokinetic	102.35
Percent Excess Air	94.55
Fuel Factor,Fo	1.146
Ultimate CO2	18.23
Concentration of Particulate (grains/acf)	0.01428
Concentration of Particulate (g/acm)	0.03267
Concentration of Particulate (grains/dscf)	0.03143
Concentration of Particulate (g/dscm)	0.07191
Concentration of Particulate (grains/dscf @12% CO2)	0.04090

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 2.00

SAMPLING PARAMETER TM

Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.37
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	323.24
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	40.68
Average Meter Pressure (in.H2O)	0.33
Average Meter Temperature (deg. F)	75.86
Moisture Collected (g)	372.60
Carbon Dioxide Concentration (%V)	7.55
Oxygen Concentration (%V)	10.01
Nitrogen Concentration (%V)	82.44
Dry Gas Meter Factor	1.00060
Pitot Constant	0.84
Particulate Catch (g)	0.04350

Average Sampling Rate (dscfm)	0.32
Standard Metered Volume,Vm(std) (dscf)	39.42
Standard Metered Volume,Vm(std) (dscm)	1.116
Standard Volume Water Vapor,Vw (scf)	17.57
Standard Volume Water Vapor,Vw (scm)	0.498
Stack Moisture (%V)	30.83
Mole Fraction Dry Stack Gas	0.692
Dry Molecular Weight	29.61
Wet Molecular Weight	26.03
Stack Gas Velocity,Vs (fpm)	3536.56
Stack Gas Velocity,Vs (mpm)	1077.94
Volumetric Flow Rate (acfm)	39196.88
Volumetric Flow Rate (acmm)	1110.056
Volumetric Flow Rate (dscfm)	17932.35
Volumetric Flow Rate (dscmm)	507.844
Percent Isokinetic	97.92
Percent Excess Air	85.04
Fuel Factor,Fo	1.442
Ultimate CO2	14.49
Concentration of Particulate (grains/acf)	0.00779
Concentration of Particulate (g/acm)	0.01783
Concentration of Particulate (grains/dscf)	0.01703
Concentration of Particulate (g/dscm)	0.03897
Concentration of Particulate (grains/dscf @12% CO2)	0.02707

```
FACILITY : Mathy #26

DATE:        9-24-91
LOCATION:    STACK
RUN NUMBER:       3.00
==================================================================
SAMPLING PARAMETER     TM
==================================================================

Total Sampling Time (min.)                                  123.50
Corrected Barometric Pressure (in. Hg)                       29.38
Absolute Stack Pressure,Ps(in. Hg)                           29.34
Stack Static Pressure (in. H2O)                              -0.58
Average Stack Temperature (deg. F)                          328.52
Stack Area (sq.in.)                                        1596.00
Metered Volume,Vm (cu.ft.)                                   40.78
Average Meter Pressure (in.H2O)                               0.35
Average Meter Temperature (deg. F)                           71.39
Moisture Collected (g)                                      341.40
Carbon Dioxide Concentration (%V)                             7.18
Oxygen Concentration (%V)                                    11.77
Nitrogen Concentration (%V)                                  81.05
Dry Gas Meter Factor                                       1.00060
Pitot Constant                                                0.84
Particulate Catch (g)                                      0.06110
------------------------------------------------------------------

Average Sampling Rate (dscfm)                                 0.32
Standard Metered Volume,Vm(std) (dscf)                       39.85
Standard Metered Volume,Vm(std) (dscm)                       1.129
Standard Volume Water Vapor,Vw (scf)                         16.10
Standard Volume Water Vapor,Vw (scm)                         0.456
Stack Moisture (%V)                                          28.77
Mole Fraction Dry Stack Gas                                  0.712
Dry Molecular Weight                                         29.62
Wet Molecular Weight                                         26.28
Stack Gas Velocity,Vs (fpm)                                3459.67
Stack Gas Velocity,Vs (mpm)                                1054.51
Volumetric Flow Rate (acfm)                               38344.71
Volumetric Flow Rate (acmm)                               1085.922
Volumetric Flow Rate (dscfm)                              17925.08
Volumetric Flow Rate (dscmm)                               507.638
Percent Isokinetic                                          100.24
Percent Excess Air                                          122.05
Fuel Factor,Fo                                               1.272
Ultimate CO2                                                 16.44
Concentration of Particulate (grains/acf)                  0.01106
Concentration of Particulate (g/acm)                       0.02531
Concentration of Particulate (grains/dscf)                 0.02366
Concentration of Particulate (g/dscm)                      0.05414
Concentration of Particulate (grains/dscf @12% CO2)        0.03954
------------------------------------------------------------------
```

APPENDIX C.2

PM_{10}/CPM

FACILITY : MATHEY 26

DATE: 9/24/91
LOCATION: STACK
RUN NUMBER: RUN 1

SAMPLING PARAMETER PM10

Total Sampling Time (min.)	64.25
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.32
Stack Static Pressure (in. H2O)	-0.80
Average Stack Temperature (deg. F)	327.81
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	17.53
Average Meter Pressure (in.H2O)	0.24
Average Meter Temperature (deg. F)	59.16
Moisture Collected (g)	148.60
Carbon Dioxide Concentration (%V)	7.06
Oxygen Concentration (%V)	12.01
Nitrogen Concentration (%V)	80.93
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.27
Standard Metered Volume,Vm(std) (dscf)	17.30
Standard Metered Volume,Vm(std) (dscm)	0.490
Standard Volume Water Vapor,Vw (scf)	7.01
Standard Volume Water Vapor,Vw (scm)	0.198
Stack Moisture (%V)	28.83
Mole Fraction Dry Stack Gas	0.712
Dry Molecular Weight	29.61
Wet Molecular Weight	26.26
Stack Gas Velocity,Vs (fpm)	3872.25
Stack Gas Velocity,Vs (mpm)	1180.26
Volumetric Flow Rate (acfm)	42917.42
Volumetric Flow Rate (acmm)	1215.421
Volumetric Flow Rate (dscfm)	20050.32
Volumetric Flow Rate (dscmm)	567.825
Percent Isokinetic	89.07
Percent Excess Air	128.15
Fuel Factor,Fo	1.259
Ultimate CO2	16.60

FACILITY : MATHEY 26

DATE: 9/25/91
LOCATION: STACK
RUN NUMBER: RUN 2

SAMPLING PARAMETER PM10

Total Sampling Time (min.)	65.10
Corrected Barometric Pressure (in. Hg)	20.98
Absolute Stack Pressure,Ps(in. Hg)	20.92
Stack Static Pressure (in. H2O)	-0.80
Average Stack Temperature (deg. F)	316.82
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	20.49
Average Meter Pressure (in.H2O)	0.30
Average Meter Temperature (deg. F)	69.02
Moisture Collected (g)	185.00
Carbon Dioxide Concentration (%V)	7.06
Oxygen Concentration (%V)	12.01
Nitrogen Concentration (%V)	80.93
Dry Gas Meter Factor	1.00320
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.22
Standard Metered Volume,Vm(std) (dscf)	14.40
Standard Metered Volume,Vm(std) (dscm)	0.408
Standard Volume Water Vapor,Vw (scf)	8.72
Standard Volume Water Vapor,Vw (scm)	0.247
Stack Moisture (%V)	37.72
Mole Fraction Dry Stack Gas	0.623
Dry Molecular Weight	29.61
Wet Molecular Weight	25.23
Stack Gas Velocity,Vs (fpm)	4895.15
Stack Gas Velocity,Vs (mpm)	1492.04
Volumetric Flow Rate (acfm)	54254.63
Volumetric Flow Rate (acmm)	1536.491
Volumetric Flow Rate (dscfm)	16048.63
Volumetric Flow Rate (dscmm)	454.497
Percent Isokinetic	91.42
Percent Excess Air	128.15
Fuel Factor,Fo	1.259
Ultimate CO2	16.60

```
FACILITY : MATHEY 26

 ___________________________________________________________________
|DATE:      9/25/91                                                 |
|LOCATION:  STACK                                                   |
|RUN NUMBER:    RUN 3                                               |
|===================================================================|
|SAMPLING PARAMETER    PM10                                         |
|===================================================================|
|                                                                   |
|Total Sampling Time (min.)                                    70.22 |
|Corrected Barometric Pressure (in. Hg)                        20.98 |
|Absolute Stack Pressure,Ps(in. Hg)                            20.92 |
|Stack Static Pressure (in. H2O)                               -0.80 |
|Average Stack Temperature (deg. F)                           304.90 |
|Stack Area (sq.in.)                                         1596.00 |
|Metered Volume,Vm (cu.ft.)                                    21.83 |
|Average Meter Pressure (in.H2O)                                0.30 |
|Average Meter Temperature (deg. F)                            70.98 |
|Moisture Collected (g)                                       185.00 |
|Carbon Dioxide Concentration (%V)                              4.74 |
|Oxygen Concentration (%V)                                     10.83 |
|Nitrogen Concentration (%V)                                   84.43 |
|Dry Gas Meter Factor                                        1.00320 |
|Pitot Constant                                                 0.84 |
|___________________________________________________________________|
|                                                                   |
|Average Sampling Rate (dscfm)                                  0.22 |
|Standard Metered Volume,Vm(std) (dscf)                        15.29 |
|Standard Metered Volume,Vm(std) (dscm)                        0.433 |
|Standard Volume Water Vapor,Vw (scf)                           8.72 |
|Standard Volume Water Vapor,Vw (scm)                          0.247 |
|Stack Moisture (%V)                                           36.33 |
|Mole Fraction Dry Stack Gas      FLOW AND ISO'S CALC'ED       0.637 |
|Dry Molecular Weight             FROM AVG OF OTHER            29.19 |
|Wet Molecular Weight             TRAINS                       25.13 |
|Stack Gas Velocity,Vs (fpm)      ----------------------     5249.47 |
|Stack Gas Velocity,Vs    ALDHDS. MTLS/PAH'S AVG             1600.04 |
|Volumetric Flow Rate (acfm)                                58181.67 |
|Volumetric Flow Rate (acmm)                                1647.705 |
|Volumetric Flow Rate (dscfm)                               17869.49 |
|Volumetric Flow Rate (dscmm)                                506.064 |
|Percent Isokinetic                                            80.80 |
|Percent Excess Air                                            94.37 |
|Fuel Factor,Fo                                                2.124 |
|Ultimate CO2                                                   9.84 |
|___________________________________________________________________|
```

APPENDIX C.3

ALDEHYDES

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 1.00

SAMPLING PARAMETER ALDEHYDE

Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.37
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	310.72
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	31.05
Average Meter Pressure (in.H2O)	0.81
Average Meter Temperature (deg. F)	71.18
Moisture Collected (g)	284.50
Carbon Dioxide Concentration (%V)	6.83
Oxygen Concentration (%V)	12.98
Nitrogen Concentration (%V)	80.19
Dry Gas Meter Factor	1.00320
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.49
Standard Metered Volume,Vm(std) (dscf)	30.47
Standard Metered Volume,Vm(std) (dscm)	0.863
Standard Volume Water Vapor,Vw (scf)	13.41
Standard Volume Water Vapor,Vw (scm)	0.380
Stack Moisture (%V)	30.57
Mole Fraction Dry Stack Gas	0.694
Dry Molecular Weight	29.61
Wet Molecular Weight	26.06
Stack Gas Velocity,Vs (fpm)	3759.20
Stack Gas Velocity,Vs (mpm)	1145.80
Volumetric Flow Rate (acfm)	41664.46
Volumetric Flow Rate (acmm)	1179.937
Volumetric Flow Rate (dscfm)	19443.32
Volumetric Flow Rate (dscmm)	550.635
Percent Isokinetic	98.83
Percent Excess Air	158.17
Fuel Factor,Fo	1.160
Ultimate CO2	18.02

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 2.00

SAMPLING PARAMETER ALDEHYDE

Total Sampling Time (min.)	38.50
Corrected Barometric Pressure (in. Hg)	29.22
Absolute Stack Pressure,Ps(in. Hg)	29.21
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	319.56
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	21.41
Average Meter Pressure (in.H2O)	1.03
Average Meter Temperature (deg. F)	79.87
Moisture Collected (g)	284.50
Carbon Dioxide Concentration (%V)	8.24
Oxygen Concentration (%V)	10.30
Nitrogen Concentration (%V)	81.46
Dry Gas Meter Factor	1.00320
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.53
Standard Metered Volume,Vm(std) (dscf)	20.57
Standard Metered Volume,Vm(std) (dscm)	0.582
Standard Volume Water Vapor,Vw (scf)	13.41
Standard Volume Water Vapor,Vw (scm)	0.380
Stack Moisture (%V)	39.47
Mole Fraction Dry Stack Gas	0.605
Dry Molecular Weight	29.73
Wet Molecular Weight	25.10
Stack Gas Velocity,Vs (fpm)	4231.62
Stack Gas Velocity,Vs (mpm)	1289.80
Volumetric Flow Rate (acfm)	46900.49
Volumetric Flow Rate (acmm)	1328.222
Volumetric Flow Rate (dscfm)	18760.40
Volumetric Flow Rate (dscmm)	531.295
Percent Isokinetic	112.25
Percent Excess Air	91.79
Fuel Factor,Fo	1.286
Ultimate CO2	16.25

FACILITY : Mathy #26

DATE: 9-24-91
LOCATION: STACK
RUN NUMBER: 3.00

SAMPLING PARAMETER ALDEHYDE

Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.34
Stack Static Pressure (in. H2O)	-0.58
Average Stack Temperature (deg. F)	315.36
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	29.71
Average Meter Pressure (in.H2O)	0.81
Average Meter Temperature (deg. F)	63.14
Moisture Collected (g)	202.90
Carbon Dioxide Concentration (%V)	6.22
Oxygen Concentration (%V)	12.93
Nitrogen Concentration (%V)	80.85
Dry Gas Meter Factor	1.00320
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.47
Standard Metered Volume,Vm(std) (dscf)	29.60
Standard Metered Volume,Vm(std) (dscm)	0.838
Standard Volume Water Vapor,Vw (scf)	9.57
Standard Volume Water Vapor,Vw (scm)	0.271
Stack Moisture (%V)	24.43
Mole Fraction Dry Stack Gas	0.756
Dry Molecular Weight	29.51
Wet Molecular Weight	26.70
Stack Gas Velocity,Vs (fpm)	3486.43
Stack Gas Velocity,Vs (mpm)	1062.66
Volumetric Flow Rate (acfm)	38641.30
Volumetric Flow Rate (acmm)	1094.322
Volumetric Flow Rate (dscfm)	19490.62
Volumetric Flow Rate (dscmm)	551.974
Percent Isokinetic	95.79
Percent Excess Air	153.37
Fuel Factor,Fo	1.281
Ultimate CO2	16.31

FACILITY : Mathy #26

DATE: 9-25-91
LOCATION: STACK
RUN NUMBER: 4.00

SAMPLING PARAMETER	ALDEHYDE
Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	28.98
Absolute Stack Pressure,Ps(in. Hg)	28.94
Stack Static Pressure (in. H2O)	-0.58
Average Stack Temperature (deg. F)	316.76
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	21.27
Average Meter Pressure (in.H2O)	0.36
Average Meter Temperature (deg. F)	71.54
Moisture Collected (g)	176.60
Carbon Dioxide Concentration (%V)	6.43
Oxygen Concentration (%V)	11.09
Nitrogen Concentration (%V)	82.48
Dry Gas Meter Factor	1.00060
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.33
Standard Metered Volume,Vm(std) (dscf)	20.50
Standard Metered Volume,Vm(std) (dscm)	0.581
Standard Volume Water Vapor,Vw (scf)	8.33
Standard Volume Water Vapor,Vw (scm)	0.236
Stack Moisture (%V)	28.88
Mole Fraction Dry Stack Gas	0.711
Dry Molecular Weight	29.47
Wet Molecular Weight	26.16
Stack Gas Velocity,Vs (fpm)	3525.45
Stack Gas Velocity,Vs (mpm)	1074.56
Volumetric Flow Rate (acfm)	39073.78
Volumetric Flow Rate (acmm)	1106.569
Volumetric Flow Rate (dscfm)	18260.28
Volumetric Flow Rate (dscmm)	517.131
Percent Isokinetic	95.96
Percent Excess Air	103.63
Fuel Factor,Fo	1.526
Ultimate CO2	13.70

APPENDIX C.4

PAH

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 1.00

SAMPLING PARAMETER PAH

Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.37
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	326.04
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	42.08
Average Meter Pressure (in.H2O)	0.37
Average Meter Temperature (deg. F)	60.70
Moisture Collected (g)	399.70
Carbon Dioxide Concentration (%V)	9.22
Oxygen Concentration (%V)	10.33
Nitrogen Concentration (%V)	80.45
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.33
Standard Metered Volume,Vm(std) (dscf)	41.42
Standard Metered Volume,Vm(std) (dscm)	1.173
Standard Volume Water Vapor,Vw (scf)	18.85
Standard Volume Water Vapor,Vw (scm)	0.534
Stack Moisture (%V)	31.27
Mole Fraction Dry Stack Gas	0.687
Dry Molecular Weight	29.89
Wet Molecular Weight	26.17
Stack Gas Velocity,Vs (fpm)	3577.70
Stack Gas Velocity,Vs (mpm)	1090.48
Volumetric Flow Rate (acfm)	39652.79
Volumetric Flow Rate (acmm)	1122.967
Volumetric Flow Rate (dscfm)	17960.14
Volumetric Flow Rate (dscmm)	508.631
Percent Isokinetic	102.72
Percent Excess Air	94.55
Fuel Factor,Fo	1.146
Ultimate CO2	18.23

FACILITY : Mathy #26

DATE: 9-23-91
LOCATION: STACK
RUN NUMBER: 2.00

SAMPLING PARAMETER PAH

Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.38
Absolute Stack Pressure,Ps(in. Hg)	29.37
Stack Static Pressure (in. H2O)	-0.17
Average Stack Temperature (deg. F)	323.28
Stack Area (sq.in.)	1596.00
Metered Volume,Vm (cu.ft.)	43.49
Average Meter Pressure (in.H2O)	0.36
Average Meter Temperature (deg. F)	72.30
Moisture Collected (g)	398.10
Carbon Dioxide Concentration (%V)	7.55
Oxygen Concentration (%V)	10.01
Nitrogen Concentration (%V)	82.44
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84

Average Sampling Rate (dscfm)	0.34
Standard Metered Volume,Vm(std) (dscf)	41.88
Standard Metered Volume,Vm(std) (dscm)	1.186
Standard Volume Water Vapor,Vw (scf)	18.77
Standard Volume Water Vapor,Vw (scm)	0.532
Stack Moisture (%V)	30.95
Mole Fraction Dry Stack Gas	0.690
Dry Molecular Weight	29.61
Wet Molecular Weight	26.02
Stack Gas Velocity,Vs (fpm)	3542.92
Stack Gas Velocity,Vs (mpm)	1079.88
Volumetric Flow Rate (acfm)	39267.33
Volumetric Flow Rate (acmm)	1112.051
Volumetric Flow Rate (dscfm)	17931.93
Volumetric Flow Rate (dscmm)	507.832
Percent Isokinetic	104.03
Percent Excess Air	85.04
Fuel Factor,Fo	1.442
Ultimate CO2	14.49

```
FACILITY : Mathy #26

DATE:        9-24-91
LOCATION:    Outlet
RUN NUMBER:       3.00
==========================================================================
SAMPLING PARAMETER     PAH
==========================================================================

Total Sampling Time (min.)                                      125.00
Corrected Barometric Pressure (in. Hg)                           29.38
Absolute Stack Pressure,Ps(in. Hg)                               29.34
Stack Static Pressure (in. H2O)                                  -0.58
Average Stack Temperature (deg. F)                              326.60
Stack Area (sq.in.)                                            1596.00
Metered Volume,Vm (cu.ft.)                                       40.52
Average Meter Pressure (in.H2O)                                   0.33
Average Meter Temperature (deg. F)                               73.41
Moisture Collected (g)                                          325.00
Carbon Dioxide Concentration (%V)                                 7.18
Oxygen Concentration (%V)                                        11.77
Nitrogen Concentration (%V)                                      81.05
Dry Gas Meter Factor                                           0.98750
Pitot Constant                                                    0.84
--------------------------------------------------------------------------

Average Sampling Rate (dscfm)                                     0.31
Standard Metered Volume,Vm(std) (dscf)                           38.93
Standard Metered Volume,Vm(std) (dscm)                           1.102
Standard Volume Water Vapor,Vw (scf)                             15.32
Standard Volume Water Vapor,Vw (scm)                             0.434
Stack Moisture (%V)                                              28.25
Mole Fraction Dry Stack Gas                                      0.718
Dry Molecular Weight                                             29.62
Wet Molecular Weight                                             26.34
Stack Gas Velocity,Vs (fpm)                                    3466.96
Stack Gas Velocity,Vs (mpm)                                    1056.73
Volumetric Flow Rate (acfm)                                   38425.48
Volumetric Flow Rate (acmm)                                   1088.210
Volumetric Flow Rate (dscfm)                                  18139.15
Volumetric Flow Rate (dscmm)                                   513.701
Percent Isokinetic                                               95.60
Percent Excess Air                                              122.05
Fuel Factor,Fo                                                   1.272
Ultimate CO2                                                     16.44
```

APPENDIX D

D.1 Multipoint Linearity/ Drift Summary Tables
D.2 CEMDAS Printouts
D.3 Stripchart Tracings

APPENDIX D.1

MULTIPOINT LINEARITY/ DRIFT SUMMARY TABLES

O_2

INSTRUMENT: THERMOX WDG III					SERIAL NUMBER: 35983-1		
SPAN GAS CONC.: 25%V							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE (%V)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~ 2	092391	0812	Span:	18.0	18.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092391	1106	MID	7.99	7.7	-0.29	-1.16
~~1~~ 2	092391	1240	MID	7.99	7.7	0	0
~~1~~ 2	092391	1240	SPAN	18.0	18.4	0.4	1.6
~~2~~ 3	092391	1445	MID	7.99	8.1	0.4	1.6
~~2~~ 4	092391	1646	MID	7.99	7.5	-0.6	-2.4

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

THC

INSTRUMENT: RATFISCH 55					SERIAL NUMBER: 2122 91		
SPAN GAS CONC.: 100 ppmv							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmv)	OBSERVED GAS CONC. (ppmv)	DIFFERENCE (ppmv)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~2	092391	0812	Span:	95.0	95.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1 RE-CAL	092391	1239	SPAN	95.0	95.0	0	0
~~1~~ 2	092391	1240	SPAN	95.3	94.5	-0.5	-0.5
~~1~~ 2	092391	1240	MID	41.4	37.5	-3.9	-3.9
~~2~~ 3	092391	1646	SPAN	95.3	102.5	8.0	8.0
~~2~~ 3	092391	1646	MID	41.4	52.2	14.7	14.7

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

SO2

INSTRUMENT: WESTERN RESEARCH 721 AT2 SERIAL NUMBER: 90-721AT2-7654-1

SPAN GAS CONC.: 500 PPMV

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (PPMV)	OBSERVED GAS CONC. (PPMV)	DIFFERENCE PPMV	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~ 2	092391	0812	Span:	295	295	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092391	1106	SPAN	295	295	0	0
2	092391	1240	SPAN	295	302	7	1.40
3	092391	1646	MID	98.0	101.0	3	0.6

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

CO

INSTRUMENT: TECO 48					SERIAL NUMBER: 48297?58 - 236		
SPAN GAS CONC.: 500 PPMV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (PPMV)	OBSERVED GAS CONC. (PPMV)	DIFFERENCE (PPMV)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~2	092391	0812	Span:	474	474	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1 RE-CAL	092391	1103	SPAN	474	474	0	0
~~1~~2 RE-CAL	092391	1105	SPAN	474	474	0	0
~~1~~3	092391	1240	SPAN	474	482	8	1.60
~~2~~3	092391	1240	MID	92.1	92.5	0.4	0.08
~~2~~4	092391	1445	MID	92.1	89.3	-3.2	-0.64
~~2~~5	092391	1646	MID	92.1	93.7	4.4	0.88
~~3~~							
~~3~~							
~~3~~							
~~4~~							
~~4~~							
~~4~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

CO_2

INSTRUMENT: BECKMAN 865 SERIAL NUMBER: 0103778

SPAN GAS CONC.: 20% V

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE (%V)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
~~MP5~~ 2	092391	0812	Span:	17.0	17.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092391	1106	MID	9.96	10.3	0.39	1.95
~~1~~ 2	092391	1240	SPAN	17.0	17.0	0	0
~~1~~ 2	092391	1240	MID	9.91	10.3	0	0
~~2~~ 3	092391	1445	MID	9.91	10.4	0.1	0.5
~~2~~ 4	092391	1646	MID	9.91	9.8	-0.6	-3.0
~~2~~							
~~2~~							
~~3~~							
~~3~~							
~~4~~							
~~4~~							
~~4~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

10x

INSTRUMENT: TECO 10 AR SERIAL NUMBER: 25558-221

SPAN GAS CONC.: 250 ppmV

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmV)	OBSERVED GAS CONC. ppmV	DIFFERENCE ppmV	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092391	0812	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~ 2	092391	0812	Span:	201	201	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092391	1106	SPAN	201	192	-9	-3.60
~~1~~ 2	092391	1240	SPAN	201	201	9	3.60
~~1~~ 3	092391	1646	MID	97.0	99.0	2	0.80
~~2~~ 3	092391	1646	LOW	44.0	44.1	0.1	0.04

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

O_2

INSTRUMENT: THERMOX WDG III SERIAL NUMBER: 35983-1

SPAN GAS CONC.: 25 %V

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE %V	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~2	092491	0724	Mid:	7.99	8.9	0.91	3.64
MP~~5~~3	092491	0721	Span:	18.0	18.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1512	MID	7.99	7.75	-1.15	-4.6

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

THC

INSTRUMENT: RATFISCH 55					SERIAL NUMBER: 212291		
SPAN GAS CONC.: 100 PPMV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (PPMV	OBSERVED GAS CONC. (PPMV)	DIFFERENCE (PPMV)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
~~MP4~~			~~Mid:~~				
MP~~5~~2	092491	0721	Span:	95.0	95.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1131	SPAN	810	807	-3.0	
~~1~~ 2	092491	1148	SPAN	810	817	7.0	
~~1~~ SPECAL	092491	1153	SPAN	810	810	0	0
~~2~~ 4	092491	1202		95.3	95.6		
~~2~~ 5	092491	1512	SPAN	810.0	811	-1.0	
~~2~~ 5	092491	1512		95.3	103.0	7.4	
~~2~~ 5	092491	1512		41.4	44.3	2.9	
~~2~~ 5	092491	1512	ZERO	0	1.2	1.2	
3							
4							
4							
4							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

SO_2

INSTRUMENT: WESTERN RESEARCH 721 AT2 SERIAL NUMBER: 90-721AT2-7654-1

SPAN GAS CONC.: 500 ppmV

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmV)	OBSERVED GAS CONC. (ppmV)	DIFFERENCE ppmV	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			Low:				
MP~~4~~ 2	092491	0724	Mid:	98.0	95.9	-2.1	-0.42
MP~~5~~ 3	092491	0721	Span:	295	295	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1512	SPAN	295	294	-1.0	-0.2
1	092491	1512	MID	98.0	105.0	9.1	1.82
~~1~~							
~~2~~							
[illegible]							
[illegible]							
[illegible]							
[illegible]							
[illegible]							
[illegible]							
[illegible]							
[illegible]							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

CO

INSTRUMENT: TECO 48					SERIAL NUMBER: 4209758-236		
SPAN GAS CONC.: 500 PPM V							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (PPMV)	OBSERVED GAS CONC. (PPMV)	DIFFERENCE (PPMV)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~ 2	092491	0724	Mid:	92.1	99.0	6.9	1.38
MP~~5~~ 3	092491	0721	Span:	474	474	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1512	MID	92.1	92.1	-6.9	-1.38
~~2~~							
~~3~~							
~~4~~							
~~5~~							
~~6~~							
~~7~~							
~~8~~							
~~9~~							
~~10~~							
~~11~~							
~~12~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

CO₂

INSTRUMENT: BECKMAN 865					SERIAL NUMBER: 0103778		
SPAN GAS CONC.: 20 % V							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE (%V)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~2	092491	0724	Mid:	9.91	9.8	-0.11	-0.55
MP~~5~~3	092491	0721	Span:	17.0	17.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1512	MID	9.91	10.2	0.4	2.0

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

x10x

INSTRUMENT: TECO 10AR				SERIAL NUMBER: 25558-221			
SPAN GAS CONC.: 250 ppmV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmV)	OBSERVED GAS CONC. (ppmV)	DIFFERENCE ppmV	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092491	0721	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
MP3			Low:				
MP~~4~~ 2	092491	0724	Mid:	97.0	99.4	2.4	0.96
MP~~5~~ 3	092491	0721	Span:	201	201	0	0
~~MP6~~			Mid:				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092491	1512	SPAN	201	193.5	-7.5	-3.0
1	092491	1512	MID	97.0	96.0	-3.4	-1.36

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

O_2

INSTRUMENT: THERMOX WDG III					SERIAL NUMBER: 35583-1		
SPAN GAS CONC.: 25% V							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE %V	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~ 2	092591	0730	Mid:	7.99	7.5	-0.49	-1.96
MP~~5~~ 3	092591	0728	Span:	18.0	18.0	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	MID	7.99	7.4	-0.1	-0.4
1	092591	1555	SPAN	18.0	18.4	0.4	1.60

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

THC

INSTRUMENT: RATFISCH 55					SERIAL NUMBER: 212291		
SPAN GAS CONC.: 1000 ppmV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmV)	OBSERVED GAS CONC. (ppmV)	DIFFERENCE (ppmV)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
MP~~3~~ 2	092591	0730	Low:	95.3	96.9	1.6	0.16
~~MP4~~			~~Mid:~~				
MP~~5~~ 3	092591	0728	Span:	810	810	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	SPAN	810	817	7	0.70
1	092591	1555	LOW	95.3	97.6	0.7	0.07
~~1~~							
~~2~~							
~~2~~							
~~3~~							
~~3~~							
~~4~~							
~~4~~							
~~5~~							
~~5~~							
~~6~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

SO2

INSTRUMENT: WESTERN ~~RESEARCH~~ 721 AT2				SERIAL NUMBER: 90-721AT2-7654-1			
SPAN GAS CONC.: 500 PPMV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (PPMV)	OBSERVED GAS CONC. (PPMV)	DIFFERENCE PPMV	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~2	092591	0730	Mid:	98 2	98.9	0.7	0.14
MP~~5~~3	092591	0728	Span:	295	293	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	SPAN	295	293	-2.0	-0.4
1	092591	1555	MID	98.2	106.4	7.5	1.5
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							
~~[illegible]~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

CO

INSTRUMENT: TECO 48					SERIAL NUMBER: 4829758-236		
SPAN GAS CONC.: 500 ppmv							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmv)	OBSERVED GAS CONC. (ppmv)	DIFFERENCE ppmv	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~ 2	092591	0730	Mid:	92.1	91.9	0.2	0.04
MP~~5~~ 3	092591	0728	Span:	474	474	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	MID	92.1	90.0	-1.9	-0.38
1	092591	1555	SPAN	474	466	-8.0	-1.6

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

CO₂

INSTRUMENT: BECKMAN 865 SERIAL NUMBER: 0103778

SPAN GAS CONC.: 20% V

RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (%V)	OBSERVED GAS CONC. (%V)	DIFFERENCE (%V)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			Low:				
MP~~4~~2	092591	0730	Mid:	10.3	10.0	-0.3	-1.50
MP~~5~~3	092591	0728	Span:	17.0	17.0	0	0
MP6			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	MID	10.3	10.5	0.5	2.5
1	092591	1555	SPAN	17.0	17.2	0.2	1.0

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

ND indicates no data is available.

NOx

INSTRUMENT: TECO 10 AR					SERIAL NUMBER: 25558-221		
SPAN GAS CONC.: 250 ppmV							
RUN NUMBER	DATE	TIME		CERTIFIED GAS CONC. (ppmV)	OBSERVED GAS CONC. (ppmV)	DIFFERENCE (ppmV)	DIFFERENCE PERCENT SCALE (%)
MULTIPOINT LINEARITY (1)							
MP1	092591	0728	Zero:	0	0	0	0
~~MP2~~			~~Low:~~				
~~MP3~~			~~Low:~~				
MP~~4~~ 2	092591	0730	Mid:	97.0	101.1	4.1	1.64
MP~~5~~ 3	092591	0728	Span:	201	201	0	0
~~MP6~~			~~Mid:~~				
REFERENCE METHOD TEST RUN DRIFT SUMMARY (2)							
1	092591	1555	MID	97.0	104.0	3.0	1.2
1	092591	1555	SPAN	201.0	200	1.0	0.4
~~2~~							
~~2~~							
~~3~~							
~~3~~							
~~4~~							
~~4~~							
~~5~~							
~~5~~							
~~6~~							
~~6~~							

(1) Difference Percent Scale = (observed conc. - certified conc.)/span value • 100%.

(2) Difference Percent Scale = (current observed conc. - previous observed conc.)/span value • 100%.

ND indicates no data is available.

APPENDIX D.2

CEMDAS PRINTOUTS

```
                           CALIBRATION SUMMARY
                           09-23-1991   08:12:27
               CALIBRATION FILE NAME =D:\CEMDATA\0923CAL3.CAL

=============================================================================
Chan. Name Units |     Zero        |     Span      |   Slope     Int.
                 | Conc.  Resp.    | Conc.  Resp.  |
=============================================================================
  1   O2    %      0.00    0.0003      18.00  0.073     246.067      -0.08
  2   THC   PPMV   0.00    0.1049      95.00  0.948     112.745     -11.83
  3   SO2   PPMV   0.00    0.0014     295.00  0.058    5226.944      -7.24
  4   CO    PPMV   0.00    0.0001     474.00  0.010   48379.250      -6.81
  5   CO2   %      0.00    0.0008      17.00  0.082     209.815      -0.17
  6   NOX   PPMV   0.00    -.0048     201.00  2.197      91.284       0.43
  7                0.00    0.0000       0.00  0.000       1.000       0.00
  8                0.00    0.0000       0.00  0.000       1.000       0.00
  9                0.00    0.0000       0.00  0.000       1.000       0.00
  10               0.00    0.0000       0.00  0.000       1.000       0.00
  11               0.00    0.0000       0.00  0.000       1.000       0.00
  12               0.00    0.0000       0.00  0.000       1.000       0.00
  13               0.00    0.0000       0.00  0.000       1.000       0.00
  14               0.00    0.0000       0.00  0.000       1.000       0.00

=============================================================================









                   Press Shift-PrtSc to Print Out Table
                           Press <C> to Continue



                       CEM INSTRUMENT DRIFT SUMMARY
                           09-23-1991   08:12:56


=============================================================================
Chan. Name Units |    Zero Conc.    |     Span         |  Drift % of Scale
                 |Actual   Observed|Actual   Observed|  Zero      Span
=============================================================================
  1   O2    %      0.000   0.321       18.00   17.98   1.29        -0.09
  2   THC   PPMV   0.000   %-7.614     95.00   95.07  -0.76         0.01
  3   SO2   PPMV   0.000   4.026      295.00  297.52   0.08         0.05
  4   CO    PPMV   0.000   8.842      474.00  469.44   0.18        -0.09
```

```
 6   NOX    PPMV    0.000   0.537     201.00  207.55   0.05          0.05
 7                  0.000   0.000       0.00    0.00   0.00          0.00
 8                  0.000   0.000       0.00    0.00   0.00          0.00
 9                  0.000   0.000       0.00    0.00   0.00          0.00
10                  0.000   0.000       0.00    0.00   0.00          0.00
11                  0.000   0.000       0.00    0.00   0.00          0.00
12                  0.000   0.000.      0.00    0.00   0.00          0.00
13                  0.000   0.000       0.00    0.00   0.00          0.00
14                  0.000   0.000       0.00    0.00   0.00          0.00

==========================================================================
```

```
                   Press Shift-PrtSc to Print Out Table
                          Press (C) to Continue

RADIAN CORPORATION      0923RUNA

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY#16
NEW RICHMOND,WI
Performed for:                EPA
Date Printed = 09-23-1991 , Current Time = 08:14:05
File Name = D:\CEMDATA\092391.PRN   Calibration File:D:\CEMDATA\0923CAL3.CAL


 09-23-1991      O2      THC     SO2     CO      CO2     NOX
 Time            %       PPMV    PPMV    PPMV    %       PPMV
==================================================================================================================================
08:14:28        -0.0    -0.4    -1.2    -5.1    -0.1    -1.1
08:15:28         2.3   316.4    29.6    -7.6     0.2    46.4
08:16:28         9.2   589.4   151.5    47.7     7.3   151.8
08:17:28         9.7   674.8   155.2    62.2     8.7   158.2
08:18:28         9.6   716.4   157.4    66.9     8.8   159.7
08:19:28         9.5   706.4   162.3    65.1     8.9   160.1
08:20:28         9.8   711.4   161.8    66.7     8.8   158.4
08:21:28         9.8   676.8   161.4    67.5     8.7   157.6
08:22:28         9.9   674.0   160.9    66.7     8.5   156.8
08:23:28        10.1   654.3   159.5    61.7     8.5   154.1
08:24:28        10.5   650.5   153.6    57.3     8.1   149.7
08:25:28        10.4   653.8   156.1    57.3     8.2   150.5
08:26:28        10.5   640.0   154.4    56.1     8.2   149.6
08:27:27        10.5   643.8   153.1    56.3     8.1   149.9
08:28:27        10.4   643.9   154.4    56.4     8.1   150.1
08:29:27        10.2   639.9   155.6    53.8     8.2   153.0
08:30:27         9.9   643.4   156.4    57.1     8.4   156.6
08:31:27         9.6   679.1   160.7    63.1     8.6   159.3
08:32:27         9.5   682.2   163.3    60.7     8.7   160.8
08:33:27         9.3   685.0   164.9    61.2     8.8   163.0
08:34:27         9.2   684.5   165.7    60.8     8.8   161.9
08:35:27         9.4   666.3   165.6    62.2     8.7   161.3
```

Start PAH Run 1
metals Run 1

```
08:37:27        10.0   638.8   156.5    55.6     8.2   152.[illegible]
08:38:27         9.9   628.8   156.5    53.0     8.2   155.5
08:39:27         9.9   604.9   156.6    49.4     8.2   154.3
08:40:26        10.1   618.3   153.2    51.2     8.0   151.2
08:41:26        10.1   603.2   153.6    48.6     8.1   152.2
08:42:26        10.3   604.2   152.2    48.5     8.1   150.6
08:43:26        10.5   605.2   152.7    49.0     8.0   147.2
08:44:26        12.2   585.6   126.1    86.2     6.6   118.2
08:45:26        10.3   633.0   141.2    81.1     7.9   151.0
08:46:26         9.8   630.7   157.8    53.4     8.3   157.2
08:47:26         9.7   625.8   159.6    49.8     9.0   156.0
08:48:26         9.8   598.5   156.8    43.6     9.1   154.4
08:49:26         9.8   589.2   155.2    42.7     9.0   153.6
08:50:26         9.9   574.5   152.6    38.3     8.8   151.2
08:51:26        10.0   529.5   148.2    35.1     8.8   149.4
08:52:26        10.3   526.3   145.9    31.1     9.3   149.2
08:53:25        10.1   526.9   145.8    40.4     9.5   148.8
08:54:25        10.2   525.5   147.3    55.7     9.4   148.4
08:55:25        10.5   529.3   148.5    55.6     9.4   146.9
08:56:25        10.6   548.0   151.7    57.8     9.4   148.8
08:57:25        10.7   532.2   151.9    45.6     9.4   146.6
08:58:25        10.4   548.3   149.7    41.0     9.6   149.4
08:59:25         9.9   542.0   154.4    36.8    10.0   154.1
```

```
09-23-1991      O2     THC     SO2     CO      CO2    NOX
Time            %      PPMV    PPMV    PPMV    %      PPMV
=====================================================================================================================
09:00:25         9.4   539.6   157.4   33.4    10.3   157.5
09:01:25         9.1   515.7   158.0   31.8    10.5   157.5
09:02:25         9.3   507.6   152.6   32.2    10.4   155.5
09:03:25         9.7   497.5   150.2   34.4    10.1   152.5
09:04:25        10.1   498.3   153.0   37.9     9.8   149.4
09:05:25        10.7   498.5   149.5   36.9     9.5   145.3
09:06:24        10.9   486.2   145.5   34.6     9.3   143.0
09:07:24        10.9   501.1   147.0   34.9     9.4   145.5
09:08:24        10.9   501.1   148.0   32.7     9.5   145.1
09:09:24        10.8   499.6   147.4   32.2     9.5   144.5
09:10:24        10.8   504.0   145.9   31.3     9.5   140.6
09:11:24        10.6   508.3   147.2   31.5     9.7   137.9
09:12:24        11.1   486.2   135.2   30.5     9.2   118.6
09:13:24        11.1   496.2   140.1   29.8     9.1   121.9
09:14:24        10.6   511.3   145.4   34.1     9.6   122.8
09:15:24        10.6   512.2   146.5   32.6     9.7   126.4
09:16:24        10.7   467.9   147.5   28.3     9.6   127.6
09:17:24        12.0   484.4   129.3   43.2     8.4   111.1
09:18:24        11.1   502.5   141.4   38.3     9.2   126.5
09:19:23        11.0   483.0   146.4   33.7     9.3   125.2
09:20:23        11.0   481.1   145.1   33.2     9.2   123.7
09:21:23        11.3   483.7   144.1   35.2     9.2   121.1
09:22:23        11.2   493.1   145.7   34.0     9.3   124.0
09:23:23        11.1   516.0   146.7   34.8     9.3   121.5
09:24:23        11.1   524.6   145.2   34.5     9.3   119.7
09:25:23        10.9   548.0   148.7   36.4     9.6   122.4
09:26:23        10.8   535.4   148.6   34.1     9.6   123.8
09:27:23        10.5   532.8   149.6   35.3     9.7   125.2
09:28:23        10.6   536.3   154.2   40.8     9.8   128.3
09:29:23        10.8   554.1   153.9   43.0     9.7   125.4
09:30:23        11.0   541.4   151.6   39.3     9.4   124.6
09:31:23        11.1   525.1   149.4   34.2     9.3   122.5
09:32:22        11.2   530.5   148.4   37.5     9.2   119.2
09:33:22        11.1   534.9   146.1   35.8     9.2   118.2
09:34:22        11.0   534.3   148.3   38.4     9.4   124.6
09:35:22        10.9   518.0   148.1   37.2     9.5   125.0
09:36:22        10.8   556.7   150.8   39.8     9.6   126.1
09:37:22        10.6   567.3   152.9   42.1     9.7   126.4
09:38:22        10.5   589.9   156.1   44.3     9.8   129.2
09:39:22        10.4   572.0   155.0   42.4     9.8   126.6
09:40:22        11.2   578.0   139.4   40.7     8.9   129.6
09:41:22         9.7   569.5   167.6   48.5    10.3   154.2
09:42:22         9.8   542.8   164.1   48.2    10.1   151.7
09:43:22         9.9   542.6   163.2   52.4     9.9   149.5
09:44:22        10.1   538.5   159.4   45.2     9.9   148.8
09:45:22        10.3   523.6   158.8   41.3     9.7   146.9
09:46:21        10.3   533.7   158.6   40.5     9.7   146.8
09:47:21        10.4   540.1   157.8   43.1     9.7   146.6
09:48:21        10.4   542.3   157.5   41.9     9.7   148.2
09:49:21        10.0   552.4   161.2   44.6     9.9   152.7
09:50:21         9.8   548.8   163.6   42.9    10.0   154.7
09:51:21         9.6   554.2   166.2   43.4    10.2   155.2
09:52:21         9.5   572.2   167.1   47.9    10.3   157.3
09:53:21         9.4   554.2   167.5   49.6    10.3   159.1
09:54:21         9.8   546.6   165.0   45.9    10.0   154.9
```

BLOWBACK
REMOVE FROM AVGS. PB

```
 09-23-1991      O2      THC     SO2     CO      CO2     NOX
 Time            %       PPMV    PPMV    PPMV    %       PPMV
==========================================================================================================================
 09:56:21        10.2    545.0   159.1   44.2     9.6   152.2
 09:57:21        10.3    540.1   157.6   39.1     9.6   150.5
 09:58:21        10.3    528.0   156.9   35.4     9.6   150.0
 09:59:20        10.3    529.7   154.4   35.9     9.5   149.3
 10:00:20        10.2    538.9   155.5   39.1     9.6   149.0
 10:01:20        10.3    538.8   156.3   39.7     9.7   150.0
 10:02:20        10.0    549.5   157.3   42.2     9.8   151.9
 10:03:20         9.8    579.0   161.1   45.3     9.9   154.1
 10:04:20         9.7    588.5   165.2   48.1    10.1   155.9
 10:05:20         9.6    594.4   165.6   47.3    10.1   155.6
 10:06:20         9.4    599.8   164.3   50.1    10.1   155.7
 10:07:20         9.5    590.7   164.2   45.3    10.1   151.9
 10:08:20         9.6    566.9   156.2   39.0    10.0   148.1
 10:09:20         9.8    532.6   151.9   32.2     9.8   143.5
 10:10:20         9.9    541.1   147.4   34.3     9.6   143.7
 10:11:20        10.0    533.0   149.1   36.8     9.6   146.6
 10:12:19        10.3    538.2   148.5   42.4     9.4   145.5
 10:13:19        10.4    540.8   148.5   43.8     9.3   143.7
 10:14:19        10.4    548.3   148.3   45.6     9.3   143.6
 10:15:19        10.2    561.9   150.2   51.2     9.6   147.2
 10:16:19         9.8    584.8   153.7   54.0     9.6   149.8
 10:17:19         9.8    597.6   153.2   54.5     9.7   150.5
 10:18:19         9.6    601.8   153.7   57.1     9.8   154.1
 10:19:19         9.9    582.1   149.0   50.8     9.4   148.3
 10:20:19        10.3    556.9   142.2   45.8     9.1   144.6
 10:21:19        10.2    562.6   144.3   43.5     9.1   145.4
 10:22:19        10.5    562.0   139.2   42.1     8.8   142.1
 10:23:19        10.9    551.8   135.7   42.7     8.5   137.7
 10:24:19        11.0    543.3   135.8   43.7     8.5   139.0
 10:25:18        11.0    540.4   133.8   42.4     8.4   135.7
 10:26:18        11.0    529.2   132.4   41.9     8.4   134.2
 10:27:18        11.0    542.1   134.3   41.4     8.4   137.2
 10:28:18        10.9    547.4   134.3   40.1     8.4   136.4
 10:29:18        10.8    555.1   135.5   36.8     8.4   134.5
 10:30:18        10.4    573.6   141.0   38.4     8.5   139.9
 10:31:18        10.2    594.3   143.3   40.3     8.6   140.5
 10:32:18        10.1    583.7   142.7   40.1     8.4   143.4
 10:33:18        10.1    574.9   144.1   38.8     8.2   140.9
 10:34:18        10.3    564.7   142.8   36.9     8.1   140.0
 10:35:18        10.5    560.2   139.6   36.6     7.8   136.0
 10:36:18        10.8    541.2   136.7   37.6     7.4   133.2
 10:37:18        10.8    534.3   138.8   38.0     7.4   135.7
 10:38:17        11.0    544.4   134.6   35.0     7.2   131.8
 10:39:17        10.9    526.6   134.3   35.5     7.2   133.2
 10:40:17        10.9    555.3   135.4   42.4     7.3   131.8
 10:41:17        10.6    559.4   141.0   44.9     7.4   138.5
 10:42:17        10.4    582.2   140.8   46.7     7.4   135.2
 10:43:17        10.2    592.0   140.8   52.2     7.4   140.0
 10:44:17        10.3    602.0    82.8   53.3     7.3   135.4
 10:45:17        12.8    599.2   103.8   43.6     5.7   109.6
 10:46:17        12.8    600.4   105.5 -4732.5    5.6   108.8
 10:47:17        13.0    587.4   105.1  -280.0    5.4   107.6
 10:48:17        13.0    580.2   106.2  -280.5    5.2   111.3
==========================================================================================================================
 Avg.=           10.3    560.6   147.7    9.0     8.9   141.2
```

end PAH Run 1
metals Run 1

```
                    CALIBRATION SUMMARY
                    09-23-1991   11:02:56
           CALIBRATION FILE NAME =D:\CEMDATA\0923CAL5.CAL

=========================================================================
Chan. Name Units ¦      Zero        ¦      Span      ¦   Slope     Int.
                 ¦  Conc.   Resp.   ¦ Conc.  Resp.   ¦
=========================================================================

  1   02    %     0.00    0.0003     18.00  0.073     246.067     -0.08
  2   THC   PPMV  0.00    0.1049     95.00  0.948     112.745    -11.83
  3   S02   PPMV  0.00    0.0014    295.00  0.058    5226.944     -7.24
  4   C0    PPMV  0.00    0.0007    474.00  0.047   10218.815     -7.57
  5   C02   %     0.00    0.0008     17.00  0.082     209.815     -0.17
  6   NOX   PPMV  0.00    -.0048    201.00  2.197      91.284      0.43
  7               0.00    0.0000      0.00  0.000       1.000      0.00
  8               0.00    0.0000      0.00  0.000       1.000      0.00
  9               0.00    0.0000      0.00  0.000       1.000      0.00
  10              0.00    0.0000      0.00  0.000       1.000      0.00
  11              0.00    0.0000      0.00  0.000       1.000      0.00
  12              0.00    0.0000      0.00  0.000       1.000      0.00
  13              0.00    0.0000      0.00  0.000       1.000      0.00
  14              0.00    0.0000      0.00  0.000       1.000      0.00

=========================================================================
```

RE-CAL CO PB

```
                Press Shift-PrtSc to Print Out Table
                      Press (C) to Continue
RADIAN CORPORATION      0923RUNB

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-23-1991  Current Time = 11:03:52
File Name = D:\CEMDATA\092391.PRN   Calibration File:D:\CEMDATA\0923CAL5.CAL

09-23-1991      02      THC      SO2      CO      CO2      NOX
Time            %       PPMV     PPMV     PPMV    %        PPMV
==========================================================================================================================
```

```
==================================================================================================================
```

~~Avg.= 0.0 0.0 0.0 0.0 0.0 0.0~~

```
==================================================================================================================


                         CALIBRATION SUMMARY
                         09-23-1991   11:04:45
              CALIBRATION FILE NAME =D:\CEMDATA\0923CAL6.CAL

 ===================================================================
 Chan. Name Units |      Zero         |     Span       |  Slope      Int.
                  | Conc.  Resp.      | Conc.  Resp.   |
 ===================================================================

   1    O2    %     0.00     0.0003     18.00  0.073     246.067     -0.08
   2    THC   PPMV  0.00     0.1049     95.00  0.948     112.745    -11.83
   3    SO2   PPMV  0.00     0.0014    295.00  0.058    5226.944     -7.24
   4    CO    PPMV  0.00     0.0006    474.00  0.047   10180.517     -5.76
   5    CO2   %     0.00     0.0008     17.00  0.082     209.815     -0.17
   6    NOX   PPMV  0.00     -.0048    201.00  2.197      91.284      0.43
   7                0.00     0.0000      0.00  0.000       1.000      0.00
   8                0.00     0.0000      0.00  0.000       1.000      0.00
   9                0.00     0.0000      0.00  0.000       1.000      0.00
  10                0.00     0.0000      0.00  0.000       1.000      0.00
  11                0.00     0.0000      0.00  0.000       1.000      0.00
  12                0.00     0.0000      0.00  0.000       1.000      0.00
  13                0.00     0.0000      0.00  0.000       1.000      0.00
  14                0.00     0.0000      0.00  0.000       1.000      0.00

 ===================================================================


                  Press Shift-PrtSc to Print Out Table
                         Press <C> to Continue

RADIAN CORPORATION      0923RUND

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-23-1991  Current Time = 11:05:38
File Name = D:\CEMDATA\092391.PRN   Calibration File:D:\CEMDATA\0923CAL6.CAL

 09-23-1991      O2      THC     SO2     CO      CO2     NOX
 Time            %       PPMV    PPMV    PPMV    %       PPMV
==================================================================================================================
```

RE-CAL CO

PB

11:05:55	0.0	29.7	-0.2	-1.7	-0.0	-0.2
11:06:05	0.0	29.6	0.2	-2.3	-0.0	-0.2
11:06:15	0.0	29.3	-0.1	-2.3	-0.0	-0.2
11:06:45	-0.0	29.3	-0.0	-2.2	-0.0	0.2
11:07:15	3.4	29.4	1.0	17.3	5.4	0.1
11:07:24	6.7	29.1	0.7	61.5	9.6	-0.1
11:07:34	7.2	29.0	0.3	76.1	10.1	-0.1
11:07:44	7.4	28.9	0.2	84.1	10.2	-0.2
11:07:54	7.4	28.7	-0.1	87.0	10.3	-0.2
11:08:04	7.5	28.5	-0.1	88.8	10.3	-0.2
11:08:14	7.5	28.5	-0.1	88.8	10.2	-0.2
11:08:24	7.6	28.3	0.0	88.8	10.3	-0.2
11:08:34	7.7	28.3	-0.1	89.9	10.4	-0.2
11:08:44	7.7	28.0	-0.1	88.8	10.4	-0.2
11:08:54	7.7	26.6	-0.2	89.9	10.3	-0.1
11:09:04	7.7	26.4	1.3	90.0	10.3	18.0
11:09:34	3.3	26.1	134.6	63.8	4.1	156.5
11:10:06	0.2	25.8	267.1	7.8	0.3	188.3
11:10:31	0.2	25.3	286.0	-2.6	0.1	190.4
11:10:41	0.0	24.8	293.2	-5.3	0.0	191.6
11:10:51	0.0	24.7	294.5	-6.2	-0.0	192.1
11:11:51	-0.0	24.2	296.9	-6.9	-0.1	192.1
11:12:51	3.2	270.9	242.0	-3.2	1.2	146.6
11:13:51	~~11.8~~	~~477.9~~	~~123.1~~	~~25.4~~	~~6.5~~	~~109.4~~
11:14:51	13.0	526.5	102.7	31.3	6.5	103.1
11:15:51	13.2	541.7	98.8	30.2	6.3	100.4
11:16:51	13.2	544.9	96.0	29.6	6.5	100.4
11:17:51	13.2	547.2	95.4	27.9	6.5	100.8
11:18:51	13.3	541.7	94.0	24.3	6.4	99.4
11:19:51	13.2	525.2	93.9	22.2	6.5	100.4
11:20:51	13.0	515.7	95.2	24.1	6.8	102.3
11:21:51	12.9	550.4	98.9	29.1	6.9	104.3
11:22:50	12.9	558.7	102.1	29.7	6.8	105.6
11:23:50	12.8	569.6	104.9	28.5	6.9	108.1
11:24:50	12.8	574.9	104.2	26.9	7.0	106.8
11:25:50	12.8	577.1	104.0	27.1	6.9	106.2
11:26:50	12.9	578.3	103.8	29.8	6.8	105.0
11:27:50	12.9	571.7	104.0	29.8	6.8	103.9
11:28:50	13.1	551.3	100.5	27.7	6.5	101.4
11:29:50	13.1	546.1	99.2	28.8	6.4	100.7
11:30:50	13.1	538.9	101.6	30.2	6.5	100.5
11:31:50	13.2	542.1	99.6	30.9	6.4	100.2
11:32:50	13.2	548.9	101.3	27.7	6.3	101.3
11:33:50	13.1	559.5	101.5	27.0	6.5	102.6
11:34:50	13.0	560.2	102.7	27.2	6.5	103.3

QC check O_2/CO/CO_2

NOx/SO_2

ON-LINE 11:11

RUN #1

Run 2

09-23-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
11:35:49	13.0	572.7	102.7	28.3	6.6	105.0
11:36:49	13.0	584.8	104.9	29.1	6.7	107.1
11:37:49	12.8	579.3	104.6	31.0	6.8	108.2
11:38:49	13.0	590.6	104.7	32.2	6.6	105.8
11:39:49	13.0	572.8	102.4	30.0	6.5	105.4
11:40:49	13.1	578.4	101.1	31.6	6.4	104.1
11:41:49	13.1	569.9	100.3	31.5	6.2	102.2
11:42:49	13.3	553.0	99.2	28.5	6.1	101.2
11:43:49	13.3	552.9	98.7	31.0	6.2	102.0
11:44:49	13.2	555.7	100.4	33.6	6.1	101.4
11:45:49	13.4	564.0	98.0	31.0	6.0	101.3
11:46:49	13.2	570.3	98.4	30.0	6.2	102.4
11:47:49	13.1	583.8	99.2	30.5	6.1	103.7
11:48:48	13.1	567.6	98.8	30.4	6.1	104.5
11:49:48	13.0	584.1	98.5	32.2	6.2	105.4
11:50:48	12.9	579.9	103.0	29.6	6.3	108.5
11:51:48	12.9	579.2	102.3	34.0	6.1	106.8
11:52:48	13.0	575.4	101.3	33.7	5.8	106.0
11:53:48	13.1	568.0	99.7	29.5	5.6	104.6
11:54:48	13.1	563.5	98.7	27.8	6.3	102.9
11:55:48	13.2	569.3	96.2	34.4	7.2	102.0
11:56:48	13.2	562.7	97.6	37.3	7.2	102.6
11:57:48	13.3	558.2	97.3	38.7	7.2	100.9
11:58:48	14.2	566.0	86.0	33.7	6.4	88.4
11:59:48	12.3	557.2	113.0	39.8	8.3	110.3
12:00:48	12.0	555.9	115.1	39.7	8.6	113.3
12:01:47	12.4	586.3	110.3	37.7	8.2	111.8
12:02:47	12.7	584.3	107.0	35.7	7.8	109.1
12:03:47	12.5	605.5	108.2	36.2	8.1	111.4
12:04:47	12.5	598.9	108.7	37.4	8.1	111.3
12:05:47	12.6	588.0	109.6	33.8	8.0	109.7
12:06:47	12.9	583.1	105.4	31.7	7.8	106.7
12:07:47	13.0	566.4	103.4	39.3	7.6	104.3
12:08:47	13.0	555.3	102.7	38.4	7.5	103.4
12:09:47	13.0	557.1	102.6	40.2	7.6	104.2
12:10:47	13.0	551.9	103.0	28.9	7.5	104.4
12:11:47	13.1	550.2	103.9	27.4	7.4	103.9
12:12:47	13.0	567.1	104.3	29.8	7.5	103.8
12:13:47	13.0	583.2	105.2	33.2	7.7	105.8
12:14:46	12.9	583.3	104.4	31.6	7.7	104.8
12:15:46	12.9	598.7	103.4	40.8	7.7	105.2
12:16:46	12.8	574.8	105.0	32.7	7.9	108.0
12:17:46	12.8	589.0	106.1	28.5	7.9	108.5
12:18:46	12.9	584.8	105.3	26.0	7.7	107.5
12:19:46	12.9	567.2	102.9	25.4	7.6	105.3
12:20:46	13.0	564.9	100.4	24.4	7.4	103.4
12:21:46	13.0	549.4	99.9	23.4	7.3	103.6
12:22:46	13.2	406.4	91.3	21.0	7.2	84.6
12:23:46	3.3	76.8	16.0	1.8	0.3	8.8
12:24:46	-0.1	40.3	-1.9	-4.2	0.3	-0.8
12:25:34	-0.1	27.8	-1.9	-4.3	0.5	-0.8
12:25:49	-0.1	20.4	-2.2	-4.3	0.5	-0.9
12:26:04	-0.1	18.7	-2.3	-4.3	0.5	-0.9
12:26:19	-0.1	-3.5	-2.0	-4.3	0.5	-0.9
12:26:34	-0.1	-16.0	-2.1	-4.4	0.5	-0.8

BLOWBACK
REMOVE FROM AVGS. RB

END ACQ #1

END RUN

```
09-23-1991       O2      THC     SO2     CO      CO2     NOX
Time             %       PPMV    PPMV    PPMV    %       PPMV
=============================================================================================================================
12:27:03          0.2     66.2   -0.5    -3.2     0.6    -0.3
12:27:18         -0.1     70.2   -1.8    -3.2     0.5    -0.7
12:27:33         -0.1     69.8   -1.8    -3.2    -0.2    -0.8
12:27:48         -0.1     69.0   -2.2    -3.2    -0.2    -0.8
12:28:03         -0.1     68.4   -2.0    -3.2    -0.2    -0.8
12:28:18         -0.1     67.8   -2.1    -3.2    -0.2    -0.8
12:28:33         -0.1     89.5   -2.2    -3.2    -0.2    -0.8
12:28:48         -0.1     90.4   -2.1    -3.2    -0.2    -0.8
12:29:03         -0.1     89.5   -2.3    -3.2    -0.2    -0.8
12:29:18         -0.1     88.3   -2.6    -3.2    -0.2    -0.8
12:29:33          0.1     96.5   -2.6    -6.0    -0.2    -0.9
12:29:48          0.2     96.3   -2.7    -7.0    -0.3    -0.9
12:30:03         -0.1     25.3   -2.2    -3.8    -0.2    -0.6
12:30:18         -0.1    -25.3   -2.4    -3.2    -0.2    -0.9
12:30:33         -0.1    -31.2   -2.6    -4.2    -0.2    -0.9
12:30:48         -0.1    -17.3   -2.4    -4.3    -0.2    -0.9
12:31:02         -0.1    -18.1   -2.4    -4.3    -0.2    -0.9
12:31:17         -0.1    -18.7   -2.2    -4.3    -0.2    -0.9
12:31:32         -0.1    -19.4   -2.4    -4.3    -0.2    -0.9
12:31:47         -0.1    -20.0   -2.3    -4.3    -0.2    -0.9
12:32:02         -0.1    -20.5   -2.1    -4.3    -0.2    -0.9
12:32:17         -0.1    -18.8   -2.3    -4.3    -0.2    -0.9
12:32:32         -0.2    -14.6   -2.5    -4.4    -0.2    -0.9
12:32:47         -0.1    -14.3   -2.6    -4.3    -0.2    -0.8
12:33:02         -0.1     51.4   -2.3    -4.3    -0.2    -0.7
12:33:17          0.2    106.7   -2.9    -7.1    -0.2    -0.8
12:33:32          0.2    108.4   -3.0    -6.9    -0.3    -0.9
12:33:47          0.2     95.3   -2.8    -6.4    -0.3    -0.9
12:34:02         -0.0     32.6   -0.8    -3.6    -0.2     1.7
12:34:17         -0.1     -7.6   -1.9    -3.2    -0.2    -0.5
12:34:32         -0.1    -14.4   -2.6    -3.7    -0.2    -0.8
12:34:47         -0.1    -15.6   -2.8    -4.3    -0.2    -0.8
12:35:02         -0.1    -16.0   -2.9    -4.3    -0.2    -0.9
=============================================================================================================================
 Avg.=            7.4    307.9   65.8    22.1     4.5    63.3
=============================================================================================================================
```

```
                        CALIBRATION SUMMARY
                        09-23-1991    12:39:10
               CALIBRATION FILE NAME =D:\CEMDATA\0923CAL8.CAL

==========================================================================
Chan. Name Units |      Zero         |       Span        |   Slope      Int.
                 |  Conc.  Resp.     | Conc.   Resp.     |
==========================================================================
  1   O2    %      0.00    0.0003      18.00  0.073      246.067       -0.08
  2   THC   PPMV   0.00    -.0422      95.00  0.912       99.585        4.20
  3   SO2   PPMV   0.00    0.0014     295.00  0.058     5226.944       -7.24
  4   CO    PPMV   0.00    0.0006     474.00  0.047    10180.517       -5.76
  5   CO2   %      0.00    0.0008      17.00  0.082      209.815       -0.17
  6   NOX   PPMV   0.00    -.0048     201.00  2.197       91.284        0.43
  7                0.00    0.0000       0.00  0.000        1.000        0.00
  8                0.00    0.0000       0.00  0.000        1.000        0.00
  9                0.00    0.0000       0.00  0.000        1.000        0.00
 10                0.00    0.0000       0.00  0.000        1.000        0.00
 11                0.00    0.0000       0.00  0.000        1.000        0.00
 12                0.00    0.0000       0.00  0.000        1.000        0.00
 13                0.00    0.0000       0.00  0.000        1.000        0.00
 14                0.00    0.0000       0.00  0.000        1.000        0.00

==========================================================================
```

RE-CAL THC

RB

```
                 Press Shift-PrtSc to Print Out Table
                        Press (C) to Continue


                     CEM INSTRUMENT DRIFT SUMMARY
                        09-23-1991    12:39:17

==========================================================================
Chan. Name Units |     Zero Conc.     |       Span         | Drift % of Scale
                 |Actual   Observed|Actual    Observed|  Zero        Span
==========================================================================
  1   O2    %     0.000   0.000        18.00    0.00    0.00        -72.00
  2   THC   PPMV  0.000   -.295        95.00   91.56   -0.03         -0.34
  3   SO2   PPMV  0.000   0.000       295.00    0.00    0.00         -5.90
  4   CO    PPMV  0.000   0.000       474.00    0.00    0.00        -47.40
```

```
 6   NOX   PPMV   0.000   0.000      201.00   0.00   0.00        -20.10
 7                0.000   0.000        0.00   0.00   0.00          0.00
 8                0.000   0.000        0.00   0.00   0.00          0.00
 9                0.000   0.000        0.00   0.00   0.00          0.00
 10               0.000   0.000        0.00   0.00   0.00          0.00
 11               0.000   0.000        0.00   0.00   0.00          0.00
 12               0.000   0.000        0.00   0.00   0.00          0.00
 13               0.000   0.000        0.00   0.00   0.00          0.00
 14               0.000   0.000        0.00   0.00   0.00          0.00

===========================================================================
```

```
                    Press Shift-PrtSc to Print Out Table
                           Press (C) to Continue

RADIAN CORPORATION      0923RUNE

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-23-1991  Current Time = 12:40:11
File Name = D:\CEMDATA\092391.PRN   Calibration File:D:\CEMDATA\0923CAL8.CAL


 09-23-1991     O2      THC     SO2     CO     CO2     NOX
 Time           %       PPMV    PPMV    PPMV   %       PPMV
==========================================================================================================================
12:40:19         0.1    94.3    -3.4    -5.6   -0.3    -0.8
12:40:29         0.1    94.2    -3.5    -6.1   -0.3    -0.8
12:40:39        -0.0    73.3    -2.6    -4.4   -0.3     1.4
12:40:49         2.7    24.8     1.2     4.8    4.3     1.6
12:40:59        11.9     6.1    -0.6    67.6   23.1    -0.2
12:41:09        15.9     0.4    -2.7   216.7   29.8    -0.5
12:41:19        17.0    -1.9    -3.1   347.9   31.4    -0.7
12:41:29        17.6    -3.4    -3.4   427.1   32.0    -0.7
12:41:39        17.9    -4.5    -3.2   466.5   32.2    -0.8
12:41:49        18.2    -4.6    -3.5   484.7   32.2    -0.8
12:41:59        18.3    -4.3    -3.5   502.6   32.2    -0.8
12:42:09        18.3    -4.8    -3.5   507.4   30.6    -0.8
12:42:19        18.4    -4.9    -3.6   503.3   16.9    -0.8
12:42:29        18.4    -4.9    -3.2   504.8   17.3    -0.8
12:42:39        18.4    -4.9    -3.0   504.8   17.3    -0.8
12:42:49        18.4    -5.0    -3.4   505.4   17.3    -0.8
12:42:58        18.4    -5.1    -3.5   505.5   17.3    -0.8
12:43:08        18.4    -5.0    -3.6   496.5   17.3    -0.8
12:43:18        18.4    -5.0    -3.6   489.0   17.7    -0.8
12:43:28        18.4    -5.1    -3.6   486.5   17.7    -0.8
12:43:38        18.4    -5.2    -3.9   481.9   17.5    -0.9
12:43:48        18.4    -5.2    -3.6   480.5   17.3    -0.8
```

THC

QC

```
12:44:08     18.4    -5.3    -3.8    484.6    16.9    -0.8
12:44:18     18.4    -5.3    -3.9    483.8    16.9    -0.8
12:44:28     18.4    -5.4    -3.7    485.2    17.0    -0.8
12:44:38     18.4    -5.6    -3.8    483.8    17.0    -0.8
12:44:48     18.4    -5.5    -3.8    481.1    16.9    -0.8
12:44:58     18.4    -5.6    -3.7    480.7    16.9    -0.8
12:45:08     18.4    -5.6    -3.6    479.7    17.2    -0.9
12:45:18     18.4    -4.5    -3.5    479.3    17.2    -0.7
12:45:28     14.9    -0.2    -3.3    460.6    14.8    -0.7
12:45:38      9.4     0.6    -3.8    386.2    10.9    -0.8
12:45:47      8.5     0.8    -3.8    276.1    10.5    -0.8
12:45:57      8.1     1.0    -3.9    183.8    10.4    -0.8
12:46:07      7.9     1.0    -4.2    128.4    10.5    -0.8
12:46:17      7.7     1.0    -4.4    103.5    10.4    -0.8
12:46:27      7.7     1.0    -4.2     96.1    10.4    -0.8
12:46:37      7.7     0.9    -4.4     93.0    10.4    -0.8
12:46:47      7.7     1.1    -4.5     92.0    10.3    -0.8
12:46:57      7.7     1.1    -4.6     91.0    10.3    -0.8
12:47:07      7.7     1.1    -4.6     90.3    10.3    -0.8
12:47:17      7.7     3.3    -4.5     90.3    10.3    -0.7
12:47:27      6.7    24.7    -4.2     88.6     9.1    -0.6
12:47:37      2.3    33.2    -4.2     78.1     2.9    -0.8
12:47:47      0.7    36.1    -4.0     53.2     0.7    -0.8
```

SPAN
O2
CO2

QC
O2
CO2

09-23-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
12:47:57	0.2	37.0	-4.2	26.4	0.0	-0.8
12:48:07	0.0	37.3	-4.3	8.7	-0.2	-0.8
12:48:17	-0.0	37.5	-4.2	0.3	-0.3	-0.8
12:48:27	-0.1	37.6	-4.4	-2.3	-0.3	-0.8
12:48:36	-0.1	37.6	-4.5	-3.1	-0.3	-0.8
12:48:46	-0.1	37.5	-4.5	-3.1	-0.3	-0.8
12:48:56	-0.1	37.5	-4.4	-3.1	-0.4	-0.8
12:49:06	-0.1	37.5	-4.2	-3.1	-0.4	-0.8
12:49:16	-0.1	37.5	-4.2	-3.1	-0.4	-0.8
12:49:26	-0.1	17.3	24.6	-4.1	-0.4	69.6
12:49:36	-0.1	0.8	185.0	-7.3	-0.3	168.3
12:49:46	-0.2	-2.5	259.5	-6.8	-0.4	186.7
12:49:56	-0.2	-4.3	285.3	-7.0	-0.4	195.6
12:50:06	-0.3	-5.0	295.9	-7.8	-0.5	198.9
12:50:16	-0.2	-5.3	300.5	-7.9	-0.5	200.4
12:50:26	-0.3	-5.5	302.9	-8.0	-0.5	201.1
12:50:36	-0.3	-5.7	303.6	-8.9	-0.5	200.6
12:50:46	-0.3	-5.6	304.1	-8.0	-0.5	200.9
12:50:56	-0.3	-5.8	304.5	-9.0	-0.5	201.9
12:51:06	-0.2	1.6	270.7	-7.6	-0.4	121.1
12:51:16	8.8	107.3	118.6	-4.7	0.4	69.2
~~12:51:25~~	~~17.6~~	~~137.8~~	~~70.1~~	~~-2.0~~	~~5.5~~	~~52.6~~
12:51:35	15.2	148.9	44.7	7.1	4.3	23.8
12:52:35	20.6	158.2	6.3	1.6	0.2	5.1
12:53:35	21.3	131.6	-0.8	-4.1	-0.2	2.0
12:54:35	21.3	73.0	-2.6	-3.1	-0.2	0.7
12:55:35	21.4	140.4	-1.1	-5.5	-0.3	1.4
12:56:35	13.5	155.9	20.2	-5.0	-0.3	16.0
12:57:35	1.1	153.5	1.4	-6.2	-0.4	0.3
12:58:35	0.8	152.0	-0.9	-6.2	-0.4	-0.2
12:59:35	1.0	112.8	0.8	-5.1	-0.4	1.3
13:00:35	-0.1	29.7	-4.0	-4.1	-0.4	-0.9
13:01:35	-0.1	15.1	-4.3	-4.3	-0.4	-0.9
13:01:50	-0.1	10.1	-4.3	-4.3	-0.4	-0.9
13:02:00	-0.1	8.7	-4.3	-4.2	-0.4	-0.9
13:02:10	-0.1	7.8	-4.3	-4.3	-0.4	-0.9
13:02:20	0.0	7.1	-4.3	-4.3	-0.4	-0.9
13:02:30	-0.0	6.1	-4.3	-4.3	-0.4	-0.9
13:02:40	-0.1	5.3	-4.6	-4.3	-0.4	-0.9
13:02:50	-0.1	4.9	-4.4	-4.3	-0.4	-0.9
13:03:00	-0.0	4.1	-4.5	-4.4	-0.4	-0.9
13:03:10	-0.0	3.4	-4.2	-4.4	-0.4	-0.9
13:03:20	-0.0	4.8	-4.2	-4.4	-0.4	-0.9
13:03:30	3.5	7.6	-4.1	-4.3	-0.4	-0.6
13:03:40	17.2	7.0	-3.6	-4.1	-0.3	-0.6
13:03:50	20.2	6.8	-3.7	-3.3	-0.3	-0.6
13:04:00	20.9	6.5	-4.1	-4.2	-0.3	-0.6
13:04:10	21.1	6.5	-4.2	-3.2	-0.3	-0.6
13:04:20	21.3	5.3	-4.3	-3.3	-0.3	-0.7
13:04:29	21.3	144.7	-4.4	-5.9	-0.4	-0.6
13:04:39	21.3	44.6	-3.9	-3.1	-0.3	-0.7
13:04:49	21.3	32.8	-4.0	-3.1	-0.3	-0.7
13:04:59	21.4	26.6	-4.1	-3.1	-0.3	-0.7
13:05:59	21.4	15.6	-4.3	-3.3	-0.3	-0.8
13:06:59	21.5	8.5	-4.5	-3.3	-0.3	-0.8

THC

SO_2
NOx

← ON-LINE
Plant Down

```
09-23-1991      O2       THC      SO2      CO       CO2      NOX
Time            %        PPMV     PPMV     PPMV     %        PPMV
==========================================================================================================================================
13:08:59        21.4      4.4     -4.7     -3.3     -0.3     -0.7
13:09:59        21.[illegible]      3.2     -4.4     -3.6     -0.3     -0.8
13:10:59        21.4      2.3     -4.2     -3.3     -0.3     -0.8
13:11:59        21.4      1.4     -4.4     -3.3     -0.3     -0.8
13:12:59        21.4      0.6     -4.4     -3.7     -0.3     -0.8
13:13:59        15.9     -0.1     -1.3     71.8      7.0      1.1
13:14:59        18.2     -0.3     -5.0    430.6     18.3     -0.9
13:15:58        18.4     -0.5     -4.9    453.5     18.3     -1.0
13:16:58        18.4     -0.9     -5.1    456.3     18.4     -1.0
13:17:58        18.3     -1.1     -5.1    460.4     18.4     -1.0
13:18:58        12.3     -1.2     -5.1    353.3     14.0     -1.0
13:19:28         7.5     -1.1     -5.[illegible]    110.1     10.5     -1.0
13:19:43         7.5     -1.1     -5.4     88.9     10.4     -1.0
13:19:58         7.4     -1.1     -5.2     88.2     10.4     -1.0
13:20:13         7.6     -0.7     -5.2     87.6     10.5     -1.0
13:20:28         7.7     -0.6     -5.2     88.2     10.5     -1.0
13:20:43         7.7     -0.8     -5.2     89.0     10.5     -1.0
13:20:58         7.7     -0.7     -4.8     88.2     10.4     -1.0
13:21:13         9.3     -0.8     -4.8     95.9     11.6     -0.9
13:21:27        16.1     -0.9     -4.9    187.9     16.8     -1.0
13:21:42        17.8     -0.9     -5.0    345.5     17.3     -1.[illegible]
13:21:57        18.3     -1.0     -4.9    413.5     17.[illegible]     -1.[illegible]
13:22:12        18.4     -1.1     -5.1    44[illegible]     17.[illegible]     -1.[illegible]
13:22:27        18.4     -1.1     -5.0    44[illegible]     17.6     -1.[illegible]
13:22:42        18.0     -1.2     -5.2    44[illegible]     17.3     -1.0
13:22:57        18.0     -1.2     -5.[illegible]    447.3     17.7     -1.0
13:23:12        18.0     -1.1     -5.2    4[illegible]7.5     17.7     -1.[illegible]
13:23:27        18.0     -1.2     -5.[illegible]    454.2     17.[illegible]     -1.0
13:23:42        18.0     -1.3     -5.2    457.7     17.2     -1.[illegible]
13:23:57        18.[illegible]     -1.3     -4.8    455.7     17.2     -1.[illegible]
13:24:12        18.[illegible]     -1.3     -4.7    455.4     17.2     -1.0
[illegible]     [illegible]     -1.3     -4.8    500.7     17.0     -1.[illegible]
[illegible]     18.[illegible]     -1.3     -4.7    457.6     16.7     -1.0
[illegible]     18.[illegible]     -1.7     -5.[illegible]    460.1     16.8     -1.0
[illegible]     18.[illegible]     -1.0     -5.0    454.7     16.9     -1.[illegible]
============================================================================
============================================
[illegible]     [illegible]      0.0     -0.0      0.0      0.0      0.[illegible]
============================================================================
============================================
```

```
RADIAN CORPORATION       0923RUNF

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
[illegible]
[illegible]
Performed For:                EPA
Date Printed = 09-23-1991  Current Time = [illegible]
File Name = C:\CEMDATA\[illegible].PRN   Calibration File:C:\CEMDATA\0923CAL9.CAL

 09-23-1991         O2       THC      SO2      CO       CO2      NOX
 Time               %        PPMV     PPMV     PPMV     %        PPMV
==========================================================================================================
13:28:36           20.9     -2.7     -4.3     60.5     [illegible] [illegible]
13:29:36           20.8     -2.4     -4.3     [illegible] [illegible] [illegible]
13:30:36           20.9     [illegible] -4.5  [illegible] [illegible] [illegible]
13:31:36           [illegible] [illegible] [illegible] [illegible] [illegible] [illegible]
13:32:36           [illegible] [illegible] [illegible] [illegible] [illegible] [illegible]
13:33:36           [illegible] [illegible] [illegible] -3.1 [illegible] [illegible]
13:34:36           20.8     -2.3     -4.4     -3.1     -0.3     -0.7
13:35:35           20.8     -2.5     -4.5     -3.1     -0.4     -0.7
13:36:35           20.9     -3.1     -4.5     -3.2     -0.4     -0.8
13:37:35           20.8     -3.7     -4.4     -3.2     -0.4     -0.8
13:38:35           20.8     -3.6     -4.4     -3.4     -0.4     -0.3
13:39:35           20.8     -3.2     -4.1     -3.1     -0.4     -0.5
13:40:35           20.8     -2.9     -4.2     -3.5     -0.4     -0.6
13:41:35           20.7     -1.9     -4.1     -3.1     -0.4     -0.5
13:42:35           20.8     -1.2     -4.2     -3.1     -0.4     -0.4
13:43:35           20.8     -0.6     -4.2     -3.1     -0.4     -0.5
13:44:35           20.8     -2.1     -4.0     -3.1     -0.4     -0.7
13:45:35           20.8     -2.9     -3.9     -3.4     -0.4     -0.8
13:46:35           20.8     -3.2     -3.5     -3.3     -0.4     -0.7
13:47:35           20.8     -3.3     -3.6     -3.4     -0.4     -0.8
13:48:34           20.8     -3.4     -3.3     -3.3     -0.4     -0.5
13:49:34           10.6     -4.3     33.8      2.4      2.0     57.6
13:50:34            9.9    106.4    122.2     37.3      7.1    133.9
13:51:34           10.6    325.9    129.2     48.6      7.1    132.8
13:52:34           10.6    558.4    134.7     54.5      7.4    141.5
13:53:34           10.3    582.4    137.9     45.0      7.6    143.9
13:54:34           10.0    591.2    140.8     47.1      7.8    145.9
13:55:34            9.7    600.7    145.0     45.2      8.0    148.4
13:56:34            9.5    607.5    147.8     43.2      8.0    149.9
13:57:34            9.5    613.0    146.9     40.2      8.1    151.9
13:58:34            9.5    599.2    146.4     44.9      8.1    150.1
13:59:34            9.8    589.2    146.1     56.8      7.7    149.1
14:00:34            9.8    593.9    147.3     50.1      7.7    148.4
14:01:34           10.0    581.5    143.7     44.8      7.7    146.5
14:02:33           10.3    561.3    142.6     43.7      7.4    143.7
14:03:33           10.1    566.7    144.1     44.6      7.4    144.6
14:04:33           10.1    572.4    145.1     57.6      7.5    146.3
14:05:33           10.0    575.1    147.1     57.4      7.5    147.3
14:06:33            9.9    580.9    147.9     44.4      7.5    148.6
14:07:33            9.5    577.5    148.5     32.8      7.8    153.3
14:08:33            9.1    584.9    146.2     27.4      8.0    156.4
14:09:33            9.0    941.5 ①  150.6    399.8 ①   8.5    150.1
14:10:33            9.4    747.8    158.7    250.7      7.8    155.9
14:11:33            9.5    692.1    156.7     53.9      7.7    155.5
```

START
ON-LINE Test #3

START #2
RAW/METALS
RB

① SPIKES OF UNKNOWN ORIGIN.
REMOVE FROM AVGS.

RB

```
14:13:33          9.8   618.8   155.4    50.9     7.1   147.5
```

```
09-23-1991        O2     THC     SO2     CO      CO2     NOX
Time              %      PPMV    PPMV    PPMV    %       PPMV
==============================================================================================================================================
14:14:33          9.9   611.6   153.6    52.0     7.0   143.5
14:15:32         10.0   580.9   152.5    46.6     7.1   147.3
14:16:32          9.7   574.9   152.4    38.0     7.2   150.3
14:17:32          9.3   578.1   151.8    41.4     7.1   149.5
14:18:32         10.1   578.6   150.8    42.3     7.0   146.5
14:19:32         10.2   576.1   151.5    42.3     6.9   145.4
14:20:32          9.9   602.7   155.9    43.4     6.9   149.1
14:21:32          9.7   600.5   159.1    43.6     7.0   151.2
14:22:32          9.8   609.8   159.1    42.7     6.2   148.3
14:23:32          9.8   608.4   150.7    47.0     6.4   151.6
14:24:32          9.6   621.3   156.2    41.0     6.7   154.0
14:25:32          9.7   621.1   153.3    39.2     6.8   151.4
14:26:32         10.0   605.5   149.3    39.3     6.3   147.8
14:27:32         10.3   596.0   146.4    39.4     5.7   144.4
14:28:31         10.4   586.8   145.3    40.0     5.9   143.3
14:29:31         10.5   575.6   143.8    40.5     5.9   142.0
14:30:31         10.7   571.6   141.4    41.0     6.0   141.0
14:31:31         10.7   573.4   142.7    38.4     5.6   140.7
14:32:31         10.6   578.4   141.9    37.5     5.3   141.4
14:33:31         10.3   588.3   143.1    36.6     4.3   144.3
14:34:31         10.1   596.9   143.5    38.0     4.2   146.7
14:35:31          9.9   600.2   144.3    38.6     4.2   147.4
14:36:31          9.9   599.9   145.1    38.3     4.2   148.6
14:37:31          9.8   596.8   147.2    38.9     4.3   150.6
14:38:31         10.0   607.2   147.6    38.5     4.2   148.4
14:39:31         10.1   593.6   144.8    38.9     4.2   146.7
14:40:31         10.3   591.6   142.9    37.8     4.1   145.0
14:41:30         10.4   587.5   142.2    37.3     4.0   144.0
14:42:30         10.3   575.2   140.1    35.0     4.1   146.6
14:43:30         10.4   575.2   139.1    34.2     4.1   145.3
14:44:30          9.0   579.3    39.7    66.9     4.8    22.4
14:45:17          8.1   595.3    -2.0    91.1     5.1    -1.0
14:45:26          8.1   615.6    -2.2    89.6     5.1    -1.0
14:45:36          8.1   617.8    -2.2    90.5     5.1    -1.0
14:45:46          8.1   619.0    -2.3    89.8     5.1    -1.0
14:45:56          8.1   606.1    -2.3    89.4     6.1    -1.1
14:46:06          8.1   599.9    -2.2    89.3    10.4    -1.1
14:46:16          8.1   601.9    -2.1    88.7    10.4    -1.1
14:46:26          8.1   609.3    -2.3    89.2    10.4    -1.1
14:46:36          8.1   606.2    -2.1    90.1    10.4    -1.1
14:47:36          9.9   615.8    46.6    76.7     8.7    70.9
14:48:36         10.0   613.0   141.0    35.5     8.4   148.8
14:49:36          9.7   609.4   147.5    31.3     8.7   154.4
14:50:36          9.4   605.0   145.7    29.2     9.0   156.7
14:51:36          9.5   606.5   144.9    29.8     8.8   153.9
14:52:36          9.5   590.0   145.3    30.8     8.8   152.4
14:53:36          9.7   559.3   142.8    28.4     8.7   149.4
14:54:35          9.8   557.1   142.4    32.4     8.4   147.2
14:55:35         10.4   531.5   140.1    35.1     8.0   138.7
14:56:35         11.0   501.1   128.9    48.9     7.6   132.5
14:57:35         11.1   511.9   132.1    53.8     7.5   132.2
14:58:35         11.2   514.1   133.6    58.9     7.4   129.8
14:59:35         11.1   525.6   133.0    58.9     7.6   132.1
15:00:35         10.9   543.6   135.4    57.7     7.8   131.7
15:01:35         10.8   556.4   138.4    57.3     7.8   135.7
```

QC CHECKS

REMOVE FROM AVGS.

RB

09-23-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
15:03:35	10.5	554.8	138.4	56.2	8.2	139.3
15:04:35	10.7	541.3	135.8	55.6	7.9	137.0
15:05:35	10.8	541.1	135.3	58.7	7.9	136.0
15:06:35	10.9	533.3	133.6	58.6	7.8	133.6
15:07:34	11.1	531.3	130.8	57.2	7.6	131.8
15:08:34	11.2	540.3	128.5	55.3	7.4	126.0
15:09:34	8.9	538.7	14.2	70.1	9.4	0.8
15:10:34	11.3	543.1	103.5	63.8	7.3	124.3
15:11:34	11.3	541.2	125.1	53.1	7.3	127.1
15:12:34	11.0	549.5	129.5	42.7	7.8	135.0
15:13:34	10.7	544.3	133.1	34.1	8.0	138.3
15:14:34	10.4	546.2	133.9	31.4	8.1	140.1
15:15:34	10.2	555.2	135.5	29.4	8.3	145.3
15:16:34	9.7	569.5	142.7	29.9	8.7	152.8
15:17:34	9.8	566.6	142.2	31.9	8.5	149.7
15:18:34	10.0	569.8	140.1	35.7	8.3	146.4
15:19:34	10.2	552.0	143.0	37.5	8.4	144.4
15:20:33	10.3	562.1	142.1	37.3	8.3	143.7
15:21:33	10.3	546.4	141.7	37.6	8.1	141.9
15:22:33	10.5	555.4	140.3	39.1	8.2	140.8
15:23:33	10.5	555.1	139.2	38.3	8.1	141.0
15:24:33	10.5	570.5	139.6	39.1	8.1	140.5
15:25:33	10.4	566.5	141.8	38.2	8.3	143.0
15:26:33	10.0	579.6	145.0	38.1	8.5	148.4
15:27:33	9.9	591.7	146.4	40.7	8.5	149.9
15:28:33	9.8	580.8	147.1	41.0	8.6	151.5
15:29:33	9.7	605.0	148.9	40.1	8.8	153.9
15:30:33	9.7	618.5	149.9	40.2	8.8	151.1
15:31:33	9.7	610.6	148.9	38.4	8.7	150.2
15:32:33	9.8	608.0	147.5	38.8	8.8	149.5
15:33:32	10.0	592.0	142.1	36.3	8.6	145.7
15:34:32	10.2	588.9	136.3	34.2	8.4	143.6
15:35:32	10.2	581.4	136.7	35.9	8.3	142.9
15:36:32	10.3	580.8	135.6	35.9	8.3	142.5
15:37:32	10.4	568.4	137.2	37.1	8.2	141.7
15:38:32	10.3	565.6	139.5	37.8	8.2	142.2
15:39:32	10.1	566.0	142.7	39.3	8.5	146.3
15:40:32	9.9	587.3	146.1	40.7	8.7	148.1
15:41:32	9.7	600.8	149.1	40.4	8.7	149.2
15:42:32	9.7	606.8	149.7	40.6	8.8	150.9
15:43:32	9.7	608.2	152.0	43.4	8.8	151.6
15:44:32	9.7	611.7	151.9	45.1	8.8	150.7
15:45:32	10.1	592.2	148.1	45.8	8.4	145.2
15:46:31	11.3	578.4	34.4	60.9	7.6	122.1
15:47:31	13.9	538.2	93.8	218.4	5.1	81.8
15:48:31	13.4	532.1	85.7	134.0	5.6	97.1
15:49:31	12.1	519.2	105.7	82.2	6.9	117.8
15:50:31	10.8	545.9	131.9	42.4	8.0	138.2
15:51:31	10.5	568.4	136.0	34.0	8.2	141.5
15:52:31	10.1	577.2	137.2	33.4	8.5	147.2
15:53:31	9.8	590.9	140.6	35.5	8.7	150.2
15:54:31	9.5	618.4	148.8	39.0	8.9	152.8
15:55:31	9.3	617.8	152.9	39.0	9.1	155.0
15:56:31	9.2	638.4	154.2	40.2	9.2	155.4
15:57:31	9.3	628.7	153.8	40.5	9.0	154.5

Fyr.ke @ 1500 hrs
O_2% ~ 11
CO_2% ~ 8

QC CHECKS
REMOVE FROM AVGS. RB

BLOW BACK RB

ACD # 2

```
09-23-1991      O2      THC     SO2     CO      CO2     NOX
Time            %       PPMV    PPMV    PPMV    %       PPMV
==========================================================================================================
15:59:30        9.6    607.4   148.4    38.5     8.9   150.9
16:00:30        9.7    594.7   145.9    39.2     8.6   148.7
16:01:30        9.8    598.6   148.0    39.6     8.6   149.2
16:02:30        9.9    597.1   147.9    40.3     8.6   147.7
16:03:30       10.4    586.7   143.1    37.3     8.0   137.2
16:04:30        9.8    593.3   153.3    39.4     8.7   151.4
16:05:30        9.8    602.5   154.7    39.3     8.7   148.8
16:06:30       10.5    593.6   140.7    38.2     7.7   125.9
16:07:30       10.4    563.5   144.6    32.3     8.2   147.1
16:08:30        9.7    584.4   152.3    37.6     8.8   150.5
16:09:30        9.6    608.9   152.9    38.3     8.9   150.8
16:10:30        9.7    600.3   151.6    38.0     8.6   147.3
16:11:30        9.9    615.8   150.8    38.7     8.6   145.9
16:12:29       10.2    607.0   148.8    39.3     8.4   143.7
16:13:29       10.3    611.4   147.0    39.7     8.2   141.9
16:14:29       10.3    612.6   146.5    39.6     8.2   141.5
16:15:29       10.4    593.1   143.2    38.9     8.2   140.5
16:16:29       10.4    609.4   141.9    39.2     8.1   140.5
16:17:29       10.3    603.6   142.9    38.7     8.1   141.5
16:18:29       10.1    608.4   145.4    39.2     8.5   144.8
16:19:29        9.9    605.6   147.2    40.7     8.6   147.1
16:20:29        9.8    620.4   148.8    42.5     8.5   147.4
16:21:29        9.7    627.4   150.0    44.0     8.6   149.6
16:22:29        9.7    632.8   151.7    44.6     8.9   150.6
16:23:29        9.8    621.9   149.9    42.9     8.6   150.4
16:24:29        9.9    619.3   149.0    41.7     8.5   148.4
16:25:29       10.3    614.7   142.5    47.4     8.2   142.1
16:26:28       10.7    596.3   139.4    48.2     7.9   137.3
16:27:28       10.7    587.1   140.2    47.4     7.9   138.0
16:28:28       10.8    574.5   137.6    47.2     7.9   137.2
16:29:28       10.8    574.2   137.3    48.6     7.9   136.4
16:30:28       10.8    580.3   138.3    49.8     7.8   136.0
16:31:28       10.7    576.3   138.5    50.2     8.1   139.6
16:32:28       10.5    582.5   139.9    52.5     8.2   141.6
16:33:28       10.2    596.2   143.0    50.9     8.3   143.2
16:34:28       10.2    615.7   144.6    53.4     8.3   144.5
16:35:28       10.0    620.7   145.8    51.6     8.5   146.0
16:36:28       10.2    612.7   143.7    44.7     8.2   143.3
16:37:28       10.2    606.4   142.5    42.1     8.2   142.8
16:38:28       10.4    608.2   140.0    42.1     8.1   140.2
16:39:27       10.6    582.5   138.8    44.2     8.0   138.2
16:40:27       10.6    590.8   136.6    42.7     7.9   138.1
16:41:27       10.6    586.2   135.9    40.5     8.0   141.9
16:42:27       16.8    385.2    67.8    23.7     2.4    38.0
16:43:27       20.9    238.8     9.8     6.1    -0.5     1.7
==========================================================================================================
Avg.=          11.3    518.9   118.0    44.3     6.8   119.2
==========================================================================================================
```

RB

END PAH/METALS

BLOW BACK
REMOVE FROM AVGS. RB

END AD #2

RADIAN CORPORATION 0923OCCX

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for: EPA
Date Printed = 09-23-1991 Current Time = 16:46:28
File Name = D:\CEMDATA\092391.PRN Calibration File:D:\CEMDATA\0923CAL9.CAL

09-23-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
16:46:32	0.1	34.1	97.4	-5.8	-0.8	98.3
16:47:07	-0.1	26.4	100.2	-6.1	-0.8	98.5
16:47:17	-0.1	23.9	100.3	-5.6	-0.8	98.5
16:47:27	-0.1	23.2	100.3	-6.3	-0.8	98.6
16:47:37	-0.1	22.3	100.9	-6.3	-0.8	98.7
16:47:47	-0.1	21.3	100.7	-6.3	-0.8	98.9
16:47:57	-0.1	20.1	100.7	-6.3	-0.8	98.8
16:48:07	-0.1	19.1	100.8	-6.3	-0.8	98.9
16:48:16	-0.1	18.3	100.8	-6.3	-0.8	98.9
16:48:26	-0.1	17.4	100.8	-6.3	-0.8	98.8
16:48:36	-0.1	16.5	100.8	-6.3	-0.8	98.8
16:48:46	-0.1	15.8	100.6	-6.3	-0.8	98.9
16:48:56	-0.1	15.2	100.6	-6.3	-0.8	98.9
16:49:06	-0.1	14.7	100.8	-6.3	-0.8	98.9
16:49:16	-0.1	14.0	101.1	-6.2	-0.8	98.9
16:49:26	-0.2	13.4	100.9	-6.1	-0.8	98.1
16:49:36	0.0	12.5	94.6	-3.6	-0.3	64.1
16:49:46	2.7	12.0	62.1	4.2	4.4	21.7
16:49:56	5.6	11.4	31.4	24.9	8.1	6.2
16:50:06	6.8	10.8	17.1	53.1	9.3	1.9
16:50:36	7.2	9.7	7.0	85.5	9.8	-0.3
16:51:06	7.4	8.8	3.0	92.9	9.9	-0.8
16:51:36	7.4	8.3	1.4	93.6	9.8	-1.0
16:52:06	7.5	7.6	0.6	93.6	9.9	-1.1
16:52:36	7.5	8.0	-0.0	94.0	9.9	-1.2
16:53:05	7.5	7.7	-0.3	93.8	9.8	-1.2
16:53:35	7.5	7.2	-0.2	93.3	9.8	-1.2
16:54:05	7.5	6.7	-0.6	93.9	9.8	-1.2
16:54:35	5.2	5.9	-0.7	85.3	6.1	23.7
16:55:05	0.4	5.4	-0.7	24.5	-0.1	42.8
16:55:24	0.0	4.9	-0.6	0.9	-0.6	43.9
16:55:34	0.1	4.4	-0.5	-1.3	-0.6	43.9
16:55:44	-0.0	4.4	-0.7	-2.1	-0.6	44.0
16:55:54	0.0	4.6	-0.6	-2.1	-0.7	44.0
16:56:04	-0.1	4.7	-0.8	-2.1	-0.7	44.1
16:56:14	-0.1	5.2	-0.8	-2.1	-0.7	44.1
16:56:24	-0.1	4.7	-0.8	-2.0	-0.7	44.1
16:56:34	-0.0	4.4	-0.7	-2.1	-0.7	44.2
16:56:44	-0.0	4.3	-0.9	-2.1	-0.7	44.2
16:56:53	0.0	4.0	-1.0	-2.1	-0.7	40.2
16:57:03	0.2	3.9	-0.7	-1.7	-0.4	16.1
16:57:13	0.2	3.8	-0.9	-1.1	-0.1	2.7
16:57:23	0.1	3.7	-1.0	-1.1	-0.2	-0.2
16:57:33	-0.1	3.8	-0.8	-1.6	-0.3	-0.6

98

92

8.0

92.

10.2

16:57:53 0.0 3.5 -0.7 -2.4 -0.3 -0.7

09-23-1991	O2	THC	SO2	CO	CO2	NOX
Time	%	PPMV	PPMV	PPMV	%	PPMV
16:58:03	-0.1	3.0	-1.0	-3.1	-0.3	-0.9
16:58:13	-0.1	3.1	-1.0	-3.1	-0.3	-1.0
16:58:23	-0.1	3.2	-0.9	-3.0	-0.3	-1.1
16:58:33	0.4	174.5	-1.2	-5.1	-0.3	-1.1
16:58:43	0.6	223.4	-1.3	-5.7	-0.4	-1.1
16:58:53	0.6	225.7	-1.3	-5.7	-0.4	-1.1
16:59:03	0.6	226.7	-1.4	-5.6	-0.4	-1.1
16:59:13	0.6	227.4	-1.3	-5.6	-0.4	-1.2
16:59:23	0.6	227.5	-1.4	-5.6	-0.4	-1.2
16:59:33	0.5	227.5	-1.2	-5.6	-0.4	-1.2
16:59:42	0.5	227.4	-1.1	-5.6	-0.4	-1.2
16:59:52	0.6	227.6	-1.6	-5.6	-0.4	-1.2
17:00:02	0.6	227.1	-1.3	-5.6	-0.4	-1.2
17:00:12	0.6	227.0	-1.3	-5.6	-0.4	-1.3
17:00:22	0.6	226.6	-1.4	-5.6	-0.4	-1.3
17:00:32	0.6	226.4	-1.1	-5.6	-0.4	-1.3
17:00:42	0.2	130.7	-1.3	-5.1	-0.4	-1.3
17:00:52	-0.1	35.4	-1.2	-3.0	-0.3	-1.3
17:01:02	-0.1	27.5	-1.0	-3.0	-0.3	-1.3
17:01:12	0.2	99.3	-1.0	-4.7	-0.3	-1.3
17:01:22	0.2	126.8	-1.3	-5.9	-0.4	-1.3
17:01:32	0.2	128.4	-1.6	-5.9	-0.4	-1.3
17:01:42	0.2	112.6	-1.5	-6.0	-0.4	-1.3
17:01:52	0.2	102.5	-1.6	-6.1	-0.4	-1.3
17:02:02	0.2	102.6	-1.6	-6.1	-0.4	-1.3
17:02:12	0.2	102.6	-1.3	-6.1	-0.4	-1.3
17:02:22	0.1	102.4	-1.5	-6.1	-0.4	-1.3
17:02:32	0.2	102.5	-1.6	-6.1	-0.4	-1.3
17:02:41	0.2	102.7	-1.6	-6.1	-0.4	-1.4
17:02:51	0.2	102.5	-1.6	-6.1	-0.4	-1.4
17:03:01	0.2	100.4	-1.7	-6.1	-0.4	-1.4
17:03:11	-0.1	73.9	-1.1	-3.5	-0.3	-1.1
17:03:21	-0.0	61.8	-1.0	-2.9	-0.2	-0.9
17:03:31	-0.0	59.1	-1.0	-2.4	-0.2	-1.2
17:03:41	-0.1	58.3	-1.3	-1.9	-0.3	-1.3
17:03:51	-0.1	57.9	-1.3	-1.9	-0.3	-1.3
17:04:01	-0.1	57.5	-1.5	-2.5	-0.3	-1.3
17:04:11	-0.1	57.5	-1.5	-2.9	-0.3	-1.3
17:04:21	-0.1	54.5	-1.5	-2.9	-0.3	-1.3
17:04:31	-0.1	52.3	-1.6	-2.9	-0.3	-1.3
17:04:41	-0.1	52.1	-1.4	-2.9	-0.3	-1.3
17:04:51	-0.1	52.1	-1.2	-2.9	-0.3	-1.4
17:05:01	-0.1	52.3	-1.5	-2.9	-0.3	-1.4
17:05:11	-0.1	52.3	-1.3	-2.9	-0.3	-1.4
17:05:21	-0.1	52.2	-1.4	-2.9	-0.3	-1.4
17:05:30	-0.1	52.2	-1.4	-2.9	-0.3	-1.4
17:05:40	-0.1	51.7	-1.2	-2.9	-0.3	-1.4
17:05:50	-0.1	31.6	-1.3	-3.0	-0.3	-1.3
17:06:00	-0.1	22.4	-0.9	-3.0	-0.3	-1.2
17:06:10	-0.1	21.3	-1.1	-3.0	-0.3	-1.3
17:06:20	-0.1	21.1	-1.2	-3.0	-0.3	-1.4
17:06:30	-0.1	20.9	-1.1	-3.0	-0.3	-1.4
17:06:40	-0.1	20.8	-1.1	-3.0	-0.3	-1.4
17:06:50	-0.1	20.6	-0.8	-3.0	-0.3	-1.4
17:07:00	-0.1	20.5	-1.4	-3.0	-0.3	-1.4

45

09-23-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
17:07:20	-0.1	20.3	-1.1	-3.0	-0.3	-1.4
17:07:30	-0.1	20.2	-1.1	-3.0	-0.3	-1.4
17:07:40	-0.1	20.2	-1.1	-3.0	-0.3	-1.4
17:07:50	-0.1	20.1	-0.8	-3.0	-0.3	-1.4
17:08:00	-0.1	20.1	-1.0	-3.0	-0.3	-1.4
17:08:10	-0.1	20.1	-1.0	-3.0	-0.3	-1.4
17:08:19	-0.1	20.0	-1.0	-3.0	-0.3	-1.4
17:08:29	-0.0	8.6	-0.9	-3.0	-0.3	-1.4
17:08:39	0.0	6.0	-0.9	-3.1	-0.3	-1.4
17:08:49	0.0	6.0	-0.9	-3.0	-0.3	-1.4
17:08:59	-0.0	6.1	-0.9	-3.0	-0.3	-1.4
17:09:09	-0.0	6.2	-0.8	-3.0	-0.3	-1.4
17:09:19	0.0	6.2	-0.8	-3.0	-0.3	-1.4
17:09:29	-0.0	6.1	-0.8	-3.0	-0.3	-1.4
17:09:39	-0.0	6.2	-1.0	-3.0	-0.3	-1.4
17:09:49	-0.0	6.2	-1.1	-3.0	-0.3	-1.4
17:09:59	0.0	6.1	-1.1	-3.0	-0.3	-1.4
17:10:09	-0.0	5.9	-1.1	-3.0	-0.3	-1.4
17:10:19	0.0	4.7	-0.9	-3.1	-0.3	-1.4
17:10:29	0.8	6.3	-1.0	-3.0	-0.3	-1.4
17:10:39	10.7	6.6	-1.4	-2.9	-0.3	-1.3
17:10:49	17.1	6.4	-1.2	-2.9	-0.3	-1.3
17:10:59	19.2	6.2	-1.8	-2.9	-0.3	-1.3
17:11:08	20.2	6.1	-1.9	-2.9	-0.3	-1.4
17:11:18	20.5	6.0	-2.3	-2.9	-0.3	-1.4
17:11:28	20.7	6.2	-2.6	-3.0	-0.3	-1.4
17:11:38	20.7	5.6	-2.8	-2.9	-0.3	-1.4
17:11:48	20.7	3.5	-2.7	-3.0	-0.3	-1.4
17:11:58	20.6	2.2	-3.0	-3.0	-0.3	-1.4
17:12:08	20.7	1.5	-2.7	-3.0	-0.3	-1.4
17:12:18	20.7	0.9	-2.9	-3.0	-0.3	-1.4
17:12:28	20.7	0.4	-3.2	-3.9	-0.2	-1.4
17:12:38	20.7	-0.0	-3.0	-3.2	-0.3	-1.4
17:12:48	20.8	-0.3	-3.1	-3.0	-0.3	-1.4
17:12:58	20.7	-0.7	-3.0	-3.0	-0.3	-1.4
17:13:08	20.8	-0.9	-3.3	-3.0	-0.3	-1.4
17:13:18	20.8	-1.2	-3.4	-3.0	-0.3	-1.4
Avg.=	3.0	45.0	12.0	3.3	0.4	14.8

```
RADIAN CORPORATION      0924QCAL1

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-24-1991  Current Time = 06:49:37
File Name = D:\CEMDATA\092491.PRN   Calibration File:D:\CEMDATA\0923CAL9.CAL


 09-24-1991      O2       THC      SO2      CO       CO2      NOX
 Time            %        PPMV     PPMV     PPMV     %        PPMV
==========================================================================================================================================
06:49:50         0.0    -18.9     -5.6     -1.9     -0.2     -1.4
06:50:50        -0.0    -18.7     -5.8     -2.1     -0.2     -1.4
06:51:50         0.4    -18.5     -6.2     -2.1     -0.3     -1.4
06:52:50         1.1    -18.3     -6.0     -2.3     -0.2     -1.4
06:53:50         0.9    -18.0     -5.9     -2.2     -0.2     -1.4
06:54:49         1.7    -17.6     -6.2     -2.1     -0.2     -1.4
06:55:49         4.0    -17.3     -6.2     -2.9     -0.2     -1.5
06:56:49         0.6    -17.0     -6.1     -3.1     -0.3     -1.5
06:57:49         1.7    -14.6     -5.7     -2.9     -0.2     -1.5
06:58:49         2.3    -14.8     -5.7     -3.3     -0.3     -1.5
06:59:49         2.5    -15.8     -5.7     -1.9     -0.3     -1.5
07:00:49        -0.1    -15.7     -5.7     -3.1     -0.3     -1.6
07:01:49        -0.1    -15.5     -5.7     -3.1     -0.3     -1.6
07:02:49        -0.1    -14.7     -5.9     -3.1     -0.3     -1.6


                               CALIBRATION SUMMARY
                               09-24-1991   07:21:23
                   CALIBRATION FILE NAME =D:\CEMDATA\0924CAL1.CAL

   =============================================================================
   Chan. Name Units |      Zero         |      Span       |   Slope      Int.
                    | Conc.  Resp.      | Conc.  Resp.    |
   =============================================================================
     1    O2   %      0.00    -.0004     18.00  0.074      242.486        0.09
     2    THC  PPMV   0.00    -.0023     95.00  0.948      100.005        0.23
     3    SO2  PPMV   0.00    0.0002    295.00  0.059     4990.611       -1.20
     4    CO   PPMV   0.00    0.0003    474.00  0.047    10059.478       -2.58
     5    CO2  %      0.00    -.0005     17.00  0.082      206.908        0.10
     6    NOX  PPMV   0.00    -.0219    201.00  2.205       90.279        1.98
     7                0.00    0.0000      0.00  0.000        1.000        0.00
     8                0.00    0.0000      0.00  0.000        1.000        0.00
     9                0.00    0.0000      0.00  0.000        1.000        0.00
    10                0.00    0.0000      0.00  0.000        1.000        0.00
    11                0.00    0.0000      0.00  0.000        1.000        0.00
    12                0.00    0.0000      0.00  0.000        1.000        0.00
    13                0.00    0.0000      0.00  0.000        1.000        0.00
    14                0.00    0.0000      0.00  0.000        1.000        0.00

   =============================================================================
```

```
                  Press Shift-PrtSc to Print Out Table
                        Press <C> to Continue



                     CEM INSTRUMENT DRIFT SUMMARY
                        09-24-1991   07:21:31

==============================================================================
Chan. Name Units |     Zero Conc.    |      Span        |  Drift % of Scale
                 |Actual   Observed|Actual   Observed|  Zero       Span
==============================================================================
  1   O2    %     0.000   -.164       18.00    18.08   -0.66          0.33
  2   THC   PPMV  0.000   3.979       95.00    98.58    0.40          0.36
  3   SO2   PPMV  0.000   %-5.975    295.00   303.00   -0.12          0.16
  4   CO    PPMV  0.000   %-3.112    474.00   471.46   -0.31         -0.25
  5   CO2   %     0.000   -.273       17.00    16.99   -1.36         -0.04
  6   NOX   PPMV  0.000   %-1.566    201.00   201.67   -0.16          0.07
  7               0.000   0.000        0.00     0.00    0.00          0.00
  8               0.000   0.000        0.00     0.00    0.00          0.00
  9               0.000   0.000        0.00     0.00    0.00          0.00
  10              0.000   0.000        0.00     0.00    0.00          0.00
  11              0.000   0.000        0.00     0.00    0.00          0.00
  12              0.000   0.000        0.00     0.00    0.00          0.00
  13              0.000   0.000        0.00     0.00    0.00          0.00
  14              0.000   0.000        0.00     0.00    0.00          0.00

==============================================================================




                  Press Shift-PrtSc to Print Out Table
                        Press <C> to Continue

RADIAN CORPORATION      0924QCCk

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:                EPA
Date Printed = 09-24-1991  Current Time = 07:24:23
File Name = D:\CEMDATA\092491.PRN   Calibration File:D:\CEMDATA\0924CAL1.CAL
```

```
09-24-1991     O2     THC     SO2     CO      CO2     NOX
Time           %      PPMV    PPMV    PPMV    %       PPMV
==========================================================================================
07:24:28       2.1    42.7    4.6     14.1    0.1     2.6
07:24:43       6.8    16.8    5.0     37.3    7.0     2.6
07:24:58       8.2    14.7    5.3     66.9    8.8     2.7
07:25:13       8.6    14.4    5.3     90.5    9.2     2.7
07:25:28       8.8    14.1    5.4     96.4    9.2     2.7
07:25:43       8.8    14.1    5.2     98.1    9.2     2.8
07:25:58       8.9    14.3    5.4     99.3    9.2     2.8
07:26:13       8.9    14.1    5.4     98.3    9.3     2.8
07:26:28       8.9    14.1    5.6     99.8    9.8     2.8
07:26:43       8.9    13.9    5.7     99.3    9.8     2.9
07:26:58       7.9     7.9   16.0     97.9    8.9    37.8
07:27:13       1.9     6.4   69.3     75.8    2.2    87.6
07:27:28       0.4     5.8   89.8     33.1    0.4    97.0
07:27:43       0.1     5.6   94.7      6.1   -0.0    99.0
07:27:58       0.1     5.5   95.8      0.2   -0.1    99.6
07:28:13       0.0     5.5   96.2     -3.2   -0.1    99.7
07:28:27       0.1     9.5   95.6     -2.1   -0.1    92.9
07:28:42       2.7     6.6   61.0      4.7    0.6    54.3
07:28:57       0.3     5.5   10.2     12.3   -0.0    46.8
07:29:12       0.1     5.3    2.3      6.5   -0.1    46.3
07:29:27       0.1     5.2    1.0      1.8   -0.1    46.1
07:29:42       0.1     5.2    0.6      0.1   -0.1    45.9
07:29:57       0.3    11.7    0.7      0.8   -0.1    34.5
07:30:12      14.1    22.5    1.7      0.6   -0.1     3.5
07:30:27      19.7    52.0    1.0      1.2   -0.1     0.9
==========================================================================================
 Avg.=         5.1    13.3   27.6     41.4    3.7    36.8
==========================================================================================
```

```
RADIAN CORPORATION      0924RUN1

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:               EPA
Date Printed = 09-24-1991  Current Time = 07:30:54
File Name = D:\CEMDATA\092491.PRN   Calibration File:D:\CEMDATA\0924CAL1.CAL


 09-24-1991      O2       THC     SO2      CO      CO2      NOX
 Time            %        PPMV    PPMV     PPMV    %        PPMV
============================================================================================================================
07:31:00        20.6     44.9     0.3      0.8     -0.1      1.4
07:31:15        20.6     43.9     0.3      0.3     -0.1      1.3
07:31:30        20.6     41.9     0.3      0.2     -0.1      0.6
07:31:45        20.6     42.0     0.3     -0.4     -0.1      0.1
07:32:00        20.6     40.4     0.0      0.2     -0.1      0.2
07:32:15        20.6     40.4    -0.1      0.3     -0.1      0.2
07:32:30        20.7     39.5     0.0     -0.5     -0.1      0.1
07:32:45        20.6     41.1    -0.2     -0.4     -0.1      0.1
07:33:00        20.6     40.5     0.1      0.9     -0.1      0.0
07:33:14        20.6     40.8     0.1      0.3     -0.1      0.0
07:33:29        20.6     40.5     0.1      0.3     -0.1      0.0
07:33:44        20.6     37.9    -0.2      0.3     -0.1      0.0
07:33:59        20.6     37.0    -0.2      0.5     -0.1      0.0
07:34:14        20.6     38.9    -0.2      0.6     -0.1      0.0
07:34:29        20.6     38.5    -0.1     -0.1     -0.1      0.0
07:34:44        20.6     37.4    -0.2      1.6     -0.1      0.0
07:34:59        20.6     36.8    -0.2     -0.4     -0.1      0.1
07:35:14        20.6     63.9    -0.2      0.3     -0.1      0.1
07:35:29        20.6     59.3    -0.2     -0.5     -0.1      0.0
07:35:44        20.7     55.6    -0.0      0.3     -0.1      0.0
07:35:59        20.7     51.4    -0.2      0.3     -0.1      0.0
07:36:14        20.7     48.9    -0.1      0.3     -0.1      0.0
07:36:29        20.7     25.3     1.7      0.2     -0.1      4.8
07:36:44        13.2     14.4    20.5     13.3      1.0     13.8
07:36:59         5.8     12.7     9.2     42.6      0.2      4.7
07:37:14         1.1     11.8     3.1     28.1     -0.1      1.1
07:37:28         0.7     11.3     2.1      7.2     -0.1      0.6
07:37:43         0.6     10.6     1.2      2.8     -0.1      0.4
07:37:58         0.5     10.3     1.0      2.0     -0.1      0.4
07:38:13         0.4     10.0     0.9      1.5     -0.1      0.3
07:38:28         0.3      9.7     0.8      1.4     -0.1      0.3
07:38:43         0.3      9.4     0.7      1.5     -0.1      0.2
07:38:58         0.2      9.0     0.8      1.8     -0.1      0.2
07:39:13         0.2      8.6     0.8      1.1     -0.1      0.2
07:40:13         0.2      8.1     0.9      1.4     -0.1      0.2
07:41:13         0.2      8.2     0.8      0.8     -0.1      0.1
07:42:13        10.0     39.5    21.3     39.8      0.8     13.6
07:43:13        18.3     40.1    39.5    117.1      1.5     21.4
07:44:13        19.8     52.4    18.2     73.1      0.5      6.7
07:45:13        19.2     50.7    30.0     82.8      1.2     17.1
07:46:13        18.7     43.7    41.2    114.1      1.5     21.8
07:47:12        19.8     38.7    22.6     76.6      0.6      8.7
07:48:12        20.6     22.8     4.7     10.7     -0.0      1.3
07:49:12        20.7     18.0     2.7      3.8     -0.1      0.8
```

07:51:12	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.4

09-24-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
07:52:12	20.7	13.5	1.4	2.1	-0.1	0.4
07:53:12	20.7	12.9	1.1	1.6	-0.1	0.3
07:54:12	20.7	11.9	1.1	1.2	-0.1	0.3
07:55:12	20.7	11.7	1.1	1.6	-0.1	0.3
07:56:12	20.7	11.1	1.2	1.3	-0.1	0.2
07:57:12	20.7	10.5	1.0	1.2	-0.1	0.2
07:58:12	20.7	10.0	0.9	1.2	-0.1	0.1
07:59:12	20.5	50.4	3.7	6.4	0.1	2.2
08:00:11	19.4	67.7	24.5	163.6	1.0	11.5
08:01:11	19.3	54.4	28.6	165.1	1.1	13.7
08:02:11	19.2	48.2	31.3	141.3	1.2	15.6
08:03:11	19.3	54.3	30.0	141.3	1.1	14.4
08:04:11	19.0	48.7	34.7	146.8	1.3	18.3
08:05:11	19.8	40.1	22.1	79.7	0.7	7.4
08:06:11	19.6	62.9	21.9	88.7	0.9	11.9
08:07:11	19.3	56.5	26.3	142.6	1.1	14.7
08:08:11	19.2	48.3	16.8	123.2	1.2	14.8
08:09:11	17.4	52.1	19.1	127.4	2.6	39.8
08:10:11	12.3	38.9	65.3	131.3	6.7	103.1
08:11:11	11.5	42.8	107.3	96.5	7.5	113.9
08:12:11	10.7	89.2	126.8	86.2	8.2	128.5
08:13:10	10.7	264.7	135.7	61.7	8.5	130.9
08:14:10	10.9	375.6	134.9	66.2	8.4	125.5
08:15:10	11.1	425.3	134.4	76.4	8.3	122.5
08:16:10	11.3	435.9	132.3	102.6	8.3	121.2
08:17:10	11.3	460.5	129.7	109.3	8.2	118.9
08:18:10	11.6	464.1	123.1	125.9	7.9	112.6
08:19:10	11.4	467.8	122.9	117.6	8.2	116.8
08:20:10	10.8	487.0	130.7	92.2	8.6	124.3
08:21:10	10.9	498.7	129.8	100.0	8.5	122.0
08:22:10	10.9	504.6	131.9	95.1	8.5	122.1
08:23:10	11.3	502.9	127.7	107.7	8.2	119.5
08:24:10	11.4	495.1	126.7	114.0	8.2	117.6
08:25:10	11.6	495.7	124.3	130.1	7.9	114.3
08:26:09	11.7	488.4	123.8	125.4	7.9	113.7
08:27:09	11.9	481.6	120.2	133.5	7.7	112.2
08:28:09	9.9	328.8	95.1	116.8	7.9	67.1
08:29:09	7.8	123.1	16.0	90.3	10.8	6.9
08:30:09	7.7	124.8	6.1	93.3	10.3	11.5
08:31:09	11.2	350.2	98.3	102.9	7.5	111.0
08:32:09	12.4	447.4	114.0	190.1	6.6	99.0
08:33:09	11.5	466.8	119.7	212.6	7.5	121.7
08:34:09	11.6	522.5	128.2	129.5	7.3	111.3
08:35:09	11.9	493.4	118.7	301.9	7.0	112.8
08:36:09	11.4	531.5	128.6	123.4	7.6	116.2
08:37:09	12.2	512.8	117.3	312.9	6.8	107.0
08:38:09	10.9	527.5	130.2	128.0	7.9	124.1
08:39:08	12.5	521.6	116.5	259.0	6.5	98.6
08:40:08	11.6	504.1	120.0	238.3	7.4	119.7
08:41:08	12.0	522.3	124.2	157.5	7.0	105.6
08:42:08	12.7	484.6	110.1	352.5	6.4	101.4
08:43:08	11.9	502.0	122.7	145.0	7.1	108.9
08:44:08	13.5	482.9	104.0	344.0	5.6	85.7
08:45:08	12.3	462.8	111.5	245.8	6.7	107.6
08:46:08	12.0	487.2	123.8	131.1	7.1	111.5

START ALDEHYDE RUN #3 (RB)

START
RUN 1

09-24-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
08:48:08	12.6	512.4	118.7	184.4	6.6	100.7
08:49:08	12.9	514.1	110.5	393.9	6.1	91.7
08:50:08	12.8	529.8	110.3	446.6	6.2	94.2
08:51:08	12.5	507.0	116.7	433.1	6.6	103.5
08:52:07	12.4	548.0	122.2	288.9	6.7	102.4
08:53:07	12.7	544.1	117.3	386.1	6.4	99.3
08:54:07	12.6	539.0	117.3	335.7	6.5	101.4
08:55:07	12.6	549.1	118.2	362.5	6.5	100.5
08:56:07	12.7	548.9	118.3	369.0	6.3	97.3
08:57:07	12.8	542.6	117.4	404.8	6.2	95.3
08:58:07	13.0	535.5	114.9	407.0	6.1	93.3
08:59:07	13.0	534.9	113.3	392.5	6.1	91.9
09:00:07	13.2	529.8	108.8	435.2	6.0	89.5
09:01:07	13.1	497.2	107.6	402.7	6.0	91.4
09:02:07	13.2	513.4	107.3	320.5	6.0	89.8
09:03:07	13.3	503.6	108.5	375.8	5.9	88.8
09:04:07	13.3	505.4	108.9	345.8	5.9	88.4
09:05:06	13.2	503.4	111.0	345.5	6.0	90.0
09:06:06	13.1	515.9	114.0	336.8	6.1	92.2
09:07:06	12.9	532.6	115.4	329.2	6.2	93.7
09:08:06	12.9	569.1	116.9	395.9	6.3	92.1
09:09:06	12.9	646.6	118.1	637.5	6.3	89.7
09:10:06	12.9	719.1	130.5	891.4	6.3	89.0
09:11:06	12.9	664.9	134.5	886.9	6.3	89.4
09:12:06	13.4	627.0	121.6	660.5	5.9	85.4
09:13:06	13.8	578.9	106.0	487.5	5.6	81.4
09:14:06	14.0	565.2	100.0	492.0	5.4	77.7
09:15:06	14.2	572.6	97.0	510.8	5.3	74.8
09:16:06	13.7	580.9	104.8	654.2	5.6	79.7
09:17:06	13.6	541.8	109.6	549.4	5.6	81.8
09:18:05	13.6	538.9	108.7	503.0	5.7	82.7
09:19:05	13.6	540.8	109.9	474.4	5.6	83.3
09:20:05	13.6	556.0	110.4	542.2	5.7	82.0
09:21:05	13.8	555.4	105.1	575.8	5.5	79.1
09:22:05	13.7	553.2	102.2	568.8	5.6	81.6
09:23:05	13.5	551.3	102.9	563.0	5.7	84.0
09:24:05	13.3	551.0	103.7	547.6	5.8	86.6
09:25:05	13.2	558.6	104.6	551.5	5.9	87.4
09:26:05	13.1	560.1	104.9	527.5	6.0	89.6
09:27:05	13.0	541.6	105.6	507.1	6.1	91.6
09:28:05	13.0	557.4	105.9	506.0	6.1	90.3
09:29:05	13.2	557.9	103.1	551.1	6.0	86.8
09:30:05	13.3	550.6	102.1	565.8	5.9	85.7
09:31:04	13.0	497.9	104.0	482.3	6.1	93.3
09:32:04	12.8	503.5	109.5	273.5	6.3	97.5
09:33:04	12.9	493.8	109.5	266.9	6.2	94.9
09:34:04	12.9	490.8	107.7	267.3	6.2	94.6
09:35:04	12.9	494.2	108.3	268.3	6.2	94.3
09:36:04	13.0	502.0	108.5	283.7	6.2	93.5
09:37:04	13.0	498.5	109.8	293.4	6.1	92.9
09:38:04	12.9	495.9	111.4	293.3	6.2	95.0
09:39:04	12.7	498.1	112.1	258.5	6.4	98.7
09:40:04	12.6	509.3	113.0	245.5	6.6	100.9
09:41:04	12.4	524.0	116.7	241.6	6.8	103.9
09:42:04	12.3	536.4	119.5	235.4	6.9	106.6

— Fyrite
O_2 13.5%
CO_2 6%

```
09-24-1991      O2      THC     SO2     CO      CO2     NOX
Time            %       PPMV    PPMV    PPMV    %       PPMV
==========================================================================================================
09:44:04        12.2    536.8   121.6   194.7   7.0     108.4
09:45:03        12.3    516.1   119.3   178.2   6.9     106.3
09:46:03        12.4    507.1   117.2   172.9   6.7     104.9
09:47:03        12.5    510.6   116.3   184.1   6.7     103.5
09:48:03        12.6    496.3   114.1   199.2   6.5     101.2
09:49:03        12.7    491.8   115.2   207.2   6.4     100.6
09:50:03        12.7    491.3   115.3   201.5   6.4     100.1
09:51:03        12.7    489.9   114.4   210.6   6.4      99.6
09:52:03        12.8    486.8   112.8   227.0   6.2      96.7
09:53:03        12.9    490.6   113.1   244.0   6.2      96.1
09:54:03        12.8    491.5   114.2   231.8   6.2      96.7
09:55:03        12.7    500.6   116.5   231.6   6.4      99.5
09:56:03        12.7    499.5   118.6   217.0   6.6     101.6
09:57:03        12.5    501.4   120.5   202.1   6.7     103.0
09:58:02        12.3    512.7   121.3   186.4   6.9     105.4   END ALDEHYDE RUN #3
09:59:02        12.3    519.5   122.8   186.7   7.0     107.1
10:00:02        12.1    525.3   123.2   177.1   7.0     107.6
10:01:02        12.1    523.6   124.3   169.4   7.1     107.6
10:02:02        12.3    510.6   121.9   182.5   6.9     105.0
10:03:02        12.4    510.0   120.4   186.3   6.8     103.6
10:04:02        12.4    516.7   120.9   187.6   6.8     103.0
10:05:02        12.6    508.1   120.2   191.9   6.6     100.7
10:06:02        12.5    510.1   120.6   190.8   6.7     102.5   START PMID # 1
10:07:02        12.5    506.1   120.4   198.1   6.7     102.0
10:08:02        12.6    504.4   118.7   206.6   6.6      99.3
10:09:02        12.7    499.0   116.1   219.3   6.4      97.3
10:10:02        12.7    505.1   115.6   200.6   6.4      97.6
10:11:01        12.7    484.6   114.8   195.2   6.5      99.1
10:12:01        12.5    479.1   115.1   175.5   6.7     101.9
10:13:01        12.3    487.1   114.0   168.6   6.8     102.9
10:14:01        12.7    470.2   109.1   169.2   6.3      95.4
10:15:01        12.7    457.6   107.2   177.5   6.4      98.7
10:16:01        12.5    458.7   110.1   165.1   6.7     101.6
10:17:01        12.2    465.5   114.7   145.5   6.9     106.9
10:18:01        11.9    485.0   122.7   118.1   7.1     109.6
10:19:01        12.2    482.2   120.3   120.5   7.0     105.6
10:20:01        12.4    470.5   113.5   133.3   6.7     101.5
10:21:01        12.4    462.7   111.1   136.1   6.7     101.2
10:22:01        12.4    451.2   111.6   147.7   6.7     102.7
10:23:01        12.3    474.5   117.0   132.3   6.8     106.6
10:24:00        12.2    479.9   119.0   130.0   6.8     106.3
10:25:00        12.3    470.6   117.6   132.7   6.8     106.5
10:26:00        12.2    466.0   118.2   135.7   6.9     108.5
10:27:00        12.2    470.9   119.9   134.0   6.9     107.8
10:28:00        12.2    474.0   118.8   142.6   6.9     108.3
10:29:00        12.2    496.8   116.3   143.5   6.9     106.0
10:30:00        11.8    501.1   122.3   126.7   7.2     113.0
10:31:00        11.7    507.3   124.3   120.6   7.3     114.6
10:32:00        11.6    506.7   124.9   111.9   7.4     115.6
10:33:00        11.5    516.3   127.1   109.7   7.4     116.9
10:34:00        11.5    509.0   128.1   105.1   7.5     117.1
10:35:00        11.5    509.2   125.2   104.2   7.5     116.5
10:36:00        11.6    520.3   123.7   105.2   7.4     115.3
10:36:59        11.7    510.6   122.7   114.0   7.4     115.0
10:37:59        11.8    501.7   121.7   114.4   7.2     112.9
```

09-24-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
10:39:59	11.8	497.7	122.1	122.6	7.2	113.4
10:40:59	11.9	495.7	122.0	122.6	7.2	112.6
10:41:59	11.9	491.6	120.9	123.8	7.1	112.6
10:42:59	11.8	490.6	121.5	123.2	7.2	113.0
10:43:59	12.0	490.5	120.2	126.6	7.1	111.7
10:44:59	11.9	489.7	119.5	126.0	7.2	113.4
10:45:59	11.8	494.6	122.4	117.5	7.3	115.5
10:46:59	11.7	500.3	124.0	111.6	7.3	116.4
10:47:59	11.6	516.1	125.9	106.0	7.5	118.0
10:48:59	11.4	527.0	126.2	102.7	7.6	120.3
10:49:58	11.3	518.4	127.2	90.4	7.6	120.7
10:50:58	11.4	514.0	125.4	85.7	7.5	118.7
10:51:58	11.7	503.4	122.6	100.3	7.4	115.3
10:52:58	11.6	507.7	120.6	106.0	7.4	116.1
10:53:58	11.7	504.9	120.5	107.3	7.4	114.6
10:54:58	11.8	498.1	119.2	112.4	7.3	114.1
10:55:58	11.7	496.6	120.8	113.2	7.3	114.7
10:56:58	11.7	497.4	119.4	110.5	7.3	114.0
10:57:58	12.0	481.2	116.8	114.0	7.1	111.2
10:58:58	12.1	483.3	116.6	120.6	7.0	110.0
10:59:58	12.1	490.0	115.8	122.0	7.0	110.1
11:00:58	12.2	481.4	115.9	123.3	7.0	110.5
11:01:58	11.9	486.4	116.6	122.0	7.2	112.9
11:02:57	11.7	492.0	118.8	106.0	7.3	114.2
11:03:57	11.7	494.6	117.9	98.2	7.4	113.6
11:04:57	11.7	498.3	117.7	111.4	7.3	113.9
11:05:57	11.6	503.6	119.5	107.3	7.4	115.1
11:06:57	11.6	494.8	119.2	108.5	7.4	115.1
11:07:57	11.7	483.5	117.5	110.3	7.3	113.3
11:08:57	11.7	482.5	116.0	112.0	7.3	112.6
11:09:57	11.7	477.9	114.9	115.4	7.1	110.8
11:10:57	12.1	464.6	113.0	124.3	7.1	109.2
11:11:57	12.1	474.2	112.3	127.2	7.1	110.1
11:12:57	11.9	471.5	114.6	112.5	7.1	110.7
11:13:57	12.2	464.2	112.3	127.1	7.0	108.7
11:14:57	12.3	463.7	110.5	129.3	6.8	106.9
11:15:56	12.2	461.7	111.0	126.2	6.9	107.4
11:16:56	12.4	454.8	109.7	131.1	6.7	105.3
11:17:56	12.3	460.0	109.5	134.3	6.8	107.0
11:18:56	12.2	458.7	111.8	123.9	7.0	108.6
11:19:56	12.0	466.7	113.2	119.7	7.1	109.9
11:20:56	11.8	472.4	114.9	113.0	7.3	113.9
11:21:56	11.6	492.2	119.5	102.4	7.5	117.9
11:22:56	11.4	553.6	119.3	94.7	7.6	116.6
11:23:56	2.8	715.9	39.8	42.6	1.2	8.5
11:24:56	1.3	689.3	10.2	4.8	0.1	3.3
11:25:56	1.2	710.6	7.2	4.1	0.0	2.0
11:26:56	1.3	742.5	6.0	4.3	0.0	1.6
11:27:56	1.3	760.3	5.7	4.7	0.0	1.5
11:28:55	1.3	767.1	5.2	4.5	-0.0	1.3
11:29:55	1.3	771.4	4.9	4.7	0.0	1.2
11:30:55	1.3	826.2	4.9	5.8	0.0	1.2
11:31:55	1.3	806.6	4.9	5.4	0.0	1.1
11:32:55	0.6	208.1	3.9	-1.9	-0.2	1.1
11:33:55	0.2	117.7	4.0	-0.9	-0.2	1.0

END PM. 10? #1

END PM10 #1 ?

THC span ✓.

```
09-24-1991      O2      THC     SO2     CO      CO2     NOX
Time            %       PPMV    PPMV    PPMV    %       PPMV
================================================================================
11:35:55        0.1     61.3    4.2     0.1     -0.1    0.9
11:36:55        1.7     77.0    3.9     -0.8    -0.1    0.7
11:37:55        0.1     60.9    2.5     -0.3    -0.2    0.5
11:38:55        0.1     44.9    2.1     -0.3    -0.2    0.5
11:39:55        0.1     37.3    2.0     -0.5    -0.2    0.5
11:40:55        0.1     32.6    2.0     -0.3    -0.2    0.5
11:41:55        0.2     76.0    1.9     -0.3    -0.2    0.5
11:42:54        1.3     749.6   2.4     3.2     -0.1    0.5
11:43:54        1.3     775.0   2.6     3.8     -0.1    0.5
11:44:54        0.8     429.3   2.4     1.5     -0.1    0.4
11:45:54        0.1     -4.8    2.2     -0.7    -0.2    0.4
11:46:54        1.1     630.7   2.6     2.3     -0.1    0.5
11:47:54        1.3     817.7   2.5     4.8     -0.1    0.5
11:48:54        1.3     816.1   2.5     4.4     -0.0    0.5
11:49:54        1.3     818.1   2.6     4.6     -0.0    0.5
11:50:54        1.1     636.7   2.2     2.5     -0.1    0.4
11:51:54        0.3     102.0   1.6     -3.1    -0.2    0.4
11:52:54        0.2     59.8    1.8     -1.8    -0.2    0.4
11:53:54        0.1     -7.5    2.0     -0.4    -0.2    0.4
================================================================================
Avg.=           11.9    384.1   79.8    159.8   4.7     70.5
================================================================================
```

THC span 810 ppm

PB

```
                  CALIBRATION SUMMARY
                  09-24-1991    11:57:52
          CALIBRATION FILE NAME =D:\CEMDATA\0924CAL2.CAL

==========================================================================
Chan. Name Units |      Zero        |      Span       |   Slope     Int.
                 |  Conc.  Resp.    | Conc.  Resp.    |
==========================================================================
  1   O2    %      0.00    -.0004     18.00  0.074     242.487      0.09
  2   THC   PPMV   0.00    -.0368    810.00  7.995     100.854      3.71
  3   SO2   PPMV   0.00    0.0002    295.00  0.059    4990.611     -1.20
  4   CO    PPMV   0.00    0.0003    474.00  0.047   10059.478     -2.58
  5   CO2   %      0.00    -.0005     17.00  0.082     206.908      0.10
  6   NOX   PPMV   0.00    -.0219    201.00  2.205      90.279      1.98
  7                0.00    0.0000      0.00  0.000       1.000      0.00
  8                0.00    0.0000      0.00  0.000       1.000      0.00
  9                0.00    0.0000      0.00  0.000       1.000      0.00
  10               0.00    0.0000      0.00  0.000       1.000      0.00
  11               0.00    0.0000      0.00  0.000       1.000      0.00
  12               0.00    0.0000      0.00  0.000       1.000      0.00
  13               0.00    0.0000      0.00  0.000       1.000      0.00
  14               0.00    0.0000      0.00  0.000       1.000      0.00

==========================================================================
```

```
            Press Shift-PrtSc to Print Out Table
                   Press (C) to Continue
```

```
               CEM INSTRUMENT DRIFT SUMMARY
                  09-24-1991    11:58:01

==========================================================================
Chan. Name Units |    Zero Conc.     |      Span         | Drift % of Scale
                 |Actual   Observed|Actual   Observed|  Zero      Span
==========================================================================
  1   O2    %     0.000   0.000       18.00     0.00    0.00      -72.00
  2   THC   PPMV  0.000   %-3.450    810.00   799.73   -0.35       -1.03
  3   SO2   PPMV  0.000   0.000      295.00     0.00    0.00       -5.90
  4   CO    PPMV  0.000   0.000      474.00     0.00    0.00      -47.40
```

```
  6   NOX   PPMV   0.000   0.000      201.00   0.00   0.00        -20.10
  7                0.000   0.000        0.00   0.00   0.00          0.00
  8                0.000   0.000        0.00   0.00   0.00          0.00
  9                0.000   0.000        0.00   0.00   0.00          0.00
 10                0.000   0.000        0.00   0.00   0.00          0.00
 11                0.000   0.000        0.00   0.00   0.00          0.00
 12                0.000   0.000        0.00   0.00   0.00          0.00
 13                0.000   0.000        0.00   0.00   0.00          0.00
 14                0.000   0.000        0.00   0.00   0.00          0.00

===================================================================================
```

```
                 Press Shift-PrtSc to Print Out Table
                          Press <C> to Continue

RADIAN CORPORATION      0924RUN2

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 15
NEW RICHMOND,WI
Performed for:                EPA
Date Printed = 09-24-1991  Current Time = 11:58:46
File Name = D:\CEMDATA\092491.PRN   Calibration File:D:\CEMDATA\0924CAL2.CAL

09-24-1991       O2      THC     SO2     CO      CO2     NOX
Time             %       PPMV    PPMV    PPMV    %       PPMV
==========================================================================================================================
11:59:27         0.9    462.2    2.1     0.3    -0.2     0.4
12:00:12         0.3    107.6    1.6    -3.1    -0.2     0.3
12:00:22         0.3    104.8    1.8    -2.8    -0.2     0.3
12:00:32         0.3    104.4    1.7    -3.1    -0.2     0.3
12:00:41         0.3    104.2    1.8    -3.1    -0.2     0.3
12:00:51         0.3    103.6    2.0    -3.1    -0.2     0.3
12:01:01         0.3    103.5    1.9    -3.1    -0.2     0.3
12:01:11         0.3    103.3    1.5    -3.0    -0.2     0.3
12:01:21         0.4    102.7    1.5    -3.1    -0.2     0.3
12:01:31         0.4    102.5    1.4    -3.1    -0.2     0.3
12:01:41         0.3    103.7    1.5    -3.1    -0.2     0.3
12:01:51         0.3     48.1    1.7    -1.1    -0.2     0.4
12:02:01        12.7     73.1    3.0    -1.1    -0.1    21.0
12:02:11        10.1     95.6   15.8     2.7     4.2    42.5
12:02:21         3.6    153.4   13.4    16.3     1.4     6.5
12:02:31         0.9    152.0    6.9    23.6     0.1     3.0
12:02:41         0.7    146.1    4.8    15.8    -0.1     2.1
12:02:51         0.6    138.9    4.1     5.0    -0.1     1.8
12:03:01         0.5    131.7    4.2    -0.2    -0.1     1.6
12:03:11         0.5    124.9    4.1    -1.9    -0.1     1.4
12:03:21         0.4    119.4    4.0    -1.9    -0.1     1.3
12:03:30         0.4    114.3    4.2    -2.0    -0.1     1.5
```

THC QC CHECK. RB

```
12:04:40          6.0   232.7    58.3    23.2     3.7    71.4
12:05:40         11.6   350.0   113.8   102.2     6.9   108.8
12:06:40         11.8   371.3   116.8   102.3     7.0   109.9
12:07:40         11.7   394.1   118.9   107.3     7.1   111.7
12:08:40         11.6   404.5   121.0   104.5     7.3   114.1
12:09:40         11.5   392.4   121.0    95.9     7.3   113.7
12:10:40         11.5   405.2   121.9    90.9     7.3   114.2
12:11:40         11.4   430.8   123.3    90.8     7.4   116.0
12:12:40         11.3   450.4   124.9    84.0     7.5   117.6
12:13:40         11.4   463.1   124.7    97.3     7.5   116.7
12:14:40         11.6   472.5   124.0   106.9     7.3   113.8
12:15:39         11.6   477.6   123.6    99.7     7.3   114.2
12:16:39         11.6   483.1   122.6    94.0     7.3   112.3
12:17:39         11.7   483.9   120.3   104.5     7.2   112.4
12:18:39         11.6   480.4   120.9   107.3     7.3   114.0
12:19:39         11.6   480.3   120.3    98.7     7.3   114.6
12:20:39         12.0   471.1   117.8   104.8     7.0   110.4
12:21:39         12.0   469.9   115.8   112.0     7.0   110.0
12:22:39         12.0   476.6   117.1   104.2     7.0   111.0
12:23:39         12.1   478.9   115.6   109.7     7.0   109.6
12:24:39         11.9   484.4   117.1   109.4     7.1   112.7
12:25:44         11.8   492.2   118.9    99.3     7.3   114.3
12:26:44         11.7   496.5   120.4    99.0     7.3   114.8
```

ON LINE

```
09-24-1991       O2      THC     SO2     CO      CO2     NOX
Time             %       PPMV    PPMV    PPMV    %       PPMV
====================================================================================================
12:27:44         11.5    499.9   122.0    93.1   7.4     117.3
12:28:44         11.4    501.6   123.0    86.5   7.5     118.3
12:29:44         11.3    509.4   123.3    86.3   7.5     119.3
12:30:44         11.3    513.0   123.8    87.6   7.6     119.4
12:31:44         11.4    517.3   122.6    88.9   7.5     119.0
12:32:44         11.4    515.0   121.8    91.5   7.5     117.6
12:33:44         11.6    511.0   121.1    96.0   7.4     115.6
12:34:44         11.7    513.5   121.0    98.3   7.2     115.2
12:35:44         11.5    512.4   121.0    96.2   7.4     117.4
12:36:44         11.6    501.8   117.9    94.2   7.3     116.6
12:37:43         11.6    504.2   117.1    89.3   7.3     115.0
12:38:43         11.7    497.7   118.5   106.4   7.2     113.1
12:39:43         12.0    492.8   115.8   111.9   7.0     111.0
12:40:43         11.9    488.2   116.1   110.5   7.1     112.5
12:41:43         11.8    495.4   118.0   104.5   7.2     114.3
12:42:43         11.6    504.7   119.5   104.8   7.4     116.8
12:43:43         11.5    512.1   121.4   100.8   7.4     117.9
12:44:43         11.5    518.6   122.6    89.7   7.4     117.3
12:45:43         11.4    521.3   123.0    87.4   7.6     120.0
12:46:43         11.3    523.0   123.8    82.5   7.6     120.2
12:47:43         11.4    527.5   123.8    77.1   7.5     118.1
12:48:43         11.5    522.1   122.7    82.6   7.5     118.7
12:49:43         11.4    521.7   122.2    80.9   7.5     118.2
12:50:42         11.6    519.2   120.5    88.6   7.3     115.6
12:51:42         11.7    512.3   118.6   102.1   7.3     113.4
12:52:42         11.9    508.1   116.0   106.7   7.2     112.8
12:53:42         11.9    507.5   116.1   101.5   7.1     111.5
12:54:42         11.9    503.3   116.0   100.8   7.1     111.9
12:55:42         12.0    492.7   115.5   104.3   7.1     111.6
12:56:42         12.0    493.8   114.9   115.9   7.0     111.1
12:57:42         11.8    500.9   117.0   120.9   7.2     112.8
12:58:42         11.7    505.5   117.7   114.1   7.4     115.5
12:59:42         11.5    518.1   120.1   112.9   7.5     116.8
13:00:42         11.4    532.0   121.3   107.0   7.5     117.9
13:01:42         11.3    533.9   123.3    93.6   7.8     120.5
13:02:42         11.1    545.4   125.5    88.0   7.8     121.9
13:03:41         11.1    540.2   125.7    84.9   7.8     121.8
13:04:41         11.3    530.5   122.9    85.7   7.7     120.0
13:05:41         11.3    526.1   122.8    90.6   7.7     120.4
13:06:41         11.4    528.3   121.2    97.1   7.6     120.0
13:07:41         11.4    527.8   121.6    92.2   7.6     119.2
13:08:41         11.4    522.8   120.1    97.0   7.6     119.5
13:09:41         11.4    520.6   119.6    94.2   7.5     118.7
13:10:41         11.6    517.6   119.3   101.9   7.4     116.3
13:11:41         11.9    502.7   116.6   113.7   7.2     112.5
13:12:41         11.8    502.5   119.4   118.5   7.2     113.2
13:13:41         11.9    499.7   118.8   119.0   7.2     112.5
13:14:41         11.8    496.6   116.9   123.7   7.2     113.8
13:15:41         11.5    703.7   119.5   210.0   7.7     113.0
13:16:41         11.2    738.6   128.4   576.3   7.8     118.4
13:17:40         11.3    646.0   129.3   107.1   7.7     121.9
13:18:40         11.2    606.3   130.5    86.2   7.8     123.2
13:19:40         14.4    482.6    93.8    67.5   4.6      65.1
13:20:40         20.6    270.4    14.4    15.1   0.0       3.7
13:21:40         20.7    185.9     9.8     7.2  -0.1       2.2
```

START PAH/METALS RUN #3 (EB)

Fyrite check

1310 O_2 = 12.5
CO_2 = 7.5

STOP PAH/METALS RUN #3 (EB)

PLANT

09-24-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
13:23:40	20.7	122.7	7.8	4.4	-0.1	2.0
13:24:40	20.7	108.4	7.2	3.6	-0.1	1.6
13:25:40	20.6	99.0	6.8	2.2	-0.1	1.1
13:26:40	20.5	94.8	6.6	3.2	-0.1	1.0
13:27:40	20.6	85.3	6.7	4.1	-0.1	0.9
13:28:40	20.5	77.5	6.8	3.4	-0.1	0.9
13:29:40	20.6	70.6	6.7	3.0	-0.1	0.8
13:30:39	20.6	65.1	6.5	2.3	-0.1	0.8
13:31:39	20.6	58.2	6.4	2.1	-0.1	0.7
13:32:39	20.6	53.8	6.4	1.2	-0.1	0.7
13:33:39	20.4	90.9	9.4	9.0	0.1	3.6
13:34:39	17.0	142.7	53.1	202.2	2.8	48.0
13:35:39	12.7	280.1	109.2	191.6	6.2	94.5
13:36:39	12.4	325.7	110.0	171.0	6.6	101.8
13:37:39	12.1	369.5	113.3	123.7	6.8	105.8
13:38:39	12.1	398.6	114.4	118.1	6.9	106.6
13:39:39	11.9	427.3	116.6	118.9	7.1	109.2
13:40:39	11.6	465.2	122.5	105.0	7.3	112.1
13:41:39	11.6	491.8	125.1	99.4	7.3	112.1
13:42:39	11.7	498.4	123.9	103.2	7.3	112.6
13:43:38	11.8	507.2	124.3	104.1	7.2	111.6
13:44:38	11.7	517.1	126.5	112.4	7.2	112.0
13:45:38	11.8	524.2	127.4	119.8	7.2	113.7
13:46:38	11.6	537.7	130.7	110.2	7.4	116.6
13:47:38	11.3	554.5	134.3	107.6	7.5	118.5
13:48:38	11.3	577.9	137.1	100.3	7.7	121.5
13:49:38	11.3	605.5	139.1	102.3	7.7	119.6
13:50:38	12.5	534.1	118.0	255.9	6.6	102.9
13:51:38	11.4	538.8	120.0	154.1	7.5	116.9
13:52:38	11.3	543.0	124.3	103.7	7.6	119.2
13:53:38	11.4	545.5	125.5	110.4	7.4	117.6
13:54:38	11.5	547.9	125.6	110.7	7.4	118.1
13:55:38	11.5	533.6	122.9	113.7	7.4	116.7
13:56:37	11.5	526.4	123.6	114.9	7.3	117.3
13:57:37	11.5	529.7	125.1	108.1	7.3	116.7
13:58:37	11.6	515.4	122.8	111.0	7.3	115.2
13:59:37	11.7	489.6	120.1	112.1	7.1	113.8
14:00:37	11.7	489.0	120.5	115.3	7.1	113.9
14:01:37	11.7	498.6	122.1	111.0	7.2	115.7
14:02:37	11.6	510.5	126.3	110.2	7.3	117.6
14:03:37	11.4	520.5	129.1	120.3	7.4	117.8
14:04:37	11.5	521.9	128.0	113.4	7.4	117.1
14:05:42	11.4	531.2	130.0	114.6	7.5	118.4
14:06:41	11.3	535.7	131.6	122.8	7.6	119.5
14:07:41	11.2	537.9	132.8	116.4	7.5	119.6
14:08:41	11.2	520.8	132.6	112.7	7.7	119.9
14:09:41	11.4	526.8	130.2	103.7	7.5	118.0
14:10:41	11.4	534.6	130.1	106.6	7.5	117.9
14:11:41	11.6	538.3	129.3	128.8	7.3	113.5
14:12:41	12.1	541.8	122.9	148.8	7.0	108.7
14:13:41	11.8	542.2	123.5	144.1	7.1	110.4
14:14:41	11.9	529.2	122.6	147.0	7.1	108.9
14:15:41	12.2	519.2	120.3	141.3	7.0	108.3
14:16:41	12.1	510.1	120.4	141.9	6.9	108.1
14:17:41	12.2	510.0	119.9	139.9	6.9	107.3

ON-LINE CONTINUE

PAH|METALS #3

09-24-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
14:19:40	12.2	507.1	120.2	159.5	6.9	107.9
14:20:40	12.1	517.0	121.7	163.9	6.9	108.4
14:21:40	11.9	515.7	122.7	148.1	7.1	111.0
14:22:40	11.7	521.2	123.9	137.8	7.3	113.2
14:23:40	11.7	523.9	125.5	137.3	7.3	113.6
14:24:40	11.6	527.2	125.6	127.1	7.3	113.9
14:25:40	11.8	522.4	123.9	129.0	7.2	111.2
14:26:40	11.9	513.7	122.2	132.5	7.1	110.8
14:27:40	12.0	506.9	121.8	129.8	7.0	109.6
14:28:40	12.1	502.4	120.1	131.0	7.0	108.3
14:29:40	12.1	500.3	120.0	127.6	7.0	109.4
14:30:40	12.0	500.8	120.7	119.7	7.0	108.9
14:31:40	12.2	526.7	119.8	133.1	7.0	107.2
14:32:39	12.0	576.2	119.8	349.5	7.0	104.2
14:33:39	12.3	528.8	120.7	160.1	6.8	106.6
14:34:39	12.4	514.2	119.8	156.0	6.8	104.7
14:35:39	12.4	508.9	119.3	159.1	6.7	105.4
14:36:39	12.3	505.5	121.6	148.1	6.8	106.9
14:37:39	12.1	527.3	122.9	146.0	6.9	106.9
14:38:39	11.8	584.0	125.0	341.2	7.2	107.9
14:39:39	11.8	539.7	126.7	152.6	7.1	112.2
14:40:39	11.7	536.6	128.7	129.3	7.2	113.5
14:41:39	11.7	536.7	128.6	119.6	7.2	112.3
14:42:39	11.9	531.0	125.5	134.2	7.1	110.1
14:43:39	12.0	533.3	125.3	146.7	6.9	108.4
14:44:39	12.0	527.7	125.1	143.2	6.9	108.0
14:45:38	12.3	521.2	123.0	149.1	6.7	106.7
14:46:38	12.0	516.5	126.6	142.4	6.8	108.4
14:47:38	12.1	512.0	126.4	147.6	6.8	107.4
14:48:38	12.2	505.3	124.0	157.2	6.7	106.2
14:49:38	12.2	504.6	123.4	164.1	6.7	106.0
14:50:38	12.2	509.0	123.2	168.6	6.7	105.3
14:51:38	12.3	506.4	121.1	177.0	6.7	104.7
14:52:38	12.2	510.7	123.5	169.3	6.8	106.9
14:53:38	12.0	530.1	126.4	169.0	6.8	108.0
14:54:38	11.8	529.8	127.8	164.9	7.0	109.7
14:55:38	11.7	531.9	130.7	157.6	7.1	111.9
14:56:38	11.7	536.8	131.4	155.2	7.1	112.9
14:57:38	11.5	533.4	131.6	137.7	7.2	113.9
14:58:38	11.6	532.2	130.1	144.7	7.1	111.7
14:59:37	11.8	531.0	127.4	153.9	7.0	110.5
15:00:37	11.8	533.1	127.3	162.8	7.0	109.9
15:01:37	11.9	532.7	126.9	161.2	6.9	108.4
15:02:37	12.1	523.7	125.5	182.4	6.8	107.2
15:03:37	12.1	522.5	125.3	180.7	6.8	106.7
15:04:37	12.1	517.6	125.0	173.1	6.7	105.5
15:05:37	12.2	509.0	122.9	178.0	6.6	104.2
Avg.=	11.2	436.3	100.7	103.6	5.9	92.4

END TEST (RB) PAH/METALS #3

END TEST 9/24/91

```
RADIAN CORPORATION      0924POS[illegible]

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-24-1991  Current Time = 15:11:53
File Name = D:\CEMDATA\092491.PRN   Calibration File:D:\CEMDATA\0924CAL2.CAL


 09-24-1991      O2      THC     SO2     CO      CO2     NOX
 Time            %       PPMV    PPMV    PPMV    %       PPMV
=====================================================================================================================================
15:12:36          1.0   304.5   275.7   -0.8    -0.1   191.8
15:13:12          1.0   305.9   291.6   -1.2    -0.1   193.9
15:13:22          1.0   283.7   293.5   -1.6    -0.1   ~~194.3~~
15:13:32          1.0   281.1   293.9   -1.7    -0.2   194.3
15:13:42          1.0   278.1   293.9   -1.8    -0.2   193.9
15:13:52          1.0   275.4   294.2   -1.8    -0.2   193.6
15:14:02          0.9   273.5   294.4   -1.9    -0.2   193.2
15:14:12          0.9   271.2   294.6   -1.9    -0.2   193.2
15:14:22          0.9   268.3   294.9   -1.9    -0.2   192.9
15:14:31          0.9   252.8   282.0   -1.7    -0.2   152.8
15:14:41          1.0   265.1   208.7   -1.9    -0.2   112.0
15:14:51          1.0   262.8   151.3   -1.9    -0.2   102.7
15:15:01          0.9   259.8   128.1   -1.9    -0.2    98.9
15:15:11          0.9   256.7   117.4   -1.9    -0.2    97.6
15:15:21          0.9   254.9   111.9   -1.9    -0.2    96.6
15:15:31          0.9   253.3   108.7   -2.0    -0.2    96.2
15:15:41          0.9   250.9   107.0   -2.0    -0.2    96.0
15:15:51          0.9   248.5   106.4   -2.0    -0.2    96.1
15:16:01          0.9   245.8   105.4   -2.0    -0.2    96.0
15:16:11          0.9   243.7   105.2   -2.0    -0.2    95.8
15:16:21          0.9   241.5   104.7   -2.0    -0.2    95.8
15:16:31          0.8   227.6   102.2   -1.7     0.2    73.1
15:16:41          2.4   222.5    81.8    4.1     4.1    39.2
15:16:51          5.3   225.1    59.2   21.4     7.2    26.2
15:17:01          6.5   220.4    44.0   45.3     8.4    16.1
15:18:00          7.5   211.1    21.9   83.8     9.8     6.9
15:18:33          7.7   203.6    15.4   90.7    10.1     4.6
15:18:43          7.7   200.2    14.3   93.0    10.2     4.4
15:18:53          7.7   199.3    14.3   92.2    10.2     4.4
15:19:02          7.7   198.5    14.3   91.6    10.2     4.6
15:19:12          7.8   197.3    14.6   93.6    10.2     4.5
15:19:22          7.7   196.7    14.1   92.1    10.2     4.6
15:19:32          7.8   195.8    13.9   91.1    10.2     4.7
15:19:42          7.8   195.5    14.1   93.9    10.2     4.5
15:20:42          7.7   192.1    13.3   91.5    10.2     4.0
15:21:42          6.6   438.2    14.8   88.5     7.8     6.0
15:22:42          1.8   934.3    15.8   23.1     0.7     5.6
15:23:42          1.7   951.9    14.1   12.3     0.4     3.9
15:24:42          1.7   931.6    14.4   11.7     0.4     4.9
15:25:42          1.5   761.1    14.2    8.7     0.4     5.0
15:26:42          0.3   109.9     9.5    0.1    -0.1     0.8
15:27:42          0.1    77.6     8.8    0.4    -0.1     0.7
15:28:42          1.3    35.4     7.8    0.4    -0.1     0.6
15:29:41          0.1    -8.4     5.0   -0.0    -0.2     0.5
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15:31:41         1.2   724.8     4.4     3.0    -0.1     0.7



09-24-1991       O2     THC     SO2      CO     CO2     NOX
Time             %      PPMV    PPMV     PPMV   %       PPMV
==========================================================================================================================================
15:32:41         1.4   837.1     4.7     5.1    -0.0     0.6
15:33:41         0.5   222.9     3.6    -2.2    -0.2     0.6
15:34:41         0.1    20.3     3.5    -0.5    -0.2     0.5
15:35:41         0.1    -9.8     3.4    -0.3    -0.2     0.5
15:36:41         0.1    -6.1     3.5    -0.1    -0.2     0.5
15:37:41         0.1    -7.8     3.4    -0.4    -0.2     0.5
15:38:41         0.1     4.5     3.2    -0.5    -0.2     0.6
15:39:41         1.0   367.7     3.0    -1.4    -0.2     0.6
15:40:41         1.2   689.5     3.3     3.0    -0.1     0.6
15:41:41         0.1    26.3     2.9    -0.3    -0.2     0.5
15:42:40         0.8   453.9     3.4     1.5    -0.1     0.6
15:43:40         1.0   588.5     3.6     2.5    -0.1     0.6
15:44:40         0.1    58.9     3.3    -0.1    -0.2     0.5
15:45:40         1.3   752.4     3.8     3.3    -0.1     0.6
15:46:40         1.2   735.1     3.7     3.4    -0.1     0.8
15:47:40         2.8    97.1    10.1     4.2     0.3     4.7
15:48:40         1.0   136.1     6.4    -2.1    -0.2     0.9
15:49:40         0.3    51.0     6.7     0.8    -0.1     0.8
15:50:40         0.1    46.8     7.0     0.2    -0.2     0.8
15:51:40         0.1    45.0     7.2     0.2    -0.1     0.8
15:52:40         0.1    22.1     7.2     0.0    -0.2     0.7
15:53:30         1.2   691.9     7.9     3.6    -0.1     0.8
15:53:40         1.4   810.3     7.9     4.4    -0.0     0.8
15:53:50         1.4   810.1     7.6     4.4    -0.0     0.8
15:54:00         1.3   810.5     8.0     5.4    -0.0     0.8
15:54:10         1.4   811.1     8.0     4.5    -0.0     0.8
15:54:20         1.4   811.2     7.8     4.5    -0.0     0.8
15:54:30         1.3   811.0     7.5     6.2    -0.0     0.8
15:54:40         1.3   810.6     7.7     3.4    -0.0     0.8
15:54:50         1.4   811.3     7.7     5.4    -0.0     0.8
15:55:00         1.3   806.9     7.8     4.4    -0.0     0.8
15:55:10         0.8   329.1     6.9    -0.7    -0.2     0.7
15:55:20         0.4   128.8     6.8    -2.0    -0.2     0.8
15:55:30         0.4   110.3     7.0    -3.0    -0.2     0.7
15:55:39         0.3   105.8     6.7    -3.0    -0.2     0.7
15:55:49         0.3   103.9     6.9    -3.0    -0.2     0.7
15:55:59         0.3   102.9     6.7    -2.9    -0.2     0.7
15:56:09         0.3   102.3     6.7    -2.1    -0.2     0.7
15:56:19         0.3    99.[illegible]     6.9    -2.8    -0.2     0.7
15:56:29         0.1    62.0     6.9     0.7    -0.2     0.8
15:56:39         0.1    50.4     7.0     0.1    -0.2     0.8
15:56:49         0.1    46.7     7.1     0.1    -0.2     0.8
15:56:59         0.1    45.4     6.8     0.2    -0.2     0.7
15:57:09         0.1    44.9     6.3     1.1    -0.2     0.7
15:57:19         0.1    44.5     6.9     0.9    -0.2     0.8
15:57:29         0.1    44.3     6.7     0.1    -0.2     0.7
15:57:39         0.1    44.0     6.8     0.2    -0.2     0.7
15:57:49         0.1    44.1     6.8     0.9    -0.2     0.8
15:57:59         0.1    44.0     6.8     0.2    -0.2     0.7
15:58:09         0.1    44.0     6.5     0.9    -0.2     0.7
15:58:19         0.1    40.2     6.7     0.1    -0.2     0.7
15:58:28         0.1    13.7     7.0     0.2    -0.2     0.7
15:58:38         0.1     3.3     7.1     0.9    -0.2     0.7
15:58:48         0.0     1.4     6.9     0.0    -0.2     0.7
15:58:58         0.0     1.2     6.9     0.0    -0.2     0.7
```

END QC check

```
RADIAN CORPORATION      09250CCK

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 16
NEW RICHMOND,WI
Performed for:              EPA
Date Printed = 09-25-1991  Current Time = 07:00:07
File Name = D:\CEMDATA\092591.PRN   Calibration File:D:\CEMDATA\0924CAL2.CAL


 09-25-1991      O2      THC     SO2      CO      CO2     NOX
 Time            %       PPMV    PPMV     PPMV    %       PPMV
=================================================================================================================
07:00:56        1.7    -57.3     4.0      0.1    -0.2      0.5
07:01:57        0.2    -57.3     4.1     -0.1    -0.2      0.4
07:02:57        0.1    -57.3     4.0     -0.1    -0.2      0.4
07:03:57        0.1    -53.2     3.9     -0.1    -0.2      0.4
07:04:57        0.1      4.6    13.2     -0.2    -0.2     22.0
07:05:57       -0.0      4.1   243.7     -4.4    -0.3    196.9
07:06:56       -0.0     19.1   252.6     -4.6    -0.3    198.6
07:07:56        0.5    203.2    90.7     -0.2    -0.2     32.9
07:08:56        1.5    682.1     9.9      2.4    -0.1      2.4
07:09:56        1.9    789.4     6.3      4.1    -0.1      0.9
07:10:56        1.3    312.4     5.1      0.7    -0.2      0.7
07:11:56        0.6     37.6     4.7     -0.2    -0.2      0.4
07:12:56        1.8    403.3     4.5      3.1    -0.2      0.4
07:13:56        2.2    796.0     4.8      4.2    -0.1      0.4
=================================================================================================================
 Avg.=          0.9    216.2    46.7      0.1    -0.2     32.1
=================================================================================================================
```

```
                    CALIBRATION SUMMARY
                    09-25-1991   07:28:36
          CALIBRATION FILE NAME =D:\CEMDATA\0925CAL1.CAL

===========================================================================
Chan. Name Units |      Zero         |      Span      |   Slope      Int.
                 | Conc.   Resp.     | Conc.  Resp.   |
===========================================================================
  1   O2    %     0.00    0.0020     18.00  0.072     256.333     -0.51
  2   THC   PPMV  0.00    0.0189    810.00  7.986     101.665     -1.92
  3   SO2   PPMV  0.00    0.0010    295.00  0.057    5243.973     -5.40
  4   CO    PPMV  0.00    0.0003    474.00  0.048    9855.925     -2.54
  5   CO2   %     0.00    -.0014     17.00  0.080     207.773      0.28
  6   NOX   PPMV  0.00    -.0190    201.00  2.143      92.983      1.77
  7               0.00    0.0000      0.00  0.000       1.000      ....
  8               0.00    0.0000      0.00  0.000       1.000      ....
  9               0.00    0.0000      0.00  0.000       .....      ..[illegible]
 10               0.00    0.0000      0.00  0.000       ..[illegible]      [illegible].00
 11               0.00    0.0000      0.00  [illegible]       .[illegible]00      0.00
 12               0.00    0.0000      [illegible]...  .[illegible]..       1.000      0.00
 13               0.00    0.0000      ....  .[illegible]00       1.000      0.00
 14               0.00    ......      ....  0.000       1.000      0.00

===========================================================================




              Press Shift-PrtSc to Print Out Table
                    Press <C> to Continue


                CEM INSTRUMENT DRIFT SUMMARY
                    09-25-1991   07:28:42

===========================================================================
Chan. Name Units :    Zero Conc.   :     Span         .  Drift % of Scale
                 :Actual   Observed:Actual   Observed.  Zero      Span
===========================================================================
  1   O2    %     [illegible]    0.570      18.00   17.50   2.28        -1.51
  2   THC   PPMV  0.000    8.811     810.00  809.14   0.56        -0.09
  3   SO2   PPMV  0.000    0.400     295.00  294.53   0.08        -0.21
  4   CO    PPMV  0.000    0.008     474.00  463.80   0.00         0.98
```

```
 [illegible]
 7                  0.000   0.000      0.00    0.00   0.00          0.00
 8                  0.000   0.000      0.00    0.00   0.00          0.00
 9                  0.000   0.000      0.00    0.00   0.00          0.00
 10                 0.000   0.000      0.00    0.00   0.00          0.00
 11                 0.000   0.000      0.00    0.00   0.00          0.00
 12                 0.000   0.000.     0.00    0.00   0.00          0.00
 13                 0.000   0.000      0.00    0.00   0.00          0.00
 14                 0.000   0.000      0.00    0.00   0.00          0.00

=========================================================================
```

```
                  Press Shift-PrtSc to Print Out Table
                        Press <C> to Continue

RADIAN CORPORATION      [illegible]

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
[illegible]
NEW RICHMOND,WI
Performed for:            EPA
Date Printed = [illegible]  Current Time = [illegible]
File Name = [illegible]   Calibration File:[illegible].CAL


 [illegible]      O2      THC     SO2      CO      CO2      NOx
 Time             %       PPMV    PPMV     PPMV    %        PPMV
==============================================================================================================
[illegible]       [illegible]  [illegible]  0.3   7.3     0.2     -0.1
[illegible]       0.5     335.0   -0.0     1.4     0.1      -0.2
[illegible]       0.4      43.2   -0.1     0.9     0.1      -0.2
07:33:10         -0.4      -0.5    0.2     1.5     0.1      -0.2
07:34:10          4.4       3.8    0.2    25.2     4.8      -0.2
07:35:16          8.8       4.1   -0.2    78.5     8.8      -0.2
07:35:31          7.6       4.4   -0.1    87.1     9.9      -0.2
07:35:46          7.4       4.4    0.1    90.6    10.0      -0.2
07:36:01          7.4       4.4    0.1    91.9    10.1      -0.2
07:36:16          7.4      26.1    0.4    92.2    10.1      -0.2
07:36:31          7.3      88.7    0.4    92.3     9.8      -0.2
07:36:40          4.9      96.5    0.3    88.7     5.3      -0.2
07:36:50          1.7      97.3   -0.5    65.4     2.0      -0.2
07:37:00          0.5      97.6   -0.5    38.3     0.7      -0.3
07:37:10          0.1      96.3   -0.6    16.9     0.4      -0.3
07:37:20         -0.2      58.1   -0.2     7.7     0.4      -0.2
07:37:30         -0.3      13.4    1.2     4.1     0.3       8.9
07:37:40          0.1       4.3   22.9     2.9     0.3      54.4
07:37:50         -0.1       0.8   60.9     2.8     0.3      84.1
07:38:00         -0.3      -0.7   82.8     1.4     0.1      94.9
07:38:10         -0.4      -1.3   92.5     0.2     0.1      99.4
07:38:20         -0.5      -1.5   96.[illegible]  -0.8   0.0     101.3
```

```
07:38:40        -0.5    -0.4    99.7    -1.8     0.0   102.2
07:38:50        -0.5    -0.5   100.2    -1.7     0.0   100.7
07:39:00        -0.5    -1.3    88.9    -0.5     0.1    81.1
07:39:10        -0.4    -1.7    53.0     0.9     0.1    61.0
07:39:20        -0.4    -1.8    24.4     1.0     0.1    52.5
07:39:29        -0.4    -2.0    10.9     1.0     0.1    49.4
07:39:39        -0.4    -2.0     5.0     1.0     0.1    48.2
07:39:49        -0.5    -1.9     2.6     1.0     0.1    47.7
07:39:59        -0.4    -2.0     1.6     1.0     0.1    47.4
07:40:09        -0.5    -1.9     1.1     1.0     0.1    47.3
07:40:19        -0.5    -2.0     0.3     1.7     0.1    47.2
07:40:29        -0.5    -1.9     0.3     0.5     0.1    47.2
07:40:39        -0.5    -1.7     0.2     0.7     0.1    47.3
==================================================================================================================================
Avg.=            1.4    49.0    23.5    22.3     2.1    36.7
==================================================================================================================================
```

RADIAN CORPORATION 0925RUN1

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY 15
NEW RICHMOND,WI
Performed for: EPA
Date Printed = 09-25-1991 Current Time = 07:40:55
File Name = D:\CEMDATA\092591.PRN Calibration File:D:\CEMDATA\0925CAL1.CAL

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
07:41:39	10.2	96.9	2.9	85.4	0.5	18.2
07:42:39	3.2	2.5	1.3	107.4	0.1	5.1
07:43:39	2.0	-0.6	0.1	4.4	0.0	0.1
07:44:39	-0.5	-1.3	-0.0	0.9	0.0	-0.2
07:45:39	-0.5	-1.5	-0.1	0.1	0.0	-0.3
07:46:39	0.7	52.6	1.7	3.0	0.1	1.3
07:47:39	18.2	103.4	20.4	185.8	0.8	9.1
07:48:38	19.9	73.9	25.8	229.0	1.0	11.5
07:49:38	19.7	59.7	32.8	163.0	1.2	14.9
07:50:38	19.6	63.1	37.4	149.7	1.3	18.6
07:51:38	19.9	46.8	32.4	115.2	0.9	11.0
07:52:38	20.5	96.1	17.3	108.4	0.7	8.7
07:53:38	20.0	83.1	26.9	203.7	1.0	12.1
07:54:38	19.9	65.6	28.1	187.4	1.1	13.0
07:55:38	19.4	50.1	38.6	146.1	1.4	19.8
07:56:38	19.4	37.7	40.0	115.1	1.3	17.5
07:57:38	20.8	80.5	12.5	50.3	0.5	6.2
07:58:38	19.9	75.6	28.5	182.0	1.1	13.4
07:59:38	19.8	72.9	30.1	184.3	1.1	14.3
08:00:38	19.8	69.2	30.9	171.3	1.1	14.9
08:01:37	19.7	63.8	31.7	168.7	1.2	15.5
08:02:37	19.7	60.7	32.6	155.6	1.2	16.1
08:03:37	19.7	55.4	33.1	152.6	1.2	16.5
08:04:37	19.7	55.0	33.5	146.5	1.2	16.9
08:05:37	19.6	53.8	34.3	140.0	1.2	17.4
08:06:37	19.6	60.5	34.7	144.4	1.3	17.6
08:07:37	19.6	55.6	35.3	157.1	1.3	17.9
08:08:37	19.6	53.2	35.8	138.7	1.3	18.4
08:09:37	19.6	57.3	36.2	144.0	1.3	18.7
08:10:37	19.6	59.1	36.9	145.8	1.3	18.9
08:11:37	19.6	61.2	37.0	158.4	1.3	19.2
08:12:37	19.5	58.8	37.6	154.2	1.3	19.7
08:13:37	19.5	60.1	38.1	142.1	1.4	20.0
08:14:36	20.5	35.9	19.8	107.8	0.6	6.6
08:15:36	20.9	80.7	10.2	35.9	0.5	7.2
08:16:36	18.9	60.6	45.6	189.7	1.8	24.3
08:17:36	20.9	64.5	14.3	72.3	0.3	3.6
08:18:36	19.5	77.1	33.4	127.3	1.4	20.2
08:19:36	20.0	43.2	27.4	139.4	0.9	9.9
08:20:36	21.3	34.9	1.8	8.8	0.1	0.6
08:21:36	21.3	19.5	0.2	3.0	0.1	0.2
08:22:36	21.3	15.1	-0.1	2.1	0.1	0.1
08:23:36	21.4	12.7	-0.5	1.8	0.1	-0.0
08:24:36	21.4	11.1	-0.7	1.3	0.1	-0.0

```
08:26:35       21.4     6.9    -0.9     1.1     0.0    -0.1



 09-25-1991     O2      THC     SO2     CO      CO2     NOX
 Time           %       PPMV    PPMV    PPMV    %       PPMV
=============================================================================================================================================
08:27:35       21.4     8.0    -1.0     1.1     0.0    -0.2
08:28:35       21.4     7.1    -1.2     1.1     0.1    -0.2
08:29:35       21.4     6.4    -1.2     1.1     0.1     0.0
08:30:35       21.3     5.8    -1.4     1.1     0.0    -0.1
08:31:35       21.4     5.3    -1.6     1.1     0.1     0.1
08:32:35       21.3     4.6    -1.6     1.1     0.0     0.1
08:33:35       21.3    13.1    -1.7     0.9     0.1     0.2
08:34:35       20.5    87.1    11.7    84.0     0.7     6.8
08:35:35       19.7    73.3    27.0   203.7     1.2    12.6
08:36:35       19.7    67.6    30.0   174.1     1.2    14.9
08:37:35       19.6    63.3    32.4   176.3     1.3    17.0
08:38:35       19.4    38.3    35.5   150.7     1.3    16.7
08:39:35       21.2    24.1     2.7    23.8     0.1     1.8
08:40:34       21.1    61.5     2.8     9.3     0.3     3.4
08:41:34       19.7    71.5    28.2   163.4     1.2    14.1
08:42:34       19.6    72.6    32.3   184.3     1.3    16.3
08:43:34       19.5    67.4    34.1   183.1     1.4    17.9
08:44:34       19.3    44.1    36.4   164.3     1.4    18.3
08:45:34       21.1    34.3     4.9    36.5     0.2     2.4
08:46:34       21.2    59.0     2.8     8.7     0.3     4.9
08:47:34       18.8    64.7    42.7   166.6     1.8    22.8
08:48:34       20.1    35.5    24.0   122.2     0.8     9.4
08:49:34       21.3    30.5     0.3     6.3     0.1     1.4
08:50:34       21.1    55.6     2.9     9.3     0.3     4.6
08:51:34       19.7    77.2    27.7   164.3     1.3    15.1
08:52:34       20.1    33.8    17.7   125.9     0.8     8.6
08:53:33       21.3    17.3     0.1    10.3     0.1     1.3
08:54:33       21.3    13.7    -1.7     2.3     0.1     0.6
08:55:33       21.3    22.9    -2.0     1.7     0.1     0.5
08:56:33       20.6   105.7    10.0    69.8     0.6     7.5
08:57:33       20.3   107.7    12.2   223.6     0.8     9.2
08:58:33       20.2   104.7     5.3   207.2     0.9     9.2
08:59:33       20.0    84.1     3.9   209.5     1.1    13.5
09:00:33       17.2   122.9    16.6   233.5     2.9    38.6
09:01:33       14.2    60.8    43.9   292.7     4.7    78.1
09:02:33       13.0    62.3    75.6   211.6     5.6    97.9
09:03:33       12.3    53.1    94.6   234.6     6.0   106.4
09:04:33       11.9   166.6   108.8   173.9     6.5   116.6
09:05:33       11.7   263.7   117.7   120.1     6.6   121.4
09:06:32       11.0   343.1   133.4    85.0     7.3   136.1
09:07:32       11.0   343.9   133.4    83.1     7.2   132.4
09:08:32       11.1   357.6   131.1    87.8     7.1   130.6
09:09:32       11.3   366.4   129.7    88.9     7.0   128.5
09:10:32       11.2   379.3   131.3    90.9     7.1   129.5
09:11:32       11.1   389.2   132.5    85.7     7.2   131.5
09:12:32       11.2   398.1   131.8    77.6     7.0   130.3
09:13:32       11.2   397.6   132.6    79.2     7.0   130.4
09:14:32       11.1   409.8   131.9    76.1     7.2   133.5
09:15:32       13.5   277.3   106.1    70.8     5.0    83.0
09:16:32       21.0   125.4     9.7    15.1     0.2     4.5
09:17:32       21.3    84.5     1.1     5.0     0.1     1.7
09:18:32       21.3    64.9    -0.2     4.2     0.1     1.2
09:19:32       20.0   137.2    18.2    33.7     1.2    28.2
09:20:31       11.9   285.7   124.3   155.1     6.6   125.3
09:21:31       11.7   339.4   122.6    99.8     6.6   121.5
```

09-25-1991	02	THC	SO2	CO	CO2	NOX
Time	%	PPMV	PPMV	PPMV	%	PPMV
09:23:31	11.8	383.1	121.9	113.0	6.6	118.8
09:24:31	11.8	388.2	121.6	112.4	6.6	119.8
09:25:31	11.4	410.1	127.9	97.7	7.0	128.3
09:26:31	11.2	425.1	133.1	79.6	7.2	132.5
09:27:31	10.8	434.3	137.9	60.9	7.5	136.9
09:28:31	10.7	441.4	140.3	53.2	7.5	136.4
09:29:31	10.5	450.3	142.5	51.5	7.7	140.9
09:30:31	10.3	457.3	144.5	53.0	7.8	142.8
09:31:31	10.2	461.7	145.2	52.1	7.7	143.1
09:32:31	10.3	473.6	146.4	53.2	7.7	142.0
09:33:30	10.7	462.8	142.6	70.8	7.6	139.0
09:34:30	10.9	456.6	136.4	87.3	7.4	132.5
09:35:30	11.1	468.9	133.2	102.8	7.2	130.1
09:36:30	11.3	455.8	131.5	109.5	7.1	126.9
09:37:30	11.4	455.8	128.9	96.5	7.0	127.9
09:38:30	11.2	461.7	132.0	90.1	7.1	131.4
09:39:30	11.1	454.6	134.3	85.0	7.2	132.4
09:40:30	11.4	440.8	130.6	106.3	7.0	127.7
09:41:30	11.6	444.5	128.5	96.4	6.9	126.6
09:42:30	11.6	439.7	128.1	95.6	6.8	124.5
09:43:30	11.7	444.8	125.5	100.3	6.7	123.5
09:44:30	11.7	452.6	125.8	111.0	6.8	124.9
09:45:30	11.5	459.6	128.2	108.9	7.0	128.0
09:46:29	11.3	477.0	128.6	102.0	7.0	128.7
09:47:29	11.2	511.4	133.8	159.1	7.1	129.5
09:48:29	11.1	527.4	141.1	182.6	7.3	132.1
09:49:29	10.9	525.1	143.7	186.8	7.4	134.1
09:50:29	10.9	516.0	142.1	118.9	7.4	135.4
09:51:29	10.9	506.7	139.8	88.3	7.4	136.9
09:52:29	10.9	500.5	137.4	73.4	7.4	134.8
09:53:29	11.0	494.5	133.6	72.8	7.3	132.2
09:54:29	10.9	487.2	135.4	70.3	7.3	133.0
09:55:29	11.0	483.3	132.6	72.0	7.3	132.5
09:56:29	11.2	481.8	130.4	82.2	7.2	131.2
09:57:29	11.3	484.8	130.8	79.4	7.1	129.0
09:58:29	11.3	487.4	131.2	80.9	7.1	128.2
09:59:28	11.4	490.4	131.1	91.8	7.0	128.3
10:00:28	11.4	484.3	131.6	100.9	6.9	127.1
10:01:28	11.6	483.2	131.6	104.5	6.9	124.5
10:02:28	11.7	483.7	131.4	122.5	6.8	124.5
10:03:28	11.8	488.5	130.3	143.0	6.8	124.2
10:04:28	11.8	499.2	131.5	165.3	6.8	124.9
10:05:28	11.6	507.6	134.0	161.0	7.0	127.3
10:06:28	11.5	521.3	133.8	135.4	6.9	127.3
10:07:28	11.4	533.1	133.2	121.9	7.1	129.3
10:08:28	11.3	531.9	135.3	126.4	7.2	131.0
10:09:28	11.2	541.7	135.8	134.7	7.2	131.5
10:10:28	11.1	546.9	137.7	141.0	7.2	132.4
10:11:28	11.2	537.8	137.0	121.6	7.2	132.5
10:12:27	11.3	517.7	133.6	109.2	7.1	129.4
10:13:27	11.4	513.5	131.4	104.8	6.9	127.2
10:14:27	11.4	501.6	128.2	102.5	7.0	128.0
10:15:27	11.7	497.9	125.5	97.8	6.8	124.2
10:16:27	11.6	494.7	126.0	99.5	6.8	124.6
10:17:27	11.5	504.6	127.0	87.3	6.9	125.9

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
10:19:27	11.8	493.1	121.3	103.3	6.7	122.8
10:20:27	11.0	534.5	134.5	90.2	7.2	139.6
10:21:27	10.7	545.8	145.4	101.4	7.5	144.4
10:22:27	10.7	560.5	146.2	149.3	7.5	139.9
10:23:27	10.6	562.5	144.8	119.7	7.6	139.3
10:24:27	10.5	573.0	142.6	94.2	7.6	137.3
10:25:26	10.5	595.8	144.1	135.0	7.6	139.0
10:26:26	10.5	595.7	143.9	160.1	7.7	137.7
10:27:26	10.7	591.3	141.2	131.0	7.5	136.4
10:28:26	10.7	594.7	139.3	142.4	7.4	132.1
10:29:26	10.9	569.7	137.5	159.6	7.3	132.2
10:30:26	10.9	556.7	137.2	117.5	7.3	135.3
10:31:26	10.9	599.7	139.5	134.4	7.3	134.1
10:32:26	10.9	662.2	148.8	289.6	7.4	132.6
10:33:26	10.4	672.8	156.0	370.6	7.7	135.9
10:34:26	10.3	653.7	155.8	316.1	7.7	135.1
10:35:26	10.8	620.3	142.5	244.0	7.3	127.6
10:36:26	10.0	620.7	154.2	199.8	8.0	149.6
10:37:26	9.6	609.2	158.1	179.0	8.2	152.1
10:38:25	9.7	590.6	153.1	140.8	8.0	149.6
10:39:25	9.8	585.5	151.6	122.8	8.0	150.2
10:40:25	10.0	571.4	148.7	134.1	7.9	145.5
10:41:25	10.1	606.6	156.2	180.7	7.8	147.1
10:42:25	10.3	603.0	160.8	266.5	7.6	142.4
10:43:25	10.7	569.1	155.1	251.1	7.5	136.4
10:44:25	10.8	549.7	149.0	167.5	7.4	136.9
10:45:25	10.8	537.9	145.5	107.1	7.4	142.1
10:46:25	10.4	543.8	145.0	96.1	7.7	146.1
10:47:25	10.2	544.8	147.1	95.9	7.8	145.8
10:48:25	10.0	542.2	148.5	105.7	7.8	141.6
10:49:25	10.0	563.1	151.4	111.1	7.9	144.3
10:50:25	10.0	553.7	152.1	133.5	7.8	142.4
10:51:24	10.3	550.5	151.5	111.1	7.6	142.0
10:52:24	10.3	545.1	153.3	104.5	7.6	143.4
10:53:24	10.5	532.7	146.4	95.3	7.5	142.5
10:54:24	10.7	528.0	142.7	83.8	7.4	139.3
10:55:24	10.6	529.0	144.1	70.4	7.5	140.8
10:56:24	10.4	530.8	143.5	70.4	7.6	140.7
10:57:24	10.6	528.9	141.8	65.4	7.5	139.6
10:58:24	10.6	523.8	141.9	62.3	7.5	140.6
10:59:24	10.3	534.8	141.9	62.5	7.6	141.1
11:00:24	10.2	547.1	143.2	71.7	7.7	142.9
11:01:24	10.0	543.6	145.2	69.8	7.8	144.7
11:02:24	9.9	543.0	146.2	83.6	7.8	145.6
11:03:24	10.0	534.2	146.8	72.2	7.7	145.2
11:04:23	10.2	540.7	143.3	85.5	7.7	143.7
11:05:23	10.4	548.4	142.8	83.3	7.6	140.0
11:06:23	10.3	546.1	144.8	94.4	7.6	140.0
11:07:23	10.6	534.0	140.9	105.6	7.4	136.4
11:08:23	10.6	546.9	139.9	85.7	7.5	138.1
11:09:23	10.4	534.4	141.6	81.1	7.5	138.4
11:10:23	10.4	521.3	141.1	69.1	7.5	138.8
11:11:23	10.5	528.7	137.8	68.2	7.5	139.2
11:12:23	10.4	536.2	138.3	71.2	7.5	136.9
11:13:23	10.3	543.4	142.0	74.3	7.5	138.4

START PM10 RUN#2 (RB)

~10:30 START 1st Test

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
11:15:23	9.9	545.8	144.4	63.7	7.9	142.2
11:16:23	10.8	545.5	130.9	59.4	7.2	128.8
11:17:23	10.5	541.0	137.7	62.6	7.5	134.9
11:18:22	10.6	539.9	137.3	55.5	7.5	134.0
11:19:22	10.8	522.8	134.0	59.6	7.3	131.1
11:20:22	10.9	537.5	133.9	65.2	7.1	128.3
11:21:22	10.9	531.1	133.8	65.3	7.2	130.7
11:22:22	10.8	518.4	134.7	56.9	7.3	135.2
11:23:22	10.9	621.4	138.3	104.3	7.2	131.0
11:24:22	11.9	916.9	149.5	452.7	7.0	114.1
11:25:22	11.5	1014.5	206.1	976.1	7.6	115.7
11:26:22	11.0	853.0	219.2	993.1	7.5	124.7
11:27:22	12.4	740.9	154.2	648.3	6.3	120.4
11:28:22	11.0	647.8	161.6	438.8	7.3	134.1
11:29:22	12.1	575.7	131.5	317.2	6.3	114.5
11:30:22	12.3	575.3	125.3	294.1	6.2	112.7
11:31:21	12.4	530.9	125.7	299.2	6.2	112.1
11:32:21	12.5	528.4	120.4	233.9	6.1	111.1
11:33:21	12.2	552.4	126.4	283.1	6.2	112.4
11:34:21	12.2	534.7	130.2	302.0	6.3	113.8
11:35:21	12.1	512.5	127.8	240.9	6.4	116.7
11:36:21	12.0	508.1	126.3	203.6	6.4	117.2
11:37:21	7.0	366.5	83.0	439.0	3.5	55.3
11:38:21	0.3	219.7	7.2	981.7	0.1	3.4
11:39:21	0.0	172.2	1.9	981.4	-0.0	1.8
11:39:51	-0.0	151.5	0.8	981.4	-0.0	1.4
11:40:01	-0.0	143.4	0.7	981.7	-0.0	1.3
11:40:11	-0.1	141.4	0.6	981.8	-0.0	1.2
11:40:21	-0.1	140.7	0.5	981.4	-0.0	1.2
11:40:31	-0.1	140.2	0.4	981.4	-0.0	1.2
11:40:41	-0.1	138.9	0.1	981.4	-0.1	1.0
11:40:51	-0.1	137.2	0.2	981.4	-0.1	1.0
11:41:01	-0.1	135.2	-0.0	981.4	-0.1	1.0
11:41:10	-0.1	134.5	0.1	981.4	-0.1	0.9
11:41:20	-0.2	132.4	0.3	981.4	-0.1	1.0
11:41:30	-0.2	129.8	0.3	981.4	-0.1	1.0
11:41:40	-0.2	127.5	-0.0	981.4	-0.1	0.9
11:41:50	-0.2	125.8	-0.1	981.4	-0.1	0.9
11:42:00	-0.1	124.2	-0.2	981.4	-0.1	0.8
11:42:10	-0.1	123.0	-0.2	981.4	-0.1	0.8
11:42:20	-0.2	120.7	-0.3	981.4	-0.1	0.8
11:42:30	0.1	165.7	-0.4	981.5	-0.1	0.7
11:42:40	0.3	266.9	0.2	982.[illegible]	-0.1	6.3
11:42:50	0.8	318.3	14.6	982.4	1.0	57.2
11:43:50	9.0	412.3	119.5	818.3	6.3	127.2
11:44:50	10.8	512.3	143.5	243.2	7.1	133.4
11:45:50	10.9	524.2	145.6	248.4	7.0	134.2
11:46:50	11.2	511.5	138.6	158.1	6.8	130.0
11:47:49	11.3	533.4	136.2	133.1	6.9	132.7
11:48:49	11.2	529.8	135.3	106.0	7.0	135.3
11:49:49	11.0	520.3	132.3	74.2	7.0	136.2
11:50:49	10.8	530.9	135.2	63.7	7.3	139.0
11:51:49	10.5	536.2	138.1	53.4	7.4	144.3
11:52:49	10.3	534.7	140.4	55.0	7.5	142.8
11:53:49	10.2	546.1	142.9	54.8	7.5	141.9

END CEMS HERE
ADJ. AVGS. (RB)

SOME FORM
OF SYSTEM UPSET

$\frac{(981.4 \times 2) - 2010}{500} =$

2010ppm CO Input

$\frac{\overset{1962.8\,ppm}{(981.4 \times 2)}_{ppm\ meter} - 2010\,ppm}{5000\,span} = \frac{47.2}{5000}$

= .0094 = .94%

OFFICIAL START OF
ALDEHYDE #4
CO's HIGH UNTIL 1149. (RB)

09-25-1991	O2	THC	SO2	CO	CO2	NOX
Time	%	PPMV	PPMV	PPMV	%	PPMV
11:55:49	10.5	538.3	145.7	59.3	7.4	140.0
11:56:49	10.4	544.9	147.6	77.5	7.4	141.7
11:57:49	10.5	548.3	147.9	80.7	7.4	140.7
11:58:49	10.8	543.4	143.7	75.1	7.2	136.1
11:59:49	10.8	542.4	144.1	66.1	7.2	137.5
12:00:48	10.7	533.8	144.6	62.4	7.2	135.0
12:01:48	10.9	548.4	145.1	65.3	7.2	135.1
12:02:48	10.9	563.8	143.9	87.4	7.1	131.5
12:03:48	10.8	564.0	151.3	125.1	7.2	137.4
12:04:48	10.7	570.1	153.9	130.5	7.3	140.1
12:05:48	10.5	607.6	157.1	165.2	7.4	142.1
12:06:48	10.3	598.3	160.3	184.0	7.5	142.7
12:07:48	10.2	595.4	158.6	145.0	7.5	143.2
12:08:48	10.4	597.6	155.4	125.6	7.5	141.8
12:09:48	10.7	605.4	152.3	118.8	7.4	141.6
12:10:48	10.8	617.1	149.3	106.8	7.2	140.2
12:11:48	10.8	588.1	150.6	108.6	7.2	141.3
12:12:48	10.8	568.5	148.6	94.5	7.2	142.3
12:13:48	10.8	553.8	147.6	77.9	7.2	143.1
12:14:47	10.8	554.4	147.8	73.8	7.1	142.9
12:15:47	10.8	537.4	147.1	61.6	7.2	143.0
12:16:47	10.9	526.0	141.0	62.4	7.2	138.2
12:17:47	10.9	550.8	139.3	64.9	7.1	137.6
12:18:47	10.9	564.4	143.8	65.6	7.1	137.7
12:19:47	10.8	563.7	143.0	63.5	7.2	138.8
12:20:47	10.9	554.5	142.9	59.6	7.1	136.8
12:21:47	11.1	552.6	135.2	56.7	6.7	124.4
12:22:47	12.1	536.0	120.5	50.6	6.2	110.5
12:23:47	12.3	531.4	122.4	67.0	6.2	108.2
12:24:47	12.3	541.9	124.3	85.9	6.1	108.6
12:25:47	12.4	536.1	123.5	75.0	6.0	108.6
12:26:47	12.4	539.0	121.3	59.8	6.1	109.5
12:27:46	11.9	532.5	129.9	62.3	6.4	124.0
12:28:46	11.6	535.4	134.3	72.4	6.5	126.1
12:29:46	11.4	539.0	137.5	80.2	6.7	131.3
12:30:46	11.3	536.6	138.5	89.7	6.7	130.9
12:31:46	11.1	568.4	140.9	108.0	6.7	131.5
12:32:46	11.1	564.7	142.0	129.4	6.7	131.7
12:33:46	11.1	546.6	139.5	89.2	6.7	131.8
12:34:46	11.2	531.6	135.4	76.5	6.5	130.0
12:35:46	11.2	528.0	135.2	74.0	6.4	129.0
12:36:46	11.4	511.7	129.9	62.7	6.3	128.4
12:37:46	11.3	508.7	129.4	64.5	5.7	129.4
12:38:46	11.4	509.7	128.6	69.8	5.0	128.2
12:39:46	11.3	517.7	128.6	63.8	4.8	129.8
12:40:45	11.4	507.1	129.6	63.8	4.8	129.3
12:41:45	11.5	513.8	128.9	61.6	4.7	126.8
12:42:45	11.4	519.0	130.0	55.2	4.8	129.7
12:43:45	11.2	514.1	131.2	52.1	4.8	132.5
12:44:45	11.0	520.5	132.1	46.7	4.9	134.7
12:45:45	11.0	530.6	132.8	44.5	4.8	134.3
12:46:45	11.0	517.9	133.2	42.8	4.8	133.0
12:47:45	11.1	512.8	130.7	47.8	4.9	135.1
12:48:45	11.1	518.9	129.9	54.0	4.7	132.1
12:49:45	11.3	509.0	126.4	71.6	4.6	127.0

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
12:51:45	11.7	492.4	123.6	110.5	4.5	123.8
12:52:45	11.7	493.8	125.0	104.1	4.5	123.3
12:53:44	11.7	493.6	124.2	100.2	4.5	121.0
12:54:44	11.8	490.1	122.7	96.2	4.5	122.7
12:55:44	11.6	496.7	124.8	102.9	4.5	123.7
12:56:44	11.5	496.3	125.5	91.8	4.5	125.5
12:57:44	11.5	500.2	128.0	90.0	4.7	129.2
12:58:44	11.3	518.9	130.4	85.6	4.8	132.2
12:59:44	11.3	513.3	129.1	78.4	4.7	128.3
13:00:44	11.3	510.0	129.4	88.5	4.7	129.2
13:01:44	11.5	494.7	127.6	93.3	4.7	127.3
13:02:44	11.6	491.9	124.5	94.0	4.5	124.8
13:03:44	11.6	494.3	123.2	89.2	4.4	120.7
13:04:44	11.7	496.3	121.4	85.7	4.6	123.2
13:05:44	11.7	499.2	121.5	83.0	4.5	121.6
13:06:43	12.2	492.8	111.2	81.7	4.1	110.2
13:07:43	11.7	494.3	123.3	72.9	4.6	128.2
13:08:43	11.2	493.1	128.6	72.4	4.8	133.8
13:09:43	11.0	492.1	129.6	69.0	4.8	135.2
13:10:43	10.9	492.7	130.2	53.2	4.8	136.2
13:11:43	10.8	504.3	132.3	51.7	5.0	138.3
13:12:43	10.9	496.6	132.5	59.0	4.8	135.5
13:13:43	11.1	506.2	133.0	68.4	4.7	132.6
13:14:43	11.2	497.8	132.2	69.5	4.7	130.9
13:15:43	11.3	485.7	132.3	85.7	4.7	132.4
13:16:43	11.3	474.7	131.0	81.4	4.6	129.4
13:17:43	11.3	475.6	131.1	74.0	4.6	129.2
13:18:43	11.4	471.9	129.2	74.0	4.6	128.6
13:19:42	11.4	467.1	129.1	75.0	4.6	128.9
13:20:42	11.2	470.7	132.6	67.1	4.6	133.6
13:21:42	11.0	472.7	133.7	55.7	4.8	137.2
13:22:42	10.8	473.4	136.3	55.1	4.9	139.8
13:23:42	10.8	496.9	137.6	61.6	4.9	141.3
13:24:42	10.6	511.9	138.7	50.5	5.0	141.8
13:25:42	10.8	512.4	137.6	45.8	4.8	138.0
13:26:42	10.8	501.2	137.5	50.1	4.9	140.7
13:27:42	10.8	498.1	137.8	48.4	4.8	142.2
13:28:42	10.8	493.7	138.6	46.8	4.8	141.6
13:29:42	10.9	485.2	135.6	49.3	4.8	139.2
13:30:42	10.8	481.4	134.2	45.6	4.8	140.0
13:31:42	10.9	477.3	135.1	48.1	4.7	138.8
13:32:41	10.9	476.2	135.2	51.1	4.7	139.5
13:33:41	10.8	486.5	136.6	46.1	4.9	142.9
13:34:41	10.7	508.9	138.6	43.5	5.0	146.1
13:35:41	10.4	522.8	140.5	42.4	5.0	147.0
13:36:41	10.4	532.3	140.5	42.5	4.9	146.1
13:37:41	10.3	524.5	140.8	43.3	5.0	148.8
13:38:41	10.4	530.7	141.1	43.4	4.9	145.9
13:39:41	10.5	517.6	140.8	43.6	4.8	144.0
13:40:41	10.7	507.3	139.3	43.2	4.8	143.6
13:41:41	10.8	498.4	136.7	46.6	4.7	142.9
13:42:41	10.8	505.2	137.1	45.8	4.7	144.5
13:43:41	10.8	497.2	135.5	46.3	4.7	143.4
13:44:41	10.8	484.0	133.0	45.7	4.8	142.3
13:45:40	10.9	497.9	133.0	47.1	4.7	140.7

END ALDEHYDE #4 PB

START PM_{10} #3

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
13:47:40	10.6	513.5	134.9	46.1	4.9	145.7
13:48:40	10.5	520.2	136.4	44.4	4.9	146.9
13:49:40	10.4	508.4	137.6	43.9	4.8	146.7
13:50:40	10.4	504.1	139.3	44.0	4.8	148.1
13:51:40	10.6	503.1	136.7	48.1	4.8	145.5
13:52:40	10.5	488.5	137.5	47.3	4.8	144.9
13:53:40	10.7	512.1	134.9	50.8	4.7	142.9
13:54:40	10.8	511.0	135.9	54.0	4.7	142.2
13:55:40	11.0	503.5	134.4	56.1	4.7	141.0
13:56:40	11.2	505.9	130.7	74.0	4.5	131.7
13:57:40	11.0	489.7	132.0	74.1	4.6	136.2
13:58:39	11.1	497.5	133.6	63.7	4.6	137.1
13:59:39	11.1	495.6	135.3	68.2	4.6	137.6
14:00:39	10.8	500.6	135.4	60.3	4.7	138.8
14:01:39	10.7	504.7	139.6	53.5	4.7	143.4
14:02:39	10.5	504.3	141.4	50.1	4.9	146.3
14:03:39	10.4	506.5	143.7	48.9	4.9	144.8
14:04:39	10.9	497.6	135.5	70.9	4.6	135.9
14:05:39	10.8	493.9	137.6	66.3	4.7	139.4
14:06:39	11.0	485.4	135.7	61.9	4.6	135.4
14:07:39	11.0	493.8	133.7	66.3	4.6	134.6
14:08:39	11.1	490.1	135.1	64.9	4.5	132.4
14:09:39	11.8	464.6	128.5	76.7	4.2	114.4
14:10:39	17.1	382.2	58.7	92.2	1.8	46.0
14:11:39	17.1	302.6	36.6	109.7	1.8	46.6
14:12:38	20.8	191.0	12.8	41.0	-0.1	5.4
14:13:38	21.3	147.4	4.2	3.9	-0.3	2.3
14:14:38	21.4	127.2	2.1	1.4	-0.3	1.4
14:15:38	21.5	116.9	1.2	-0.2	-0.3	1.1
14:16:38	21.4	112.0	0.7	-1.1	-0.3	1.2
14:17:38	21.5	108.0	0.5	-1.1	-0.3	1.0
14:18:38	21.3	101.8	0.2	-1.1	-0.4	0.9
14:19:38	21.4	94.7	0.2	-1.2	-0.3	1.0
14:20:38	21.4	87.7	0.2	0.1	-0.3	1.0
14:21:38	21.3	82.2	0.6	1.2	-0.3	0.9
14:22:38	21.3	77.2	0.5	1.5	-0.3	0.9
14:23:38	21.3	72.3	0.3	1.2	-0.3	0.8
14:24:38	21.3	69.2	0.2	1.2	-0.3	1.0
14:25:37	21.3	70.3	0.1	1.0	-0.3	1.0
14:26:37	21.2	68.7	-0.1	1.1	-0.3	0.7
14:27:37	21.3	75.4	-0.4	0.9	-0.3	0.6
14:28:37	20.7	131.3	8.1	76.6	0.1	6.7
14:29:37	18.8	139.3	36.6	230.4	1.0	22.1
14:30:37	19.1	170.5	30.6	148.0	1.0	35.7
14:31:37	12.2	279.0	93.8	144.9	3.8	120.4
14:32:37	11.8	316.9	118.0	73.2	4.0	122.9
14:33:37	11.4	355.7	121.2	67.2	4.2	131.8
14:34:37	11.1	379.8	127.0	58.3	4.3	135.3
14:35:37	11.0	407.3	129.8	57.3	4.4	137.8
14:36:37	10.9	436.5	132.6	57.2	4.4	138.6
14:37:37	10.9	446.4	134.8	55.9	4.4	139.7
14:38:36	10.8	447.5	133.8	54.0	4.4	140.4
14:39:36	10.8	456.1	136.6	54.1	4.5	141.8
14:40:36	10.8	450.7	138.1	50.8	4.5	142.1
14:41:36	10.9	454.6	136.1	56.6	4.4	138.9

END PM10 #3

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
14:43:36	10.9	474.2	140.3	62.9	4.4	140.9
14:44:36	10.8	481.4	143.1	56.7	4.5	144.1
14:45:36	10.8	495.3	141.0	53.5	4.5	139.6
14:46:36	10.6	499.8	143.5	48.0	4.5	141.3
14:47:36	10.8	513.0	142.5	49.7	4.5	140.6
14:48:36	10.4	515.5	146.1	50.9	4.6	145.0
14:49:36	10.9	504.8	141.8	52.7	4.3	137.1
14:50:36	10.9	498.6	141.0	59.1	4.3	137.8
14:51:35	11.2	485.0	137.5	71.3	4.3	134.7
14:52:35	11.3	493.9	135.0	69.6	4.2	132.8
14:53:35	11.2	501.1	134.9	67.3	4.2	131.8
14:54:35	11.7	485.6	128.0	86.0	4.0	121.2
14:55:35	12.2	476.8	117.7	118.3	3.9	115.9
14:56:35	12.2	468.5	114.2	110.9	3.8	117.8
14:57:35	11.6	477.4	120.6	66.5	4.0	124.6
14:58:35	11.3	489.4	126.4	46.4	4.1	129.8
14:59:35	10.8	496.7	132.3	53.6	4.3	133.2
15:00:35	10.8	510.2	132.5	64.9	4.2	128.5
15:01:35	10.7	528.4	136.7	69.8	4.2	134.0
15:02:35	10.7	544.8	138.4	61.7	4.2	135.5
15:03:35	11.1	534.4	132.2	56.7	3.9	129.5
15:04:34	10.8	514.0	135.4	58.5	3.8	134.2
15:05:34	11.0	515.0	130.8	56.0	3.6	125.8
15:06:34	11.1	521.6	129.8	62.3	3.6	129.5
15:07:34	11.0	527.3	132.7	58.3	3.5	132.3
15:08:34	11.0	509.5	132.4	50.9	3.8	132.3
15:09:34	11.2	511.6	130.1	42.9	3.8	131.7
15:10:34	10.9	511.8	131.3	42.4	3.9	134.0
15:11:34	10.9	526.9	131.6	40.2	3.8	129.9
15:12:34	10.8	527.7	132.6	38.5	3.9	133.8
15:13:34	10.7	543.1	136.2	36.3	3.9	136.1
15:14:34	10.7	563.7	133.7	36.5	3.8	131.8
15:15:34	10.8	562.8	130.2	37.6	3.7	127.2
15:16:34	10.9	537.6	130.1	36.0	3.7	132.0
15:17:33	11.0	532.8	127.3	38.0	3.6	131.2
15:18:33	11.0	516.2	127.4	39.6	3.5	131.7
15:19:33	11.0	517.5	128.1	39.7	3.5	131.4
15:20:33	11.7	511.1	[illegible]	42.3	3.2	122.4
15:21:33	10.9	509.8	134.8	48.3	3.5	141.6
15:22:33	10.8	495.0	[illegible]	57.4	4.1	141.5
15:23:33	11.0	476.5	133.5	67.7	4.0	132.9
15:24:33	11.5	456.6	126.8	89.9	3.9	126.2
15:25:33	11.6	455.9	128.4	101.1	3.8	122.3
15:26:33	11.6	459.6	127.9	112.2	3.9	124.2
15:27:33	11.7	458.3	125.8	111.3	3.9	120.5
15:28:33	11.9	454.8	125.4	132.4	3.8	117.4
15:29:33	12.3	460.7	124.6	146.0	3.7	110.7
15:30:32	12.9	438.4	115.4	232.2	3.4	101.1
15:31:32	12.9	429.6	112.6	221.5	3.4	101.6
15:32:32	12.9	412.8	109.5	233.3	3.4	101.7
15:33:32	12.7	409.9	112.0	177.5	3.5	106.7
15:34:32	12.8	409.8	114.2	172.3	3.4	104.4
15:35:32	12.8	407.2	115.7	173.1	3.5	107.0
15:36:32	12.6	416.7	117.8	163.7	3.6	110.0
15:37:32	12.4	400.9	119.8	158.7	3.5	110.9

1455 Plant Back on-Line

END Testing

post Test QC

09-25-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
15:39:32	21.2	181.7	6.5	6.4	-0.3	2.7
15:40:32	21.3	147.6	3.0	3.1	-0.4	1.7
15:41:32	21.3	129.9	2.1	1.3	-0.4	1.4
15:42:32	21.2	118.5	1.7	0.5	-0.4	1.0
15:43:31	21.2	108.4	0.9	-0.6	-0.4	0.6
15:44:31	21.2	99.0	0.7	-1.1	-0.4	0.6
15:45:31	21.2	97.2	0.7	-0.6	-0.4	1.9
15:46:31	5.0	101.0	8.4	692.6	-0.4	6.5
15:47:31	0.6	95.7	-0.8	982.0	-0.5	0.7
15:48:31	0.3	90.3	3.2	578.1	-0.3	2.0
15:49:31	-0.5	78.4	-0.9	14.9	-0.4	0.0
15:50:31	-0.5	84.3	22.8	7.8	-0.4	42.5
15:51:31	-0.3	79.7	275.5	3.7	-0.5	207.8
15:51:41	-0.3	77.9	305.6	-0.1	-0.5	213.2
15:51:51	-0.3	77.9	308.1	0.3	-0.5	213.9
15:52:01	-0.3	70.9	308.5	-1.2	-0.5	214.0
15:52:11	-0.4	64.5	306.3	-0.5	-0.5	204.5
15:52:21	0.4	65.1	264.9	0.2	-0.5	108.0
15:52:31	8.9	64.5	159.6	3.1	-0.4	35.7
15:52:41	16.5	64.1	82.1	3.2	-0.4	13.0
15:52:51	19.4	63.7	43.5	3.2	-0.4	5.8
15:53:01	20.3	63.6	25.6	3.0	-0.4	3.5
15:53:11	20.7	63.5	16.7	3.0	-0.4	2.2
15:53:20	20.9	65.1	11.5	2.9	-0.4	1.7
15:53:30	21.0	63.8	8.4	2.3	-0.4	1.2
15:53:40	21.0	63.3	7.2	3.1	-0.4	1.0
15:53:50	20.9	62.6	6.0	2.9	-0.4	0.8
15:54:00	21.1	61.3	4.6	2.2	-0.4	0.6
15:54:10	21.1	61.7	3.1	2.3	-0.4	0.5
15:54:20	21.1	67.9	3.6	2.2	-0.4	9.0
15:54:30	20.2	66.9	38.6	0.8	-0.4	107.1
15:55:00	11.6	66.3	130.7	-1.4	-0.5	175.4
15:55:10	1.4	64.7	271.2	-1.1	-0.5	197.2
15:55:20	1.1	64.8	283.0	-2.3	-0.5	198.1
15:55:30	1.0	64.4	287.9	-2.3	-0.5	198.9
15:55:40	0.9	63.4	290.1	-2.3	-0.5	199.9
15:55:50	0.8	62.6	292.3	-2.3	-0.5	200.4
15:56:00	0.8	62.1	293.7	-2.4	-0.5	200.7
15:56:10	0.8	63.0	[illegible]	-2.4	-0.5	201.1
15:56:20	-0.9	63.6	[illegible]	-2.3	-0.5	193.5
15:56:30	0.8	63.0	260.7	-1.3	-0.5	146.8
15:56:40	0.1	62.2	193.2	0.3	-0.5	118.1
15:56:50	-0.1	61.1	147.7	1.3	-0.5	109.5
15:56:59	-0.2	60.8	126.6	1.0	-0.5	106.7
15:57:09	-0.2	60.2	116.0	0.0	-0.5	105.7
15:57:19	-0.2	59.5	111.5	-0.7	-0.5	105.3
15:57:29	-0.2	59.2	108.9	-0.7	-0.5	105.0
15:57:39	-0.2	59.2	107.6	-0.7	-0.5	104.9
15:57:49	-0.3	59.0	107.0	-1.0	-0.5	104.5
15:57:59	-0.3	58.5	106.3	-1.3	-0.5	102.2
15:58:09	-0.3	58.3	105.5	-0.7	-0.5	103.5
15:58:19	-0.3	55.2	105.3	-0.9	-0.5	103.8
15:59:19	4.2	52.9	48.7	36.8	3.3	26.2
16:00:19	7.4	50.8	4.2	89.8	5.5	0.6
16:00:34	7.4	49.6	1.7	89.0	10.6	0.2

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09-25-1991      O2      THC     SO2      CO      CO2     NOX
Time            %       PPMV    PPMV     PPMV    %       PPMV
==================================================================================================
16:00:53        7.4     49.2     1.2     90.5    10.5     0.1
16:01:03        7.4     49.0     0.9     90.3    10.6     0.1
16:01:13        7.4     48.6     1.1     89.4    10.5     0.1
16:01:23        7.4     48.4     0.7     89.4    10.4     0.1
16:01:33        7.4     48.4     0.5     90.4    10.6     0.1
16:02:33       10.7     41.2     0.8    132.4    10.9     0.5
16:03:33       18.2     41.7     0.3    363.7    15.4     0.1
16:04:33       18.4     42.6    -0.3    397.4    15.5     0.0
16:04:55       18.5     42.5    -0.2    467.1    17.5     0.0
16:05:05       18.5     42.6    -0.4    467.6    17.7     0.0
16:05:15       18.5     43.1    -0.4    479.2    17.3     0.0
16:05:25       18.4     43.3    -0.2    448.6    17.6     0.0
16:05:35       18.5     43.4    -0.2    468.7    17.6     0.0
16:05:45       18.4     43.2    -0.2    461.8    17.4    -0.0
16:05:55       18.5     43.1    -0.3    457.0    17.6    -0.0
16:06:05       18.4     42.7    -0.4    538.4    17.9    -0.0
16:06:15       18.4     42.5    -0.5    414.6    17.8    -0.0
16:06:25       18.4     42.3    -0.5    493.4    17.0     0.0
16:06:35       18.5     41.9    -0.5    477.0    17.3     0.0
16:06:45       18.5     41.4    -0.7    464.4    17.3     0.0
16:06:54       18.4     40.7    -0.7    455.1    17.1     0.0
16:07:04       18.4     40.1    -0.6    463.4    17.2    -0.0
16:07:14       18.4     39.6    -0.6    471.7    17.2    -0.0
16:08:14       18.5     53.8    -0.7    464.2    17.1    -0.0
16:08:57       15.6    760.3     0.5    332.2    12.3     0.3
16:09:07        2.0    815.5     0.2     70.1     1.5    -0.0
16:09:17        1.5    816.6     0.4     36.1     0.7    -0.1
16:09:27        1.3    818.9     0.3     21.2     0.4    -0.0
16:09:37        1.2    817.5     0.4     15.4     0.2    -0.0
16:09:47        1.2    810.2     0.3     12.3     0.1    -0.1
16:09:57        0.7    419.3    -0.7      5.6    -0.1    -0.1
16:10:07        0.3    164.9    -0.9      2.6    -0.2    -0.1
16:10:17        0.3    148.6    -0.6      2.1    -0.2    -0.1
16:10:27        0.3    144.1    -0.6      2.1    -0.2    -0.1
16:10:37        0.3    141.3    -0.7      2.1    -0.2    -0.1
16:10:47        0.3    140.0    -0.4      1.7    -0.3    -0.1
16:10:57        0.3    139.1    -0.8      1.1    -0.3    -0.1
16:11:07        0.2    126.9    -0.6      1.1    -0.3    -0.1
16:11:17        0.1    104.8    -1.0      0.7    -0.3    -0.1
16:11:26       -0.1     97.6    -1.0     -0.1    -0.4    -0.1
16:11:36        0.1     97.6    -1.1     -0.1    -0.3    -0.1
16:11:46       -0.0     97.6    -0.6     -0.2    -0.3    -0.1
16:11:56       -0.0     97.4    -0.8     -0.1    -0.4    -0.1
16:12:06       -0.2     71.2    -0.6      1.0    -0.4    -0.1
16:12:16       -0.1      9.2    -0.9      2.1    -0.3    -0.1
16:12:26       -0.2      3.7    -0.8      3.0    -0.3    -0.1
16:12:36       -0.3      3.2    -0.8      2.1    -0.3    -0.1
16:12:46       -0.3      2.6    -0.7      3.1    -0.3    -0.1
16:12:56       -0.4      2.6    -0.4      2.1    -0.3    -0.1
16:13:06       -0.4      2.8    -0.5      2.1    -0.4    -0.1
==================================================================================================
Avg.=          11.6    346.7    95.9    133.3     4.4    90.2
==================================================================================================
```

7.99

9.9

O_2 / CO / CO_2

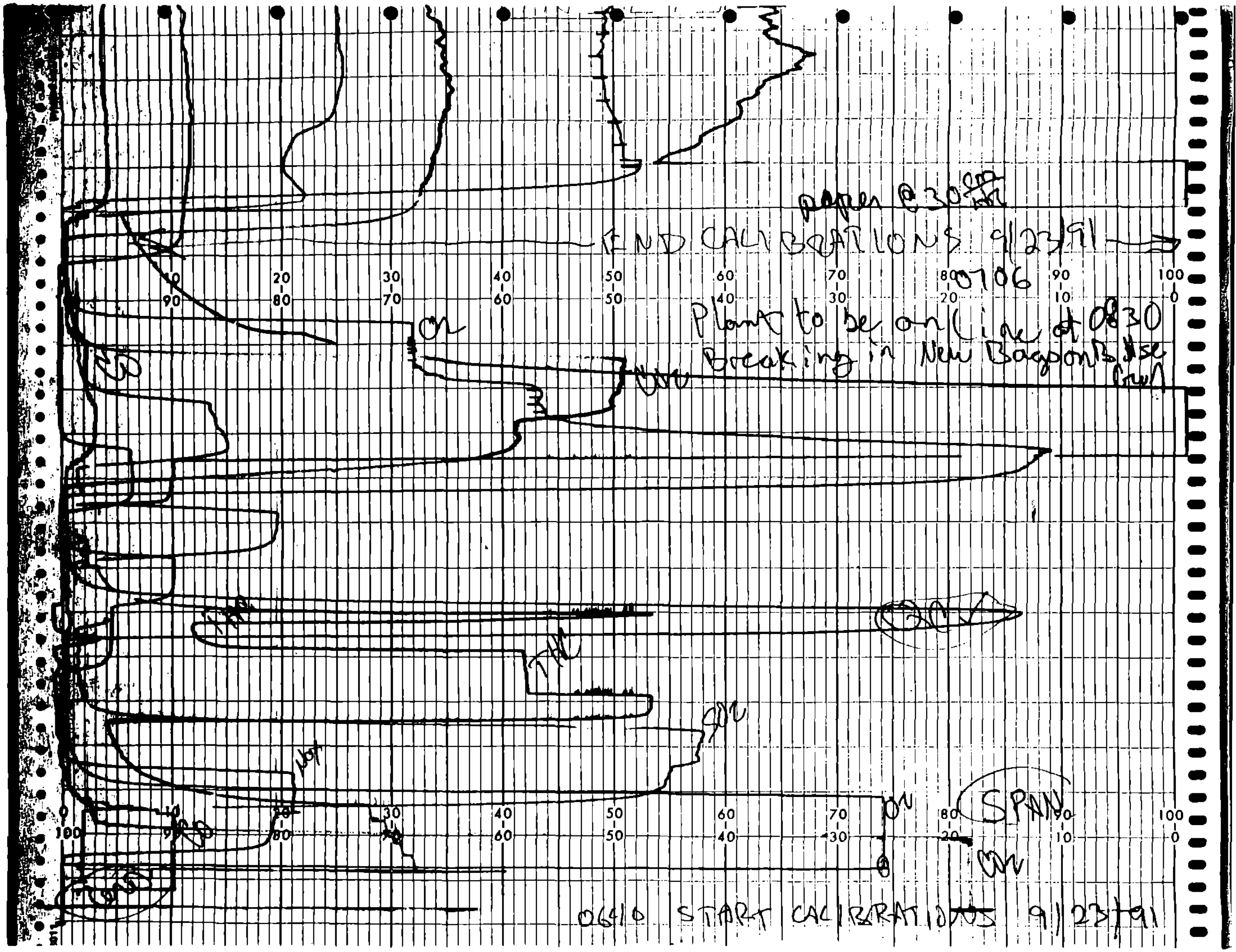
18 / 444 / 17

THC ≈ 810

95 THC

APPENDIX D.3

STRIPCHART TRACINGS



paper @ 30 cm/hr

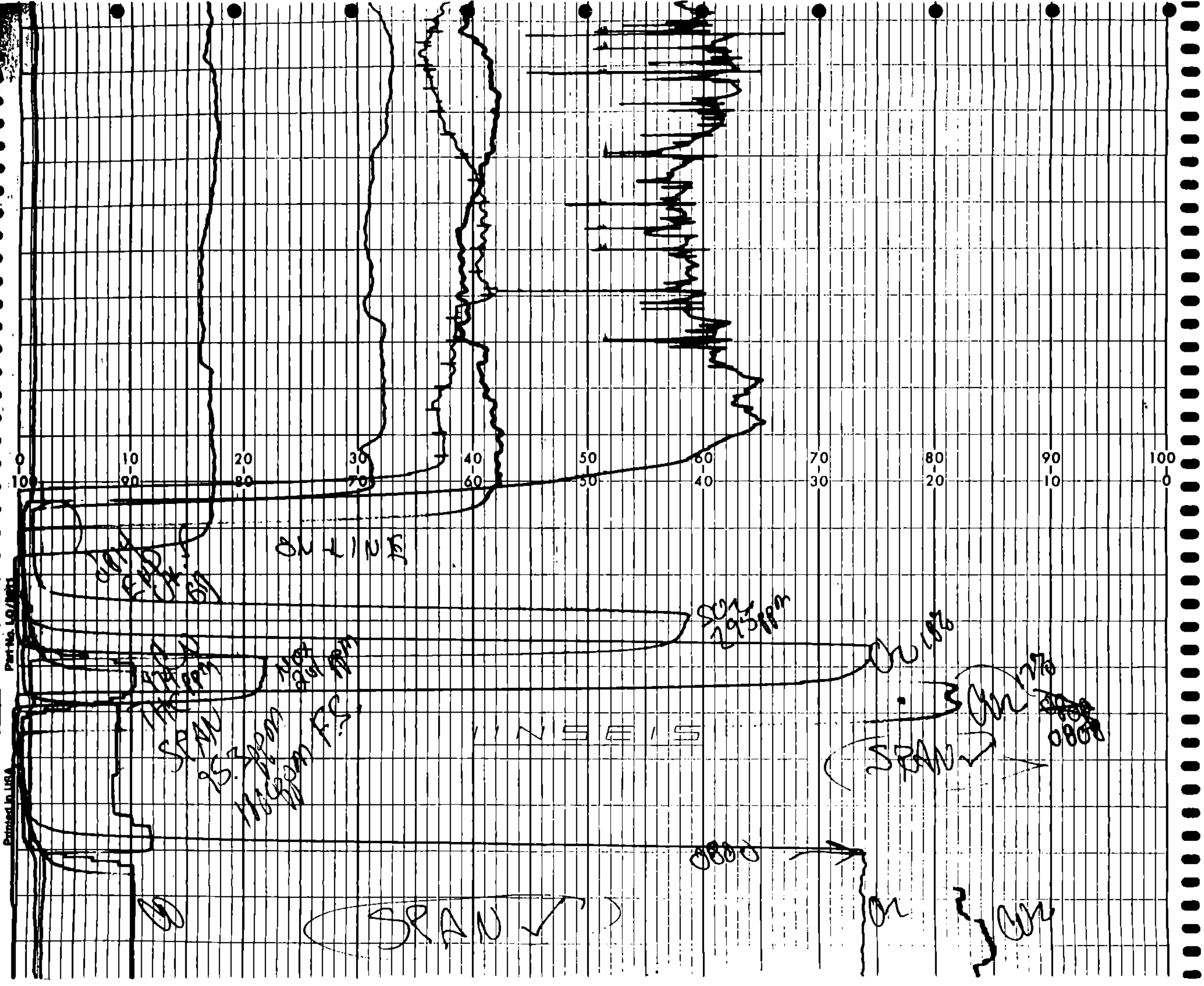
END CALIBRATIONS 9/23/91

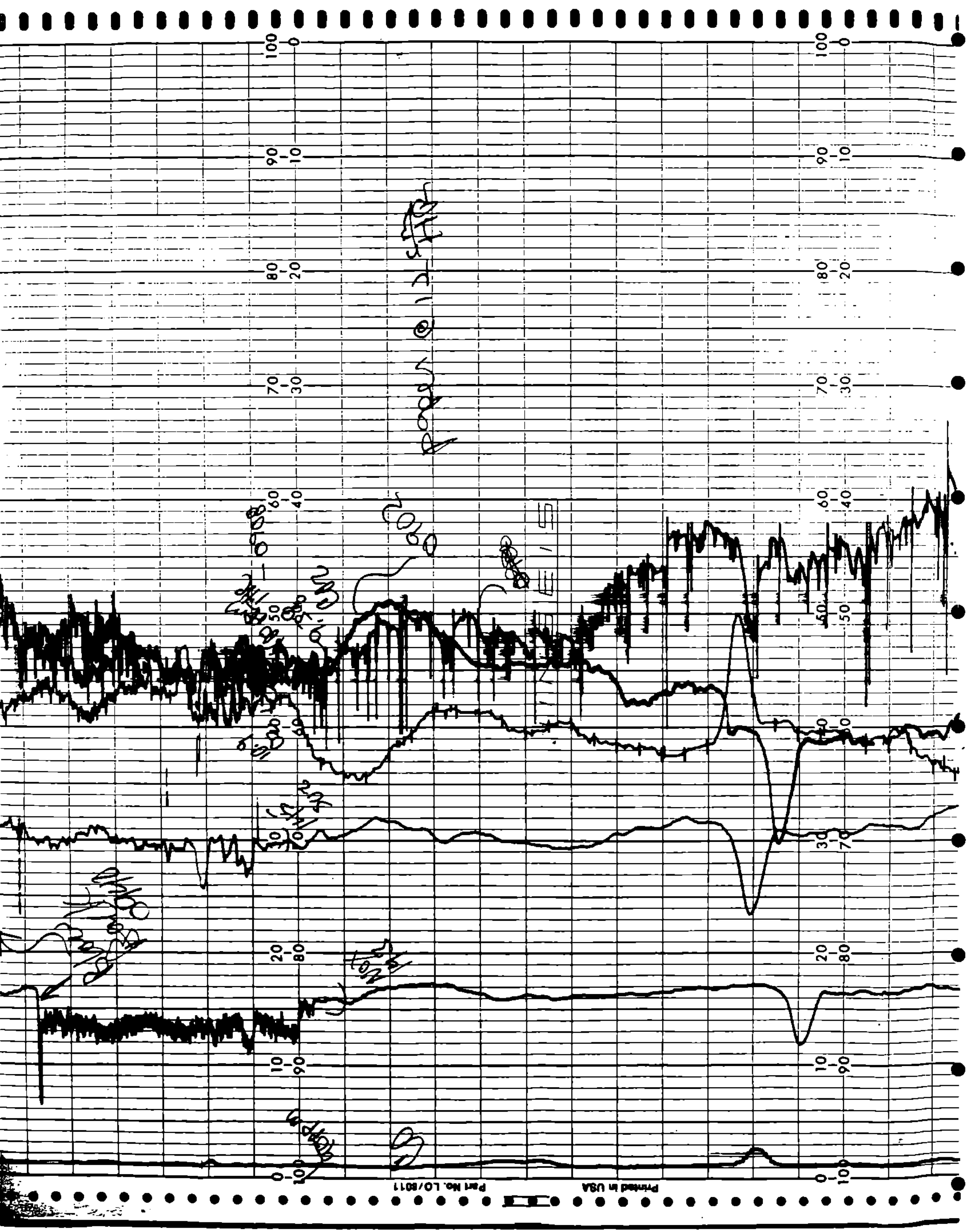
0706

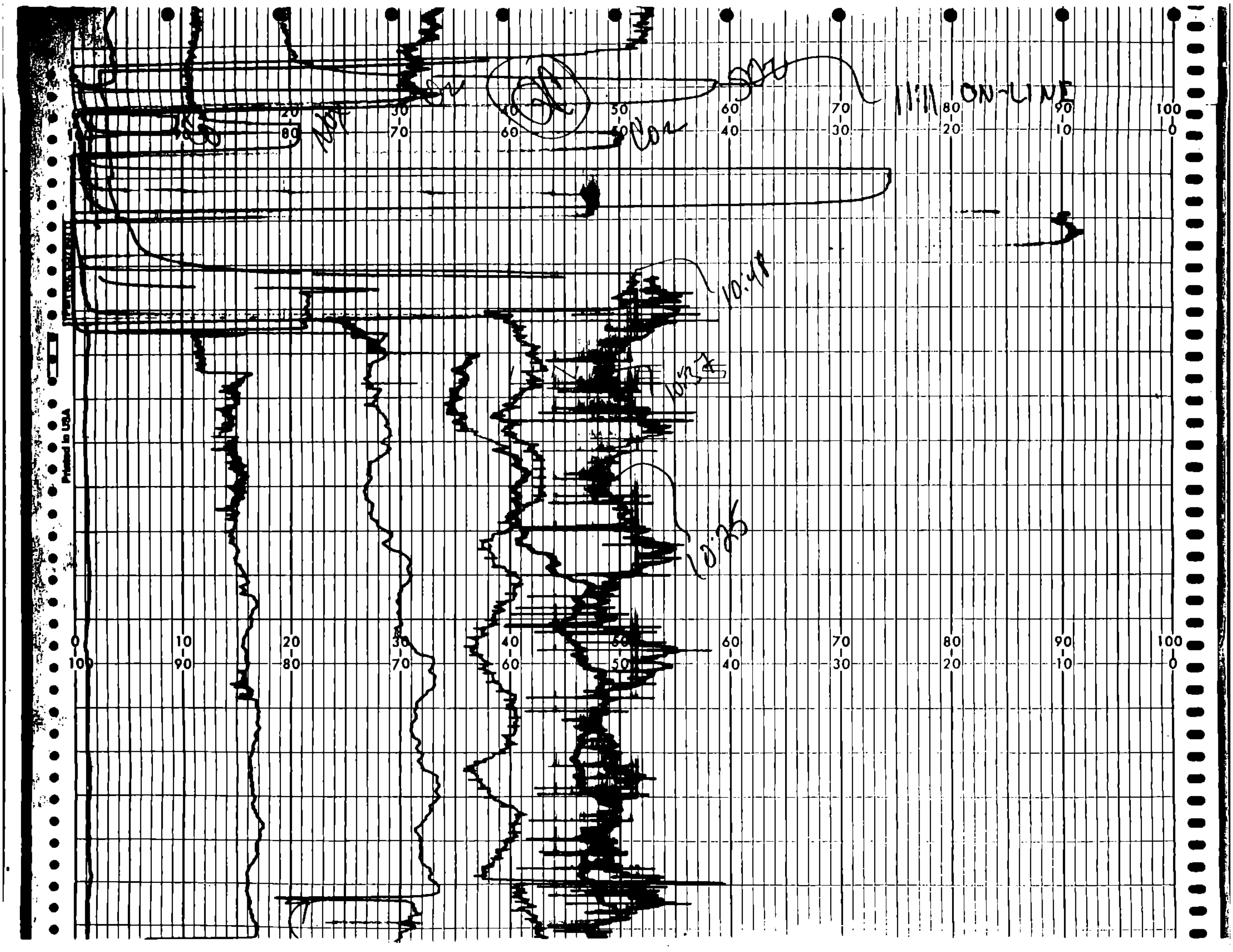
Plant to be on Line at 0830
Breaking in New Bags on B Hse
Crw

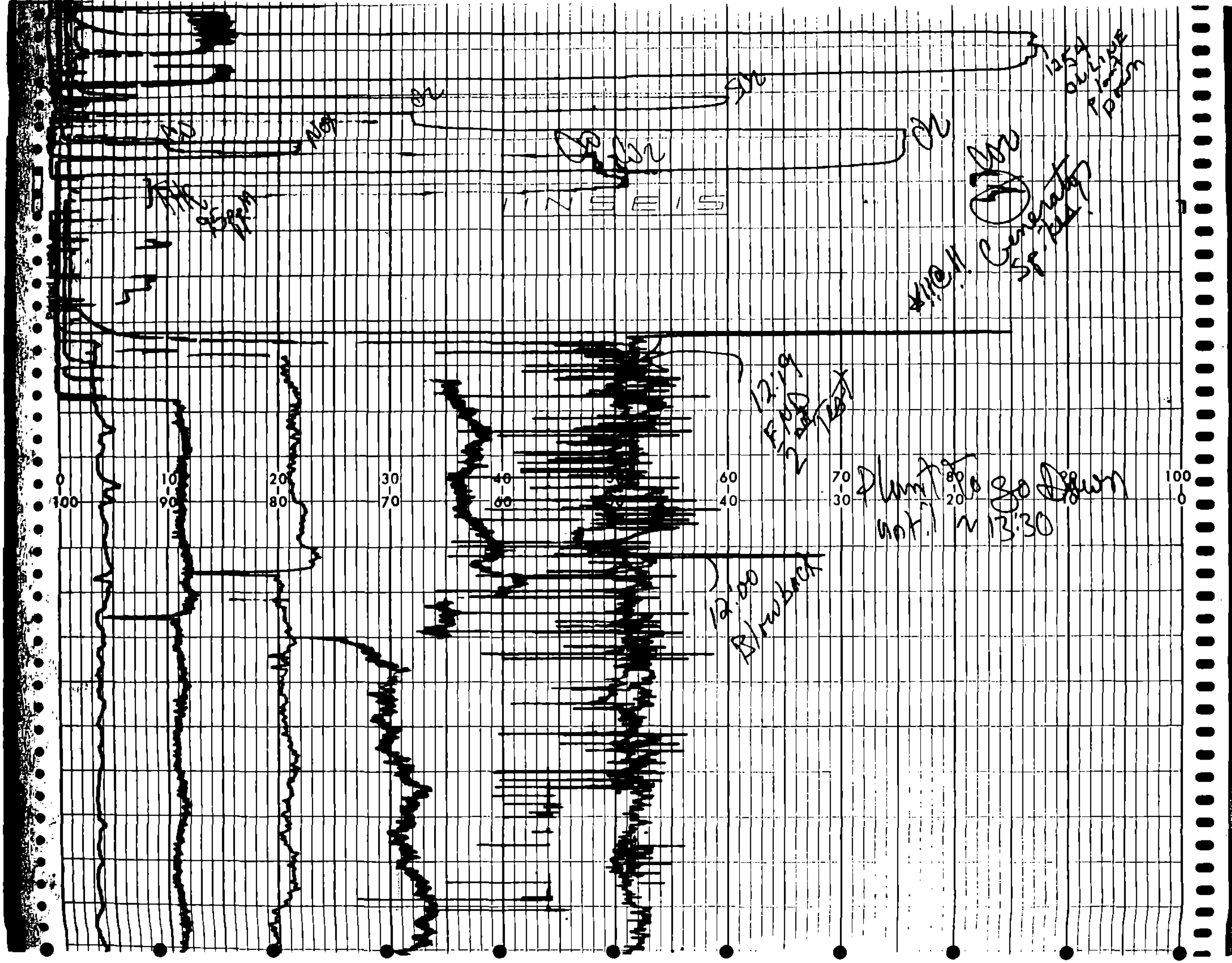
0610 START CALIBRATIONS 9/23/91

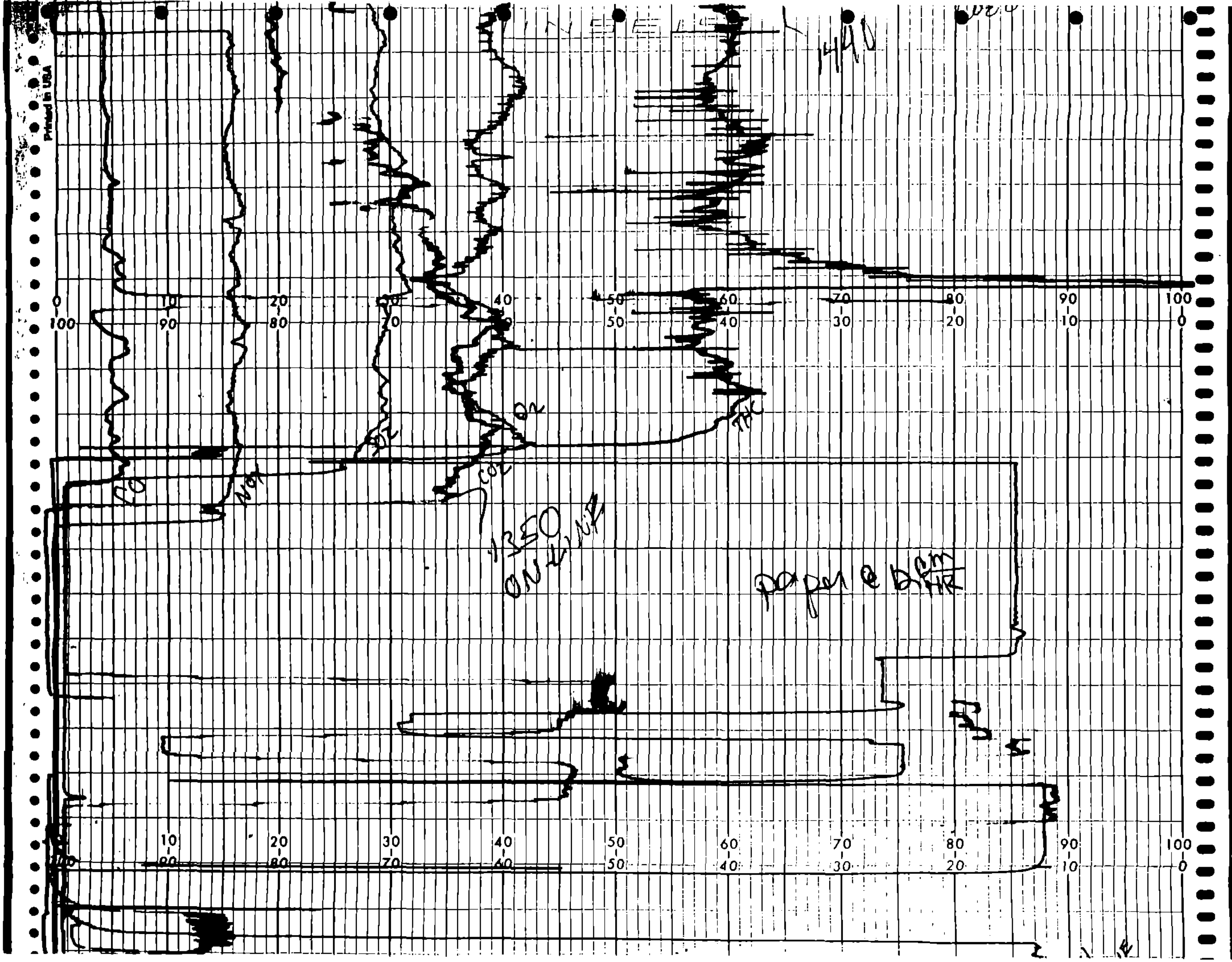

100 0
90 10
80 20
70 30
60 40
50 50
40 60
30 70
20 80
10 90
0 100
100 0
90 10
80 20
70 30
60 40
50 50
40 60
30 70
20 80
10 90
0 100
Part No. LO/8011
USA

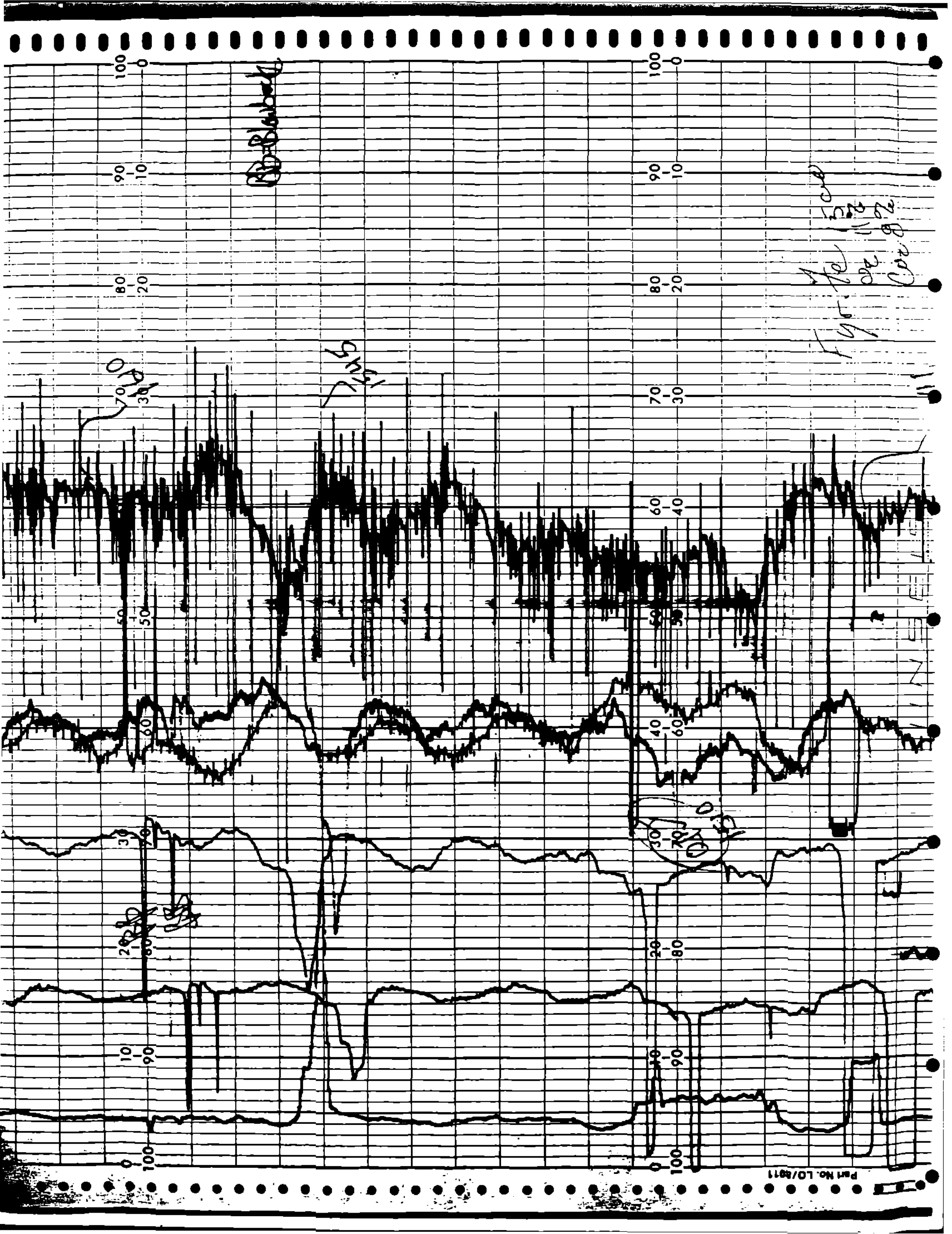

0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0
ON LINE
SO2 293ppm
O2 18%
Cm 17%
0800
SPAN ✓
NO 241 PPM
SPAN 253ppm
FS?
0800
SPAN ✓
ON
Cm
Part No. L0/1881
Printed in USA

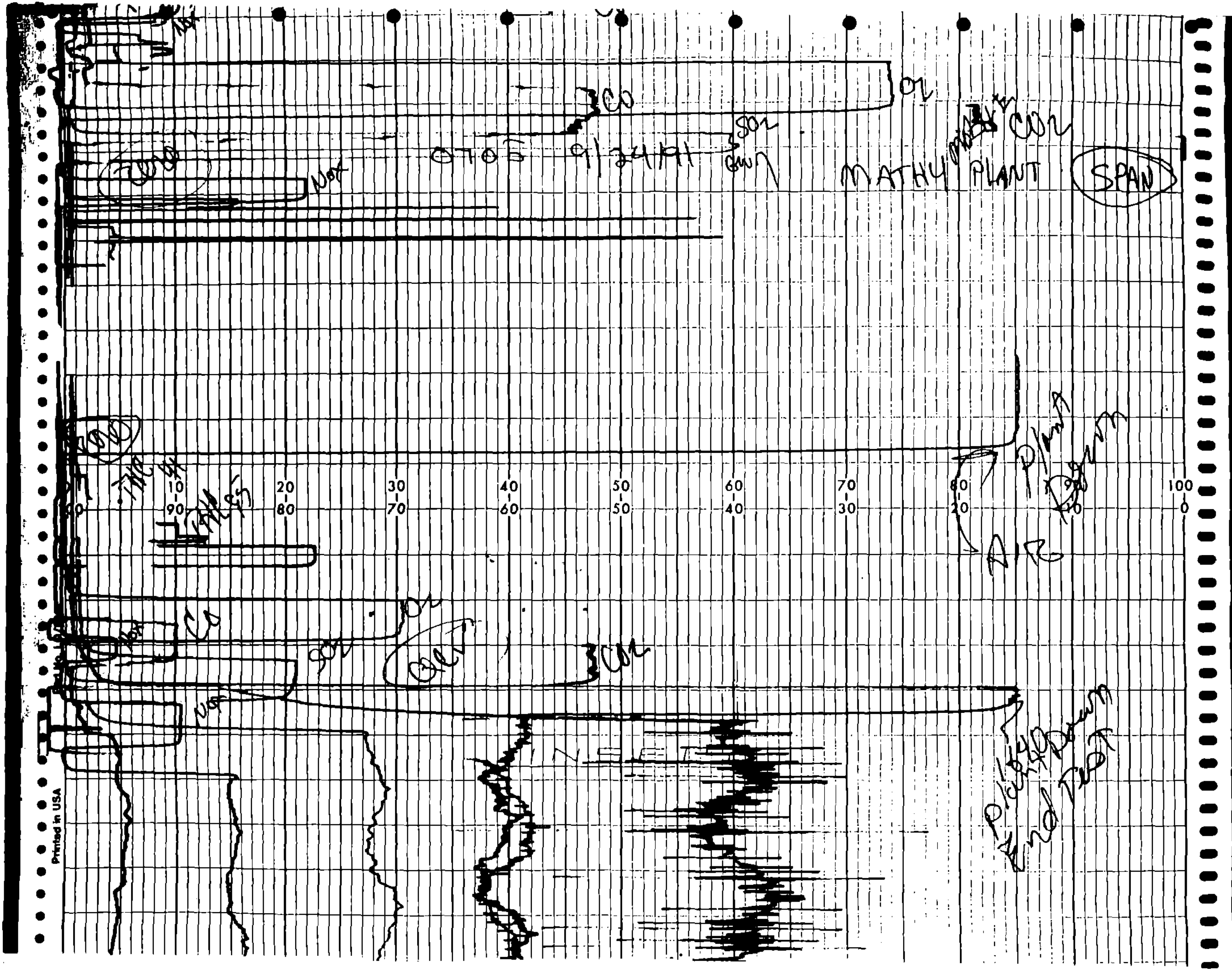

Part No. LO/8011
Printed in USA
0902
0908

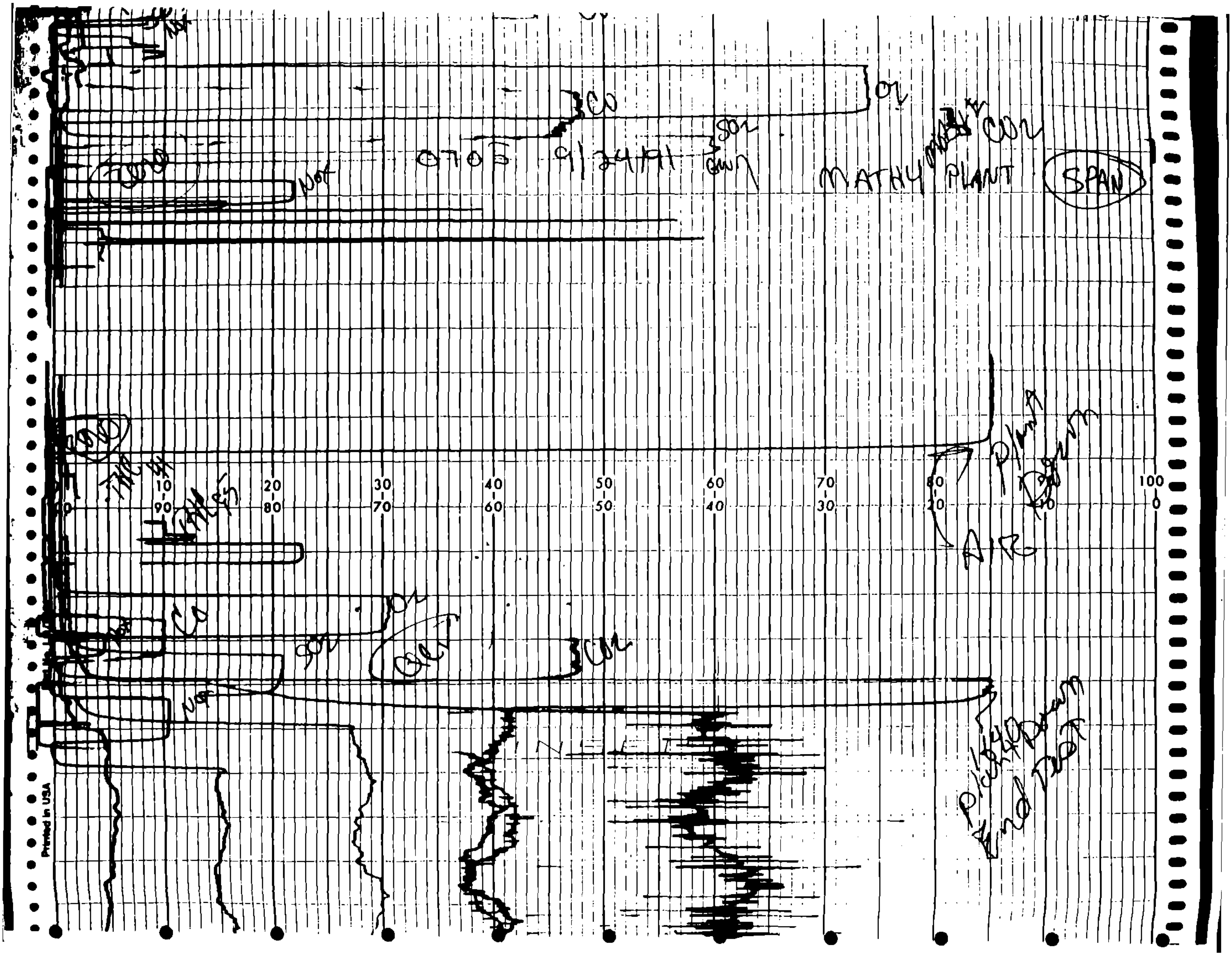

0
10
20
30
40
50
60
70
80
90
100
100
90
80
70
60
50
40
30
20
10
0
11:11 ON-LINE
10:48
10:37
10:25
50%
40%
30%
20%
Printed in USA

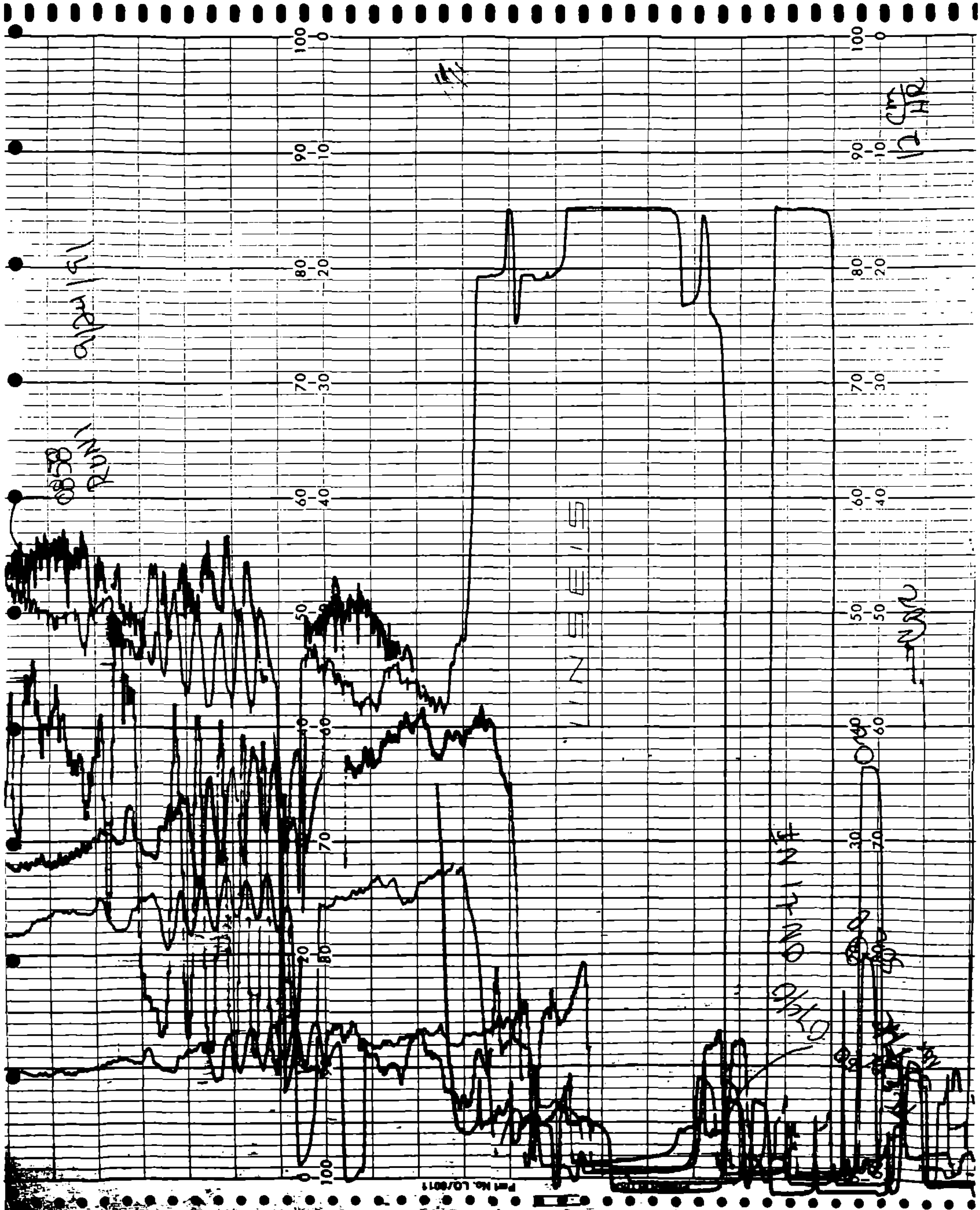

0 100
10 90
20 80
30 70
40 60
60 40
70 30
80 20
90 10
100 0
LINSEIS
O2
CO2
CO
NOx
THC
95 ppm
12:54
ON LINE
P Dwn
High Cal Generator
58 ppm
12:19
END 2nd TEST
12:00
Blowback
Plume To 80 Down
until ~13:30


Printed in USA
0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0
1440
CO
NOx
SO2
CO2
O2
THC
1350 ON LINE
90 ppm @ 6 cm/HR

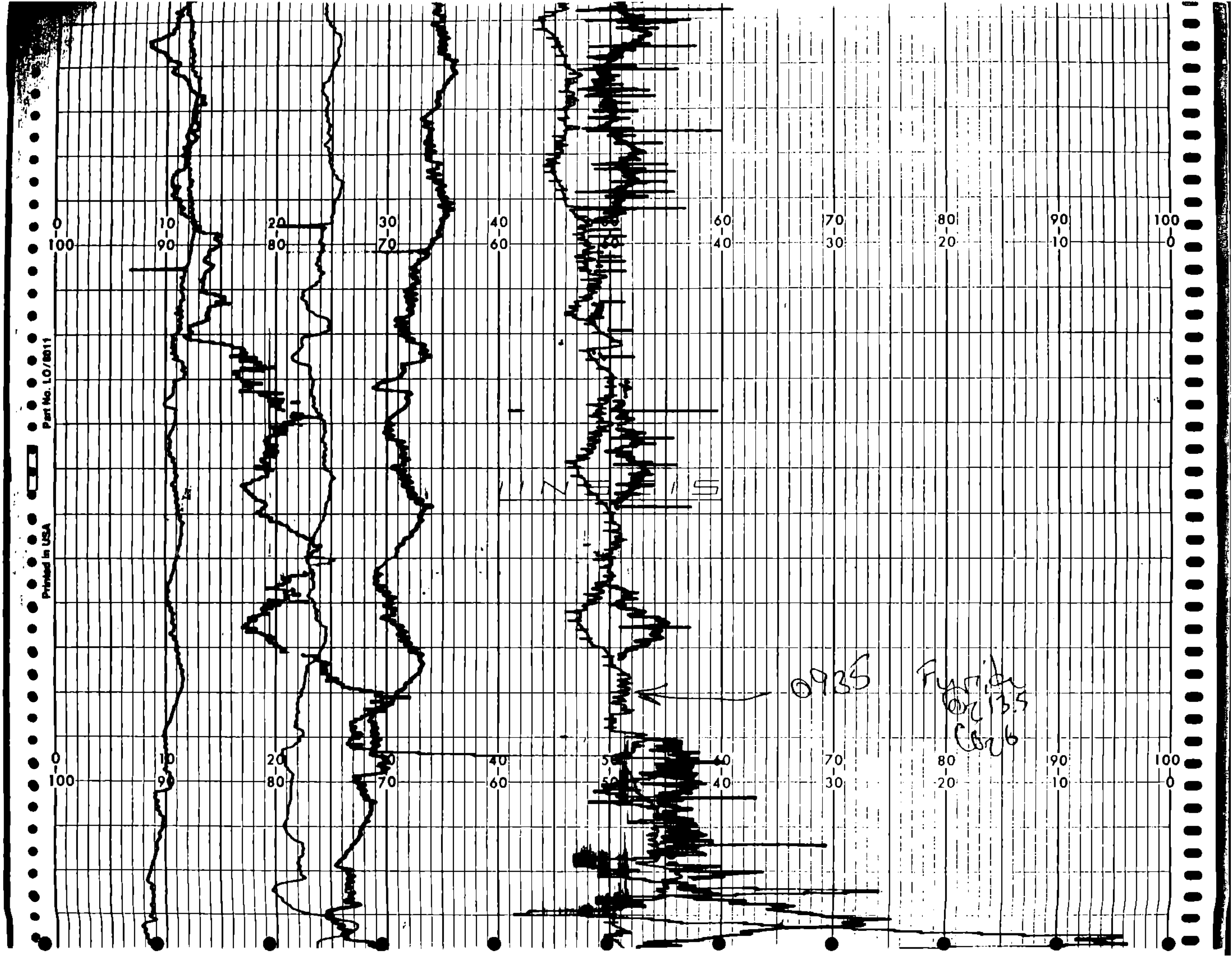

100 0
90 10
80 20
70 30
60 40
50 50
40 60
30 70
20 80
10 90
0 100
Rb-Blackwell
1540
1545
Fyr Ye 15cc
02 11/25
Cor 82
Part No. LO/8011

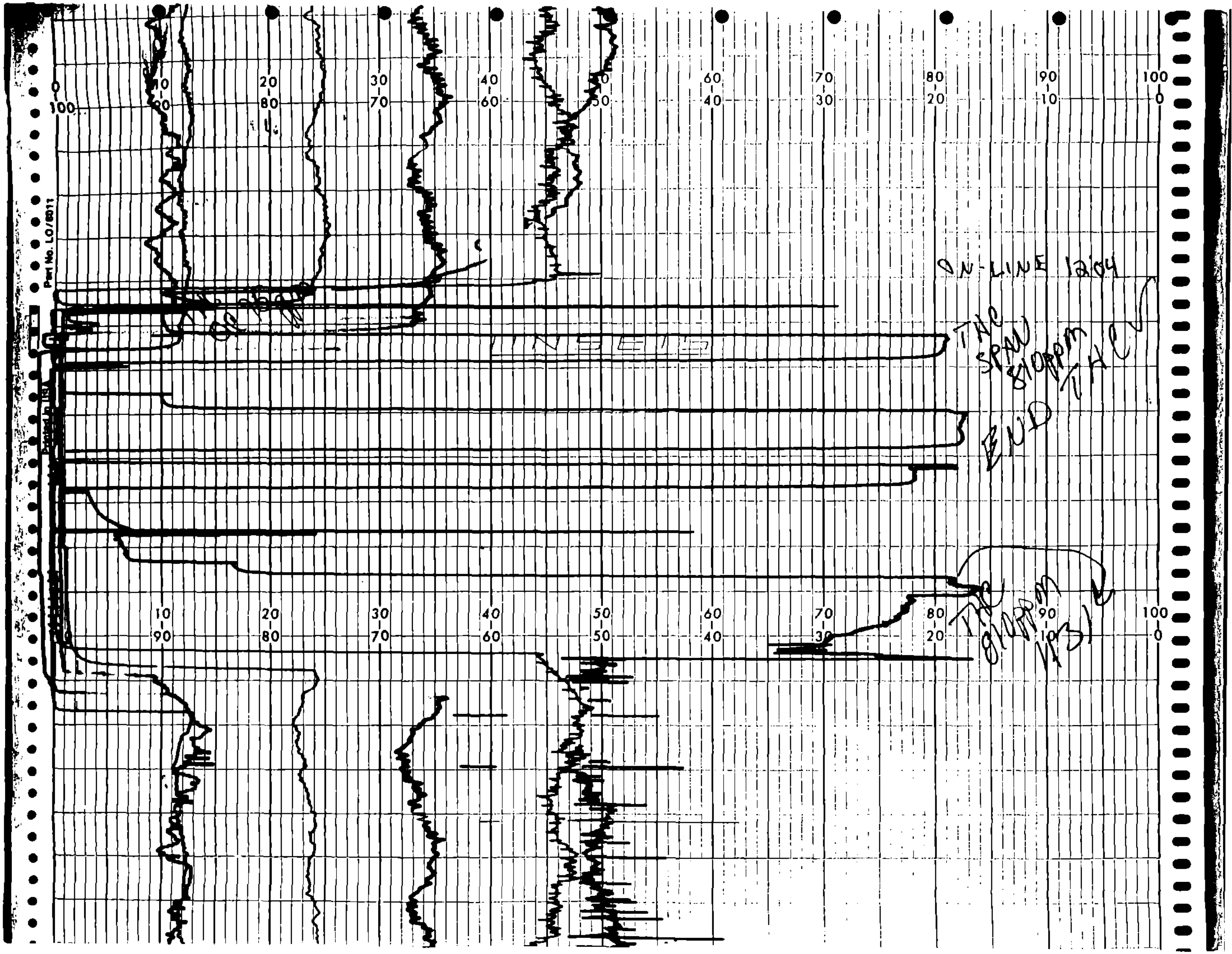

NOx
O2
CO
SO2
CO2
0705 9/24/91
MATHY PLANT
SPAN
ZERO
NOx
ZERO
THC
CALIBS
10 90
20 80
30 70
40 60
50 50
60 40
70 30
80 20
90 10
100 0
Plant Down
AIR
CO
O2
NOx
SO2
QCV
CO2
NOx
Plant 1440 Down
End Test
Printed in USA


O2
CO
SO2
CO2
0705 9/24/91 GWN
MATHY PLANT
SPAN
NOx
Zero
Plant Down
AIR
Zero
THC
CO
O2
SO2
CO2
NOx
Pick up and Test
100
0
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
Printed in USA



9/24/91
0858 RUN 1
12 cm/HR
0740 ON-LINE
LINSEIS
Part No. LO/8011
100 0
90 10
80 20
70 30
60 40
50 50
40 60
30 70
20 80
0 100


0
100
10
90
20
80
30
70
40
60
50
60
40
70
30
80
20
90
10
100
0
Printed in USA
Part No. LO/8011
0935
Fundus @ 13.5
Ca26

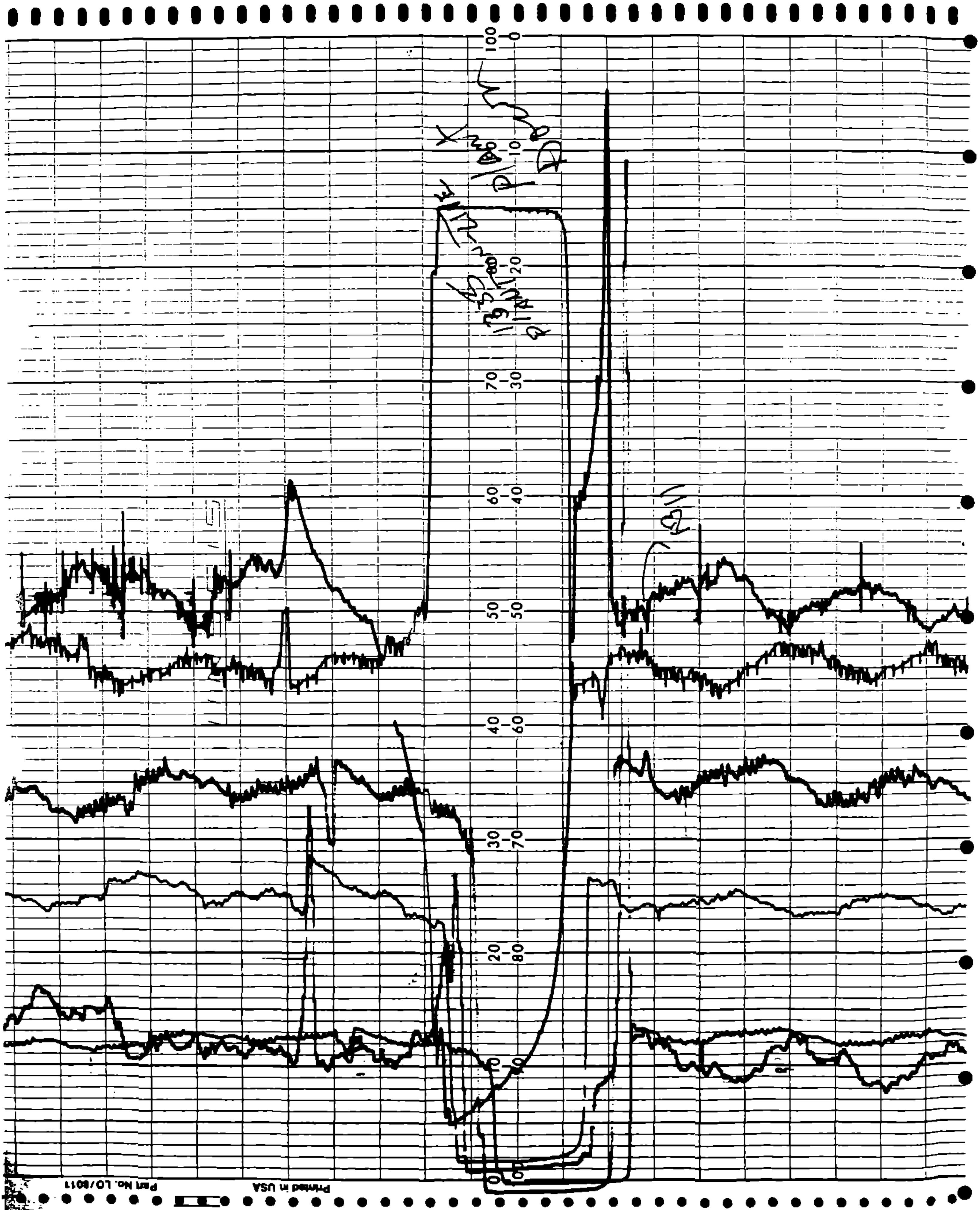


ON-LINE 12:04

THC SPAN 810PPM THC

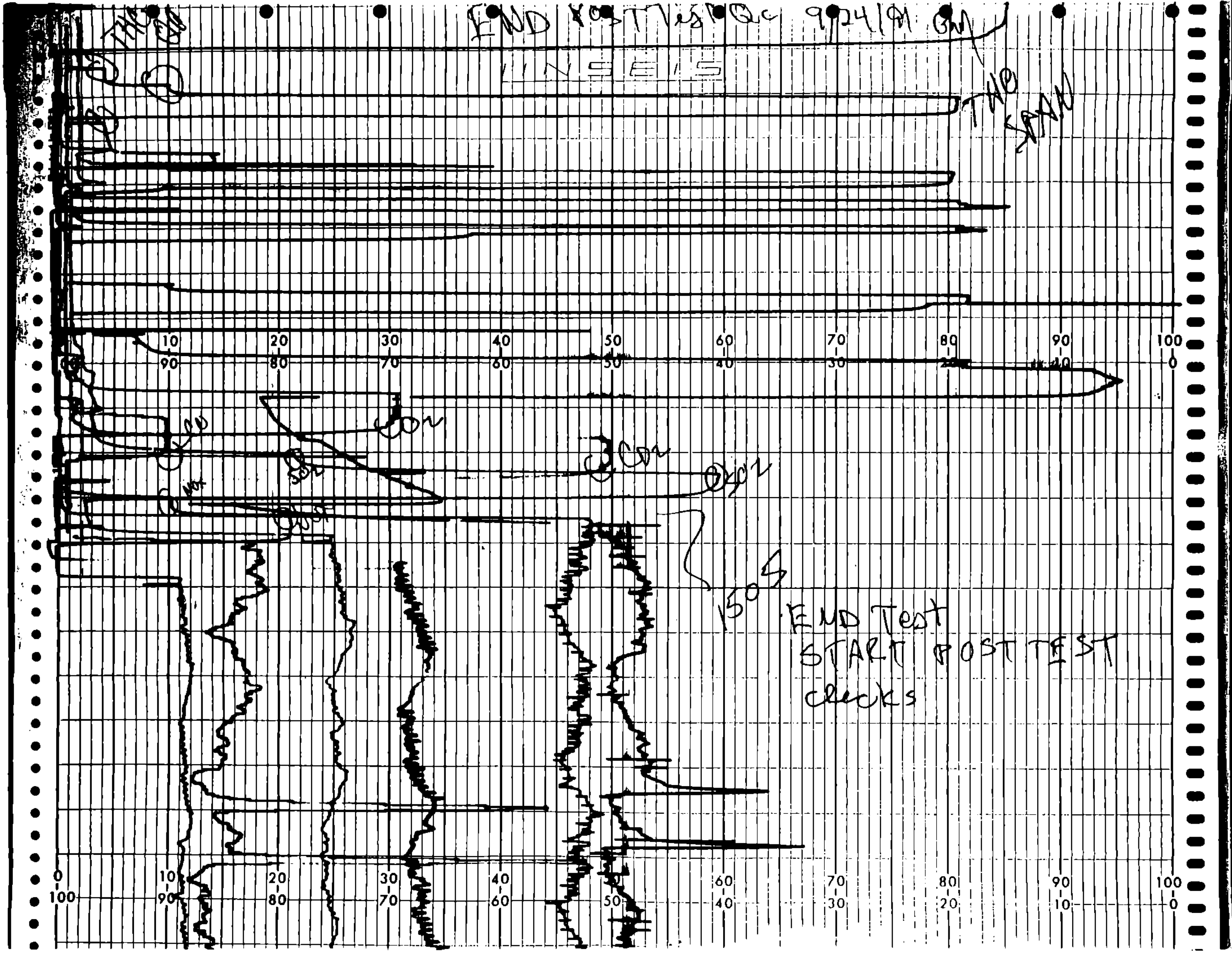
END

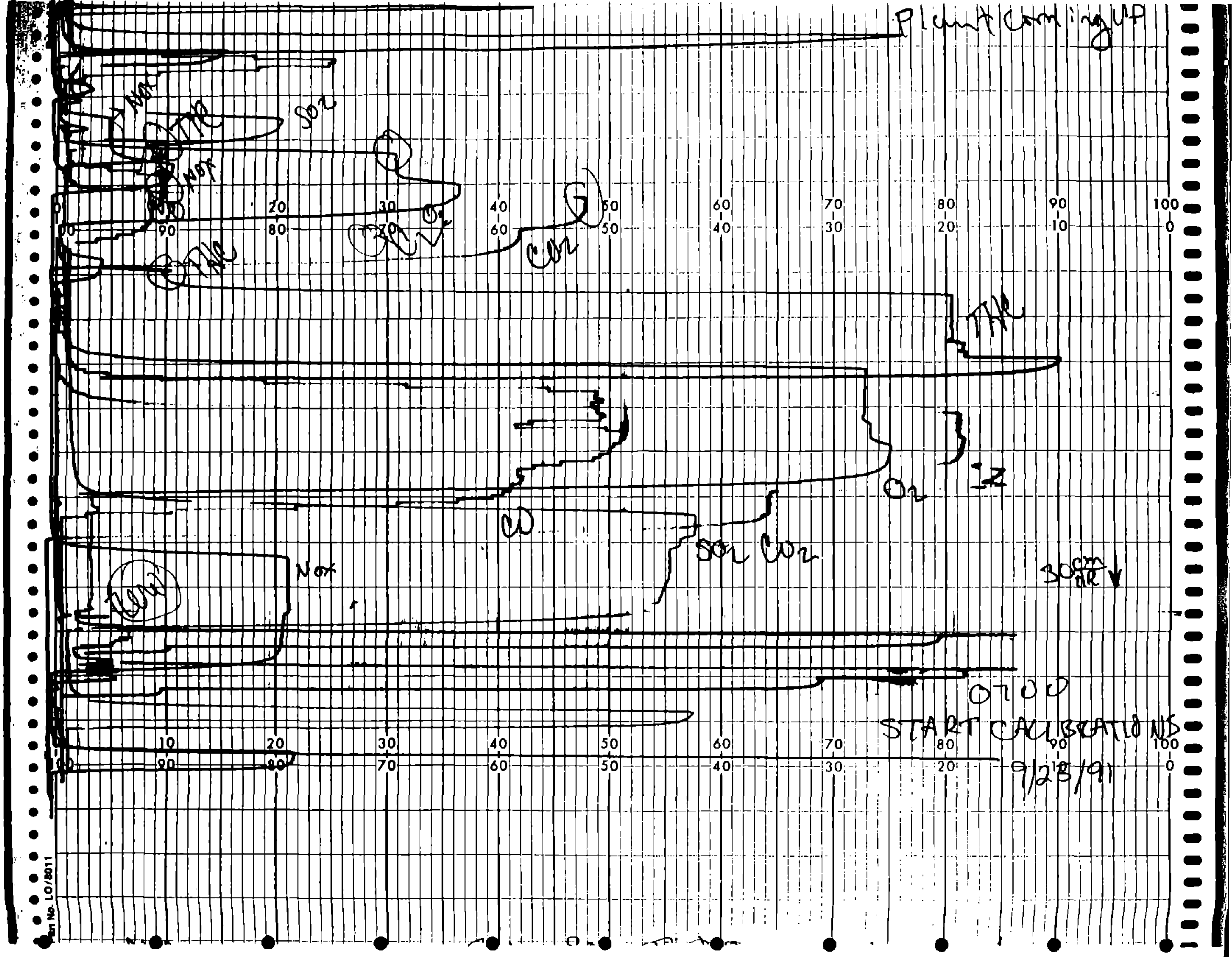
THC 810PPM 1231



Part No. LO/8011

Printed in USA

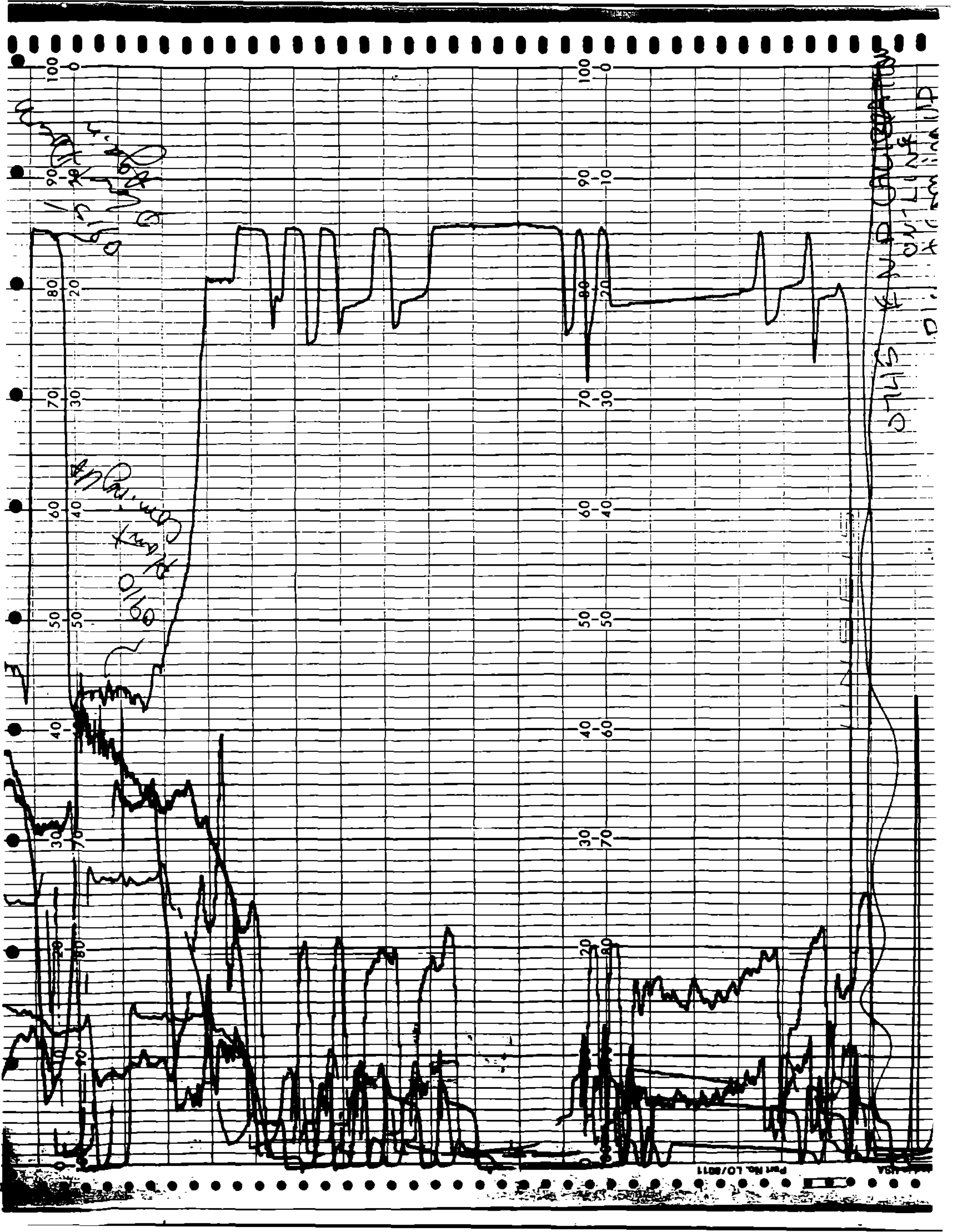

END POSTTEST QC 9/24/91 GW
LINSEIS
THC SPAN
THC
CO
CO2
O2
SO2
NOx
1505
END Test
START POST TEST
checks
0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0

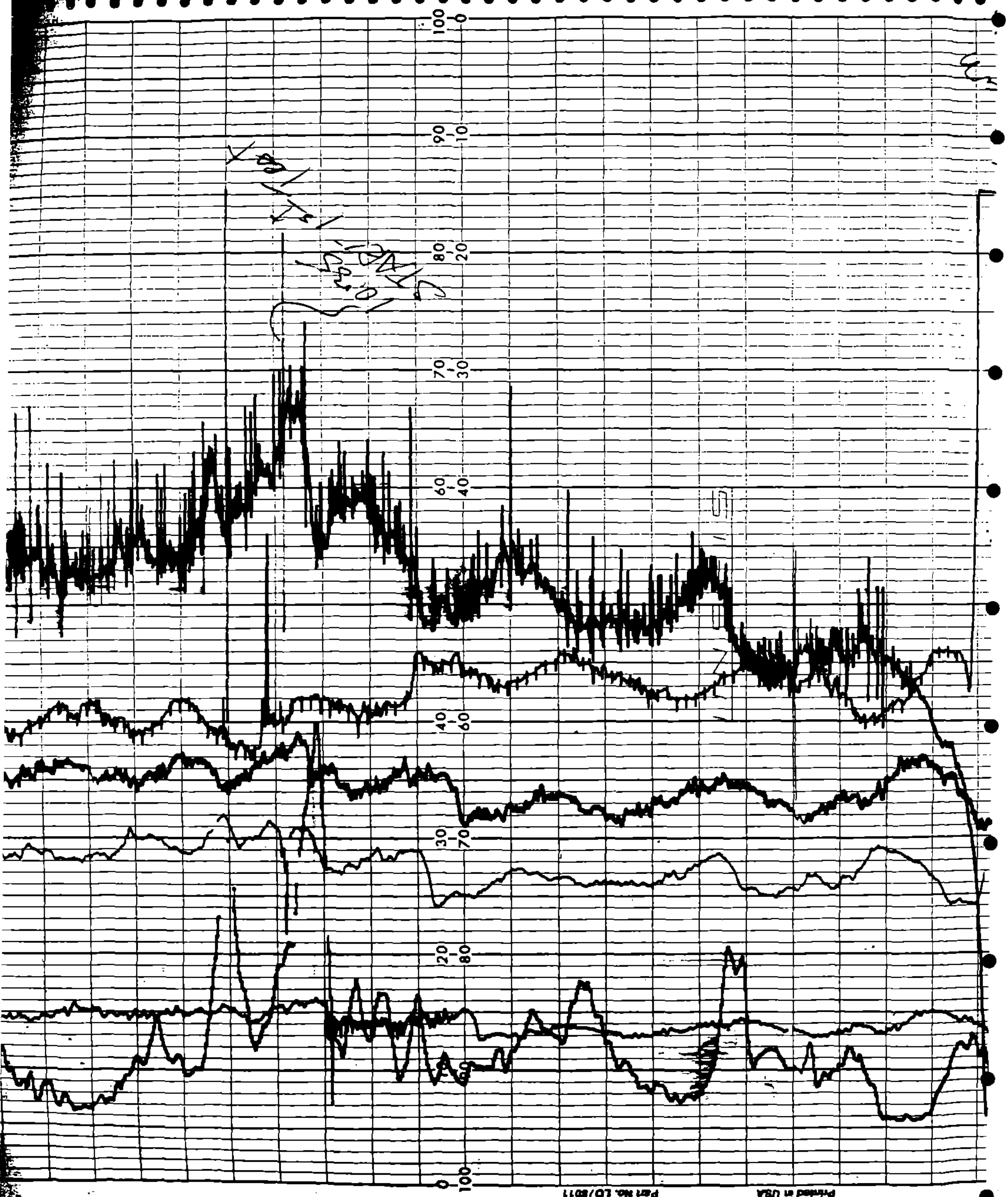


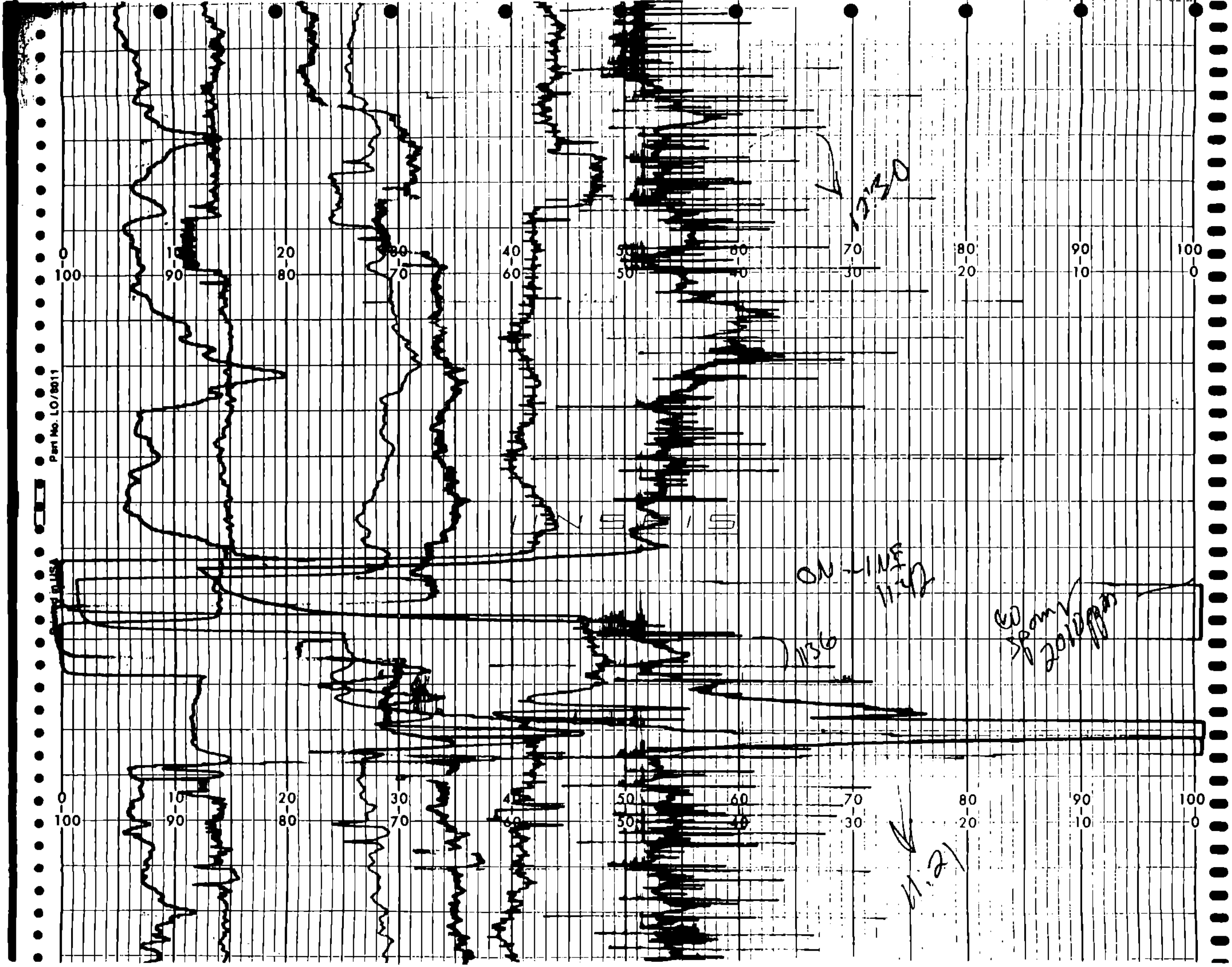
Plant Coming UP

0700 START CALIBRATIONS 9/28/91

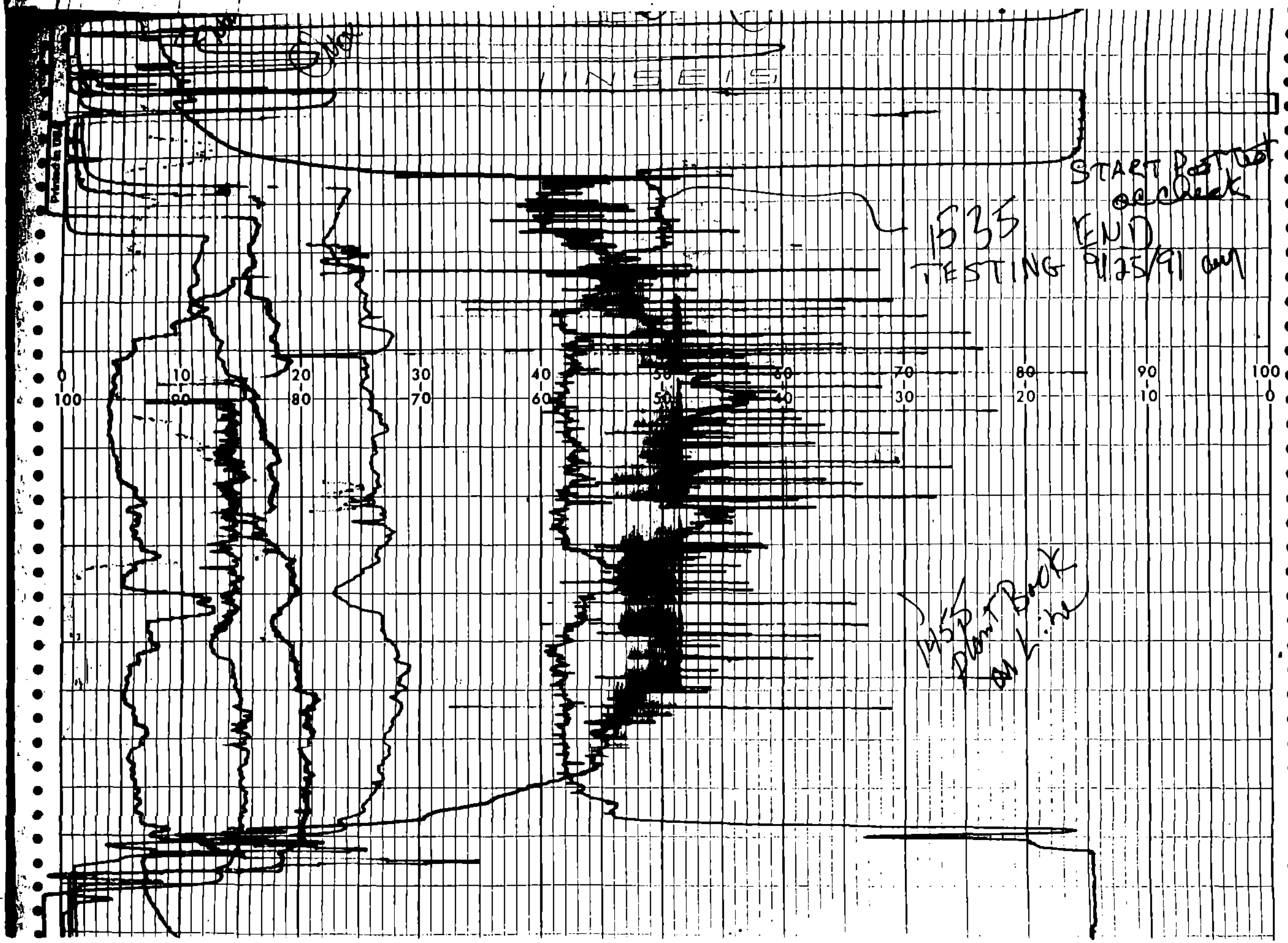
30 cm/hr


100
90
80
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
70
80
90
100
0910
THIS END UP CAUTION
ON-LINE
Part No. LO/8011


100 0
90 10
80 20
70 30
60 40
40 60
30 70
20 80
0 100
Part No. LO/8011
Printed in USA

12:30
ON-LINE
11:42
CO
Span
2010 ppm
11:36
11:21
0
100
10
90
20
80
30
70
40
60
50
50
60
40
70
30
80
20
90
10
100
0
Part No. LO/8011
Printed in USA

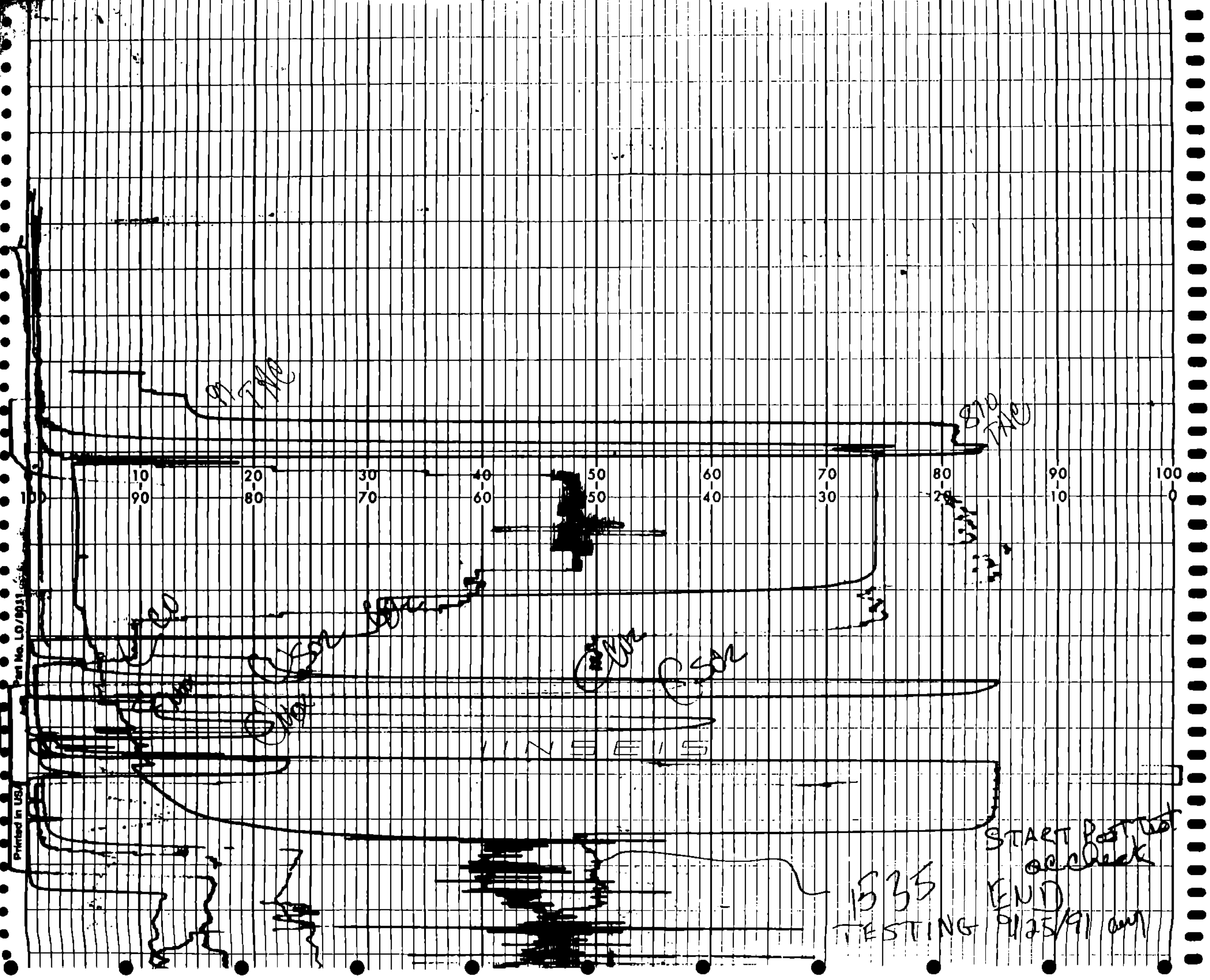

10 90
20 80
30 70
40 60
50 50
60 40
70 30
80 20
90 10
100 0
0 100
13:15



START Post test @ check

1535 END TESTING 9/25/91 am

1455 Plant Back on line

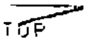

100
10
90
20
80
30
70
40
60
50
50
60
40
70
30
80
20
90
10
100
0
LINSEIS
Part No. LO/8011
Printed in USA
START Post Test check
1533
TESTING
END
9/25/91 am

APPENDIX E

GC DATA

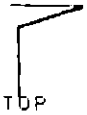
```
CHT SP =  1.0
AR REJ =  0
THRSH  =  0
PK WD  =  0.04


*  AR REJ 500  0
*  ATT 2^ 3  0
*  PLOT
```



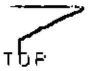
```
STOP


*  PLOT
```



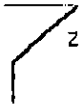
```
STOP


*  PLOT
```



```
STOP


*  THRSH 2  0
*  PLOT
```

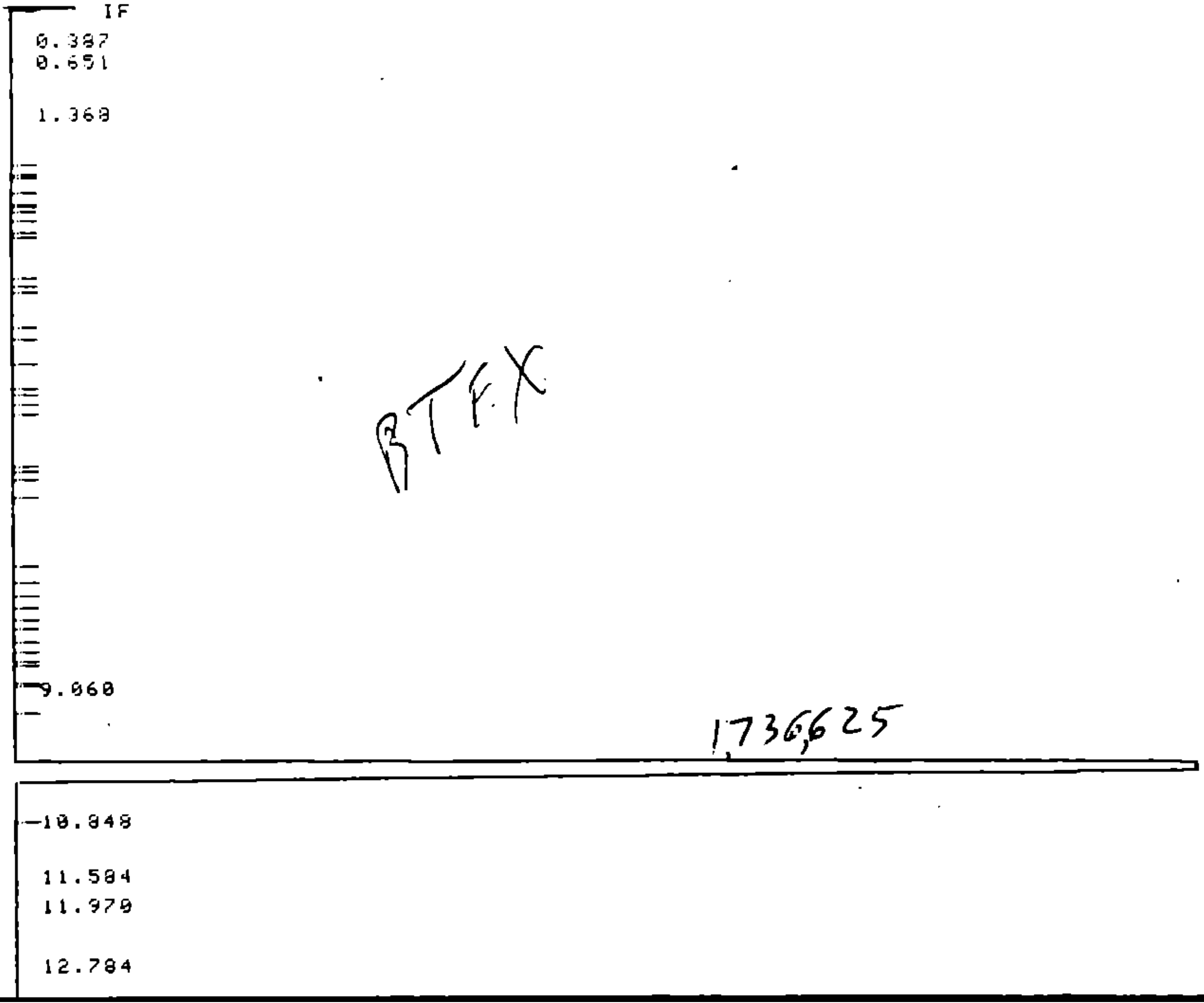


```
ZE=  0, 0.180

             STOP
```

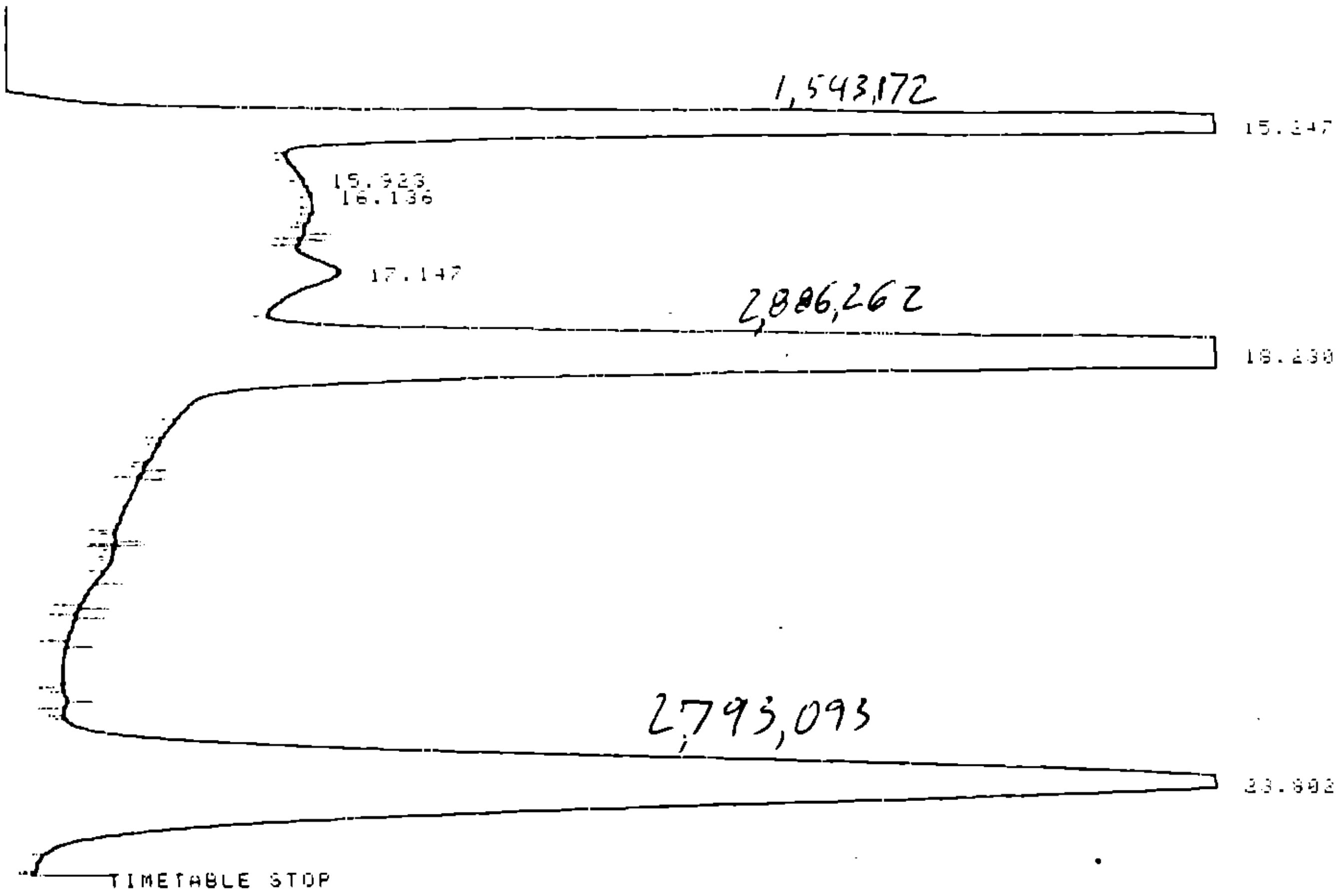
pg ①

```
*  RUN #    2      SEP 22, 1991  15:02:04
START
```



BTEX

1,736,625



lg ②

```
RUN#      2             SEP 22, 1991   15:02:04


MATHY CONSTRUCTION

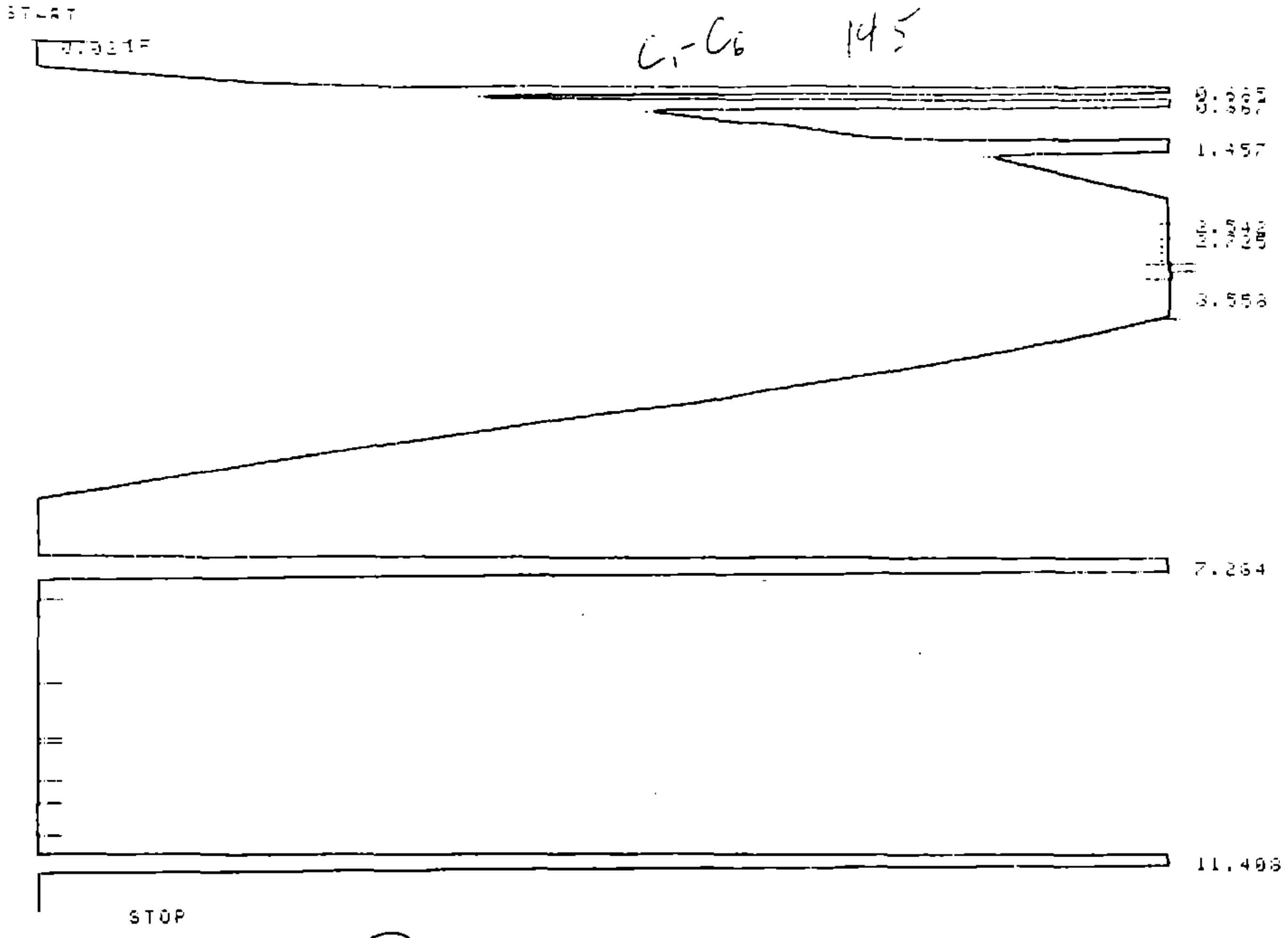
AREA%
        RT         AREA TYPE  WIDTH       AREA%
      .387          974   VB   .091      .01078
      .651         4153   BB   .159      .04595
     1.368         1359   PB   .328      .01504
    10.226      1736625   PB   .216    19.21357
    11.584          694   PB   .065      .00768
    15.247      1543172   BB   .174    17.07326
    16.136          725   BB   .076      .00802
    17.147        71479   BB   .382      .79082
    18.230      2886262   BB   .387    31.93286
    23.802      2793093   VB   .683    30.90204

TOTAL AREA=9.0385E+06
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.04
```

C1-C6 145



③

```
RUN#      3             SEP 22, 1991  15:30:02


MATHY CONSTRUCTION

AREA%
      RT         AREA TYPE  WIDTH        AREA%
    .027         1878   PB   .022       .02139
    .685       412048   BB   .086      4.69397
    .867       828131   BB   .087      9.43392
   1.457      1245052   BB   .099     14.18341
   2.542        41126   BB  3.873       .46850
   2.725         1367   BB   .133       .01557
   3.558      1716034   BB   .161     19.54874
   7.284      2106210   BB   .202     23.99354
  11.408      2426386   BB   .222     27.64094

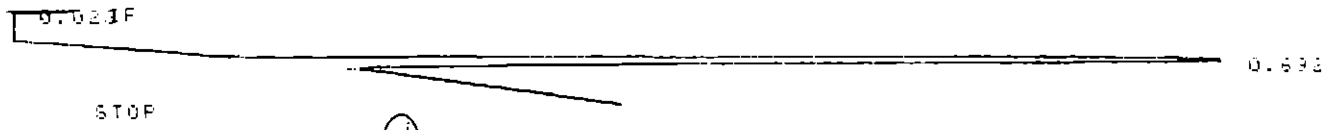
TOTAL AREA=8.7782E+06
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.04
```

METHANE =
9,96

```
+  RUN #     5      SEP 22, 1991  15:44:57
START
```



④

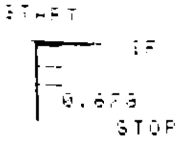
```
RUN#     5            SEP 22, 1991  15:44:57


MATHY CONSTRUCTION

AREA%
      RT        AREA TYPE  WIDTH       AREA%
     .023       1008   PB    .016      .32345
     .692     310631   BB    .095    99.67654

TOTAL AREA= 311639
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```



⑤

METHANE
.996 ppm

```
RUN#      6            SEP 22, 1991  15:43:40

MATH/ CONSTRUCTION

AREA%
      RT        AREA TYPE  WIDTH       AREA%
     .673      35999 I 6P    .096  100.00000

TOTAL AREA=  35999
MUL FACTOR=1.0000E+00

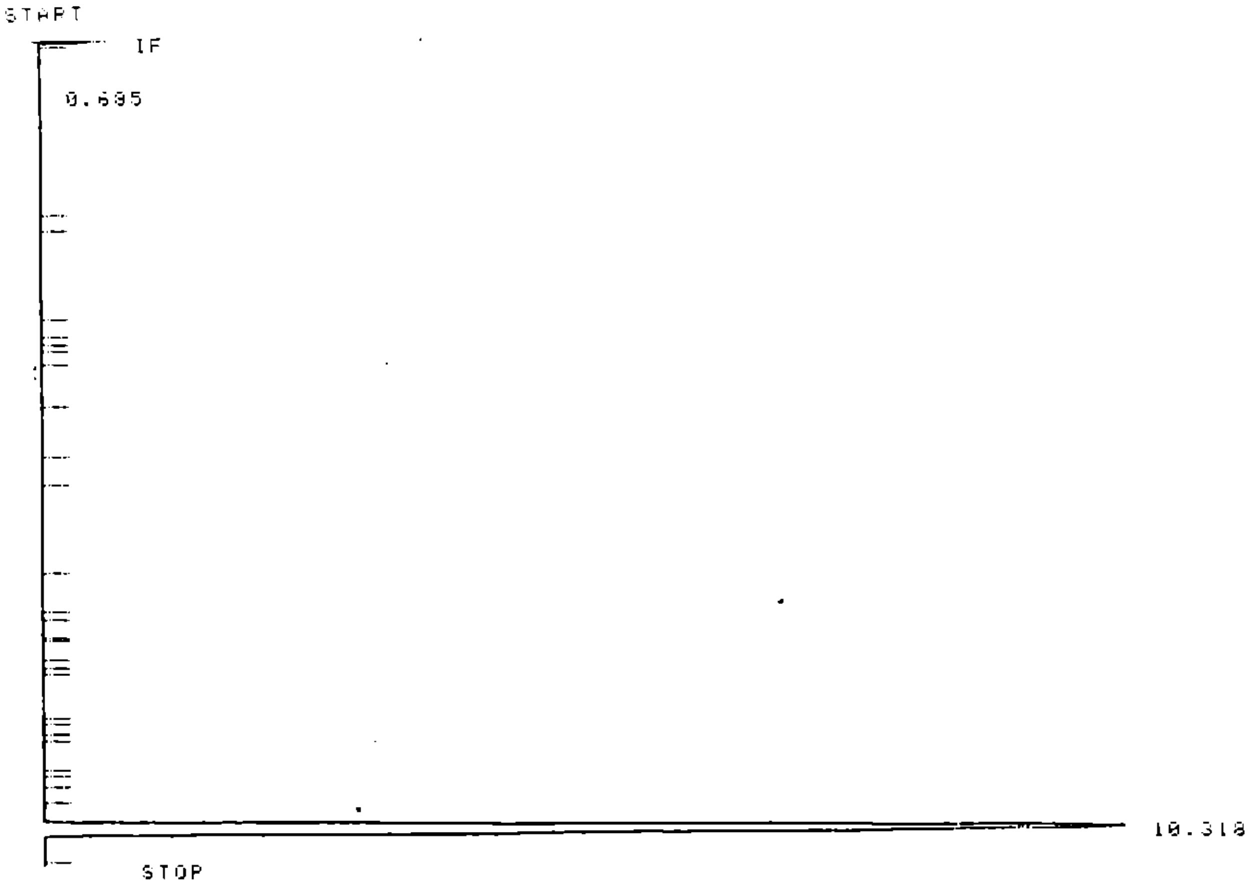


RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.04
```

BTEX 10

⑥

```
*  RUN #     7       SEP 22, 1991   15:52:18
```



```
RUN#      7            SEP 22, 1991   15:52:18


MATHY CONSTRUCTION

AREA%
        RT        AREA TYPE  WIDTH       AREA%
      .685         884   PB   .087      .05109
    10.318     1729492   BB   .215    99.94893

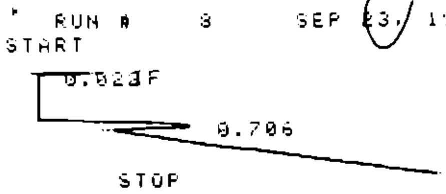
TOTAL AREA=1730376
MUL FACTOR=1.0000E+00
```

```
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```

METHANE 1.0 ppm

(1) MON

```
 RUN #    8     SEP 23, 1991  06:25:05
```



```
RUN#     8          SEP 23, 1991  06:25:05


MATHY CONSTRUCTION

AREA%
      RT       AREA TYPE  WIDTH       AREA%
    .022        885   PB   .012     1.27512
    .706      68520   BB   .184    98.72490

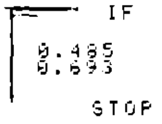
TOTAL AREA=  69405
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```

METHANE
1.0 ppmv

```
*  RUN #    9      SEP 23, 1991  06:27:42
START
```



⑧

```
RUN#     9          SEP 23, 1991  06:27:42


MATHY CONSTRUCTION

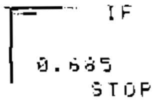
AREA%
        RT        AREA TYPE  WIDTH       AREA%
      .485        7306   PB   .381    18.18408
      .693       32872   VB   .089    81.81590

TOTAL AREA=  40178
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```

METHANE
1.0 ppmv

```
*  RUN #    10      SEP 23, 1991  06:29:55
START
```



⑨

```
RUN#    10          SEP 23, 1991  06:29:55


MATHY CONSTRUCTION

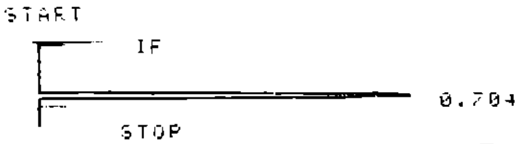
AREA%
     RT        AREA TYPE  WIDTH       AREA%
    .685      36414 I BP   .099  100.00000

TOTAL AREA=  36414
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^ =   3
CHT SP =   1.0
AR REJ =   500
THRSH   =  2
PK WD   =  0.04
```

METHANE
9.96 ppmv
(DRY/NO PRESSURE
IN CANISTER)

```
*  RUN #   11      SEP 23, 1991  06:32:13
```



⑩

```
RUN#    11          SEP 23, 1991  06:32:13


MATHY CONSTRUCTION

AREA%
      RT       AREA TYPE  WIDTH      AREA%
    .704     267305   BB    .088  100.00000

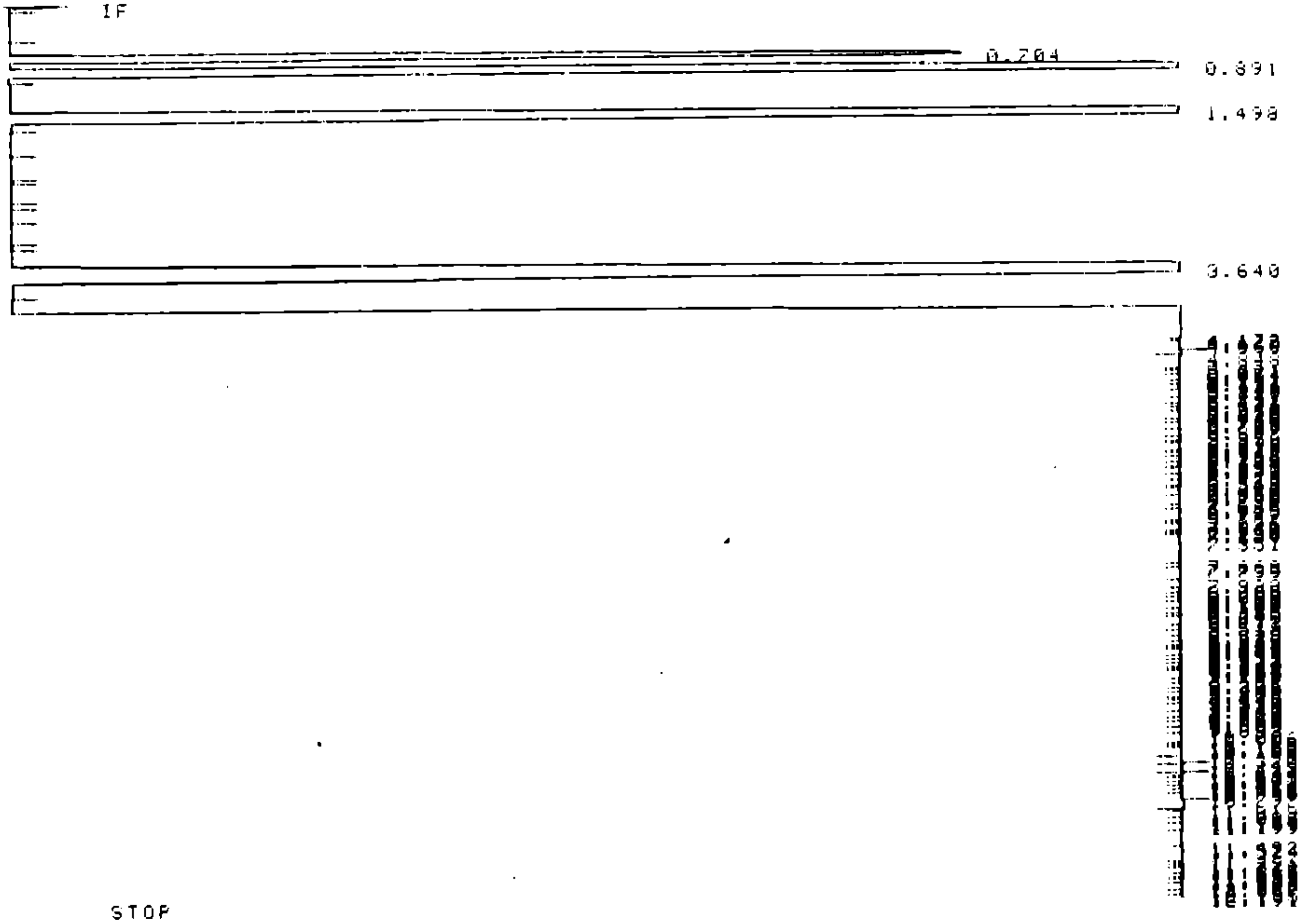
TOTAL AREA= 267305
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```

C_1-C_6
~14.5 ppmv

⑪

```
*  RUN #    12        SEP 23, 1991  06:36:23
START
```



```
          STOP


RUN#     12           SEP 23, 1991  06:36:23


MATHY CONSTRUCTION

AREA%
        RT        AREA TYPE  WIDTH       AREA%
      .704      410147   PB   .090      .29439
      .891      818294   BB   .091      .58734
     1.498     1206782   BB   .101      .86618
     3.640     1675756   BB   .161     1.20279
     4.122   118335880 >SBB   .231    84.93690
```

```
4.666   1442655   BB    .042   1.03351
4.843   2529200   SBB   .103   1.83863
5.021    116315   SPB   .016    .08348
5.054    218312   SPB   .029    .15670
5.111    530658   SPB   .048    .38089
5.194    258109   SPB   .024    .18526
5.240    507908   SPB   .041    .36456
5.321    177608   SPB   .036    .12748
5.431    726929   SPB   .063    .52176
5.515     90536   SPB   .019    .06498
5.550    634551   SPB   .045    .45546
5.625   1214899   SPB   .070    .87201
5.735    288533   SPB   .026    .20710
5.765    144196   SPB   .025    .10350
5.847    156288   SPB   .050    .11218
5.950    191963   SPB   .035    .13778
6.018     39909   SPB   .013    .02865
6.077    457305   SPB   .049    .32824
6.142     51105   SPB   .015    .03668
6.166    103645   SPB   .020    .07439
6.226    157346   SPB   .029    .11294
6.295    168443   SPB   .032    .12090
6.351     34195   SPB   .012    .02454
6.378    162124   SPB   .072    .11637
6.473    116264   SPB   .021    .08345
6.545    102920   SPB   .025    .07387
6.620     85188   SPB   .017    .06114
6.650    238242   SPB   .047    .17100
6.748     81237   SPB   .035    .05831
6.885     92234   SPB   .024    .06620
6.925    207534   SPB   .138    .14896
7.096     17521   SPB   .012    .01258
7.140     57702   SPB   .028    .04142
7.230     24787   SPB   .018    .01779
7.254     18404   SPB   .012    .01321
7.351   1234910   SPB   .170    .88637
7.668     37302   SPB   .025    .02677
7.730    560149   SPB   .130    .40205
7.923    111069   SPB   .036    .07972
8.005     56856   SPB   .048    .04081
8.085     29846   SPB   .019    .02142
8.130    113090   SPB   .046    .08117
8.197     91178   SPB   .088    .06544
8.283     78554   SPB   .044    .05638
8.348     24025   SPB   .027    .01724
8.411     87074   SPB   .044    .06250
8.518     32961   SPB   .031    .02366
8.575    122790   SPB   .049    .08813
8.678     25392   SPB   .021    .01823
8.725     43136   SPB   .024    .03096
8.784     48168   SPB   .021    .03457
8.818      6278   SPB   .012    .00451
8.844     58472   SPB   .034    .04197
8.932     59021   SPB   .021    .04236
8.973     34888   SPB   .019    .02504
9.042     33250   SPB   .025    .02387
9.070     23863   SPB   .027    .01713
9.107     13480   SPB   .010    .00968
9.140     46244   SPB   .032    .03319
9.235     60390   SPB   .058    .04335
9.306     27716   SPB   .032    .01989
9.380     32608   SPB   .025    .02340
9.428     34309   SPB   .032    .02463
9.510     28746   SPB   .019    .02063
```

⑫

```
     9.655       47645   SPB    .060       .03434
     9.766       33367   SPB    .025       .02105
     9.820       39912   SPB    .036       .02865
     9.893       62791   SPB    .042       .04507
    10.006      129304   SPB    .049       .09317
    10.105       28657   SPB    .020       .02057
    10.152       50748   SPB    .019       .03642
    10,165       46709  ISPB    .078       .03353
    10.320       19692    BB    .028       .01413
    10.433       41660    BB    .027       .03005
    10.535       24353    PB    .016       .01748
    10.571        3737    BB    .009       .00268
    10.593       26838    BB    .021       .01926
    10.661       17693    BB    .019       .01270
    10.715        5594    BB    .020       .00402
    10.745       20924    BB    .026       .01502
    10.797       56102    BB    .052       .04027
    11.019        9405    PB    .014       .00675
    11.060      105694    BB    .046       .07586
    11.151       63174    BB    .041       .04534
    11.199       44945    BB    .043       .03226
    11.492      107622    BB    .124       .07725
    11.524      434779    BB    .163       .31207
    11.721       19248    BB    .027       .01382
    11.772       16814    BB    .021       .01207
    11.815       55947    BB    .045       .04016
    11.895       27350    BB    .014       .01963
    11.939       12857    BB    .014       .00923
    11.967       20298    BB    .023       .01457
    12.067       47804    BB    .035       .03431
    12.135       20558    BB    .022       .01476
    12.191       18971 I  BP    .028       .01362

TOTAL AREA=1.3932E+08
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.04
```

(13)

```
*  PREP  CALIB   @

E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E*/I/N]:

REF % RTW [   5.000]:
NON-REF % RTW [   5.000]:

RF BASED ON AREA OR HEIGHT [A*/H]:
CAL#     RT          AMT          NAME
  1  :.704       :14.99        :METHANE
  2  :.891       :14.5         :ETHANE
  3  :1.498      :14.2         :PROPANEE
  4  :3.640      :14.6         :BUTANE
  5  :
REF PK CAL#:  1
REF PK CAL#:  1
REF PK CAL#:

GROUP PEAKS [Y/N*]:

CALIBRATION OPTIONS
RF of uncalibrated peaks [0.0000E+00 ]: 3.655E-05
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

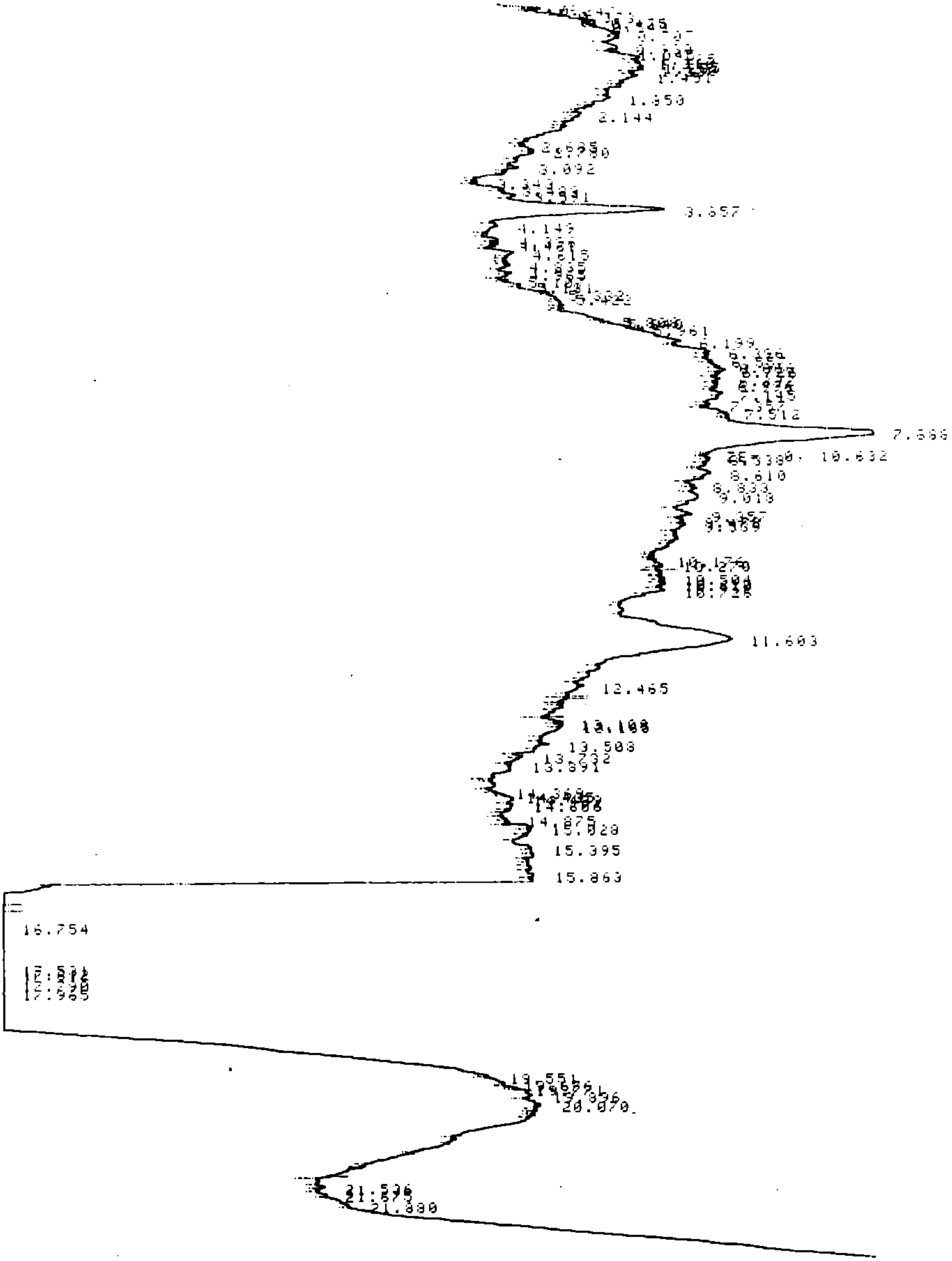
*  LIST: LIST
PEAK CAPACITY:  1234

ZERO   =  0, 26.894
ATT 2^ =  3
CHT SP =  1.0
AR REJ =  500
THRSH  =  2
PK WD  =  0.04
```

(14)

```
*ZERO   BREAK

*  RUN #   13     SEP 23, 1991  07:18:54
START
```



```
STOP
```

(15)

```
RUN#    13           SEP 23, 1991  07:18:54


MATHY CONSTRUCTION


ESTD-AREA
      RT          AREA TYPE  CAL#  CONCENTRAT
    .158          6851   BB               .250
```

```
   .373      915   BB              .034
   .475     4257   BB              .156
   .545      565   BB              .021
   .707     2684   FB    1F        .098
   .930     1114   FB    2         .020
  1.045      745   FB              .027
  1.125     4205   BB              .154
  1.210      597   BB              .022
  1.265     1429   BB              .052
  1.330     1730   BB              .063
  1.451     4525   VB    3         .053
  1.850     4194   VB              .153
  2.144      827   FB              .030
  2.635      605   FB              .022
  2.780     2383   BB              .087
  3.092     3092   FB              .113
  3.343      901   FB              .033
  3.489     4241   BB              .155
  3.591     3894   BB    4         .034
  3.857   134747   BB             4.925
  4.149     3949   BB              .144
  4.396     3442   FB              .126
  4.461     1480   BB              .054
  4.615     5368   PB              .196
  4.835     2758   VB              .101
  4.965     1200   BB              .044
  5.107      800   FB              .029
  5.181     1714   BB              .063
  5.422     1016   BB              .037
  5.808     3507   PB              .128
  5.840      508   BB              .019
  5.961     1511   BB              .055
  6.199     8143   BB              .298
  6.396     5284   PB              .193
  6.557     1270   PB              .046
  6.666     1352   BB              .049
  6.726     2001   BB              .073
  6.892     2207   FB              .081
  6.996     1588   BB              .058
  7.145     2327   PB              .085
  7.357      780   PB              .029
  7.512     2552   PB              .093
  7.888   203172   VB             7.426
  8.338      520   PB              .019
  8.610     4048   PB              .148
  8.833      546   PB              .020
  9.010     6086   PB              .222
  9.357     6592   PB              .241
  9.472     1838   BB              .067
  9.539     3258   BB              .119
 10.504      702   PB              .026
 10.610     1398   VB              .051
 10.726     4267   BB              .156
 11.603   267639   PB             9.782
 12.465     5203   PB              .190
 13.108     2934   PB              .107
 13.165     1403   BB              .051
 13.508     2238   PB              .082
 13.732     1228   PB              .045
 13.891      690 I PB              .025
 14.435      683   BB              .025
 14.489     2937   BB              .107
 14.606     2040   BB              .075
 15.028     6056   PB              .221
 15.863     2215   PB              .081
```

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```
   17.531       1392    PB                 .051
   17.611       1534    BB                 .056
   17.739        768    PB                 .028
   17.965        523    BB                 .019
   19.551     239711    BB                8.761
   19.676       2625    BB                 .096
   19.771        874    PB                 .032
   19.896       6054    BB                 .221
   20.070       4805    BB                 .176
   21.536       1316    PB                 .048
   21.675        602    PB                 .022
   21.880       2106    BB                 .077

 TOTAL AREA=1036230
 MUL FACTOR=1.0000E+00
```

⑫

```
 RUN PARAMETERS
 ZERO    =  0
 ATT 2^  =  3
 CHT SP  =  1.0
 AR REJ  =  500
 THRSH   =  2
 PK WD   =  0.04
```

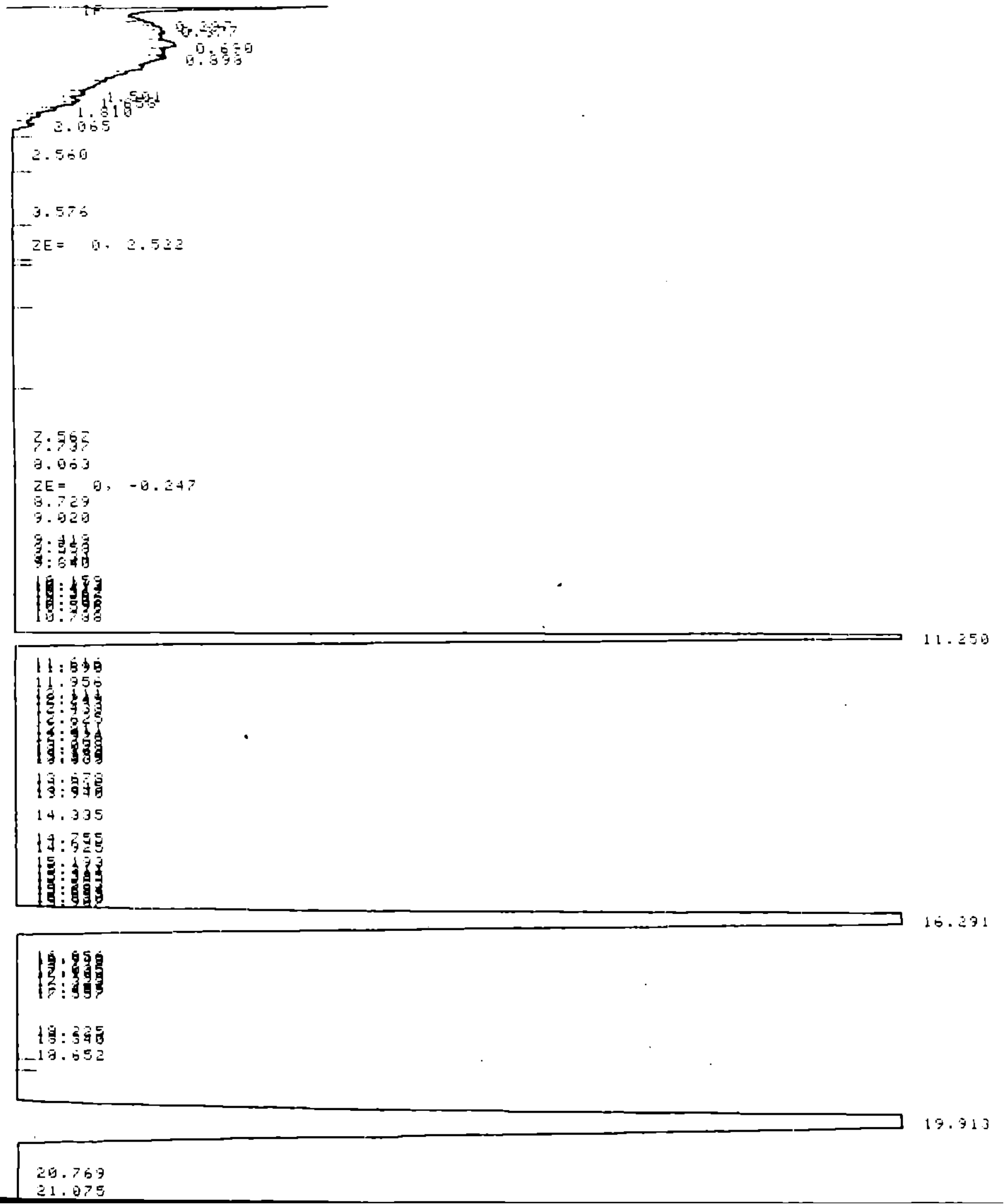
BTEX
~10ppmv

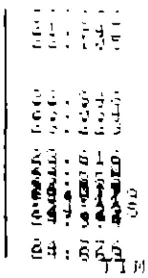
```
REPORT OPTIONS

Suppress local report [Y/N*]:
HEIGHT% report [Y/N*]:
Replace report title [Y/N*]:
Replace amount label [Y/N*]: Y
Amount label:  CONC.
Report uncalibrated peaks [Y*/N]:
Extended report [Y/N*]: Y

*  PK WD .07  0
*  RUN #   14      SEP 23, 1991  07:49:57
START
```

(18)





```
   TIMETABLE STOP
```

⑲

```
RUN#     14          SEP 23, 1991  07:49:57


MATHY CONSTRUCTION

ESTD-AREA
     RT TYPE         AREA  WIDTH       HEIGHT CAL# CONC.          NAME
    .297   BB         880  .042          352              .032
    .377   BB         697  .057          205              .025
    .690   PB        9554  .124         1289   1R         .349    METHANE
    .898   VB       13949  .154         1512   2          .247    ETHANE
   1.501   PB        2750  .069          663   3          .032    PROPANEE
   1.658   BB        2061  .048          721              .075
   1.810   PB        2770  .054          856              .101
   2.065   VB       21861  .183         1994              .799
   2.560   PB        4496  .104          721              .164
   3.576   VB        4576  .070         1086   4          .040    BUTANE
   7.567   PB        4223  .089          795              .154
   7.737   BB        3440  .069          826              .126
   8.063 I PB         793  .030          439              .029
   8.729   VB       11299  .146         1293              .413
   9.020   BB        5904  .122          807              .216
   9.419   PB        5200  .082         1057              .190
   9.558   BB        1588  .040          663              .058
   9.734   PB         819  .022          622              .030
   9.840   BB         552  .031          299              .020
  10.159   PB        2255  .078          481              .082
  10.213   BB        2381  .053          744              .087
  10.362   BB        5522  .084         1099              .202
  10.508   BB        3802  .079          802              .139
  10.596   BB        1568  .050          527              .057
  10.788   BB        9024  .093         1621              .330
  11.250   BB     1547092  .211       122462            56.546
  11.690   BB        1595  .048          554              .058
  11.956   BB        5529  .168          550              .202
  12.111   BB        7549  .062         2029              .276
  12.249   BB        3681  .076          811              .135
  12.438   BB        2204  .092          400              .081
  12.625   BB       11057  .150         1229              .404
  12.926   BB        1831  .081          379              .067
  13.098   PB        1641  .030          900              .060
  13.163   BB        1499  .057          438              .055
  13.279   BB         903  .020          756              .033
  13.335   BB        2955  .055          893              .108
  13.678   PB        2256  .138          272              .082
  13.940   BB        1528  .031          833              .056
  14.335   BB        2897  .076          637              .106
  14.755   BB       15931  .127         2098              .582
  14.925   BB        5487  .164          556              .201
  15.193   BB        5938  .097         1020              .217
  15.311   BB        1099  .036          504              .040
  15.385   BB        4258  .041         1725              .156
  15.563   BB        3233  .064          848              .118
  15.684   BB        3949  .096          683              .144
```

```
  16.231    BB    1877132   .267      117230            55.609
  16.356    BB       7063   .070        1631              .256
  16.943    BB       3566   .049        1223              .131
  17.035    VB      10764   .141        1277              .394
  17.163    BB       2542   .032        1331              .093
  17.329    BB       2295   .034        1110              .084
  17.455    BB       2542   .046         915              .093
  17.557    BB      11695   .153        1272              .427
  18.225    PB      11454   .140        1364              .419
  18.340    BB       6847   .094        1220              .250
  18.652    PB      13564   .124        1826              .496
  19.913    PB    2526840   .445       94657            92.356
  20.769    PB       1811   .049         611              .066
  21.075    PB       2994   .053         949              .109
  21.335    PB       4098   .055        1237              .150
  21.447    BB       2223   .026        1444              .081
  21.494    BB       2353   .052         758              .086
  21.626    BB       3475   .054        1072              .127
  21.947    PB       6679   .058        1911              .244
  22.105    BB       3910   .079         822              .143
  22.848    PB        703   .033         350              .026
  23.034    BB       4096   .148         460              .150
  23.223    PB       2142   .087         411              .078
  23.616    PB        806   .024         564              .029
  23.770    BB       1847   .085         362              .068
  23.888    BB        755   .042         298              .028
  24.019    PB       1666   .052         538              .061
  24.077    BB       1156   .027         719              .042
  24.322    PB       1347   .031         715              .049
  24.394    BB       2230   .032        1150              .082
  24.776    PB       2572   .068         635              .094
  24.863    BB       1475   .024        1020              .054


TOTAL AREA=6283574
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO     =   0
ATT 2^   =   3
CHT SP   =   1.0
AR REJ   =   500
THRSH    =   2
PK WD    =   0.07
```

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```
*  PLOT
```

```
STOP

*  PK WD   BREAK

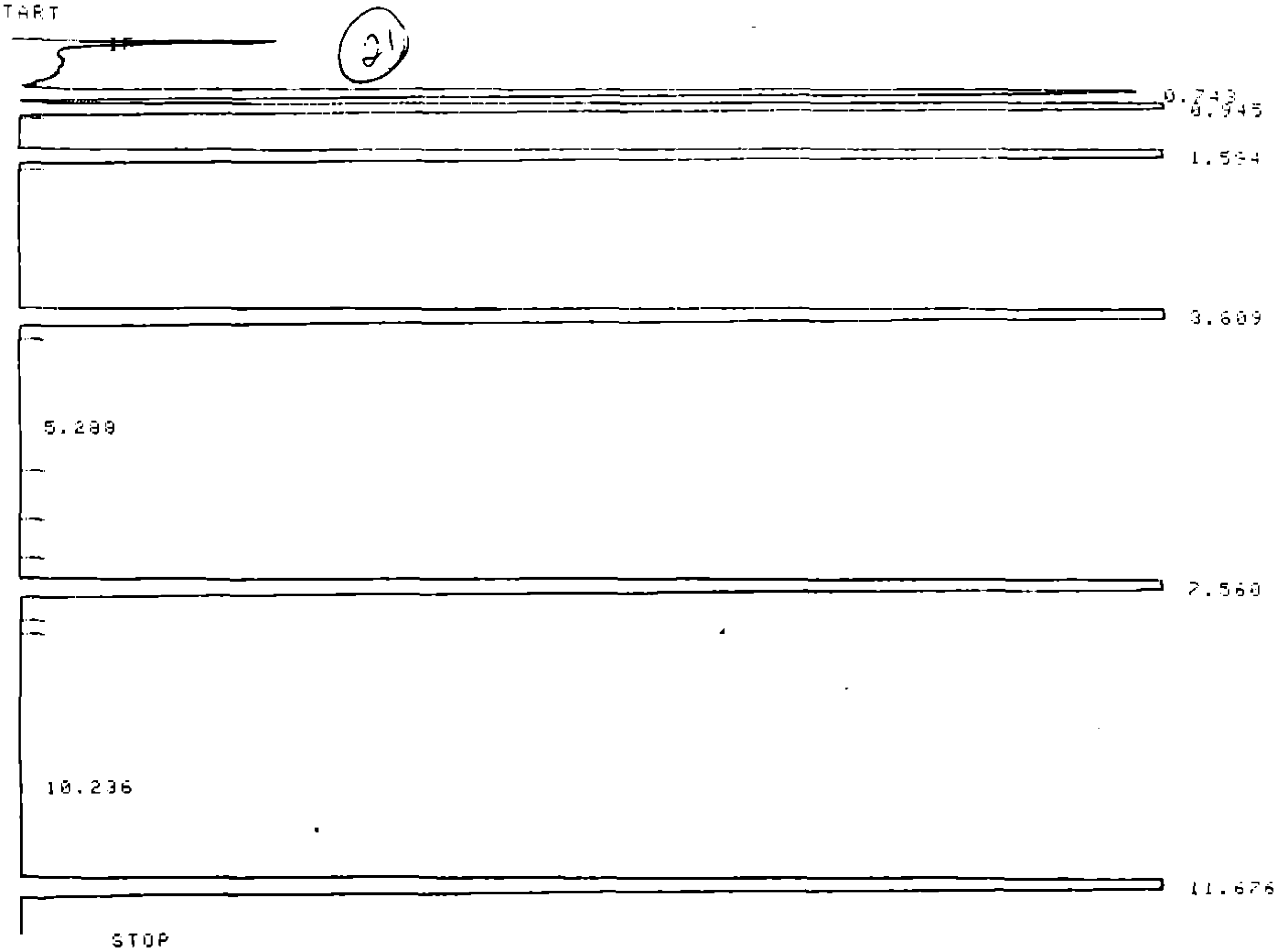
*  LIST: PK WD =  0.07

*  PK WD .12  0
*  LIST: ZERO =  0, 11.894
```

C6 ~14.5

```
*  RUN #   15      SEP 23, 1991  08:29:41
START
```

㉑



```
     STOP


RUN#    15          SEP 23, 1991  08:29:41


MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
     RT        AREA TYPE  WIDTH        AREA%
   .743      382193   BB   .097      4.66599
   .945      767635   BB   .097      9.37164
  1.594     1137188   BB   .108     13.88332
  3.809     1582630   BB   .160.    19.32147
  5.288       16048   BB   .141       .19592
```

```
  10.336       4722   PB   .201      .05765
  11.576    2045950   PB   .221    33.04716

TOTAL AREA=6191040
MUL FACTOR=1.0000E-00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

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```
*  LIST:
BREAK

*DEL  BREAK

*  DELETE  CALIB  @
DELETE ALL  [Y/N*]: Y

*  PREP  CALIB   @

E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E*/I/N]:

REF % RTW [    5.000]:
NON-REF % RTW [    5.000]:

RF BASED ON AREA OR HEIGHT [A*/H]:
CAL#      RT           AMT          NAME
  1   :.743※※※※-.743
                  :15.0         :METHANE
  2   :.945       :14.5         :ETHANE
  3   :1.594      :14.2         :PROPANE
  4   :3.809      :14.6         :PENTANE
  5   :7.560      :14.6         :PENTANE
  6   :11.676     :14.5         :HEXANE
  7   :

GROUP PEAKS [Y/N*]:

CALIBRATION OPTIONS
RF of uncalibrated peaks [0.0000E+00 ]: 3.922E-05
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

*  LIST: TIME   @

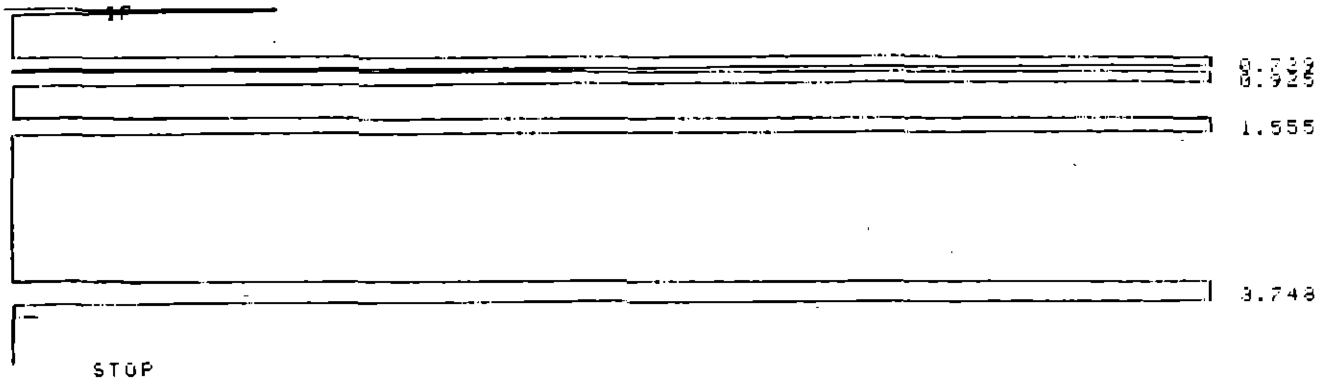
   0.000 INTG # =  2
   0.000 INTG # =  8
  25.000 STOP

*  DELETE  TIME 25  @

*  TIME 27  STOP
*  RUN #   16     SEP 23, 1991  08:49:08
START
```

C1-C6
~ 100 ppmv

(23)



24

```
RUN#     16           SEP 23, 1991  05:49:08


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .729   PB     3656069   .096      462900   1R    104.243  METHANE
    .925  BPB     5381096   .096      935835   2     101.645  ETHANE
   1.555  BPB     7997978   .104     1286288   3      99.870  PROPANE
   3.746   BB    10938824   .162     1124710   4     100.912  PENTANE


TOTAL AREA=2.7974E+07
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

```
*  PREP  CALIB 3  @
CAL#      RT          AMT
  1      0.739     :102.5
  2      0.940     :
  3      1.584     :000#100.4
  4      3.793     :97.31
  5      7.560     :
  6     11.676     :

*  EDIT  CALIB 3  @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

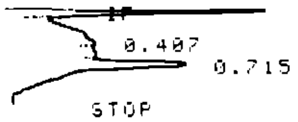
SECTION TO BE EDITED: 3

CAL#:  2
RT:
AMT:  101.1
AMT/AREA:


SECTION TO BE EDITED:

*  RUN #    17       SEP 23, 1991  08:59:07
START
```

METHANE .996 ppmv



```
        STOP
```

(25)

```
RUN#     17           SEP 23, 1991  08:59:07


MATHY CONSTRUCTION

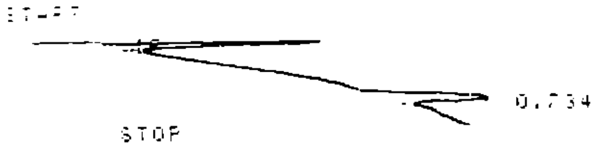
ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .407   PB       11094   .293         630              .435
    .715   VB       57473   .137        6974   1R        2.256  METHANE


TOTAL AREA=  68567
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

```
*  RUN #    18      SEP 23, 1991  09:03:11
```



1 ppm METHANE

```
RUN#     18            SEP 23, 1991  09:03:11

MATHY CONSTRUCTION
```

26

```
ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .734   PB        57621   .169        5684   1R       3.261  METHANE


TOTAL AREA=  57621
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

```
*  PREP  CALIB 3  @
CAL#       RT          AMT
  1       0.753    1.996
  2       0.940    :
  3       1.584    :
  4       3.293    :
  5       7.560    :
  6      11.676    :

*
*
```

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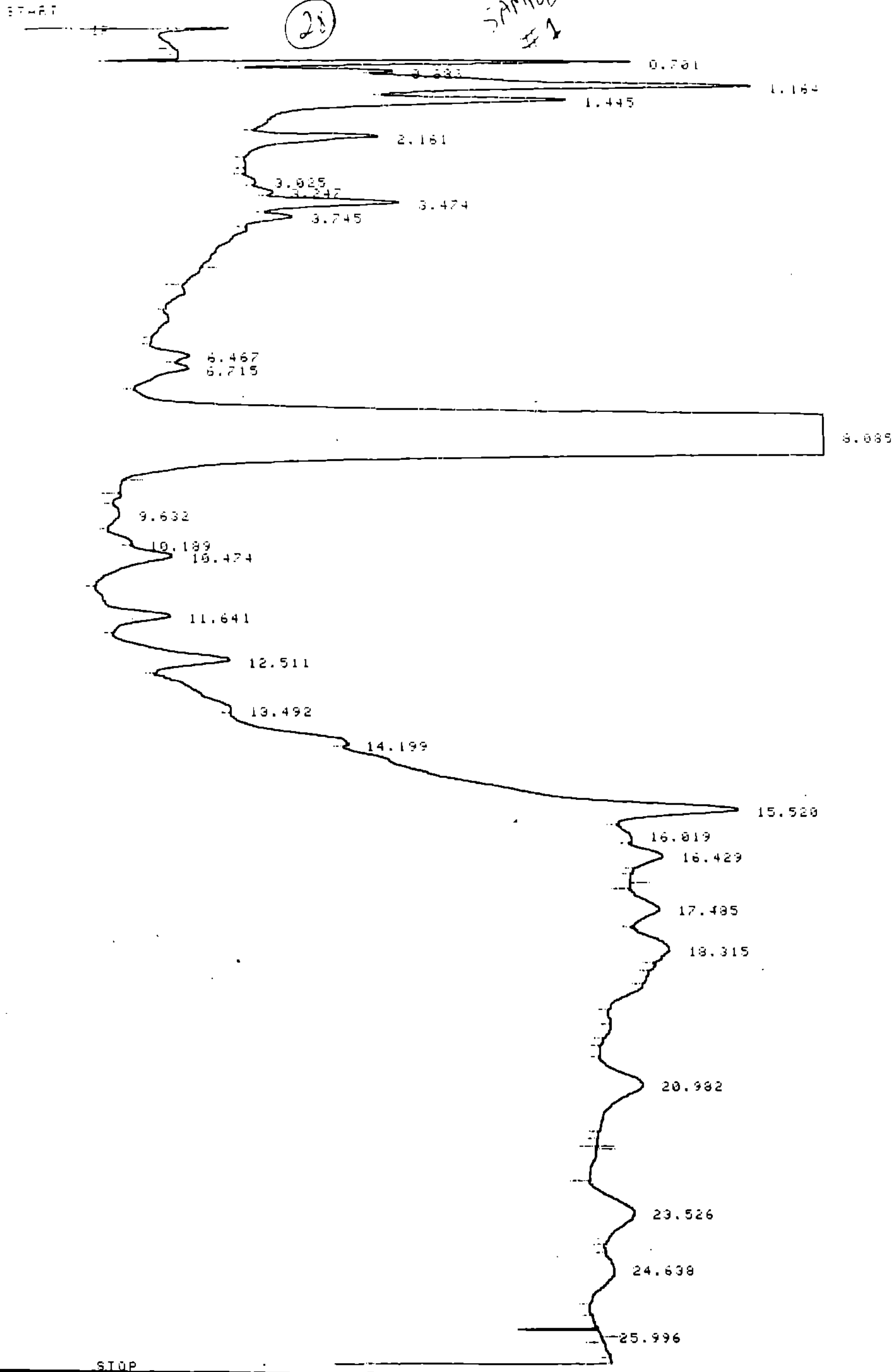
(21)

SAMPLE #1


START
0.781
0.883
1.164
1.445
2.161
3.025
3.247
3.474
3.745
6.467
6.715
8.085
9.632
10.189
10.474
11.641
12.511
13.492
14.199
15.520
16.019
16.429
17.485
18.315
20.982
23.526
24.638
25.996
STOP

```
RUN#     19          SEP 23, 1991   09:20:15

                               29

MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH       HEIGHT CAL# CONC.          NAME
     .701   PB      176764   .073        40405   1R         6.137  METHANE
     .833   BB       26900   .084         5345   2           .508  ETHANE
    1.164   BB      313106   .164        31844             12.280
    1.445   BB       83293   .086        17513   3          1.115  PROPANE
    2.161   BB      103236   .157        10948              4.049
    3.025   PB        4720   .131          602               .185
    3.247   BB        5419   .104          866               .213
    3.474   BB      100431   .146        11469              3.939         TTS
    3.745   BB       22642   .123         3078   4           .209  ~~PENTANE~~ BUTANE
    6.467   PB       15914   .132         2013               .624
    6.715   BB       21019   .178         1963               .824
    8.085   BB    16957376   .482       586847            665.068
    9.632   PB       16009   .273          979               .628
   10.189   BB        5266   .155          566               .207
   10.474   BB       71945   .271         4429              2.822
   11.641   BB       84622   .254         5560   6           .534  HEXANE
   12.511   BB      134633   .292         7686              5.280
   13.492   BB       20349   .760          446               .799
   15.520   BB      254630   .288        14738              9.987
   16.019   BB        7594   .186          681               .298
   16.429   BB       43805   .262         2785              1.718
   17.465   PB       54251   .357         2530              2.128
   18.315   BB       53982   .412         2185              2.117
   20.982   PB      131631   .569         3857              5.163
   23.526   BB      122163   .607         3355              4.791
   24.638   VB       49506   .560         1423              1.942


TOTAL AREA=1.8887E+07
MUL FACTOR=1.0000E+00



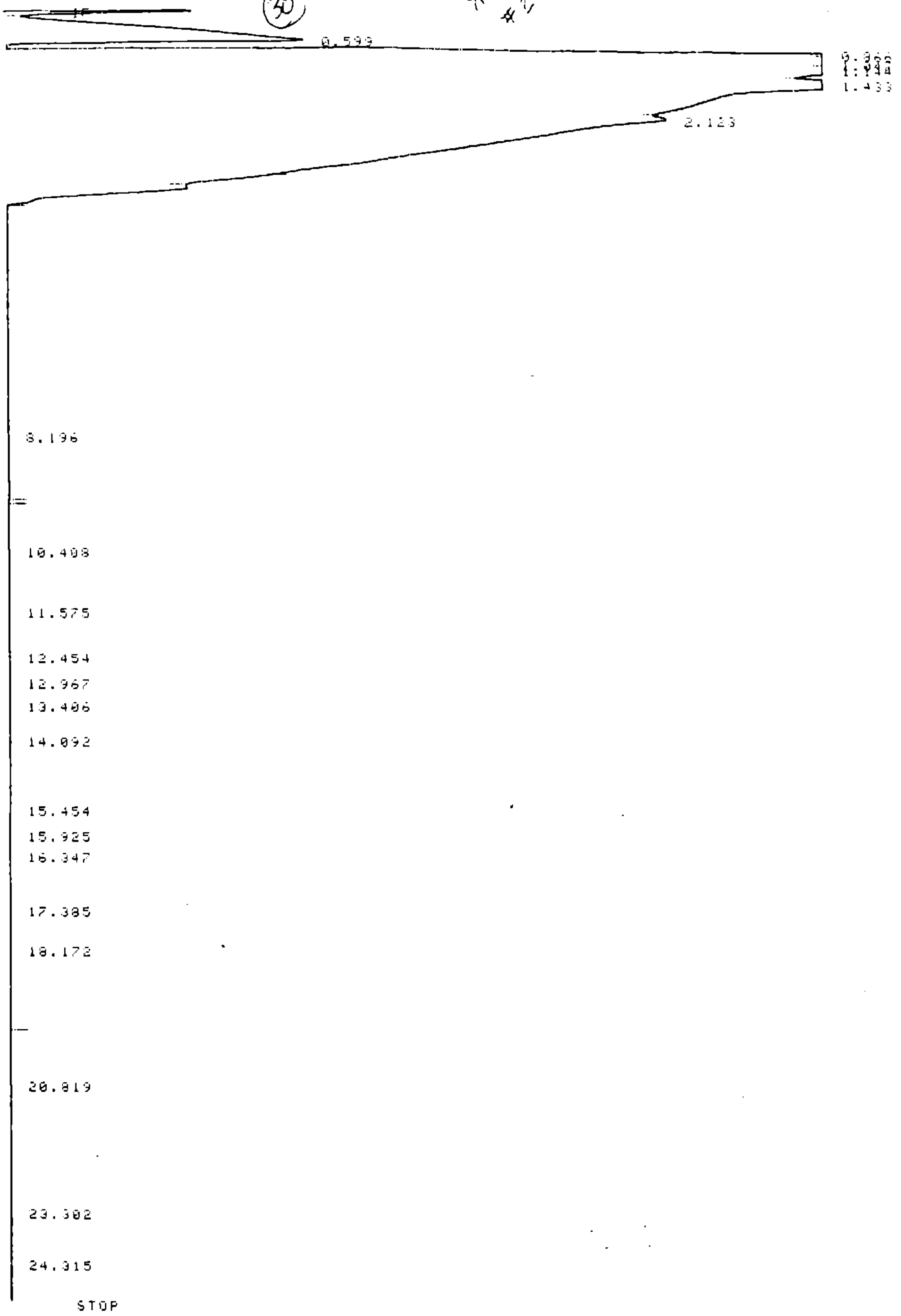

RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

* RUN # 30 SEP 23, 1991 09:51:38

START

(30)

Sample #2



(31)

```
MATH. CONSTRUCTION

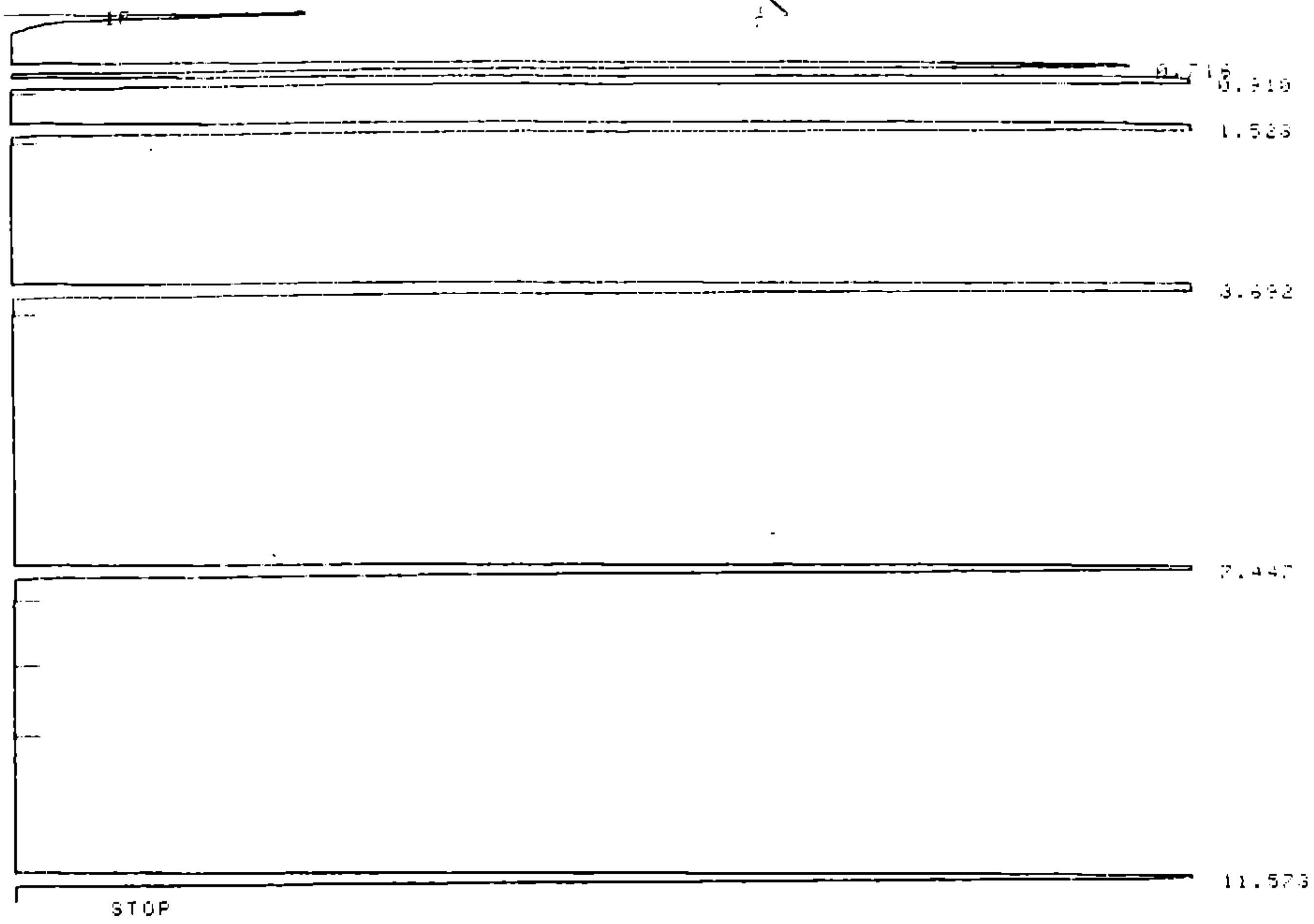
ESTD-AREA
       RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
     .599   BB     1167645   .287       67795          45.795
     .666   BB      221666   .125       30050           8.694
    1.047   BB       16265   .086        3146            .638
    1.144   BB       37329   .097        6404           1.464
    1.433   BB      128392   .163       13103           5.036
    2.123   BB      140377   .652        3587           5.506
    6.196   BB       20923   .145        2413            .821
   10.408   BB       27766   .140        3295           1.089
   11.575   PB       31173   .228        5924    6       .512   HEXANE
   12.454   BB      146710   .262        5666           5.754
   12.967   BB        1915   .074         434            .075
   13.406   BB       10445   .268         649            .410
   15.454   BB      220960   .235       15679           8.666
   15.925   BB       10582   .179         983            .415
   16.347   BB       47005   .286        2717           1.844
   17.385   VB       29216   .331        1473           1.146
   18.172   BB       94079   .531        2954           3.690
   20.319   BB      132660   .579        3819           5.203
   24.315   PB        1318   .182         121            .052


TOTAL AREA=2536429
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO     =   0
ATT 2^   =   3
CHT SP   =   1.0
AR REJ   =   500
THRSH    =   2
PK WD    =   0.12
```

```
* RUN #    21       SEP 23, 1991  10:27:32
START
```



```
RUN#    21          SEP 23, 1991  10:27:32

MATHY CONSTRUCTION          (32)

ESTD-AREA
      RT TYPE        AREA   WIDTH      HEIGHT CAL# CONC.          NAME
    .716   BB      395837   .092        71842   1R      15.525  METHANE
    .910   BB      793967   .092       143500   2       14.997  ETHANE
   1.528   BB     1171181   .103       189262   3       14.627  PROPANE
   3.692   BB     1621547   .161       167710   4       14.944  PENTANE ← BUTANE
   7.447   BB     2041939   .203       167388   5       14.982  PENTANE
  11.573 I BH     2355725   .222       176606   6       14.868  HEXANE

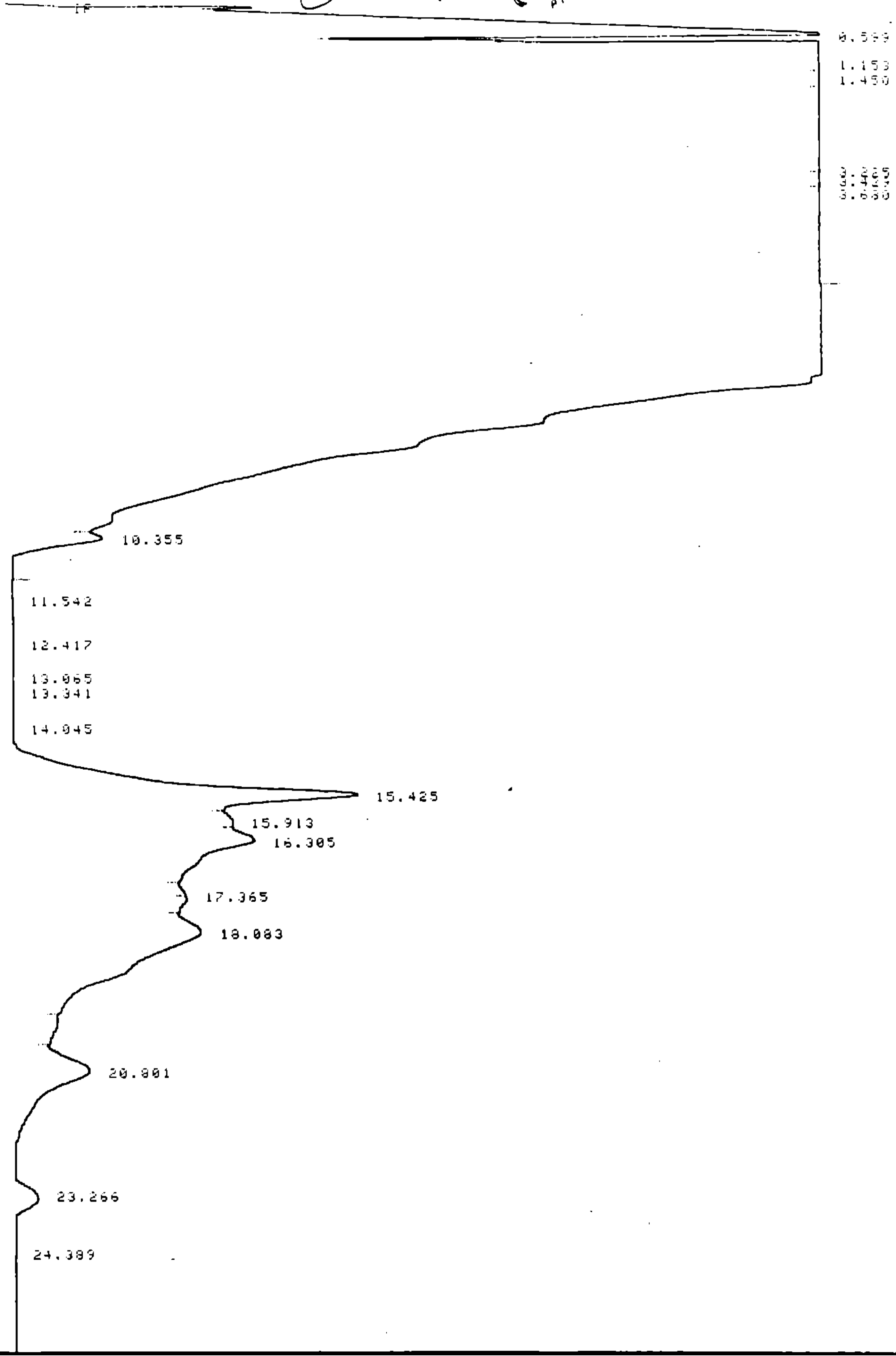
TOTAL AREA=8380195
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

* RUN # 33 SEP 25, 1991 10:41:53
START

(33)

SAMPLE #5 (SAMPLED AT 10:42)



```
RUN#     22              SEP 23, 1991  10:41:53

MATHY CONSTRUCTION

ESTD-AREA
     RT  TYPE        AREA   WIDTH        HEIGHT CAL# CONC.        NAME
    .599   PB      736557    .293         44697            30.349
   1.153   BB     1130099    .541         30692            46.253
   1.450   BB       97366    .162          9992             3.619
   3.225   BB      101107   1.149          1467             3.965
   3.429   BB       34620    .140          4129             1.358
   3.660   BB      821683   2.405          5694    4        7.530 ~~PENTANE~~ ITS BUTANE
  11.542   BB       50244    .176          4746    6         .317 HEXANE
  12.417   BB      134170    .274          6152             5.262
  13.341   BB        8307    .210           658              .326
  14.045   BB       11543    .064          2277              .453
  15.425   BB      308794    .230         15103             6.189
  15.913   BB        4042    .166           406              .159
  16.305   BB       45401    .252          3002             1.781
  17.365   BB         834    .101           137              .033
  18.085   VB      207036    .772          4468             8.120
  20.801   VB      187651    .690          4532             7.360
  23.266   BB      116676    .614          3168             4.576
  24.369   BB       56555    .646          1460             2.218

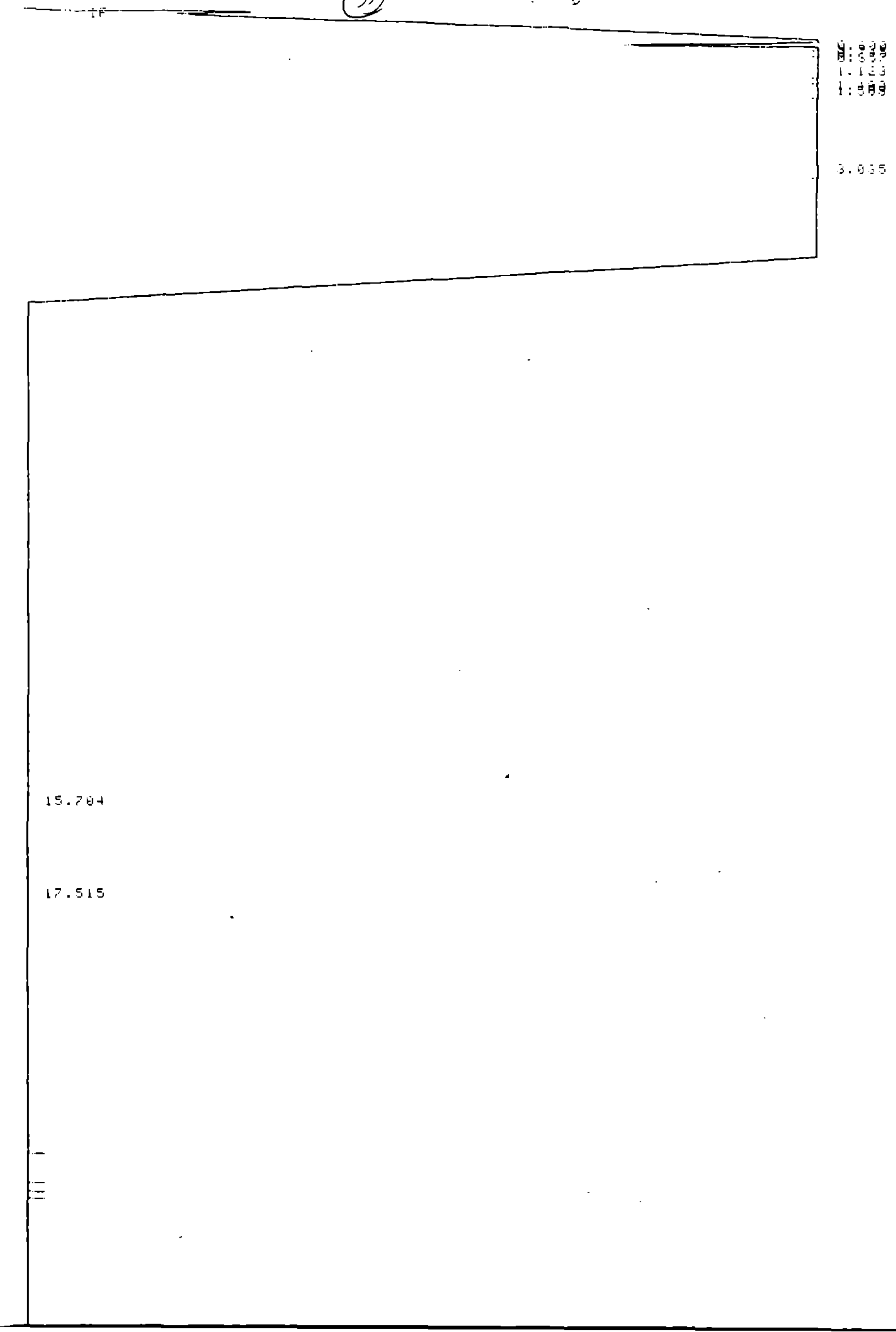
TOTAL AREA=4052685
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

(34)

* RUN # 33 SEP 25, 1991 [illegible]
START

33



```
RUN#    23           SEP 23, 1991  11:11:41
```

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```
MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .600   FB        450160   .345       21771            17.655
    .745   BB         31165   .045       11454   1R         .539  METHANE
    .857   BB         35667   .023       25633             1.407
   1.123   BB        219821   .201       18265             8.621
   1.400   BB          2956   .060         823              .116
   1.506   BB          9499   .033        4153              .373
   3.035   BB        272443   .666        6516            10.665
  17.515   FB          5073   .237         357              .199

TOTAL AREA=1027004
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

```
*   OF # 3

CALIBRATION OPTIONS
RF of uncalibrated peaks [3.9220E-05 ]:
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

*   OF # 3

CALIBRATION OPTIONS
RF of uncalibrated peaks [3.9220E-05 ]:
Replace calibration fit [Y/N*]: Y

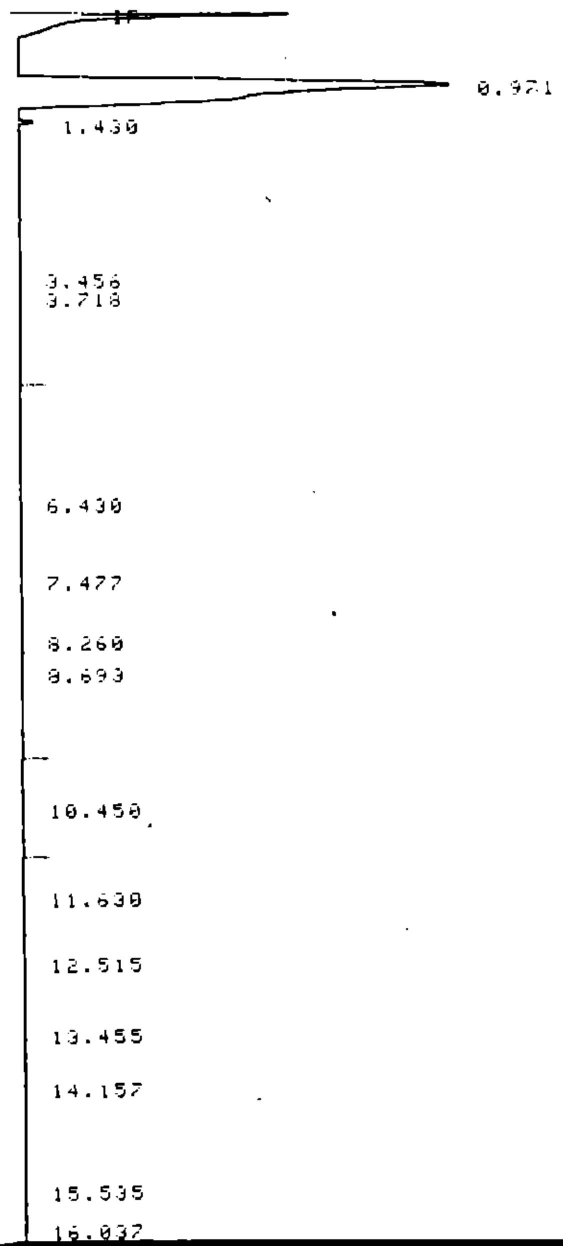
P = point-to-point
L = linear (least square)
N = non-linear (quadratic)

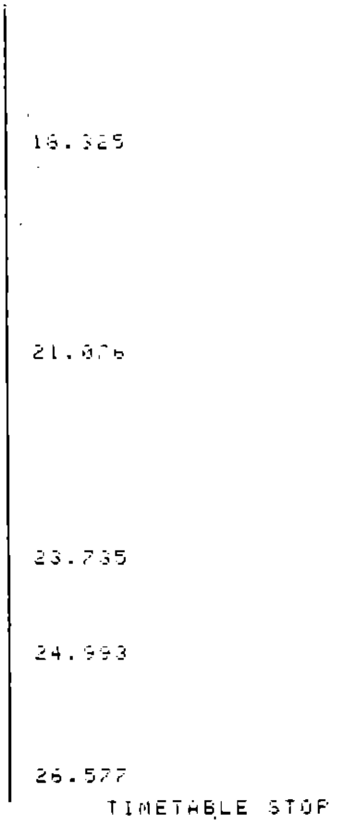
Calibration fit [N/L/P*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:
```

(37)

```
*  RUN #    24      SEP 23, 1991  11:48:56
START
```

SAMPLE #5





```
     TIMETABLE STOP


RUN#     24             SEP 23, 1991  11:48:56


MATHY CONSTRUCTION

ESTD-AREA
     RT TYPE         AREA   WIDTH      HEIGHT CAL# CONC.        NAME
    .971    BB    1166900    .377       51618            45.766
   1.430    BB      11458    .025        7668              .449
   3.456    PB      21914    .132        2758              .859
   6.430    BB      32624    .182        2991             1.280
   7.477    BB      45080    .188        3989    5         .329  PENTANE
   8.260    BB      36819    .200        3071             1.444
   8.693    BB      57329    .367        2606             2.248
  10.450    VB      54613    .241        3776             2.142
  11.630    BB      65364    .218        4996    6         .413  HEXANE
  12.515    BB     128000    .286        7457             5.020
  13.455    BB      11550    .271         710              .453
  14.157    BB       6338    .053        1980              .249
  15.535    BB     300334    .341       14668            11.779
  16.037    BB       6448    .181         595              .253
  16.474    BB      43546    .280        2591             1.708
  18.325    VB     235606    .985        3986             9.240
  21.076    BB     181625    .748        4049             7.123
  23.735    BB      81235    .560        2416             3.186
  24.993    PB      25858    .499         863             1.014


TOTAL AREA=2512642
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
```

㊳

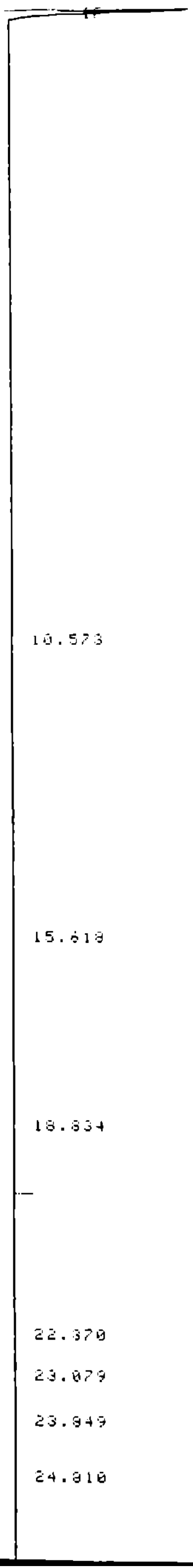
```
[illegible] = 500
[illegible] = 3
[illegible] = 0.12
```

RUN # 25 SEP 23, 1991 12:25:05

START

(40)

BTEX
~1.0 ppmv



```
        STOP

RUN#     25            SEP 23, 1991  12:25:35
```

41

```
MATH, CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT        AREA TYPE  WIDTH        AREA%
  10.575     106620    PB   .144   10.00456
  15.618     533799    BB   .378   50.08838
  18.634     197906    PB   .392   18.57027
  22.370       2635    PB   .636     .24725
  23.079       2829    BB   .437     .26546
  23.849       2864    BB   .442     .26874
  24.610     219061    BB   .701   20.55533

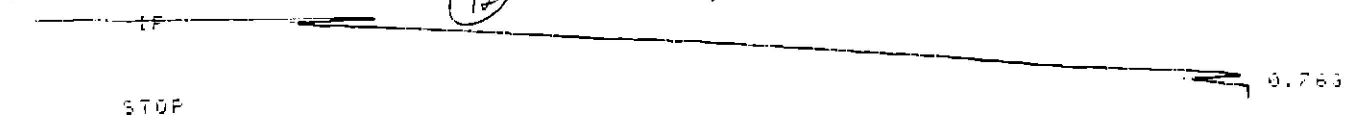
TOTAL AREA=1065714
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

```
* RUN #   26      SEP 23, 1991  12:55:55
START
```

(42)

METHANE



```
        STOP


RUN#     26            SEP 23, 1991  12:55:55


MATHY CONSTRUCTION

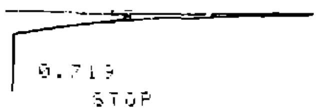
ESTD-AREA
       RT TYPE          AREA  WIDTH       HEIGHT CAL# CONC.         NAME
    .763   F6         129094   .351         6135   1R       4.030  METHANE


TOTAL AREA= 129094
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

```
* RUN #   27        SEP 23, 1991  13:03:03
START
```



(43) Methane 1.0 ppm

```
RUN#    27            SEP 23, 1991  13:03:03


MATHY CONSTRUCTION

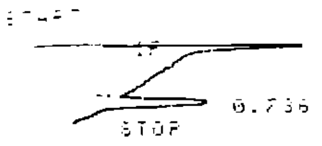
ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .719 I BP        32346   .101        .5348   1R        .559  METHANE


TOTAL AREA=  32346
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

```
- RUN #    25       SEP 25, 1991  13:04:39
```



47

```
RUN#     25             SEP 25, 1991  13:04:39


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH       HEIGHT CAL# CONC.         NAME
    .736 I BP         34165   .107         5333   1R        .591  METHANE

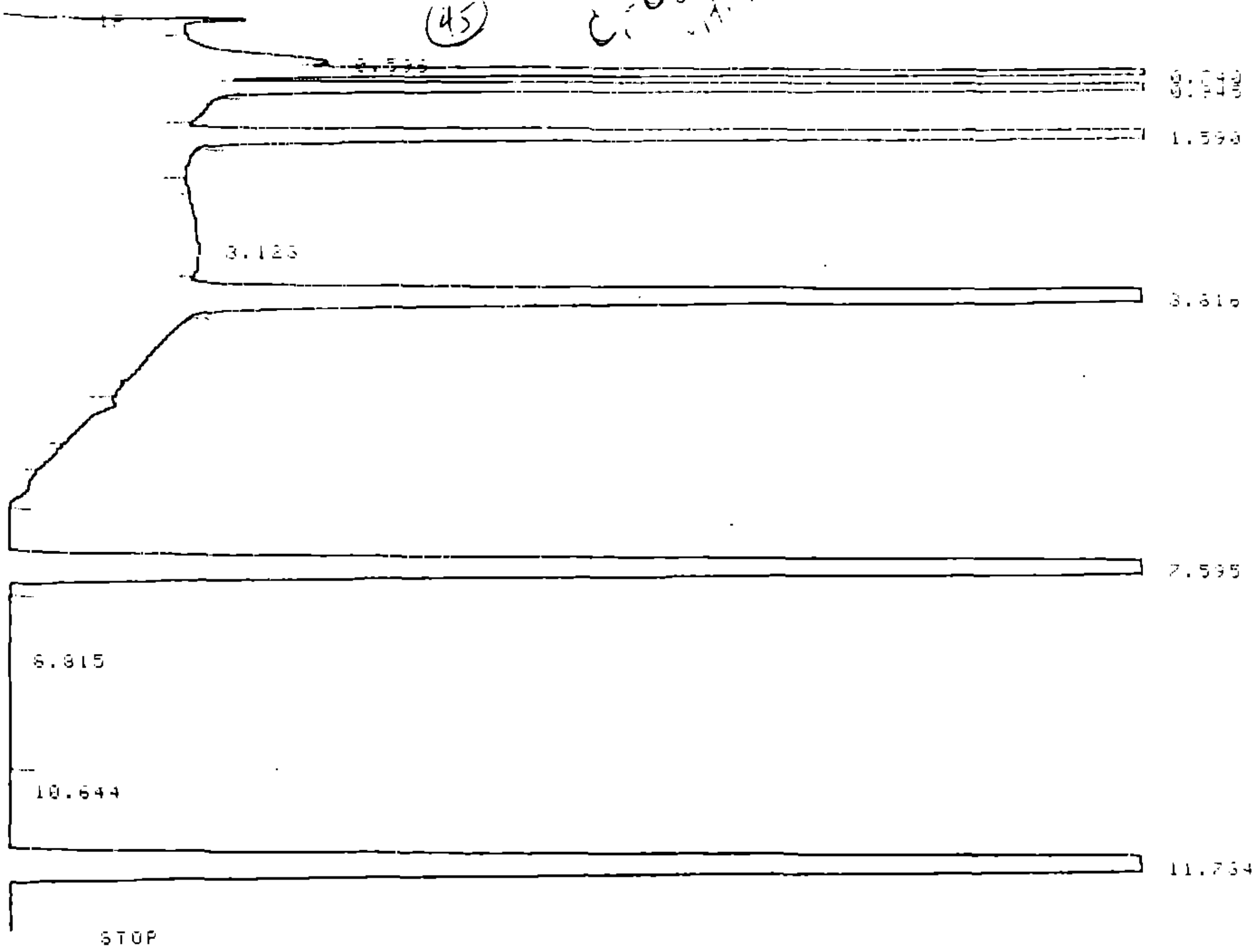

TOTAL AREA=  34165
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2   =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

```
* RUN #   29      SEP 23, 1991  13:08:12
START
```

(45) C1-C6



```
RUN#    29          SEP 23, 1991  13:08:12


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .740   BB      379577   .097       65384   1R      14.887  METHANE
    .945   BB      776304   .097      132791   2       14.664  ETHANE
   1.590   PB     1150298   .108      177341   3       14.365  PROPANE
   3.123   VB       28049   .746         627            1.100
   3.316   BB     1608069   .163      164311   4       14.825  PENTANE
   7.595   BB     2017564   .203      165918   5       14.704  PENTANE
   8.815   BB        3788   .070         902             .149
  10.644   BB        5962   .202         493             .234
  11.734   BB     2313738   .220      175322   6       14.603  HEXANE


TOTAL AREA=8283350
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
```

[illegible] = 0.12

46

```
*  EDIT  CALIB 3  0

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  1
RT:
AMT:
AMT/AREA:  23[illegible].99E-05
NAME:

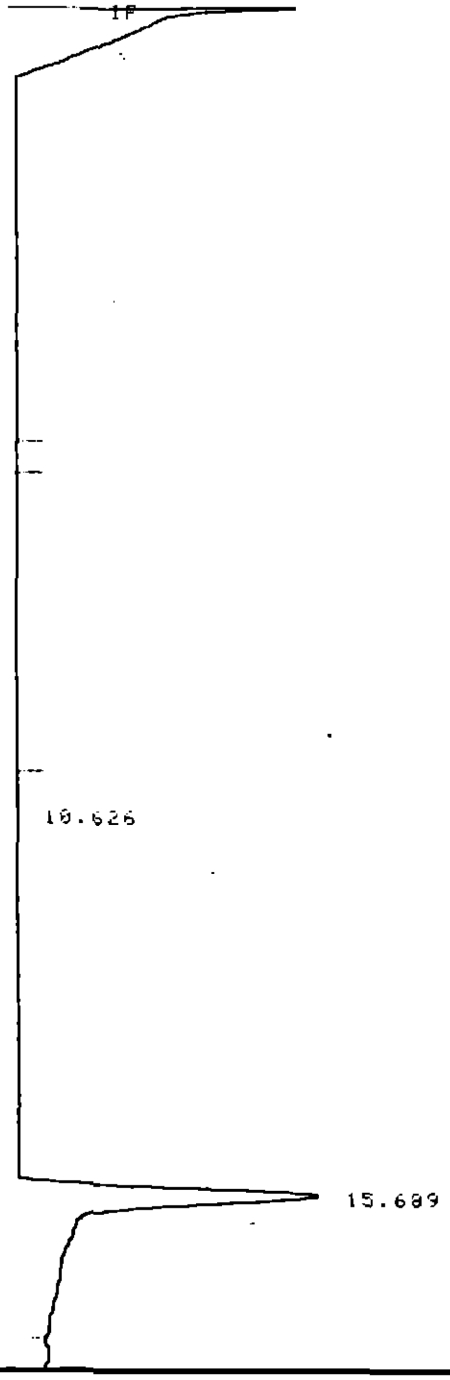
CAL#:

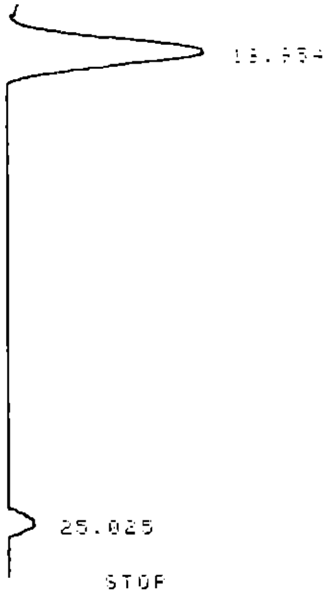
SECTION TO BE EDITED:

*  RUN #   30     SEP 23, 1991  13:30:20
START
```

BTEX
~1.0 ppmv

(47)





㊽

```
RUN#     30            SEP 23, 1991  13:30:20


MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT        AREA TYPE  WIDTH       AREA%
  10.626     177579   PB    .214    11.81853
  15.689     758208   BB    .442    50.46152
  18.954     286953   PB    .418    19.09777
  25.025     279807 I PP    .684    18.62218

TOTAL AREA=1502547
MUL FACTOR=1.0000E+00

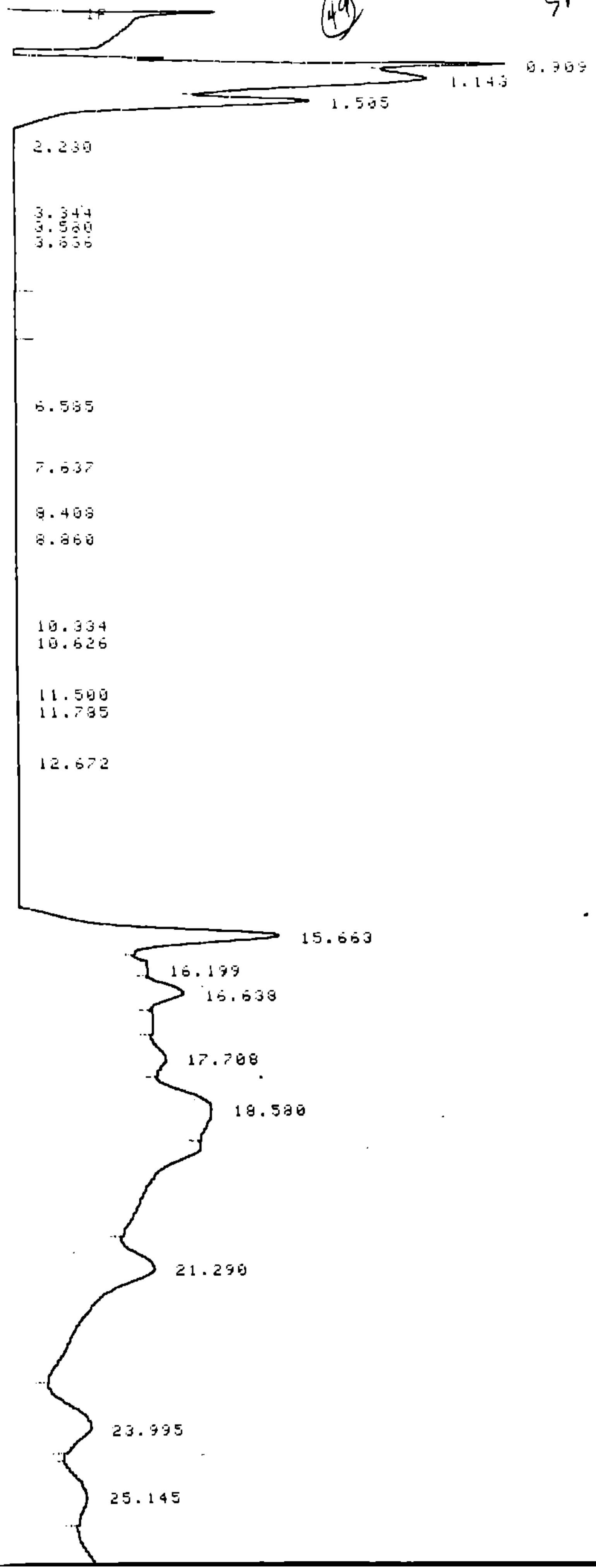

RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

* RUN # 31 SEP 23, 1991 14:07:09

START

(49)

SAMPLE #6



(50)

```
TIMETABLE STOP

RUN#    31          SEP 23, 1991   14:07:09

MATHY CONSTRUCTION

ESTD-AREA
      RT  TYPE         AREA   WIDTH      HEIGHT CAL# CONC.        NAME
     .909  PB        126672   .115        16390   2        2.397  ETHANE
    1.143  BB        110516   .310         8785            4.334
    1.505  BB         40301   .063        10660   3         .503  PROPANE
    2.230  BB         10491   .091         1919             .411
    3.344  PB          7733   .100         1289             .303
    3.560  BB         30557   .135         3760            1.198
    3.656  BB         22735   .129         2935   4         .310  ~~PENTANE~~ ← BUTANE
    6.585  BB         71269   .357         3329            2.795
    7.637  BB         71718   .254         4711   5         .523  PENTANE
    8.408  BB         34590   .195         2952            1.357
    9.360  BB         18317   .148         2069             .718
   10.334  PB          6537   .168          649             .256
   10.626  BB         52002   .243         3569            2.040
   11.500  BB           903   .104          145             .035
   11.785  BB         65005   .214         5284   6         .429  HEXANE
   12.672  BB        107496   .244         7351            4.216
   15.663  BB        112087   .126        14822            4.396
   16.199  BB          6854   .208          548             .269
   16.638  BB         45201   .233         2661            1.773
   17.708  PB         17186   .318          902             .674
   18.580  BB         81923   .548         2493            3.213
   21.290  PB        145060   .651         3713            5.689
   23.995  BB         90649   .606         2492            3.555
   25.145  PB         40436   .534         1262            1.586

TOTAL AREA=1319438
MUL FACTOR=1.0000E+00

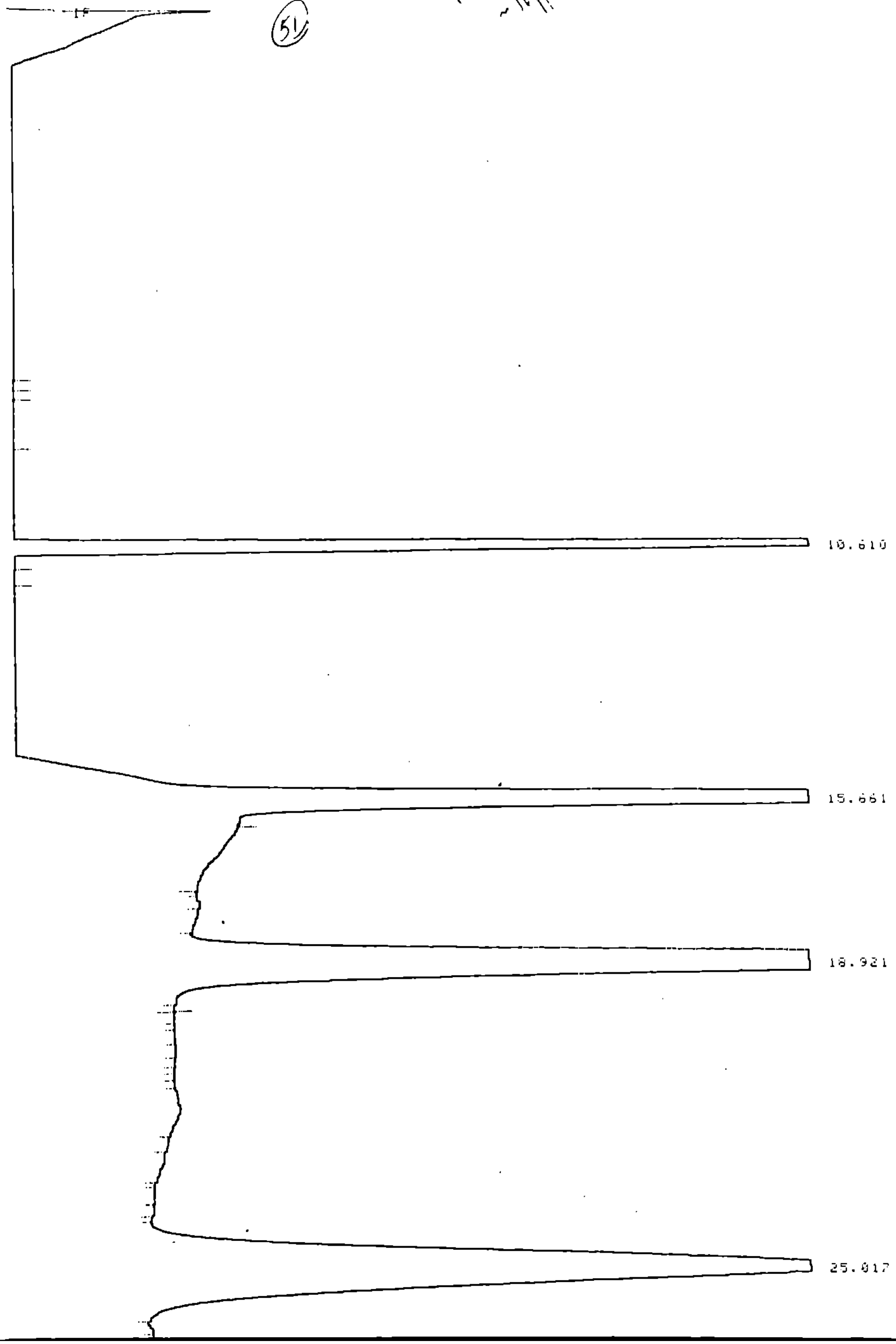


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

* RUN # 32 SEP 23, 1991 14:47:44
START

BTEX
~10 ppm

(51)



```
        TIMETABLE STOP


RUN#     32            SEP 23, 1991  14:47:44
```

52

```
MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT         AREA TYPE  WIDTH       AREA%
  10.610   1716856   BB    .215   21.39965
  15.661    875614   PB    .104   10.91404
  16.921   2806920   VB    .416   34.98667
  25.017   2623434   PB    .725   32.69963

TOTAL AREA=8022826
MUL FACTOR=1.0000E+00

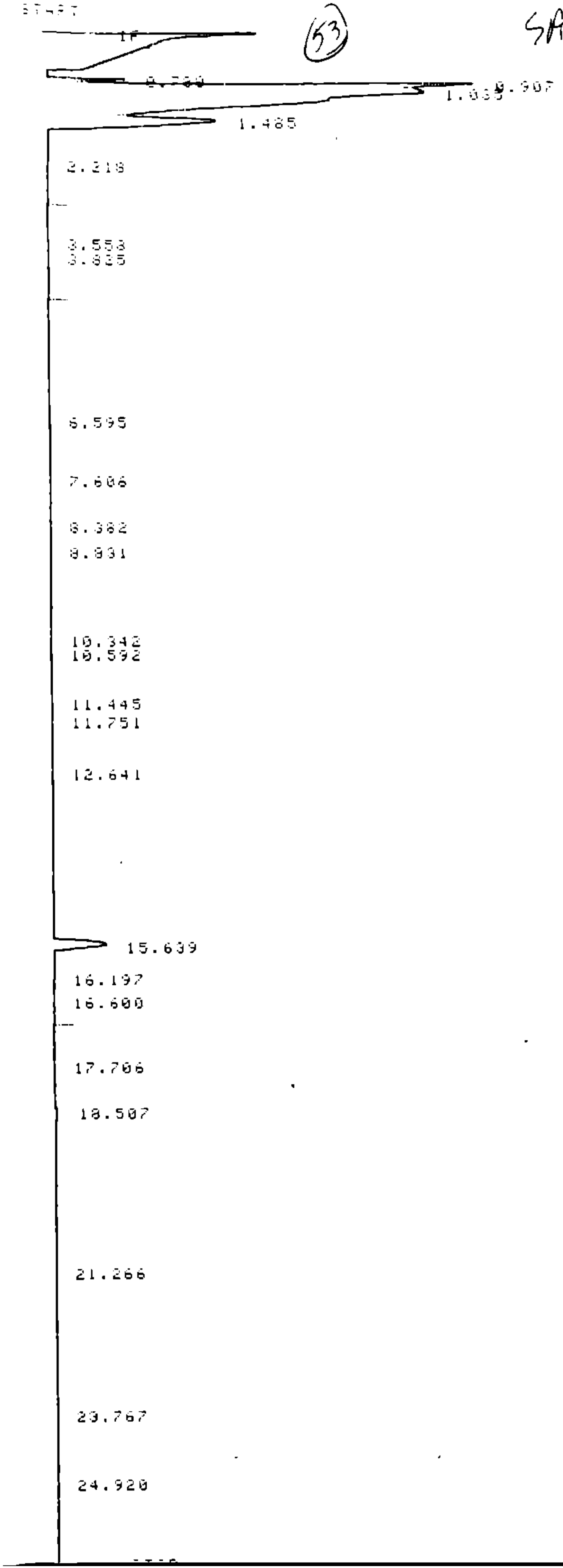


RUN PARAMETERS
ZERO     =  0
ATT 2^   =  3
CHT SP   =  1.0
AR REJ   =  500
THRSH    =  2
PK WD    =  0.12
```

RUN # 53 SEP 23, 1991 15:25:36

(53)

SAMPLE #7



```
RUN#    53          SEP 23, 1991  15:25:38

                        (54)

MATHY CONSTRUCTION

ESTD-AREA
      RT  TYPE          AREA  WIDTH        HEIGHT CAL# CONC.          NAME
     .730   PB         27441   .053          8560             1.076
     .907   BB         31503   .041         12927    2         .595   ETHANE
    1.035   BB         56967   .202          4702             2.234
    1.455   BB         31630   .061          8633    3         .396   PROPANE
    2.218   BB         11626   .116          1671              .456
    3.558   VB         24811   .141          2924              .973
    3.825   BB         15777   .107          2456    4         .146   PENTANE <—BUTANE
    6.595   BB         17269   .177          1624              .677
    7.606   PB         63886   .279          3818    5         .466   PENTANE
    8.362   BB         28081   .190          2457             1.101
    8.831   BB         21677   .209          1731              .850
   10.342   PB          1832   .122           251              .072
   10.592   BB         46933   .292          2793             1.919
   11.445   BB          2343   .207           189              .092
   11.751   BB         54989   .223          4105    6         .347   HEXANE
   12.641   BB         89965   .260          5768             3.528
   16.197   BB          4589   .180           425              .180
   16.600   BB         35004   .290          2012             1.373
   17.706   PB         13644   .321           718              .543
   18.507   BB         65499   .557          1959             2.569
   21.266   PB        223696   .996          3743             8.773
   24.920   PB          1710   .108           265              .067

TOTAL AREA= 873137
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

55

```
*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  3  BREAK

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  4
RT:
AMT:
AMT/AREA:
NAME:  BUTANE

CAL#:

SECTION TO BE EDITED:

*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1            RECALIBRATIONS: 1
LEVEL: 2            RECALIBRATIONS: 1
LEVEL: 3            RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1R     0.734     1 1.5000E+01   3.9247E-05
                   2 1.0250E+02   3.8591E-05
                   3 9.9600E-01   2.9900E-05
  2      0.927     1 1.4500E+01   1.8889E-05
  3      1.554     1 1.4200E+01   1.2487E-05
                   2 1.0040E+02   1.2553E-05
  4      3.772     1 1.4600E+01   9.2252E-06
                   2 9.7310E+01   8.8958E-06
  5      7.547     1 1.4600E+01   7.2881E-06
  6     11.665     1 1.4500E+01   6.3116E-06

CAL#     NAME
  1  METHANE
  2  ETHANE
  3  PROPANE
  4  BUTANE
  5  PENTANE
  6  HEXANE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
```

```
MUL FACTOR .................... 1.0000E+00

*  LIST: CALIB 3  @

ILLEGAL FILE NAME

*  LIST: CALIB 3  @

ILLEGAL FILE NAME

*  LIST: THRSH =  2

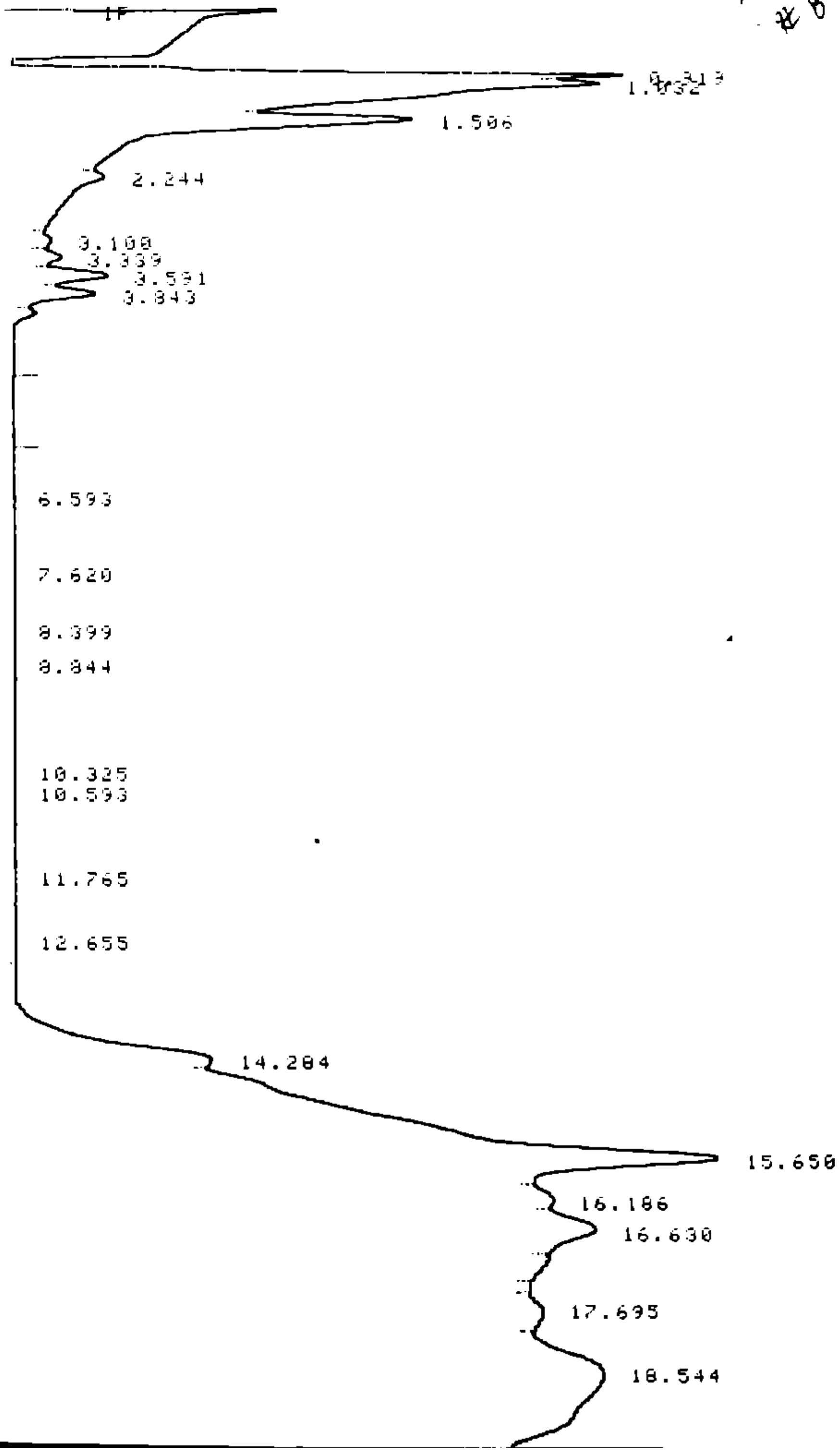
*  LIST: CALIB LEVEL 2  @

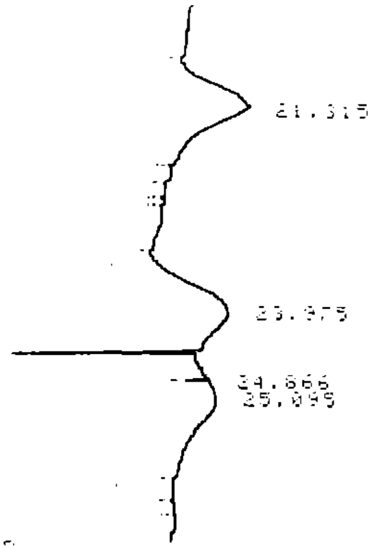
ILLEGAL FILE NAME

*  RUN #    34      SEP 23, 1991   16:02:50
START
```

(56)

SAMPLE
#8





57

```
TIMETABLE STOP


RUN#    34          SEP 23, 1991  16:02:50


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .919   PB       24444   .040       10164   2         .462  ETHANE
   1.032   BB       41600   .130        5344            1.632
   1.506   BB       50668   .081       10458   3         .633  PROPANE
   2.244   BB        5432   .076        1154             .213
   3.100   BB        3404   .111         510             .134
   3.339   BB        6670   .113         985             .262
   3.591   BB       28070   .138        3383            1.101
   3.843   BB       23553   .130        3029   4         .217  BUTANE
   6.593   BB       45044   .281        2671            1.767
   7.620   BB       75283   .266        4715   5         .549  PENTANE
   8.399   BB       36338   .201        3013            1.425
   8.844   BB       28868   .206        2336            1.132
  10.325   PB        2214   .062         597             .087
  10.593   BB       59824   .269        3713            2.346
  11.765   BB       66695   .219        5087   6         .421  HEXANE
  12.655   BB      106530   .246        7229            4.178
  15.650   BB      374514   .419       14886           14.688
  16.186   BB        6110   .174         584             .240
  16.630   BB       51506   .301        2856            2.020
  17.695   PB       11609   .253         750             .455
  18.544   BB      285316   .995        4780           11.190
  21.315   BB      144318   .593        4054            5.660
  23.975   VB      623919  1.049        9912           24.470
  24.866   BB      116327   .941        2060            4.562
  25.095   BB       70544   .623        1887            2.767


TOTAL AREA=2288800
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
```

F. NO = 5.12

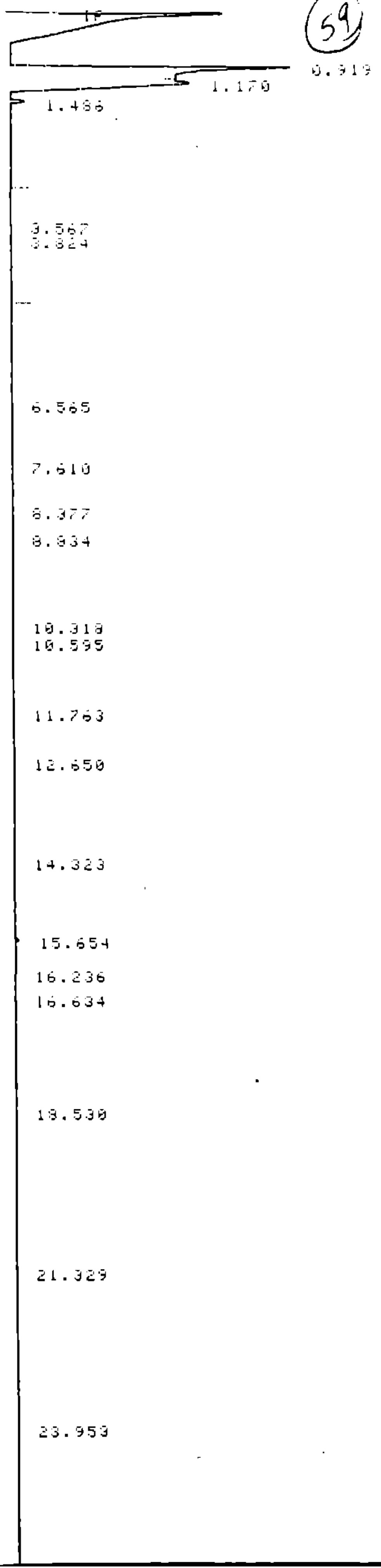
(58)

* RUN # 55 SEP 23, 1991 16:33:14

START

59

SAMPLE #9



```
| 26.809 TIMETABLE STOP


RUN#     35              SEP 23, 1991  16:39:14
```

60

```
MATHY CONSTRUCTION

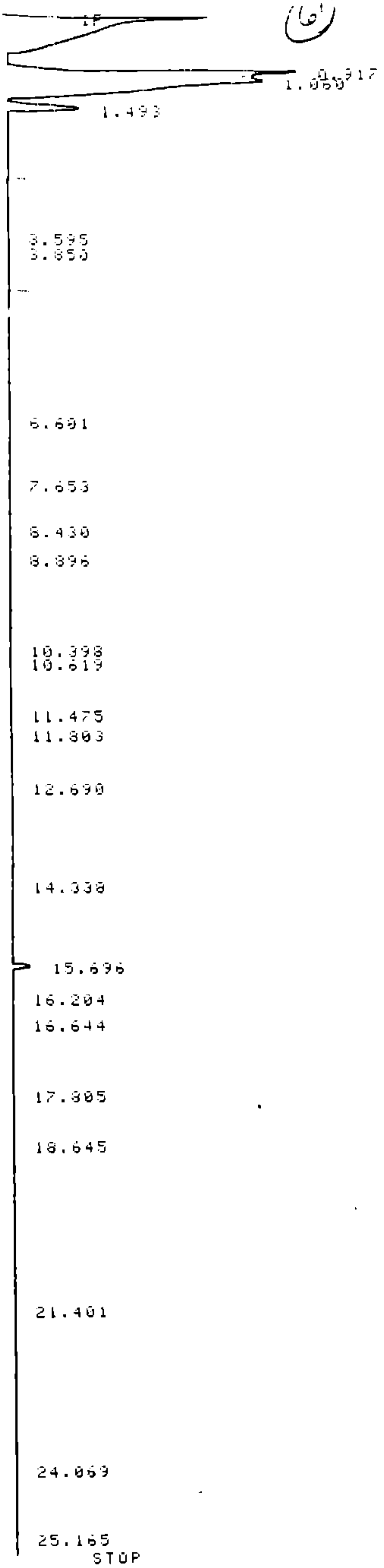
ESTD-AREA
      RT TYPE          AREA   WIDTH        HEIGHT CAL# CONC.         NAME
     .919   PB       147055   .115         21224    3       2.778  ETHANE
    1.170   BB        61973   .147          7019            2.431
    3.567   BB        23073   .134          2873             .905
    3.824   BB        26277   .137          3205    4        .242  BUTANE
    6.565   BB        62331   .328          3169            2.445
    7.610   BB        74658   .261          4773    5        .544  PENTANE
    8.377   BB        36971   .199          3094            1.450
    8.834   BB        26072   .202          2156            1.023
   10.318   PB         1968   .085           385             .077
   10.595   BB        53273   .275          3227            2.089
   11.763   BB        73008   .228          5332    6        .461  HEXANE
   12.650   BB       119972   .270          7412            4.705
   15.654   BB       341737   .406         14020           13.403
   16.236   BB         1910   .149           213             .075
   16.634   BB        51250   .301          2840            2.010
   18.530   PB        54164   .477          1891            2.124
   21.329   PB       188763   .714          4407            7.403
   23.953   PB       121183   .670          3015            4.753
   26.809   PB         3549   .261           227             .139


TOTAL AREA=1469187
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

* RUN # 36 SEP 23, 1991 17:16:41
START



6'

```
RUN#    36           SEP 23, 1991  17:16:41
```

62

```
MATHY CONSTRUCTION

ESTD-AREA
        RT TYPE          AREA  WIDTH       HEIGHT CAL# CONC.        NAME
      .917   PB         55188   .088        10410   2        1.042  ETHANE
     1.060   BB         46173   .160         4807            1.811
     3.595   PB         22729   .135         2902             .891
     3.850   BB         12113   .072         2815   4         .112  BUTANE
     6.601   BB         62376   .326         3186            2.446
     7.653   BB         69195   .244         4729   5         .504  PENTANE
     8.430   BB         36363   .198         3061            1.426
     8.896   BB         29641   .205         2407            1.163
    10.398   PB          2660   .144          308             .104
    10.619   BB         44520   .247         3006            1.746
    11.475   BB          1150   .107          179             .045
    11.803   BB         69960   .220         5304   6         .442  HEXANE
    12.690   BB        115840   .262         7361            4.543
    15.696   BB        310300   .372        13896           12.170
    16.204   BB         10180   .195          869             .399
    16.644   BB         38621   .252         2552            1.515
    17.805   BB         15906   .293          904             .624
    18.645   BB         64957   .509         2128            2.548
    21.401   PB         94280   .482         3257            3.698
    24.069   PB        114476   .645         2959            4.490
    25.165 I VH         24317   .446          909             .954


TOTAL AREA=1240945
MUL FACTOR=1.0000E+00

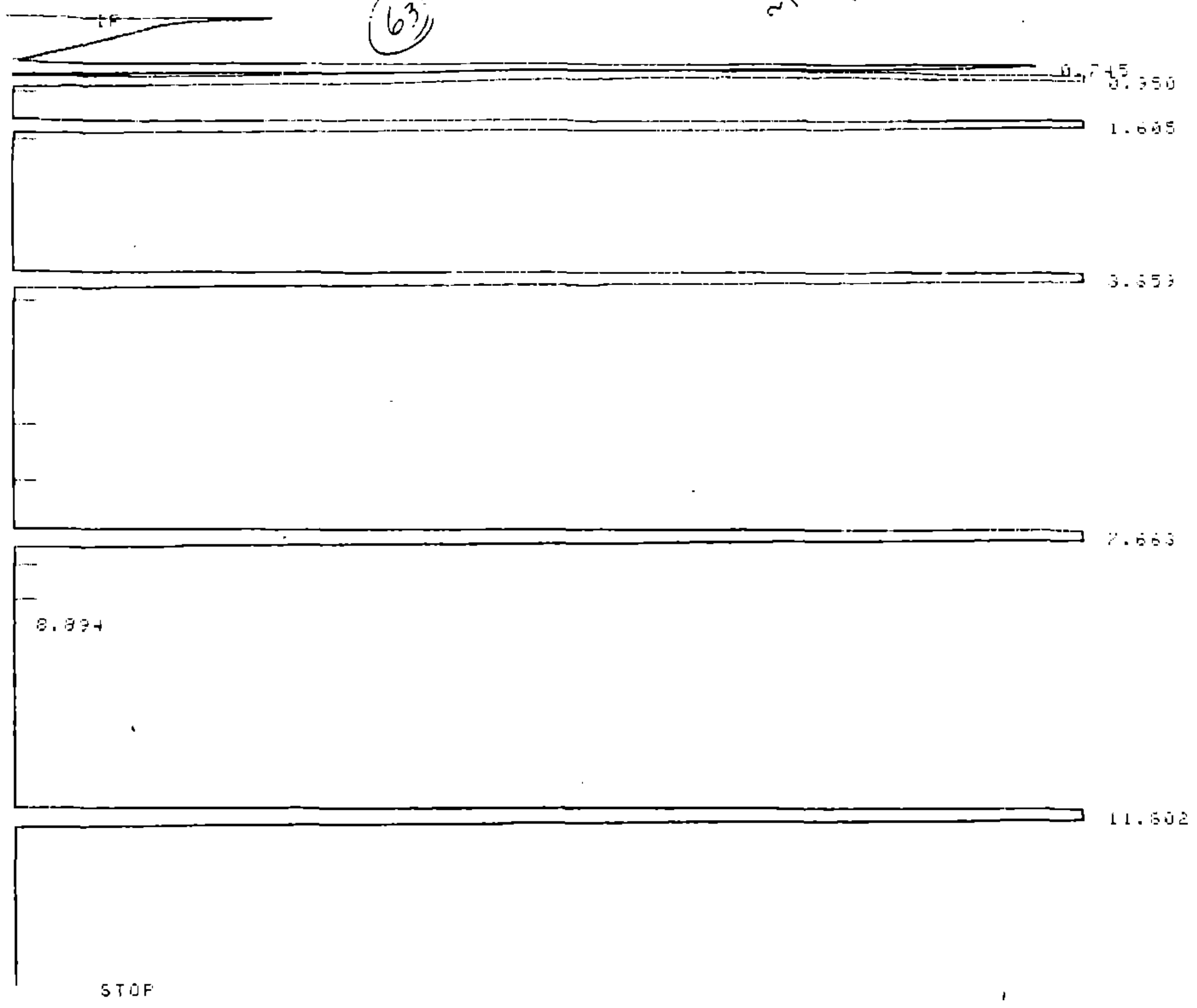


RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  2
PK WD   =  0.12
```

```
* RUN #    37      SEP 23, 1991  17:50:50
START
```

(63)

C1-C6
~14.5 ppm



```
RUN#     37           SEP 23, 1991   17:50:50


MATHY CONSTRUCTION

ESTD-AREA
      RT  TYPE        AREA   WIDTH      HEIGHT  CAL#  CONC.        NAME
     .745    BB      390619    .100       65201    1R      15.324  METHANE
     .950    BB      784042    .100      130551    2       14.810  ETHANE
    1.605    BB     1160382    .110      175048    3       14.491  PROPANE
    3.859    BB     1602791    .164      163040    4       14.778  BUTANE
    7.663    BB     2023450    .203      165812    5       14.747  PENTANE
    8.994    BB        4718    .080         981             .185
   11.802    PB     2298394    .219      174970    6       14.507  HEXANE


TOTAL AREA=8264397
MUL FACTOR=1.0000E+00

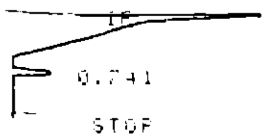


RUN PARAMETERS
ZERO    =   0
ATT 2^ =   3
```

```
HP REL  =  500
[illegible]  =  1
R, WD   =  0.14
```

64

```
*  RUN #   36      SEP 23, 1991  16:13:02
START
```



```
      0.741
       STOP
```

65

.998 ppm METHANE

```
RUN#    36           SEP 23, 1991  16:13:03


MATH, CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH       HEIGHT CAL# CONC.         NAME
    .741   BB        23526   .083         4747   1R         .703  METHANE

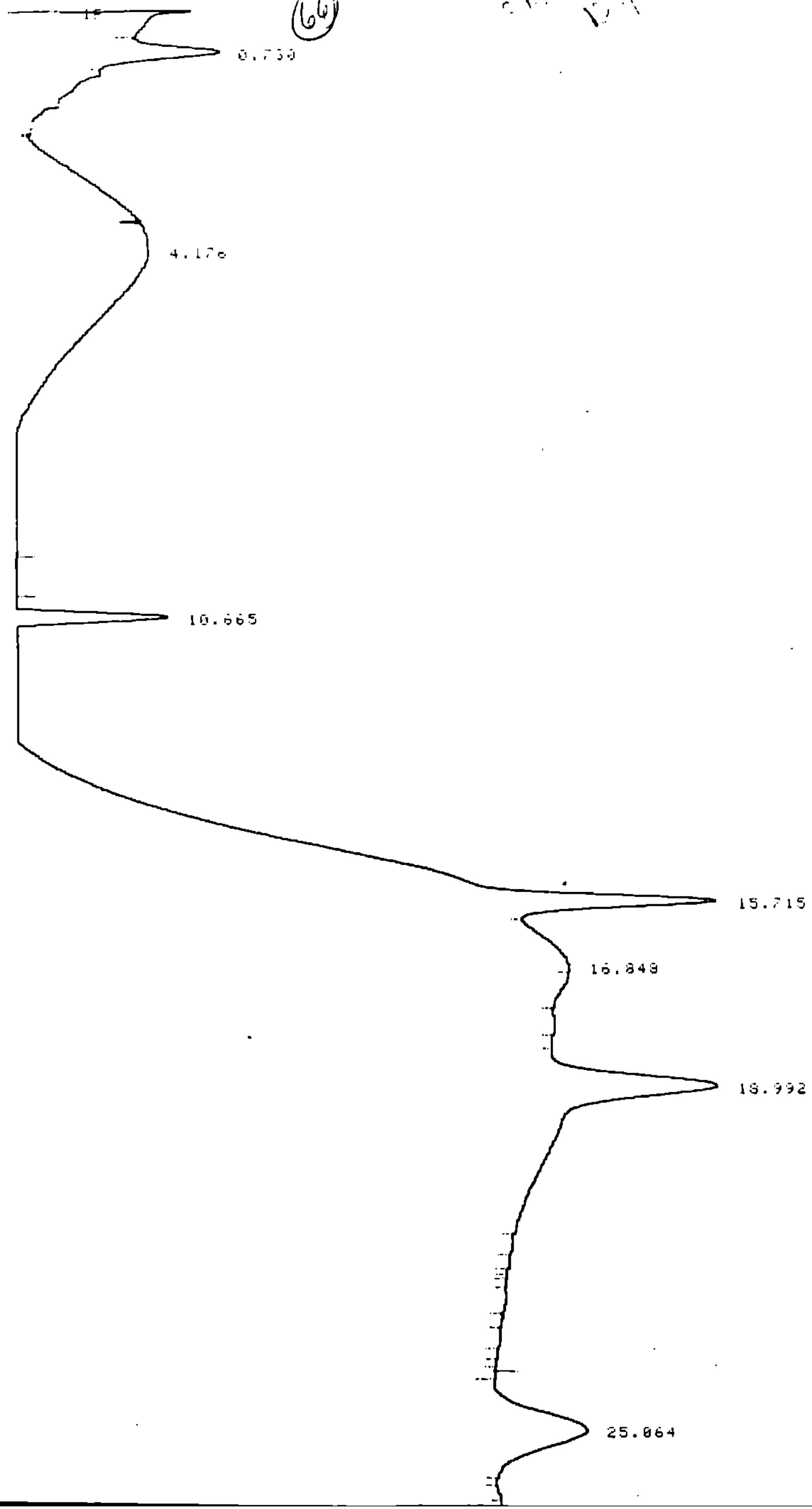

TOTAL AREA=  23526
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

* RUN # 39 SEP 23, 1991 15:16:34
START

(66)



```
     TIMETABLE STOP


RUN#    39          SEP 23, 1991  15:16:34

MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .730   BB      105555   .225        7322   1R       3.896  METHANE
   4.176   PB     1514176  2.669        9457           59.386
  10.665   BB      179310   .213       14030            7.033
  16.848   BB       25223   .858         490             .989
  18.992   PB      475559   .597       13368           18.651
  25.064   PB      291628   .695        6992           11.438


TOTAL AREA=2591451
MUL FACTOR=1.0000E+00

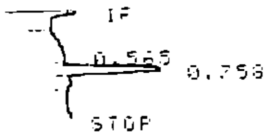


RUN PARAMETERS
ZERO    =   0
ATT 2^  =   3
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   2
PK WD   =   0.12
```

(62)

9/23 monday

```
*  RUN #    43        SEP 24, 1991  06:49:43
START: not read
```



18

```
RUN#     43             SEP 24, 1991  06:49:43


MATHY CONSTRUCTION

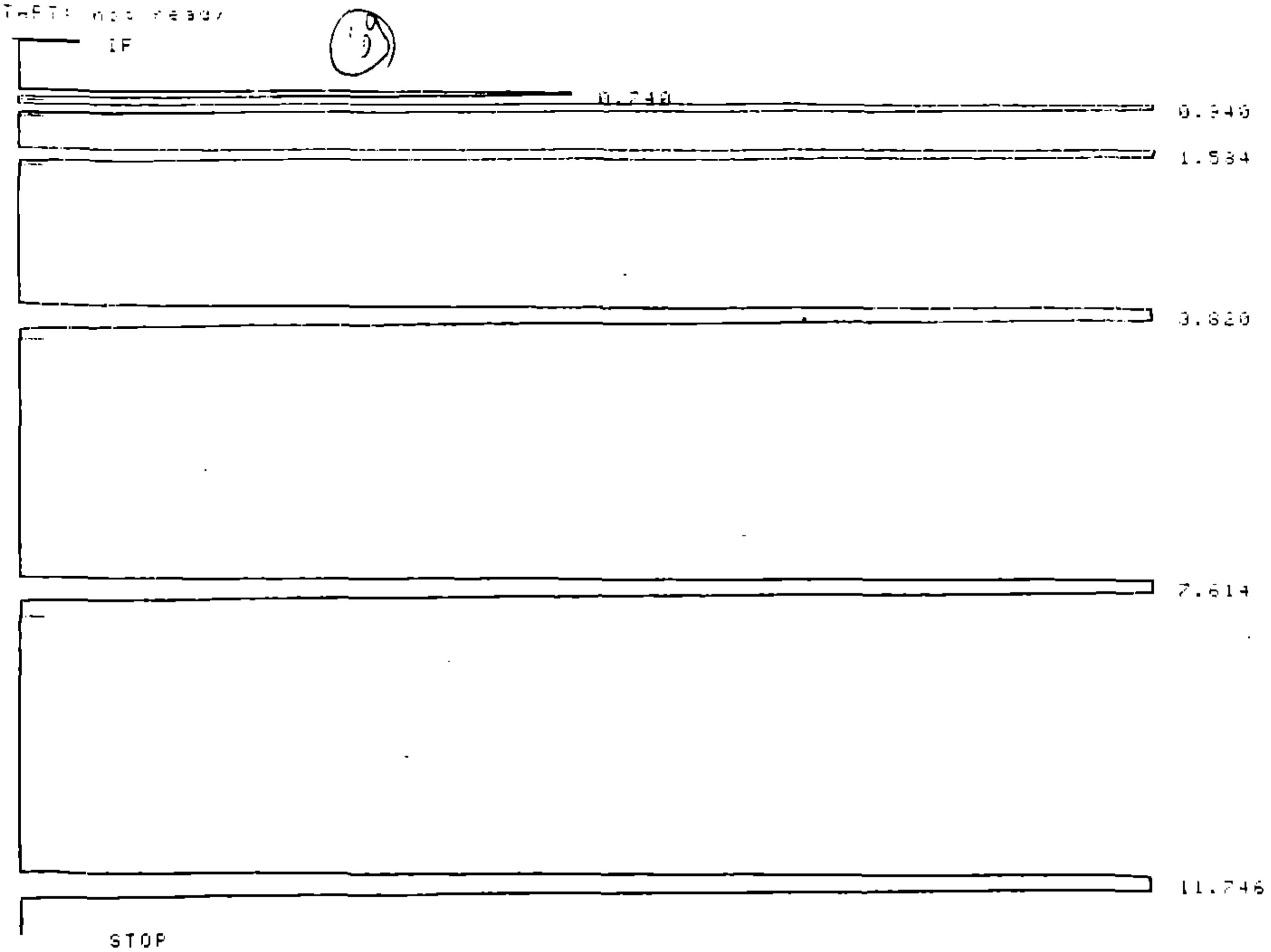
ESTD-AREA
      RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.          NAME
    .565   PB          3838   .251        255              .151
    .758   BB         31613   .099       5303   1R         .945  METHANE


TOTAL AREA=  35451
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  1
PK WD   =  0.12
```

```
*  RUN #    47        SEP 24, 1991  07:04:09
START: not ready
```



```
RUN#    47           SEP 24, 1991  07:04:09


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .740   BB       280278   .086       54453   1R       10.909  METHANE
    .940   BB       669257   .092      121640   2        12.642  ETHANE
   1.584   BB      1060181   .105      168556   3        13.238  PROPANE
   3.820   PB      2067947   .208      166069   4        18.390  BUTANE
   7.614   BB      1971943   .201      163485   5        14.372  PENTANE
  11.746   PB      2263114   .219      172129   6        14.284  HEXANE


TOTAL AREA=8312720
MUL FACTOR=1.0000E+00

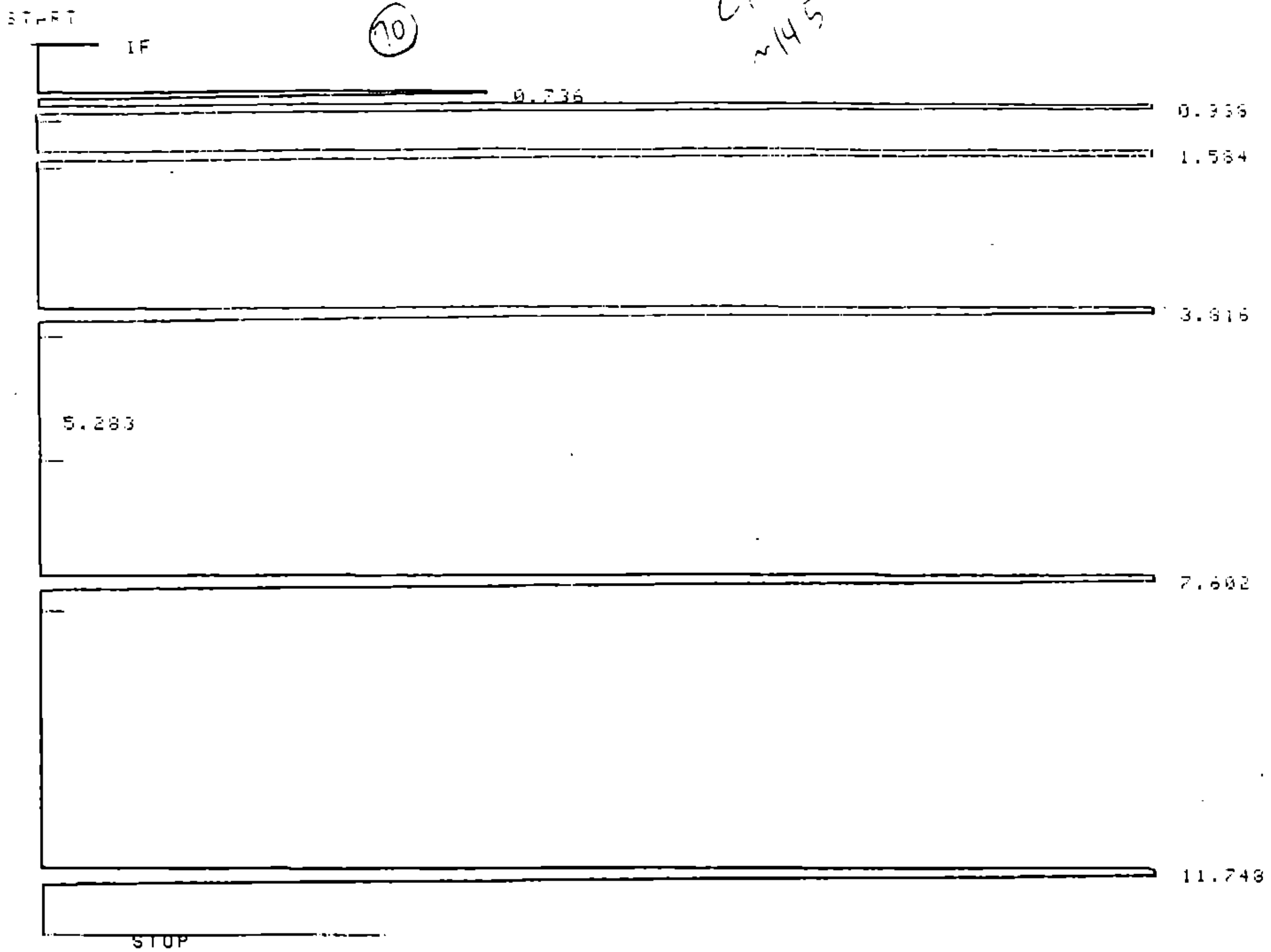


RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  0
PK WD   =  0.12
```

```
*  RUN #    48       SEP 24, 1991  07:24:40
```

(70)

C_1-C_6
~145



```
RUN#    48           SEP 24, 1991  07:24:40


MATHY CONSTRUCTION

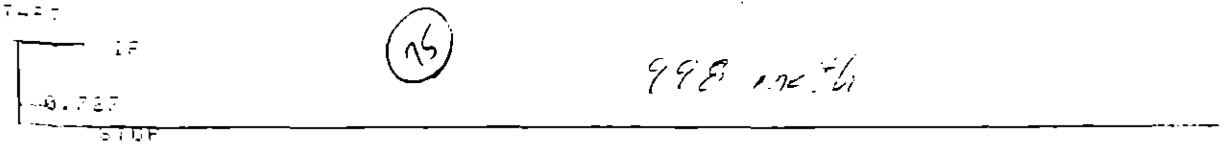
ESTD-AREA
       RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.         NAME
     .736    BB     389761   .096       67321   1R       15.291  METHANE
     .938    BB     779866   .097      133510   2        14.731  ETHANE
    1.584    BB    1151474   .108      177725   3        14.379  PROPANE
    3.816    BB    1594563   .163      163324   4        14.705  BUTANE
    5.283    BB       2118   .059         596              .083
    7.602    BB    1982490   .202      163956   5        14.449  PENTANE
   11.748    PB    2262450   .219      171959   6        14.280  HEXANE

TOTAL AREA=8162723
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  3
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  0
```

```
*  RUN #    62      SEP 24, 1991  08:23:19
START
```



(75)

998 mg/l

```
RUN#     62           SEP 24, 1991  08:23:19


MATHY CONSTRUCTION

ESTD-AREA
       RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.          NAME
     .727   BB         25177   .079        5282   1R          .753  METHANE


TOTAL AREA=  25177
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =   1
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   0
PK WD   =   0.08
```

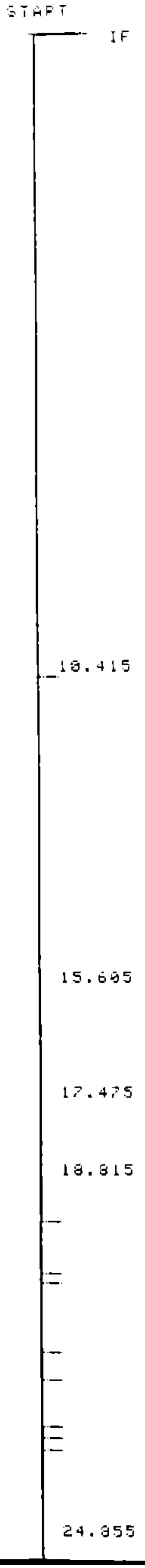
* THRSH BREAK

(16)

* RUN # 77 SEP 24, 1991 09:06:27

EX

C



```
TIMETABLE STOP


RUN#    77           SEP 24, 1991  09:08:27
```

77

```
MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT        AREA TYPE  WIDTH       AREA%
  10.415      66805   BB    .146    11.04814
  17.475     131763   BB    .800    21.79082
  18.815     212610   BB    .404    35.16122
  24.855     193494   PB    .689    31.99983

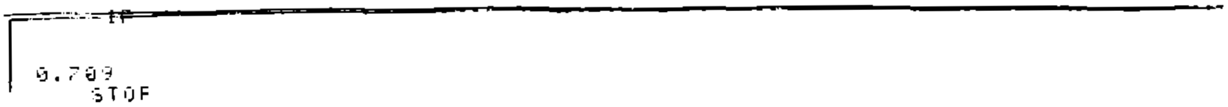
TOTAL AREA= 604672
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

HC T71
[illegible]

```
*  RUN #    78        SEP 24, 1991  09:46:54
START
```



78

```
RUN#    78           SEP 24, 1991  09:46:54


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .709 I BP        33610   .104        5399   1R       1.006  METHANE

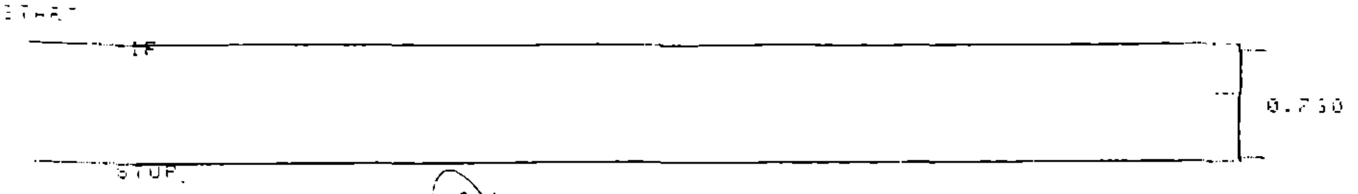

TOTAL AREA=  33610
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

1ppm

```
*  Run #    79        SEP 24, 1991  09:48:57
```



79

```
RUN#     79            SEP 24, 1991  09:48:57


MATHY CONSTRUCTION

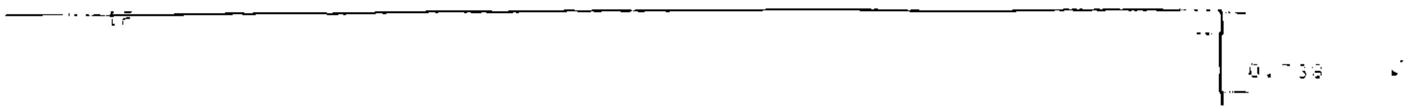
ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .730   BB       31484   .096        5456   1R       .941  METHANE


TOTAL AREA=  31484
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*  RUN #   60     SEP 24, 1991  09:52:15
START
```



```
      STOP
```

60

```
RUN#    60           SEP 24, 1991  09:52:15


MATHY CONSTRUCTION

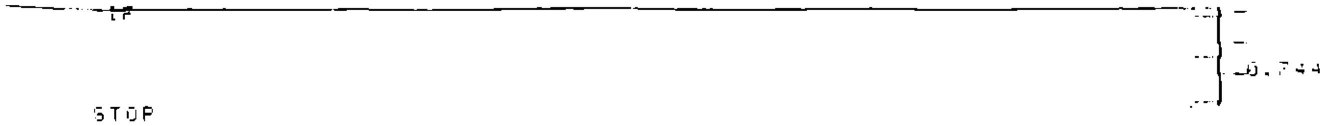
ESTD-AREA
       RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .738    BB      359426   .118       50680   1R      14.086  METHANE


TOTAL AREA= 359426
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*  RUN #    81       SEP 24, 1991  09:54:54
START
```



```
       STOP
```

(81)

```
RUN#     81             SEP 24, 1991  09:54:54


MATHY CONSTRUCTION

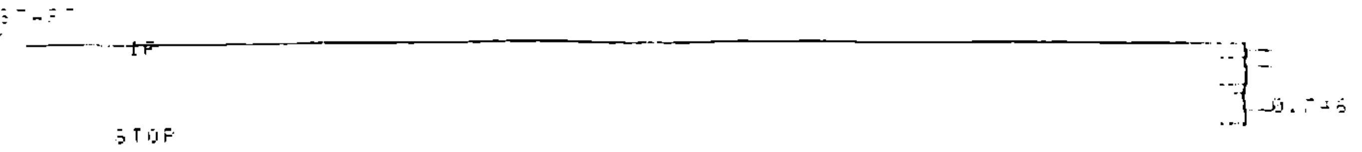
ESTD-AREA
       RT TYPE         AREA   WIDTH      HEIGHT CAL# CONC.         NAME
     .744   BB       259106    .098        44133   1R      10.059  METHANE


TOTAL AREA= 259106
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

```
*  RUN #   82        SEP 24, 1991  09:57:42
```



METHANE
905
ppm

(82)

```
RUN#    82           SEP 24, 1991  09:57:42


MATHY CONSTRUCTION

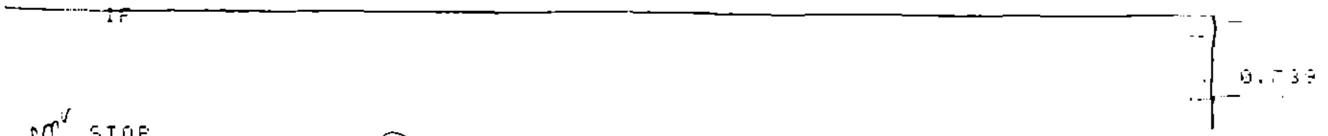
ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .746   PB      252516   .096       43964   1R       9.795  METHANE


TOTAL AREA= 252516
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*  RUN #    83        SEP 24, 1991   10:00:27
START
```



METZ
0.993 ppmv

```
STOP
```

(83)

```
RUN#     83              SEP 24, 1991   10:00:27


MATHY CONSTRUCTION

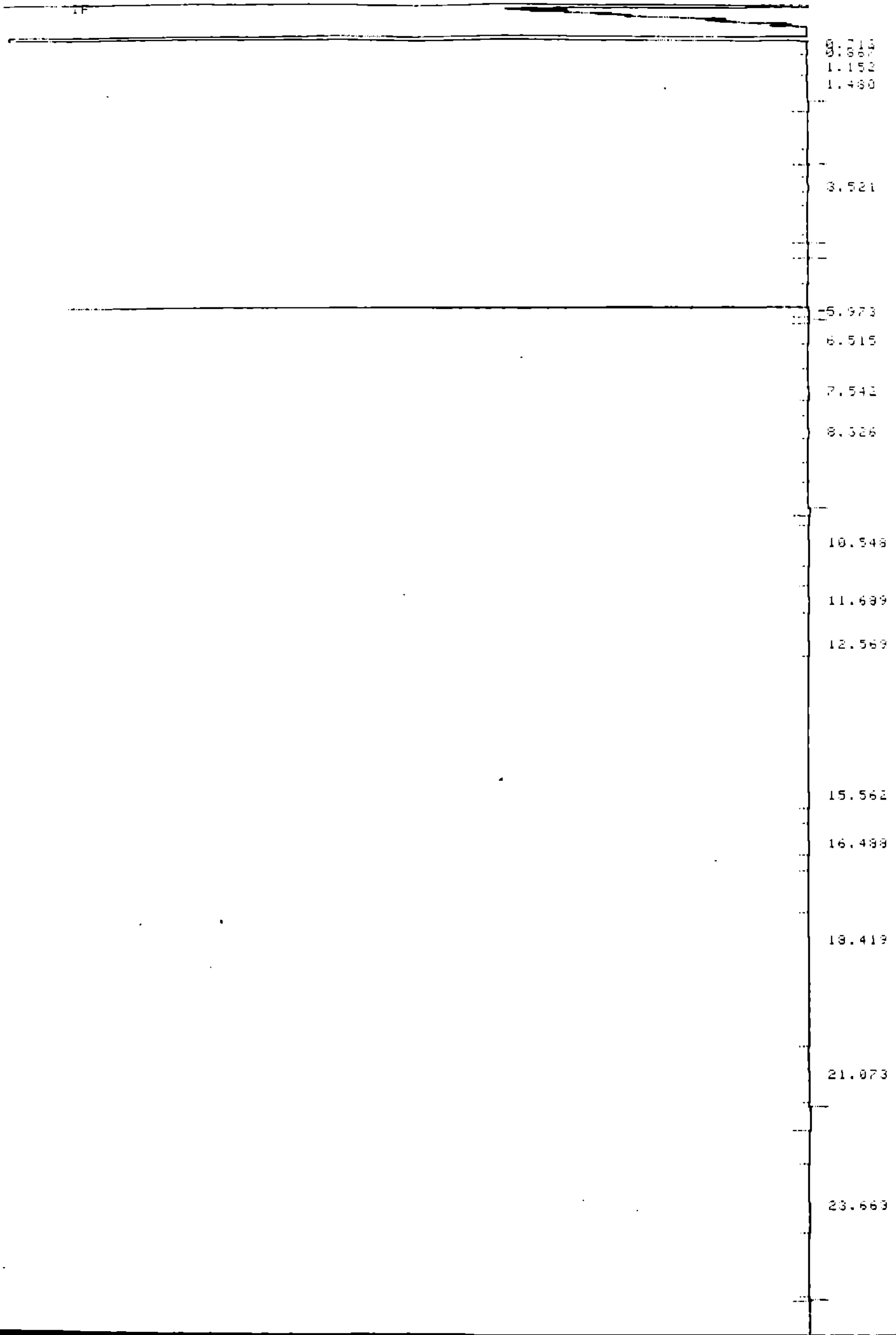
ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .739   BB        33715   .103        5448   1R       1.012  METHANE


TOTAL AREA=  33715
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

* RUN # 54 SEP 24, 1991 10:05:19 SAMPLE #1
START



(85)

```
          TIMETABLE STOP

RUN#     64          SEP 24, 1991  10:05:19


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE       AREA  WIDTH     HEIGHT CAL# CONC.       NAME
    .713   PB       1160   .014       1386   1R       .035  METHANE
    .867   BB     204035   .099      34264   2       8.854  ETHANE
   1.152   BB     115788   .177      10897           4.541
   1.480   BB      92201   .112      13781   3       1.151  PROPANE
   3.521   VB      45218   .151       5000   4        .417  BUTANE
   5.973   PB       9803   .046       3584            .384
   6.515   PB      13107   .158       1386            .514
   7.542   PB      36496   .211       2880   5        .266  PENTANE
   8.326   PB      20743   .190       1823            .814
  10.548   VB      95204   .316       5016           3.734
  11.669   PB      47026   .219       3577   6        .297  HEXANE
  12.569   BB      76889   .253       5072           3.016
  16.488   VB      42066   .323       2168           1.650
  18.419   VB     309760  1.340       3852          12.149
  21.073   BB      72808   .498       2435           2.856
  23.663   PB      88731   .623       2373           3.480

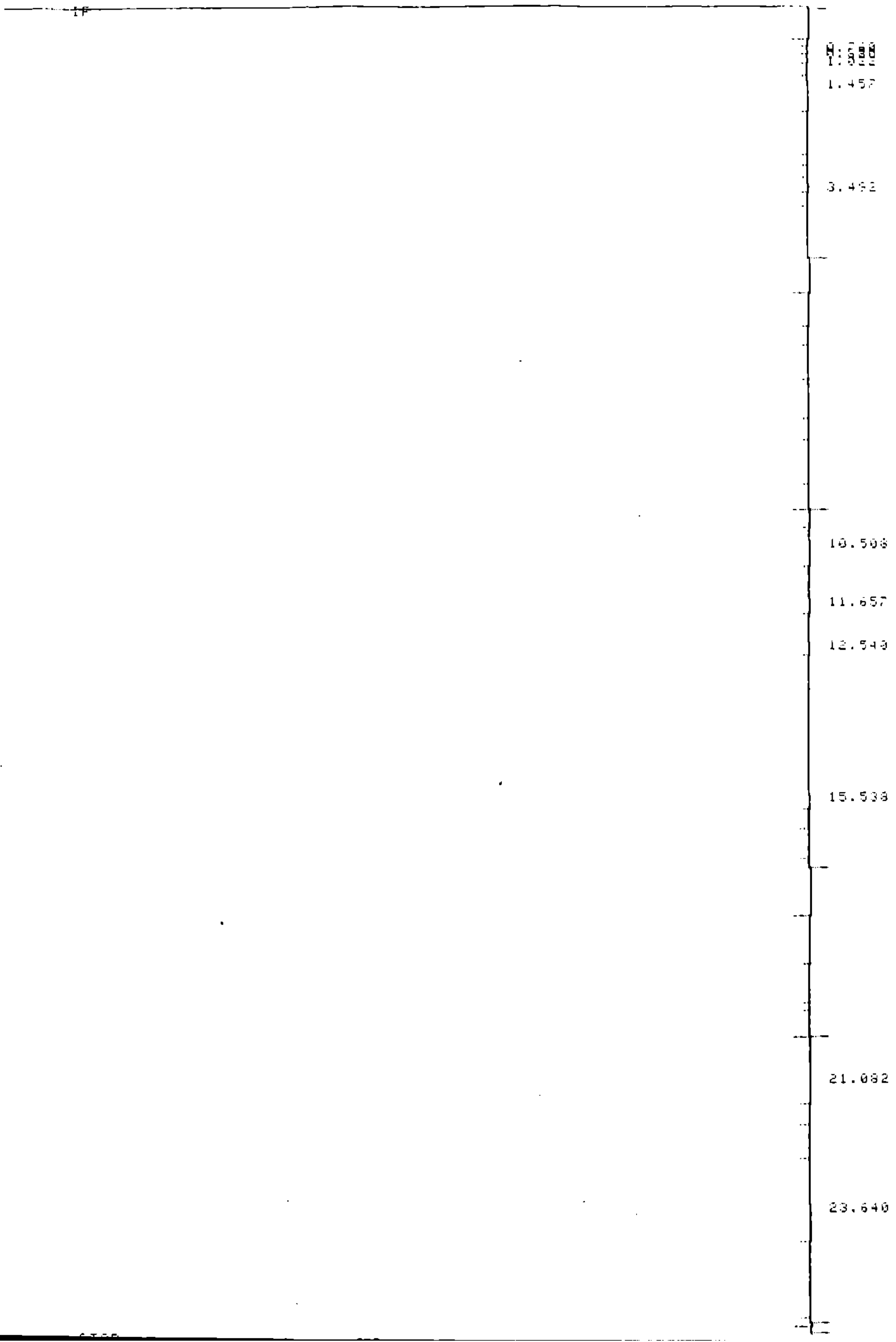

TOTAL AREA=1271035
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

* RUN # 85 ⑧ SEP 24, 1991 10:44:20 SAMPLE # 2

START



```
RUN#     65            SEP 24, 1991  10:44:20

MATHY CONSTRUCTION

ESTD-AREA
       RT  TYPE          AREA   WIDTH       HEIGHT  CAL#  CONC.         NAME
     .760    PB         17632    .050         5659    1R          .527  METHANE
     .880    BB         69309    .074        15636               2.718
    1.022    BB         24195    .061         4927                .949
    1.457    PB         76044    .094        13479               2.982
    3.492    VB         39776    .143         4625               1.560
   10.508    VB         90833    .355         4265               3.562
   11.657    BB         47909    .219         3647               1.879
   12.540    BB         72312    .241         5007     6          .456  HEXANE
   21.082    PB         73624    .514         2366               2.888
   23.640    PB        103506    .667         2586               4.060


TOTAL AREA= 615140
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO     =  0
ATT 2^   = -2
CHT SP   =  1.0
AR REJ   =  500
THRSH    =  4
PK WD    =  0.04
```

87

(88)

```
*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1              RECALIBRATIONS: 1
LEVEL: 2              RECALIBRATIONS: 1
LEVEL: 3              RECALIBRATIONS: 1

CAL#     RT      LV    AMT         AMT/AREA
  1R    0.736    1 1.5000E+01  3.9247E-05
                 2 1.0250E+02  3.8591E-05
                 3 9.9600E-01  2.9900E-05
  2     0.916    1 1.4500E+01  1.8889E-05
  3     1.549    1 1.4200E+01  1.2487E-05
                 2 1.0040E+02  1.2553E-05
  4     3.732    1 1.4600E+01  9.2252E-06
                 2 9.7310E+01  8.8958E-06
  5     7.577    1 1.4600E+01  7.2881E-06
  6    11.918    1 1.4500E+01  6.3116E-06

CAL#     NAME
  1  METHANE
  2  ETHANE
  3  PROPANE
  4  BUTANE
  5  PENTANE
  6  HEXANE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  EDIT  CALIB 3  @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  11
RT:
AMT:
AMT/AREA:  3.435E-05
NAME:

CAL#:

SECTION TO BE EDITED:

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS
```

```
CAL#:  1
RT:
AMT:
AMT/AREA:  3.549E-05
NAME:

CAL#:  2
RT:
AMT:
AMT/AREA:  2.013E-05
NAME:

CAL#:  3
RT:
AMT:
AMT/AREA:  1.286E-05
NAME:

CAL#:  4
RT:
AMT:
AMT/AREA:  9.156E-06
NAME:

CAL#:  5
RT:
AMT:
AMT/AREA:  7.383E-06
NAME:

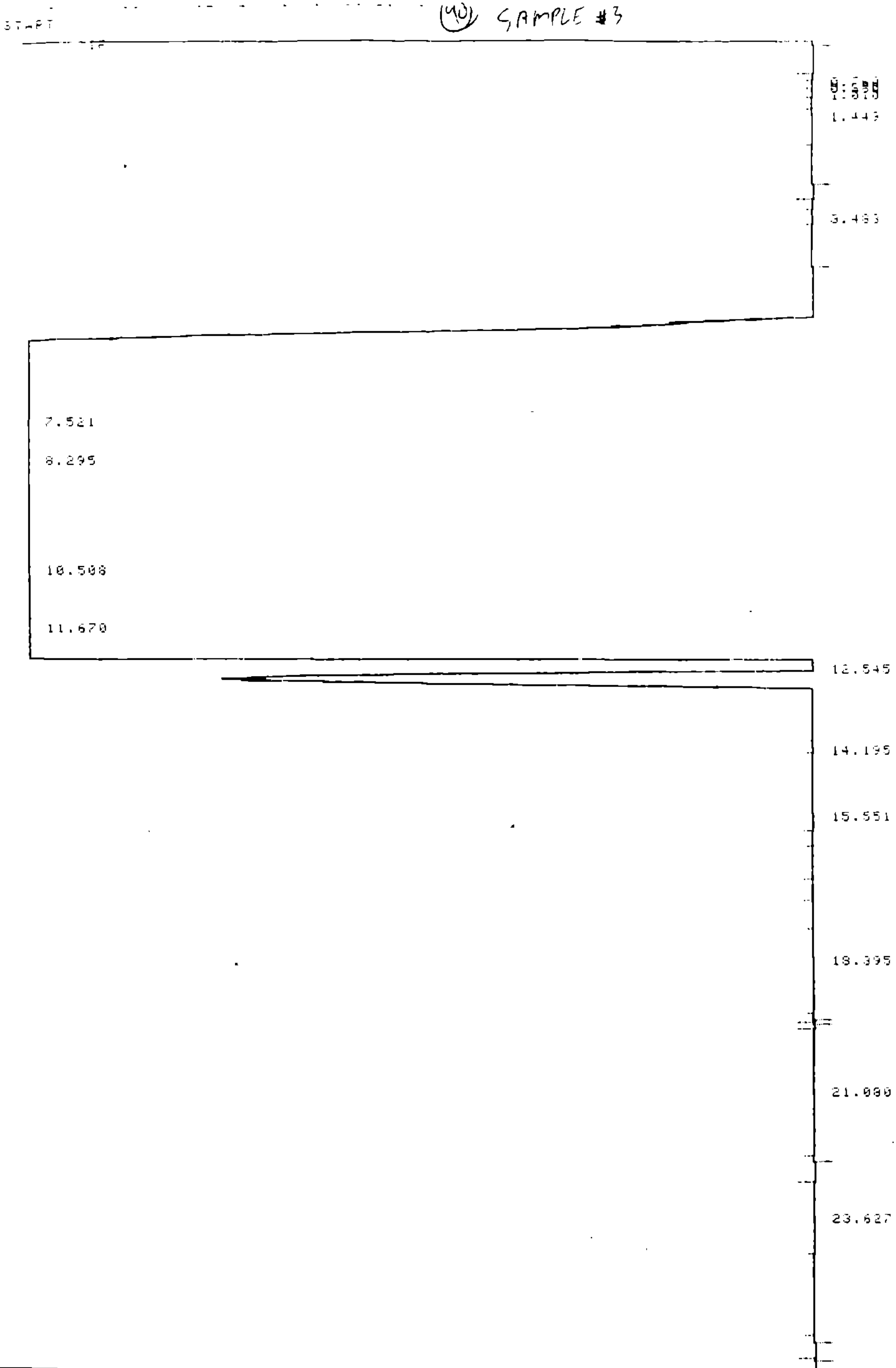
CAL#:  6
RT:
AMT:
AMT/AREA:  6.4075E-06
NAME:

CAL#:

SECTION TO BE EDITED:

*
```

(40) SAMPLE #3


START
0.588
1.315
1.443
3.483
7.521
8.295
10.508
11.670
12.545
14.195
15.551
18.395
21.080
23.627

```
RUN#    66          SEP 24, 1991  11:21:43
```

91

```
MATHY CONSTRUCTION

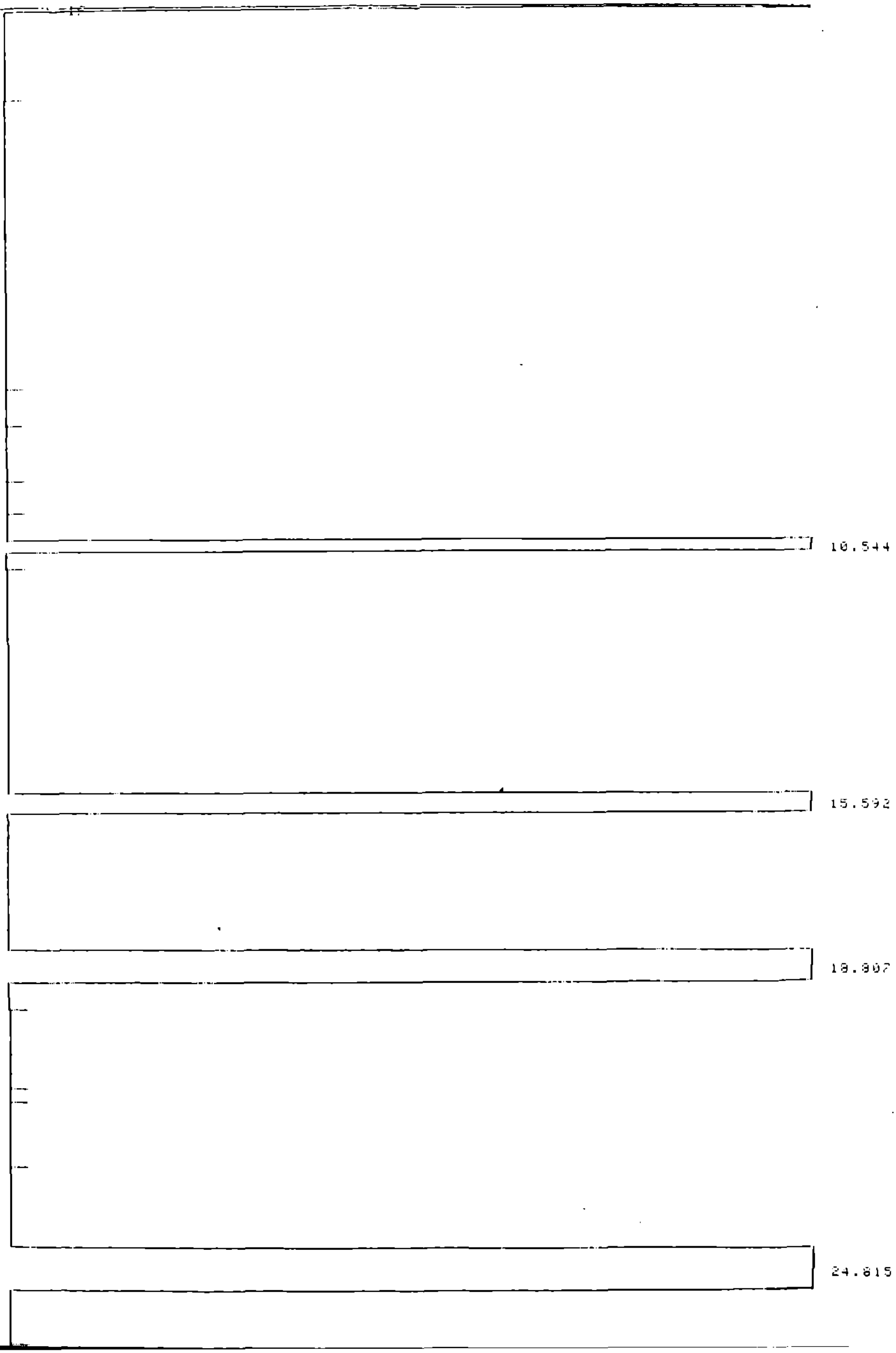
ESTD-AREA
      RT  TYPE         AREA  WIDTH       HEIGHT  CAL#  CONC.        NAME
    .760    PB        10532   .031         5657    1R      .362     METHANE
    .880    BB        47682   .064        12367           1.870
   1.010    BB        21136   .083         4286            .829
   1.449    PB        60404   .085        11607           2.369
   5.463    VB        40666   .144         4724           1.596
   7.521    PB        44141   .221         3324    5       .326     PENTANE
   8.295    BB        26983   .193         2325           1.058
  10.508    PB       108873   .418         4343           4.270
  11.670    BB        52913   .218         4046           2.076 ← TI7
  12.545    BB        83517   .243         5664    6       .529     HEXANE
  15.551    BB       251107   .355        11797           9.848
  18.395    VB       243151   .981         4131           9.536
  21.060    PB       176263   .760         3864           6.913
  23.627    BB       107577   .654         2743           4.219


TOTAL AREA=1273977
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

• RUN # 67 SEP 24, 1991 11:53:50 (92) BTEX ~16 ppmv
START



```
RUN#     97              SEP 24, 1991  11:59:50

MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
        RT        AREA TYPE  WIDTH        AREA%
   10.544    1680151    PB    .214    22.53135
   15.592     652621    PB    .080     8.75165
   18.807    2598200    PB    .410    34.84267
   24.815    2525976    PB    .733    33.87413

TOTAL AREA=7456947
MUL FACTOR=1.0000E+00



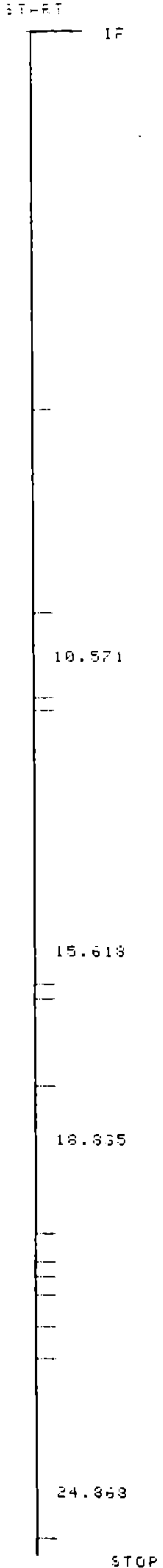
RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

93

RUN # 38 SEP 24, 1991 12:36:35

(94)

BTFA ~1.0 ppm



```
RUN#     55             SEP 24, 1991  12:36:35
```

(95)

```
MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
       RT          AREA TYPE  WIDTH        AREA%
   10.571       156072    BB   .209    25.90270
   16.835       226206    BB   .411    37.67483
   24.368       216252    PB   .694    36.22246

TOTAL AREA= 602532
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO      =   0
ATT 2^    =  -2
CHT SP    =   1.0
AR REJ    =   500
THRSH     =   4
PK WD     =   0.04
```

```
*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1              RECALIBRATIONS: 1
LEVEL: 2              RECALIBRATIONS: 1
LEVEL: 3              RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1F     0.743     1 1.5000E+01   3.8490E-05
                   2 1.0250E+02   3.8591E-05
                   3 9.9600E-01   3.4350E-05
  2      0.916     1 1.4500E+01   2.0130E-05
  3      1.549     1 1.4200E+01   1.2860E-05
                   2 1.0040E+02   1.2553E-05
  4      3.732     1 1.4600E+01   9.1560E-06
                   2 9.7310E+01   6.8956E-06
  5      7.563     1 1.4600E+01   7.3830E-06
  6     12.074     1 1.4500E+01   6.4075E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 2

REF % RTW [   5.000]:   BREAK

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:   6
RT:  11.748
AMT:
AMT/AREA:
NAME:

CAL#:
```

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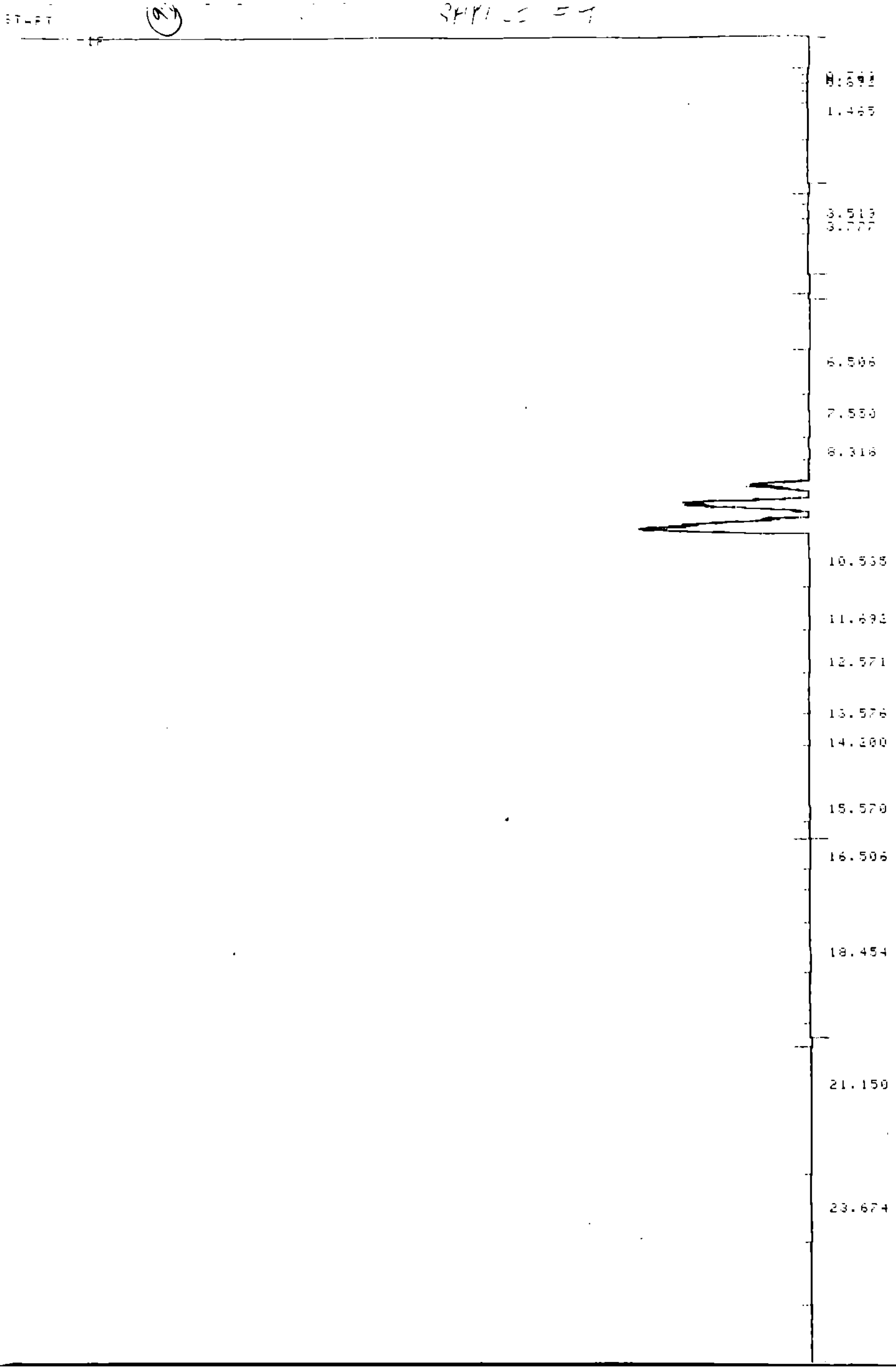
START
0.591
0.692
1.465
3.513
3.777
6.506
7.550
8.316
10.535
11.693
12.571
13.576
14.200
15.570
16.506
18.454
21.150
23.674

98

```
RUN#     59          SEP 24, 1991  13:16:35


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.        NAME
     .769   PB         6494   .024        4572   1R       .223   METHANE
     .892   BB        53276   .066       13439           2.089
    1.465   PB        67710   .097       11590           2.656
    3.519   VB        40633   .142        4759           1.594
    5.777   BB        22942   .130        3932   4        .210   BUTANE
    6.506   PB        62445   .346        3006           2.449
    7.550   BB        66802   .252        4559   5        .505   PENTANE
    8.318   BB        35230   .195        3004           1.382
   10.535   PB       130677   .410        5314           5.125
   11.692   BB        70829   .229        5158           2.778
   12.571   BB       115941   .262        7382   6        .743   HEXANE
   13.576   BB        11741   .535         366            .460
   15.570   BB       288949   .340       14147          11.333
   16.506   BB        42657   .279        2545           1.673
   18.454   PB        71923   .527        2275           2.821
   21.150   BB       215030   .606        4444           8.433
   23.674   BB       114801   .641        2985           4.502


TOTAL AREA=1420080
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1               RECALIBRATIONS: 1
LEVEL: 2  .            RECALIBRATIONS: 1
LEVEL: 3               RECALIBRATIONS: 1

CAL#      RT      LV    AMT        AMT/AREA
  1R    0.749    1 1.5000E+01  3.8490E-05
                 2 1.0250E+02  3.8591E-05
                 3 9.9600E-01  3.4350E-05
  2     0.918    1 1.4500E+01  2.0130E-05
  3     1.549    1 1.4200E+01  1.2860E-05
                 2 1.0040E+02  1.2553E-05
  4     3.743    1 1.4600E+01  9.1560E-06
                 2 9.7310E+01  8.8958E-06
  5     7.559    1 1.4600E+01  7.3830E-06
  6    11.953    1 1.4500E+01  6.4075E-06

CAL#     NAME
  1  METHANE
  2  ETHANE
  3  PROPANE
  4  BUTANE
  5  PENTANE
  6  HEXANE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  6
RT:
AMT:
AMT/AREA:
NAME:

CAL#:  6
RT:  11.748
AMT:
AMT/AREA:
NAME:

CAL#:

SECTION TO BE EDITED:

*  LIST: CALIB   @

ESTD
```

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```
LEVEL: 1              RECALIBRATIONS: 1
LEVEL: 2              RECALIBRATIONS: 1
LEVEL: 3              RECALIBRATIONS: 1

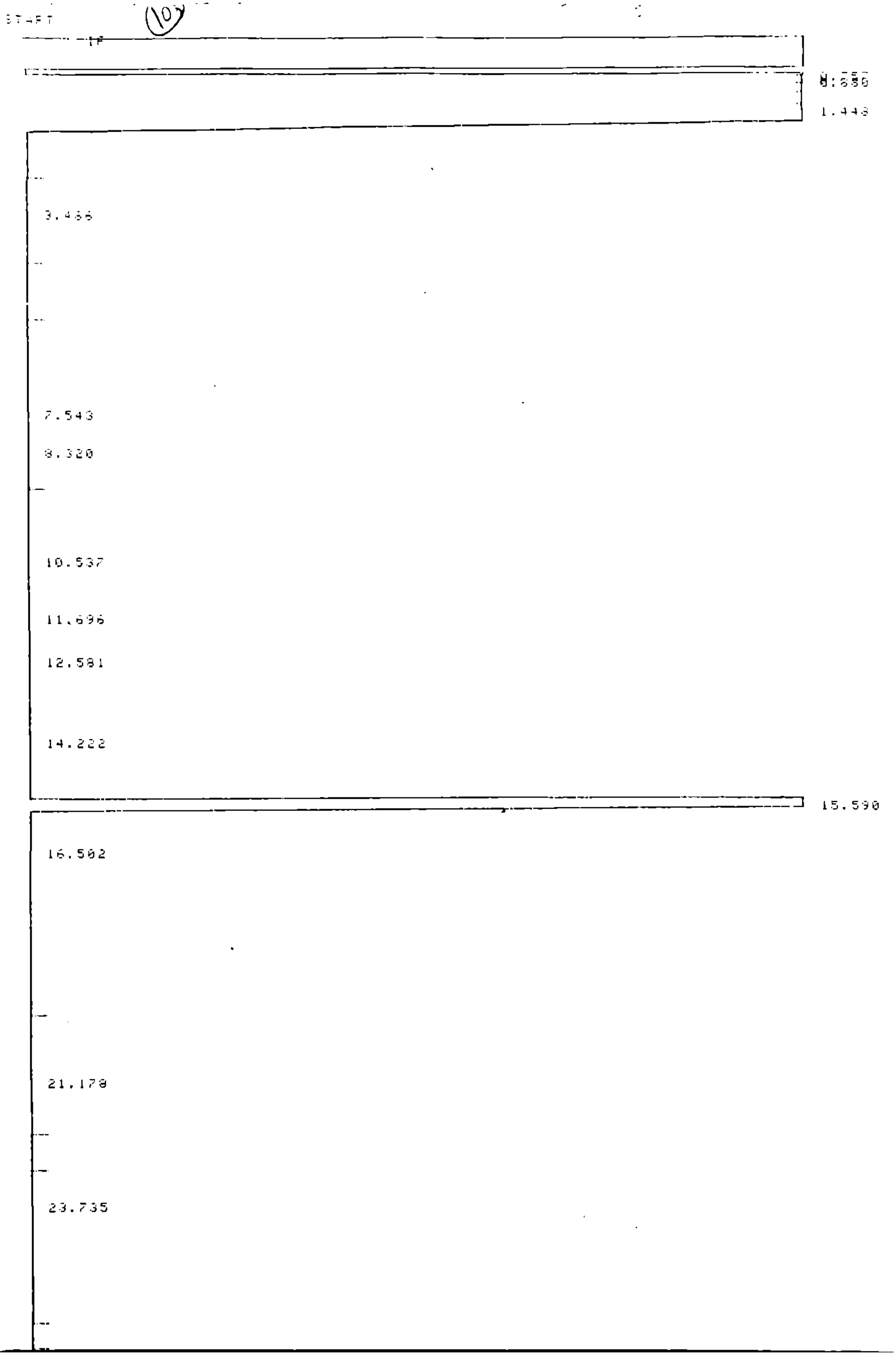
CAL#     RT      LV     AMT          AMT/AREA
 1R     0.749     1 1.5000E+01   3.6490E-05
                  2 1.0250E+02   3.5591E-05
                  3 9.9600E-01   3.4350E-05
 2      0.918     1 1.4500E+01   2.0130E-05
 3      1.549     1 1.4200E+01   1.2860E-05
                  2 1.0040E+02   1.2553E-05
 4      3.743     1 1.4600E+01   9.1560E-06
                  2 9.7310E+01   8.8958E-06
 5      7.559     1 1.4600E+01   7.3830E-06
 6     11.748     1 1.4500E+01   6.4075E-06

CAL#      NAME
 1   METHANE
 2   ETHANE
 3   PROPANE
 4   BUTANE
 5   PENTANE
 6   HEXANE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00
```

100

(10)



START
0.550
1.448
3.466
7.543
8.320
10.537
11.696
12.581
14.222
15.590
16.502
21.178
23.735

102

```
RUN#     90          SEP 24, 1991   13:54:36


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH       HEIGHT CAL# CONC.        NAME
    .757    FB         3626   .012         4691   1R         .125  METHANE
    .860    BB        36979   .059        10403             1.450
   1.446    FB        68842   .101        11344             2.700
   3.486    BB        32532   .140         3884             1.276
   7.543    FB        63785   .259         4107   5          .471  PENTANE
   9.320    BB        30957   .199         2594             1.214
  10.537    FB       118130   .424         4640             4.633
  11.696    BB        57965   .215         4485   6          .372  HEXANE
  12.561    BB       105891   .266         6578             4.153
  15.590    BB       305924   .374        13633            11.998
  16.502    FB        31999   .262         2062             1.255
  21.178    FB       116525   .588         3304             4.570
  23.735    BB       113998   .657         2890             4.471


TOTAL AREA=1087173
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:   7
RT:   10.544
AMT:   1
AMT/AREA:   6.099E-06
NAME:   BENZENE

CAL#:   8
RT:   18.607
AMT:   1.14
AMT/AREA:   4.85E-06
NAME:   ETHYLBENZEN▒NE

CAL#:   9
RT:   24.815
AMT:   1.08
AMT/AREA:   4.922E-06
NAME:   O-XYLENE

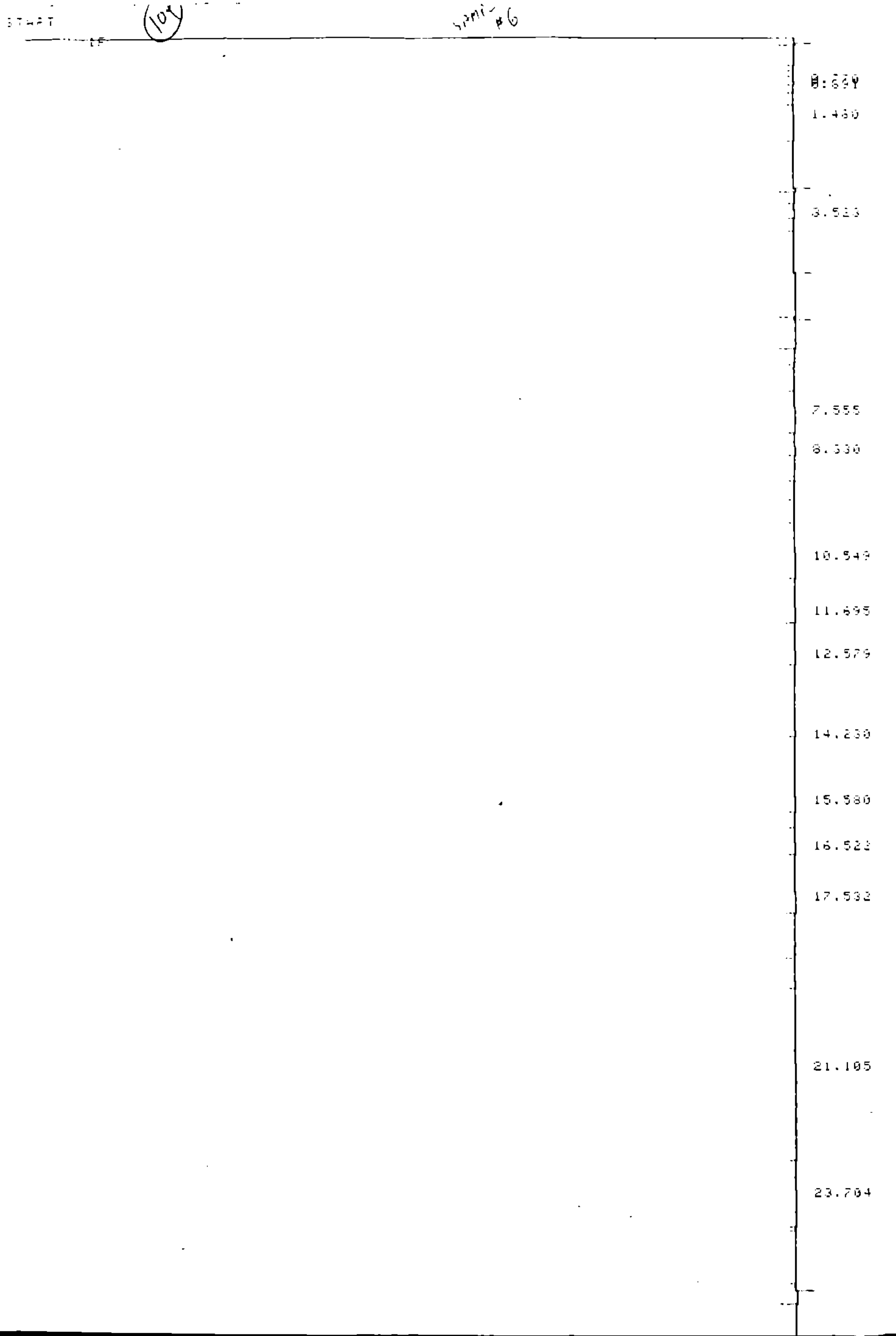
CAL#:

SECTION TO BE EDITED:

*
```

(104)

spmi #6


START
0.639
1.460
3.523
7.555
8.330
10.549
11.695
12.579
14.230
15.580
16.522
17.532
21.105
23.704

105

```
RUN#     91            SEP 24, 1991  14:35:30


MATHY CONSTRUCTION

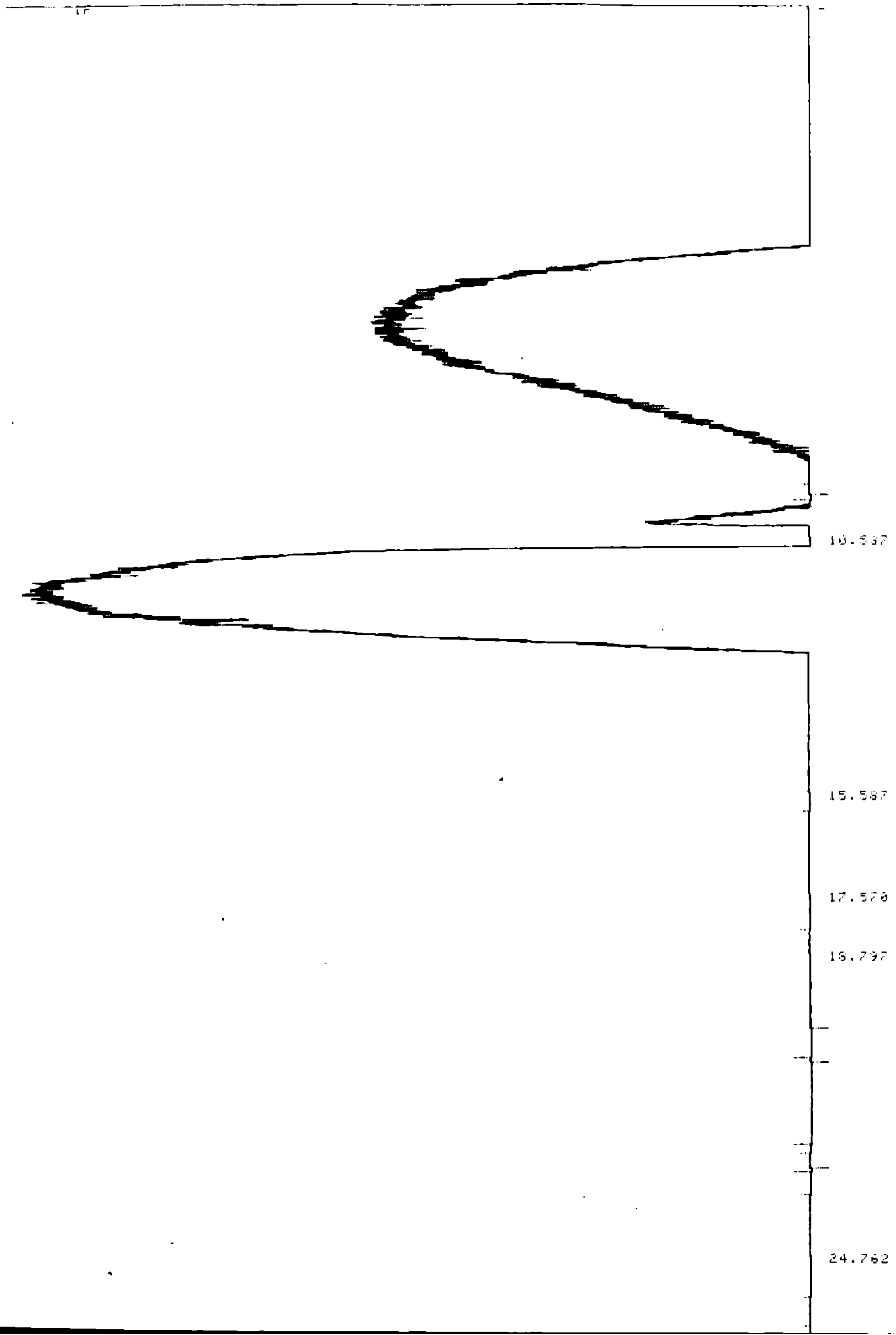
ESTD-AREA
     RT TYPE          AREA  WIDTH        HEIGHT CAL# CONC.        NAME
    .770   PB        16345   .049          5579   1R          .561  METHANE
    .891   BB       110271   .084         21911              4.335
   1.480   PB        98662   .110         14993              3.870
   3.523   VB        54260   .150          6025              2.128
   7.555   PB        56743   .275          3443   5           .419  PENTANE
   8.330   BB        24114   .193          2079               .946
  10.549   PB       118957   .387          5118   7           .726  BENZENE
  11.695   BB        62174   .256          4014   6           .398  HEXANE
  12.579   BB        88235   .263          5590              3.461
  15.580   BB       272178   .382         11861             10.675
  16.522   PB        28013   .266          1758              1.099
  17.532   BB        75636   .661          1906              2.966
  21.105   VB       484795  1.412          5723             19.014
  23.704   BB        92851   .619          2500              3.642


TOTAL AREA=1583234
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

* RUN # 93 (10) SEP 24, 1991 15:14:33 1.0 ppm/ [illegible]
START



109

```
RUN#    52          SEP 24, 1991  15:14:33


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.         NAME
  10.537   BB       167562   .215       13005    7         1.022  BENZENE
  17.570   BB       150541   .973        2579              5.904
  19.797   BB       276628   .431       10695    8         1.342  ETHYLBENZENE
  24.782   PB       277901   .713        6495    9         1.368  O-XYLENE

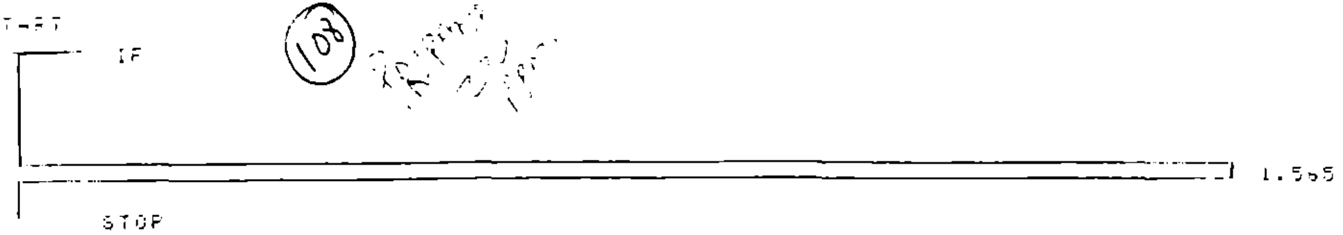

TOTAL AREA= 872632
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2   = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
* RUN #    93        SEP 24, 1991  15:49:42
```

(108)



```
RUN#     93          SEP 24, 1991  15:49:42


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH     HEIGHT CAL# CONC.       NAME
   1.565 ISBF   22443944   .109    3440062   3     281.020  PROPANE


TOTAL AREA=2.2443E+07
MUL FACTOR=1.0000E+00

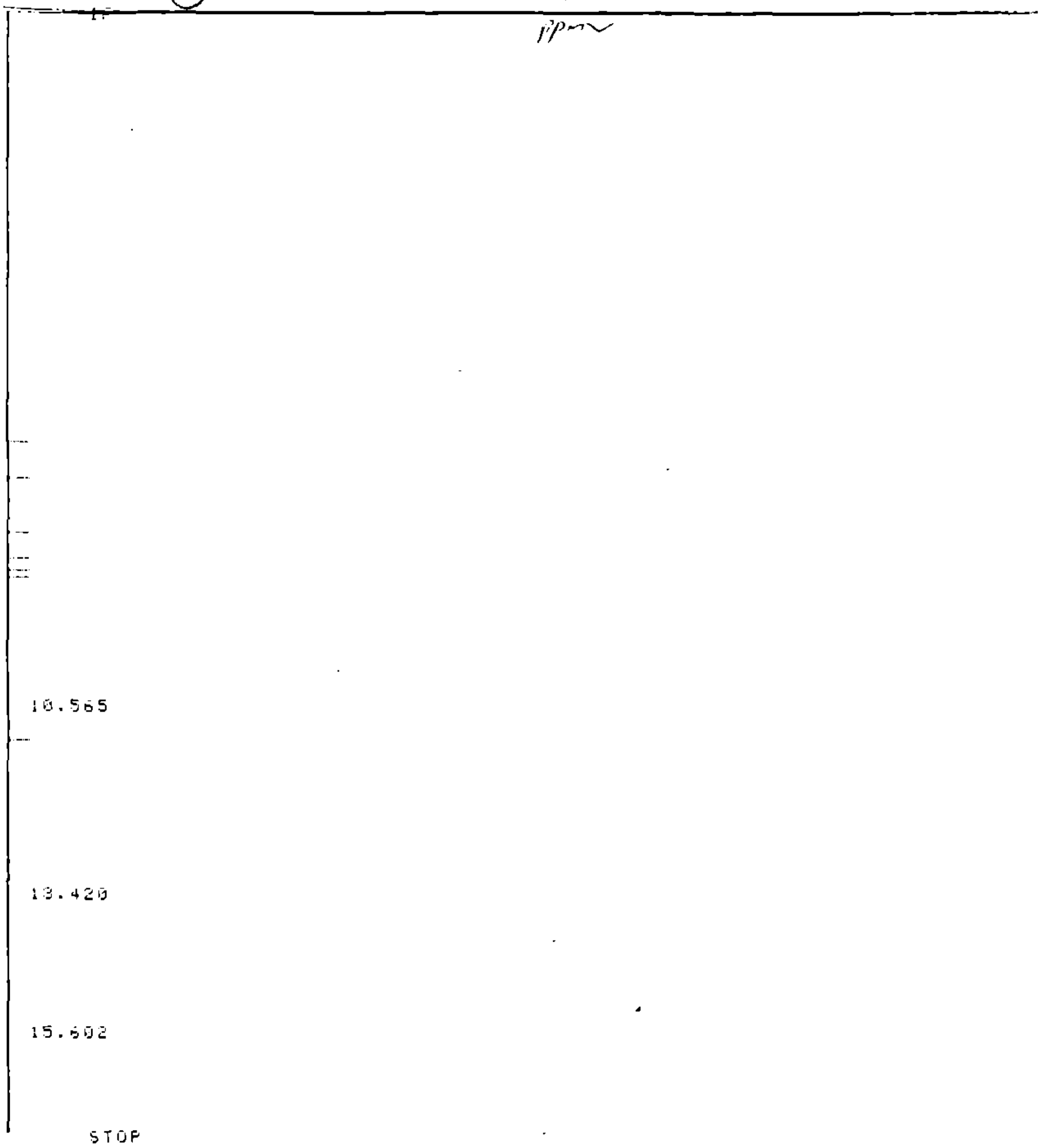


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
* RUN #    95          SEP 24, 1991   16:22:42
START
```

(109)

1.0 BTEX
ppm



```
RUN#    95          SEP 24, 1991  16:22:42

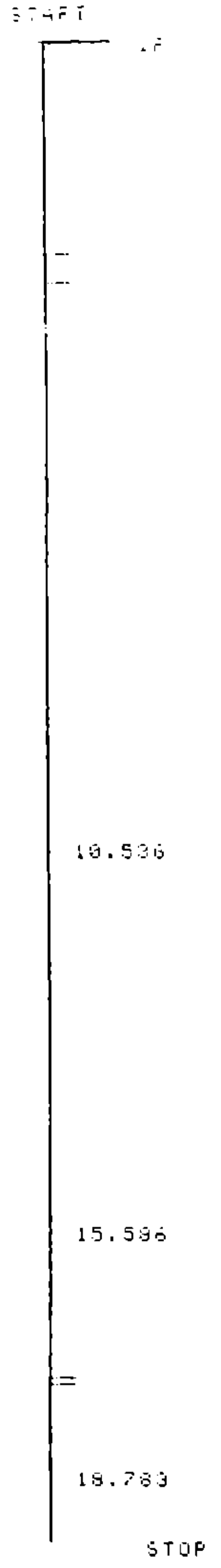

MATHY CONSTRUCTION

ESTD-AREA
     RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.         NAME
  10.565   BB     162043   .209       12930   7          .988  BENZENE
  13.420   PB      76054   .304        4170             2.983
  15.602   BB     352503   .351       16735            13.825


TOTAL AREA= 590600
MUL FACTOR=1.0000E+00
```

110

1.0 BTX



```
RUN#    96           SEP 24, 1991  16:54:50


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.          NAME
  10.536   PB      153090   .190       13395   7          .934  BENZENE
  15.586   BB     1486323   .915       27085            58.294
  18.783 I BP     2119573  1.251       28234   8        10.280  ETHYLBENZENE


TOTAL AREA=3758986
```

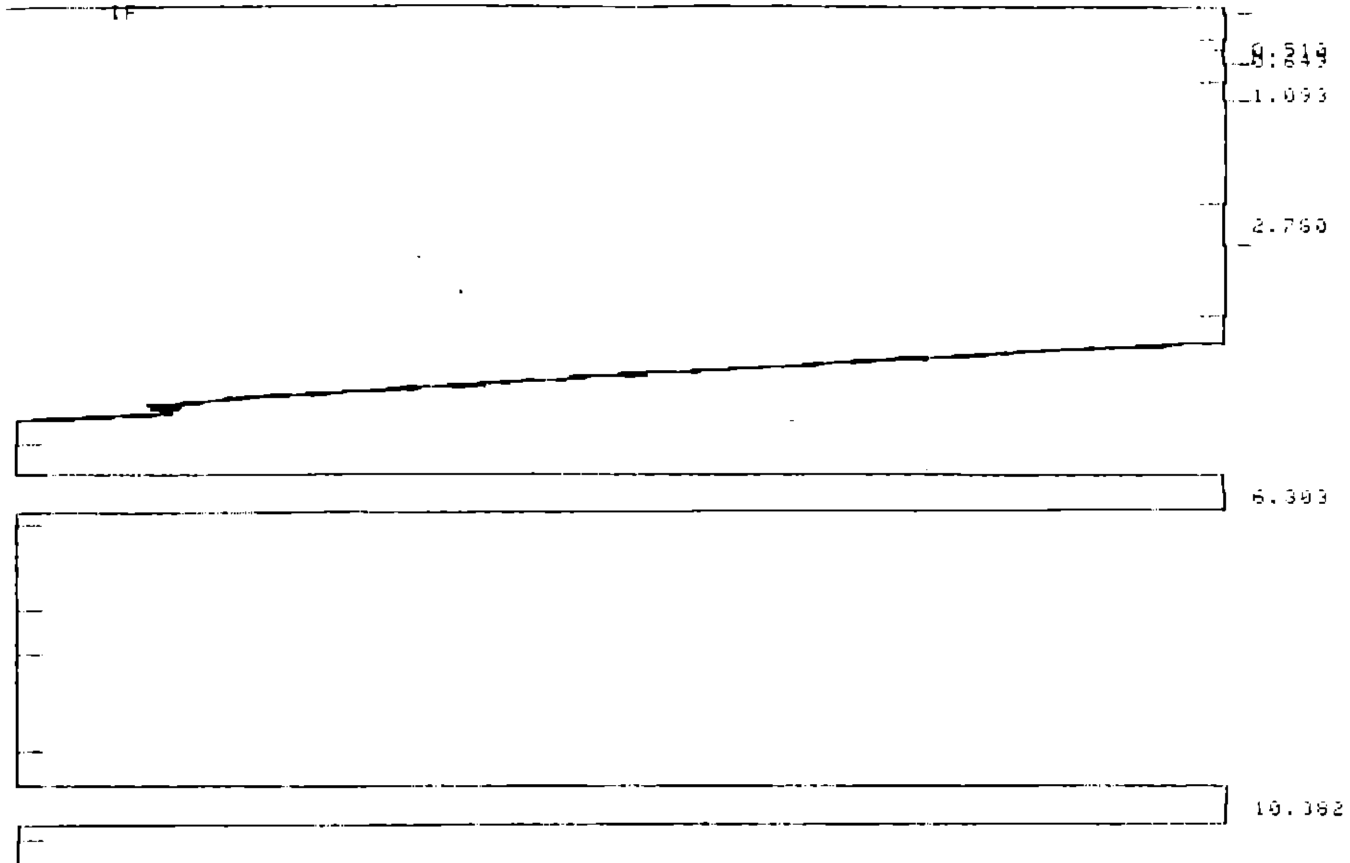
```
RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

111

```
*NOTEPAD

*USE BREAK OR CONTROL-C TO END*
14.5-15.0 ppmv Standard
* RUN #   98       SEP 25, 1991  07:24:51
START
```

112



```
STOP


RUN#    98          SEP 25, 1991  07:24:51


MATHY CONSTRUCTION

ESTD-AREA
     RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .510   PB     466330   .056      137645           18.289
    .649   BB     920924   .058      265206           36.119
   1.093   BB    1353954   .071      315662           53.102
   2.780   BB    1880067   .152      205839           73.736
   6.303   BB    2338526   .209      186836           91.717
  10.382   PB    2685480   .234      190901    7      16.379  BENZENE

TOTAL AREA=9.6453E+06
MUL FACTOR=1.0000E+00



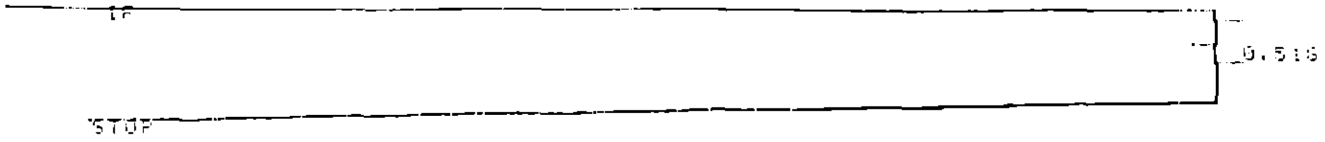
RUN PARAMETERS
ZERO    =  0
ATT 2^  =  -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*NOTEPAD

<USE BREAK OR CONTROL-Y TO END>
<996 ppmv Methane STANDARD

*  RUN #    99      SEP 25, 1991  07:44:10
START
```

(113)



```
RUN#     99            SEP 25, 1991  07:44:10


MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT        AREA TYPE  WIDTH       AREA%
    .518      40727   BB    .062  100.00000

TOTAL AREA=  40727
MUL FACTOR=1.0000E+00



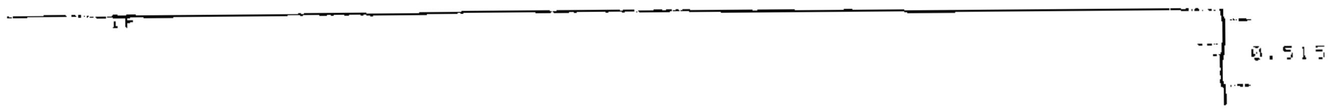
RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*NOTEPAD

 USE BREAK OR CONTROL-Y TO END)
.998 PPMV METHNS STNDARD

*  RUN #  100      SEP 25, 1991  07:47:45
START
```

(114)



```
      STOP


RUN#   100          SEP 25, 1991  07:47:45


MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
        RT        AREA TYPE  WIDTH       AREA%
      .515       36957   PB    .056  100.00000

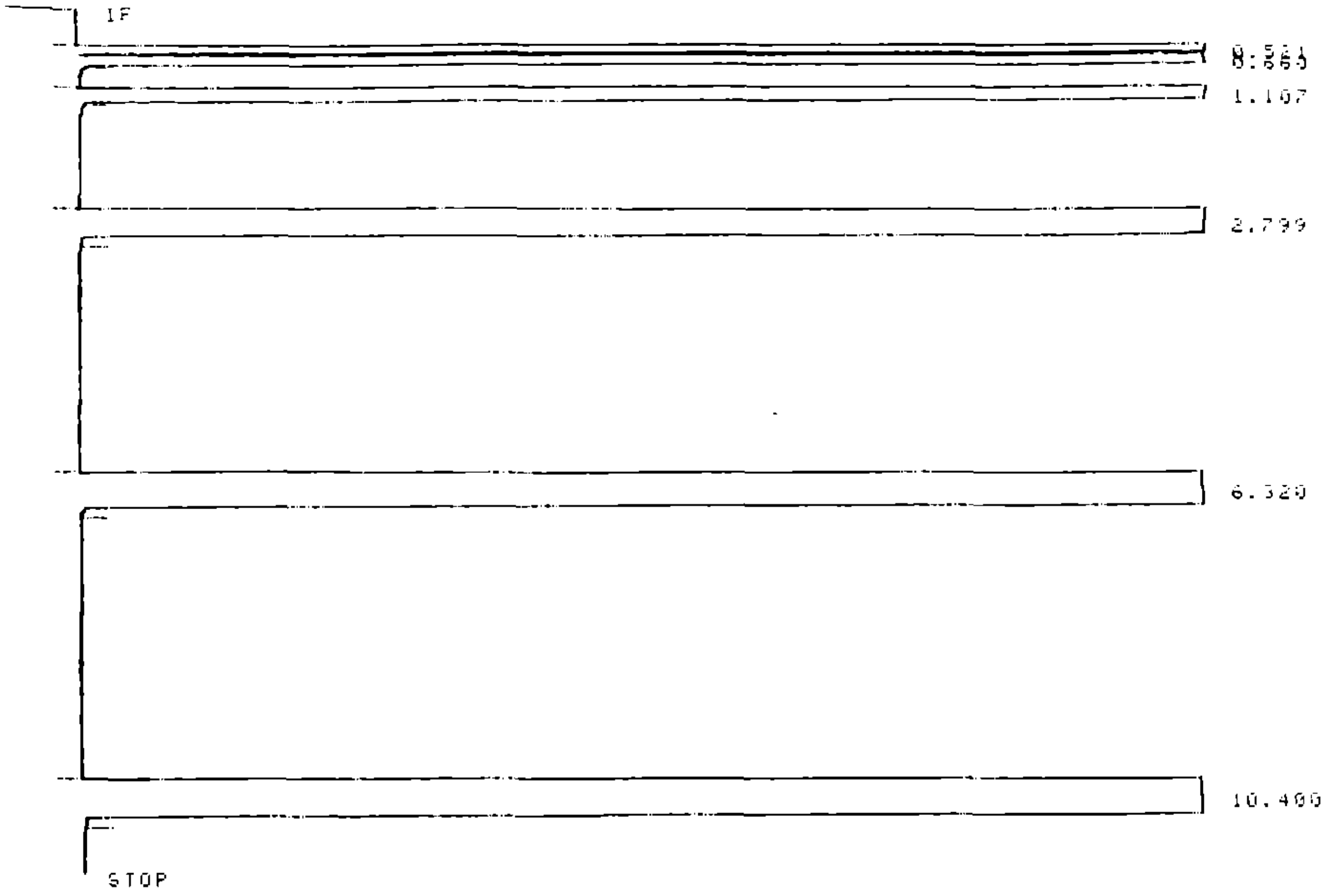
TOTAL AREA=  36957
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*NOTEPAD

<USE BREAK OR CONTROL-C TO END>
C1-C6 14.5 PPMV STANDARD
*  RUN #  101       SEP 25, 1991  07:56:03
START
```



```
RUN#   101          SEP 25, 1991  07:56:03
```

115

```
MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.          NAME
    .521    BB     463275   .057      134879             18.170
    .660    BB     930479   .059      263785             36.493
   1.107    PB    1367897   .071      319205             53.649
   2.799    PB    1875819   .149      209526             73.570
   6.320    BB    2269278   .201      188020             99.001
  10.400    BB    2602210   .225      192434    7        15.871  BENZENE

TOTAL AREA=9.5090E+06
MUL FACTOR=1.0000E+00



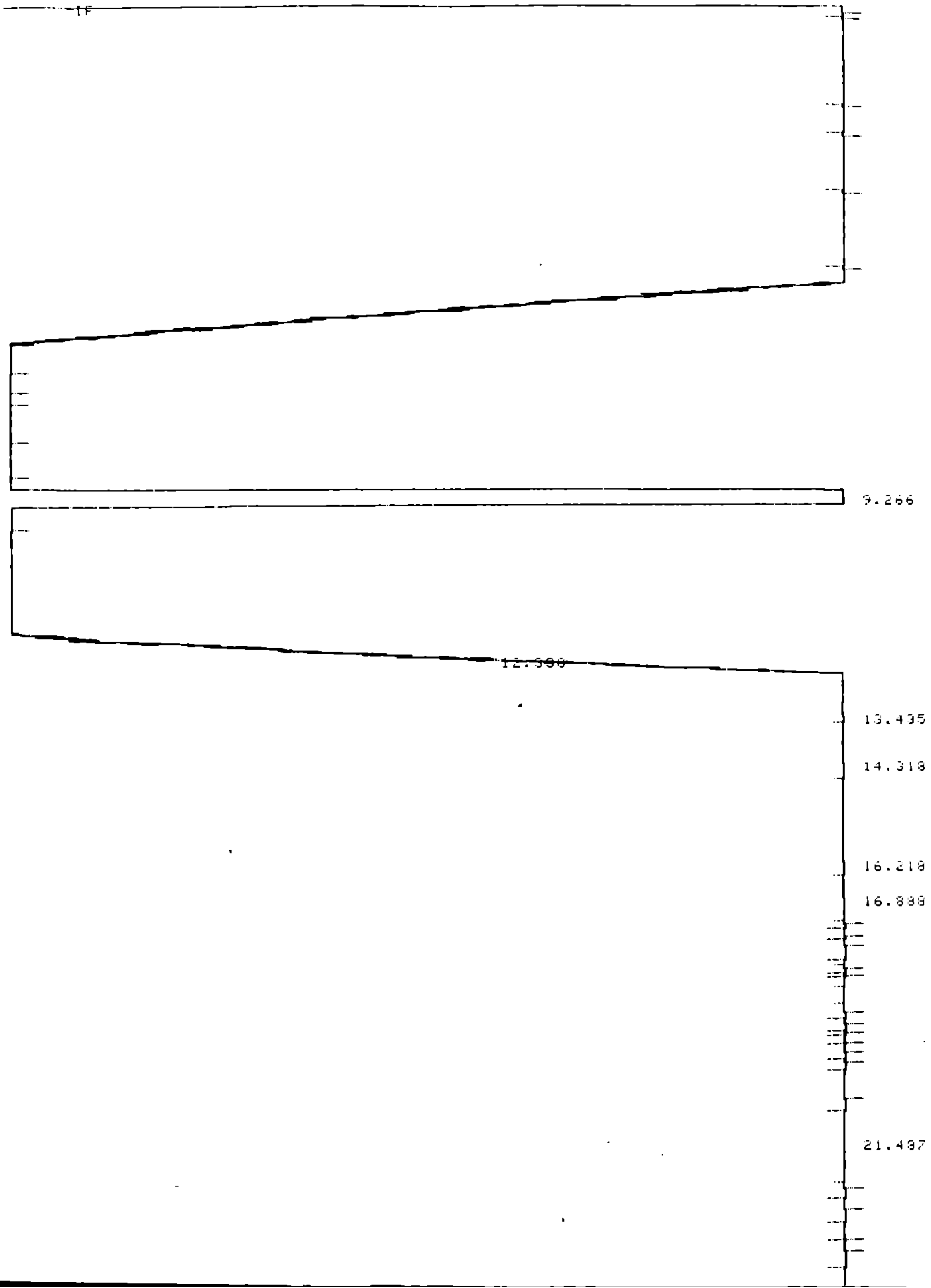
RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*NOTEPAD

<USE BREAK OR CONTROL-Y TO END>
BTEX STANDRD, 1 PPMV

* RUN # 102      SEP 25, 1991  08:17:01
START
```

116



```
        STOP


RUN#    102             SEP 25, 1991   08:17:01
```

112

```
MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
       RT        AREA TYPE  WIDTH       AREA%
    9.266      203032   BB   .225    14.48682
   14.318      205889   BB   .197    14.69068
   16.218      366983   BB  3.201    26.18514
   16.888      319113   BB   .336    22.76949
   21.487      306477   BB   .583    21.86787

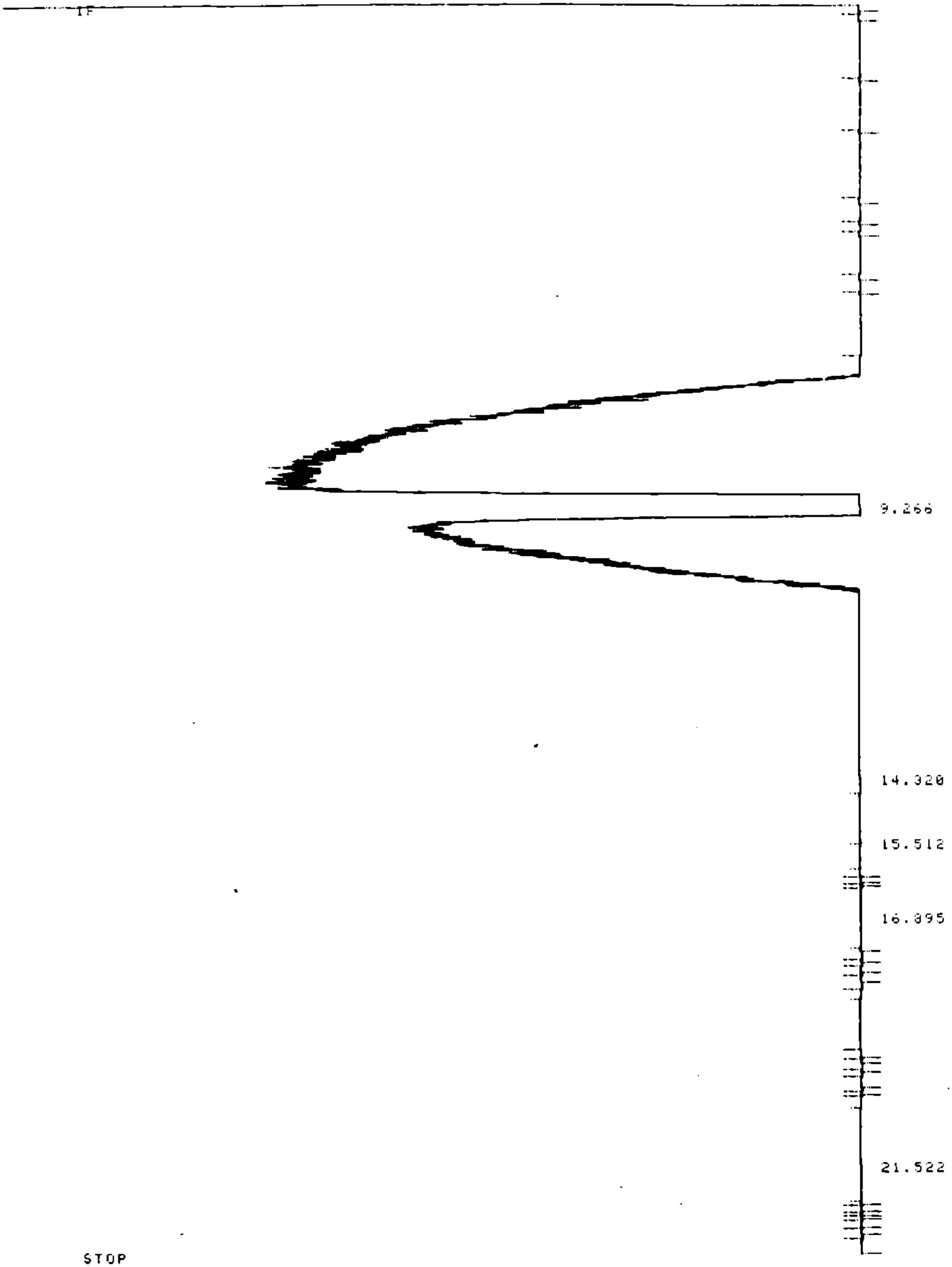
TOTAL AREA=1401494
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
-NOTEP-G

\USE BREAK OR CONTROL-Y TO END/
BTE *X  1.0 ppmv  STANDARD
*  RUN #  103       SEP 25, 1991  08:54:43
START
```

118



```
STOP


RUN#   103              SEP 25, 1991  08:54:43
```

```
MATRL CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
      RT        AREA TYPE  WIDTH       AREA%
   9.266      193072   BB   .226   24.40480
  15.512       35785   BB  1.671    4.52332
  16.895      278921   PB   .337   35.25634
  21.522      283345   VB   .592   35.81554

TOTAL AREA= 791123
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

112

* RUN # 104 SEP 25, 1991 09:29:06

(120)

START

```
RUN#   104            SEP 25, 1991  09:29:06
```

121

```
MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
       RT        AREA TYPE  WIDTH        AREA%
    9.256     185628   PB    .227     21.57687
   15.971     152838   BB   9.400     17.76546
   16.882     260538   VB    .345     30.28419
   21.466     261306   VB    .592     30.37347

TOTAL AREA= 860310
MUL FACTOR=1.0000E+00

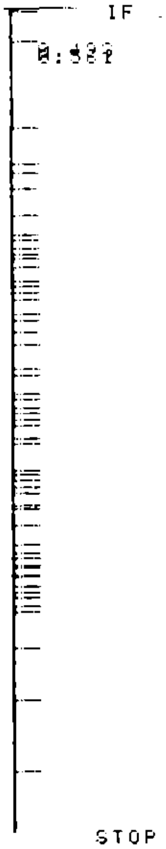


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
*NOTEPOAD

<USE BREAK OR CONTROL-Y TO END>
BLANK
*  RUN #  105       SEP 25, 1991  10:01:27
START
```

122



```
STOP


RUN#   105            SEP 25, 1991   10:01:27


MATHY CONSTRUCTION

NO CALIB PEAKS FOUND
AREA%
        RT       AREA TYPE  WIDTH       AREA%
      .493     150534   PB   .036    42.50165
      .561     203650   BB   .102    57.49835

TOTAL AREA= 354184
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

```
* LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1              RECALIBRATIONS: 1
LEVEL: 2              RECALIBRATIONS: 1
LEVEL: 3              RECALIBRATIONS: 1

CAL#       RT      LV     AMT        AMT/AREA
  1R     0.750     1 1.5000E+01   3.8490E-05
                   2 1.0250E+02   3.8591E-05
                   3 9.9600E-01   3.4350E-05
  2      0.922     1 1.4500E+01   2.0130E-05
  3      1.554     1 1.4200E+01   1.2860E-05
                   2 1.0040E+02   1.2553E-05
  4      3.752     1 1.4600E+01   9.1560E-06
                   2 9.7310E+01   8.8958E-06
  5      7.468     1 1.4600E+01   7.3830E-06
  6     11.725     1 1.4500E+01   6.4075E-06
  7     10.576     1 1.0000E+00   6.0990E-06
  8     18.807     1 1.1400E+00   4.8500E-06
  9     24.815     1 1.0800E+00   4.9220E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   ETHYLBENZENE
  9   O-XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  DELETE  CALIB  3  @

*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1              RECALIBRATIONS: 1
LEVEL: 2              RECALIBRATIONS: 1

CAL#       RT      LV     AMT        AMT/AREA
  1R     0.750     1 1.5000E+01   3.8490E-05
                   2 1.0250E+02   3.8591E-05
  2      0.922     1 1.4500E+01   2.0130E-05
  3      1.554     1 1.4200E+01   1.2860E-05
                   2 1.0040E+02   1.2553E-05
  4      3.752     1 1.4600E+01   9.1560E-06
                   2 9.7310E+01   8.8958E-06
  5      7.468     1 1.4600E+01   7.3830E-06
  6     11.725     1 1.4500E+01   6.4075E-06
  7     10.576     1 1.0000E+00   6.0990E-06
  8     18.807     1 1.1400E+00   4.8500E-06
  9     24.815     1 1.0800E+00   4.9220E-06
```

123

```
CAL#      NAME
  1  METHANE
  2  ETHANE
  3  PROPANE
  4  BUTANE
  5  PENTANE
  6  HEXANE
  7  BENZENE
  8  ETHYLBENZENE
  9  O-XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  1
RT:  .521
AMT:
AMT/AREA:  3.2275E-05
NAME:

CAL#:  2
RT:  .660
AMT:
AMT/AREA:  1.55665[illegible]665
NAME:

CAL#:  2
RT:  .660
AMT:
AMT/AREA:  1.5665E-05
NAME:

CAL#:  3
RT:  1.107
AMT:
AMT/AREA:  1.0435E-05
NAME:

CAL#:  4
RT:  2.799
AMT:
AMT/AREA:  7.774E-06
NAME:

CAL#:  5
RT:  6.332[illegible]20
AMT:
AMT/AREA:  6.330E-06
NAME:

CAL#:  6
```

124

```
AMT:
AMT/AREA:  5.4355E-06
NAME:

CAL#:  7
RT:  9.266
AMT:  1.00
AMT/AREA:  5.164E-06
NAME:

CAL#:  8
RT:  14.316
AMT:  1.05
AMT/AREA:  5.985E-06
NAME:

CAL#:  8
RT:
AMT:
AMT/AREA:
NAME:  TOLUENE

CAL#:  9
RT:  16.888
AMT:  1.14
AMT/AREA:  4.011E-06
NAME:  ETY#HYLBENZENE

CAL#:  10
RT:  21.487
AMT:  1.08
AMT/AREA:  3.822E-06
NAME:  o-XYLENE

CAL#:

SECTION TO BE EDITED:

*  DELETE
BREAK

*  EDIT  CALIB 2  BREAK

*  DELETE  CALIB  2  @

*  EDIT  CALIB 2  @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  1
RT:
AMT:  .998
AMT/AREA:  2.575E-05
NAME:

CAL#:

SECTION TO BE EDITED:
```

125

```
ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1                RECALIBRATIONS: 1
LEVEL: 2                RECALIBRATIONS: 1

CAL#      RT     LV    AMT         AMT/AREA
  1      0.521    1 1.5000E+01  3.2275E-05
                  2 9.9800E-01  2.5750E-05
  2      0.660    1 1.4500E+01  1.5665E-05
  3      1.107    1 1.4200E+01  1.0435E-05
  4      2.799    1 1.4600E+01  7.7740E-06
  5      6.320    1 1.4600E+01  6.3380E-06
  6     10.400    1 1.4500E+01  5.4855E-06
  7      9.266    1 1.0000E+00  5.1640E-06
  8     14.316    1 1.0500E+00  5.9650E-06
  9     16.888    1 1.1400E+00  4.0110E-06
 10     21.487    1 1.0800E+00  3.8220E-06

CAL#     NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   TOLUENE
  9   ETHYLBENZENE
 10   o-XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.9220E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00
```

```
POST-RUN LIST OPTIONS
Store post-run report [Y/N*]:    BREAK

*  OP # 4

REPORT OPTIONS

Suppress local report [Y/N*]:    BREAK

*  OP # 3

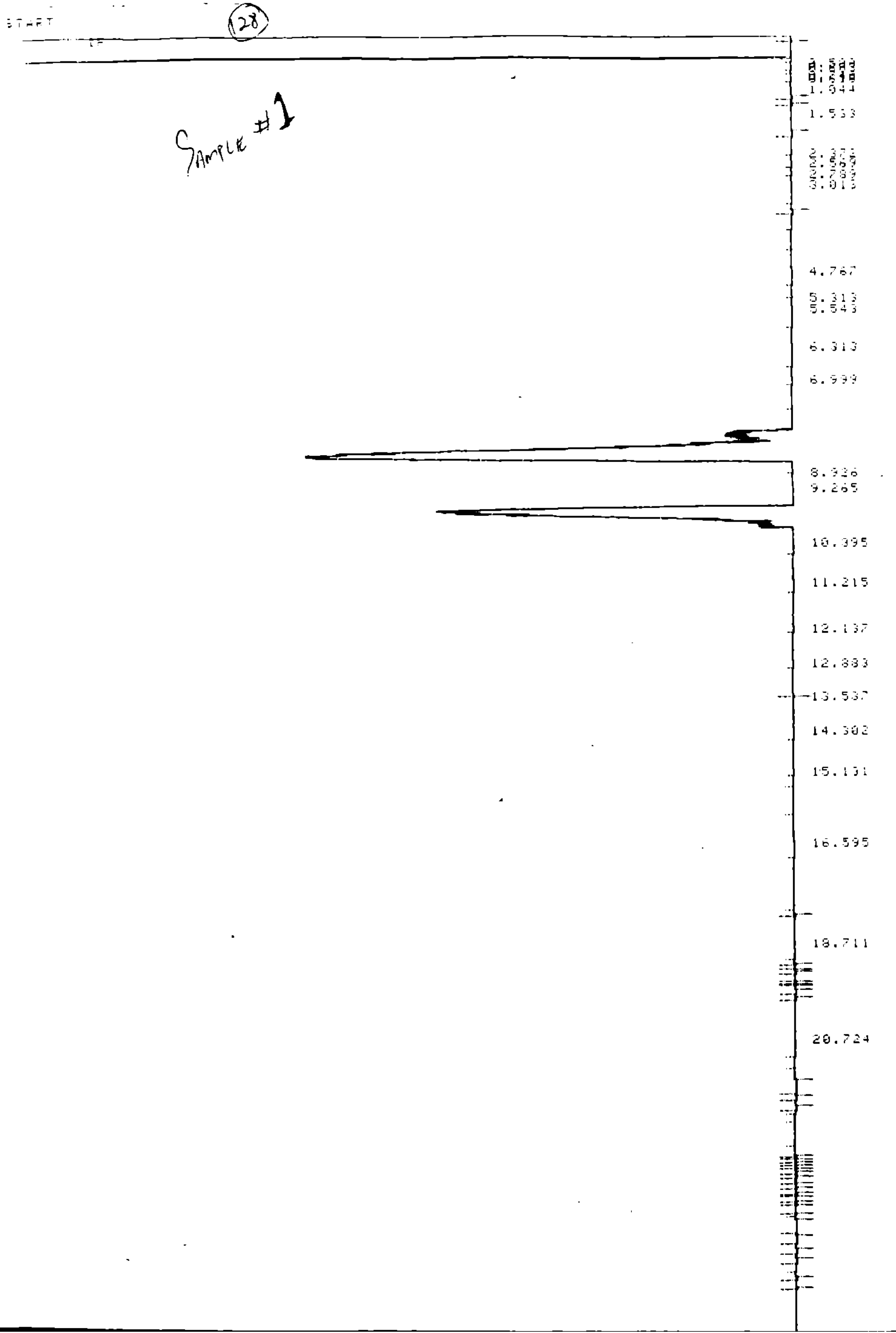
CALIBRATION OPTIONS
RF of uncalibrated peaks [3.9220E-05 ]: 3.2275E-05
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

*
```

127

(128)

Sample #1


START
1.944
1.533
3.013
4.767
5.313
5.543
6.313
6.999
8.926
9.265
10.395
11.215
12.137
12.883
13.537
14.302
15.131
16.595
18.711
20.724

```
RUN#   106          SEP 25, 1991  10:37:16

MATHY CONSTRUCTION

ESTD-AREA
       RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.        NAME
     .500    PB        70032   .040       29332    1        2.028  METHANE
     .603    BB       757234   .060      209347            24.440
     .748    BB         6817   .046        2474              .220
     .818    BB        20534   .054        6337              .663
    1.044    BB       429730   .087       81881            13.870
    1.533    BB        29821   .123        4055              .962
    2.372    PB        34554   .095        6091             1.115
    2.569    BB        81458   .120       11327             2.629
    2.789    BB        12583   .090        2325    4         .098  BUTANE
    3.013    BB        87616   .134       10934             2.828
    4.767    VB        73555   .345        3550             2.374
    5.313    BB         5285   .110         799              .171
    5.543    BB        51421   .199        4307             1.660
    6.313    BB       102579   .352        4854    5         .650  PENTANE
    6.999    BB        22573   .180        2092              .729
    8.926    PB         7775   .138         936              .251
    9.265    BB       191682   .348        9168    7         .990  BENZENE
   10.395    BB        83918   .260        5388    6         .460  HEXANE
   11.215    BB       115812   .279        6929             3.738
   12.137    BB        20665   .253        1359              .667
   12.883    BB        10085   .081        2077              .325
   13.537    BB        15780   .296         889              .509
   14.302    PB       167492   .232       12021    8        1.002  TOLUENE
   15.131    BB        38521   .273        2351             1.243
   16.595    PB        65109   .439        2474    9         .261  ETHYLBENZENE
   18.711    BB        87020   .383        3785             2.809
   20.724    BB       115508   .500        3951   10         .441  o-XYLENE


TOTAL AREA=2705219
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

129

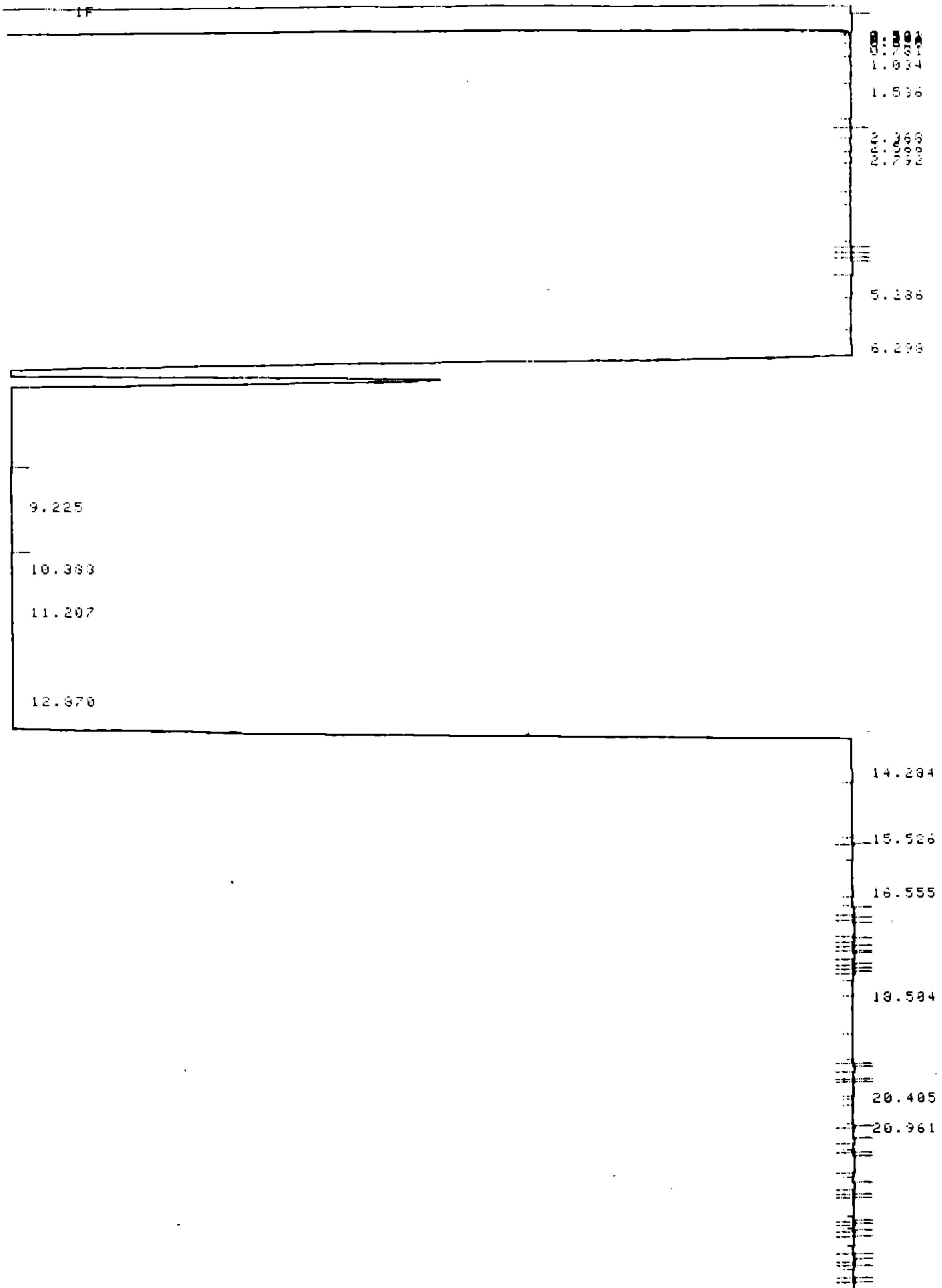
*NOTEP-C

(USE BREAK OR CONTROL-Y TO END) ㉚

SMPLE #2

+ RUN # 107 SEP 25, 1991 11:15:06

START



STOP

```
RUN#   107            SEP 25, 1991   11:15:06
```

(131)

```
MATHY CONSTRUCTION

ESTD-AREA
       RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.         NAME
     .501    PB        15431   .025       10253              .498
     .528    BB         4968   .017        4879   1          .128  METHANE
     .620    BB       135615   .065       34668             4.377
     .781    BB        63136   .127        8254             2.038
    1.034    BB       122115   .078       26074             3.941
    1.536    BB        15720   .083        3145              .507
    2.368    PB         8877   .097        1527              .287
    2.566    BB        36354   .120        5056             1.173
    2.792    BB        18045   .107        2808   4          .140  BUTANE
    5.186    PB        11284   .133        1416              .364
    6.298    PB        65646   .271        4041   5          .416  PENTANE
    9.225    VB       100078   .361        4615   7          .517  BENZENE
   10.363    BB        57826   .225        4280   6          .317  HEXANE
   11.207    BB        94331   .276        5659             3.045
   14.284    BB        22008   .043        8467   8          .132  TOLUENE
   15.526    BB        95782  3.645         438             3.091
   16.555    PB        16912   .251        1121   9          .068  ETHYLBENZENE
   20.961    PB         9396   .029        5321  10          .036  o-XYLENE

TOTAL AREA= 993524
MUL FACTOR=1.0000E+00

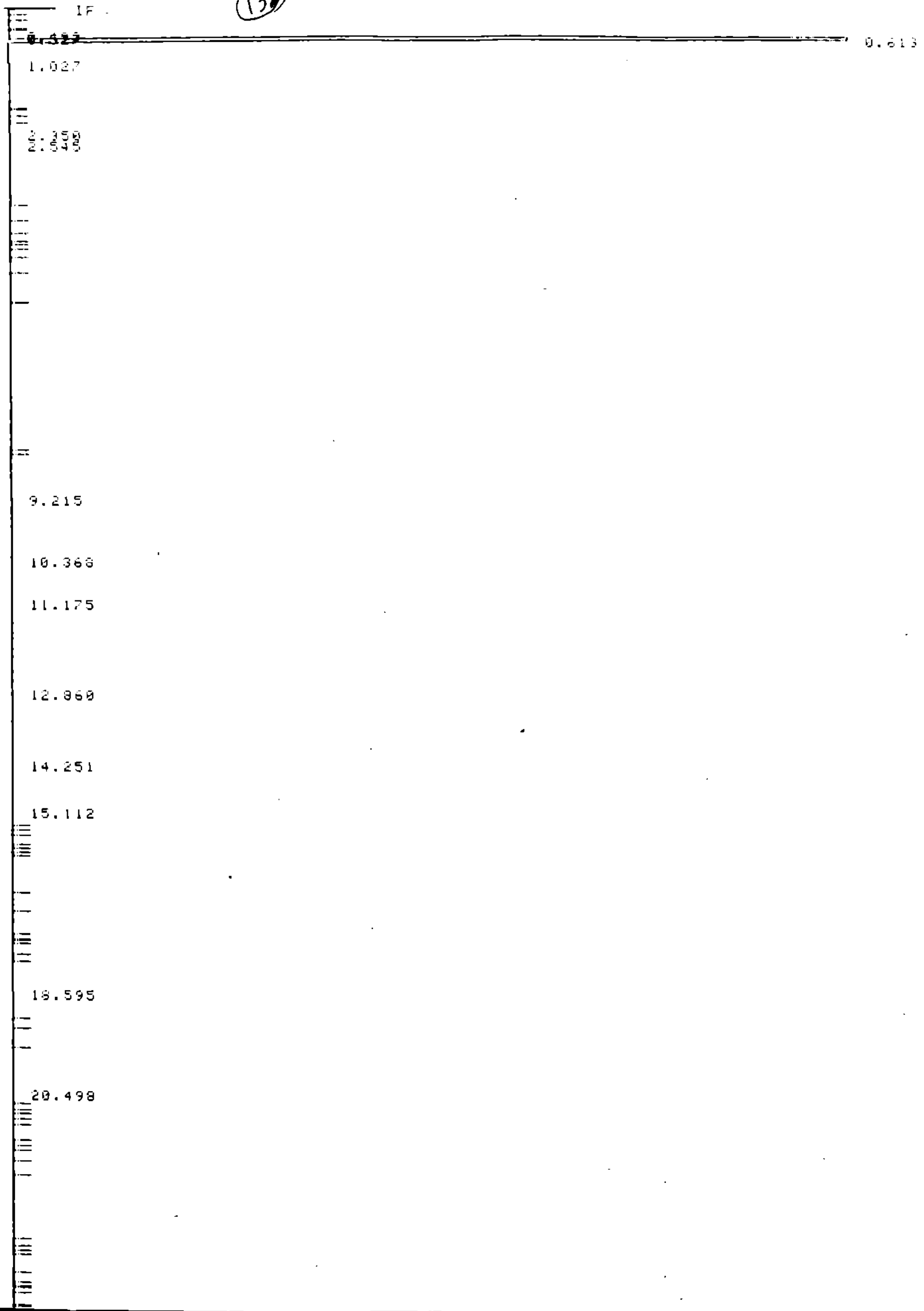


RUN PARAMETERS
ZERO    =   0
ATT 2^  =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   4
PK WD   =   0.04
```

```
*NOTEPAD

<USE BREAK OR CONTROL-Y TO END>
SAMOPLE #3
*  RUN #  108      SEP 25, 1991  11:49:26
START
```

137



```
     TIMETABLE STOP


RUN#   108           SEP 25, 1991  11:49:26

MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE         AREA  WIDTH      HEIGHT CAL# CONC.         NAME
    .493   PB        32105   .026       20610             1.036
    .527   BB         6012   .017        5906   1          .155  METHANE
    .613   BB       131423   .057       38457             4.242
   1.027   PB       115761   .080       24109             3.736
   2.350   PB        12975   .105        2067              .419
   2.545   BB        33816   .120        4715             1.091
   9.215   VB        73266   .351        3479   7          .378  BENZENE
  10.368   BB        47903   .248        3214   6          .263  HEXANE
  11.175   BB        65881   .272        4043             2.126
  15.112   BB        26311   .380        1155              .849
  18.595   PB        13230   .228         965              .427

TOTAL AREA= 558685
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

(133)

-NOTEPAD

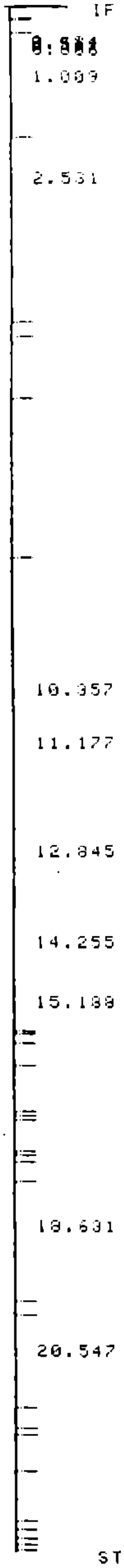
(USE BREAK OR CONTROL-Y TO END)

SAMPLE #4

* RUN # 109 SEP 25, 1991 12:28:14

START

(134)



STOP

```
RUN#   163            SEP 25, 1991  12:28:14
```

35

```
MATHY CONSTRUCTION

ESTD-AREA
       RT TYPE           AREA   WIDTH       HEIGHT CAL# CONC.         NAME
     .484    PB         20969    .027        13119             .677
     .511    BB          6274    .017         5994    1        .162  METHANE
     .606    BB         86003    .064        22303            2.776
    1.009    PB        117242    .089        21885            3.784
    2.531    VB         29413    .120         4101             .949
   10.357    PB         51974    .246         3516    6        .265  HEXANE
   11.177    BB         75909    .277         4571            2.450
   15.166    BB         20753    .581          995             .670
   16.631    VB        141011    .615         3620            4.551
   20.547    PB         67750    .438         3579   10        .259  o-XYLENE


TOTAL AREA= 617297
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

*NOTEPAD

USE BREAK OR CONTROL-Y TO END

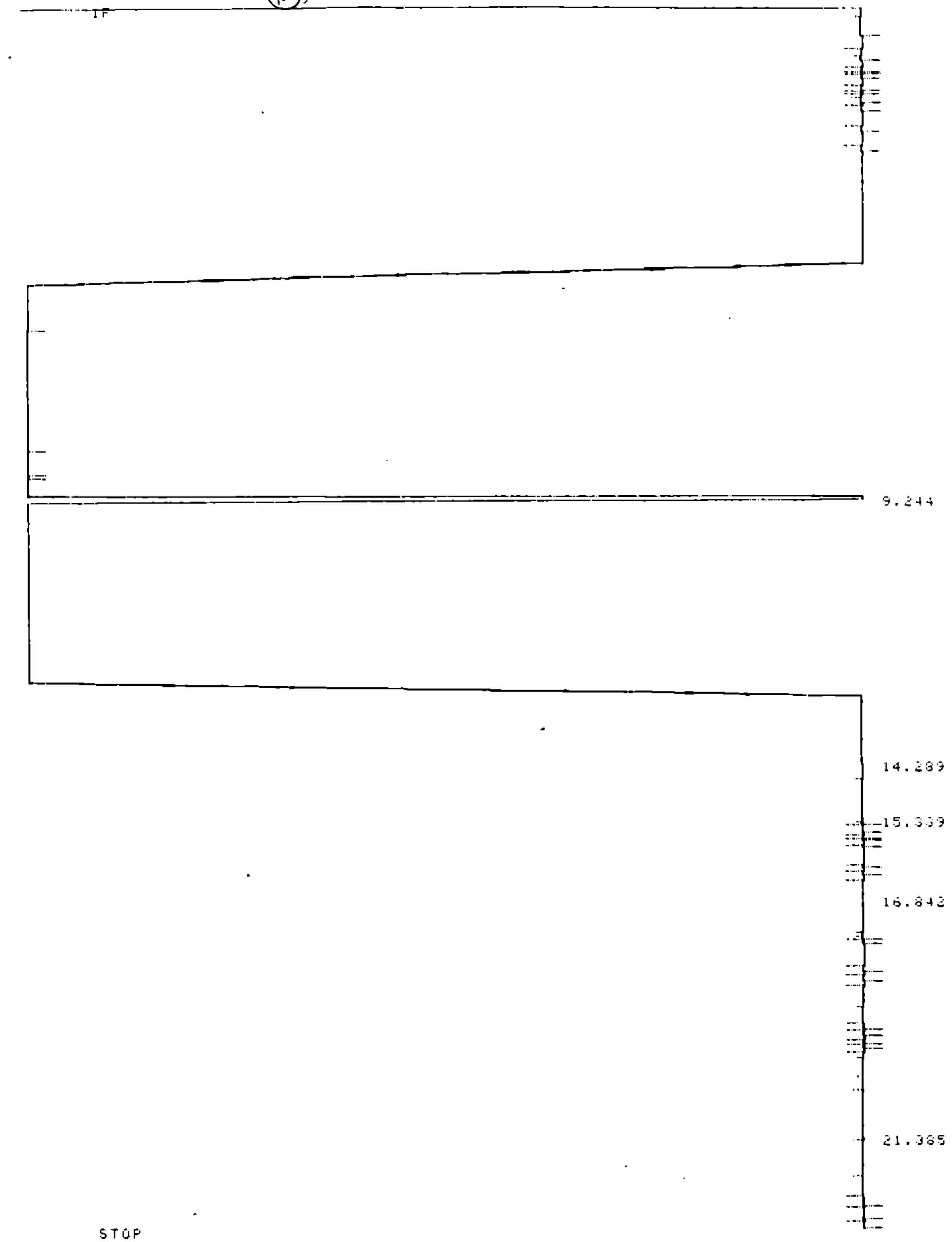
BTEX*
~1.0 ppm

[redacted]

* RUN # 110 SEP 25, 1991 13:04:42

START

(124)



STOP

```
MAIN CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.         NAME
   9.244    BB      198288   .225       14694    7          1.024  BENZENE
  15.339    BB       10185   .331         513                .329
  16.842    BB      285352   .331       14380    9          1.145  ETHYLBENZENE


TOTAL AREA= 493825
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

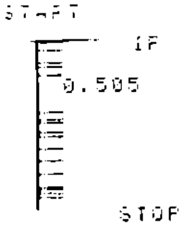
137

```
*  RUN #  111       SEP 25, 1991  13:38:28
```

(138)

.996
ppmv

METHANE



```
RUN#   111              SEP 25, 1991  13:38:28


MATHY CONSTRUCTION

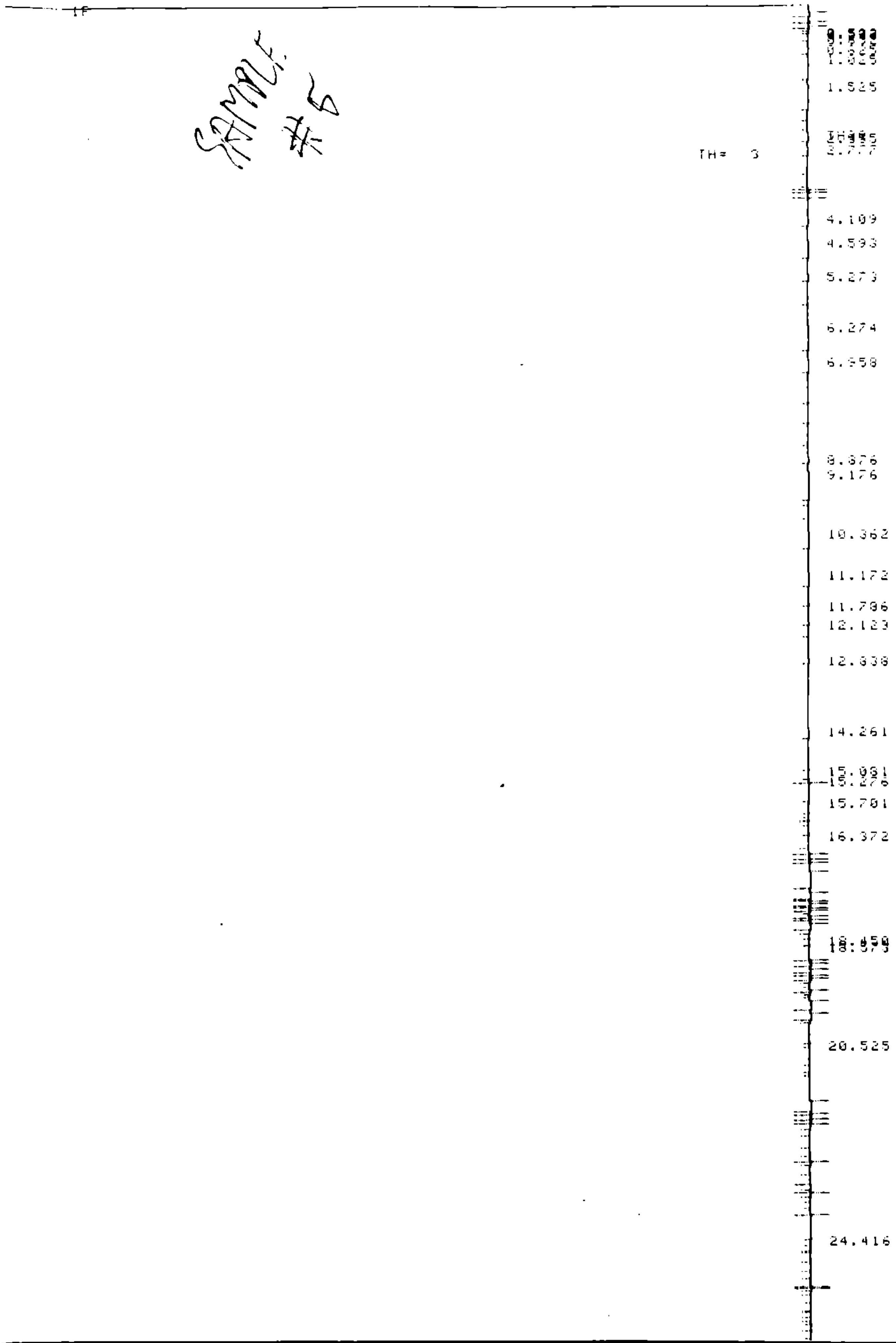
ESTD-AREA
       RT TYPE        AREA   WIDTH      HEIGHT CAL# CONC.       NAME
    .505    BB       36395    .056       10893   1         .950  METHANE


TOTAL AREA=  36395
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =   0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  4
PK WD   =  0.04
```

* RUN # 112 (13) SEP 25, 1991 13:42:53
START



SAMPLE #5

```
RUN#   112              SEP 25, 1991  13:42:53

                          143
MATHY CONSTRUCTION

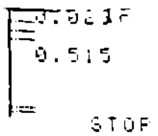
ESTD-AREA
      RT  TYPE          AREA   WIDTH       HEIGHT CAL# CONC.          NAME
     .502   PB         16080   .025         15601   1           .414  METHANE  #3
     .528   BB          4701   .017          4545               .152
     .632   BB         42738   .070         10177   2           .669  ETHANE
     .825   BB         93419   .091         17934              3.176
    1.025   BB         93535   .075         20684              3.019
    1.525   BB         15512   .091          2851               .501
    2.555   PB         30127   .118          4249               .972
    2.777   BB         21977   .118          3098   4           .171  BUTANE
    4.109   BB         10107   .223           754               .326
    4.593   BB         13416   .347           906               .433
    5.273   BB          7606   .100          1262               .245
    6.274   PB         74997   .284          4395   5           .475  PENTANE
    6.958   BB         24392   .194          2098               .787
    8.876   PB          7632   .146           870               .246
    9.176   BB         43453   .320          2261   7           .224  BENZENE
   10.362   VB         63208   .233          4525   6           .347  HEXANE
   11.172   BB         92236   .279          5504              2.977
   11.786   BB           714   .165            72               .023
   12.123   BB          4934   .199           414               .159
   12.838   PB         20324   .196          1725               .656
   15.701   BB          3240   .157           345               .105
   16.372   PB           929   .034           461   9           .004  ETHYLBENZENE
   18.573   BB          1700   .051           552               .055
   20.525   PB          1239   .090           229  10           .005  o-XYLENE
   24.416   BB          1989   .080           227               .035

TOTAL AREA= 694305
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO     =   0
ATT 2^   =  -2
CHT SP   =   1.0
AR REJ   =   500
THRSH    =   3
PK WD    =   0.04
```

```
* RUN #  113        SEP 25, 1991  14:20:44
START
```



```
          STOP
```

(141)

.99% ppm
METHANE

```
RUN#   113            SEP 25, 1991  14:20:44


MATHY CONSTRUCTION

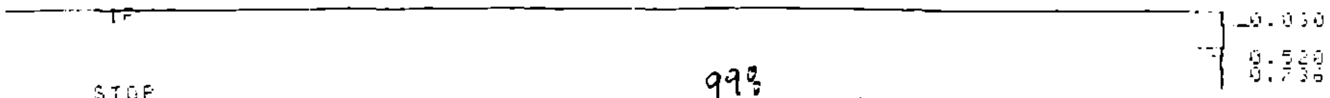
ESTD-AREA
     RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.          NAME
   .023   PB        1147   .011       1769              .037
   .515   BB       38680   .060      10819    1         .996   METHANE


TOTAL AREA=  39827
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  3
PK WD   =  0.04
```

```
*  RUN #  114     SEP 25, 1991  14:22:51
START
```



```
     STOP
```

.99%
METHANE

```
RUN#   114          SEP 25, 1991  14:22:51
```

(142)

```
MATHY CONSTRUCTION

ESTD-AREA
      PT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .030   BB        2654   .016        2719              .086
    .520   BB       36601   .056       10800   1          .942 METHANE
    .736 I BP      118769   .165       12005             3.833

TOTAL AREA= 158024
MUL FACTOR=1.0000E+00

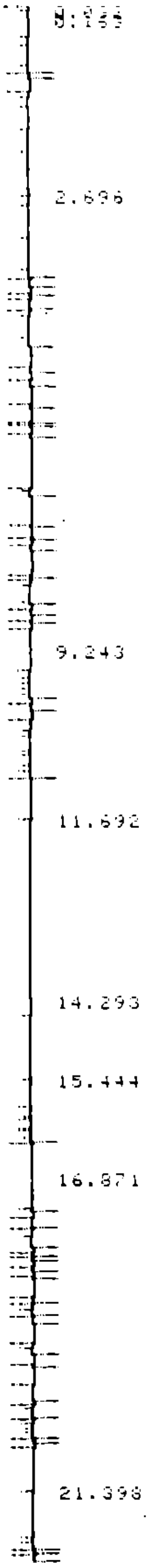



RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  3
PK WD   =  0.04
```

RUN # 115 (143) SEP 25, 1991 14:25:30
START

BTEX
~1.0 ppm

TH= 3



STOP

RUN# 115 SEP 25, 1991 14:25:30

MATHY CONSTRUCTION

ESTD-AREA

```
     .032    BB        2857   .027        1559                .066
     .153    BB       57153   .133        7153               1.351
    9.243    BB      189197   .225       14007    7           .977  BENZENE
   15.444    BB       28983   .220        2133                .935
   16.671    PB      255283   .339       12556    9          1.024  ETHYLBENZENE


TOTAL AREA= 533473
MUL FACTOR=1.0000E+00

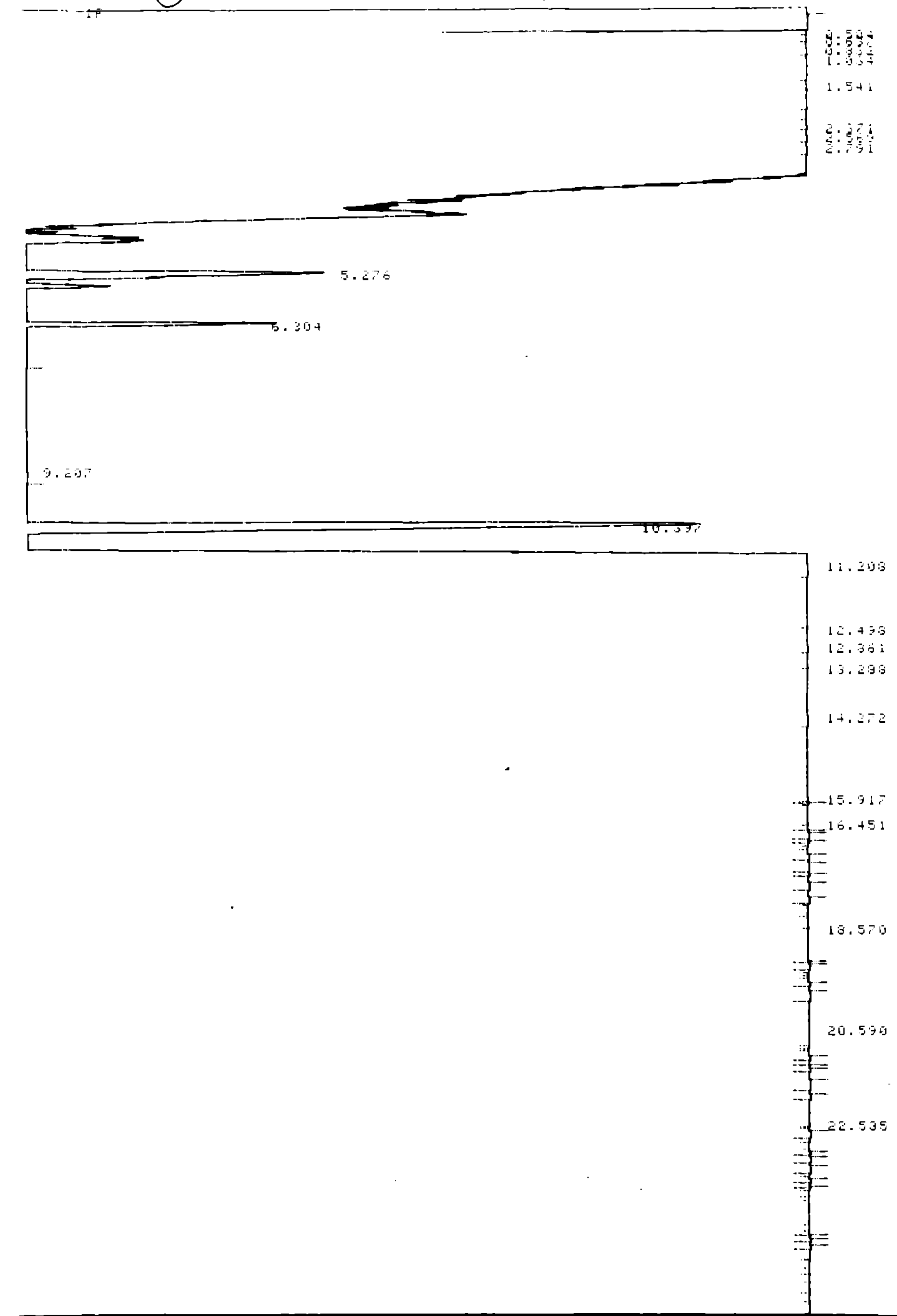



RUN PARAMETERS
ZERO    =  0
ATT 2^  = -2
CHT SP  =  1.0
AR REJ  =  500
THRSH   =  3
PK WD   =  0.04
```

* RUN # 116 (145) SEP 25, 1991 15:04:46

SAMPLE #6

START



```
      TIMETABLE STOP


RUN#   116       (14)  SEP 25, 1991  15:54:46


MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH       HEIGHT CAL# CONC.          NAME
     .504   PB        78553   .047        27675   1         2.306   METHANE
     .637   BB        39859   .072         9180   2          .624   ETHANE
     .832   BB        59994   .095        15831             2.901
    1.034   BB        80092   .073        18247             2.585
    1.541   BB        17226   .100         2863              .556
    2.371   PB         5458   .096          945              .176
    2.569   BB        29641   .119         4164              .957
    2.791   BB        18300   .115         2650   4          .142   BUTANE
    5.276   PB        13520   .159         1415              .436
    6.304   PB        38369   .203         3149   5          .243   PENTANE
    9.207   BB        10430   .161         1082   7          .054   BENZENE
   10.397   PB        45233   .217         3478   6          .248   HEXANE
   11.208   BB        69869   .259         4492             2.255
   12.498   BB        19576  2.589          126              .632
   12.861   BB        22705   .240         1577              .733
   13.288   BB         2126   .270          131              .069
   14.272   BB        34557   .088         6530   8          .207   TOLUENE
   15.917   BB       261842  3.775         1156             8.451
   16.451   PB         4042   .123          549   9          .016   ETHYLBENZENE
   18.570   PB          918   .070          218              .030
   20.590   BB        63562   .449         2362  10          .243   o-XYLENE
   22.535   PB         1284   .096          222              .041

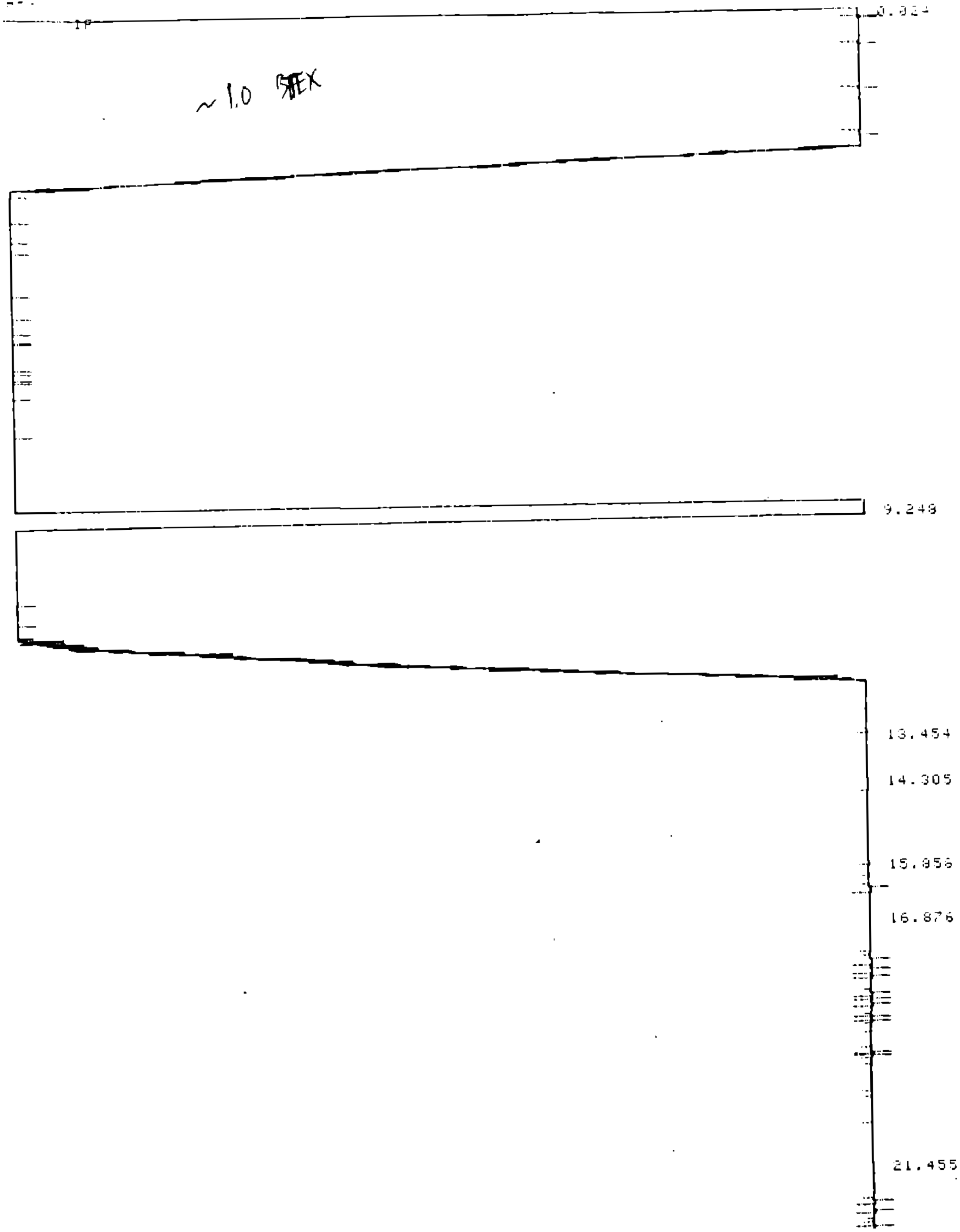

TOTAL AREA= 947056
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO     =   0
ATT 2^   =  -2
CHT SP   =   1.0
AR REJ   =   500
THRSH    =   3
PK WD    =   0.04
```

RUN # 117 [illegible] SEP 25, 1991 15:44:26

START



~1.0 BTEX

STOP

RUN# 117 SEP 25, 1991 15:44:28

MATHY CONSTRUCTION

ESTD-AREA

```
   9.246    PB      186644    .226      13904    7         .974  BENZENE
  14.305    BB      163920    .139      13716    8         .981  TOLUENE
  15.356    BB      151350   6.354        337             4.685
  16.676    BB      257657    .355      12106    9        1.034  ETHYLBENZENE
  21.455    VB      244979    .604       6757   10         .936  o-XYLENE


TOTAL AREA=1006750
MUL FACTOR=1.0000E+00

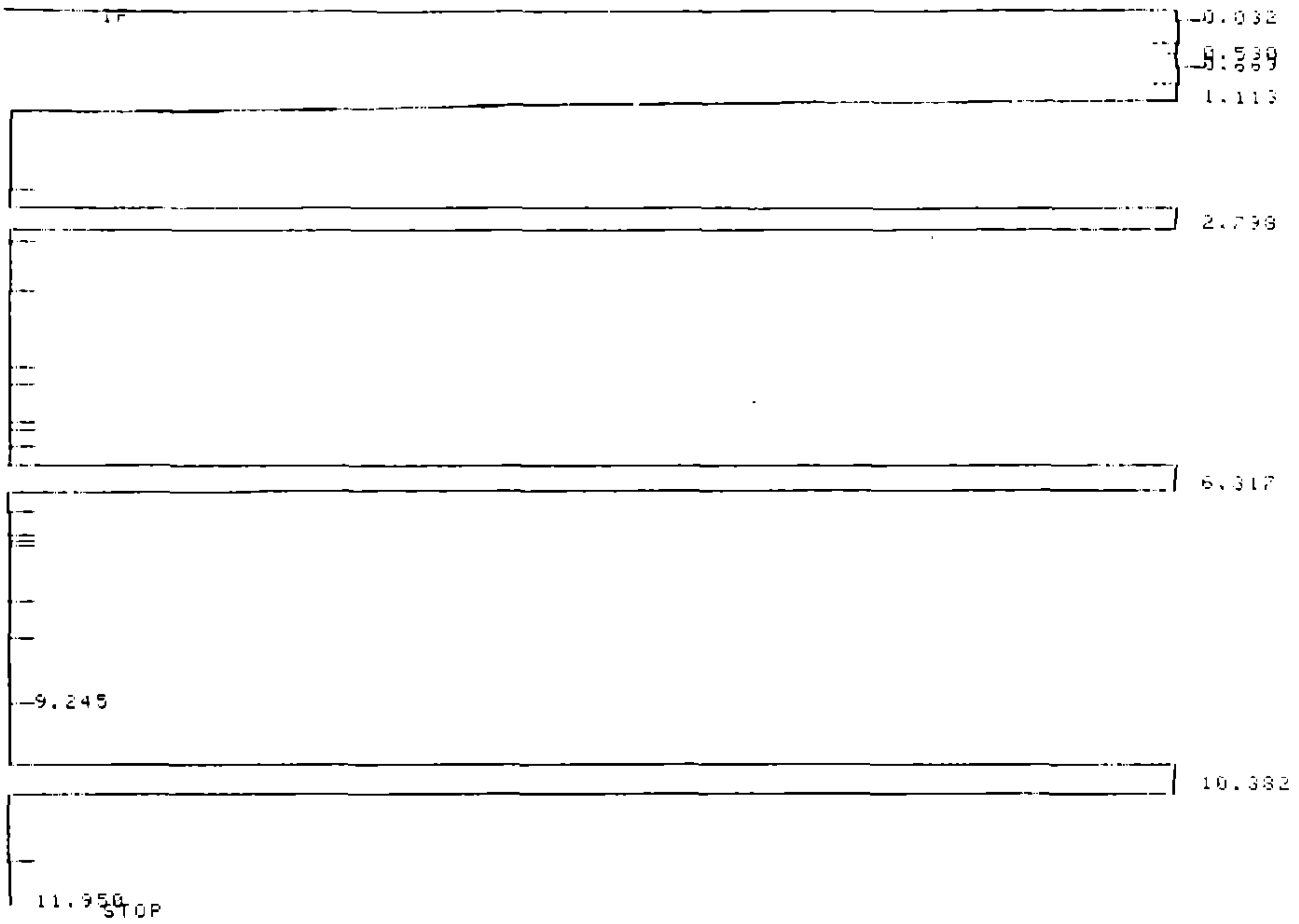


RUN PARAMETERS
ZERO    =   0
ATT 2   =  -2
CHT SP  =   1.0
AR REJ  =   500
THRSH   =   3
PK WD   =   0.04
```

14.5 ppm C1-C6

```
*NOTEPAD

<USE BREAK OR CONTROL-Y TO END>
C1-C6 14.5ppmv STANDARD
*  RUN #  119      SEP 25, 1991  16:19:00
START
```



(149)

```
RUN#   119          SEP 25, 1991  16:19:00

MATHY CONSTRUCTION

ESTD-AREA
      RT TYPE          AREA  WIDTH      HEIGHT CAL# CONC.         NAME
    .530   BB        510518   .061      138622   1        16.504  METHANE
    .669   BB       1009215   .062      269918   2        15.909  ETHANE
   1.113   BB       1491188   .074      334436   3        15.561  PROPANE
   2.798   BB       2078894   .154      225472   4        16.161  BUTANE
   6.317   PB       2588520   .209      206261   5        16.406  PENTANE
  10.382   PB       2970293   .235      210689   6        16.294  HEXANE
  11.950   PB          1168   .161         121              .038


TOTAL AREA=1.0650E+07
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  0
ATT 2^  =  -2
CHT SP  =  1.0
```

```
[illegible]
[illegible]   =   1
[illegible]   =   0,04
```

150

```
PEAK CAPACITY:  1331

ZERO    =  0, -10.570
ATT 2^ = -2
CHT SP =  1.0
AR REJ =  500
THRSH  =  3
PK WD  =  0.04


*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:    5.000

LEVEL: 1             RECALIBRATIONS: 1
LEVEL: 2             RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1      0.515     1 1.5000E+01  3.2275E-05
                   2 9.9800E-01  2.5750E-05
  2      0.654     1 1.4500E+01  1.5665E-05
  3      1.106     1 1.4200E+01  1.0435E-05
  4      2.792     1 1.4600E+01  7.7740E-06
  5      6.306     1 1.4600E+01  6.3380E-06
  6     10.379     1 1.4500E+01  5.4855E-06
  7      9.230     1 1.0000E+00  5.1640E-06
  8     14.299     1 1.0500E+00  5.9850E-06
  9     16.706     1 1.1400E+00  4.0110E-06
 10     20.988     1 1.0800E+00  3.8220E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   TOLUENE
  9   ETHYLBENZENE
 10   o-XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.2275E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  LIST: TIME   @

   0.000 INTG # =  2
   0.000 INTG # =  8
  27.000 STOP


*
```

(151)

APPENDIX F

ANALYTICAL DATA

F.1	PM/Metals
F.2	PM_{10}/CPM
F.3	Aldehydes
F.4	PAH
F.5	Sample Log

APPENDIX F.1

PM/METALS

Run # m26

Client

Date 12/10/91

Plant mpthY

Technician JWood

Sample type MET Filters (m26)

Sheet 2 of 45

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
met 1	m26			0.6406	0.6921		
	33			0.6406	0.6924		
	991 5			0.6406	0.6923 ✓	0.0517	✓ ✓
met 2	m26			0.6144	0.6462		
	34			0.6142	0.6463		
	991 14			0.6143	0.6462 ✓	0.0319	✓
met 3	m26			0.6186	~~0.6544~~		
	69			0.6183	0.6538		
					0.6535		
	991 18			0.6185	0.6537 ✓	0.0352	0.0352 ✓
met FB	m26			0.6414	0.6421		
	17			0.6413	0.6416		
	99 20			0.6414	0.6419 ✓	0.0005	✓
							Reviewed by Julie D Lopez 11/4/91

1181672R

Method 5 Analysis Data Sheet

Run # m26
Client
Date 10/14/91
Plant [illegible] m26
Technician T. WOOD
Sample type Filters (Pm10 M5
Sheet 3 of 45

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
Pm10 1	Pm10 m26 53			0.2152	0.2221		TOTAL =
				0.2152	0.2222		
	~~[illegible]~~ Pm10 19						
				0.2152	0.2222	0.0070	✓
Pm10 2	M26 76			0.2137	0.2215		
				0.2136	0.2216		
	Pm10 14						
				0.2136	0.2216	0.0080	✓
Pm10 3	m26 82			0.2124	0.2212		
				0.2123	0.2208		
	Pm10 12						
				0.2124	0.2210	0.0086	✓
Pm10 FB	m26 ~~m10~~ 4			0.2152	0.2158		
				0.2152	0.2154		
	Pm10 04						
				0.2152	0.2156	0.0004	✓
MET 1	m26 33			0.6406	0.6921		TOTAL = 0.0842
				0.6406	0.6924		
	M5 591 5						
				0.6406	0.6922	0.0516	✓
MET 2	m26 34			0.6144	0.6462		TOTAL = 0.0435
				0.6142	0.6463		
	M5 591 14						
				0.6143	0.6462	0.0319	✓
MET 3	m26 69			~~0.6279~~	~~0.6538~~	~~0.0538~~	TOTAL = 0.0611
				0.6183	0.6538	~~[illegible]~~	
	M5 591 18			0.6186	0.6538		
				0.6184	0.6538	0.0354	✓
MET FB	m26 17			0.6414	0.6421		Reviewed by
				0.6413	0.6426		
	M5 591 20						
				0.6414	0.6418	0.0004	✓ 11/4/91

Method 5 Analysis Data Sheet

1181672R

Run # m26

Client

Date

Plant math

Technician TWood

Sample type STACK Aceton MET PM10 CYC

Sheet 5 of 5

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. ~~(mg)~~ g	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
MET 1	m26	200mL	0.0005	108.2770	108.3100		Blank Corrected
	31			108.2768	108.3102		
	Beaker #						
	RX92			108.2769	108.3101	0.0332	✓ 0.0327
MET 2	m26	75mL	0.0002	104.8617	104.8735		Blank Corrected
	35			104.8617	104.8735		
	Beaker #						
	RX 34			104.8617	104.8735	0.0118	✓ 0.0116
MET 3	m26	340mL	0.0009	103.6004	103.6270		Blank Corrected
	67			103.6005	103.6270		
	Beaker #						
	LL16			103.6004	103.6270	~~0.0266~~	0.0266 ✓ 0.0257
MET FB ~~B~~	m26	225ml		105.7070	105.7075		
	15			105.7071	105.7077		
	Beaker #						
	LL24			105.7070	105.7076	0.0006	✓ 2.6667×10^{-6} g/ml
CYC 1	m26	350ml	0.0005	169.7014	169.7222		Blank Corrected
	57			169.7017	169.7224		
	Beaker #						0.0202
	LL07			169.7016	169.7223	0.0207	✓
CYC 2	m26	150mL	0.0002	101.0530	101.0825		Blank Corrected
	75			101.0530	101.0827		
	Beaker #						0.0294
	LL18			101.0530	101.0826	0.0296	✓
CYC 3	m26	150mL	0.0002	112.9999	113.0285		Blank Corrected
	~~[illegible]~~			112.9997	113.0287		0.0286
	~~Beaker #~~						
	m6			112.9998	113.0286	0.0288	✓
CYC FB	m26	325ml	168.9493	~~[illegible]~~	168.5955		Reviewed by
	3		~~168.9468~~	~~[illegible]~~	168.9457		
	Beaker #		168.9499	168.9491			11/4/91
	LL10		168.9497	~~[illegible]~~	168.9456	0.0004	0.0005 ✓

1.5385×10^{-6} g/ml

Method 5 Analysis Data Sheet

MATHY - Asphalt: Front 1/2
Results in ug/L.

SAM Number	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF
P1-10-010-01a	M26 - 15,16,17 FB	< 6.00	180	* 1.60	* 4.70	* 97.9	* 10.4	221	61.0	* 447	32.5	* 37.5	< 16.0	< 100	278	64.3
P1-10-010-02a	M26 - 31,32,33 Run 1	* 20.7	257	* 2.20	* 9.00	* 403	92.0	328	551	* 1261	124	* 60.3	< 16.0	< 100	997	93.8
P1-10-010-03a	M26 - 34,35,36 Run 2	* 29.8	160	* 1.70	12.4	419	77.6	376	399	* 1222	113	* 47.2	< 16.0	< 100	1073	80.6
P1-10-010-04a	M26 - 67,68,69 Run 3	* 20.2	319	* 1.10	18.8	111	148	420	53.1	* 1440	147	* 38.3	< 16.0	< 100	1265	108
P1-10-010-05a	M6 - 2,3,4 FB	* 16.2	19.2	< 1.00	* 6.30	* 15.1	< 4.00	19.7	* 13.2	< 300	* 6.96	* 54.4	< 16.0	< 100	86.5	< 4.00
P1-10-010-06a	M6 - 25,26,27 Run 1	* 28.0	36.6	< 1.00	11.7	* 31.0	< 4.00	232	180	< 300	* 7.67	* 56.2	< 16.0	< 100	95.2	< 4.00
P1-10-010-07a	M6 - 43,44,45 Run 2	* 21.4	29.9	< 1.00	* 3.30	* 12.8	< 4.00	116	* 9.70	< 300	< 3.00	* 48.2	< 16.0	< 100	* 61.5	< 4.00
P1-10-010-08a	M6 - 60,61,62 Run 3	< 6.00	38.0	< 1.00	* 5.70	* 13.5	< 4.00	166	* 14.0	< 300	25.4	* 48.2	< 16.0	< 100	140	< 4.00
P1-10-010-09a-M	Method Spike	745	914	475	1000	996	935	961	999	892	978	1004	990	1025	1034	874
P1-10-010-10a-M	Method Spike-dup	247	910	476	999	994	939	962	1002	921	1020	1001	996	1002	1036	954
P1-10-010-11a-B	Method Blank	< 6.00	* 1.00	< 1.00	< 2.00	< 6.00	< 4.00	* 3.30	< 3.00	< 300	< 3.00	< 15.0	< 16.0	< 100	* 24.1	< 4.00
P1-10-003-	LCS	2145	1832	974	2083	2007	1928	1942	2048	1980	50.3	2185	2121	1970	2105	45.7

mathy
26

* less than 5 times the detection limit.

Table 2.
MATHY - Asphalt: Front 1/2
Results in total ug.

SAM Number	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF	Final Volume
P1-10-010-01a	M26 - 15,16,17	< 1.50	45.0	* 0.400	* 1.18	* 24.5	* 2.60	55.3	15.3	* 112	8.13	* 9.38	< 4.00	< 25.0	69.5	16.1	0.250
P1-10-010-02a	M26 - 31,32,33	* 5.18	64.3	* 0.550	* 2.25	* 101	23.0	82.0	138	* 315	31.0	* 15.1	< 4.00	< 25.0	249	23.5	0.250
P1-10-010-03a	M26 - 34,35,36	* 7.45	40.0	* 0.425	3.10	105	19.4	94.0	99.8	* 306	28.3	* 11.8	< 4.00	< 25.0	268	20.2	0.250
P1-10-010-04a	M26 - 67,68,69	* 5.05	79.8	* 0.275	4.70	27.8	37.0	105	13.3	* 360	36.8	* 9.58	< 4.00	< 25.0	316	27.0	0.250
P1-10-010-05a	M6 - 2,3,4	* 4.05	4.80	< 0.250	* 1.58	* 3.78	< 1.00	4.93	* 3.30	< 75.0	* 1.74	* 13.6	< 4.00	< 25.0	21.6	< 1.00	0.250
P1-10-010-06a	M6 - 25,26,27	* 7.00	9.15	< 0.250	2.93	* 7.75	< 1.00	58.0	45.0	< 75.0	* 1.92	* 14.1	< 4.00	< 25.0	23.8	< 1.00	0.250
P1-10-010-07a	M6 - 43,44,45	* 5.35	7.48	< 0.250	* 0.83	* 3.20	< 1.00	29.0	* 2.43	< 75.0	< 0.750	* 12.1	< 4.00	< 25.0	* 15.4	< 1.00	0.250
P1-10-010-08a	M6 - 60,61,62	< 1.50	9.50	< 0.250	* 1.43	* 3.38	< 1.00	41.5	* 3.50	< 75.0	6.35	* 12.1	< 4.00	< 25.0	35.0	< 1.00	0.250
P1-10-010-09a-M	Method Spike	74.5%	91.4%	95.0%	100%	99.6%	93.5%	96.1%	99.9%	89.2%	97.8%	100%	99.0%	102%	103%	87.4%	0.100
P1-10-010-10a-M	Method Spike-dup	24.7%	91.0%	95.2%	99.9%	99.4%	93.9%	96.2%	100%	92.1%	102%	100%	99.6%	100%	104%	95.4%	0.100
P1-10-010-11a-B	Method Blank	< 0.600	* 0.100	< 0.100	< 0.200	< 0.600	< 0.400	* 0.330	< 0.300	< 30.0	< 0.300	< 1.50	< 1.60	< 10.0	* 2.41	< 0.40	0.100
P1-10-003-	LCS	107%	91.6%	97.4%	104%	100%	96.4%	97.1%	102%	99.0%	101%	109%	106%	98.5%	105%	91.4%	54.0

} Mathy 2b

* less than 5 times the detection limit.

CALCULATIONS:
Sample Result (ug/L) X Final Volume (L) = Total ug

Reviewed by
11/1/11

MATHY - Asphalt: Back 1/2
Results in ug/L.

SAM Number	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF
P1-10-010-16a	M6 - 1	* 6.30	9.10	< 1.00	< 2.00	* 7.30	< 4.00	* 11.3	* 8.60	* 698	* 3.36	< 15.0	< 16.0	< 100	* 20.2	< 4.00
P1-10-010-17a	M6 - 24	< 6.00	7.80	< 1.00	* 2.70	* 17.9	59.6	26.9	20.8	* 565	* 8.48	< 15.0	< 16.0	< 100	154	< 4.00
P1-10-010-18a	M6 - 42	< 6.00	100	< 1.00	96.0	104	1876	384	290	1815	280	< 15.0	< 16.0	< 100	2326	* 10.4
P1-10-010-19a	M6 - 59	< 6.00	13.5	< 1.00	* 6.66	12.2	56.3	31.8	16.4	* 697	15.8	< 15.0	< 16.0	< 100	127	< 4.00
P1-10-010-12a	M26 - 14	< 6.00	< 1.00	< 1.00	< 2.00	< 6.00	< 4.00	* 4.00	* 3.30	* 449	< 3.00	< 15.0	< 16.0	< 100	< 15.0	< 4.00
P1-10-010-13a	M26 - 30	< 6.00	12.5	< 1.00	* 2.40	* 11.9	* 10.1	17.4	24.5	* 614	* 3.36	< 15.0	< 16.0	< 100	136	< 4.00
P1-10-010-14a	M26 - 37	< 6.00	15.8	< 1.00	* 6.80	* 29.2	27.5	236	44.6	* 695	33.3	< 15.0	< 16.0	< 100	231	< 4.00
P1-10-010-15a	M26 - 66	< 6.00	12.6	< 1.00	< 2.00	* 12.4	< 4.00	19.1	19.4	* 642	* 9.21	< 15.0	< 16.0	< 100	* 55.5	< 4.00
P1-10-010-20a-M	Method Spike	410	882	417	985	970	901	940	881	782	886	868	853	947	907	860
P1-10-010-21a-M	Method Spike-dup	304	784	467	885	865	793	849	985	865	988	968	958	975	1010	978
P1-10-010-22a-B	Method Blank	< 6.00	* 1.00	< 1.00	< 2.00	< 6.00	< 4.00	22.4	* 5.40	< 300	* 7.02	< 15.0	< 16.0	< 100	< 15.0	< 4.00
P1-10-010-	LCS	2145	1832	974	2083	2007	1928	1942	2048	1980	48.0	2185	2121	1970	2105	44.0

* less than 5 times the detection limit.

} Mathy 26

Reviewed by
[illegible]

Table 2.
MATHY - Asphalt: Back 1/2
Results in total ug.

SAM Number	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF	Final Volume	Initial Volume	Digested Volume
P1-10-010-16a	M6 - 1 FB	* 0.700	1.01	< 0.111	< 0.222	* 0.811	< 0.444	* 1.25	* 0.955	* 77.5	* 0.373	< 1.67	< 1.78	< 11.1	* 2.24	< 0.444	0.100	0.5030	0.4530
P1-10-010-17a	M6 - 24 Run 1	< 0.627	0.815	< 0.104	* 0.282	* 1.87	6.23	2.81	2.17	* 59.0	* 0.886	< 1.57	< 1.67	< 10.4	16.1	< 0.418	0.100	1.1615	1.1115
P1-10-010-18a	M6 - 42 Run 2	< 0.633	10.5	< 0.105	10.1	11.0	198	40.5	30.6	191	29.5	< 1.58	< 1.69	< 10.5	245	* 1.10	0.100	0.9605	0.9105
P1-10-010-19a	M6 - 59 Run 3	< 0.631	1.42	< 0.105	* 0.700	1.28	5.92	3.34	1.72	* 73.3	1.66	< 1.58	< 1.68	< 10.5	13.4	< 0.421	0.100	1.0191	0.9691
P1-10-010-12a	M26 - 14 FB	< 0.671	< 0.112	< 0.112	< 0.224	< 0.671	< 0.447	* 0.447	* 0.369	* 50.2	< 0.335	< 1.68	< 1.79	< 11.2	< 1.68	< 0.447	0.100	0.4755	0.4255
P1-10-010-13a	M26 - 30 Run 1	< 0.634	1.32	< 0.106	* 0.254	* 1.26	* 1.07	1.84	2.59	* 64.9	* 0.355	< 1.59	< 1.69	< 10.6	14.4	< 0.423	0.100	0.9246	0.8746
P1-10-010-14a	M26 - 37 Run 2	< 0.631	1.66	< 0.105	* 0.716	* 3.07	2.89	24.8	4.69	* 73.1	3.50	< 1.58	< 1.68	< 10.5	24.3	< 0.421	0.100	1.0069	0.9569
P1-10-010-15a	M26 - 66 Run 3	< 0.631	1.33	< 0.105	< 0.210	* 1.30	< 0.421	2.01	2.04	* 67.5	* 0.969	< 1.58	< 1.68	< 10.5	* 5.84	< 0.421	0.100	1.0163	0.9663
P1-10-010-20a-M	Method Spike	41.0%	88.2%	83.4%	98.5%	97.0%	90.1%	94.0%	88.1%	78.2%	88.6%	86.8%	85.3%	94.7%	90.7%	86.0%	0.100	0.100	0.100
P1-10-010-21a-M	Method Spike-dup	30.4%	78.4%	93.4%	88.5%	86.5%	79.3%	84.9%	98.5%	86.5%	98.8%	96.8%	95.8%	97.5%	101%	97.8%	0.100	0.100	0.100
P1-10-010-22a-B	Method Blank	< 0.636	* 0.106	< 0.106	< 0.212	< 0.636	< 0.424	2.37	* 0.572	< 31.8	* 0.744	< 1.59	< 1.70	< 10.6	< 1.59	< 0.424	0.100	0.8834	0.8334
P1-10-010-	LCS	107%	91.6%	97.4%	104%	100%	96.4%	97.1%	102%	99.0%	96.0%	109%	106%	98.5%	105%	107%			

Mathy 26

* less than 5 times the detection limit.

CALCULATIONS:

Sample Result (ug/L) X Final Volume (L) X Initial Volume / Digested Volume = Total ug

Reviewed by
11/1/91

APPENDIX F.2

PM_{10}/CPM

Run # m26
Client
Date 10/10/91
Plant marthy
Technician T Ward
Sample type pm10 filters (M 26)
Sheet 1 of ~~4~~ 5

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
1	pm10 m26 58			0.2152	0.2221		
				0.2152	0.2222		
	filter #						
pm10	pm10 19			0.2152	0.2222	0.0070	✓
2	m26 76			0.2137	0.2215		
				0.2136	0.2216		
	filter #						
pm10	14			0.2136	0.2216	0.0080	✓
3	m26 82			0.2124	0.2212		
				0.2123	0.2208		
	filter #						
pm10	12			0.2124	0.2210	0.0086	✓
FB	m26 84			0.2152	0.2158		
				0.2152	0.2154		
	filter #						
pm10	04			0.2152	0.2156	0.0004	✓
							Reviewed by G Lopez 11/4/91

Method 5 Analysis Data Sheet

Run # m26

Client ______

Date ______

Plant mathi

Technician TLW

Sample type STACK Aceton MET Small CYC

Sheet 5 of 5

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. ~~(ml)~~ g	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
MET	m26	200mL	0.0005	108.2770	108.3100		BLANK CORRECTED
1	31			108.2768	108.3102		
	Beaker #						
	RX 92			108.2769	108.3101	0.0332	✓ 0.0327
MET	m26	75mL	0.0002	104.8617	104.8735		BLANK CORRECTED
2	35			104.8617	104.8735		
	Beaker #						
	RX 34			104.8617	104.8735	0.0118	✓ 0.0116
MET	m26	340mL	0.0009	103.6004	103.6270		BLANK CORRECTED
3	67			103.6005	103.6270		
	Beaker #						
	LL 16			103.6004	103.6270	~~0.0206~~	0.0266 ✓ 0.0257
MET FB B	m26	225ml		105.7070	105.7075		
	15			105.7071	105.7077		
	Beaker #						
	LL24			105.7070	105.7076	0.0006	✓ 2.6667 × 10⁻⁶ g/ml
CYC	m26	350mL	0.0005	169.7014	169.7222		BLANK CORRECTED
1	57			169.7017	169.7224		
	Beaker #						
	LL07			169.7016	169.7223	0.0207	✓ 0.0202
CYC	m26	150mL	0.0002	101.0530	101.0825		BLANK CORRECTED
2	75			101.0530	101.0827		
	Beaker #						
	LL18			101.0530	101.0826	0.0296	✓ 0.0294
CYC	m26	150mL	0.0002	112.9999	113.0285		BLANK CORRECTED
3	81			112.9997	113.0287		
	Beaker #						
	ml			112.9998	113.0286	0.0288	✓ 0.0286
CYC	m26	335ml	168.9453	~~[illegible]~~	168.9455		Reviewed by
FB	3		~~168.9446~~ 169.9489	~~[illegible]~~	168.9497		Julio Olopez
	Beaker #		168.9497	168.9491			11/4/91
	LL10			~~[illegible]~~	168.9496	0.0004	0.0005 ✓

1.5385 × 10⁻⁶ g/ml

Method 5 Analysis Data Sheet

1181672R

Run # m26

Client

Date 10/10/91

Plant MPHY

Technician T WOOD

Sample type Pmio mech + H2O (M26)

Sheet 4 of ~~4~~ 5

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
mech	m26	575 ml	0.0003	169.9897	169.9944		Blank Corrected
1	56			169.9896	169.9944		
	Beaker #						
	XX60			169.9896	169.9944	0.0048	✓ 0.0040
H2O	m26	850 ml	0.0009	169.8825	164.9184		Blank Corrected
1	55			169.8822	164.9187		
	Beaker #						
	XX51			169.8823	164.9186	~~0.0363~~	0.0363 ✓ 0.0354
mech	m26	500 ml	0.0007	114.0453	114.0538		Blank Corrected
2	74			114.0452	114.0540		
	Beaker #						
	M9			114.0452	114.0539	0.0087	✓ 0.0080
H2O	m26	870 ml	0.0009	167.5352	167.5980		Blank Corrected
2	73			167.5352	167.5981		
	Beaker #						
	LL01			167.5352	167.5980	0.0628	✓ 0.0619
mech	m26	320 ml	0.0004	102.4440	102.4474		Blank Corrected
3	80			102.4439	102.4476		
	Beaker #						
	LL23			102.4440	102.4475	0.0035	✓ 0.0031
H2O	m26	760 ml	0.0008	171.4287	171.4573		Blank Corrected
3	79			171.4287	171.4575		
	Beaker #						
	LL03			171.4287	171.4574	0.0287	✓ 0.0279
mech	m26	450 ml		174.9800	174.9805		
FB	2			174.9800	174.9807		
	Beaker #						
	XX61			174.9800	174.9806	0.0006	✓ 1.3333 x 10-6 g/ml
H2O	m26	770 ml		167.6630	167.6642		Reviewed By
FB	1			167.6634	167.6638		Julie Ooly
	Beaker #						11/4/91
	LL12			167.6632	167.6640	0.0008	✓

0.0394

0.0699

0.0310

1.0390 x 10^{-6} g/ml

1181672R

Method 5 Analysis Data Sheet

APPENDIX F.3

ALDEHYDES

ALn

Radian Work Order P1-10-001

Analytical Report
12/16/91

EMB

EMB
Radian Corporation
RTP, NC

Larry Romesburg

Customer Work Identification EMB Asphalt Test Site 26
Purchase Order Number 275-026-48-17

Contents:

Radian Analytical Services
900 Perimeter Park
Morrisville, NC 27560

919-481-0212

Client Services Coordinator: LJROMESBURG

Certified by: ______________________________

Method:Aldehydes, Mod TO11, HPLC (1)
List:Compendium Method TO-11

Sample ID:	M26-0922-ALD-- FB (IC, PR)	M26-0923-ALD-- 1 (IC, PR)	M26-0923-ALD-- 2 (IC, PR)	M26-0924-ALD-- 3 (IC, PR)
Factor:	10	10	10	10
Results in:	Total ug 01A	Total ug 02A	Total ug 03A	Total ug 04A
Matrix:	Stack	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acetaldehyde	ND	11	3530	110	1220	110	5880	270
Acetone	396	14	2180	140	2280	140	2280	340
Acetophenone/o-Tolualdehyde	ND	23	ND	23	ND	23	ND	23
Acrolein	ND	13	107	13	ND	13	ND	13
Benzaldehyde	ND	21	86.4	2.1	65.3 •	21	757	21
Butyraldehyde/Isobutyraldehyde	ND	18	484	18	253	18	633	18
Crotonaldehyde	ND	18	109	18	48.1 •	18	561	18
2,5-Dimethylbenzaldehyde	ND	24	ND	24	ND	24	ND	26
Formaldehyde	14.5 •	8.9	2070	89	2100	89	11800	220
Hexanal	ND	20	190	20	136	20	519	20
Isophorone	ND	18	ND	18	ND	18	ND	18
Isovaleraldehyde	ND	18	75.1 •	18	ND	18	138	18
MIBK/p-Tolualdehyde	ND	23	ND	23	ND	23	ND	23
Methyl Ethyl Ketone	ND	18	36.9 •	18	ND	18	130	18
Propionaldehyde	ND	14	241	14	105	14	759	14
Quinone	ND	18	122	18	155	18	818	18
m-Tolualdehyde	ND	23	ND	23	ND	23	ND	23
Valeraldehyde	ND	18	89.1 •	18	62.9 •	18	344	18

ND Not detected at specified detection limit

• Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-10-001

Method:Aldehydes, Mod TO11, HPLC (1)
List:Compendium Method TO-11

Sample ID:	M26-0925-ALD--4 (IC, PR)	METHOD BLANK	METHOD SPIKE	Calibration Check QC
Factor:	10	.54	1	1
Results in:	Total ug	Total ug	%	%
	05A	06A	07A	20A
Matrix:	Stack	DNPH	DNPH	ACN

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acetaldehyde	2560	110	ND	0.58	90		115	
Acetone	2710	140	2.74 *	0.73	83		NS	
Acetophenone/o-Tolualdehyde	ND	23	ND	1.2	70 Q		NS	
Acrolein	134	13	ND	0.71	23 Q		NS	
Benzaldehyde	135	21	ND	1.1	119		NS	
Butyraldehyde/Isobutyraldehyde	237	18	ND	0.94	NA		103	
Crotonaldehyde	102	18	ND	0.94	25 Q		NS	
2,5-Dimethylbenzaldehyde	ND	24	ND	1.3	85		NS	
Formaldehyde	3050	89	1.24 *	0.48	87		105	
Hexanal	200	20	ND	1.1	117		NS	
Isophorone	ND	18	ND	0.94	91		NS	
Isovaleraldehyde	85.3 *	18	ND	0.98	NS		NS	
MIBK/p-Tolualdehyde	ND	23	ND	1.2	106		NS	
Methyl Ethyl Ketone	ND	18	ND	0.94	NA		NS	
Propionaldehyde	181	14	ND	0.73	107		165 Q	
Quinone	344	18	ND	0.94	64 Q		NS	
m-Tolualdehyde	ND	23	ND	1.2	93		NS	
Valeraldehyde	134	18	ND	0.98	82		NS	

ND Not detected at specified detection limit
Q Outside control limits
NS Not spiked
* Est. result less than 5 times detection limit
NA Not analyzed

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-10-001

Sample Identifications and Dates

Sample ID	M26-0922-ALD-- FB (IC, PR)	M26-0923-ALD-- 1 (IC, PR)	M26-0923-ALD-- 2 (IC, PR)	M26-0924-ALD-- 3 (IC, PR)	M26-0925-ALD-- 4 (IC, PR)	METHOD BLANK
Date Sampled	09/22/91	09/23/91	09/23/91	09/24/91	09/25/91	
Date Received	10/01/91	10/01/91	10/01/91	10/01/91	10/01/91	10/01/91
Matrix	Stack	Stack	Stack	Stack	Stack	DNPH
	01	02	03	04	05	06
Aldehydes, Mod TO11, HPLC						
Prepared	10/08/91	10/08/91	10/08/91	10/08/91	10/08/91	10/08/91
Analyzed	10/18/91	10/19/91	10/19/91	10/19/91	10/19/91	10/18/91
Analyst	LKK	LKK	LKK	LKK	LKK	LKK
File ID	OWEN130	OWEN131/179	OWEN132/178	OWEN133/177	OWEN134/176	OWEN129
Blank ID	OWEN129	OWEN129	OWEN129	OWEN129	OWEN129	OWEN129
Instrument	V5000	V5000	V5000	V5000	V5000	V5000
Report as	received	received	received	received	received	received

Appendix A

Comments, Notes and Definitions

EMB
Radian Work Order: P1-10-001

General Comments

05-A: The XAD-2/Filter portion of the samples was lost during preparation.

NQ = Not quantitated.

PAH samples were diluted because of high hydrocarbon content.

EMB
Radian Work Order: P1-10-001

J Indicates an estimated value for GC/MS data. This flag is used either when estimating a concentration for tentatively identified compounds where a response factor of 1 is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit.

NA This analyte was not analyzed.

ND This flag (or <) is used to denote analytes which are not detected at or above the specified detection limit. The value to the right of the < symbol is the method specified detection limit for the sample.

NS This analyte or surrogate was not added (spiked) to the sample for this analysis.

Q This quality control standard is outside method or laboratory specified control limits. This flag is applied to matrix spike, analytical QC spike, and surrogate recoveries; and to RPD(relative percent difference) values for duplicate analyses and matrix spike/matrix spike duplicate result.

* The asterisk(*) is used to flag results which are less than five times the method specified detection limit. Studies have shown that the uncertainty of the analysis will increase exponentially as the method detection limit is approached. These results should be considered approximate.

TERMS USED IN THIS REPORT:
Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units -	ug/L	micrograms per liter (parts per billion);liquids/water
	ug/kg	micrograms per kilogram (parts per billion); soils/solids
	ug/M3	micrograms per cubic meter; air samples
	mg/L	milligrams per liter (parts per million);liquids/water
	mg/kg	milligrams per kilogram (parts per million);soils/solids
	%	percent; usually used for percent recovery of QC standards
	uS/cm	conductance unit; microSiemans/centimeter
	mL/hr	milliliters per hour; rate of settlement of matter in water
	NTU	turbidity unit; nephelometric turbidity unit
	CU	color unit; equal to 1 mg/L of chloroplatinate salt

EMB
Radian Work Order: P1-10-001

Sample Identifications and Dates

Sample ID	Method Spike	Calibration Check QC				
Date Sampled						
Date Received	10/01/91	10/01/91				
Matrix	XAD-2/Fi	ACN				
	19	20				
SW8270-Semi-Volatiles						
Prepared	10/11/91					
Analyzed	11/05/91					
Analyst	RK					
File ID	4586534.TI					
Blank ID	4586533.TI					
Instrument	GC/MS B					
Report as	received					
Aldehydes, Mod TO11, HPLC						
Prepared						
Analyzed		10/19/91				
Analyst		LKK				
File ID		OWEN180				
Blank ID						
Instrument		V5000				
Report as		received				

Compound	Run 1	Run 2	Run 3
Phenol	34.63		23.97
2-Chlorophenol			
1,4-Dichlorobenzene			
N-Nitroso-di-n-propylamine			
Acetophenone	93.5	83.405	113.565
3-Methylphenol	5.695	3.465	2.745
Indene	420.855	387.09	426.24
Cyclohexane			995.545
Isophorone			
1,2,4-Trichlorobenzene			
Naphthalene	416.255	776.14	1470.645
4-Chloro-3-methylphenol			
2-Methynaphthalene	407.595	347.54	416.865
1-Methynaphthalene	249.355	212.975	262.12
Acetoaphthylene	41.765	37.745	48.13
Acetoaphthene	14.94	50	50
4-Nitrophenol			
2,4-Dinitrotoluene			
Diethylphthalate		12.245	17.875
Fluorene			
N-nitrosodiphenylamine			
Pentachlorophenol			
Phenanthrene	9.035	7.935	8.49
Anthracene	10.24	8.99	9.62
Di-n-butylphthalate			
Fluoranthene	50	50	50
Diphenylamine			
Pyrene	1	0.625	1
Butylbenzylphthalate			
Bis-(2-ethylhexyl)-phthalate	47.4	390.355	36.87
Di-n-octyphthalate		0.66	1.17

Total ug

		M-26-0922-FB	M-26-923-1	M-26-923-2	M-26-924-3	M-26-Blank	Spike	M-6-918-FB	M-6-919-1	M-6-919-2	M-6-919-3	M-6-Blank
	Dilution Factor	1	100	100	100	1	1	1	1	1	1	1
	Final Extract Volume	5	10	10	6	5	5	5	5	5	5	5
	Target Compounds											
9	Nitrobenzene-d5 (SS)	304.85	779	777	928.2	625.085	536.04	491.05	573.825	478.18	575.765	445.09
10	2-Fluorobiphenyl (SS)	462.095	1023	834	1052.4	771.33	741.09	729.41	757.145	642.93	794.995	675.51
12	Terphenyl-d14 (SS)	341.985	650	690	655.8	773.88	647.43	568.67	566.49	568.265	614.345	564.045
14	Phenol	NA	NA	NA	NA	NA	848.275	NA	34.63	NA	23.97	NA
17	2-Chlorophenol	NA	NA	NA	NA	NA	941.64	NA	NA	NA	NA	NA
19	1,4-Dichlorobenzene	NA	NA	NA	NA	NA	477.905	NA	NA	NA	NA	NA
25	N-Nitroso-di-n-propylamine	NA	NA	NA	NA	NA	520.855	NA	NA	NA	NA	NA
32	Acetophenone	NA	NA	1047	NA	NA	NA	NA	93.54	83.405	113.565	NA
33	3-Methylphenol	NA	NA	NA	NA	NA	NA	NA	5.695	3.465	2.745	NA
37	Indene	NA	NA	NA	NA	NA	NA	NA	420.855	387.09	426.24	NA
39	Cyclohexane	NA	16763	9731	NA	NA	NA	NA	NA	NA	995.545	NA
43	Isophorone	2.235	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
49	1,2,4-Trichlorobenzene	NA	NA	NA	NA	NA	546.905	NA	NA	NA	NA	NA
50	Naphthalene	87.39	NA	NA	1502.4	0.37	533.02	751.275	416.255	776.14	1470.645	259.75
53	4-Chloro-3-methylphenol	NA	NA	NA	NA	NA	999.405	NA	NA	NA	NA	NA
54	2-Methylnaphthalene	NA	3035	2256	1983	NA	1.265	2.36	407.595	347.54	416.865	0.68
63	1-Methylnaphthalene	NA	2016	1451	1217.4	NA	1.165	1.935	249.355	212.875	262.12	0.365
73	Acenaphthylene	NA	NA	NA	NA	NA	NA	NA	41.765	37.745	48.13	NA
75	Acenaphthene	NA	NA	NA	54.6	NA	546.725	NA	14.84	NA	NA	NA
77	4-Nitrophenol	NA	NA	NA	NA	NA	529.995	NA	NA	NA	NA	NA
80	2,4-Dinitrotoluene	NA	NA	NA	NA	NA	491.895	NA	NA	NA	NA	NA
81	Diethylphthalate	5.685	911	112	43.8	12.98	14.41	7.27	NA	12.245	17.875	22.26
83	Fluorene	NA	NA	120	54	NA	NA	NA	NA	NA	NA	0.375
97	N-nitrosodiphenylamine	NA	NA	NA	134.4	NA	NA	0.72	NA	NA	NA	NA
100	Pentachlorophenol	NA	NA	NA	NA	NA	266.17	NA	NA	NA	NA	NA
101	Phenanthrene	NA	290	430	207.6	NA	NA	0.275	9.035	7.935	8.49	NA
102	Anthracene	NA	NA	75	NA	NA	NA	NA	10.24	8.99	9.62	NA
103	Di-n-butylphthalate	7.9	1654	2049	1102.2	6.34	10.77	9.435	NA	NA	NA	11.655
104	Fluoranthene	NA	NA	73	NA	NA	NA	NA	NA	NA	NA	NA
107	Diphenylamine	NA	NA	330	134.4	NA	NA	0.72	NA	NA	NA	NA
110	Pyrene	NA	NA	NA	36	NA	676.415	NA	1.265	0.625	0.805	0.805
111	Butylbenzylphthalate	NA	NA	NA	NA	NA	NA	NA	0.67	NA	NA	NA
114	Bis-(2-ethylhexyl)-phthalate	3.125	716	1069	590.4	13.02	8.5	11.435	47.43	390.355	36.87	19.465
131	Di-n-octylphthalate	NA	NA	NA	NA	NA	NA	NA	0.66	NA	1.17	NA

APPENDIX F.4

PAH

PAH

RADIAN
CORPORATION

Radian Work Order P1-10-001

Analytical Report
01/13/92

EMB

EMB
Radian Corporation
RTP, NC

Larry Romesburg

Customer Work Identification EMB Asphalt Test Site 26
Purchase Order Number 275-026-48-17

Contents:

Radian Analytical Services
900 Perimeter Park
Morrisville, NC 27560

919-481-0212

Client Services Coordinator: LJROMESBURG

Certified by:

Previously Reported on 12/16/91.

RADIAN CORPORATION

EMB
Radian Work Order: P1-10-001

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	M26-0922-PAH-- FB	M26-0923-PAH-1	M26-0923-PAH-2	M26-0924-PAH-3
Factor:	5	1000	1000	600
Results in:	Total ug	Total ug	Total ug	Total ug
	14A	15A	16A	17A
Matrix:	Acetone	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acenaphthene	ND	50	ND	10000	ND	10000	54.6 J	6000
Acenaphthylene	ND	50	ND	10000	ND	10000	ND	6000
Anthracene	ND	50	ND	10000	75.0 J	10000	ND	6000
Benzo(a)anthracene	ND	50	ND	10000	ND	10000	ND	6000
Benzo(a)pyrene	ND	50	ND	10000	ND	10000	ND	6000
Benzo(b)fluoranthene	ND	50	ND	10000	ND	10000	ND	6000
Benzo(g,h,i)perylene	ND	50	ND	10000	ND	10000	ND	6000
Benzo(k)fluoranthene	ND	50	ND	10000	ND	10000	ND	6000
Chrysene	ND	50	ND	10000	ND	10000	ND	6000
Dibenz(a,h)anthracene	ND	50	ND	10000	ND	10000	ND	6000
Dibenzofuran	ND	50	ND	10000	ND	10000	ND	6000
7,12-Dimethylbenz(a)anthracene	ND	100	ND	20000	ND	20000	ND	12000
Fluoranthene	ND	50	ND	10000	73.0 J	10000	ND	6000
Fluorene	ND	50	ND	10000	120 J*	10000	54.0 J	6000
Indeno(1,2,3-cd)pyrene	ND	50	ND	10000	ND	10000	ND	6000
2-Methylnaphthalene	ND	50	3040 J	10000	2260 J	10000	1980 J	6000
Naphthalene	87.4 *	50	1900 J	10000	3100 J	10000	1500 J	6000
Phenanthrene	ND	50	290 J	10000	430 J*	10000	208 J*	6000
Pyrene	ND	50	ND	10000	ND	10000	36.0 J	6000

(See next page for tentatively identified compounds.)

ND Not detected at specified detection limit
J Detected at less than detection limit

* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-10-001

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	M26-0922-PAH--FB	M26-0923-PAH-1	M26-0923-PAH-2	M26-0924-PAH-3
Factor:	5	1000	1000	600
Results in:	Total ug	Total ug	Total ug	Total ug
	14A	15A	16A	17A
Matrix:	Acetone	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
<u>Surrogate Recovery(%)</u>								
2-Fluorobiphenyl	59.4		132 Q		107		135 Q	
Control Limits: 30 to 115								
Nitrobenzene-d5	39.4		102		102		122 Q	
Control Limits: 23 to 120								
Terphenyl-d14	44.8		85.1		90.3		85.8	
Control Limits: 18 to 137								

(See next page for tentatively identified compounds.)

Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-10-001

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	Method Blank	Method Spike
Factor:	5	5
Results in:	Total ug	%
	18A	19A
Matrix:	Stack	XAD-2/Fi

	Method Blank Result	Method Blank Det. Limit	Method Spike Result	Method Spike Det. Limit
Acenaphthene	ND	50	109	
Acenaphthylene	ND	50	NS	
Anthracene	ND	50	NS	
Benzo(a)anthracene	ND	50	NS	
Benzo(a)pyrene	ND	50	NS	
Benzo(b)fluoranthene	ND	50	NS	
Benzo(g,h,i)perylene	ND	50	NS	
Benzo(k)fluoranthene	ND	50	NS	
Chrysene	ND	50	NS	
Dibenz(a,h)anthracene	ND	50	NS	
Dibenzofuran	ND	50	NS	
7,12-Dimethylbenz(a)anthracene	ND	100	NS	
Fluoranthene	ND	50	NS	
Fluorene	ND	50	NS	
Indeno(1,2,3-cd)pyrene	ND	50	NS	
2-Methylnaphthalene	ND	50	NS	
Naphthalene	0.37 J	50	NS	
Phenanthrene	ND	50	NS	
Pyrene	ND	50	135	

ND Not detected at specified detection limit
J Detected at less than detection limit
NS Not spiked

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-10-001

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	Method Blank	Method Spike
Factor:	5	5
Results in:	Total ug	%
	18A	19A
Matrix:	Stack	XAD-2/Fi

	Result	Det. Limit	Result	Det. Limit		
Surrogate Recovery(%)						
2-Fluorobiphenyl	99.2		143 Q			
Control Limits: 30 to 115						
Nitrobenzene-d5	81.8		105			
Control Limits: 23 to 120						
Terphenyl-d14	101		127			
Control Limits: 18 to 137						

Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

RADIAN CORPORATION

Analytical Data Summary

EMB
Radian Work Order: P1-10-001

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
M26-0922-PAH-FB				
	Isophorone	2.24 J*	Total ug	
	Diethylphthalate	5.68 J*	Total ug	
	Di-n-butylphthalate	7.9 J*	Total ug	
	Bis-(2-ethylhexyl)-phthalate	3.12 J*	Total ug	
M26-0923-PAH-1				
	Cyclohexane	16800	Total ug	
	1-Methylnaphthalene	2020 J*	Total ug	
	Diethylphthalate	911 J*	Total ug	
	Di-n-butylphthalate	1650 J*	Total ug	
	Bis-(2-ethylhexyl)-phthalate	716 J*	Total ug	
M26-0923-PAH-2				
	Acetophenone	1050 J*	Total ug	
	Cyclohexane	9730 J*	Total ug	
	1-Methylnaphthalene	1450 J*	Total ug	
	Diethylphthalate	112 J*	Total ug	
	Di-n-butylphthalate	2050 J*	Total ug	
	Diphenylamine	330 J*	Total ug	
	Bis-(2-ethylhexyl)-phthalate	1070 J*	Total ug	
M26-0924-PAH-3				
	1-Methylnaphthalene	1220 J*	Total ug	

EMB
Radian Work Order: P1-10-001

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	Diethylphthalate	43.8 J*	Total ug	
	N-nitrosodiphenylamine	134 J*	Total ug	
	Di-n-butylphthalate	1100 J*	Total ug	
	Diphenylamine	134 J*	Total ug	
	Bis-(2-ethylhexyl)-phthalate	590 J*	Total ug	
Method Blank				
	Diethylphthalate	13.0 J*	Total ug	
	Di-n-butylphthalate	6.34 J*	Total ug	
	Bis-(2-ethylhexyl)-phthalate	13.0 J*	Total ug	
Method Spike				
	Phenol	84.8	%	
	2-Chlorophenol	94.2	%	
	1,4-Dichlorobenzene	95.6	%	
	N-Nitroso-di-n-propylamine	104	%	
	1,2,4-Trichlorobenzene	109	%	
	4-Chloro-3-methylphenol	99.9	%	
	4-Nitrophenol	53.0	%	
	2,4-Dinitrotoluene	98.4	%	
	Pentachlorophenol	26.6	%	
	Di-n-butylphthalate	2.15	%	

EMB
Radian Work Order: P1-10-001

Method:Twenty TICS to be reported (1)
List:

Sample ID:	M26-0922-PAH--FB	M26-0923-PAH-1	M26-0923-PAH-2	M26-0924-PAH-3
Factor:	5	1000	1000	600
Results in:	Total ug	Total ug	Total ug	Total ug
	14A	15A	16A	17A
Matrix:	XAD-2/Fi	Stack	Stack	Stack

	Result Det. Limit	Result Det. Limit	Result Det. Limit	Result Det. Limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-10-001

Method: Twenty TICS to be reported (1) List:	
Sample ID:	Method Blank
Factor:	5
Results in:	Total ug
	18A
Matrix:	XAD-2/Fi

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
M26-0922-PAH-FB				
	Cyclohexene	NQ B	Total ug	
	Oxygenated hydrocarbons	NQ	Total ug	
	2-Hexanol	NQ	Total ug	
	Methylpentenone isomers	NQ B	Total ug	
	Unknown alkoxy alcohol	NQ	Total ug	
	2,2,4,4-Tetramethyl-3-	NQ	Total ug	
	pentanone	NQ	Total ug	
	2,2'-Oxybis-ethanol diacetate	NQ B	Total ug	
	1,3-Diethylbenzene	NQ	Total ug	
	1-Ethenyl-4-ethylbenzene	NQ	Total ug	
	Unknown alcohol	NQ	Total ug	
M26-0923-PAH-1				
	Cyclohexene	NQ B	Total ug	
	Unknown oxygenated hydrocarbon	NQ	Total ug	
	Unknown branched alkane	NQ	Total ug	
	Decane	NQ	Total ug	
	Dimethylundecanes	NQ	Total ug	
	6-Methyldodecane	NQ	Total ug	
	Trimethyldodecanes	NQ	Total ug	

EMB
Radian Work Order: P1-10-001

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
	Dimethylheptadecanes	NQ	Total ug	
	2-Ethyl-1-Decanol	NQ	Total ug	
M26-0923-PAH-2				
	Cyclohexene	NQ B	Total ug	
	Unknown oxygenated hydrocarbon	NQ	Total ug	
	Decane	NQ	Total ug	
	Dimethylundecane isomers	NQ	Total ug	
	2,3,7-Trimethyloctane	NQ	Total ug	
	2,7,10-Trimethyldodecane	NQ	Total ug	
	2,6,11-Trimethyldodecane	NQ	Total ug	
	Branched alkanes	NQ	Total ug	
M26-0924-PAH-3				
	Cyclohexene	NQ B	Total ug	
	4-Methyl-3-penten-2-one	NQ B	Total ug	
	4-Methyloctane	NQ	Total ug	
	1-Ethyl-2-methylbenzene	NQ	Total ug	
	Decane	NQ	Total ug	
	Dimethylundecane isomers	NQ	Total ug	
	Branched alkanes	NQ	Total ug	
	6-Methyldodecane	NQ	Total ug	

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
	Dimethylheptadecane isomers	NQ	Total ug	
Method Blank				
	Cyclohexene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	Oxygenated hydrocarbons	NQ	Total ug	
	2,2'-Oxybis-ethanol diacetate	NQ	Total ug	

EMB
Radian Work Order: P1-10-001

Sample Identifications and Dates

Sample ID	METHOD SPIKE	M26-0922-PAH--FB	M26-0923-PAH-1	M26-0923-PAH-2	M26-0924-PAH-3	Method Blank
Date Sampled		09/22/91	09/23/91	09/23/91	09/24/91	
Date Received	10/01/91	10/01/91	10/01/91	10/01/91	10/01/91	10/01/91
Matrix	DNPH	XAD-2/Fi	Stack	Stack	Stack	XAD-2/Fi
	07	14	15	16	17	18
SW8270-Semi-Volatiles						
Prepared		10/11/91	10/11/91	10/11/91	10/11/91	10/11/91
Analyzed		11/05/91	11/05/91	11/06/91	11/06/91	11/05/91
Analyst		RK	RK	RK	RK	RK
File ID		45B6539.TI	45B6540.TI	45B6545.TI	45B6546.TI	45B6533.TI
Blank ID		45B6533.TI	45B6533.TI	45B6533.TI	45B6533.TI	45B6533.TI
Instrument		GC/MS B	GC/MS B	GC/MS B	GC/MS B	GC/MS B
Report as		received	received	received	received	received
Aldehydes, Mod TO11, HPLC						
Prepared	09/30/91					
Analyzed	10/04/91					
Analyst	LKK					
File ID	LLWA76					
Blank ID	LLWA75					
Instrument	V5000					
Report as	received					
Twenty TICS to be reported						
Prepared		10/11/91	10/11/91	10/11/91	10/11/91	10/11/91
Analyzed		11/05/91	11/05/91	11/06/91	11/06/91	11/05/91
Analyst		RK	RK	RK	RK	RK
File ID		45B6539.TI	45B639.TI	45B6545.TI	45B6546.TI	45B6533.TI
Blank ID		45B6533.TI	45B633.TI	45B6533.TI	45B6533.TI	45B6533.TI
Instrument		GC/MS B	GC/MS B	GC/MS B	GC/MS B	GC/MS B
Report as		received	received	received	received	received

EMB
Radian Work Order: P1-10-001

Sample Identifications and Dates

Sample ID	Method Spike	Calibration Check QC
Date Sampled		
Date Received	10/01/91	10/01/91
Matrix	XAD-2/Fi	ACN
	19	20
SW8270-Semi-Volatiles		
Prepared	10/11/91	
Analyzed	11/05/91	
Analyst	RK	
File ID	4586534.TI	
Blank ID	4586533.TI	
Instrument	GC/MS B	
Report as	received	
Aldehydes, Mod TO11, HPLC		
Prepared		
Analyzed		10/19/91
Analyst		LKK
File ID		OWEN180
Blank ID		
Instrument		V5000
Report as		received

Appendix A

Comments, Notes and Definitions

EMB
Radian Work Order: P1-10-001

General Comments

05-A: The XAD-2/Filter portion of the samples was lost during preparation.

NQ = Not quantitated.

PAH samples were diluted because of high hydrocarbon content.

J Indicates an estimated value for GC/MS data. This flag is used either when estimating a concentration for tentatively identified compounds where a response factor of 1 is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit.

NA This analyte was not analyzed.

ND This flag (or <) is used to denote analytes which are not detected at or above the specified detection limit. The value to the right of the < symbol is the method specified detection limit for the sample.

NS This analyte or surrogate was not added (spiked) to the sample for this analysis.

Q This quality control standard is outside method or laboratory specified control limits. This flag is applied to matrix spike, analytical QC spike, and surrogate recoveries; and to RPD(relative percent difference) values for duplicate analyses and matrix spike/matrix spike duplicate result.

* The asterisk(*) is used to flag results which are less than five times the method specified detection limit. Studies have shown that the uncertainty of the analysis will increase exponentially as the method detection limit is approached. These results should be considered approximate.

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units -	ug/L	micrograms per liter (parts per billion);liquids/water
	ug/kg	micrograms per kilogram (parts per billion); soils/solids
	ug/M3	micrograms per cubic meter; air samples
	mg/L	milligrams per liter (parts per million);liquids/water
	mg/kg	milligrams per kilogram (parts per million);soils/solids
	%	percent; usually used for percent recovery of QC standards
	uS/cm	conductance unit; microSiemans/centimeter
	mL/hr	milliliters per hour; rate of settlement of matter in water
	NTU	turbidity unit; nephelometric turbidity unit
	CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX F.5

SAMPLE ID LOG

TITLE Mathy #26 Sample Log

Project No.
Book No. 25284

RADIAN CORPORATION

From Page No.

	Sample ID	Date	Sample soln / type	Sample wt.	Analysis	Commen
1	M26-0922-PM10-IC-FB	9/22	DI H_2O + probe & imp. rinses	757.0	PM10	Field Bl
2	M26-0922-PM10-ME-FB 70		$MeCl_2$ probe/imp rinse	399.5		
3	M26-0922-PM10-CYC-FB 71		cyclone contents + Acetone rinses	287.5		
4	M26-0922-PM10-F-FB 72		Filter	—		#Pa
5	M26-0922-ALD-IC-FB 73		DNPH	367.9	Aldehydes	
6	M26-0922-ALD-PR-FB 74		$MeCl_2$ probe/nozzle rinse	314.2		
7	M26-0922-PAH-IC-FB 75		HPLC H_2O	944.8	PAH's	
8	M26-0922-PAH-MCR-FB 76		$MeCl_2$ imp/coil rinse	652.3		
9	M26-0922-PAH-MPR-FB 77		$MeCl_2$ probe/nozzle rinse	253.3		
10	M26-0922-PAH-ACR-FB 78		Acetone imp/coil rinse	309.3		
11	M26-0922-PAH-APR-FB 79		Acetone probe/nozzle rinse	81.5		
12	M26-0922-PAH-SM-FB 80		XAD trap	—		
13	M-26-0922-PAH-F-FB 81		Filter	—		
14	M-26-0922-MET-IC-FB 82		HNO_3/H_2O_2	475.5	Metals	
15	M26-0922-MET-PR-FB 83		Acetone probe/nozzle rinse	154.3	~~Metals~~/particulates	
16	M26-0922-MET-APR-FB 84		Nitric probe/nozzle rinse	164.3	Metals	
17	M26-0922 MET-F-FB 85		Filter	—	Metals	
18	M26-0923-WOF-O-1 86	9/23	Waste Oil Fuel			A-1 of
19	M26-0923-WOF-O-1 87					B-2 o
20	M26-0923-WOF-O-1 88					C-3 o
2	~~M26-0923-WOF-O-2~~			VLL		~~A-1 o~~
	~~M26-0923-WOF-O-2~~					~~B-2 o~~
	~~M26-0923-WOF-O-2~~					~~C-3 o~~
21	M26-0923-PAH-IC-1 89		HPLC H_2O	591.2	PAH's	Run
22	M26-0923-PAH-MPR-1 90		$MeCl_2$ probe/nozzle rinse	433.2		
23	M26-0923-PAH-APR-1 91		Acetone probe/nozzle rinse	387.8		
24	M26-0923-PAH-MCR-1 92		$MeCl_2$ imp/coil rinse	379.3		
25	M26-0923-PAH-ACR-1 93		Acetone imp/coil rinse	199.3		
26	M26-0923-PAH-SM-1 94		XAD trap	—		
27	M26-0923-PAH-F-1 95		Filter	—		This filt used for as well (a
28	M26-0923-ALD-IC-1 96		DNPH	1073.3	Aldehydes	
29	M26-0923-ALD-PR-1 97		$MeCl_2$ probe/nozzle rinse	299.0		
30	M26-0923-MET-IC-1 98		HNO_3/H_2O_2	924.6	Metals VLL	
31	M26-0923-MET-PR-1 99		Acetone probe/nozzle rinse	165.2	part./~~Metals~~	
32	M26-0923-MET-APR-1 100		Nitric probe/nozzle rinse	210.3	Metals	

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

Project No.

Book No. 25284 TITLE Mathy #26 Sample Log

From Page No.

	Sample ID		Date	Sample sdn./type	Sample wt.	Analysis	Comm
33	M26-0923-MET-F-1	101	9/23	Filter	—	part/Metals	Run
34	M26-0923-MET-F-2	102		Filter	—	part/Metals	Run
35	M26-0923-MET-PR-2	103		Acetone probe/nozzle rinse	57.7	particulates	
36	M26-0923-MET-APR-2	104		Nitric probe/nozzle rinse	111.6	Metals	
37	M26-0923-MET-IC-2	105		HNO_3/H_2O_2	1006.9	⇓	
38	M26-0923-PAH-F-2	106		Filter	—	PAH's	1
39	M26-0923-PAH-IC-2	107		HPLC H_2O	569.2		
40	M26-0923-PAH-MPR-2	108		$MeCl_2$ probe/nozzle rinse	192.0		
41	M26-0923-PAH-APR-2	109		Acetone probe/nozzle rinse	74.0		
42	M26-0923-PAH-MCR-2	110		$MeCl_2$ imp./coil rinse	279.3		
43	M26-0923-PAH-ACR-2	111		Acetone imp./coil rinse	356.1		
44	M26-0923-PAH-SM-2	112		XAD trap	—	↓	↓
45	M26-0923-BHF 1	113		Baghouse ash	—		A-10
46	M26-0923-BHF-1	114			—		B-20
47	M26-0923-BHF-1	115	↓	↓	—		C-30
48	M26-0923-ALD-IC-2	116	9/23	DNPH	1055.8	Aldehydes	Run
49	M26-0923-ALD-PR-2	117	↓	$MeCl_2$ probe/nozzle rinse	333.9	⇓	⇓
50	M26-0924-ALD-IC-3	118	9/24	DNPH	1074.0	Aldehydes	Run
51	M26-0924-ALD-PR-3	119		$MeCl_2$ probe/nozzle rinse	262.9	⇓	
52	M26-0924-ASC-1	120		Asphalt Cement	—		A-10
53	M26-0924-ASC-1	121			—		B-20
54	M26-0924-ASC-1	122		↓	—		C-30
55	M26-0924-PM10-IC-1	123		DIH_2O + probe + imp rinses	849.8	PM-10	Run
56	M26-0924-PM10-ME-1	124		$MeCl_2$ probe/imp rinses	548.8		
57	M26-0924-PM10-CYC-1	125		Cyclone contents + Acetone rinses	311.7		
58	M26-0924-PM10-F-1	126		Filter	—	↓	↓ #1
59	M26-0924-PAH-IC-3	127		HPLC H_2O	494.2	PAH's	Run
60	M26-0924-PAH-ACR-3	128		Acetone imp/coil rinse	231.8		
61	M26-0924-PAH-MCR-3	129		$MeCl_2$ imp./coil rinse	360.6		
62	M26-0924-PAH-APR-3	130		Acetone probe/nozzle rinse	431.9		
63	M26-0924-PAH-MPR-3	131		$MeCl_2$ probe/nozzle rinse	365.5		
64	M26-0924-PAH-SM-3	132		XAD trap	—		
65	M26-0924-PAH-F-3	133		Filter	—	↓	↓

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

From Page No.__

	Sample ID	Date	Sample soln./type	Sample wt.	Analysis	Co
66	M26-0924-MET-IC-3 134	9/24	HNO$_3$/H$_2$O$_2$ rinse	1016.3	Metals	Ru.
67	M26-0924-MET-PR-3 135		Acetone probe/nozzle	260.5	particulates	
68	M26-0924-MET-APR-3 136		Nitric probe/nozzle rinse	384.2	Metals	
69	M26-0924-MET-F-3 137		Filter	—	part./Metals	\
70	M26-0924-WOF-O-2 138		Waste Oil Fuel	728.3		A-
71	M26-0924-WOF-O-2 139			751.1		B-
72	M26-0924-WOF-O-2 140			785.3		C-
73	M26-0925-PM10-IC-2 141	9/25	DIH$_2$O + probe contents IMP rinse	876.5	PM10	Ru
74	M26-0925-PM10-ME-2 142		MeCl$_2$ probe/imp. rinse	475.4		
75	M26-0925-PM10-CYC-2 143		Cyclone contents + ACE RINSE	111.1		
76	M26-0925-PM10-F-2 144		FILTER TARE= 0.2136	—		
77	M26-925-ALD-IC-4 145		DNPH	1124.8	ALDEHYDE	Ru
78	M26-0925-ALD-PR-4 146		MeCl$_2$ probe/nozzle rins	263.0	ALDEHYDE	Ru
79	M26-0925-PM10-IC-3 147		DIH$_2$O impcontents & rinse	761.9	PM10	
80	M26-0925-PM10-ME-3 148		MECl$_2$ Probe/imp. rinse	366.8		
81	M26-0925-PM10-CYC-3 149		Cyclone contents + ACE RINSE	110.2		

To Pag

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

APPENDIX G

CALIBRATION DATA SHEETS

Meter Box Calibration

Post Test - Mathey

Date	10/3/91	Cal Meter #	6830284	Pbar	29.9
Box #	N-30	Cal Meter Yd	0.9967	Vaccum	10 "Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Cal. Temp Out	Meter Temp In	Meter Temp Out	Time	Yd	Delta H@
1.1	-0.07	Final	43.149	52.743	68	67	70	68	6	1.0164	1.92
		Init.	39.743	49.400	68	67	71	69			
		Total	3.406	3.343	Avg.	67.5	Avg.	69.5			
1.1	-0.07	Final	49.823	59.250	66	67	71	68	11	1.0252	1.87
		Init.	43.515	53.100	66	67	70	69			
		Total	6.308	6.150	Avg.	66.5	Avg.	69.5			
1.1	-0.07	Final	57.947	67.182	67	67	70	69	14.25	1.0237	1.90
		Init.	49.823	59.250	68	68	73	70			
		Total	8.124	7.932	Avg.	67.5	Avg.	70.5			
									Average	1.0218	1.90

Meter Box Calibration

Post Test - Mathey

Date 10/4/91
Cal Meter # 6830284
Pbar 29.88

Box # N-32
Cal Meter Yd 0.9967
Vaccum 12 "Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Out	Meter Temp In	Out	Time	Yd	Delta H@
1.0	-0.06	Final	32.507	47.087	65	65	71	69	18	1.0028	1.95
		Init.	22.882	37.450	66	66	73	70			
		Total	9.625	9.637	Avg.	65.5	Avg.	70.8			
1.0	-0.06	Final	37.323	51.885	66	66	73	70	9	1.0083	1.95
		Init.	32.507	47.087	67	67	74	71			
		Total	4.816	4.798	Avg.	66.5	Avg.	72.0			
1.0	-0.06	Final	44.398	58.962	67	67	73	71	13.25	1.0042	1.96
		Init.	37.323	51.885	67	67	75	71			
		Total	7.075	7.077	Avg.	67.0	Avg.	72.5			
									Average	1.0051	1.95

Meter Box Calibration

Post Test - Mathey

Date	10/15/91	Cal Meter #	6830284	Pbar	29.88
Box #	N-33	Cal Meter Yd	0.9967	Vaccum	11 " Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Out	Meter Temp In	Out	Time	Yd	Delta H@
1.1	-0.07	Final	67.375	76.428	70	70	73	70	11	0.9810	1.89
		Init.	61.057	70.000	72	72	78	72			
		Total	6.318	6.428	Avg.	71.0	Avg.	73.3			
1.1	-0.07	Final	73.116	82.298	73	73	80	75	10	0.9788	1.89
		Init.	67.375	76.428	72	72	77	73			
		Total	5.741	5.870	Avg.	72.5	Avg.	76.3			
1.1	-0.007	Final	79.410	88.777	73	73	82	78	11	0.9765	1.90
		Init.	73.116	82.298	72	72	79	75			
		Total	6.294	6.479	Avg.	72.5	Avg.	78.5			
									Average	0.9788	1.89

APPENDIX H

SAMPLE EQUATIONS

```
                                        SAMPLE CALCULATIONS
                                        -------------------

COMPANY             : BORGESS MEDICAL CENTER
PLANT SITE          : KALAMAZOO, MI           INPUT PARAMETERS:
SAMPLING LOCATION   : BAGHOUSE OUTLET        : ==================================================================================
DATE                : 09/07/91               : As        =  551.55  sq.in.    Ps       =   29.48 in. Hg   PM COLLECT.=  0.0102 grams :
                                             : Cp        =    0.84            %CO2     =    4.69     AVG SQRT DEL.P =  0.2546 IN H2O:
EXAMPLE #1: PM/Metals , RUN 02               : Dn        =   0.313 in.        %N2      =   80.26          MOISTURE    =  211.60 grams :
                                             : Kp        =   84.59            %O2      =   15.05           COLLECTED                 :
STANDARD CONDITIONS: 68 F, 29.92 in Hg       : P(std)    =   29.92 in. Hg     T(std)   =   68.00 F        SAMPL TIME  =     240 min.  :
                                             : Pb        =   29.52 in. Hg     Tm(avg)  =  110.09 F        Ts(avg)     =  294.42 F     :
                                             : Pmg(avg)=      0.45 in. H2O    Vm       =   91.40 ft^3     Y           =  1.0108       :
                                             : ==================================================================================
    1)  Volume of dry gas sampled at standard conditions:
                                                           Pm = Pmg/13.6 + Pb
                        Y * Vm * (T(std) + 460) * Pm       Pm =       29.5530
           Vm(std) =  ------------------------------
                           P(std) * (Tm(avg) + 460)

           Vm(std) =      (1.011 * 91.40 * (68 + 460)  * 29.55) / (29.92 * ( 110.09 + 460))

           Vm(std) =              84.52 dscf


    2)  Volume of water vapor at standard conditions:


           Vw(gas) = 0.04707 ft3/g * (moisture collected)

           Vw(gas) =      ( 0.04707 * 211.60 )

           Vw(gas) =               9.95 scf


    3)  Percent moisture in stack:

                      100 * Vw(gas)
           %V =     -------------------
                    Vm(std) + Vw(gas)

           %V =     (100 *9.95) / (84.52 + 9.95)

           %V =        10.53


    4)  Mole fraction of dry stack gas:

                     100 - %V
           MFd =     --------
                       100

           MFd =    (100 - 10.53) / 100

           MFd =       0.895
```

DEFINITION OF TERMS

SYMBOL	DEFINITIONS	UNITS
As	AREA OF STACK	in.^2
Cp	PITOT COEFFICIENT	--
Ca	CONCENTRATION OF PARTICULATE	grains/ft^3
Dn	DIAMETER OF SAMPLING NOZZLE	in.
ER	EMISSION RATE OF PARTICULATE	lb/hr
Kp	PITOT TUBE COEFFICIENT	--
MFd	MOLE FRACTION OF DRY STACK GAS	--
MWd	MOLECULAR WEIGHT OF DRY STACK GAS	lb/lb-mole
MWw	MOLECULAR WEIGHT OF WET STACK GAS	lb/lb-mole
	MOISTURE COLLECTED IN IMPINGERS	grams
P(std)	STANDARD PRESSURE (29.92 in. Hg)	in. Hg
Pb	BAROMETRIC PRESSURE	in. Hg
Pmg(avg)	AVERAGE GAUGE METER PRESSURE	in. Hg
Ps	ABSOLUTE STACK PRESSURE	in. Hg
	PARTICULATE CATCH	grams
Qsd	AVERAGE STACK DRY VOLUMETRIC FLOW RATE	dry ft^3/min.
%CO2	PERCENT CO2 IN STACK GAS	--
%N2	PERCENT N2 IN STACK GAS	--
%O2	PERCENT O2 IN STACK GAS	--
%V	PERCENT MOISTURE IN STACK	--
%XS	PERCENT EXCESS AIR	--
	TOTAL SAMPLING TIME	min.
T(std)	STANDARD TEMPERATURE (68 F)	F
Tm(avg)	AVERAGE TEMPERATURE OF THE METER	F
Ts(avg)	AVERAGE TEMPERATURE OF THE STACK	F
Vm	TOTAL METERED VOLUME	ft^3
Vm(std)	STANDARD METERED VOLUME	dry standard ft^3
Vw(gas)	VOLUME OF WATER IN STACK GAS	standard ft^3
Vs	VELOCITY OF STACK GAS	ft/min
Y	TEST METER CALIBRATION COEFFICIENT	--

10) Excess air (%):

$$\%XS = \frac{100 * \%O2}{(0.264 * \%N2) - \%O2}$$

%XS = (100 * 15.05) / ((0.264 * 80.26) - 15.05)

%XS = 244.53

11) Concentration of particulate:

Ca = (particulate catch) / Vm(std) / 453.59 * 7000

Ca = 0.0102 / 84.52 / 453.59 * 7000

Ca = 0.00186 grains/dscf

12) Particulate Emissions Rate:

ER = (concentration) * (Qsd) * 60 / 7000

ER = 0.0019 * 2444.95 * 60 / 7000

ER = 0.039 lb/hr

5) Average molecular weight of dry stack gas:

```
MWd = (0.44 * %CO2) + (0.32 * %O2) + (0.28 * %N2)

MWd =  (0.44 * 4.69) + (0.32 * 15.05) + (0.28 * 80.26)

MWd =     29.35 lb/lb-mole
```

6) Average molecular weight of wet stack gas:

```
MWw = MWd * MFd + 18.0 * (1.0 - MFd)

MWw = 29.35 * 0.895 + 18.0 * (1.0 - 0.895)

MWw =    28.16 lb/lb-mole
```

7) Stack velocity (feet/min) at stack conditions:

```
    Vs = Kp*Cp*[SQRT(dP)]avg*(SQRT[(Ts)avg])*[SQRT(1/Ps*MWw)]*60

    Vs =  84.59 * 0.84 * 0.25 * SQRT[(294.42 +460) / (29.48 * 28.16)]

Vs =      17.24665 fps
          1034.799 fpm
```

8) Average stack dry volumetric flow rate:

```
                 Vs * As * MFd * (T(std)+460) * Ps
Qsd =     --------------------------------------
          144 sq.in./cu.ft. * (Ts(avg) + 460) * P(std)

Qsd =     17.25 * 551.55 * 0.895 * (68.0 + 460) * 29.48 / (144 * (294.42 + 460) * 29.92)

Qsd =      2444.95 dscfm
          69.24238 dscmm
```

9) Isokinetic sampling rate (%):

```
       1039.5746 * Vm(std) * (Ts(avg) + 460)
%I =  ---------------------------------------
      Vs * samp. time * Ps * MFd * (Dn)^2

%I =      (1039.5746 * 84.52 * (294.42 + 460)) / (1034.80 * 240 * 29.48 * 0.895 * (0.313)^2)

%I =        103.24
```

APPENDIX I

PROJECT PARTICIPANTS

PROJECT PARTICIPANTS

RADIAN CORPORATION

Rod Brown
Geoff Johnson
Jack Johnson
Vince Laura
Julie Lopez
Tom McDonald
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Judy Smith

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Dennis Holzschuh

APPENDIX J

SAMPLING AND ANALYTICAL PROTOCOLS

J.1 PM/Metals
J.2 PM_{10}/CPM
J.3 Aldehydes
J.4 PAH
J.5 CEM and GC

APPENDIX J.1

PM/METALS

METHODOLOGY FOR THE DETERMINATION OF METALS EMISSIONS IN EXHAUST GASES FROM INCINERATION PROCESSES

1. Applicability and Principle

1.1 Applicability. This method is applicable for the determination of arsenic (As), beryllium (Be), cadmium (Cd), total chromium (Cr), lead (Pb), mercury (Hg), nickel (Ni), and zinc (Zn) emissions from municipal waste incinerators and similar combustion processes. These elements are referred to hereafter as the primary metals. This method may also be used for the determination of antimony (Sb), barium (Ba), copper (Cu), manganese (Mn), phosphorus (P), selenium (Se), silver (Ag), and thallium (Tl) emissions from these sources. These elements are referred to hereafter as the secondary metals.

In addition, the method may be used to determine particulate emissions by following the additional procedures described. Modifications to the sample recovery and analysis procedures described in this protocol for the purpose of determining particulate emissions may potentially impact the front half mercury determination.*

1.2 Principle. The stack sample is withdrawn isokinetically from the source, with particulate emissions collected in the probe and on a heated filter and gaseous emissions collected in a series of chilled impingers containing a solution of dilute nitric acid in hydrogen peroxide in two impingers, and acidic potassium permanganate solution in two (or one) impingers. Sampling train components are recovered and digested in separate front and back half fractions. Materials collected in the sampling train are digested with acid solutions to dissolve inorganics and to remove organic constituents that may create analytical interferences. Acid digestion is performed using conventional Parr® Bomb or microwave digestion techniques. The

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*Field tests to date have shown that of the total amount of mercury measured by the method, only 0 to <2% was measured in the front half. Therefore, it is tentatively concluded, based on the above data, that particulate emissions may be measured by this train, without significantly altering the mercury results.

nitric acid and hydrogen peroxide impinger solution, the acidic potassium permanganate impinger solution, and the probe rinse and digested filter solutions are analyzed for mercury by cold vapor atomic absorption spectroscopy (CVAAS). Except for the permanganate solution, the remainder of the sampling train fractions are analyzed for As, Be, Cd, Cr, Pb, Ni, and Zn (and Sb, Ba, Cu, Mn, P, Se, Ag, and Tl, if desired) by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of As, Cd, and Pb (and Sb, Se, and Tl, when measured) if these elements require greater analytical sensitivity than can be obtained by ICAP. Additionally, if desired, the tester may use AAS for analyses of all target metals if the resulting in-stack method detection limits (combined sampling and analytical detection limits) meet the data quality objectives of the testing program. For convenience, aliquots of each digested sample fraction can be combined proportionally for a single analytical determination. The efficiency of the analytical procedure is quantified by the analysis of spiked quality control samples containing each of the target metals including actual sample matrix effects checks.

2. Range, Sensitivity, Precision, and Interferences

2.1 Range. For the analyses described in this methodology and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per milliliter (ng/ml) to micrograms per milliliter (ug/ml) range in the analytical finish solution can be analyzed using this technique. Samples containing greater than approximately 50 ug/ml of arsenic, chromium, or lead should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20 ug/ml of cadmium should be diluted to that level before analysis.

2.2 Analytical Sensitivity. ICAP analytical detection limits for the primary [and secondary] metals in the sample solutions (based on SW-846, Method 6010) are approximately as follows: As (53 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Pb (42 ng/ml), Ni (15 ng/ml), Zn (2 ng/ml) [Sb (32 ng/ml), Ba (2 ng/ml), Cu (6 ng/ml), P (75 ng/ml), Mn (2 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Tl (40 ng/ml)]. The actual method detection limits are sample dependent and may vary as the sample matrix may affect the limits. The analytical detection limits for the primary [and secondary] metals in sample solutions analyzed by direct aspiration AAS (based on SW-846, Method 7000) are approximately as follows: As (2 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Pb (100 ng/ml), Ni (40 ng/ml), Zn (5 ng/ml) [Sb (200 ng/ml), Ba (100 ng/ml), Cu (20 ng/ml), Mn (10 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml)]. The detection limit for mercury by CVAAS is approximately 0.2 ng/ml. The use of GFAAS can give added sensitivity compared to the use of direct aspiration AAS for the following primary and secondary metals: As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Pb (1 ng/ml), Sb (3 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml).

Using (1) the procedures described in this method, (2) the analytical detection limits described in the previous paragraph, (3) a volume of 300 ml for the front half and 150 ml for the back half samples, and (4) a stack gas sample volume of 1.25 m^3, the corresponding in-stack method detection limits are presented in Table A-1 and calculated as shown:

$$\frac{A \times B}{C} = D$$

where: A = analytical detection limit, ug/ml.
B = volume of sample prior to aliquot for analysis, ml.
C = stack sample volume, dscm (dsm^3).
D = in-stack detection limit, ug/m^3.

Values in Table A-1 are calculated for the front and back half and/or the total train.

To ensure optimum sensitivity in obtaining the measurements, the concentrations of target metals in the solutions are suggested to be at least ten times the analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, this concentration can be as low as approximately three times the analytical detection limit. In all cases, repetitive analyses, method of standard additions (MSA), serial dilution, or matrix spike addition should be used to establish the quality of the data.

Actual in-stack method detection limits will be determined based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be made more sensitive than those shown in Table A-1 for a specific test by using one or more of the following options:

TABLE A-1. IN-STACK METHOD DETECTION LIMITS (ug/m^3) FOR TRAIN FRACTIONS USING ICAP AND AAS

Metal	Front Half Fraction 1 Probe and Filter	Back Half$_1$ Fraction 2 Impingers 1-3	Back Half$_2$ Fraction 3 Impingers 4-5	Total Train
		Primary Metals		
Arsenic	12.7 (0.3)*	6.4 (0.1)*		19.1 (0.4)*
Beryllium	0.07 (0.05)*	0.04 (0.03)*		0.11 (0.08)*
Cadmium	1.0 (0.02)*	0.5 (0.01)*		1.5 (0.03)*
Chromium	1.7 (0.2)*	0.8 (0.1)*		2.5 (0.3)*
Lead	10.1 (0.2)*	5.0 (0.1)*		15.1 (0.3)*
Mercury	0.05**	0.03**	0.03**	0.11**
Nickel	3.6	1.8		5.4
Zinc	0.5	0.3		0.8
		Secondary Metals		
Antimony	7.7 (0.7)*	3.8 (0.4)*		11.5 (1.1)*
Barium	0.5	0.3		0.8
Copper	1.4	0.7		2.1
Manganese	0.5 (0.2)*	0.2 (0.1)*		0.7 (0.3)*
Phosphorus	18	9		27
Selenium	18 (0.5)*	9 (0.3)*		27 (0.8)*
Silver	1.7	0.9		2.6
Thallium	9.6 (0.2)*	4.8 (0.1)*		14.4 (0.3)*

( )* Detection limit when analyzed by GFAAS.

** Detection limit when analyzed by CVAAS.

Actual method in-stack detection limits will be determined based on actual source sampling parameters and analytical results as described earlier in this section.

- A normal 1-hour sampling run collects a stack gas sampling volume of about 1.25 m^3. If the sampling time is increased and 5 m^3 are collected, the in-stack method detection limits would be one fourth of the values shown in Table A-1 (this means that with this change, the method is four times more sensitive than normal).
- The in-stack detection limits assume that all of the sample is digested (with exception of the aliquot for mercury) and the final liquid volumes for analysis are 300 ml for the front half and 150 ml for the back half sample. If the front half volume is reduced from 300 ml to 30 ml, the front half in-stack detection limits would be one tenth of the values shown above (ten times more sensitive). If the back half volume is reduced from 150 ml to 25 ml, the in-stack detection limits

would be one sixth of the above values. Matrix effects checks are necessary on analyses of samples and typically are of greater significance for samples that have been concentrated to less than the normal sample volume. A volume less than 25 ml may not allow resolubilization of the residue and may increase interference by other compounds.

- When both of the above two improvements are used on one sample at the same time, the resultant improvements are multiplicative. For example, where stack gas volume is increased by a factor of five and the total liquid sample digested volume of both the front and back halves is reduced by factor of six, the in-stack method detection limit is reduced by a factor of thirty (the method is thirty times more sensitive).
- Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase limits. The front half and back half samples (Fractions 1 and 2) can be combined prior to analysis. The resultant liquid volume (excluding Fraction 3, which must be analyzed separately) is recorded. Combining the sample as described does not allow determination (whether front or back half) of where in the train the sample was captured. The in-stack method detection limit then becomes a single value for all target metals except mercury, for which the contribution of Fraction 3 must be considered.
- The above discussion assumes no blank correction. Blank corrections are discussed later in this method.

2.3 Precision. The precisions (relative standard deviations) for each of the primary and secondary metals detected in a method development test at a sewage sludge incinerator, are as follows: As (13.5%), Cd (11.5%), Cr (11.2%), Pb (11.6%), Zn (11.8%), Sb (12.7%), Ba (20.6%), Cu (11.5%), P (14.6%), Se (15.3%), and Tl (12.3%). The precision for nickel was 7.7% for another test conducted at a source simulator. Beryllium, manganese and silver were not detected in the tests; however, based on the analytical sensitivity of the ICAP for these metals, it is assumed that their precisions should be similar to those for the other metals, when detected at similar levels.

2.4 Interferences. Iron can be a spectral interference during the analysis of arsenic, chromium, and cadmium by ICAP. Aluminum can be a spectral interference during the analysis of arsenic and lead by ICAP. Generally, these interferences can be reduced by diluting the sample, but this increases the in-stack method detection limit. Refer to EPA Method 6010 (SW-846) for details on potential interferences for this method. For all GFAAS analyses, matrix modifiers should be used to limit interferences, and standards should be matrix matched.

3. Apparatus

3.1 Sampling Train. A schematic of the sampling train is shown in Figure A-1. It is similar to the Method 5 train. The sampling train consists of the following components.

3.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as Method 5, Sections 2.1.1 and 2.1.2. Glass nozzles are requiredunless an alternate probe tip prevents the possibility of contamination or interference of the sample with its materials of construction. If a probe tip other than glass is used, no correction of the stack sample test results can be made because of the effect on the results by the probe tip.

3.1.2 Pitot Tube and Differential Pressure Gauge. Same as Method 2, Sections 2.1 and 2.2, respectively.

3.1.3 Filter Holder. Glass, same as Method 5, Section 2.1.5, except that a Teflon filter support must be used to replace the glass frit.

3.1.4 Filter Heating System. Same as Method 5, Section 2.1.6.

3.1.5 Condenser. The following system shall be used for the condensation and collection of gaseous metals and for determining the moisture content of the stack gas. The condensing system should consist of four to six impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is optional and is recommended as a water knockout trap for use during test conditions which require such a trap. The impingers to be used in the metals train are now described. When the first impinger is used as a water knockout, it shall be appropriately-sized for an expected large moisture catch and constructed generally as described for the first impinger in Method 5, Paragraph 2.1.7. The second impinger (or the first HNO_3/H_2O_2 impinger) shall also be as described for the first impinger in Method 5. The third impinger (or the impinger used as the second HNO_3/H_2O_2 impinger) shall be the same as the Greenburg Smith impinger with the standard tip described as the second impinger in Method 5, Paragraph 2.1.7. All other impingers used in the metals train are the same as the second impinger (the first HNO_3/H_2O_2 impinger) previously described in this paragraph. In summary,

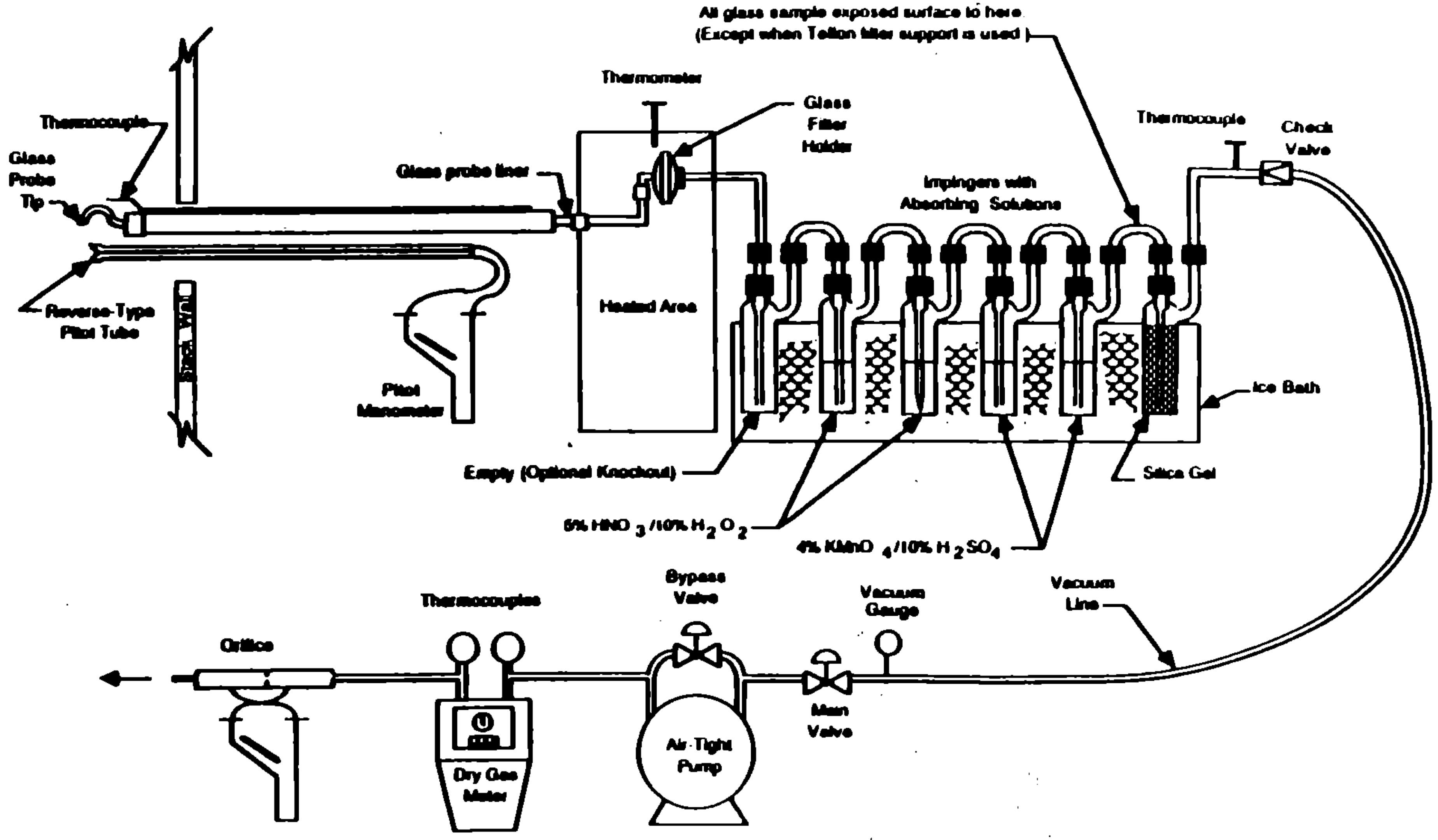

Figure A-1 Schematic of multiple metals sampling train.

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the first impinger should be empty, the second and third shall contain known quantities of a nitric acid/hydrogen peroxide solution (Section 4.2.1), the fourth (and fifth, if required) shall contain a known quantity of acidic potassium permanganate solution (Section 4.2.2), and the last impinger shall contain a known quantity of silica gel or equivalent desiccant. A thermometer capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger. When the water knockout impinger is not needed, it is removed from the train and the other impingers remain the same. If mercury analysis is not needed, the potassium permanganate impingers are removed.

3.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1 through 2.1.10, respectively.

3.1.7 Teflon Tape. For capping openings and sealing connections on the sampling train.

3.2 Sample Recovery. Same as Method 5, Sections 2.2.1 through 2.2.8 (Nonmetallic Probe-Liner and Probe-Nozzle Brushes, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

3.2.1 Nonmetallic Probe-Liner and Probe-Nozzle Brushes. For quantitative recovery of materials collected in the front half of the sampling train. Description of acceptable all-Teflon component brushes to be included in EPA's Emission Measurement Technical Information Center (EMTIC) files.

3.2.2 Sample Storage Containers. Glass bottles with Teflon-lined caps, 1000- and 500-ml, shall be used for $KMnO_4$-containing samples and blanks. Polyethylene bottles may be used for other sample types.

3.2.3 Graduated Cylinder. Glass or equivalent.

3.2.4 Funnel. Glass or equivalent.

3.2.5 Labels. For identification of samples.

3.2.6 Polypropylene Tweezers and/or Plastic Gloves. For recovery of the filter from the sampling train filter holder.

3.3 Sample Preparation and Analysis. For the analysis, the following equipment is needed:

3.3.1 Volumetric Flasks, 100 ml, 250 ml, and 1000 ml. For preparation of standards and sample dilution.

3.3.2 Graduated Cylinders. For preparation of reagents.

3.3.3 Parr® Bombs or Microwave Pressure Relief Vessels with Capping

Station (CEM Corporation model or equivalent).

3.3.4 Beakers and Watchglasses. 250-ml beakers for sample digestion with watchglasses to cover the tops.

3.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

3.3.6 Filter Funnels. For holding filter paper.

3.3.7 Whatman 541 Filter Paper (or equivalent). For filtration of digested samples.

3.3.8 Disposable Pasteur Pipets and Bulbs.

3.3.9 Volumetric Pipets.

3.3.10 Analytical Balance. Accurate to within 0.1 mg.

3.3.11 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures.

3.3.12 Hot Plates.

3.3.13 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

3.3.13.1 Graphite Furnace Attachment. With As, Cd, and Pb (and Sb, Se, and Tl, if measured) hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). Same as EPA Methods 7060 (As), 7131 (Cd), 7421 (Pb), 7041 (Sb), 7740 (Se), and 7841 (Tl).

3.3.13.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL. The equipment needed for the cold vapor mercury attachment includes an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp should be capable of raising the ambient temperature at the quartz cell by 10°C such that no condensation forms on the wall of the quartz cell. Same as EPA Method 7470.

3.3.14 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. Same as EPA Method 6010.

4. Reagents

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

4.1 Sampling. The reagents used in sampling are as follows:

4.1.1 Filters. The filters shall contain less than 1.3 ug/in.^{2} of each of the metals to be measured. Analytical results provided by filter manufacturers are acceptable. However, if no such results are available, filter blanks must be analyzed for each target metal prior to emission testing. Quartz fiber or glass fiber filters without organic binders shall be used. The filters should exhibit at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986-71 (incorporated by reference). For particulate determination in sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3, as described in EPA Method 5. Quartz fiber filters meeting these requirements are recommended.

4.1.2 Water. To conform to ASTM Specification D1193.77, Type II (incorporated by reference). Analyze the water for all target metals prior to field use. All target metals should be less than 1 ng/ml.

4.1.3 Nitric Acid. Concentrated. Baker Instra-analyzed or equivalent.

4.1.4 Hydrochloric Acid. Concentrated. Baker Instra-analyzed or equivalent.

4.1.5 Hydrogen Peroxide, 30 Percent (V/V).

4.1.6 Potassium Permanganate.

4.1.7 Sulfuric Acid. Concentrated.

4.1.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.4, respectively.

4.2 Pretest Preparation for Sampling Reagents.

4.2.1 Nitric Acid (HNO_3)/Hydrogen Peroxide (H_2O_2) Absorbing Solution, 5 Percent HNO_3/10 Percent H_2O_2. Add 50 ml of concentrated HNO_3 and 333 ml of 30 percent H_2O_2 to a 1000-ml volumetric flask or graduated cylinder containing approximately 500 ml of water. Dilute to volume with water. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.2 Acidic Potassium Permanganate ($KMnO_4$) Absorbing Solution, 4 Percent $KMnO_4$ (W/V). Prepare fresh daily. Dissolve 40 g of $KMnO_4$ in sufficient 10 percent H_2SO_4 to make 1 liter. Prepare and store in glass bottles to prevent degradation. The reagent shall contain less than 2 ng/ml of Hg.

Precaution: To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper. Also, due to reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottle; these bottles should not be fully filled and should be vented both to relieve excess pressure and prevent explosion due to pressure buildup. Venting is highly recommended, but should not allow contamination of the sample: a No. 70-72 hole drilled in the container cap and Teflon liner has been used.

4.2.3 Nitric Acid, 0.1 N. Add 6.3 ml of concentrated HNO_3 (70 percent) to a graduated cylinder containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.4 Hydrochloric Acid (HCl), 8 N. Add 690 ml of concentrated HCl to a graduated cylinder containing 250 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of Hg.

4.3 Glassware Cleaning Reagents.

4.3.1 Nitric Acid, Concentrated. Fisher ACS grade or equivalent.

4.3.2 Water. To conform to ASTM Specifications D1193-77, Type II.

4.3.3 Nitric Acid, 10 Percent (V/V). Add 500 ml of concentrated HNO_3 to a graduated cylinder containing approximately 4000 ml of water. Dilute to 5000 ml with water.

4.4 Sample Digestion and Analysis Reagents.

4.4.1 Hydrochloric Acid, Concentrated.

4.4.2 Hydrofluoric Acid, Concentrated.

4.4.3 Nitric Acid, Concentrated. Baker Instra-analyzed or equivalent.

4.4.4 Nitric Acid, 10 Percent (V/V). Add 100 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

4.4.5 Nitric Acid, 5 Percent (V/V). Add 50 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Reagent shall contain less than 2 ng/ml of each target metal.

4.4.6 Water. To conform to ASTM Specifications D1193-77, Type II.

4.4.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See EPA Method 7470 for preparation.

4.4.8 Stannous Chloride.

4.4.9 Potassium Permanganate, 5 Percent (W/V).

4.4.10 Sulfuric Acid, Concentrated.

4.4.11 Nitric Acid, 50 Percent (V/V).

4.4.12 Potassium Persulfate, 5 Percent (W/V).

4.4.13 Nickel Nitrate, $Ni(NO_3)_2 \cdot 6H_2O$.

4.4.14 Lanthanum Oxide, La_2O_3.

4.4.15 AAS Grade As Standard, 1000 ug/ml.

4.4.16 AAS Grade Be Standard, 1000 ug/ml.

4.4.17 AAS Grade Cd Standard, 1000 ug/ml.

4.4.18 AAS Grade Cr Standard, 1000 ug/ml.

4.4.19 AAS Grade Pb Standard, 1000 ug/ml.

4.4.20 AAS Grade Hg Standard, 1000 ug/ml.

4.4.21 AAS Grade Ni Standard, 1000 ug/ml.

4.4.22 AAS Grade Zn Standard, 1000 ug/ml.

4.4.23 AAS Grade Al Standard, 1000 ug/ml.

4.4.24 AAS Grade Fe Standard, 1000 ug/ml.

4.4.25 AAS Grade Sb Standard, 1000 ug/ml. Optional.

4.4.26 AAS Grade Ba Standard, 1000 ug/ml. Optional.

4.4.27 AAS Grade Cu Standard, 1000 ug/ml. Optional.

4.4.28 AAS Grade Mn Standard, 1000 ug/ml. Optional.

4.4.29 AAS Grade P Standard, 1000 ug/ml. Optional.

4.4.30 AAS Grade Se Standard, 1000 ug/ml. Optional.

4.4.31 AAS Grade Ag Standard, 1000 ug/ml. Optional.

4.4.32 AAS Grade Tl Standard, 1000 ug/ml. Optional.

4.4.33 The metals standards may also be made from solid chemicals as described in EPA Method 200.7. EPA Method 7470 or Standard Methods for the Analysis of Water and Wastewater. 15th Edition, Method 303F should be referred to for additional information on mercury standards.

4.4.34 Mercury Standards and Quality Control Samples. Prepare fresh weekly a 10 ug/ml intermediate mercury standard by adding 5 ml of 1000 ug/ml mercury stock solution to a 500-ml volumetric flask; dilute to 500 ml by first adding 20 ml of 15 percent HNO_3 and then adding water. Prepare a working mercury standard solution fresh daily: add 5 ml of the 10 ug/ml intermediate standard to a 250 ml volumetric flask and dilute to 250 ml with 5 ml of 4 percent $KMnO_4$, 5 ml of 15 percent HNO_3, and then water. At least six separate aliquots of the working mercury standard solution should be used to prepare the standard curve. These aliquots should contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution. Quality control samples should be prepared by making a separate 10 ug/ml standard and diluting until in the range of the calibration.

4.4.35 ICAP Standards and Quality Control Samples. Calibration standards for ICAP analysis can be combined into four different mixed standard solutions as shown below.

MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

Solution	Elements (secondary metals in parantheses)
I	As, Be, Cd, Pb, Zn (Mn, Se)
II	Fe (Ba, Cu)
III	Al, Cr, Ni
IV	(Sb, P, Ag, Tl)

Prepare these standards by combining and diluting the appropriate volumes of the 1000 ug/ml solutions with 5 percent nitric acid. A minimum of one standard and a blank can be used to form each calibration curve. However, a separate quality control sample spiked with known amounts of the target metals in quantities in the midrange of the calibration curve should be prepared. Suggested standard levels are 50 ug/ml for Al, 25 ug/ml for Cr and Pb, 15 ug/ml for Fe, and 10 ug/ml for the remaining elements. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.36 Graphite Furnace AAS Standards for Arsenic, Cadmium, and Lead (and Antimony, Selenium, and Thallium). Prepare a 10 ug/ml standard by adding 1 ml of 1000 ug/ml standard to a 100-ml volumetric flask. Dilute to 100 ml with 10 percent nitric acid. For graphite furnace AAS, the standards must be matrix matched; e.g., if the samples contain 6 percent nitric acid and 4 percent hydrofluoric acid, the standards should also be made up with 6 percent nitric acid and 4 percent hydrofluoric acid. Prepare a 100 ng/ml standard by adding 1 ml of the 10 ug/ml standard to a 100-ml volumetric flask and dilute to 100 ml with the appropriate matrix solution. Other standards should be prepared by dilution of the 100 ng/ml standards. At least five standards should be used to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Quality control samples should be prepared by making a separate 10 ug/ml standard and diluting until it is in the range of the samples. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.37 Matrix Modifiers.

4.4.37.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of $Ni(NO_3)_2 \cdot 6H_2O$ in approximately 50 ml of water in a 100-ml volumetric flask.

Dilute to 100 ml with water.

4.4.37.2 Nickel Nitrate, One-tenth Percent (V/V). Dilute 10 ml of 1 percent nickel nitrate solution to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for As.

4.4.37.3 Lanthanum. Dissolve 0.5864 g of La_2O_3 in 10 ml of concentrated HNO_3 and dilute to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for Pb.

5. Procedure

5.1 Sampling. The complexity of this method is such that, to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. All sampling train glassware should first be rinsed with hot tap water and then washed in hot soapy water. Next, glassware should be rinsed three times with tap water, followed by three additional rinses with water. All glassware should then be soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, rinsed a final time with acetone, and allowed to air dry. All glassware openings where contamination can occur should be covered until the sampling train is assembled, prior to sampling.

5.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2.

5.1.3 Preparation of Sampling Train. Follow the same general procedures given in Method 5, Section 4.1.3, except place 100 ml of the nitric acid/hydrogen peroxide solution (Section 4.2.1) in the two HNO_3/H_2O_2 impingers (normally the second and third impingers), place 100 ml of the acidic potassium permanganate solution (Section 4.2.2) in the fourth and fifth impinger, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to train assembly.

Several options are available to the tester based on the sampling conditions. The use of an empty first impinger can be eliminated if the moisture to be collected in the impingers is calculated or determined to be less than 150 ml. The tester shall include two impingers containing the acidic potassium permanganate solution for the first test run, unless past testing experience at the same or similar sources has shown that only one is necessary. The last permanganate impinger may be discarded if both permanganate impingers have retained their original deep purple permanganate color. A maximum of 200 ml in each permanganate impinger (and a maximum of three permanganate impingers) may be used, if necessary, to maintain the desired color in the last permanganate impinger.

Retain for reagent blanks, 100 ml of the nitric acid/hydrogen peroxide solution and 100 ml of the acidic potassium permanganate solution. These solutions should be labeled and treated as described in Section 7. Set up the sampling train as shown in Figure A-1. If necessary to ensure leak-free sampling train connections, Teflon tape should be used instead of silicone grease to prevent contamination.

Precaution: Extreme care should be taken to prevent contamination within the train. Prevent the mercury collection reagent (acidic potassium permanganate) from contacting any glassware of the train which is washed and analyzed for Mn. Prevent hydrogen peroxide from mixing with the acidic potassium permanganate.

5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

5.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

5.2 Sample Recovery. Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period.

The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling. This normally causes a vacuum to form in the filter holder, thus causing the undesired result of drawing liquid from the impingers into the filter.

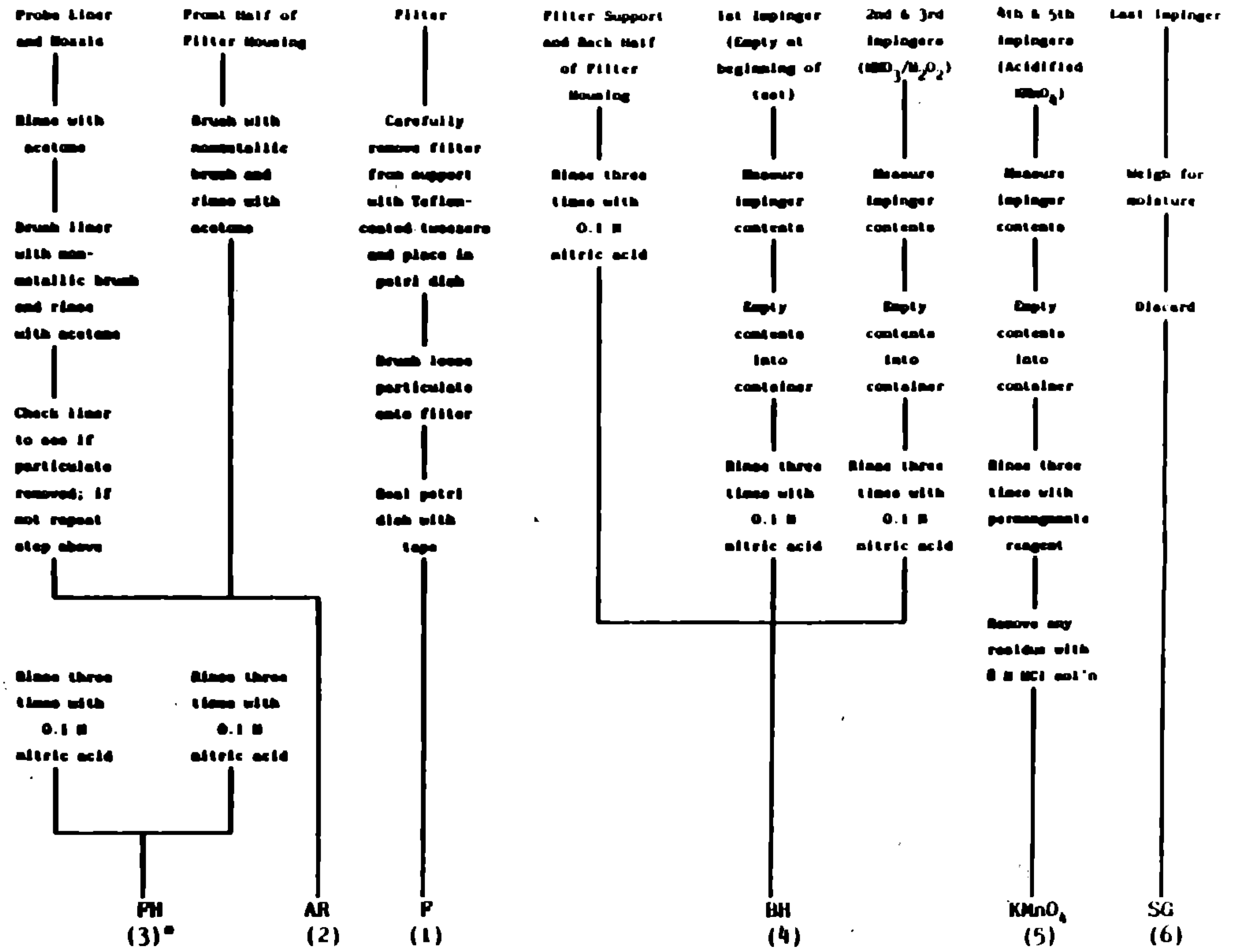
Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the impinger. Cap off the filter holder outlet and impinger inlet. Use noncontaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon tape to close these openings.

Alternatively, the train can be disassembled before the probe and filter holder/oven are completely cooled, if this procedure is followed: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. The sample is recovered and treated as follows (see schematic in Figure A-2). Assure that all items necessary for recovery of the sample do not contaminate it.

5.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. Acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water should be used to handle the filters. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metal-containing materials when recovering this train. Seal the labeled petri dish.

5.2.2 Container No. 2 (Acetone Rinse). Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 100 ml of acetone and placing the wash in a glass container. <u>Note</u>: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nonmetallic brush. Brush until



* Number in parantheses indicates container number.

Figure A-2. Sample recovery scheme.

the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a nonmetallic probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a samplecontainer underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the front half of the filter holder by rubbing surfaces with a nonmetallic nylon bristle brush and rinsing with aceton Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container clearly to identify its contents.

5.2.3 Container No. 3 (Probe Rinse). Rinse the probe liner, probe nozzle, and front half of the filter holder thoroughly with 100 ml of 0.1 N nitric acid and place the wash into a sample storage container. <u>Note</u>: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Perform the rinses as described in Method 12, Section 5.2.2. Record the volume of the combined rinse. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during transport. Seal the container and clearly label the contents. Finally, rinse the nozzle, probe liner, and front half of the filter holder with water followed by acetone and discard these rinses.

5.2.4 Container No. 4 (Impingers 1 through 3, Contents and Rinses). Due to the large quantity of liquid involved, the tester may place the impinger solutions in more than one container. Measure the liquid in the first three impingers volumetrically to within 0.5 ml using a graduated cylinder. Record the volume of liquid present. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N nitric acid as described in Method 12, Section 5.2.4. Note: The use of exactly 100 ml of 0.1 N nitric acid rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the volume. Calculate the 0.1 N nitric acid rinse volume by difference. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. Seal the container and clearly label the contents.

5.2.5 Container No. 5 (Acidified Potassium Permanganate Solution and Rinses, Impingers No. 4 & 5). Pour all the liquid from the permanganate impingers (fourth and fifth, if two permanganate impingers are used) into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Using 100 ml total of the acidified potassium permanganate solution, rinse the permanganate impinger(s) and connecting glass pieces a minimum of three times. Combine the rinses with the permanganate impinger solution. Finally, rinse the permanganate impinger(s) and connecting glassware with 50 ml of 8 N HCl to remove any residue. Note: The use of exactly 100 ml and 50 ml for the two rinses is necessary for the subsequent blank correction procedures. Place the combined rinses and impinger contents in a labeled glass storage bottle. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport. See the following note and the Precaution in Paragraph 4.2.2 and properly seal the bottle and clearly label the contents.

Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles should not be filled full and should be vented to relieve excess pressure. Venting is highly recommended. A No. 70-72 hole drilled in the container cap and Teflon liner has been found to allow adequate venting without loss of sample.

5.2.6 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

5.2.7 Container No. 7 (Acetone Blank). Once during each field test, place 100 ml of the acetone used in the sample recovery process into a labeled container for use in the front half field reagent blank. Seal the container.

5.2.8 Container No. 8 (0.1 N Nitric Acid Blank). Once during each field test, place 200 ml of the 0.1 N nitric acid solution used in the sample recovery process into a labeled container for use in the front half and back half field reagent blanks. Seal the container.

5.2.9 Container No. 9 (5% Nitric Acid/10% Hydrogen Peroxide Blank). Once during each field test, place 200 ml of the 5% nitric acid/10% hydrogen peroxide solution used as the nitric acid impinger reagent into a labeled container for use in the back half field reagent blank. Seal the container.

5.2.10 Container No. 10 (Acidified Potassium Permanganate Blank). Once during each field test, place 300 ml of the acidified potassium permanganate solution used as the impinger solution and in the sample recovery process into a labeled container for use in the back half field reagent blank for mercury analysis. Seal the container.

Note: This container should be vented, as described in Section 5.2.4, to relieve excess pressure.

5.2.11 Container No. 11 (8 N HCl Blank). Once during each field test, place 50 ml of the 8 N hydrochloric acid used to rinse the acidified potassium permanganate impingers into a labeled container for use in the back half reagent blank for mercury.

5.2.12 Container No. 12 (Filter Blank). Once during each field test, place an unused filter from the same lot as the sampling filters in a labeled petri dish. Seal the petri dish. This will be used in the front half field reagent blank.

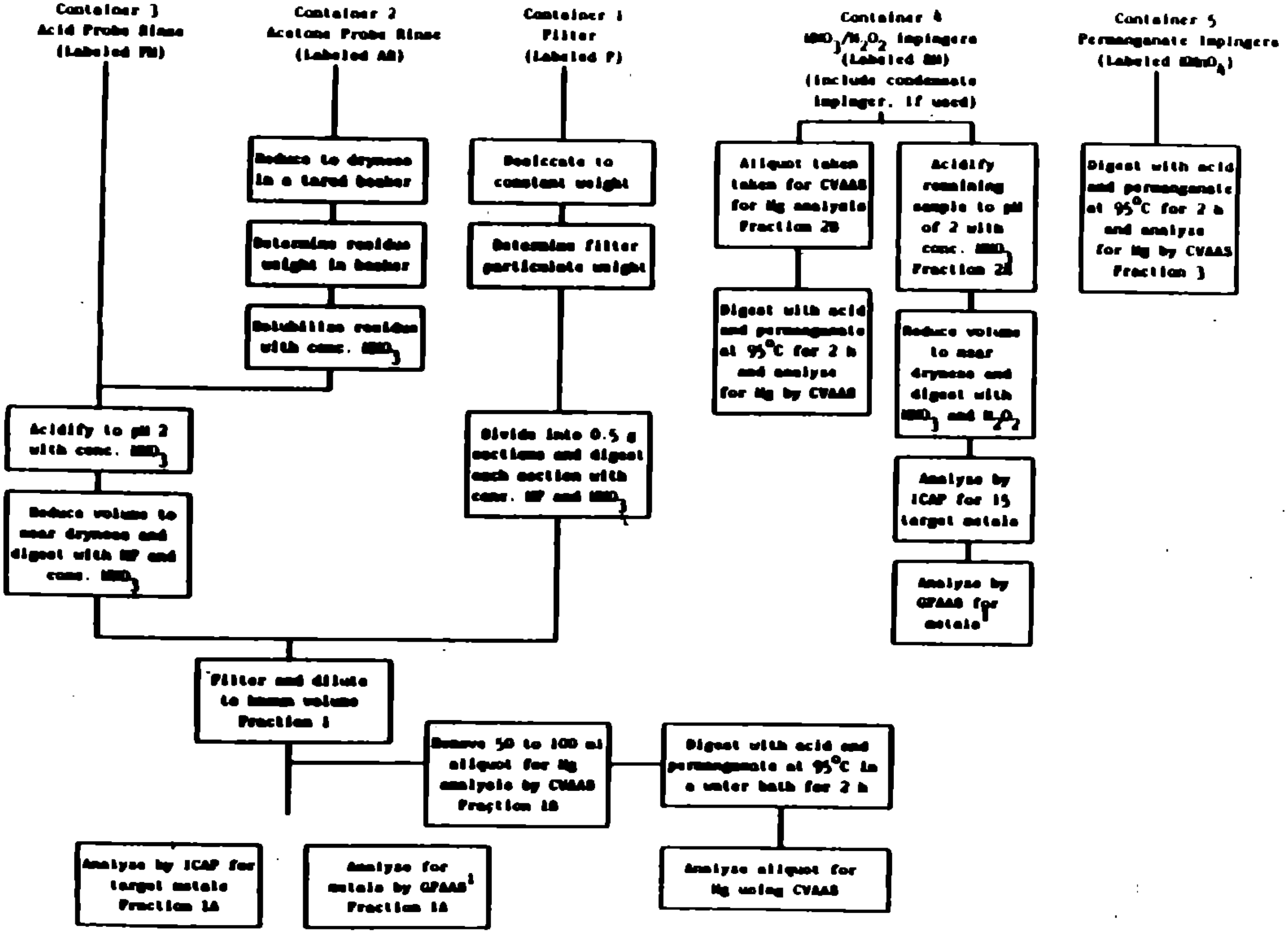
5.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure A-3.

5.3.1 Container No. 1 (Filter). If particulate emissions are being determined, then desiccate the filter and filter catch without heat and weigh to a constant weight as described in Section 4.3 of Method 5. For analysis of metals, divide the filter with its filter catch into portions containing approximately 0.5 g each and place into the analyst's choice of either individual microwave pressure relief vessels or Parr® Bombs. Add 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid to each vessel. For microwave heating, microwave the sample vessels for approximately 12-15 minutes in intervals of 1 to 2 minutes at 600 Watts. For conventional heating, heat the Parr Bombs at 140°C (285°F) for 6 hours. Then cool the samples to room temperature and combine with the acid digested probe rinse as required in Section 5.3.3, below.

Notes: 1. Suggested microwave heating times are approximate and are dependent upon the number of samples being digested. Twelve to 15 minute heating times have been found to be acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by sorbent reflux within the vessel.

2. If the sampling train uses an optional cyclone, the cyclone catch should be prepared and digested using the same procedures described for the filters and combined with the digested filter samples.

5.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to an acid-cleaned tared 250-ml beaker and evaporate to dryness at ambient Figure



[1] Analysis by AAS for metals found at less than 2 ug/ml in digestate solution, if desired. Or analyse for each metal by AAS, if desired

Figure A-3. Sample preparation and analysis scheme.

temperature and pressure. (If particulate emissions are being determined, desiccate for 24 hours without heat, weigh to a constant weight according to the procedures described in Section 4.3 of Method 5, and report the results to the nearest 0.1 mg.) Resolubilize the residue with concentrated nitric acid and combine the resultant sample including all liquid and any particulate matter with Container No. 3 prior to beginning the following Section 5.3.3.

5.3.3 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. If the pH is higher, the sample should be acidified with concentrated nitric acid to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 50 ml by heating on a hot plate at a temperature just below boiling. Inspect the sample for visible particulate matter, and depending on the results of the inspection, perform one of the following. If no particulate matter is observed, combine the sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. If particulate matter is observed, digest the sample in microwave vessels or Parr® Bombs following the procedures described in Section 5.3.1; then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. The resultant combined sample is referred to as Fraction 1. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. Measure and record the combined volume of the Fraction 1 solution to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as Fraction 1B. Label the remaining 250-ml portion as Fraction 1A. Fraction 1A is used for ICAP or AAS analysis. Fraction 1B is used for the determination of front half mercury.

5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample (Fraction 2) to within 0.5 ml. Remove a 50-ml aliquot for mercury analysis and label as Fraction 2B. Label the remaining portion of Container No. 4 as Fraction 2A. The Fraction 2B aliquot should be prepared and analyzed as described in Section 5.4.3. Fraction 2A shall be pH 2 or lower. If necessary, use concentrated nitric acid to lower Fraction 2A to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Then follow either of the digestion procedures described in Sections

5.3.4.1 and 5.3.4.2, below.

5.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent nitric acid and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent hydrogen peroxide and heat for 10 more minutes. Add 50 ml of hot water and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water.

5.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent nitric acid and heat for 6 minutes in intervals of 1 to 2 minutes at 600 Watts. Allow the sample to cool. Add 10 ml of 3 percent hydrogen peroxide and heat for 2 more minutes. Add 50 ml of hot water and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water.

Note: All microwave heating times given are approximate and are dependent upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.

5.3.5 Container No. 5 (Impingers 4 & 5). Measure and record the total volume of this sample to within 0.5 ml. This sample is referred to as Fraction 3. Follow the analysis procedures described in Section 5.4.3.

5.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

5.4 Sample Analysis. For each sampling train, five individual samples are generated for analysis. A schematic identifying each sample and the prescribed sample preparation and analysis scheme is shown in Figure A-3. The first two samples, labeled Fractions 1A and 1B, consist of the digested samples from the front half of the train. Fraction 1A is for ICAP or AAS analysis as described in Sections 5.4.1 and/or 5.4.2. Fraction 1B is for determination of front half mercury as described in Section 5.4.3.

The back half of the train was used to prepare the third through fifth samples. The third and fourth samples, labeled Fractions 2A and 2B, contain the digested samples from the H_2O and HNO_3/H_2O_2 Impingers 1 through 3. Fraction 2A is for ICAP or AAS analysis. Fraction 2B will be analyzed for mercury.

The fifth sample, labeled Fraction 3, consists of the impinger contents and rinses from the permanganate Impingers 4 and 5. This sample is analyzed for mercury as described in Section 5.4.3. The total back half mercury catch is determined from the sum of Fraction 2B and Fraction 3.

5.4.1 ICAP Analysis. Fraction 1A and Fraction 2A are analyzed by ICAP using EPA Method 200.7 (40 CFR 136, Appendix C). Calibrate the ICAP, and set up an analysis program as described in Method 200.7. The quality control procedures described in Section 7.3.1 of this method shall be followed. Recommended wavelengths for use in the analysis of the primary, secondary, and interferring metals are listed below.

Element	Wavelength (nm)
Arsenic	193.696
Beryllium	313.042
Cadmium	226.502
Chromium	267.716
Lead	220.353
Nickel	231.604
Zinc	213.856
Antimony	206.833
Barium	455.403
Copper	324.754
Manganese	257.610
Selenium	196.026
Silver	328.068
Thallium	190.864
Aluminum	308.215
Iron	259.940

The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

Initially, analyze all samples for the target metals plus iron and aluminum. If iron and aluminum are present in the sample, the sample may have to be diluted so that each of these elements is at a concentration of less than 50 ppm to reduce their spectral interferences on arsenic and lead.

<u>Note</u>: When analyzing samples in a hydrofluoric acid matrix, an alumina torch should be used; since all front half samples will contain hydrofluoric acid, use an alumina torch.

5.4.2 AAS by Direct Aspiration and/or Graphite Furnace. If analysis of metals in Fraction 1A and Fraction 2A using graphite furnace or direct aspiration AAS is desired, Table A-2 should be used to determine which techniques and methods should be applied for each target metal. Table A-2 should also be consulted to determine possible interferences and techniques to be followed for their minimization. Calibrate the instrument according to Section 6.3 and follow the quality control procedures specified in Section 7.3.2.

5.4.3 Cold Vapor AAS Mercury Analysis. Fraction 1B, Fraction 2B, and Fraction 3 should be analyzed for mercury using cold vapor atomic absorption spectroscopy following the method outlined in EPA Method 7470 or in Standard Methods for Water and Wastewater Analysis, 15th Edition, Method 303F. Set up the calibration curve as described in Section 7.3 of Method 303F. Add approximately 5 ml of each sample to BOD bottles. Record the amount of sample added. The amount used is dependent upon the expected levels of mercury. Dilute to approximately 120 ml with mercury-free water. Add approximately 15 ml of 5 percent potassium permanganate solution to the Fraction 2B and Fraction 3 samples. Add 5 percent potassium permanganate solution to the Fraction 1B sample as needed to produce a purple solution lasting at least 15 minutes. A minimum of 25 ml is suggested. Add 5 ml of 50 percent nitric acid, 5 ml of concentrated sulfuric acid, and 9 ml of 5 percent potassium persulfate to each sample and each standard. Digest the solution in the capped BOD bottle at 95°C (205°F) in a convection oven or water bath for 2 hours. Cool. Add 5 ml of hydroxylamine hydrochloride solution and mix the sample. Then add 7 ml of stannous chloride to each sample and analyze immediately.

6. Calibration

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in Section 4.4. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures using the

TABLE A-2. APPLICABLE TECHNIQUES, METHODS, AND MINIMIZATION OF INTEFERENCE FOR AAS ANALYSIS

Metal	Technique	Method No.	Wavelength (nm)	Interference	
				Cause	Minimization
Sb	Aspiration	7040	217.6	1000 mg/ml Pb Ni, Cu, or acid	Use secondary wavelenght of 231.1 nm. Match sample & standards acid concentration or use nitrous oxide/acetylene flame
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction
As	Furnace	7060	193.7	Arsenic volati - zation Aluminium	Spiked samples & add nickel nitrate solution to digestates prior to analyses Use Zeeman background correction
Ba	Aspiration	7080	553.6	Calcium Barium ionization	High hollow cathode current & narrow band set 2 mL of KCl per 100 mL of sample
Be	Aspiration	7090	234.9	500 ppm Al High Mg & Si	Add 0.1% flouride Use method of standard additions
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects
Cd	Aspiration	7130	228.8	Absorption & light scattering	Background correction is required
Cd	Furnace	7131	228.8	As above Excess chloride Pipet tips	As above Ammonium phosphate used as a matrix modifier Use cadmium-free tips
Cr	Aspiration	7190	357.9	Alkali metal Absorption & scatt	KCl ionization suppressant in sample & stand Consult manufacturer's literature
Cr	Furnace	7191	357.9	200 mg/L calcium & phosphate	All calcium nitrate for a know constant effect and to eliminate effect of phosphate

(continued)

TABLE A-2 (CONTINUED)

Metal	Technique	Method No.	Wavelength (nm)	Interference Cause	 Minimization
Cu	Aspiration	7210	324.7	Absorpt & scatter	Consult manufacturer's manual
Fe	Aspiration	7380	248.3	Contamination	Great care taken to aviod contamination
Pb	Aspiration	7420	283.3	217.0 nm alternat	Background correction required
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 uL of phosphorus acid to 1-mL of prepared sample in sampler cup
Mn	Aspiration	7460	279.5	403.1 nm alternat	Background correction required
Ni	Aspiration	7520	232.0	352.4 nm alternat	Background correction required
				Fe, Co, & Cr	Matrix matching or a nitrous-oxide/acety flame
				Nonlinear respons	Sample dilution or use 352.4 nm line
Se	Furnace	7740	196.0	Volitality	Spike samples & reference materials & add nickel nitrate to minimize volatilization
				Adsorpt & scatter	Background correction is required & Zeeman background correction can be useful
Ag	Aspiration	7760	328.1	Absorpt & scatter	Background correction is required
				AgCl insoluble	Avoid hydrochloric acid unless silver is in solution as a chloride complex
				Viscosity	Sample & standards monitored for apiration rate
Tl	Aspiration	7840	276.8		Background correction is required Hydrochloric acid should not be used
Tl	Furnace	7841	276.8		Background correction is required
				Hydrochloric acid or chloride	Verify that losses are not occuring for volitization by spiked samples or standad addt Palladium is a suitable matrix modifier
Zn	Aspiration	7950	213.9	High Si, Cu & P	Strontium removes Cu and phosphate
				Contamination	Care should be taken to avid contamination

above standards. The instrument calibration should be checked once per hour. If the instrument does not reproduce the concentrations of the standard within 10 percent, the complete calibration procedures should be performed.

6.3 Atomic Absorption Spectrometer - Direct Aspiration, Graphite Furnace and Cold Vapor Mercury Analyses. Prepare the standards as outlined in Section 4.4. Calibrate the spectrometer using these prepared standards. Calibration procedures are also outlined in the EPA methods referred to in Table A-2 and in Standard Methods for Water and Wastewater, 15th Edition, Method 303F (for mercury). Each standard curve should be run in duplicate and the mean values used to calculate the calibration line. The instrument should be recalibrated approximately once every 10 to 12 samples.

7. Quality Control

7.1 Sampling. Field Reagent Blanks. The blank samples in Container Numbers 7 through 12 produced previously in Sections 5.2.7 through 5.2.11, respectively, shall be processed, digested, and analyzed as follows. Digest and process Container No. 12 contents per Section 5.3.1, Container No. 7 per Section 5.3.2, and half of Container No. 8 per Section 5.3.3. This produces Fraction Blank 1A and Fraction Blank 1B from Fraction Blank 1. Combine the remaining half of Container No. 8 with the contents of Container No. 9 and digest and process the resultant volume per Section 5.3.4. This produces Fraction Blank 2A and Fraction Blank 2B from Fraction Blank 2. Container No. 10 and Container No. 11 contents are Fraction Blank 3. Analyze Fraction Blank 1A and Fraction Blank 2A per Section 5.4.1 and/or 5.4.2. Analyze Fraction Blank 1B, Fraction Blank 2B, and Fraction Blank 3 per Section 5.4.3. The analysis of Fraction Blank 1A produces the front half reagent blank correction values for the metals except mercury; the analysis of Fraction Blank 1B produces the front half reagent blank correct value for mercury. The analysis of Fraction Blank 2A produces the back half reagent blank correction values for the metals except mercury, while separate analysis of Fraction Blanks 2B and 3 produce the back half reagent blank correction value for mercury.

7.2 An attempt may be made to determine if the laboratory reagents used in Section 5.3 caused contamination. They should be analyzed by the procedures in Section 5.4. The Administrator will determine whether or not the laboratory blank reagent values can be used in the calculation of the stationary source test results.

7.3 Quality Control Samples. The following quality control samples should be analyzed.

7.3.1 ICAP Analysis. Follow the quality control shown in Section 8 of Method 6010. For the purposes of a three run test series, these requirements have been modified to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (must be within 25% or analyze by standard addition), one quality control sample to check the accuracy of the calibration standards (must be within 25% of calibration), and one duplicate analysis (must be within 5% of average or repeat all analysis).

7.3.2 Direct Aspiration and/or Graphite Furnace AAS Analysis for Arsenic, Beryllium, Cadmium, Chromium, Lead, Mercury, Nickel, and Zinc (and Antimony, Barium, Copper, Manganese, Phosphorus, Selenium, Silver, and Thallium, if measured). All samples should be analyzed in duplicate. Perform a matrix spike on one front half sample and one back half sample or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the method of additions. A quality control sample should be analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration repeated.

7.3.3 Cold Vapor AAS Analysis for Mercury. All samples should be analyzed in duplicate. A quality control sample should be analyzed to check the accuracy of the calibration standards (within 10% or repeat calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). Additional information on quality control can be obtained from EPA Method 7470 or in <u>Standard Methods for Water and Wastewater</u>, 15th Edition, Method 303F.

8. Calculations

8.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

8.2 Volume of Water Vapor and Moisture Content. Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

8.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

8.4 Metals (Except Mercury) in Source Sample.

8.4.1 Fraction 1A, Front Half, Metals (except Hg). Calculate the amount of each metal collected in Fraction 1 of the sampling train using the following equation:

$$M_{fh} = C_a \; F_d \; V_{soln,1} \qquad \text{Eq. 1*}$$

where:

M_{fh} = total mass of each metal (except Hg) collected in the front half of the sampling train (Fraction 1), ug.
C_a = concentration of metal in sample Fraction 1A as read from the standard curve (ug/ml).
F_d = dilution factor (F_d = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument to produce the reading C_a. For example, when the dilution of Fraction 1A is from 2 to 10 ml, F_d = 5).
$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.

8.4.2 Fraction 2A, Back Half, Metals (except Hg). Calculate the amount of each metal collected in Fraction 2 of the sampling train using the following equation.

$$M_{bh} = C_a \; F_a \; V_a \qquad \text{Eq. 2*}$$

where:

M_{bh} = total mass of each metal (except Hg) collected in the back half of the sampling train (Fraction 2), ug.
C_a = concentration of metal in sample Fraction 2A, as read from the standard curve (ug/ml).
F_a = aliquot factor, volume of Fraction 2 divided by volume of aliquot Fraction 2A.
V_a = volume of digested sample analyzed (concentrated Fraction 2A), ml.

8.4.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{fh} - M_{fhb}) + (M_{bh} - M_{bhb}) \qquad \text{Eq. 3*}$$

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*If Fractions 1A and 2A are combined, proportional aliquots must be used. Appropriate changes must be made in Equations 1-3 to reflect this approach.

where:

M_t = total mass of each metal (separately stated for each metal) collected in the sampling train, ug.
M_{fhb} = blank correction value for mass of metal detected in front half field reagent blank, ug.
M_{bhb} = blank correction value for mass of metal detected in back half field reagent blank, ug.

Note: If the measured blank value for the front half (m_{fhb}) is in the range 0.0 to A ug [where A ug equals the value determined by multiplying 1.4 ug per square inch (1.4 ug/in.^{2}) times the actual area in square inches (in.^{2}) of the filter used in the emission sample], m_{fhb} may be used to correct the emission sample value (m_{fh}): if m_{fhb} exceeds A ug, the greater of the two following values (either I. or II.) may be used:

I. A ug, or

II. the lesser of (a) m_{fhb}, or (b) 5 percent of m_{fh}.

If the measured blank value for the back half (m_{bhb}) is in the range 0.0 to 1 ug, m_{bhb} may be used to correct the emission sample value (m_{bh}): if m_{bhb} exceeds 1 ug, the greater of the two following values may be used: 1 ug or 5 percent of m_{bh}.

8.5 Mercury in Source Sample.

8.5.1 Fraction 1B, Front Half, Hg. Calculate the amount of mercury collected in the front half, Fraction 1, of the sampling train using the following equation:

$$Hg_{fh} = \frac{Q_{fh}}{V_{f1B}} \times V_{soln,1} \qquad \text{Eq. 4}$$

where:

Hg_{fh} = total mass of mercury collected in the front half of the sampling train (Fraction 1), ug.
Q_{fh} = quantity of mercury in analyzed sample, ug.
$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.
V_{f1B} = volume of Fraction 1B analyzed, ml. See the following Note.

Note: V_{f1B} is the actual amount of Fraction 1B analyzed. For example, if 1 ml of Fraction 1B were diluted to 100 ml to bring it into the proper analytical range, and 1 ml of the 100-ml dilution was analyzed, V_{f1B} would be 0.01.

8.5.2 Fraction 2B and Fraction 3, Back Half, Hg. Calculate the amount of mercury collected in Fractions 2 and 3 using Equations 5 and 6, respectively.

Calculate the total amount of mercury collected in the back half of the sampling train using Equation 7.

$$Hg_{bh2} = \frac{Q_{bh2}}{V_{f1B}} \times V_{soln,2} \qquad \text{Eq. 5}$$

where:

Hg_{bh2} = total mass of mercury collected in Fraction 2, ug.
Q_{bh2} = quantity of mercury in analyzed sample, ug.
V_{f2B} = volume of Fraction 2B analyzed, ml (see Note in Section 8.5.1).
$V_{soln,2}$ = total volume of Fraction 2, ml.

$$Hg_{bh3} = \frac{Q_{bh3}}{V_{f2B}} \times V_{soln,3} \qquad \text{Eq. 6}$$

where:

Hg_{bh3} = total mass of mercury collected in Fraction 3, ug.
Q_{bh3} = quantity of mercury in analyzed sample, ug.
V_{f3} = volume of Fraction 3 analyzed, ml (see Note in Section 8.5.1).
$V_{soln,3}$ = total volume of Fraction 3, ml.

$$Hg_{bh} = Hg_{bh2} + Hg_{bh3} \qquad \text{Eq. 7}$$

where:

Hg_{bh} = total mass of mercury collected in the back half of the sampling train, ug.

8.5.3 **Total Train Mercury Catch.** Calculate the total amount of mercury collected in the sampling train using Equation 8.

$$M_t = (Hg_{fh} - Hg_{fhb}) + (Hg_{bh} - Hg_{bhb}) \qquad \text{Eq. 8}$$

where:

M_t = total mass of mercury collected in the sampling train, ug.
Hg_{fhb} = blank correction value for mass of mercury detected in front half field reagent blank, ug.

Hg_{bhb} = blank correction value for mass of mercury detected in back half field reagent blank, ug.

Note: If the total of the measured blank values ($Hg_{fhb} + Hg_{bhb}$) is in the range of 0 to 3 ug, then the total may be used to correct the emission sample value ($Hg_{fh} + Hg_{bh}$); if it exceeds 3 ug, the greater of the following two values may be used: 3 ug or 5 percent of the emission sample value ($Hg_{fh} + Hg_{bh}$).

8.6 Metal Concentration of Stack Gas. Calculate the concentrations of arsenic, beryllium, cadmium, total chromium, lead, mercury, nickel, and zinc (and antimony, barium, copper, manganese, phosphorus, selenium, silver, and thallium, if measured) in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_s = K_4 \ (M_t/V_{m(std)}) \qquad \text{Eq. 9}$$

where:

C_s = concentration of each metal in the stack gas, mg/dscm.
K_4 = 10^{-3} mg/ug.
M_t = total mass of each metal collected in the sampling train, ug.
$V_{m(std)}$ = volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

8.7 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

9. Bibliography

9.1 Method 303F in Standard Methods for the Examination of Water Wastewater, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street N.W., Washington, D.C. 20036.

9.2 EPA Methods 6010, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U. S. Environmental Protection Agency, Washington, D.C. 20460.

9.3 EPA Method 200.7, Code of Federal Regulations, Title 40, Part 136, Appendix C. July 1, 1987.

9.4 EPA Methods 1 through 5, Code of Federal Regulations, Title 40, Part 60, Appendix A, July 1, 1987.

APPENDIX J.2

PM$_{10}$/CPM

Sampling Rate, for directions in the use of this equation for Q in the setup calculations.

5.4 Cascade Impactor. The purpose of calibrating a cascade impactor is to determine the empirical constant (STK_{50}), which is specific to the impactor and which permits the accurate determination of the cut size of the impactor stages at field conditions. It is not necessary to calibrate each individual *impactor*. Once an impactor has been calibrated, the calibration data can be applied to other impactors of identical design.

5.4.1 Wind Tunnel. Same as in Section 5.2.1 of this method.

5.4.2 Particle Generation System. Same as in Section 5.2.2 of this method.

5.4.3 Hardware Configuration for Calibrations. An impaction stage constrains an aerosol to form circular or rectangular jets, which are directed toward a suitable substrate where the larger aerosol particles are collected. For calibration purposes, three stages of the cascade impactor shall be discussed and designated calibration stages 1, 2, and 3. The first calibration stage consists of the collection substrate of an impaction stage and all upstream surfaces up to and including the nozzle. This may include other preceding impactor stages. The second and third calibration stages consist of each respective collection substrate and all upstream surfaces up to but excluding the collection substrate of the preceding calibration stage. This may include intervening impactor stages which are not designated as calibration stages. The cut size, or D_{50}, of the adjacent calibration stages shall differ by a factor of not less than 1.5 and not more than 2.0. For example, if the first calibration stage has a D_{50} of 12 μm, then the D_{50} of the downstream stage shall be between 6 and 8 μm.

5.4.3.1 It is expected, but not necessary, that the complete hardware assembly will be used in each of the sampling runs of the calibration and performance determinations. Only the first calibration stage must be tested under isokinetic sampling conditions. The second and third calibration stages must be calibrated with the collection substrate of the preceding calibration *stage in place, so that* gas flow patterns existing in field operation will be simulated.

5.4.3.2 Each of the PM_{10} stages should be calibrated with the type of collection substrate, *viscid material (such as grease) or* glass fiber, used in PM_{10} measurements. Note that most materials used as substrates at elevated temperatures are not viscid at normal laboratory conditions. The substrate *material used for calibrations* should minimize particle bounce, yet be viscous enough to withstand erosion or deformation by the impactor jets and not interfere with the procedure for measuring the collected PM.

5.4.4 Calibration Procedure. Establish test particle generator operation and verify particle size microscopically. If monodispersity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements shall be made at this time. Measure in triplicate the PM collected by the calibration stage (m) and the PM on all surfaces downstream of the respective calibration stage (m') for all of the flow rates and particle size combinations shown in Table 2 of this method. Techniques of mass measurement may include the use of a dye and spectrophotometer. Particles on the upstream side of a jet plate shall be included with the substrate downstream, except agglomerates of particles, which shall be included with the preceding or upstream substrate. Use the following formula to calculate the collection efficiency (E) for each stage.

5.4.4.1 Use the formula in Section 5.2.5.3 of this method to calculate the standard deviation (σ) for the replicate measurements. If σ exceeds 0.10, repeat the replicate runs.

5.4.4.2 Use the following formula to calculate the average collection efficiency (E_{avg}) for each set of replicate measurements.

$$E_{avg} = (E_1 + E_2 + E_3)/3$$

where E_1, E_2, and E_3 are replicate measurements of E.

5.4.4.3 Use the following formula to calculate Stk for each E_{avg}.

$$Stk = \frac{D^2 Q}{9 \mu A d_j}$$

where:

D = Aerodynamic diameter of the test particle, cm $(g/cm^3)^{1/2}$.

Q = Gas flow rate through the calibration stage at inlet conditions, cm^3/sec.

μ = Gas viscosity, micropoise.

A = Total cross-sectional area of the jets of the calibration stage, cm^2.

d_j = Diameter of one jet of the calibration stage, cm.

5.4.4.4 Determine Stk_{50} for each calibration stage by plotting E_{avg} versus Stk on log-log paper. Stk_{50} is the Stk number at 50 percent efficiency. Note that particle bounce can cause efficiency to decrease at high values of Stk. Thus, 50 percent efficiency can occur at multiple values of Stk. The calibration data should clearly indicate the value of Stk_{50} for minimum particle bounce. Impactor efficiency versus Stk with minimal particle bounce is characterized by a monotonically increasing function with constant or increasing slope with increasing Stk.

5.4.4.5 The Stk_{50} of the first calibration stage can potentially decrease with decreasing nozzle size. Therefore, calibrations should be performed with enough nozzle sizes to provide a measured value within 25 percent of any nozzle size *used in PM_{10} measurements.*

5.4.5 Criteria For Acceptance. Plot E_{avg} for the first calibration stage versus the square root of the ratio of Stk to Stk_{50} on Figure 9 of this method. Draw a smooth curve through all of the points. The curve shall be within the banded region.

6. Calculations

6.1 Nomenclature.

B_{ws} = Moisture fraction of stack, by volume, dimensionless.

C_1 = Viscosity constant, 51.12 micropoise for °K (51.05 micropoise for °R).

C_2 = Viscosity constant, 0.372 micropoise/°K (0.207 micropoise/°R).

C_3 = Viscosity constant, 1.05×10^{-4} micropoise/°K^2 (3.24×10^{-5} micropoise/°R^2).

C_4 = Viscosity constant, 53.147 micropoise/fraction O_2.

C_5 = Viscosity constant, 74.143 micropoise/fraction H_2O.

D_{50} = Diameter of particles having a 50 percent probability of penetration, μm.

f_o = Stack gas fraction O_2, by volume, dry basis.

K_1 = 0.3858 °K/mm Hg (17.64 °R/in. Hg).

M_w = Wet molecular weight of mixed gas through the PM_{10} cyclone, g/g-mole (lb/lb-mole).

M_d = Dry molecular weight of stack gas, g/g-mole (lb/lb-mole).

P_{bar} = Barometric pressure at sampling site, mm Hg (in. Hg).

P_s = Absolute stack pressure, mm Hg (in. Hg).

Q_s = Total cyclone flow rate at wet cyclone conditions, m^3/min (ft^3/min).

$Q_{s(std)}$ = Total cyclone flow rate at standard conditions, dscm/min (dscf/min).

T_m = Average absolute temperature of dry meter, °K (°R).

T_s = Average absolute stack gas temperature, °K (°R).

$V_{w(std)}$ = Volume of water vapor in gas sample (standard conditions), scm (scf).

θ = Total sampling time, min.

μ_{cyc} = Viscosity of mixed cyclone gas, micropoise.

μ_{std} = Viscosity of standard air, 180.1 micropoise.

6.2 Analysis of Cascade Impactor Data. Use the manufacturer's recommended procedures to analyze data from cascade impactors.

6.3 Analysis of Cyclone Data. Use the following procedures to analyze data from a single stage cyclone.

6.3.1 PM_{10} Weight. Determine the PM catch in the PM_{10} range from the sum of the weights obtained from Container Numbers 1 and 3 less the acetone blank.

6.3.2 Total PM Weight (optional). Determine the PM catch for greater than PM_{10} from the weight obtained from Container Number 2 less the acetone blank, and add it to the PM_{10} weight.

6.3.3 PM_{10} Fraction. Determine the PM_{10} fraction of the total particulate weight by dividing the PM_{10} particulate weight by the total particulate weight.

6.3.4 Aerodynamic Cut Size. Calculate the stack gas viscosity as follows:

$$\mu_{cyc} = C_1 + C_2 T_s + C_3 T_s^2 + C_4 f_{o2} - C_5 B_{ws}$$

6.3.4.1 The PM_{10} flow rate, at actual cyclone conditions, is calculated as follows:

$$Q_s = \frac{T_s}{K_1 P_s} \left[Q_{s(std)} + \frac{V_{w(std)}}{\theta} \right]$$

6.3.4.2 Calculate the molecular weight on a wet basis of the stack gas as follows:

$$M_w = M_d(1 - B_{ws}) + 18.0(B_{ws})$$

6.3.4.3 Calculate the actual D_{50} of the cyclone for the given conditions as follows:

$$D_{50} = \beta_1 \left[\frac{T_s}{M_w P_s} \right]^{0.2091} \left[\frac{\mu_s}{Q_s} \right]^{0.4091}$$

where $\beta_1 = 0.027754$ for metric units (0.15625 for English units).

6.3.5 Acceptable Results. The results are acceptable if two conditions are met. The first is that 9.0 μm ≤ D_{50} ≤ 11.0 μm. The second is that no sampling points are outside Δp_{min} and Δp_{max}, or that 80 percent ≤ I ≤ 120 percent and no more than one sampling point is outside Δp_{min} and Δp_{max}. If D_{50} is less than 9.0 μm, reject the results and repeat the test.

7. Bibliography

1. Same as Bibliography in Method 5.

2. McCain, J.D., J.W. Ragland, and A.D. Williamson. Recommended Methodology for the Determination of Particle Size Distributions in Ducted Sources. Final Report. Prepared for the California Air Resources Board by Southern Research Institute. May 1986.

3. Farthing, W.E., S.S. Dawes, A.D. Williamson, J.D. McCain, R.S. Martin, and J.W. Ragland. Development of Sampling Methods for Source PM_{10} Emissions. Southern Research Institute for the Environmental Protection Agency. April 1989. NTIS PB 89 190375. EPA/600/3–88–056.

4. *Application Guide for Source PM_{10} Measurement with Constant Sampling Rate.* EPA/600/3–88–057.

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assembly such that flow disturbances are minimized.

4.1.2.2 The setup calculations can be performed by using the following procedures.

4.1.2.2.1 In order to maintain a cut size of 10 μm in the sizing device, the flow rate through the sizing device must be maintained at a constant, discrete value during the run. If the sizing device is a cyclone that meets the design specifications in Figure 3 of this method, use the equations in Figure 4 of this method to calculate three orifice heads (ΔH): one at the average stack temperature, and the other two at temperatures ±28 °C (±50 °F) of the average stack temperature. Use ΔH calculated at the average stack temperature *as the pressure head for the sample flow rate* as long as the stack temperature during the run is within 28 °C (50 °F) of the average stack temperature. If the stack temperature varies by more than 28 °C (50 °F), then use the appropriate ΔH.

4.1.2.2.2 If the sizing device is a cyclone that does not meet the design specifications in Figure 3 of this method, use the equations in Figure 4 of this method, except use the procedures in Section 5.3 of this method to determine Q_s, the correct cyclone flow rate for a 10 μm size.

4.1.2.2.3 To select a nozzle, use the equations in Figure 5 of this method to calculate Δp_{min} and Δp_{max} for each nozzle at all three temperatures. If the sizing device is a cyclone that does not meet the design specifications in Figure 3 of this method, the example worksheets can be used.

4.1.2.2.4 Correct the Method 2 pitot readings to Method 201A pitot readings by multiplying the Method 2 pitot readings by the *square of a ratio of the Method 201A pitot* coefficient to the Method 2 pitot coefficient. Select the nozzle for which Δp_{min} and Δp_{max} bracket all of the corrected Method 2 pitot readings. If more than one nozzle meets this requirement, select the nozzle giving the greatest symmetry. Note that if the expected pitot reading for one or more points is near a limit for a chosen nozzle, it may be outside the limits at the time of the run.

4.1.2.2.5 Vary the dwell time, or sampling time, at each traverse point proportionately with the point velocity. Use the equations in Figure 6 of this method to calculate the dwell time at the first point and at each subsequent point. It is recommended that the number of minutes sampled at each point be rounded to the nearest 15 seconds.

4.1.3 Preparation of Collection Train. Same as in Method 5, Section 4.1.3, except omit directions about a glass cyclone.

4.1.4 Leak-Check Procedure. The sizing device is removed before the post-test leak-check to prevent any disturbance of the collected sample prior to analysis.

4.1.4.1 Pretest Leak-Check. A pretest leak-check of the entire sampling train, including the sizing device, is required. Use the leak-check procedure in Method 5, Section 4.1.4.1 to conduct a pretest leak-check.

4.1.4.2 Leak-Checks During Sample Run. Same as in Method 5, Section 4.1.4.1.

4.1.4.3 Post-Test Leak-Check. A leak-check is required at the conclusion of each sampling run. Remove the cyclone before the leak-check to prevent the vacuum created by the cooling of the probe from disturbing the collected sample and use the procedure in Method 5, Section 4.1.4.3 to conduct a post-test leak-check.

4.1.5 Method 201A Train Operation. Same as in Method 5, Section 4.1.5, except use the procedures in this section for isokinetic sampling and flow rate adjustment. Maintain the flow rate calculated in Section 4.1.2.2.1 of this method throughout the run provided the stack temperature is within 28 °C (50 °F) of the temperature used to calculate ΔH. If stack temperatures vary by more than 28 °C (50 °F), use the appropriate ΔH value calculated in Section 4.1.2.2.1 of this method. Calculate the dwell time at each traverse point as in Figure 6 of this method.

4.1.6 *Calculation of Percent Isokinetic* Rate and Aerodynamic Cut Size (D_{50}). Calculate percent isokinetic rate and D_{50} (see Calculations, Section 6 of this method) to determine whether the test was valid or another test run should be made. If there was difficulty in maintaining isokinetic sampling rates within the prescribed range, or if the D_{50} is not in its proper range because of source conditions, the Administrator may be consulted for possible variance.

4.2 Sample Recovery. If a cascade impactor is used, use the manufacturer's recommended procedures for sample recovery. If a cyclone is used, use the same sample recovery as that in Method 5, Section 4.2, except an increased number of sample recovery containers is required.

4.2.1 *Container Number 1* (In-Stack Filter). The recovery shall be the same as that for Container Number 1 in Method 5, Section 4.2.

4.2.3 *Container Number 2* (Cyclone or *Large PM Catch). This step is optional. The* anisokinetic error for the cyclone PM is theoretically larger than the error for the PM_{10} catch. Therefore, adding all the fractions to get a total PM catch is not as accurate as Method 5 or Method 201. Disassemble the cyclone and remove the nozzle to recover the large PM catch. Quantitatively recover the PM from the interior surfaces of the nozzle and cyclone, excluding the "turn around" cup and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.4 *Container Number 3* (PM_{10}). Quantitatively recover the PM from all of the surfaces from the cyclone exit to the front half of the in-stack filter holder, including the "turn around" cup inside the cyclone and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.6 *Container Number 4* (Silica Gel). The *recovery shall be the same as that for* Container Number 3 in Method 5, Section 4.2.

4.2.7 *Impinger Water*. Same as in Method 5, Section 4.2, under "Impinger Water."

4.3 Analysis. Same as in Method 5, Section 4.3, except handle Method 201A Container Number 1 like Container Number 1, Method 201A Container Numbers 2 and 3 like Container Number 2, and Method 201A Container Number 4 like Container Number 3. Use Figure 7 of this method to record the weights of PM collected. Use Figure 5–3 in Method 5, Section 4.3, to record the volume of water collected.

4.4 Quality Control Procedures. Same as in Method 5, Section 4.4.

5. Calibration

Maintain an accurate laboratory log of all calibrations.

5.1 Probe Nozzle, Pitot Tube, Metering System, Probe Heater Calibration, Temperature Gauges, Leak-check of Metering System, and Barometer. Same as in Method 5, Section 5.1 through 5.7, respectively.

5.2 Probe Cyclone and Nozzle Combinations. The probe cyclone and nozzle combinations need not be calibrated if both meet design specifications in Figures 2 and 3 of this method. If the nozzles do not meet design specifications, then test the cyclone and nozzle combinations for conformity with performance specifications (PS's) in Table 1 of this method. If the cyclone does not meet design specifications, then the cyclone and nozzle combination shall conform to the PS's and calibrate the cyclone to determine the relationship between flow rate, gas viscosity, and gas density. Use the procedures in Section 5.2 of this method to conduct PS tests and the procedures in Section 5.3 of this method to calibrate the cyclone. The purpose of the PS tests are to conform that the cyclone and nozzle combination has the desired sharpness of cut. Conduct the PS tests in a wind tunnel described in Section 5.2.1 of this method and particle generation system described in Section 5.2.2 of this method. Use five particle sizes and three wind velocities as listed in Table 2 of this method. A minimum of three replicate measurements of collection efficiency shall be *performed for each of the* 15 conditions listed, for a minimum of 45 measurements.

5.2.1 Wind Tunnel. Perform the calibration and PS tests in a wind tunnel (or equivalent test apparatus) capable of establishing and maintaining the required gas stream velocities within 10 percent.

5.2.2 Particle Generation System. The particle generation system shall be capable of producing solid monodispersed dye particles *with the mass median aerodynamic* diameters specified in Table 2 of this method. Perform the particle size distribution verification on an integrated sample obtained during the sampling period of each test. An acceptable alternative is to verify the size distribution of samples obtained before and after each test, with both samples required to meet the diameter and monodispersity requirements for an acceptable test run.

5.2.2.1 Establish the size of the solid dye particles delivered to the test section of the wind tunnel by using the operating parameters of the particle generation system, and verify them during the tests by microscopic examination of samples of the particles collected on a membrane filter. The particle size, as established by the operating parameters of the generation system, shall be within the tolerance specified in Table 2 of this method. The precision of the particle size verification technique shall be at least ±0.5 μm, and particle size determined by the verification technique shall not differ by more than 10 percent from that established by the operating parameters of the particle generation system.

5.2.2.2 Certify the monodispersity of the particles for each test either by microscopic inspection of collected particles on filters or by other suitable monitoring techniques such as an optical particle counter followed by a multichannel pulse height analyzer. If the proportion of multiplets and satellites in an aerosol exceeds 10 percent by mass, the particle generation system is unacceptable for the purpose of this test. Multiplets are particles that are agglomerated, and satellites are particles that are smaller than the specified size range.

5.2.3 Schematic Drawings. Schematic drawings of the wind tunnel and blower system and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques shall be furnished with calibration data to the reviewing agency.

5.2.4 Flow Measurements. Measure the cyclone air flow rates with a dry gas meter and a stopwatch, or a calibrated orifice system capable of measuring flow rates to within 2 percent.

5.2.5 Performance Specification Procedure. Establish test particle generator operation and verify particle size microscopically. If monodisperity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements may be made at this time.

5.2.5.1 The cyclone cut size, or D_{50}, of a cyclone is defined here as the particle size having a 50 percent probability of penetration. Determine the cyclone flow rate at which D_{50} is 10 μm. A suggested procedure is to vary the cyclone flow rate while keeping a constant particle size of 10 μm. Measure the PM collected in the cyclone (m_c), the exit tube (m_t), and the filter (m_f). Calculate cyclone efficiency (E_c) for each flow rate as follows:

$$E_c = \frac{m_c}{(m_c+m_t+m_f)} \times 100$$

5.2.5.2. Do three replicates and calculate the average cyclone efficiency [$E_{c(avg)}$] as follows:

$$E_{c(avg)} = (E_1 + E_2 + E_3)/3$$

Where E_1, E_2, and E_3 are replicate measurements of E_c.

5.2.5.3 Calculate the standard deviation (σ) for the replicate measurements of E_c as follows:

$$\left[\sigma = \left[\frac{(E_1{}^2+E_2{}^2+E_3{}^2) - \dfrac{(E_1+E_2+E_3)^2}{3}}{2}\right]^{1/2}\right.$$

If σ exceeds 0.10, repeat the replicated runs.

5.2.5.4 Measure the overall efficiency of the cyclone and nozzle, E_o, at the particle sizes and nominal gas velocities in Table 2 of this method using the following procedure.

5.2.5.5 Set the air velocity and particle size from one of the conditions in Table 2 of this method. Establish isokinetic sampling conditions and the correct flow rate in the cyclone (obtained by procedures in this section) such that the D_{50} is 10 μm. Sample long enough to obtain ±5 percent precision on total collected mass as determined by the precision and the sensitivity of measuring technique. Determine separately the nozzle catch (m_n), cyclone catch (m_c), cyclone exit tube (M_t), and collection filter catch (m_f) for each particle size and nominal gas velocity in Table 2 of this method. Calculate overall efficiency (E_o) as follows:

$$E_o = \frac{(m_n+m_c)}{(m_n+m_c+m_t+m_f)} \times 100$$

5.2.5.6 Do three replicates for each combination of gas velocity and particle size in Table 2 of this method. Use the equation below to calculate the average overall efficiency [$E_{o(avg)}$] for each combination following the procedures described in this section for determining efficiency.

$$E_{o(avg)} = (E_1 + E_2 + E_3)/3$$

Where E_1, E_2, and E_3 are replicate measurements of E_o.

5.2.5.7 Use the formula in Section 5.2.5.3 to calculate σ for the replicate measurements. If σ exceeds 0.10 or if the particle sizes and nominal gas velocities are not within the limits specified in Table 2 of this method, repeat the replicate runs.

5.2.6 Criteria for Acceptance. For each of the three gas stream velocities, plot the $E_{o(avg)}$ as a function of particle size on Figure 8 of this method. Draw smooth curves through all particle sizes. $E_{o(avg)}$ shall be within the banded region for all sizes, and the $E_{c(avg)}$ shall be 50±0.5 percent at 10 μm.

5.3 Cyclone Calibration Procedure. The purpose of this procedure is to develop the relationship between flow rate, gas viscosity, gas density, and D_{50}.

5.3.1 Calculate Cyclone Flow Rate. Determine flow rates and D_{50}'s for three different particle sizes between 5 μm and 15 μm, one of which shall be 10 μm. All sizes must be determined within 0.5 μm. For each size, use a different temperature within 60 °C (108 °F) of the temperature at which the cyclone is to be used and conduct triplicate runs. A suggested procedure is to keep the particle size constant and vary the flow rate.

5.3.1.1. On log-log graph paper, plot the Reynolds number (Re) on the abscissa, and the square root of the Stokes 50 number [$(Stk_{50})^{1/2}$] on the ordinate for each temperature. Use the following equations to compute both values:

$$Re = \frac{4\,\rho\,Q_{cyc}}{d_{cyc}\,\pi\,\mu_{cyc}}$$

$$(Stk_{50})^{1/2} = \left[\frac{4\,Q_{cyc}\,(D_{50})^2}{9\,\pi\,\mu_{cyc})^3}\right]^{1/2}$$

where:

Q_{cyc} = Cyclone flow rate, cm³/sec.

ρ = Gas density, g/cm³.

d_{cyc} = Diameter of cyclone inlet, cm.

μ_{cyc} = Viscosity of gas through the cyclone, micropoise.

D_{50} = Aerodynamic diameter of a particle having a 50 percent probability of penetration, cm.

5.3.1.2. Use a linear regression analysis to determine the slope (m) and the Y-intercept (b). Use the following formula to determine Q, the cyclone flow rate required for a cut size of 10 μm.

$$Q_s = \frac{\pi\,\mu_{cyc}}{4}\left[(3000)(K_1)-b\right]^{-1/(0.5-m)}\left[\frac{T_s}{M_s\,P_s}\right]^{m/(m-0.5)} d^{(m-1.5)/(m-0.5)}$$

where:

m = Slope of the calibration line.

b = y-intercept of the calibration line.

Q_s = Cyclone flow rate for a cut size of 10 μm, cm³/sec.

d = Diameter of nozzle, cm.

T_s = Stack gas temperature, °R.

P_s = Absolute stack pressure, in. Hg.

M_s = Molecular weight of the stack gas, lb/lb-mole.

$K_1 = 4.077 \times 10^{-3}$.

5.3.1.3 Refer to the Method 201A operators manual, entitled *Application Guide for Source PM₁₀ Measurement with Constant*

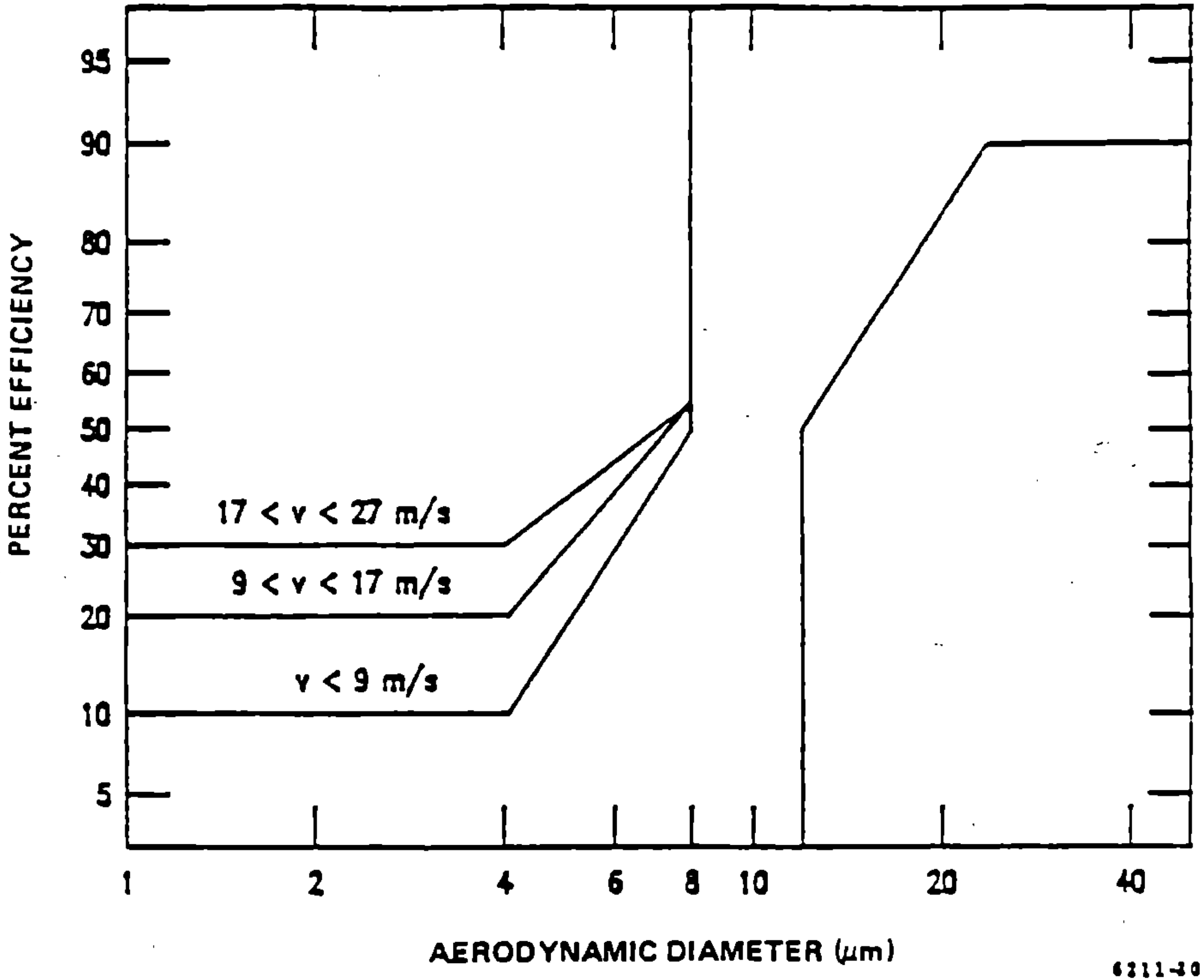


Figure 13. Efficiency envelope for the PM_{10} cyclone.

BILLING CODE 6560-50-C

Emission Gas Recycle. Data Reduction. Version 3.4 MAY 1986

Test ID. Code: Chapel Hill 2.
Test Location: Baghouse Outlet.
Test Site: Chapel Hill.
Test Date: 10/20/86.
Operators(s): JB RH MH.

Entered Run Data

Temperatures:	
T(STK)	[illegible] F
T(RCL)	[illegible] F
T(LFE)	81.0 F
T(DGM)	78.0 F
System Pressures:	
DH(ORI)	1.10 INWG
DP(TOT)	1.91 INWG
P(INL)	12.15 INWG
DP(RCL)	2.21 INWG
DP(PTO)	0.00 INWG
Miscellaneous:	
P(BAR)	29.98 INWG

Entered Run Data—Continued

DP(STK)	0.10 INWG
V(DGM)	13.744 FT3
TIME	60.00 MIN
% CO2	8.00
	20.00
[illegible]	0.2500
...ntent:	
Estimate	0.0%
or	
Condenser	7.0 ML
Column	0.0 GM
Raw Masses:	
Cyclone 1	21.7 MG
Filter	11.7 MG
Impinger Residue	0.0 MG
Blank Values:	
CYC Rinse	0.0 MG
Filter Holder Rinse	0.0 MG
Filter Blank	0.0 MG
Impinger Rinse	0.0 MG

Calibration Values	
CP(TOT)	0.840
DH@ORI	10.990
M(TOT LFE)	0.2290
B(TOT LFE)	−.0058
M(RCL LFE)	0.0848
B(RCL LFE)	−.0007
DGM GAMMA	0.9840

Reduced Data

Stack Velocity (FT/SEC)	15.9[illegible]
Stack Gas Moisture (%)	2.4
Sample Flow Rate (ACFM)	0.3104
Total Flow Rate (ACFM)	0.5819
Recycle Flow Rate (ACFM)	0.2785
Percent Recycle	46.7
Isokinetic Ratio (%)	95.1

	(Particulate) (UM)	(Particulate) (% <)	(MG/DNCM)	(GR/ACF)	(GR/DCF)	(LB/DSCF) (X 1E6)
Cyclone 1	10.15	35.8	56.8	0.01794	0.02470	3.53701
Backup Filter			30.5	0.00968	0.01332	1.907
Particulate Total			87.2	0.02762	0.03802	5.444

Note: Figure 14. Example inputs and outputs of the EGR reduction program.

Method 201A—Determination of PM_{10} Emissions (Constant Sampling Rate Procedure)

1. Applicability and Principle

1.1 Applicability. This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 (PM_{10}) from stationary sources. The EPA recognizes that condensible emissions not collected by an in-stack method are also PM_{10}, and that emissions that contribute to ambient PM_{10} levels are the sum of condensible emissions and emissions measured by an in-stack PM_{10} method, such as this method or Method 201. Therefore, for establishing source contributions to ambient levels of PM_{10}, such as for emission inventory purposes, EPA suggests that source PM_{10} measurement include both in-stack PM_{10} and condensible emissions. Condensible emissions may be measured by an impinger analysis in combination with this method.

1.2 Principle. A gas sample is extracted at a constant flow rate through an in-stack sizing device, which separates PM greater than PM_{10}. Variations from isokinetic sampling conditions are maintained within well-defined limits. The particulate mass is determined gravimetrically after removal of uncombined water.

2. Apparatus

Note: Methods cited in this method are part of 40 CFR part 60, appendix A.

2.1 Sampling Train. A schematic of the Method 201A sampling train is shown in Figure 1 of this method. With the exception of the PM_{10} sizing device and in-stack filter, this train is the same as an EPA Method 17 train.

2.1.1 Nozzle. Stainless steel (316 or equivalent) with a sharp tapered leading edge. Eleven nozzles that meet the design specification in Figure 2 of this method are recommended. A larger number of nozzles with small nozzle increments increase the likelihood that a single nozzle can be used for the entire traverse. If the nozzles do not meet the design specifications in Figure 2 of this method, then the nozzles must meet the criteria in Section 5.2 of this method.

2.1.2 PM_{10} Sizer. Stainless steel (316 or equivalent), capable of determining the PM_{10} fraction. The sizing device shall be either a cyclone that meets the specifications in Section 5.2 of this method or a cascade impactor that has been calibrated using the procedure in Section 5.4 of this method.

2.1.3 Filter Holder. 63-mm, stainless steel. An Andersen filter, part number SE274, has been found to be acceptable for the in-stack filter. Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.4 Pitot Tube. Same as in Method 5, Section 2.1.3. The pitot lines shall be made of heat resistant tubing and attached to the probe with stainless steel fittings.

2.1.5 Probe Liner. Optional, same as in Method 5, Section 2.1.2.

2.1.6 Differential Pressure Gauge, Condenser, Metering System, Barometer, and Gas Density Determination Equipment. Same as in Method 5, Sections 2.1.4, and 2.1.7 through 2.1.10, respectively.

2.2 Sample Recovery.

2.2.1 Nozzle, Sizing Device, Probe, and Filter Holder Brushes. Nylon bristle brushes with stainless steel wire shafts and handles, properly sized and shaped for cleaning the nozzle, sizing dedvice, probe or probe liner, and filter holders.

2.2.2 Wash Bottles, Glass Sample Storage Containers, Petri Dishes, Graduated Cylinder and Balance, Plastic Storage Containers, Funnel and Rubber Policeman, and Funnel. Same as in Method 5, Sections 2.2.2 through 2.2.8, respectively.

2.3 Analysis. Same as in Method 5, Section 2.3.

3. Reagents

The reagents for sampling, sample recovery, and analysis are the same as that specified in Method 5, Sections 3.1, 3.2, and 3.3, respectively.

4. Procedure

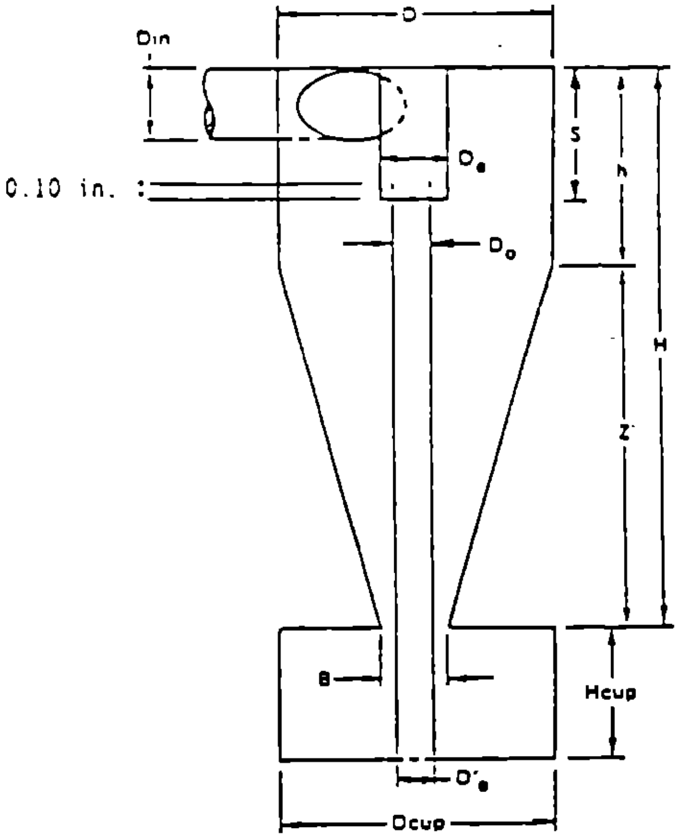
4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. Same as in Method 5, Section 4.1.1.

4.1.2 Preliminary Determinations. Same as in Method 5, Section 4.1.2, except use the directions on nozzle size selection and sampling time in this method. Use of any nozzle greater than 0.16 in. in diameter require a sampling port diameter of 6 inches. Also, the required max[illegible]ber of traverse points at [illegible] be 12.

4.1.2.1 The sizing [illegible] be in-stack or maintained at stack temperature during sampling. The blockage effect of the CSR sampling assembly will be minimal if the cross-sectional area of the sampling assembly is 3 percent or less of the cross-sectional area of the duct. If the cross-sectional area of the assembly is greater than 3 percent of the cross-sectional area of the duct, then either determine the pitot coefficient at sampling conditions or use a standard pitot with a known coefficient in a configuration with the CSR sampling

Cyclone Interior Dimensions



	Dimensions (±0.02 cm, ±0.01 in.)											
	Din	D	De	B	H	h	Z	S	Hcup	Dcup	D_e'	D_o
cm	1.27	4.47	1.50	1.88	6.95	2.24	4.71	1.57	2.25	4.45	1.02	1.24
inches	0.50	1.76	0.59	0.74	2.74	0.88	1.85	0.62	0.89	1.75	0.40	0.49

Figure 12. Cyclone design specifications.

TABLE 1. PERFORMANCE SPECIFICATIONS FOR SOURCE PM_{10} Cyclones and Nozzle Combinations

Parameter	Units	Specification
1. Collection efficiency	Percent	Such that collection efficiency falls within envelope specified by Section 5.7.6 and Figure 13.
2. Cyclone cut size (D_{50}).	μm	10±1 μm aerodynamic diameter.

TABLE 2. PARTICLE SIZES AND NOMINAL GAS VELOCITIES FOR EFFICIENCY

Particle size (μm)[a]	Target gas velocities (m/sec)		
	7±1.0	15±1.5	25±2.5
5±0.5			
7±0.5			
10±0.5			
14±1.0			
20±1.0			

(a) Mass median aerodynamic diameter.

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Run Code	Date	Stack Temperature (°F)	Gas Composition % CO_2 % O_2 % CO
Sampler ID	Start Time	Stack Static Pressure (in H_2O)	Moisture Content
Filter ID	End Time	Ambient Temperature (°F)	
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in Hg)	
Sampling Location	DGM (Initial)	Gas Velocity	Pilot Leak Check (Pos) (Neg)
Nozzle Diameter-ID (in.)	DGM (final)	System Leak Check (≥ 15 in. Hg)	Notes
Operator (s)	Sample Volume (ft^3)		
Dual Manometer Leveled and Zeroed?			
Magnehelics Zeroed?			

Run Time	Port No / Trav. Pt.	ΔP Pilot	ΔH Sample	DGM Volume	ΔP Total	P Inlet	ΔP Recycle	T_1 Stack	T_2 Recycle	T_3 Probe	T_4 LFE	T_5 DGM

Figure 10. Example EGR Procedure data sheet.

BILLING CODE 6560-50-C

Plant ____________________
Date ____________________
Run no. ____________________
Filter no. ____________________
Amount liquid lost during transport______
Acetone blank volume, ml______
Acetone wash volume, ml (2)______(3)______
Acetone blank conc., mg/mg (Equation 5–4, Method 5)______
Acetone wash blank, mg (Equation 5–5, Method 5)______

Container number	Weight of particulate matter, mg		
	Final weight	Tare weight	Weight gain
1			
3			
Total			
Less acetone blank			
Weight of PM_{10}			
2			
Less acetone blank			

Container number	Weight of particulate matter, mg		
	Final weight	Tare weight	Weight gain
Total particulate weight			

Figure 11. ECR method analysis sheet.

BILLING CODE 6560-50-M

Molecular weight of stack gas, wet basis, M_w lb/lb mole. = ______

Pressure upstream of LFE, in. Hg. = 0.6

Gas analysis:

%O_2 .... = ______

Fraction moisture content, B_{ws}. = ______

Calibration data:

Nozzle diameter, D_n, in. = ______

Pitot coefficient, C_p .... = ______

Total LFE calibration constant, X_t. = ______

Total LFE calibration constant, Y_t. = ______

Absolute pressure upstream of LFE:

$P_{LFE} = P_{bar} + 0.6$ .... = ______ in. Hg

Viscosity of gas in total LFE:

$\mu_{LFE} = 152.418 + 0.2552\ T_m + 3.2355 \times 10^{-5}\ T_m^2 + 0.53147\ (\%O_2)$. = ______

Viscosity of dry stack gas:

$\mu_d = 152.418 + 0.2552\ T_s + 3.2355 \times 10^{-5}\ T_s^2 + 0.53147\ (\%O_2)$. = ______

Constants:

$$K_1 = 1.5752 \times 10^{-5} \frac{\mu_{LFE}\ T_m\ P_s^{0.7051}\ \mu_d}{P_{LFE}\ M_d^{0.2949}\ T_s^{0.7051}} = \_\_\_\_\_\_$$

$$K_2 = 0.1539 \frac{\mu_{LFE}\ T_m\ D_n^2\ C_p}{P_{LFE}} \left[\frac{P_s}{T_s}\right]^{1/2}$$

$$K_3 = \frac{B_{ws}\ \mu_d\ [1 - 0.2949\ (1 - 18/M_d)] + 74.143\ B_{ws}\ (1 - B_{ws})}{\mu_d - 74.143\ B_{ws}} = \_\_\_\_\_\_$$

Figure 8. Example worksheet 2, total LFE pressure head.

$$A_t \frac{K_1}{X_t} = \frac{\mu_{LFE}\ Y_t}{180.1\ X_t} = \_\_\_\_\_\_$$

$$B_t = \frac{K_2\ K_3}{(M_w)^{1/2}\ X_t} = \_\_\_\_\_\_$$

Total LFE pressure head:

$\Delta p_t = A_t - B_t\ (\Delta p)^{1/2}$ = ______ in. H_2O

Barometric pressure, P_{bar}, in. Hg = ______

Absolute stack pressure, P_s in. Hg = ______

Average stack temperature, T_s, °R = ______

Meter temperature, T_m, °R = ______

Molecular weight of stack gas, dry basis, M_d, lb/lb mole = ______

Viscosity of LFE gas, μ_{LFE}, poise = ______

Viscosity of dry stack gas, μ_d, poise = ______

Absolute pressure upstream of LFE, P_{LFE}, in. Hg = ______

Calibration data:

Nozzle diameter, D_n, in. = ______

Pitot coefficient, C_p = ______

Recycle LFE calibration constant, X_r = ______

Recycle LFE calibration constant, Y_r = ______

$$K_1 = 1.5752 \times 10^{-5} \frac{\mu_{LFE}\ T_m\ P_s^{0.7051}\ \mu_d}{P_{LFE}\ M_d^{0.2949}\ T_s^{0.7051}} = \_\_\_\_\_\_$$

$$K_2 = 0.1539 \frac{M_{LFE}\ T_m\ D_n^2\ C_p}{P_{LFE}} = \left[\frac{P_s}{T_s} = \right]^{1/2}$$

$$K_4 = \frac{\mu_d}{M_w^{0.7051}\ M_d^{0.2949}\ (\mu_d - 74.143\ B_{ws})} = \_\_\_\_\_\_$$

$$A_r = \frac{K}{X_r} = \frac{\mu_{LFE}\ _r}{180.1\ X_r} = \_\_\_\_\_\_$$

$$B_r = \frac{K_2}{X_r} = \_\_\_\_\_\_$$

Pressure head for recycle LFE:

$\Delta P_r = A_r - B_r\ (\Delta p)^{1/2}$ = ______ in. H_2O

Figure 9. Example worksheet 3. recycle LFE pressure head.

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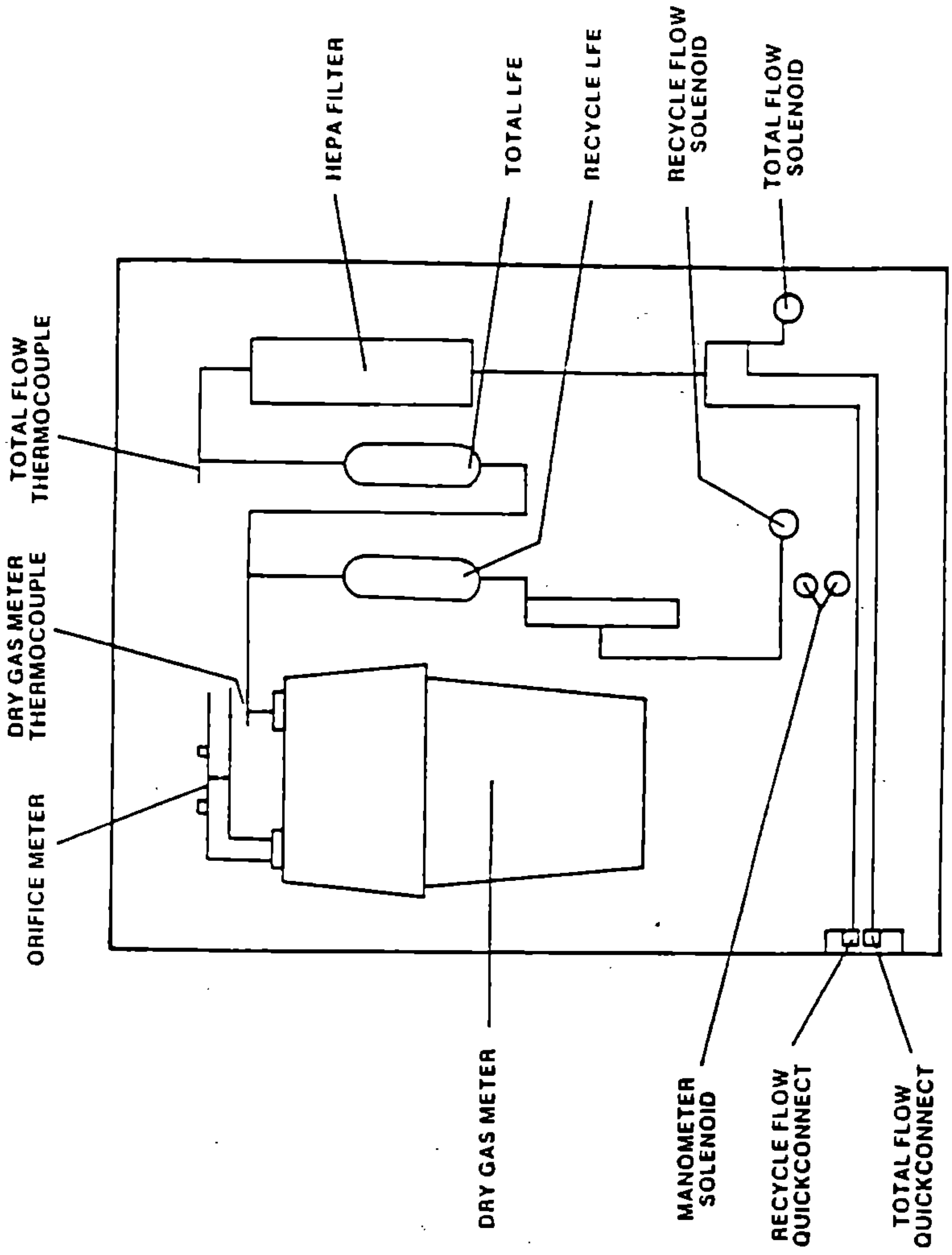

Figure 5. Example EGR control module (rear view) showing principle components.

EXAMPLE EMISSION GAS RECYCLE SETUP SHEET

VERSION 3.1 MAY 1986

TEST I.D.: SAMPLE SETUP
RUN DATE: 11/24/86
LOCATION: SOURCE SIM
OPERATOR(S): RH JB
NOZZLE DIAMETER (IN): .25

STACK CONDITIONS:

AVERAGE TEMPERATURE (F): 200.0
AVERAGE VELOCITY (FT/SEC): 15.0
AMBIENT PRESSURE (IN HG): 29.92
STACK PRESSURE (IN H20): .10

GAS COMPOSITION:
H20 = 10.0% .... MD = 28.84
02 = 20.9% .... MW = 27.75
CO2 = .0% .... (LB/LB MOLE)

TARGET PRESSURE DROPS

TEMPERATURE (F)

DP(PTO)	150	161	172	183	194	210	217	228
0.026	SAMPLE	.49	.49	.48	.47	.46	.45	.45
	TOTAL	1.90	1.90	1.91	1.92	1.92	1.92	1.93
	RECYCLE	2.89	2.92	2.94	2.97	3.00	3.02	3.05
	%RCL	61%	61%	62%	62%	63%	63%	63%
.031	.58	.56	.55	.55	.55	.54	.53	.52
	1.88	1.89	1.89	1.90	1.91	1.91	1.91	1.92
	2.71	2.74	2.77	2.80	2.82	2.85	2.88	2.90
	57%	57%	58%	58%	59%	59%	60%	60%
.035	.67	.65	.64	.63	.62	.61	.60	.59
	1.88	1.88	1.89	1.89	1.90	1.90	1.91	1.91
	2.57	2.60	2.63	2.66	2.69	2.72	2.74	2.74
	54%	55%	55%	56%	56%	57%	57%	57%
.039	.75	.74	.72	.71	.70	.69	.67	.66
	1.87	1.88	1.88	1.89	1.89	1.90	1.90	1.91
	2.44	2.47	2.50	2.53	2.56	2.59	2.62	2.57
	51%	52%	52%	53%	53%	54%	54%	55%

Figure 6. Example ECR setup sheet.

Barometric pressure, P_{bar}, in. Hg = ________
Stack static pressure, P_g, in. H_2O = ________
Average stack temperature, t_s, °F = ________
Meter temperature, t_m, °F = ________
Gas analysis:
%CO_2 = ________
%O_2 = ________
%N_2 + %CO = ________
Fraction moisture content, B_{ws} = ________

Calibration data:
Nozzle diameter, D_n in. = ________
Pitot coefficient, C_p = ________
$\Delta H@$, in. H_2O = ________
Molecular weight of stack gas, dry basis:
$M_d = 0.44 (\%CO_2) + 0.32 (\%O_2) + 0.28 (\%N_2 + \%CO)$ = ____ lb/lb mole

Molecular weight of stack gas, wet basis:
$M_s = M_d (1 - B_{ws}) + 18 B_{ws}$ = ________ lb/lb mole
Absolute stack pressure:
$P_s = P_{bar} + (P_g/13.6)$ = ________ in. Hg

$$K = 846.72\, D_n^4\, \Delta H@\, C_p^2\, (1 - B_{ws})^2\, \frac{M_d (t_m + 460) P_s}{M_s (t_s + 460) P_{bar}} = \_\_\_\_\_\_$$

Desired meter orifice pressure (ΔH) for velocity head of stack gas (Δp):
$\Delta H = K\, \Delta p$ = ________ in. H_2O

Figure 7. Example worksheet 1, meter orifice pressure head calculation.

Barometric pressure, P_{bar} in. Hg = ________

Absolute stack pressure, P_s, in. Hg = ________
Average stack temperature, T_s, °R = ________
Meter temperature, T_m, °R = ________

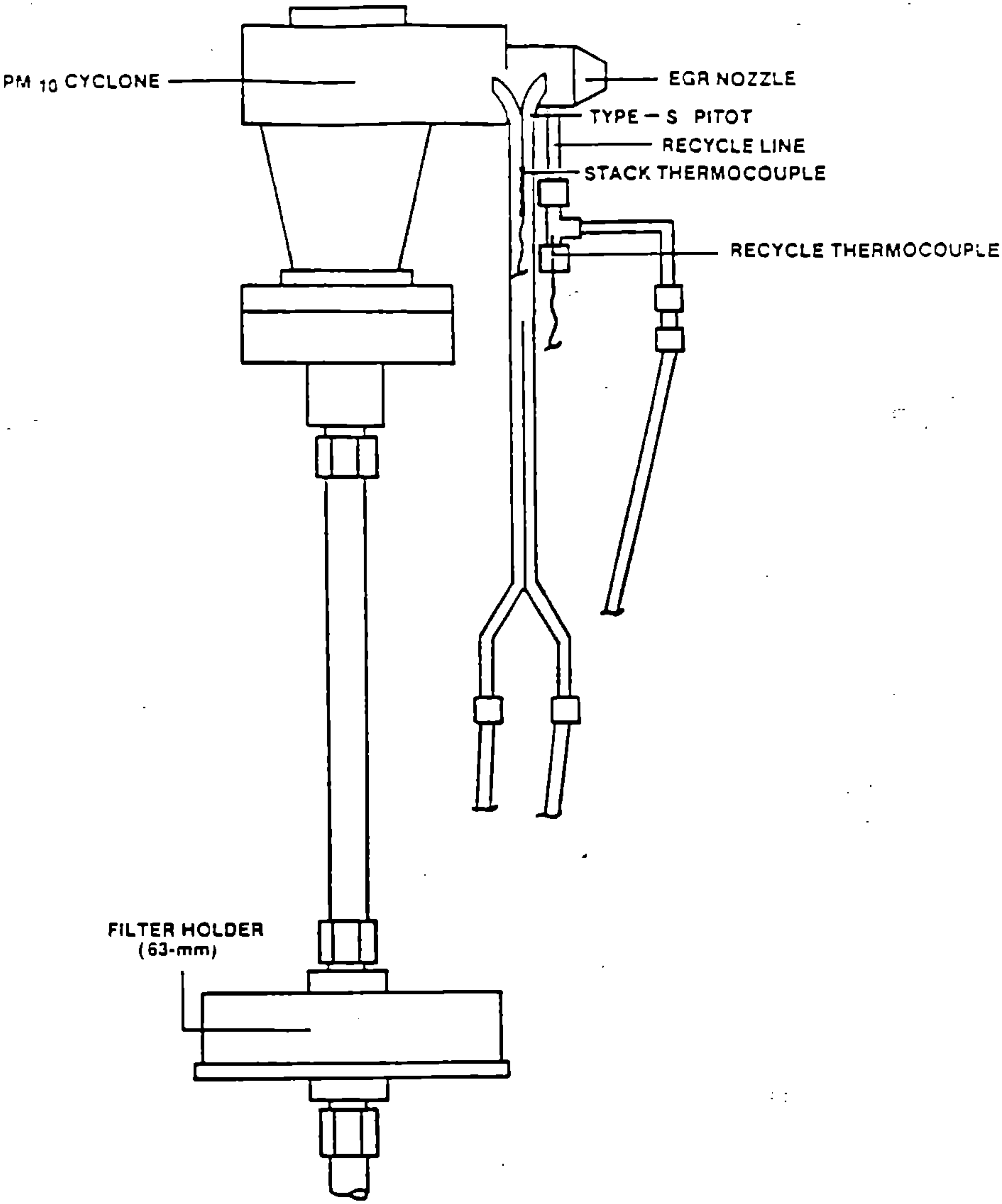

Figure 3. EGR PM_{10} cyclone sampling device.

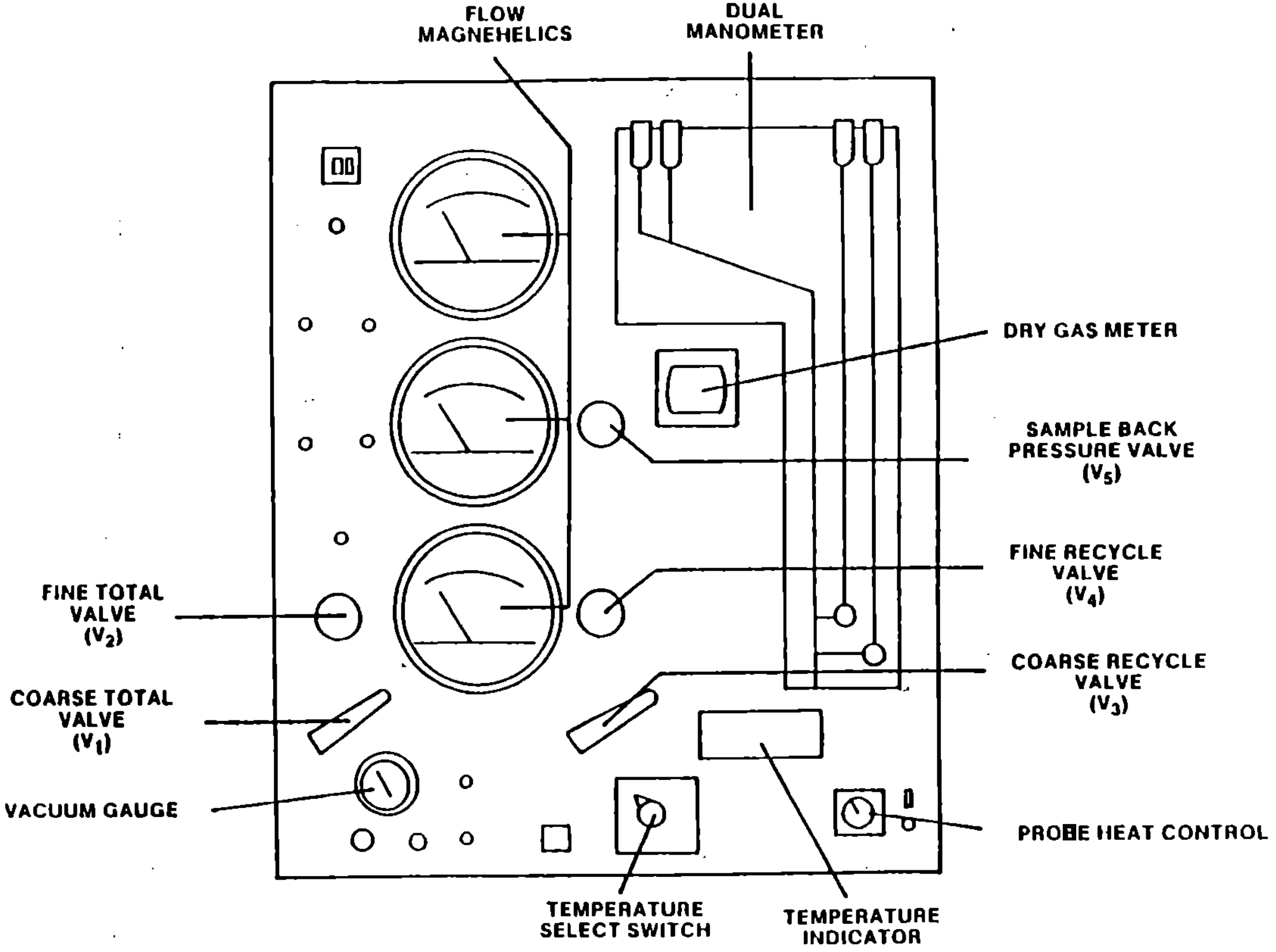

Figure 4. Example EGR control module (front view) showing principle components.

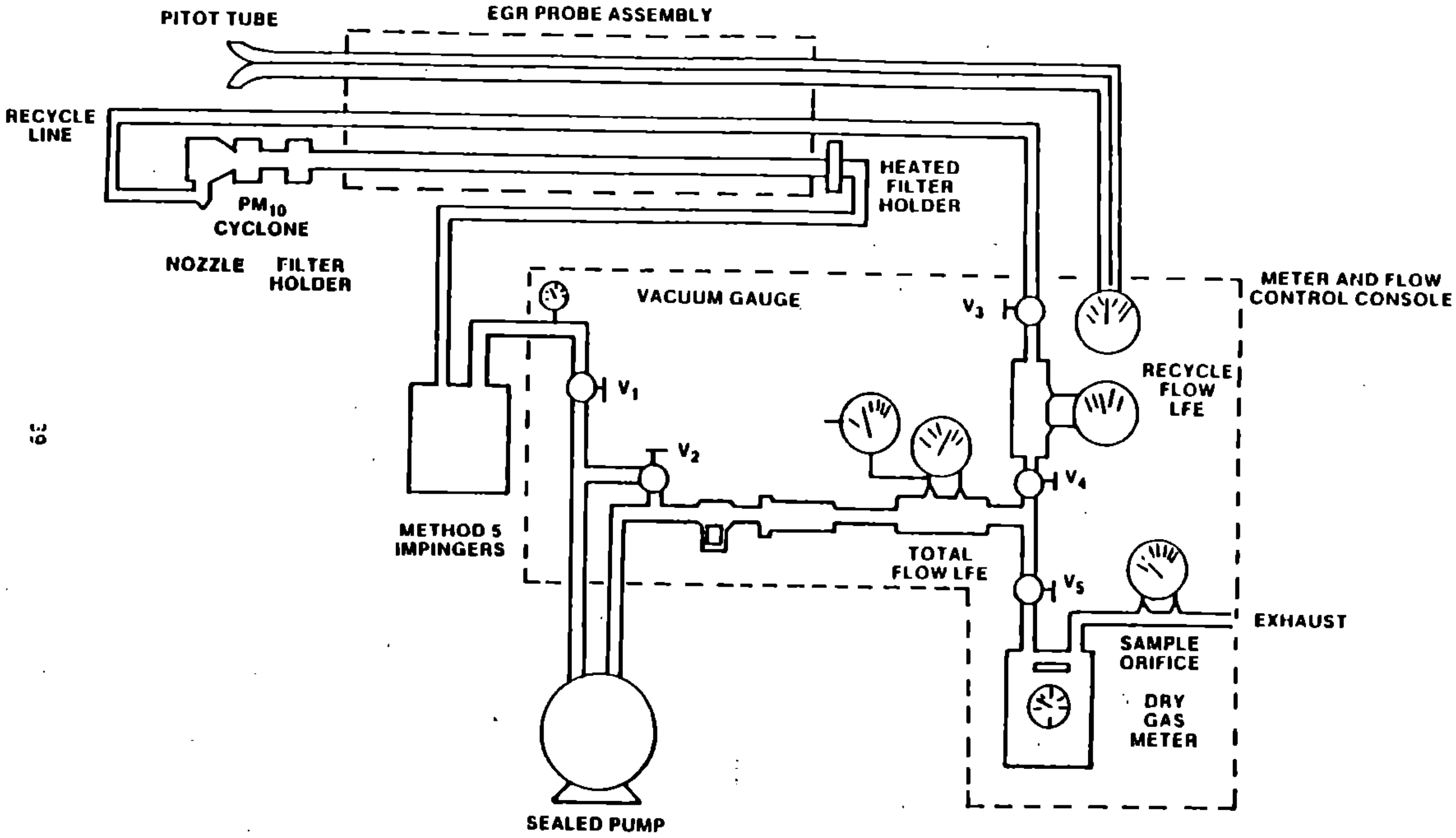

Figure 1. Schematic of the exhaust gas recycle train.

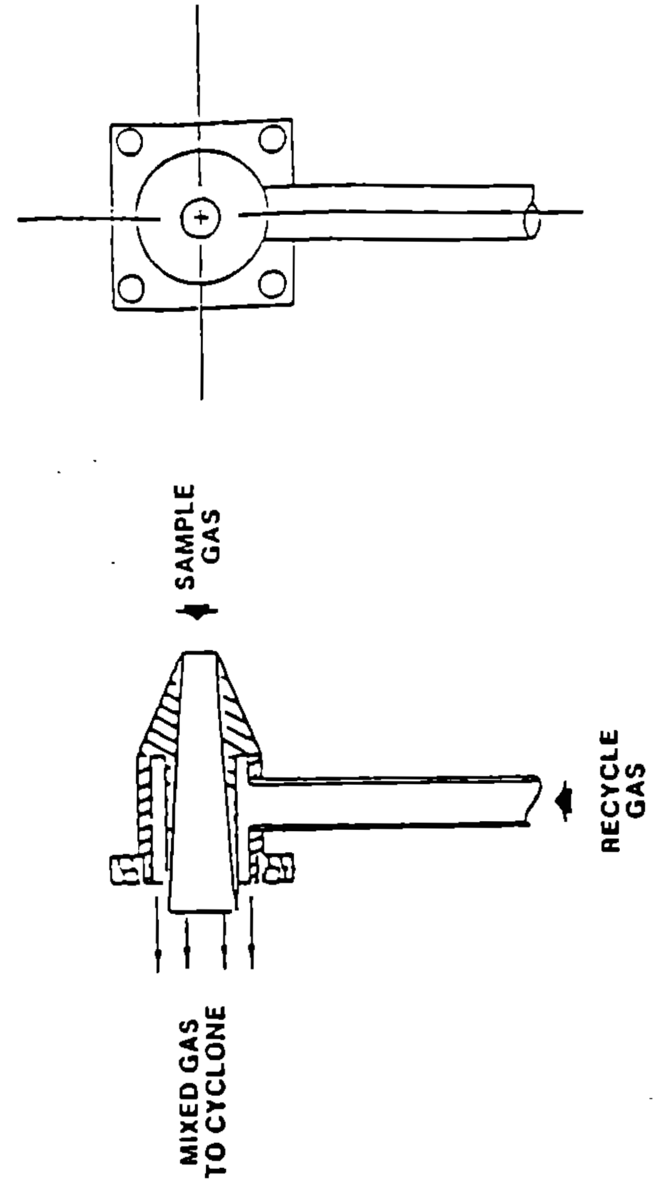

Figure 2. Schematic of EGR nozzle assembly.

$$E_o = \frac{(m_a + m_d)}{(m_a + m_r + m_t + m_f)} \times 100$$

5.7.5.7 Do three replicates for each combination of gas velocities and particle sizes in Table 2 of this method. Calculate E_o for each particle size following the procedures described in this section for determining efficiency. Calculate the standard deviation (σ) for the replicate measurements. If σ exceeds 0.10, repeat the replicate runs.

5.7.6 Criteria for Acceptance. For each of the three gas stream velocities, plot the average E_o as a function of particle size on Figure 13 of this method. Draw a smooth curve for each velocity through all particle sizes. The curve shall be within the banded region for all sizes, and the average E_o for a D_{50} for 10 μm shall be 50 ± 0.5 percent.

5.8 Cyclone Calibration Procedure. The purpose of this section is to develop the relationship between flow rate, gas viscosity, gas density, and D_{50}. This procedure only needs to be done on those cyclones that do not meet the design specifications in Figure 12 of this method.

5.8.1 Calculate cyclone flow rate. Determine the flow rates and D_{50}'s for three different particle sizes between 5 μm and 15 μm, one of which shall be 10 μm. All sizes must be within 0.5 μm. For each size, use a different temperature within 60°C (108°F) of the temperature at which the cyclone is to be used and conduct triplicate runs. A suggested procedure is to keep the particle size constant and vary the flow rate. Some of the values obtained in the PS tests in Section 5.7.5 may be used.

5.8.1.1 On log-log graph paper, plot the Reynolds number (Re) on the abscissa, and the square root of the Stokes 50 number $[(STK_{50})^{1/2}]$ on the ordinate for each temperature. Use the following equations:

$$Re = \frac{4\rho K_{cyc}}{d_{cyc} \pi \mu_{cyc}}$$

$$(Stk_{50})^{1/2} = \left[\frac{4\, Q_{cyc} (D_{50})^2}{9\, \pi\, \mu_{cyc} (d_{cyc})^3} \right]^{1/2}$$

where:

Q_{cyc} = Cyclone flow rate cm³/sec.

ρ = Gas density, g/cm³.

d_{cyc} = Diameter of cyclone inlet, cm.

μ_{cyc} = Viscosity of gas through the cyclone, poise.

D_{50} = Cyclone cut size, cm.

5.8.1.2 Use a linear regression analysis to determine the slope (m), and the y-intercept (b). Use the following formula to determine Q, the cyclone flow rate required for a cut size of 10 μm.

$$Q = \frac{\pi \mu_{cyc}}{4} \left[(3000)(K_1)^b \right] -(0.5-m) \left[\frac{T_s}{M_s P_s} \right]^{m/(m-0.5)} d^{(m-1.5)/(m-0.5)}$$

where:

Q = Cyclone flow rate for a cut size of 10 μm, cm³/sec.

T_s = Stack gas temperature, °K.

d = Diameter of nozzle, cm.

K_1 = 4.077 X 10⁻³.

5.8.2 Directions for Using Q. Refer to Section 5 of the EGR operators manual for directions in using this expression for Q in the setup calculations.

6. Calculations

6.1 The EGR data reduction calculations are performed by the EGR reduction computer program, which is written in IBM BASIC computer language and is available through NTIS, Accession number PB90-500000, 5285 Port Royal Road, Springfield, Virginia 22161. Examples of program inputs and outputs are shown in Figure 14 of this method.

6.1.1 Calculations can also be done manually, as specified in Method 5, Sections 6.3 through 6.7, and 6.9 through 6.12, with the addition of the following:

6.1.2 Nomenclature.

B_{ws} = Moisture fraction of mixed cyclone gas, by volume, dimensionless.

C_1 = Viscosity constant, 51.12 micropoise for °K (51.05 micropoise for °R).

C_2 = Viscosity constant, 0.372 micropoise/°K (0.207 micropoise/°R).

C_3 = Viscosity constant, 1.05 X 10⁻⁴ micropoise/°K² (3.24 X 10⁻⁵ micropoise/°R²).

C_4 = Viscosity constant, 53.147 micropoise/fraction O_2.

C_5 = Viscosity constant, 74.143 micropoise/fraction H_2O.

D_{50} = Diameter of particles having a 50 percent probability of penetration, μm.

f_{O2} = Stack gas fraction O_2, by volume, dry basis.

K_1 = 0.3858 °K/mm Hg (17.64 °R/in. Hg).

M_c = Wet molecular weight of mixed gas through the PM_{10} cyclone, g/g-mole (lb/lb-mole).

M_d = Dry molecular weight of stack gas, g/g-mole (lb/lb-mole).

P_{bar} = Barometer pressure at sampling site, mm Hg (in. Hg).

P_{inlet} = Gauge pressure at inlet to total LFE, mm H_2O (in. H_2O).

P_s = Absolute stack pressure, mm Hg (in. Hg).

Q_T = Total cyclone flow rate at wet cyclone conditions, m³/min (ft³/min).

$Q_{T(std)}$ = Total cyclone flow rate at standard conditons, dscm/min (dscf/min).

T_m = Average temperature of dry gas meter, °K (°R).

T_s = Average stack gas temperature, °K (°R).

$V_{w(std)}$ = Volume of water vapor in gas sample (standard conditions), scm (scf).

X_T = Total LFE linear calibration constant, m³/[(min)(mm H_2O)] {ft³/[(min)(in. H_2O)]}.

Y_T = Total LFE linear calibration constant, dscm/min (dscf/min).

ΔP_T = Pressure differential across total LFE, mm H_2O, (in. H_2O).

θ = Total sampling time, min.

μ_{cyc} = Viscosity of mixed cyclone gas, micropoise.

μ_{LFE} = Viscosity of gas laminar flow elements, micropoise.

μ_{std} = Viscosity of standard air, 180.1 micropoise.

6.2 PM_{10} Particulate Weight. Determine the weight of PM_{10} by summing the weights obtained from Container Numbers 1 and 3, less the acetone blank.

6.3 Total Particulate Weight. Determine the particulate catch for PM greater than PM_{10} from the weight obtained from Container Number 2 less the acetone blank, and add it to the PM_{10} particulate weight.

6.4 PM_{10} Fraction. Determine the PM_{10} fraction of the total particulate weight by dividing the PM_{10} particulate weight by the total particulate weight.

6.5 Total Cyclone Flow Rate. The average flow rate at standard conditions is determined from the average pressure drop across the total LFE and is calculated as follows:

$$Q_{T(std)} = K_1 \left[X_T\, \Delta P \frac{\mu_{std}}{\mu_{LFE}} + Y_T \right] \frac{P_{bar} + P_{inlet}/13.6}{T_m}$$

The flow rate, at actual cyclone conditions, is calculated as follows:

$$Q_s = \frac{T_s}{K_1 P_s}\left[Q_{s(std)} + \frac{V_{w(std)}}{\Theta}\right]$$

6.6.1 Determine the water fraction of the mixed gas through the cyclone by using the equation below.

$$B_c = \frac{V_{w(std)}}{Q_{s(std)}\,\Theta + V_{w(std)}}$$

6.6.2 Calculate the cyclone gas viscosity as follows:
$\mu_{cyc} = C_1 + C_2 T_s + C_3 T_s^2 + C_4 f_{O2} - C_5 B_c$

6.6.3 Calculate the molecular weight on a wet basis of the cyclone gas as follows:
$M_c = M_d(1 - B_c) + 18.0(B_c)$

6.6.4 If the cyclone meets the design specification in Figure 12 of this method, calculate the actual D_{50} of the cyclone for the run as follows:

$$D_{50} = \beta_1\left[\frac{T_s}{M_c P_s}\right]^{0.2091}\left[\frac{\mu_{cyc}}{Q_s}\right]^{0.7091}$$

where $\beta_1 = 0.1562$.

6.6.5 If the cyclone does not meet the design specifications in Figure 12 of this method, then use the following equation to calculate D_{50}.

$$D_{50} = (3)(10)^b\,(7.376 \times 10^{-4})^m\left[\frac{M_c P_s}{T_s}\right]\left[\frac{4\,Q_s}{\pi\,\mu_{cyc}}\right]d^{(1.5-m)}$$

6.6 Aerodynamic Cut Size. Use the following procedure to determine the aerodynamic cut size (D_{50}).

where:

m = Slope of the calibration curve obtained in Section 5.8.2.

b = y-intercept of the calibration curve obtained in Section 5.8.2.

6.7 Acceptable Results. Acceptability of anisokinetic variation is the same as Method 5, Section 6.12.

6.7.1 If 9.0 μm < D_{50} < 11 μm and 90 < I < 110, the results are acceptable. If D_{50} is greater than 11 μm, the Administrator may accept the results. If D_{50} is less than 9.0 μm, reject the results and repeat the test.

7. Bibliography

1. Same as Bibliography in Method 5.

2. McCain, J.D., J.W. Ragland, and A.D. Williamson. Recommended Methodology for the Determination of Particles Size Distributions in Ducted Sources. Final Report. Prepared for the California Air Resources Board by Southern Research Institute. May 1986.

3. Farthing, W.E., S.S. Dawes, A.D. Williamson, J.D. McCain, R.S. Martin, and J.W. Ragland. Development of Sampling Methods for Source PM-10 Emissions. Southern Research Institute for the Environmental Protection Agency. April 1989.

4. *Application Guide for the Source PM₁₀ Exhaust Gas Recycle Sampling System*. EPA/600/3-88-058.

BILLING CODE 6560-50-M

4.1.4.3 Post-Test Leak-Check. A leak-check is required at the conclusion of each sampling run. Remove the cyclone before the leak-check to prevent the vacuum created by the cooling of the probe from disturbing the collected sample and use the following procedure to conduct a post-test leak-check.

4.1.4.3.1 The sample-side leak-check is performed as follows: After removing the cyclone, seal the probe with a leak-tight stopper. Before starting pump, close the coarse total valve and both recycle valves, and open completely the sample back pressure valve and the fine total valve. After turning the pump on, partially open the coarse total valve slowly to prevent a surge in the manometer. Adjust the vacuum to at least 381 mm Hg (15.0 in. Hg) with the fine total valve. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

Caution: Do not decrease the vacuum with any of the valves. This may cause a rupture of the filter.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

4.1.4.3.2 Leak rates in excess of 0.00057 m^3/min (0.020 ft^3/min) are unacceptable. If the leak rate is too high, void the sampling run.

4.1.4.3.3 To complete the leak-check, slowly remove the stopper from the nozzle until the vacuum is near zero, then immediately turn off the pump. This procedure sequence prevents a pressure surge in the manometer fluid and rupture of the filter.

4.1.4.3.4 The recycle-side leak-check is performed as follows: Close the coarse and fine total valves and sample back pressure valve. Plug the sample inlet at the meter box. Turn on the power and the pump, close the recycle valves, and open the total flow valves. Adjust the total flow fine adjust valve until a vacuum of 25 inches of mercury is achieved. If the desired vacuum is exceeded, either leak-check at this higher vacuum, or end the leak-check and start over. Minimum acceptable leak rates are the same as for the sample-side. If the leak rate is too high, void the sampling run.

4.1.5 EGR Train Operation. Same as in Method 5, Section 4.1.5, except omit references to nomographs and recommendations about changing the filter assembly during a run.

4.1.5.1 Record the data required on a data sheet such as the one shown in Figure 10 of this method. Make periodic checks of the manometer level and zero to ensure correct ΔH and Δp values. An acceptable procedure for checking the zero is to equalize the pressure at both ends of the manometer by pulling off the tubing, allowing the fluid to equilibrate and, if necessary, to re-zero. Maintain the probe temperature to within 11 °C (20 °F) of stack temperature.

4.1.5.2 The procedure for using the example EGR setup sheet is as follows: Obtain a stack velocity reading from the pitot manometer (Δp), and find this value on the ordinate axis of the setup sheet. Find the stack temperature on the abscissa. Where these two values intersect are the differential pressures necessary to achieve isokineticity and 10 μm cut size (interpolation may be necessary).

4.1.5.3 The top three numbers are differential pressures (in. H_2O), and the bottom number is the percent recycle at these flow settings. Adjust the total flow rate valves, coarse and fine, to the sample value (ΔH) on the setup sheet, and the recycle flow rate valves, coarse and fine, to the recycle flow on the setup sheet.

4.1.5.4 For startup of the EGR sample train, the following procedure is recommended. Preheat the cyclone in the stack for 30 minutes. Close both the sample and recycle coarse valves. Open the fine total, fine recycle, and sample back pressure valves halfway. Ensure that the nozzle is properly aligned with the sample stream. After noting the Δp and stack temperature, select the appropriate ΔH and recycle from the EGR setup sheet. Start the pump and timing device simultaneously. Immediately open both the coarse total and the coarse recycle valves slowly to obtain the approximate desired values. Adjust both the fine total and the fine recycle valves to achieve more precisely the desired values. In the EGR flow system, adjustment of either valve will result in a change in both total and recycle flow rates, and a slight iteration between the total and recycle valves may be necessary. Because the sample back pressure valve controls the total flow rate through the system, it may be necessary to adjust this valve in order to obtain the correct flow rate.

Note: Isokinetic sampling and proper operation of the cyclone are not achieved unless the correct ΔH and recycle flow rates are maintained.

4.1.5.5 During the test run, monitor the probe and filter temperatures periodically, and make adjustments as necessary to maintain the desired temperatures. If the sample loading is high, the filter may begin to blind or the cyclone may clog. The filter or the cyclone may be replaced during the sample run. Before changing the filter or cyclone, conduct a leak-check (Section 4.1.4.2 of this method). The total particulate mass shall be the sum of all cyclone and the filter catch during the run. Monitor stack temperature and Δp periodically, and make the necessary adjustments in sampling and recycle flow rates to maintain isokinetic sampling and the proper flow rate through the cyclone. At the end of the run, turn off the pump, close the coarse total valve, and record the final dry gas meter reading. Remove the probe from the stack, and conduct a post-test leak-check as outlined in Section 4.1.4.3 of this method.

4.1.6 Calculation of Percent Isokinetic Rate and Aerodynamic Cut Size. Calculate percent isokinetic rate and the aerodynamic cut size (D_{50}) (see Calculations, Section 6 of this method) to determine whether the test was valid or another test run should be made. If there was difficult in maintaining isokinetic rates or a D_{50} of 10 μm because of source conditions, the Administrator may be consulted for possible variance.

4.2 Sample Recovery. Allow the probe to cool. When the probe can be safely handled, wipe off all external PM adhering to the outside of the nozzle, cyclone, and nozzle attachment, and place a cap over the nozzle to prevent losing or gaining PM. Do not cap the nozzle tip tightly while the sampling train is cooling, as this action would create a vacuum in the filter holder. Disconnect the probe from the umbilical connector, and take the probe to the cleanup site. Sample recovery should be conducted in a dry indoor area or, if outside, in an area protected from wind and free of dust. Cap the ends of the impingers and carry them to the cleanup site. Inspect the components of the train prior to and during disassembly to note any abnormal conditions. Disconnect the pitot from the cyclone. Remove the cyclone from the probe. Recover the sample as follows:

4.2.1 *Container Number 1* (Filter). The recovery shall be the same as that for Container Number 1 in Method 5, Section 4.2.

4.2.2 *Container Number 2* (Cyclone or Large PM Catch). The cyclone must be disassembled and the nozzle removed in order to recover the large PM catch. Quantitatively recover the PM from the interior surfaces of the nozzle and the cyclone, excluding the "turn around" cup and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.3 *Container Number 3* (PM_{10}). Quantitatively recover the PM from all of the surfaces from cyclone exit to the front half of the in-stack filter holder, including the "turn around" cup and the interior of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.4 *Container Number 4* (Silica Gel). Same as that for Container Number 3 in Method 5, Section 4.2.

4.2.5 *Impinger Water.* Same as in Method 5, Section 4.2, under "Impinger Water."

4.3 Analysis. Same as in Method 5, Section 4.3, except handle EGR Container Numbers 1 and 2 like Container Number 1 in Method 5, EGR Container Numbers 3, 4, and 5 like Container Number 3 in Method 5, and EGR Container Number 6 like Container Number 3 in Method 5. Use Figure 11 of this method to record the weights of PM collected.

4.4 Quality Control Procedures. Same as in Method 5, Section 4.4.

5. Calibration

Maintain an accurate laboratory log of all calibrations.

5.1 Probe Nozzle. Same as in Method 5, Section 5.1.

5.2 Pitot Tube. Same as in Method 5, Section 5.2.

5.3 Meter and Flow Control Console.

5.3.1 Dry Gas Meter. Same as in Method 5, Section 5.3.

5.3.2 LFE Gauges. Calibrate the recycle, total, and inlet total LFE gauges with a manometer. Read and record flow rates at 10, 50, and 90 percent of full scale on the total and recycle pressure gauges. Read and record flow rates at 10, 20, and 30 percent of full scale on the inlet total LFE pressure gauge. Record the total and recycle readings to the nearest 0.3 mm (0.01 in.). Record the inlet total LFE readings to the nearest 3 mm (0.1 in.). Make three separate measurements at each setting and calculate the average. The maximum difference between the average pressure reading and the average manometer reading shall not exceed 1 mm (0.05 in.). If the differences exceed the limit specified, adjust or replace the pressure gauge. After each field use, check the calibration of the pressure gauges.

5.3.3 Total LFE. Same as the metering system in Method 5, Section 5.3.

5.3.4 Recycle LFE. Same as the metering system in Method 5, Section 5.3, except completely close both the coarse and fine recycle valves.

5.4 Probe Heater. Connect the probe to the meter and flow control console with the umbilical connector. Insert a thermocouple into the probe sample line approximately half the length of the probe sample line. Calibrate the probe heater at 66 °C (150 °F), 121 °C (250 °F), and 177 °C (350 °F). Turn on the power, and set the probe heater to the specified temperature. Allow the heater to equilibrate, and record the thermocouple temperature and the meter and flow control console temperature to the nearest 0.5 °C (1 °F). The two temperatures should agree within 5.5 °C (10 °F). If this agreement is not met, adjust or replace the probe heater controller.

5.5 Temperature Gauges. Connect all thermocouples, and let the meter and flow contol console equilibrate to ambient temperature. All thermocouples shall agree to within 1.1 °C (2.0 °F) with a standard mercury-in-glass thermometer. Replace defective thermocouples.

5.6 Barometer. Calibrate against a standard mercury-in-glass barometer.

5.7 Probe Cyclone and Nozzle Combinations. The probe cyclone and nozzle combinations need not be calibrated if the cyclone meets the design specifications in Figure 12 of this method and the nozzle meets the design specifications in appendix B of the *Application Guide for the Source PM₁₀ Exhaust Gas Recycle Sampling System*, EPA/600/3–88–058. This document may be obtained from Roy Huntley at (919) 541-1060. If the nozzles do not meet the design specifications, then test the cyclone and nozzle combination for conformity with the performance specifications (PS's) in Table 1 of this method. The purpose of the PS tests is to determine if the cyclone's sharpness of cut meets minimum performance criteria. If the cyclone does not meet design specifications, then, in addition to the cyclone and nozzle combination conforming to the PS's, calibrate the cyclone and determine the relationship between flow rate, gas viscosity, and gas density. Use the procedures in Section 5.7.5 of this method to conduct PS tests and the procedures in Section 5.8 of this method to calibrate the cyclone. Conduct the PS tests in a wind tunnel described in Section 5.7.1 of this method and using a particle generation system described in Section 5.7.2 of this method. Use five particle sizes and three wind velocities as listed in Table 2 of this method. Perform a minimum of three replicate measurements of collection efficiency for each of the 15 conditions listed, for a minimum of 45 measurements.

5.7.1 Wind Tunnel. Perform calibration and PS tests in a wind tunnel (or equivalent test apparatus) capable of establishing and maintaining the required gas stream velocities within 10 percent.

5.7.2 Particle Generation System. The particle generation system shall be capable of producing solid monodispersed dye particles with the mass median aerodynamic diameters specified in Table 2 of this method. The particle size distribution verification should be performed on an integrated sample obtained during the sampling period of each test. An acceptable alternative is to verify the size distribution of samples obtained before and after each test, with both samples required to meet the diameter and monodispersity requirements for an acceptable test run.

5.7.2.1 Establish the size of the solid dye particles delivered to the test section of the wind tunnel using the operating parameters of the particle generation system, and verify the size during the tests by microscopic examination of samples of the particles collected on a membrane filter. The particle size, as established by the operating parameters of the generation system, shall be within the tolerance specified in Table 2 of this method. The precision of the particle size verification technique shall be at least ±0.5 μm, and the particle size determined by the verification technique shall not differ by more than 10 percent from that established by the operating parameters of the particle generation system.

5.7.2.2 Certify the monodispersity of the particles for each test either by microscopic inspection of collected particles on filters or by other suitable monitoring techniques such as an optical particle counter followed by a multichannel pulse height analyzer. If the proportion of multiplets and satellites in an aerosol exceeds 10 percent by mass, the particle generation system is unacceptable for purposes of this test. Multiplets are particles that are agglomerated, and satellites are particles that are smaller than the specified size range.

5.7.3 Schematic Drawings. Schematic drawings of the wind tunnel and blower system and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques shall be furnished with calibration data to the reviewing agency.

5.7.4 Flow Rate Measurement. Determine the cyclone flow rates with a dry gas meter and a stopwatch, or a calibrated orifice system capable of measuring flow rates to within 2 percent.

5.7.5 Performance Specification Procedure. Establish the test particle generator operation and verify the particle size microscopically. If mondispersity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements may be made at this time.

5.7.5.1 The cyclone cut size (D_{50}) is defined as the aerodynamic diameter of a particle having a 50 percent probability of penetration. Determine the required cyclone flow rate at which D_{50} is 10 μm. A suggested procedure is to vary the cyclone flow rate while keeping a constant particle size of 10 μm. Measure the PM collected in the cyclone (m_c), exit tube (m_t), and filter (m_f). Compute the cyclone efficiency (E_c) as follows:

$$E_c = \frac{m_c}{(m_c + m_t + m_f)} \times 100$$

5.7.5.2 Perform three replicates and calculate the average cyclone efficiency as follow:

$$E_{avg} = \frac{(E_1 + E_2 + E_3)}{3}$$

where E_1, E_2 and E_3 are replicate measurements of E_c.

5.7.5.3 Calculate the standard deviation (σ) for the replicate measurements of E_c as follows:

$$\sigma = \left[\frac{(E_1{}^2 + E_2{}^2 + E_3{}^2) - \frac{(E_1 + E_2 + E_3)^2}{3}}{2} \right]^{1/2}$$

if σ exceeds 0.10, repeat the replicate runs.

5.7.5.4 Using the cyclone flow rate that produces D_{50} for 10 μm, measure the overall efficiency of the cyclone and nozzle, E_o, at the particle sizes and nominal gas velocities in Table 2 of this method using this following procedure.

5.7.5.5 Set the air velocity in the wind tunnel to one of the nominal gas velocities from Table 2 of this method. Establish isokinetic sampling conditions and the correct flow rate through the sampler (cyclone and nozzle) using recycle capacity so that the D_{50} is 10 μm. Sample long enough to obtain ±5 percent precision on the total collected mass as determined by the precision and the sensitivity of the measuring technique. Determine separately the nozzle catch (m_n), cyclone catch (m_c), cyclone exit tube catch (m_t), and collection filter catch (m_f).

5.7.5.6 Calculate the overall efficiency (E_o) as follows:

them to be erroneously measured as PM_{10}.

Drying and shrinking is not thought to be a problem. Should it be considered a problem, the tester could choose Method 201A in which there is no recycle gas.

Another commenter said that the use of recycle gas increases velocity in the cyclone which could cause friable particles to break up, becoming PM_{10}.

Prior to size classification by a PM_{10} cyclone, there is no known or suspected mechanism by which friable particles, should they exist, may break up. When particles greater than 10 μm aerodynamic size reach the cyclone wall due to their inertia, they are collected.

One commenter said that no consideration is made in either method of the gas density, gas viscosity, or of the density of the particulate matter being measured.

Gas density and viscosity are compensated for in the calculations for both PM_{10} methods. Because the aerodynamic diameter of PM_{10} emissions is used in both PM_{10} methods, determination of particle densities, volumes, or shapes is not necessary and would be redundant.

Another commenter said if the PM_{10} measurement is made downstream of an electrostatic precipitater (ESP), then the particles will carry an electric charge and the measurement of PM_{10} by these methods will be affected.

The effect of an ESP on particle sizing when using Method 201 or 201A is considered to be negligible.

There was concern by the commenters that the particulate matter may settle out inside the sample train.

The trains have been calibrated with test aerosols and the relative accuracies to each other have been established. These tests results and the operation principles of both methods have shown that "settling" is not a problem. The particles travel only 1.5 to 3 in. (nozzle length) prior to size classification by the trains.

IV. Administrative

The docket is an organized and complete file of all the information considered by EPA in the development of this rulemaking. The docket is a dynamic file since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to identify readily and locate documents so that they can effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated test method revisions and EPA responses to significant comments, the contents of the docket, except for interagency review materials, will serve as the record in case of judicial review (section 307(d)(7)(A)).

Under Executive Order 12291, EPA is required to judge whether a regulation is a "major rule" and, therefore, subject to the requirements of a regulatory impact analysis. The Agency has determined that this regulation would result in none of the adverse economic effects set forth in section 1 of the Order as grounds for finding a regulation to be a "major rule." The Agency has, therefore, concluded that this regulation is not a "major rule" under Executive Order 12291.

The Regulatory Flexibility Act (RFA) of 1980 requires the identification of potentially adverse impacts of Federal regulations upon small business entities. The Act specifically requires the completion of a RFA analysis in those instances where small business impacts are possible. Because this rulemaking imposes no adverse economic impacts, an analysis has not been conducted.

List of Subjects in 40 CFR Part 51

Administrative practice and procedure, Air pollution control, Carbon monoxide, Intergovernmental relations, Lead, Nitrogen dioxide, Ozone, Particulate matter, Reporting and recordkeeping requirements, Sulfur oxides, Volatile organic compounds.

Dated: March 22, 1990.

William K. Reilly,

Administrator.

The EPA amends title 40, chapter I, part 51 of the Code of Federal Regulations as follows:

PART 51—[AMENDED]

1. The authority citation for part 51 is revised to read as follows:

Authority: 42 U.S.C. 7401(b)(1), 7410, 7470–7479, 7501–7508, and 7601(a), unless otherwise noted.

2. Subpart K, § 51.212 is amended by adding paragraph (c) to read as follows:

§ 51.212 Testing, inspection, enforcement, and complaints.

* * * * *

(c) Enforceable test methods for each emission limit specified in the plan. As an enforceable method, States may use:

(1) Any of the appropriate methods in appendix M to this part, Recommended Test Methods for State Implementation Plans; or

(2) An alternative method following review and approval of that method by the Administrator; or

(3) Any appropriate method in appendix A to 40 CFR part 60.

3. Appendix M is added to part 51 to read as follows:

Appendix M—Recommended Test Methods for State Implementation Plans

Method 201—Determination of PM_{10} Emissions (Exhaust Gas Recycle Procedure).

Method 201A—Determination of PM_{10} Emissions (Constant Sampling Rate Procedure).

Presented herein are recommended test methods for measuring air pollutants emanating from an emission source. They are provided for States to use in their plans to meet the requirements of Subpart K—Source Surveillance.

The State may also choose to adopt other methods to meet the requirements of Subpart K of this part, subject to the normal plan review process.

The State may also meet the requirements of Subpart K of this part by adopting, again subject to the normal plan review process, any of the relevant methods in appendix A to 40 CFR part 60.

Method 201—Determination of PM_{10} Emissions

(Exhaust Gas Recycle Procedure)

1. Applicability and Principle

1.1 Applicability. This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 μm (PM_{10}) from stationary sources. The EPA recognizes that condensible emissions not collected by an in-stack method are also PM_{10}, and that emissions that contribute to ambient PM_{10} levels are the sum of condensible emissions and emissions measured by an in-stack PM_{10} method, such as this method or Method 201A. Therefore, for establishing source contributions to ambient levels of PM_{10}, such as for emission inventory purposes, EPA suggests that source PM_{10} measurement include both in-stack PM_{10} and condensible emissions. Condensible missions may be measured by an impinger analysis in combination with this method.

1.2 Principle. A gas sample is isokinetically extracted from the source. An in-stack cyclone is used to seperate PM greater than PM_{10}, and an in-stack glass fiber filter is used to collect the PM_{10}. To maintain isokinetic flow rate conditions at the tip of the probe and a constant flow rate through the cyclone, a clean, dried portion of the sample gas at stack temperature is recycled into the nozzle. The particulate mass is determined gravimetrically after removal of uncombined water.

2. Apparatus

Note: Method 5 as cited in this method refers to the method in 40 CFR part 60, appendix A.

2.1 Sampling Train. A schematic of the exhaust of the exhaust gas recycle (EGR) train is shown in Figure 1 of this method.

2.1.1 Nozzle with Recycle Attachment. Stainless steel (316 or equivalent) with a sharp tapered leading edge, and recycle attachment welded directly on the side of the nozzle (see schematic in Figure 2 of this method). The angle of the taper shall be on the outside. Use only straight sampling nozzles. "Gooseneck" or other nozzle extensions designed to turn the sample gas flow 90°, as in Method 5 are not acceptable. Locate a thermocouple in the recycle attachment to measure the temperature of the recycle gas as shown in Figure 3 of this method. The recycle attachment shall be made of stainless steel and shall be connected to the probe and nozzle with stainless steel fittings. Two nozzle sizes, e.g., 0.125 and 0.160 in., should be available to allow isokinetic sampling to be conducted over a range of flow rates. Calibrate each nozzle as described in Method 5, Section 5.1.

2.1.2 PM_{10} Sizer. Cyclone, meeting the specifications in Section 5.7 of this method.

2.1.3 Filter Holder. 63mm, stainless steel. An Andersen filter, part number SE274, has been found to be acceptable for the in-stack filter.

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.4 Pitot Tube. Same as in Method 5, Section 2.1.3. Attach the pitot to the pitot lines with stainless steel fittings and to the cyclone in a configuration similar to that shown in Figure 3 of this method. The pitot lines shall be made of heat resistant material and attached to the probe with stainless steel fittings.

2.1.5 EGR Probe. Stainless steel, 15.9-mm (⅝-in.) ID tubing with a probe liner, stainless steel 9.53-mm (⅜-in.) ID stainless steel recycle tubing, two 6.35-mm (¼-in.) ID stainless steel tubing for the pitot tube extensions, three thermocouple leads, and one power lead, all contained by stainless steel tubing with a diameter of approximately 51 mm (2.0 in.). Design considerations should include minimum weight construction materials sufficient for probe structural strength. Wrap the sample and recycle tubes with a heating tape to heat the sample and recycle gases to stack temperature.

2.1.6 Condenser. Same as in Method 5, Section 2.1.7.

2.1.7 *Umbilical Connector. Flexible tubing* with thermocouple and power leads of sufficient length to connect probe to meter and flow control console.

2.1.8 Vacuum Pump. Leak-tight, oil-less, noncontaminating, with an absolute filter, "HEPA" type, at the pump exit. A Gast Model 0522-V103 G18DX pump has been found to be satisfactory.

2.1.9 Meter and Flow Control Console. System consisting of a dry gas meter and calibrated orifice for measuring sample flow rate and capable of measuring volume to ±2 percent, calibrated laminar flow elements (LFE's) or equivalent for measuring total and sample flow rates, probe heater control, and *manometers and magnehelic gauges (as* shown in Figures 4 and 5 of this method), or equivalent. Temperatures needed for calculations include stack, recycle, probe, dry gas meter, filter, and total flow. Flow measurements include velocity head (Δp), orifice differential pressure (ΔH), total flow, recycle flow, and total back-pressure through the system.

2.1.10 Barometer. Same as in Method 5, Section 2.1.9.

2.1.11 Rubber Tubing. 6.35-mm (¼-in.) ID flexible rubber tubing.

2.2 Sample Recovery.

2.2.1 Nozzle, Cyclone, and Filter Holder Brushes. Nylon bristle brushes property sized and shaped for cleaning the nozzle, cyclone, filter holder, and probe or probe liner, with stainless steel wire shafts and handles.

2.2.2 Wash Bottles, Glass Sample Storage Containers, Petri Dishes, Graduated Cylinder and Balance, Plastic Storage Containers, and Funnels. Same as Method 5, Sections 2.2.2 through 2.2.6 and 2.2.8, respectively.

2.3 Analysis. Same as in Method 5, Section 2.3.

3. *Reagents*

The reagents used in sampling, sample recovery, and analysis are the same as that specified in Method 5, Sections 3.1, 3.2, and 3.3, respectively.

4. *Procedure*

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. Same as in Method 5, Section 4.1.1.

4.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2, except use the directions on nozzle size selection in this section. Use of the EGR method may require a minimum sampling port diameter of 0.2 m (6 in.). Also, the required maximum number of sample traverse points at any location shall be 12.

4.1.2.1 The cyclone and filter holder must be in-stack or at stack temperature during sampling. The blockage effects of the EGR sampling assembly will be *minimal if the* cross-sectional area of the sampling assembly is 3 percent or less of the cross-sectional *area of the duct and a pitot* coefficient of 0.84 may be assigned to the pitot. If the cross-sectional area of the *assembly is greater than 3 percent* of the cross-sectional area of the duct, then either determine the pitot coefficient at sampling conditions or use a standard pitot with a known coefficient in a configuration with the EGR sampling assembly such that flow disturbances are minimized.

4.1.2.2 Construct a setup of pressure drops for various Δp's and temperatures. A computer is useful for these calculations. An example of the output of the EGR setup program is shown in Figure 6 of this method, and directions on its use are in section 4.1.5.2 of this method. Computer programs, written in IBM BASIC computer language, to do these types of setup and reduction calculations for *the EGR procedure, are available through the* National Technical Information Services (NTIS), Accession number PB90-500000, 5285 *Port Royal Road, Springfield, Virginia 22161.*

4.1.2.3 The EGR setup program allows the tester to select the nozzle size based on anticipated average stack conditions and prints a setup sheet for field use. The amount of recycle through the nozzle should be between 10 and 80 percent. Inputs for the EGR setup program are stack temperature (minimum, maximum, and average), stack velocity (minimum, maximum, and average), atmospheric pressure, stack static pressure, meter box temperature, stack moisture, percent O_2, and percent CO_2 in the stack gas, pitot coefficient (C_p), orifice $\Delta H_@$, flow rate measurement calibration values (slope (m) and y-intercept (b) of the calibration curve), and the number of nozzles available and their diameters.

4.1.2.4 A less rigorous calculation for the setup sheet can be done manually using the equations on the example worksheets in Figures 7, 8, and 9 of this method, or by a Hewlett-Packard HP41 calculator using the program provided in appendix D of the EGR operators manual, entitled *Applications Guide for Source PM_{10} Exhaust Gas Recycle Sampling System*. This calculation uses an approximation of the total flow rate and agrees within 1 percent of the exact solution for pressure drops at stack temperatures from 38 to 260 °C (100 to 500 °F) and stack moisture up to 50 percent. Also, the example worksheets use a constant stack temperature in the calculation, ingoring the complicated temperature dependence from all three pressure drop equations. Errors for this at stack temperatures ±28 °C (±50 °F) of the temperature used in the setup calculations are within 5 percent for flow rate and within 5 percent for cyclone cut size.

4.1.2.5 The pressure upstream of the LFE's is assumed to be constant at 0.6 in. *Hg in the* EGR setup calculations.

4.1.2.6 The setup sheet constructed using this procedure *shall be similar to Figure 8 of* this method. Inputs needed for the calculation are the same as for the setup computer except that stack velocities are not needed.

4.1.3 Preparation of Collection Train. Same as in Method 5, Section 4.1.3, except use the *following directions to set up the* train.

4.1.3.1 Assemble the EGR sampling *device, and attach it* to probe as shown in Figure 3 of this method. If stack temperatures exceed 260 °C (500 °F), then assemble the EGR *cyclone* without the O-ring and reduce the vacuum requirement to 130 mm Hg (5.0 in. Hg) in the leak-check procedure in Section 4.1.4.3.2 of this method.

4.1.3.2 Connect the proble directly to the filter holder and condenser as in Method 5. Connect the condenser and probe to the meter and flow control console with the umbilical connector. Plug in the pump and attach pump lines to the meter and flow control console.

4.1.4 Leak-Check Procedure. The leak-check for the EGR Method consists of two parts: the sample-side and the recycle-side. The sample-side leak-check is required at the beginning of the run with the cyclone attached, and after the run with the cyclone removed. The cyclone is removed before the post-test leak-check to prevent any disturbance of the collected sample prior to analysis. The recycle-side leak-check tests the leak tight integrity of the recycle components and is required *prior to the first* test run and after each shipment.

4.1.4.1 Pretest Leak-Check. A pretest leak-check of the entire *sample-side, including* the cyclone and nozzle, is required. Use the leak-check procedure in Section 4.1.4.3 of this method to *conduct a pretest* leak-check.

4.1.4.2 Leak-Checks During Sample Run. Same as in Method 5, Section 4.1.4.1.

Moisture Determination

Volume or weight of liquid in impingers ____________ ml or g
Weight of moisture in silica gel ____________ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport ____________ ml
Final volume ____________ ml
pH of sample prior to analysis ____________
Addition of NH_4OH required? ____________
Sample extracted 2X with 75 ml $MeCl_2$? ____________

For Titration of Sulfate

Normality of NH_4OH ____________ N
Volume of sample titrated ____________ ml
Volume of titrant ____________ ml

Sample Analysis

Container number	Weight of Condensible Particulate, mg		
	Final Weight	Tare Weight	Weight Gain
4 (Inorganic) 4 & 5 (Organic)			

Total ____________
Less Blank ____________
Weight of Condensible Particulate ____________

Figure 3. Analytical data sheet.

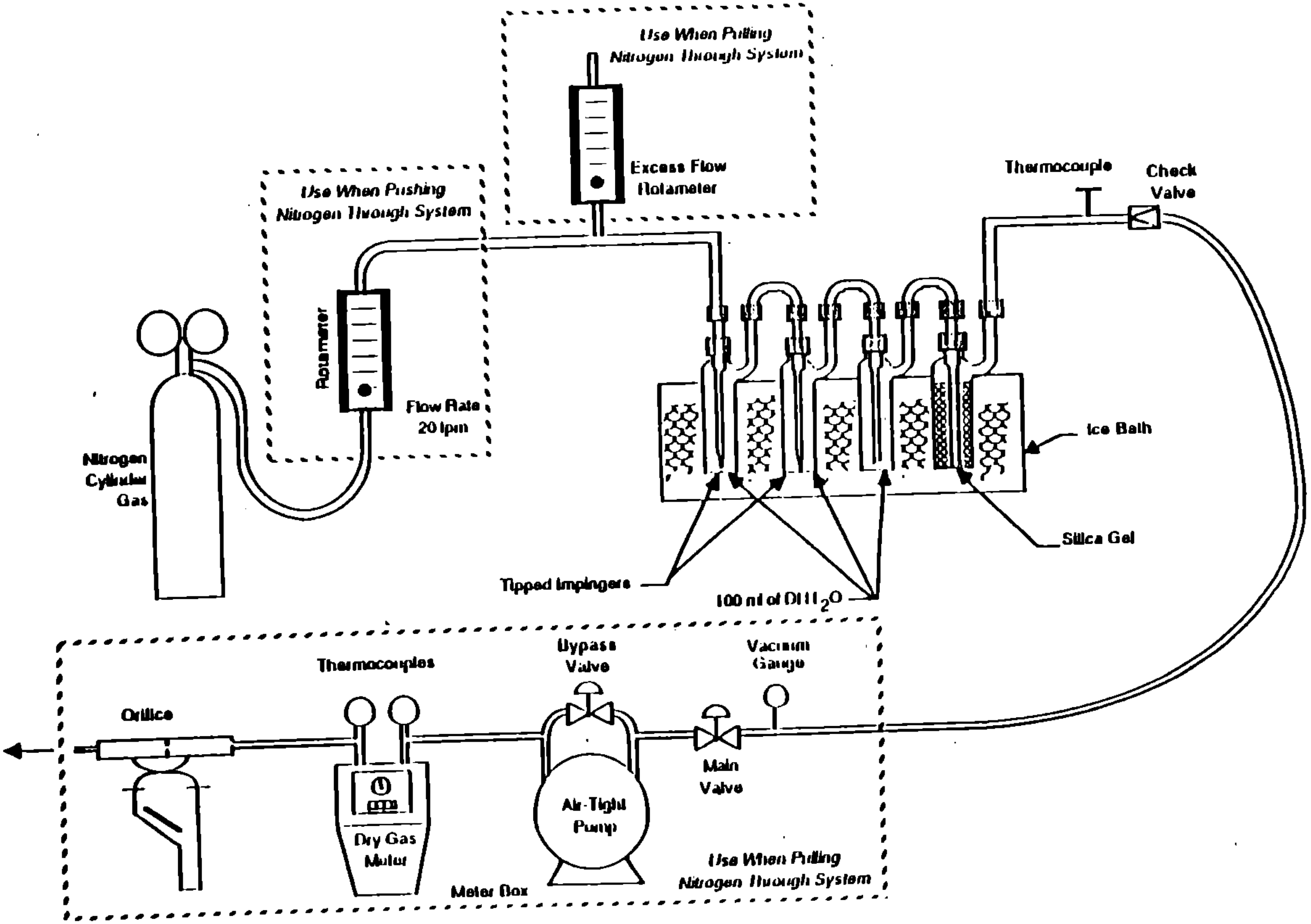

Figure 2. Schematic of post-test nitrogen purge system.

8.2 Analysis of Chlorides by IC. At the conclusion of the final weighing as described in Section 5.3.2.3, redissolve the inorganic fraction in 100 ml of water. Analyze an aliquot of the redissolved sample for chlorides by IC using techniques similar to those described in Method 5F for sulfates. Previous drying of the sample should have removed all HCl. Therefore, the remaining chlorides measured by IC can be assumed to be NH_4Cl, and this weight can be subtracted from the weight determined for CPM.

8.3 Air Purge to Remove SO_2 from Impinger Contents. As an alternative to the post-test N_2 purge described in Section 5.2.1, the tester may opt to conduct the post-test purge with air at 20 liter/min. **Note:** The use of an air purge is not as effective as a N_2 purge.

9. BIBLIOGRAPHY

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3. Texas Air Control Board, Laboratory Division. "Determination of Particulate in Stack Gases Containing Sulfuric Acid and/or Sulfur Dioxide." Laboratory Methods for Determination of Air Pollutants. Modified December 3, 1976.

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7.3 Mass of Inorganic CPM.

$$m_{rc} = m_r \left[\frac{V_{ic}}{V_{ic} - V_b}\right] - m_b + m_c \qquad \text{Eq. 2}$$

7.4 Concentration of CPM.

$$c_{cpm} = \frac{m_o + m_i}{V_{m\ std}} \qquad \text{Eq. 3}$$

8. ALTERNATIVE PROCEDURES

8.1 Determination of NH_4^- Retained in Sample by Titration.

8.1.1 An alternative procedure to determine the amount of NH_4^- added to the inorganic fraction by titration may be used. After dissolving the inorganic residue in 100 ml of water, titrate the solution with 0.1 N NH_4OH to a pH of 7.0, as indicated by a pH meter. The 0.1 N NH_4OH is made as follows: Add 7 ml of concentrated (14.8 M) NH_4OH to 1 liter of water. Standardize against standardized 0.1 N H_2SO_4 and calculate the exact normality using a procedure parallel to that described in Section 5.5 of Method 6 (Appendix A, 40 CFR Part 60). Alternatively, purchase 0.1 N NH_4OH that has been standardized against a National Institute of Standards and Technology reference material.

8.1.2 Calculate the concentration of $SO_4^=$ in the sample using the following equation.

$$C_{SO4} = \frac{48.03\ V_t\ N}{100} \qquad \text{Eq. 4}$$

where:

N = Normality of the NH_4OH, mg/ml.

V_t = Volume of NH_4OH titrant, ml.

48.03 = mg/meq.

100 = Volume of solution, ml.

8.1.3 Calculate the CPM as described in Section 7.

5.3.4 Analysis of Acetone Blank (Container No. 8). Same as in Method 5, Section 4.3.

6. CALIBRATION

Same as in Method 5, Section 5, except calibrate the IC according to the procedures in Method 5F, Section 5.

7. CALCULATIONS

Same as in Method 5, Section 6, with the following additions:

7.1 Nomenclature. Same as in Method 5, Section 6.1 with the following additions.

c_{cpm} = Concentration of the CPM in the stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

C_{SO4} = Concentration of $SO_4^=$ in the sample, mg/ml.

m_b = Sum of the mass of the water and $MeCl_2$ blanks, mg.

m_c = Mass of the NH_4^- added to sample to form ammonium sulfate, mg.

m_i = Mass of inorganic CPM matter, mg.

m_o = Mass of organic CPM, mg.

m_r = Mass of dried sample from inorganic fraction, mg.

m_{rc} = Mass of dried sample from inorganic fraction corrected for volume of aliquot taken for IC analysis, mg.

V_b = Volume of aliquot taken for IC analysis, ml.

V_{ic} = Volume of impinger contents sample, ml.

7.2 Correction for NH_4^- and H_2O. Calculate the correction factor to delete the NH_4^- retained in the sample and to add the combined water removed by the acid-base reaction based on the IC $SO_4^=$.

$$m_c = K\, C_{SO4}\, V_{ic} \qquad \text{Eq. 1}$$

where:

K = 0.020502

(Note: Do not use this aliquot to determine chlorides since the HCl will be evaporated during the first drying step; Section 8.2 details a procedure for this analysis.)

5.3.2.1 **Extraction.** Separate the organic fraction of the sample by adding the contents of Container No. 5 ($MeCl_2$) to the contents of Container No. 4 in a 1000-ml separatory funnel. After mixing, allow the aqueous and organic phases to fully separate, and drain off most of the organic/$MeCl_2$ phase. Then add 75 ml of $MeCl_2$ to the funnel, mix well, and drain off the lower organic phase. Repeat with another 75 ml of $MeCl_2$. This extraction should yield about 250 ml of organic extract. Each time, leave a small amount of the organic/$MeCl_2$ phase in the separatory funnel ensuring that no water is collected in the organic phase. Place the organic extract in a tared 350-ml weighing tin.

5.3.2.2 **Organic Fraction Weight Determination (Organic Phase from Container Nos. 4 and 5).** Evaporate the organic extract at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg.

5.3.2.3 **Inorganic Fraction Weight Determination.** Using a hot plate, or equivalent, evaporate the aqueous phase to approximately 50 ml; then evaporate to dryness in a 105°C oven. Redissolve the residue in 100 ml of water. Add five drops of phenolphthalein to this solution, then add concentrated (14.8 M) NH_4OH until the sample turns pink. Any excess NH_4OH will be evaporated during the drying step. Evaporate the sample to dryness in a 105°C oven, desiccate the sample for 24 hours, weigh to a constant weight, and record the results to the nearest 0.1 mg. (Note: The addition of NH_4OH is recommended, but is optional when no or little SO_2 is present in the gas stream, i.e., when the pH of the impinger solution is greater than 4.5, the addition of NH_4OH is not necessary.)

5.3.2.4 **Analysis of Sulfate by IC to Determine Ammonium Ion (NH_4^+) Retained in the Sample. (Note: If NH_4OH is not added, omit this step.)** Determine the amount of sulfate in the aliquot taken from Container No. 4 earlier as described in Method 5F (Appendix A, 40 CFR Part 60). Based on the IC $SO_4^=$ analysis of the aliquot, calculate the correction factor to delete the NH_4^+ retained in the sample and to add the combined water removed by the acid-base reaction (see Section 7.2).

5.3.3 Analysis of Water and $MeCl_2$ Blanks (Container Nos. 6 and 7). Analyze these sample blanks as described above in Sections 5.3.2.3 and 5.3.2.2, respectively. The sum of the values for the water blank and the $MeCl_2$ blank must be less than 2 mg or 5 percent of the mass of the CPM ($m_i + m_r$), whichever is greater. If the sum of the actual blank values is greater, then subtract 2 mg or 5 percent of the mass of the CPM, whichever is greater.

is operating at greater than ambient pressure and prevents that possibility of passing ambient air (rather than N_2) through the impingers. Continue the purge under these conditions for 1 hour, checking the rotameter and ΔH value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

5.2.2 Sample Handling.

5.2.2.1 Container Nos. 1, 2, and 3. If filter catch is to be determined, as detailed in Method 5, Section 4.2.

5.2.2.2 Container No. 4 (Impinger Contents). Measure the liquid in the first three impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas. Quantitatively transfer this liquid into a clean sample bottle (glass or plastic); rinse each impinger and the connecting glassware, including probe extension, twice with water, recover the rinse water and add it to the same sample bottle. Mark the liquid level on the bottle.

5.2.2.3 Container No. 5 ($MeCl_2$ Rinse). Follow the water rinses of each impinger and the connecting glassware, including the probe extension with two rinses of $MeCl_2$; save the rinse products in a clean, glass sample jar. Mark the liquid level on the jar.

5.2.2.4 Container No. 6 (Water Blank). Once during each field test, place 500 ml of water in a separate sample container.

5.2.2.5 Container No. 7 ($MeCl_2$ Blank). Once during each field test, place in a separate glass sample jar a volume of $MeCl_2$ approximately equivalent to the volume used to conduct the $MeCl_2$ rinse of the impingers.

5.2.2.6 Container No. 8 (Acetone Blank). As described in Method 5, Section 4.2.

5.3 Analysis. Record the data required on a sheet such as the one shown in Figure 3. Handle each sample container as follows:

5.3.1 Container Nos. 1, 2, and 3. If filter catch is analyzed, as detailed in Method 5, Section 4.3.

5.3.2 Container Nos. 4 and 5. Note the level of liquid in the containers and confirm on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container No. 4 either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Remove a 5-ml aliquot and set aside for later ion chromatographic (IC) analysis of sulfates.

4.2.1 N_2 Gas. N_2 gas at delivery pressures high enough to provide a flow of 20 liters/min for 1 hour through the sampling train.

4.2.2 Methylene Chloride.

4.2.3 Water. Same as in Section 4.1.

4.3 Analysis. Same as in Method 5, Section 3.3, with the following additions:

4.3.1 Methylene Chloride.

4.3.2 Ammonium Hydroxide. Concentrated (14.8 M) NH_4OH.

4.3.3 Water. Same as in Section 4.1.

4.3.4 Phenolphthalein. The pH indicator solution, ~~1.0~~ .05 percent in ~~60~~ 50 percent alcohol.

5. PROCEDURE

5.1 Sampling. Same as in Method 5, Section 4.1, with the following exceptions:

5.1.1 Place 100 ml of water in the first three impingers.

5.1.2 The use of silicone grease in train assembly is not recommended. Teflon tape or similar means may be used to provide leak-free connections between glassware.

5.2 Sample Recovery. Same as in Method 17, Section 4.2 with the addition of a post-test N_2 purge and specific changes in handling of individual samples as described below.

5.2.1 Post-test N_2 Purge for Sources Emitting SO_2. (Note: This step is recommended, but is optional. When no or little SO_2 is present in the gas stream, i.e., the pH of the impinger solution is greater than 4.5, purging has been found to be unnecessary.) As soon as possible after the post-test leak check, detach the probe and filter from the impinger train. Leave the ice in the impinger box to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature below 20°C. With no flow of gas through the clean purge line and fittings, attach it to the input of the impinger train (see Figure 2). To avoid over- or under-pressurizing the impinger array, slowly commence the N_2 gas flow through the line while simultaneously opening the meter box pump valve(s). Adjust the pump bypass and N_2 delivery rates to obtain the following conditions: (1) 20 liters/min or $\Delta H_@$ and (2) an overflow rate through the rotameter of less than 2 liters/min. Condition (2) guarantees that the N_2 delivery system

3.1.2 A Teflon filter support shall be used.

3.1.3 Both the first and second impingers shall be of the Greenburg-Smith design with the standard tip.

3.1.4 All sampling train glassware shall be cleaned prior to the test with soap and tap water, water, and rinsed using tap water, water, acetone, and finally, $MeCl_2$. It is important to remove completely all silicone grease from areas that will be exposed to the $MeCl_2$ during sample recovery.

3.2 Sample Recovery. Same as in Method 5, Section 2.2, with the following additions:

3.2.1 N_2 Purge Line. Inert tubing and fittings capable of delivering 0 to 28 liters/min of N_2 gas to the impinger train from a standard gas cylinder (see Figure 2). Standard 0.95 cm (3/8-inch) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

3.2.2 Rotameter. Capable of measuring gas flow at 20 liters/min.

3.3 Analysis. The following equipment is necessary in addition to that listed in Method 5, Section 2.3:

3.3.1 Separatory Funnel. Glass, 1-liter.

3.3.2 Weighing Tins. 350-ml.

3.3.3 Drying Equipment. Hot plate and oven with temperature control.

3.3.4 ~~Burette. 5-ml size with 0.01-ml graduations~~.

3.3.5 Pipets. 5-ml.

3.3.6 Ion Chromatograph. Same as in Method 5F, Section 2.1.6.

4. REAGENTS

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

4.1 Sampling. Same as in Method 5, Section 3.1, with the addition of deionized distilled water to conform to the American Society for Testing and Materials Specification D 1193-74, Type II.

4.2 Sample Recovery. Same as in Method 5, Section 3.2, with the following additions:

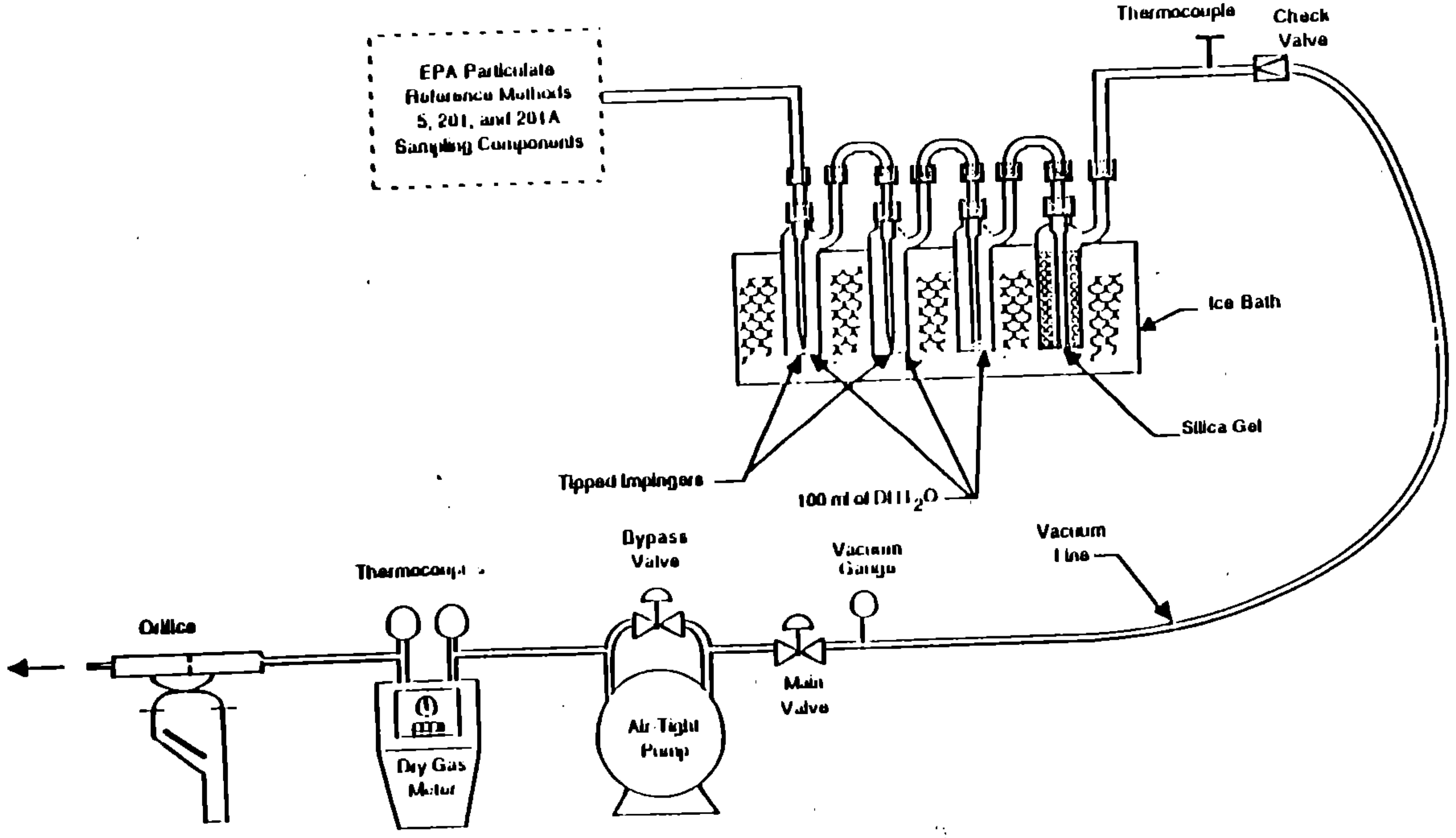

Figure 1. Schematic of condensible particulate sampling train.

EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER
CONDITIONAL TEST METHOD

Determination Of Condensible Emissions From Stationary Sources

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method applies to the determination of condensible particulate matter (CPM) emissions from stationary sources. It is intended to represent condensible matter as material that condenses after passing through an in-stack filter (Note: The filter catch can be analyzed according to Method 17 procedures). This method may be used in conjunction with Method 201 or 201A if the probes are glass lined. This method may also be modified to measure material that condenses at other temperatures by specifying the filter temperature.

1.2 Principle. The CPM is collected in the impinger portion of a Method 17 (Appendix A, 40 CFR Part 60) type sampling train. The impinger contents are immediately purged after the run with nitrogen (N_2) to remove dissolved sulfur dioxide (SO_2) gases from the impinger contents. The impinger solution is then extracted with methylene chloride ($MeCl_2$). The organic and aqueous fractions are then taken to dryness and the residues weighed. The total of both fractions represents the CPM.

2. PRECISION AND INTERFERENCE

2.1 Precision. The precisions based on method development tests at a wood waste burner and two coal-fired boilers are 13.0 ± 2.1 mg/m^3, 3.5 ± 1.1 mg/m^3, and 39.5 ± 9.0 mg/m^3, respectively.

2.2 Interference. Ammonia (e.g., in sources that use ammonia injection as a control technique) interferes by reacting with the hydrogen chloride (HCl) in the gas stream to form ammonium chloride (NH_4Cl) which would be measured as CPM. The sample may be analyzed for chloride and the equivalent amount of NH_4Cl can be subtracted from the CPM weight.

3. APPARATUS

3.1 Sampling Train. Same as in Method 17, Section 2.1, with the following exceptions noted below (see Figure 1). Note: Mention of trade names or specific products does not constitute endorsement by EPA.

3.1.1 The probe extension shall be glass-lined.

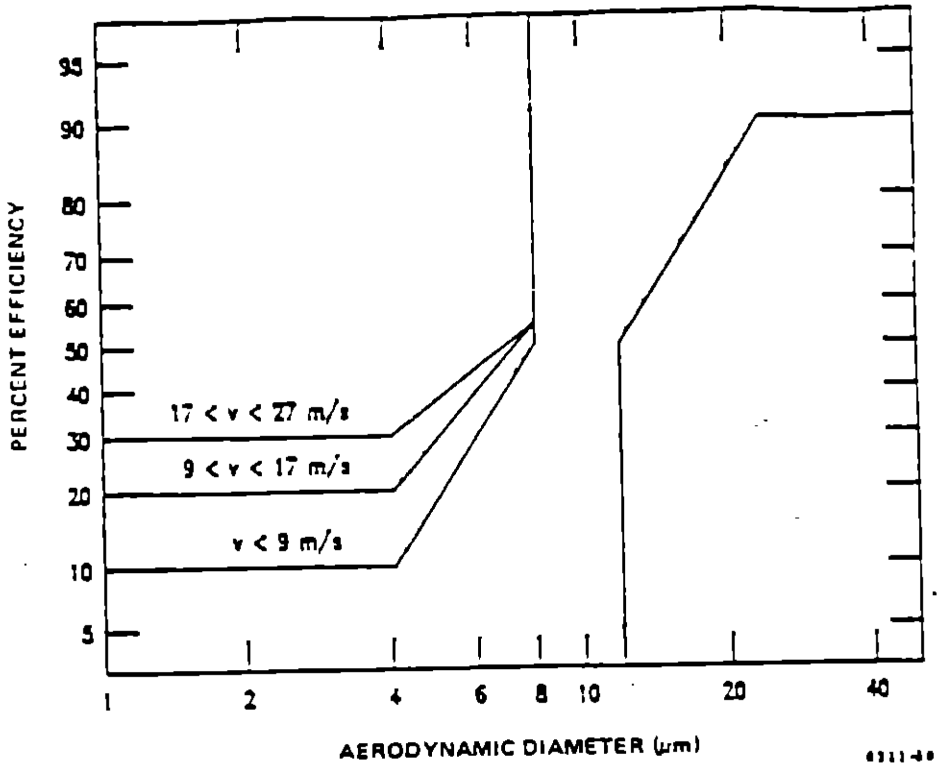

Figure 8. Efficiency envelope for the PM_{10} cyclone.

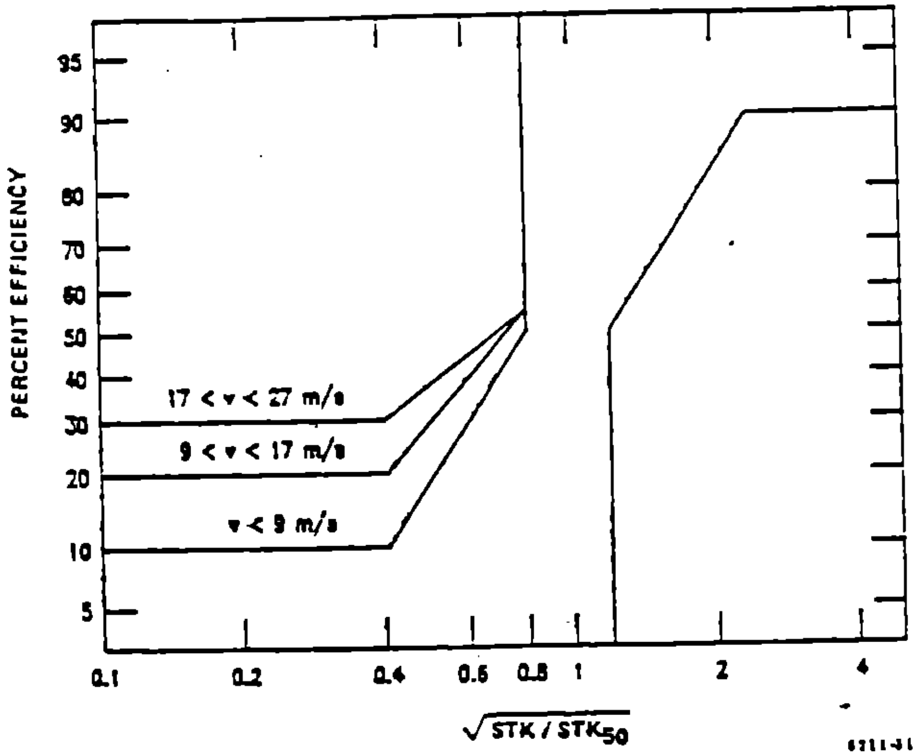

Figure 9. Efficiency envelope for first calibration stage.

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TABLE 2.—PARTICLE SIZES AND NOMINAL GAS VELOCITIES FOR EFFICIENCY

Particle size (μm)[a]	Target gas velocities (m/sec)		
	7 ± 1.0	15 ± 1.5	25 ± 2.5
5 ± 0.5			
7 ± 0.5			
10 ± 0.5			
14 ± 1.0			
20 ± 1.0			

[a] Mass median aerodynamic diameter.

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$$\Delta p_{max} = 1.3686 \times 10^{-4} \frac{P_s M_s (v_{max})^2}{(t_s + 460) C_p^2} = \_\_\_\_\_\_ \text{ in. } H_2O$$

Nozzle No.				
D_n, in.				
v_n, ft/sec				
v_{min}, ft/sec				
v_{max}, ft/sec				
Δp_{min}, in. H_2O				
Δp_{max}, in. H_2O				

Velocity traverse data:

$$\Delta p(\text{Method 201A}) = \Delta p(\text{Method 2}) \left[\frac{C_p}{C_p'} \right]^2$$

Total run time, minutes = ______

Number of traverse points = ______

$$t_1 = \left[\frac{\Delta p'_1}{\Delta p'_{avg}} \right]^{1/2} \frac{\text{(Total run time)}}{\text{(Number of points)}}$$

where:

t_1 = dwell time at first traverse point, minutes.

$\Delta p'_1$ = the velocity head at the first traverse point (from a previous traverse), in. H_2O.

$\Delta p'_{avg}$ = the square of the average square root of the Δp's (from a previous velocity traverse), in. H_2O.

At subsequent traverse points, measure the velocity Δp and calculate the dwell-time by using the following equation:

$$t_n = \frac{t_1}{(\Delta p_n)^{1/2}} (\Delta p_1)^{1/2},\ n = 2,3, \ldots \text{ total number of sampling points}$$

where:

t_n = dwell time at traverse point n, minutes.

Δp_n = measured velocity head at point n, in. H_2O.

Δp_1 = dwell time at first traverse point, minutes.

Figure 6. Example worksheet 3, dwell time.

Point No.	Port							
	Δp	t	Δp	t	Δp	t	Δp	t
1								
2								
3								
4								
5								
6								

Plant ______

Date ______

Run no. ______

Filter no. ______

Amount of liquid lost during transport ______

Acetone blank volume, ml ______

Acetone wash volume, ml (4) ______ (5) ______

Acetone blank conc., mg/mg (Equation 5-4, Method 5) ______

Acetone wash blank, mg (Equation 5-5, Method 5) ______

Container No.	Weight of PM_{10} (mg)		
	Final weight	Tare weight	Weight gain
1			
3			
Total			
Less acetone blank			
Weight of PM_{10}			

Figure 7. Method 201A analysis sheet.

TABLE 1.—PERFORMANCE SPECIFICATIONS FOR SOURCE PM_{10} CYCLONES AND NOZZLE COMBINATIONS

Parameter	Units	Specifications
1. Collection efficiency.	Percent	Such that collection efficiency falls within envelope specified by Section 5.2.6 and Figure 8.
2. Cyclone cut size (D_{50}).	μm	10 ± 1 μm aerodynamic diameter.

Barometric pressure,
P_{bar}, in. Hg = ________
Stack static pressure,
P_g, in. H_2O = ________
Average stack temperature,
t_s, °F = ________
Meter temperature, t_m, °F = ________
Orifice $\Delta H_@$, in. H_2O = ________
Gas analysis:
$\%CO_2$ = ________
$\%O_2$ = ________
$\%N_2 + \%CO$ = ________
Fraction moisture content,
B_{ws} = ________

Molecular weight of stack gas, dry basis:

$M_d = 0.44\ (\%CO_2) + 0.32\ (\%O_2) + 0.28\ (\%N_2 + \%CO)$ = ________ lb/lb mole

Molecular weight of stack gas, wet basis:

$M_w = M_d\ (1-B_{ws}) + 18\ (B_{ws})$ = ________ lb/lb mole

Absolute stack pressure:

$$P_s = P_{bar} + \frac{P_g}{13.6} = \_\_\_\_\_\_\_\_ \text{ in. Hg}$$

Viscosity of stack gas:

$\mu_s = 152.418 + 0.2552\ t_s + 3.2355 \times 10^{-5}\ t_s^2 + 0.53147\ (\%O_2) - 74.143\ B_{ws}$ = ________ micropoise

Cyclone flow rate:

$$Q_s = 0.002837\ \mu_s \left[\frac{(t_s - 460)}{M_w\ P_s} \right]^{0.2949} = \_\_\_\_\_\_\_\_ \text{ ft}^3\text{/min}$$

Figure 4. Example worksheet 1, cyclone flow rate and ΔH.

Orifice pressure head (ΔH) needed for cyclone flow rate:

$$\Delta H = \left[\frac{Q_s\ (1-B_{ws})\ P_s}{t_s + 460} \right]^2 \frac{t_m\ M_s\ 1.083\ \Delta H_@}{P_{bar}} = \_\_\_\_\_\_\_\_ \text{ in. } H_2O$$

Calculate ΔH for three temperatures:

t_s, °F			
ΔH, in. H_2O			

Stack viscosity, μ_s,
micropoise = ________
Absolute stack pressure,
P_s, in. Hg = ________
Average stack temperature,
t_s, °F = ________
Meter temperature, t_m, °F = ________
[illegible] A pitot coefficient,
C ________
Cyclone flow rate, ft³/min,
Q_s = ________
Method 2 pitot coefficient,
C_p' = ________
Molecular weight of stack gas, wet basis,
M_w = ________
Nozzle diameter, D_n, in. = ________

Nozzle velocity:

$$v_n = \frac{3.056\ Q_s}{D_n^2} = \_\_\_\_\_\_\_\_ \text{ ft/sec}$$

Maximum and minimum velocities:

$$v_{min} = v_n \left[0.2457 + \left[0.3072 - \frac{0.2603\ Q_s^{1/2}\ \mu_s}{v_n^{1.5}} \right]^{1/2} \right] = \_\_\_\_\_\_\_\_ \text{ ft/sec}$$

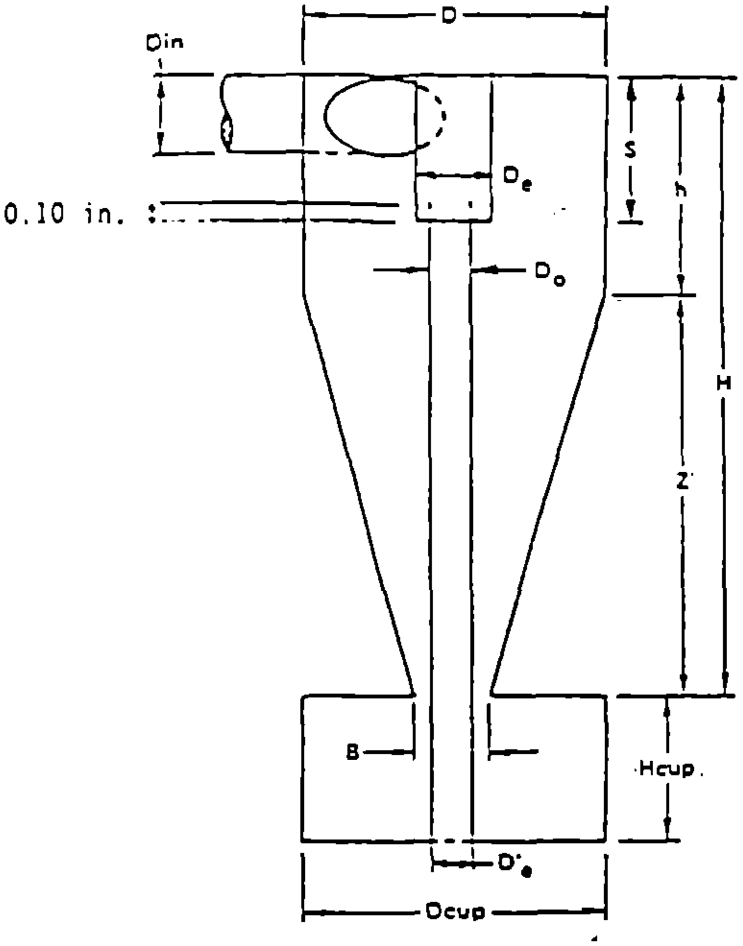
$$v_{max} = v_n \left[0.4457 + \left[0.5690 - \frac{0.2603\ Q_s^{1/2}\ \mu_s}{v_n^{1.5}} \right]^{1/2} \right] = \_\_\_\_\_\_\_\_ \text{ ft/sec}$$

Figure 5. Example worksheet 2, nozzle selection.

Maximum and minimum velocity head values:

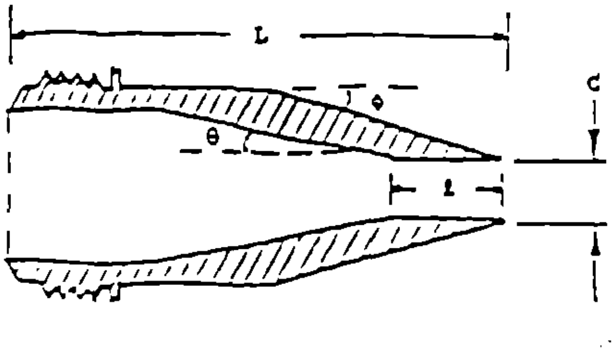
$$\Delta p_{min} = 1.3686 \times 10^{-4} \frac{P_s\ M_w\ (v_{min})^2}{(t_s + 460)\ C_p^2} = \_\_\_\_\_\_\_\_ \text{ in. } H_2O$$

Cyclone Interior Dimensions



	Dimensions (±0.02 cm, ±0.01 in.)											
	Din	D	De	B	H	h	Z	S	Hcup	Dcup	D'_e	D_o
cm	1.27	4.47	1.50	1.88	6.95	2.24	4.71	1.57	2.25	4.45	1.02	1.24
inches	0.50	1.76	0.59	0.74	2.74	0.88	1.85	0.62	0.89	1.75	0.40	0.49

Figure 3. Cyclone design specifications.



Nozzle Diameter (inches)	Cone Angle, θ (degrees)	Outside taper, ϕ (degrees)	Straight inlet length, ℓ (inches)	Total Length L (inches)
0.136	4	15	<0.05	2.653±0.05
0.150	4	15	<0.05	2.553±0.05
0.164	5	15	<0.05	1.970±0.05
0.180	6	15	<0.05	1.572±0.05
0.197	6	15	<0.05	1.491±0.05
0.215	6	15	<0.05	1.45 ±0.05
0.233	6	15	<0.05	1.45 ±0.05
0.264	5	15	<0.05	1.45 ±0.05
0.300	4	15	<0.05	1.48 ±0.05
0.342	4	15	<0.05	1.45 ±0.05
0.390	3	15	<0.05	1.45 ±0.05

Figure 2. Nozzle design specifications.

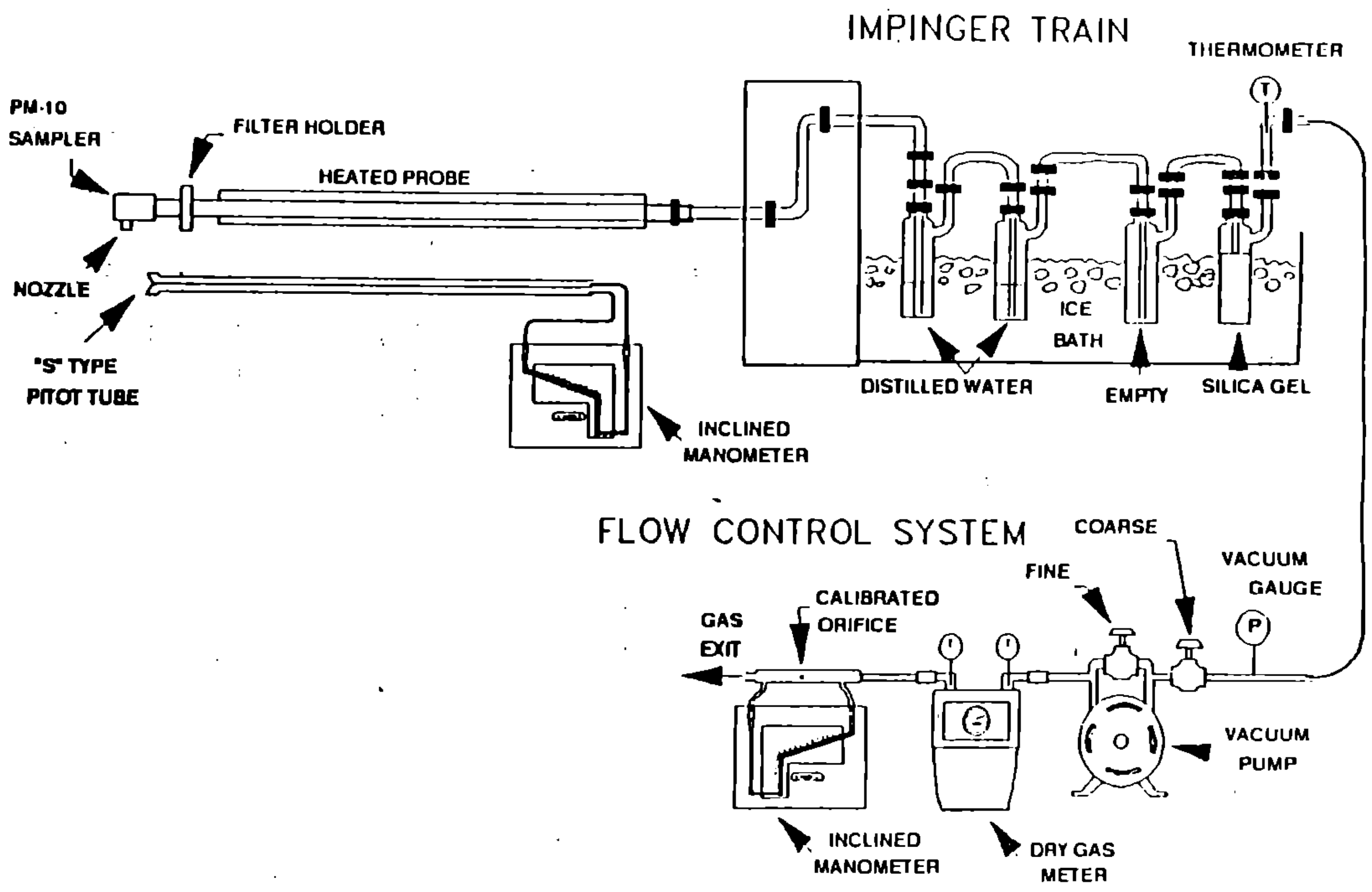


Figure 1. CSR Sampling Train

Moisture Determination

Volume or weight of liquid in impingers ____________ ml or g
Weight of moisture in silica gel ____________ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport ____________ ml
Final volume ____________ ml
pH of sample prior to analysis ____________
Addition of NH_4OH required? ____________
Sample extracted 2X with 75 ml $MeCl_2$? ____________

For Titration of Sulfate

Normality of NH_4OH ____________ N
Volume of sample titrated ____________ ml
Volume of titrant ____________ ml

Sample Analysis

Container number	Weight of Condensible Particulate, mg		
	Final Weight	Tare Weight	Weight Gain
4 (Inorganic)			
4 & 5 (Organic)			

Total ____________
Less Blank ____________
Weight of Condensible Particulate ____________

Figure 3. Analytical data sheet.

APPENDIX J.3

ALDEHYDES

Formaldehyde, acetone, and 2,4-dinitroaniline contamination of the aqueous acidic 2,4-dinitrophenyl-hydrazine (DNPH) reagent is frequently encountered. The reagent must be prepared within five days of use in the field and must be stored in an uncontaminated environment both before and after sampling in order to minimize blank problems. Some concentration of acetone contamination is unavoidable, because acetone is ubiquitous in laboratory and field operations. However, the acetone contamination must be minimized.

3.5.4 Apparatus and Materials

3.5.4.1 A schematic of the sampling train is shown in Figure 3.5-1. This sampling train configuration is adapted from EPA Method 4 procedures. The sampling train consists of the following components: Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment.

3.5.4.1.1 **Probe Nozzle:** Quartz or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant inner diameter. The nozzle shall be buttonhook or elbow design. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.15 cm(1/16 in), e.g., 0.32 to 1.27 cm (1/8 to 1/2 in), of larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Section 3.5.8.1

3.5.4.1.2 **Probe Liner:** Borosilicate glass or quartz shall be used for the probe liner. The tester should not allow the temperature in the probe to exceed 120 ± 14°C (248 ± 25°F).

3.5.4.1.3 **Pitot Tube:** The Pitot tube shall be Type S, as described in Section 2.1 of EPA Method 2, or any other appropriate device. The pitot tube shall be attached to the probe to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plan (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.

3.5 Sampling for Aldehyde and Ketone Emissions from Stationary Sources (Method 0011)

3.5.1 Scope and Application

This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of formaldehyde, CAS Registry number 50-00-0, and possibly other aldehydes and ketones from stationary sources as specified in the regulations. The methodology has been applied specifically to formaldehyde however, many laboratories have extended the application to other aldehydes and ketones. Compounds derivatized with 2,4-dinitrophenyl-hydrazine can be detected as low as 6.4 x 10^{-8} lbs/cu ft (1.8 ppbv) in stack gas over a 1 hr sampling period, sampling approximately 45 cu ft.

3.5.2 Summary of Method

3.5.2.1 Gaseous and particulate pollutants are withdrawn isokinetically from an emission source and are collected in aqueous acidic 2,4-dinitrophenyl-hydrazine. Formaldehyde present in the emissions reacts with the 2,4-dinitrophenyl-hydrazine to form the formaldehyde dinitrophenylhydrazone derivative. The dinitrophenylhydrazone derivative is extracted, solvent-exchanged, concentrated, and then analyzed by high performance liquid chromatography.

3.5.3 Interferences

3.5.3.1 A decomposition product of 2,4-dinitrophenyl-hydrazine, 2,4-dinitroaniline, can be an analytical interferant if concentrations are high. 2,4-Dinitroaniline can coelute with 2,4-dinitrophenylhydrazone of formaldehyde under high performance liquid chromatography conditions, which may be used for the analysis. High concentrations of highly-oxygenated compounds, especially acetone, that have the same retention time or nearly the same retention time as the dinitrophenylhydrazone of formaldehyde, and that also absorb at 360 nm, will interfere with the analysis.

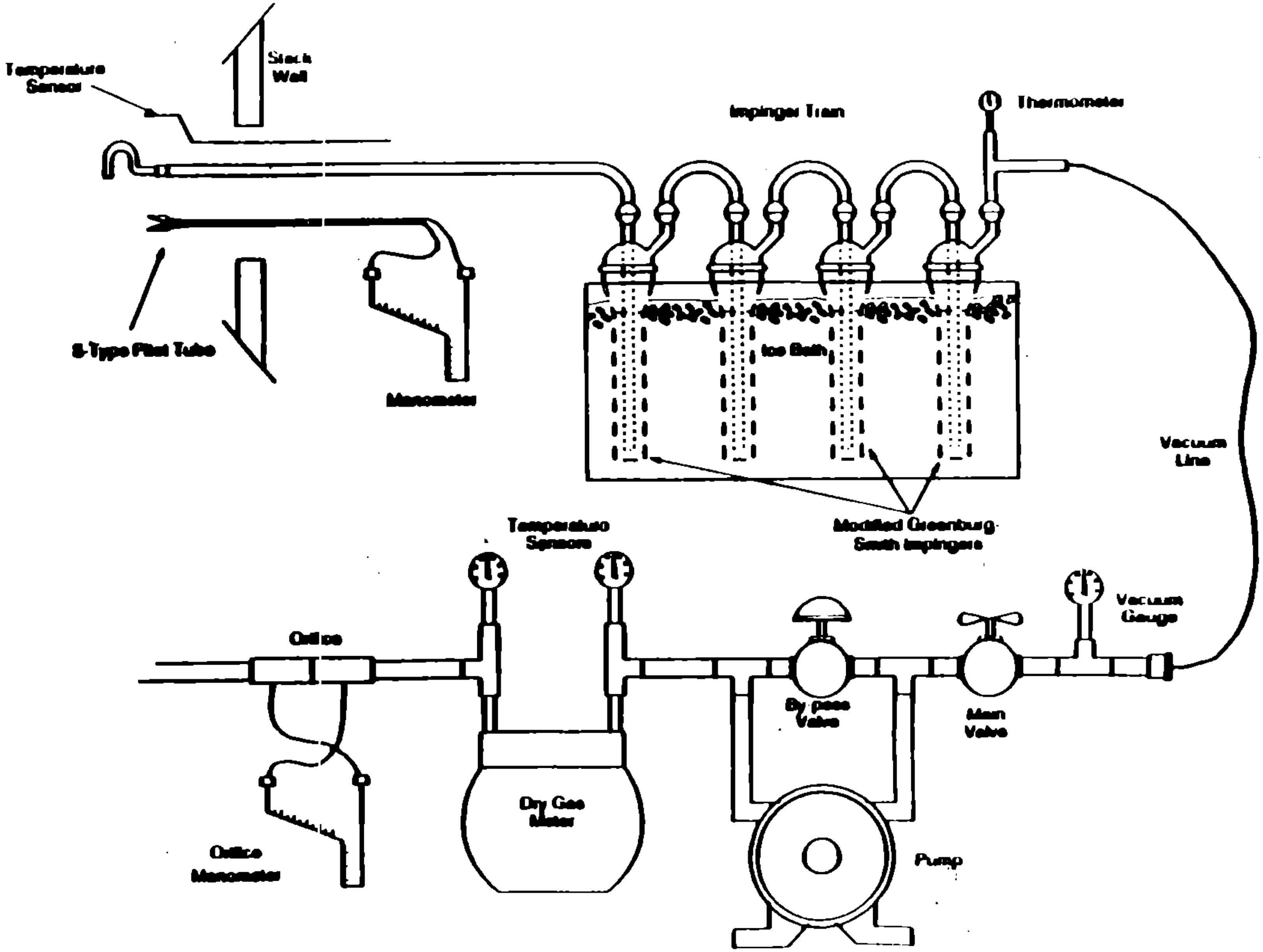
3.5.4.1.4 Differential Pressure Gauge: The differential pressure gauge shall be an inclined manometer or equivalent device as described in Section 2.2 of EPA Method 2. One manometer shall be used for velocity-head reading and the other for orifice differential pressure readings.

3.5.4.1.5 Impingers: The sampling train requires a minimum of four impingers, connected as shown in Figure 3.5-1, with ground glass (or equivalent) vacuum-tight fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm inside diameter (1/2 in) glass tube extending to 1.3 cm (1/2 in) from the bottom of the flask. For the second impinger, use a Greenburg-Smith impinger with the standard tip. Place a thermometer capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger for monitoring purposes.

3.5.4.1.6 Metering System: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment as shown in Figure 3.5-1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems may be used which are capable of maintaining sample volumes to within 2%. The metering system may be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates.

3.5.4.1.7 Barometer: The barometer may be mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service Station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in Hg) per 30 m (100 ft) elevation increases (vice versa for elevation decrease).

3.5.4.1.8 Gas Density Determination Equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.3 of EPA Method 2), and



Formaldehyde Sampling Train

Figure 3.5-4

3.5.4.2.5 Rubber Policeman and Funnel: A rubber policeman and funnel are required to aid in the transfer of material into and out of containers in the field.

3.5.4.3 Reagent Preparation

3.5.4.3.1 Bottles/Caps: Amber 1- or 4-L bottles with Teflon-lined caps are required for storing cleaned DNPH solution. Additional 4-L bottles are required to collect waste organic solvents.

3.5.4.3.2 Large Glass Container: At least one large glass (8 to 16 L) is required for mixing the aqueous acidic DNPH solution.

3.5.4.3.3 Stir Plate/Large Stir Bars/Stir Bar Retriever: a magnetic stir plate and large stir bar are required for the mixing of aqueous acidic DNPH solution. A stir bar retriever is needed for removing the stir bar from the large container holding the DNPH solution.

3.5.4.3.4 Buchner Filter/Filter Flask/Filter Paper: A large filter flask (2-4 L) with a buchner filter, appropriate rubber stopper, filter paper and connecting tubing are required for filtering the aqueous acidic DNPH solution prior to cleaning.

3.5.4.3.5 Separatory Funnel: At least one large separatory funnel (2 L) is required for cleaning the DNPH prior to use.

3.5.4.3.6 Beakers: Beakers (150 ml, 250 ml, and 400 ml) are useful for holding/measuring organic liquids when cleaning the aqueous acidic DNPH solution and for weighing DNPH crystals.

3.5.4.3.7 Funnels: At lease one large funnel is needed for pouring the aqueous acidic DNPH into the separator funnel.

gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot openings (see EPA Method 2, Figure 2-7). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

3.5.4.2 Sample Recovery

3.5.4.2.1 **Probe Liner:** Probe nozzle and brushes; Teflon bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel, Teflon, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the impingers.

3.5.4.2.2 **Wash Bottles:** Three wash bottles are required. Teflon or glass wash bottles are recommended; polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

3.5.4.2.3 **Graduate Cylinder and/or Balance:** A graduated cylinder or balance is required to measure condensed water to the nearest 1 ml or 1 g. Graduated cylinders shall have division not >2 ml. Laboratory balances capable of weighing to ±0.5 g are required.

3.5.4.2.4 **Amber Glass Storage Containers:** One-liter wide-mouth amber flint glass bottles with Teflon-lined caps are required to store impinger water samples. The bottles must be sealed with Teflon tape.

3.5.5.4 2,4-dinitrophenylhydrazine (DNPH), $[2,4\text{-}(O_2N)_2C_6H_3]NHNH_2$ - The quantity of water may vary from 10 to 30%.

3.5.5.4.1 The 2,4-dinitrophenylhydrazine reagent must be prepared in the laboratory within five days of sampling use in the field. Preparation of DNPH can also be done in the field, with consideration of appropriate procedures required for safe handling of solvent in the field. When a container of prepared DNPH reagent is opened in the field, the contents of the opened container should be used within 48 hours. All laboratory glassware must be washed with detergent and water and rinsed with water, methanol, and methylene chloride prior to use.

NOTE: DNPH crystals or DNPH solution should be handled with plastic gloves at all times with prompt and extensive use of running water in case of skin exposure

3.5.5.4.2 Preparation of Aqueous Acidic DNPH Derivatizing Reagent: Each batch of DNPH reagent should be prepared and purified within five days of sampling, according to the procedures described below.

NOTE: Reagent bottles for storage of cleaned DNPH derivatizing solution must be rinsed with acetonitrile and dried before use. Baked glassware is not essential for preparation of DNPH reagent. The glassware must not be rinsed with acetone or an unacceptable concentration of acetone contamination will be introduced. If field preparation of DNPH is performed, caution must be exercised in avoiding acetone contamination.

3.5.5.4.2.1 Place an 8 L container under a fume hood on a magnetic stirrer. Add a large stir bar and fill the container half full of organic-free reagent water. Save the empty bottle from the organic-free reagent water. Start the stirring bar and adjust the stir rate to be as fast as possible. Using a graduated cylinder, measure 1.4 ml of concentrated hydrochloric acid. Slowly pour the acid into the stirring water. Fumes may be generated and the water may become warm. Weight the DNPH crystals on a

3.5.4.3.8 Graduated Cylinders: At least one large graduated cylinder (1 to 2 L) is required for measuring organic-free reagent water and acid when preparing the DNPH solution.

3.5.4.3.9 Top-Loading Balance: A one-place top loading balance is needed for weighing out the DNPH crystals used to prepare the aqueous acidic DNPH solution.

3.5.4.3.10 Spatulas: Spatulas are needed for weighing out DNPH when preparing the aqueous DNPH solution.

3.5.4.4 Crushed Ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending upon ambient temperature. Samples which have been taken must be stored and shipped cold; sufficient ice for this purpose must be allowed.

3.5.5 Reagents

3.5.5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.5.5.2 Organic-free reagent water: All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

3.5.5.3 Silica Gel: Silica gel shall be indicating type, 6-16 mesh. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

Table 3.5-1

APPROXMIATE AMOUNT OF CRYSTALLINE DNPH USED TO PREPARE A SATURATED SOLUTION

Amount of Moisture in DNPH	Weight Required per 8 L of Solution
10 weight percent	31 g
15 weight percent	33 g
30 weight percent	40 g

Table 3.5-2

INSTRUMENT DETECTION LIMITS AND REAGENT CAPACITY FOR FORMALDEHYDE ANALYSIS[1]

Analyte	Detection Limit, ppbv[2]	Reagent Capacity, ppmv
Formaldehyde	1.8	66
Acetaldehyde	1.7	70
Acrolein	1.5	75
Acetone/Propionaldehyde	1.5	75
Butyraldehyde	1.5	79
Methyl ethyl ketone	1.5	79
Valeraldehyde	1.5	84
Isovaleraldehyde	1.4	84
Hexaldehyde	1.3	88
Benzaldehyde	1.4	84
o-/m-/p-Tolualdehyde	1.3	89
Dimethylbenzaldehyde	1.2	93

---

[1] Oxygenated compounds in addition to formaldehyde are included for comparison with formaldehyde; extension of the methodology to other compounds is possible.

[2] Detection limits are determined in solvent. These values therefore represent the optimum capability of the methodology.

one-place balance (see Table 3.5-1 for approximate amounts) and add to the stirring acid solution. Fill the 8-L container to the 8-L mark with organic-free reagent water and stir overnight. If all of the DNPH crystals have dissolved overnight, add additional DNPH and stir for two more hours. Continue the process of adding DNPH with additional stirring until a saturated solution has been formed. Filter the DNPH solution using vacuum filtration. Gravity filtration may be used, but a much longer time is required. Store the filtered solution in an amber bottle at room temperature.

3.5.5.4.2.2 Within five days of proposed use, place about 1.6 L of the DNPH reagent in a 2-L separatory funnel. Add approximately 200 ml of methylene chloride and stopper the funnel. Wrap the stopper of the funnel with paper towels to absorb any leakage. Invert and vent the funnel. Then shake vigorously for 3 minutes. Initially, the funnel should be vented frequently (every 10 -15 sec). After the layers have separated, discard the lower (organic) layer.

3.5.5.4.2.3 Extract the DNPH a second time with methylene chloride and finally with cyclohexane. When the cyclohexane layer has separated from the DNPH reagent,the cyclohexane layer will be the top layer in the separatory funnel. Drain the lower layer (the cleaned extract DNPH reagent solution) into an amber bottle that has been rinsed with acetonitrile and allowed to dry.

3.5.5.4.3 **Quality Control:** Take two aliquots of the extracted DNPH reagent. The size of the aliquots is dependent upon the exact sampling procedure used, but 100 ml is reasonably representative. To ensure that the background in the reagent is acceptable for field use, analyze one aliquot of the reagent according to the procedure of Method 8315. Save the other aliquot of aqueous acidic DNPH for use as a method blank when the analysis is performed.

3.5.5.4.4 **Shipment to the Field:** Tightly cap the bottle containing extracted DNPH reagent using a Teflon-lined cap. Seal the bottle with Teflon disposal purposes. 2,4-dinitrophenylhydrazine is a flammable solid when dry, so water should not be evaporated from the solution of the reagent.

3.5.5.5 Field Spike Standard Preparation: To prepare a formaldehyde field spiking standard at 4.01 mg/ml, use a 500 µl syringe to transfer 0.5 ml to 37% by weight of formaldehyde (401 mg/ml) to a 50 ml volumetric flask containing approximately 50 ml of methanol. Dilute to 50 ml with methanol.

3.5.5.6 Hydrochloric Acid, HCL: Reagent grade hydrochloric acid (approximately 12N) is required for acidifying the aqueous DNPH solution.

3.5.5.7 Methylene Chloride, CH_2Cl_2: Methylene chloride (suitable for residue and pesticide analysis, GC/MS, HPLC, GC, Spectrophotometry or equivalent) is required for cleaning the aqueous acidic DNPH solution, rinsing glassware, and recovery of sample trains.

3.5.5.8 Cyclohexane, C_6H_{12}: Cyclohexane (HPLC grade) is required for cleaning the aqueous acidic DNPH solution.

NOTE: Do not use spectroanalyzed grades of cyclohexane if this sampling methodology is extended to aldehydes and ketones with four or more carbon atoms.

3.5.5.9 **Methanol, CH_3OH:** Methanol (HPLC grade or equivalent) is required for rinsing glassware.

3.5.5.10 Acetonitrile, CH_3CN: Acetonitrile (HPLC grade or equivalent) is required for rinsing glassware.

3.5.5.11 Formaldehyde, HCHO: Analytical grade or equivalent formaldehyde is required for preparation of standards. If other aldehydes or ketones are used, analytical grade or equivalent is required.

tape. After the bottle is labeled, the bottle may be placed in a friction-top can (paint can or equivalent) containing a 1-2 inch layer of granulated charcoal and stored at ambient temperature until use.

3.5.5.4.4.1 If the DNPH reagent has passed the Quality Control criteria, the reagent may be packaged to meet necessary shipping requirements and sent to the sampling area. If the Quality Control criteria are not met the reagent solution may be re-extracted or the solution may be re-prepared and the extraction sequence repeated.

3.5.5.4.4.2 If the DNPH reagent is not used in the field within five days of extraction, an aliquot may be taken and analyzed as described in Method 0011A. If the reagent meets the Quality Control requirements, the reagent may be used. If the reagent does not meet the Quality Control requirements, the reagent must be discarded and new reagent must be prepared and tested.

3.5.5.4.5 Calculation of Acceptable Concentrations of Impurities in DNPH Reagent: The acceptable impurity concentration (AIC, µg/ml) is calculated from the expected analyte concentration in the sampled gas (EAC, ppbv), the volume of air that will be sampled at standard conditions (SVOL, L), the formula weight of the analyte (FW, g/mol), and the volume of DNPH reagent that will be used in the impingers (RVOL, ml):

$$AIC = 0.1 \times [EAC \times SVOL \times FW/22.4 \times (FW + 180)/FW](RVOL \times 1,000)$$

where:

0.1 is the acceptable contaminant concentration,
22.4 is a factor relating ppbv to g/L,
180 is a facto relating underivatized to derivatized analyte
1,000 is a unit conversion factor.

3.5.5.4.6 Disposal of Excess DNPH Reagent: Excess DNPH reagent may be returned to the laboratory and recycled or treated as aqueous waste for change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2. of EPA Method 2)

3.5.6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

3.5.6.3.4 A minimum of 45 ft^3 of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of formaldehyde from incineration systems (45 ft^3 is equivalent to one hour of sampling at 0.75 dscf). Additional sample volume shall be collected as necessitated by the capacity of the DNPH reagent and analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.

3.5.6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus 0.5 min.

3.5.6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-volume samples. In these cases, careful documentation must be maintained in order to allow accurate calculation of concentrations.

3.5.6.4 Preparation of Collection Train:

3.5.6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.

3.5.6 Sample Collection, Preservation, and Handling

3.5.6.1 Because of the complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

3.5.6.2 Laboratory Preparation:

3.5.6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified.

3.5.6.2.2 Weigh several 200 to 300 g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

3.5.6.3 Preliminary Field Determinations:

3.5.6.3.1 Select the sampling site and the minimum number of sampling point according to EPA Method 1 or other relevant criteria. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. A leak-check of the pitot lines according to EPA Method 2, Section 3.1, must be performed. Determine the stack gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

3.5.6.3.2 Select a nozzle size based on the range of velocity heads so that is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 L/min (1.0 cfm). During the run, do not

3.5.6.5 Leak-Check Procedures:

3.5.6.5.1 Pre-test Leak Check

3.5.6.5.1.1 After the sampling train has been assembled, turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize. If a Viton-A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 381 mm Hg (15 in Hg) vacuum.

NOTE: A lower vacuum may be used, provided that the lower vacuum is not exceeded during the test.

3.5.6.5.1.2 If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first attaching a carbon-filled leak check impinger to the inlet and then plugging the inlet and pulling a 381 mm Hg (15 in Hg) vacuum. (A lower vacuum any be used if this lower vacuum is not exceeded during the test.) Next connect the probe to the train and leak-check at about 25 mm Hg (1 in Hg) vacuum. Alternatively, leak-check the probe with the rest of the sampling train in one step at 381 mm Hg (15 in Hg) vacuum. Leakage rates in excess of (a) 4% of the average sampling rate or (b) >0.00057 m^3/min (0.02 cfm), are unacceptable.

3.5.6.5.1.3 The following leak check instructions for the sampling train descried in ADPT-0576 and APTD-0581 may be helpful. Start the pump with the fine-adjust valve fully open and coarse-valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do <u>not</u> reverse direction of the fine-adjust valve, as liquid will back up into the train. If the desired vacuum is exceeded, either perform the leak check at this higher vacuum or end the leak check, as shown below, and start over.

3.5.6.4.2 Place 100 ml of cleaned DNPH solution in each of the first two impingers, and leave the third impinger empty. If additional capacity is required for high expected concentrations of formaldehyde in the stack gas 200 ml of DNPH per impinger may be used or additional impingers may be used for sampling. Transfer approximately 200 to 300 g of pre-weighed silica gel from its container to the fourth impinger. Care should be taken to ensure that the silica gel is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place or later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

3.5.6.4.3 With a glass or quartz liner, install the selected nozzle using a Viton-A O-ring with stack temperatures are <260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connection systems utilizing either 316 stainless steel or Teflon ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

3.5.6.4.4 Assemble the train as shown in Figure 3.5-1. During assembly, do not use any silicone grease on ground-glass joints upstream of the impingers. Use Teflon tape, if required. A very light coating of silicone grease may be used on ground-glass joints downstream of the impingers, but the silicone grease should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone grease contamination. If necessary, Teflon tape may be used to seal leaks. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperatures.

3.5.6.4.5 Place crushed ice all around the impingers.

3.5.6.4.6 Turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize.

m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable. If, however, a higher leakage rate is obtained, the tester shall record the leakage rate and void the sampling run.

3.5.6.6 Sampling Train Operation:

3.5.6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, below 20 L/min (1.0 cfm). Maintain a temperature around the probe of 120°C (248° ± 25°F).

3.5.6.6.2 For each run, record the data on a data sheet such as the one shown in Figure 3.5-2. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 2 at least once at each sample point during each time increment and additional readings when significant adjustments 20% variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse

3.5.6.6.3 Clean the stack access ports prior to the test run to eliminate the change of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot tube coefficient is 0.84 ± 0.02 and the stack gas equivalent density (dry molecular weight) is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If the stack gas molecular weight and the pitot tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps are taken to compensate for the deviations.

3.5.6.5.1.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe. When the vacuum drops to 127 mm (5 in Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed to prevent the liquid in the impingers from being forced backward in the sampling line and silica gel from being entrained backward into the third impinger.

3.5.6.5.2 Leak Checks During Sampling Run:

3.5.6.5.2.1 If, during the sampling run, a component change (i.e., impinger) becomes necessary, a leak check shall be conducted immediately after the interruption of sampling and before the change is made. The leak check shall be done according to the procedure described in Section 3.5.6.5.1 except that is shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm or 4% of the average sampling rate (whichever is less), the results are acceptable. If a higher leakage rate is obtained, the tester must void the sampling run.

NOTE: Any correction of the sample volume by calculation reduces the integrity of the pollutant concentration data generated and must be avoided.

3.5.6.5.2.2 Immediately after a component change and before sampling is reinitiated, a leak check similar to a pre-test leak check must also be conducted.

3.5.6.5.3 Post-test Leak Check:

3.5.6.5.3.1 A leak check is mandatory at the conclusion of each sampling run. The leak check shall be done with the same procedures as the pre-test leak check, except that the post-test leak check shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057

3.5.6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack in order to prevent liquid from backing up through the train. If necessary, the pump may be turned on with the coarse-adjust valve closed.

3.5.6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent unrepresentative dilution of the gas stream.

3.5.6.6.6 Traverse the stack cross section, as required by EPA Method 1, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

3.5.6.6.7 During the test run, make periodic adjustments to keep the temperature around the probe at the proper levels. Add more ice and, if necessary, salt, to maintain a temperature of <20°C (68°F) at the silica gel outlet. Also, periodically check the level and zero of the manometer.

3.5.6.6.8 A single train shall be used for the entire sampling run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. An additional train or additional trains may also be used for sampling when the capacity of a single train is exceeded.

3.5.6.6.9 When two or more trains are used, separate analyses of components from each train shall be performed. If multiple trains have been used because the capacity of a single train would be exceeded, first impingers from each train may be combined, and second impingers from each train may be combined.

Plant ____________________

Location ____________________

Operator ____________________

Date ____________________

Run No. ____________________

Sample Box No. ____________________

Meter Box No. ____________________

Meter H@ ____________________

C Factor ____________________

Pitot Tube Coefficient C_p ____________________

Schematic of Stack Cross Section

Ambient Temperature ____________________

Barometric Pressure ____________________

Assumed Moisture % ____________________

Probe Length, m(ft) ____________________

Nozzle Identification No. ____________________

Average Calibrated Nozzle Diameter, cm (in) ____________________

Probe Heating Setting ____________________

Leak Rate, m^3/min. (cfm) ____________________

Probe Liner Material ____________________

Static Pressure, mm Hg (in. Hg) ____________________

Filter No. ____________________

Traverse Point Number	Sampling Time (θ) Min.	Vacuum mm Hg (in. Hg)	Stack Temperature (T_s) °C (°F)	Velocity Head (P_s) mm (in) H_2O	Pressure Differential Across Orifice Meter mm (H_2O) (in H_2O)	Gas Sample Volume m^3 (ft^3)	Gas Sample Temp. at Dry Gas Meter		Filter Holder Temperature °C(°F)	Temperature of Gas Leaving Last Impinger °C(°F)
							Inlet °C(°F)	Outlet °C(°F)		
Total							Avg	Avg		
Average										

Figure [illegible] Field Data Sheet

3.5.7.1.5 Save a portion of all washing solution (methylene chloride, water) used for cleanup as a blank. Transfer 200 ml of each solution directly from the wash bottle being used and place each in a separate, prelabeled sample container.

3.5.7.2 Sample Containers:

3.5.7.2.1 Container 1: Probe and Impinger Catches. Using a graduated cylinder, measure to the nearest ml, and record the volume of the solution in the first three impingers. Alternatively, the solution may be weighed to the nearest 0.5 g. Include any condensate in the probe in this determination. Transfer the impinger solution from the graduated cylinder into the amber flint glass bottle. Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all surfaces to which the sample is exposed (including the probe nozzle, probe fitting, probe liner, first impinger, and impinger connector) with methylene chloride. Use less than 500 ml for the entire wash (250 ml would be better, if possible). Add the washing to the sample container.

3.5.7.2.1.1 Carefully remove the probe nozzle and rinse the inside surface with methylene chloride from a wash bottle. Brush with a Teflon bristle brush, and rinse until the rinse shows no visible particles or yellow color, after which make a final rinse of the inside surface. Brush and rinse the inside parts of the Swagelok fitting with methylene chloride in a similar way.

3.5.7.2.1.2 Rinse the probe liner with methylene chloride. While squirting the methylene chloride into the upper end of the probe, tilt and rotate the probe so that all inside surfaces will be wetted with methylene chloride. Let the methylene chloride drain from the lower end into the sample container. The tester may use a funnel (glass or polyethylene) to aid in transferring the liquid washes to the container. Follow the rinse with a Teflon brush. Hold the probe in an inclined position, and squirt methylene chloride into the upper end as the probe brush is being pushed with a twisting action through the probe. Hold the sample container underneath the lower end

3.5.6.6.10 At the end of the sampling run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak check. Also, leak check the pitot lines as described in EPA Method 2. The lines must pass this leak check in order to validate the velocity-head data.

3.5.6.6.11 Calculate percent isokineticity (see Method 3) to determine whether the run was valid or another test should be made.

3.5.7 <u>Sample Recovery</u>

3.5.7.1 Preparation:

3.5.7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be handled safely, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling because a vacuum will be created drawing liquid from the impingers back through the sampling train.

3.5.7.1.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train, and cap the open outlet, being careful not to lose any condensate that might be present. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used, let any condensed water or liquid drain into the impingers. Cap off any open impinger inlets and outlets. Ground glass stoppers, Teflon caps or caps of other inert materials may be used to seal all openings.

3.5.7.1.3 Transfer the probe and impinger assembly to an area that is clean and protected from wind so that the chances of contaminating or losing the sample are minimized.

3.5.7.1.4 Inspect the train before and during disassembly, and note any abnormal conditions.

hydrazone is a solid which floats and froths on top of the impinger solution. Any physical carryover of collected moisture into the second impinger will invalidate a breakthrough assessment.

3.5.7.2.2 Container 2: Sample Blank. Prepare a blank by using an amber flint glass container and adding a volume of DNPH reagent and methylene chloride equal to the total volume in Container 1. Process the blank in the same manner as Container 1.

3.5.7.2.3 Container 3: Silica Gel. Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. The impinger containing the silica gel may be used as a sample transport container with both ends sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon caps maybe used. The silica gel impinger should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If the silica gel is removed from the impinger, the tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use water or other liquids to transfer the silica gel. If a balance is available in the field, the spent silica gel (or silica gel plus impinger) maybe weighed to the nearest 0.5 g.

3.5.7.2.4 Sample containers should be placed in a cooler, cooled by (although not in contact with) ice. Sample containers must be placed vertically and, since they are glass, protected from breakage during shipment. Samples should be cooled during shipment so they will be received cold at the laboratory.

of the probe, and catch any methylene chloride, water, and particulate matter that is brushed from the probe. Run the brush through the probe three times or more. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since there may be small crevices in which particulate matter can be entrapped. Rinse the brush with methylene chloride or water, and quantitatively collect these washing in the sample container. After the brushing, make a final rinse of the probe as described above.

NOTE: Two people should clean the probe in order to minimize sample losses. Between sampling runs, brushes must be kept clean and free from contamination.

3.5.7.2.1.3 Rinse the inside surface of each of the first three impingers (and connecting tubing) three separate times. Use a small portion of methylene chloride for each rinse, and brush each surface to which the sample is exposed with a Teflon bristle brush to ensure recovery of fine particulate matter. Water will be required for the recovery of the impingers in addition to the specified quantity of methylene chloride. There will be at least two phases in the impingers. This two-phase mixture does not pour well and a significant amount of the impinger catch will be left on the walls. The use of water as a rinse makes the recovery quantitative. Make a final rinse of each surface and of the brush, using both methylene chloride and water.

3.5.7.2.1.4 After all methylene chloride and water washing and particulate matter have been collected in the sample container, tighten the lid so the solvent, water, and DNPH reagent will not leak out when the container is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Seal the container with Teflon tape. Label the container clearly to identify its contents.

3.5.7.2.1.5 If the first two impingers are to be analyzed separately to check for breakthrough, separate the contents and rinses of the two impingers into individual containers. Care must be taken to avoid physical carryover from the first impinger to the second. The formaldehyde valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more the 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

3.5.8.3.3 Leak check of metering system: The portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. Use the following procedure: Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 - 18 cm (5 - 7 in) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series must be voided or calculations for test series must be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

3.5.8.4 **Probe Heater:** The probe heating system must be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

3.5.8.5 **Temperature gauges:** Each thermocouple must be permanently and uniquely marked on the casting. All mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at the ambient air temperatures, with and without the extension lead, must be noted

3.5.8 Calibration

3.5.8.1 Probe Nozzle: Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in). when the nozzles become nicked or corroded, they shall be replaced and calibrated before use. Each nozzle must be permanently and uniquely identified.

3.5.8.2 Pitot Tube: The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of EPA Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked or corroded and if it meets design and intercomponent spacing specifications.

3.5.8.3 Metering System

3.5.8.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm pumps, the normal leak check procedure will not detect leakages with the pump. For these cases, the following leak check procedure will apply: make a ten-minute calibration run at 0.00057 m^3/min (0.02 cfm). At the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

3.5.8.3.2 After each field use, check the calibration of the metering system by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). Set the vacuum at the maximum value reached during the test series. To adjust the vacuum, insert a

3.5.9.1 Calculation of Total Formaldehyde: To determine the total formaldehyde in mg, use the following equation:

$$\text{Total mg formaldehyde} = C_d \times V \times DF \times \frac{[\text{g/mole aldehyde}]}{[\text{g/mole DNPH derivative}]} \times 10^3 \text{ mg}/\mu\text{g}$$

where:

C_d = measured concentration of DNPH - formaldehyde derivative, μg/ml
V = organic extract volume ml
DF = dilution factor

3.5.9.2 Formaldehyde concentration in stack gas:

Determine the formaldehyde concentration in the stack gas using the following equation:

$$C_f = K \text{ [total formaldehyde, mg] } V_{m(std)}$$

where:

K = 35.31 ft^3/m^3 if $V_{m(std)}$ is expressed in English units
= 1.00 m^3/m^3 if $V_{m(std)}$ is expressed in metric units
$V_{m(std)}$ = volume of gas sample a measured by dry gas meter, corrected to standard conditions, dscm (dscf)

3.5.9.3 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop are obtained from the data sheet.

3.5.9.4 Dry Gas Volume: Calculate $V_{m(std)}$ and adjust for leakage, if necessary, using the equation in Section 6.3 of EPA Method 5.

3.5.9.5 Volume of Water Vapor and Moisture Content: Calculate the volume of water vapor and moisture content from equations 5-2 and 5-3 of EPA Method 5.

3.5.10 <u>Determination of Volume to be Sampled</u>

To determine the minimum sample volume to be collected, use the following sequence of equations.

and recorded. Correction is necessary if the use of an extension lead produces a change >1.5%.

3.5.8.5.1 Impinger and dry-gas meter thermocouples: For the thermocouples used to measure the temperature of the gas leaving the impinger train, three-point calibration at ice water, room air, and boiling water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2C (3.6°F) with those of the absolute value of the reference thermometer.

3.5.8.5.2 Probe and stack thermocouple: for the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice water, boiling water, and hot oil bath temperatures must be performed. Use of a point at room air temperature is recommended. The thermometer and thermocouple must agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

3.5.8.6 Barometer: Adjust the barometer initially and before each test series to agree to within ±2.5 mm Hg (0.1 in Hg) of the mercury barometer or the correct barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

3.5.8.7 Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class S standard weights. The weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

3.5.9 Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

3.5.10.4 The following analytical detection limits and DNPH Reagent Capacity (based on a total volume of 200 ml in two impingers) must also be considered in determining a volume to be sampled.

3.5.11 Quality Control

3.5.11.1 Sampling: See EPA Manual 600/4-77-02b for Method 5 quality control.

3.5.11.2 Analysis: The quality assurance program required for this method includes the analysis of the field and method blanks, procedure validations, and analysis of field spikes. The assessment of combustion data and positive identification and quantitation of formaldehyde are dependent on the integrity of the samples received and the precision and accuracy of the analytical methodology. Quality assurance procedures for this method are designed to monitor the performance of the analytical methodology and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

3.5.11.2.1 Field Blanks: Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample-recovery solvents, methylene chloride and water, and unused DNPH reagent. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling are, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual sampling train). The probe of the blank train must be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the blank sampling train.

3.5.11.2.2 Method Blanks: A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

3.5.10.1 From prior analysis of the waste feed, the concentration of formaldehyde (FORM) introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the amount of FORM allowed to be present in the effluent. This amount may be expressed as:

$$\text{Max FORM Mass} = [\,(\text{WF})\,(\text{FORM conc})\,(100 - \%\text{DRE})\,]\,/100$$

where:

WF = mass flow rate of waste feed per h, g/h (lb/h)

FORM = concentration of FORM (wt %) introduced into the combustion process

DRE = percent Destruction and Removal Efficiency required

Max FORM = mass flow rate (g/h [lb/]) of FORM emitted from the combustion sources

3.5.10.2 The average discharge concentration of the FORM in the effluent gas is determined by comparing the Max FORM with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary EPA Method 1 - 4 determinations:

$$\text{Max FORM conc} = [\text{Max FORM Mass}]\,/\,DV_{eff(std)}$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf)

FORM conc = anticipated concentration of the FORM in the exhaust gas stream, g/dscm (lb/dscf)

3.5.10.3 In making this calculation, it is recommended that a safety margin of at least ten be included.

$$[LDL_{FORM} \times 10\,/\,\text{FORM conc}] = V_{tbc}$$

where:

LDL_{FORM} = detectable amount of FORM in entire sampling train

V_{tbc} = minimum dry standard volume to be collected at dry-gas meter

Table 3.5-3

EXPECTED METHOD PERFORMANCE FOR FORMALDEHYDE

Parameter	Precision[1]	Accuracy[2]	Detection Limit[3]
Matrix: Dual trains	±15% RPD	±20%	1.5×10^{-7} lb/ft^3 (1.8 ppbv)

---

[1] Relative percent difference limit for dual trains.

[2] Limit for field spike recoveries.

[3] The lower reporting limit having less than 1% probability of false positive detection.

3.5.11.2.3 Field Spike: A field spike is performed by introducing 200 µL of the Field Spike Standard into an impinger containing 200 ml of DNPH solution. Standard impinger recovery procedures are followed and the spike is used as a check on field handling and recovery procedures. An aliquot of the field spike standard is retained in the laboratory for derivatization and comparative analysis.

3.5.12 Method Performance

3.5.12.1 Method performance evaluation: The expected method performance parameters for precision, accuracy, and detection limits are provided in Table 3.5-3.

Addition of a Filter to the Formaldehyde Sampling Train

As a check on the survival of particulate material through the impinger system, a filter can be added to the impinger train either after the second impinger or after the third impinger. Since the impingers are in an ice bath there is no reason to heat the filter at this point.

Any suitable medium (e.g., paper, organic membrane) may be used for the filter if the material conforms to the following specifications:

1) the filter has at least 95% collection efficiency (<5% penetration) for 3 µm dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

2) the filter has a low aldehyde blank value (<0.015 mg formaldehyde/cm^2 of filter area). Before the test series, determine the average formaldehyde blank value of at least three filters (from the lot to be used for sampling) using the applicable analytical procedures.

3.6 Analysis for Aldehydes and Ketones by High Performance Liquid Chromatography (HPLC) (Method 0011A)

3.6.1 Scope and Application

3.6.1.1 Method 0011A covers the determination of free formaldehyde in the aqueous samples and leachates and derived aldehydes/ketones collected by Method 0011.

Compound Name	CAS No.*
Formaldehyde	50-00-0
Acetaldehyde	75-07-0

* Chemical Abstract Services Registry Number

3.6.1.2 Method 0011A is a high performance liquid chromatographic (HPLC) method optimized for the determination of formaldehyde and acetaldehyde in aqueous environmental matrices and leachates of solid samples and stack samples collected by Method 0011. When this method is used to analyze unfamiliar sample matrices, compound identification should be supported by at least one additional qualitative technique. A gas chromatograph/mass spectrometer (GC/MS) may be used for the qualitative confirmation of results from the target analytes, using the extract produced by this method.

3.6.1.3 The method detection limits (MDL) are listed in Tables 3.6-1 and 3.6-2. The MDL for a specific sample may differ from that listed, depending upon the nature of interferences in the sample matrix and the amount of sample used in the procedure.

3.6.1.4 The extraction procedure for solid samples is similar to that specified in Method 1311 (1). Thus, a single sample may be extracted to measure the analytes included in the scope of other appropriate methods. The analyst is allowed the flexibility to select chromatographic conditions

Recover the exposed filter into a separate clean container and return the container over ice to the laboratory for analysis. If the filter is being analyzed for formaldehyde, the filter may be recovered into a container of DNPH reagent for shipment back to the laboratory. If the filter is being examined for the presence of particulate material, the filter may be recovered into a clean dry container and returned to the laboratory.

appropriate for the simultaneous measurement of contaminations of these analytes

3.6.1.5 This method is restricted to use by, or under the supervision of analysts experienced in the use of chromatography and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

3.6.1.6 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available.

3.6.1.7 Formaldehyde has been tentatively classified as a known or suspected, human or mammalian carcinogen.

3.6.2 Summary of Method

3.6.2.1 Environmental Liquids and Solid Leachates

3.6.2.1.1 For wastes comprised of solids or for aqueous wastes containing significant amounts of solid material, the aqueous phase, if any, is separated from [illegible] solid phase and stored for later analysis. If necessary, the particle size of the solids in the waste is reduced. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase of the waste. A special extractor vessel is used when testing for volatiles. Following extraction, the aqueous extract is separated from the solid phase by filtration employing 0.6 to 0.8 µm glass fiber filters.

Table 3.6-1

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS USING SOLID SORBENT EXTRACTION

Analyte	Retention Time (minutes)	MDL (µg/L)*
Formaldehyde	7.1	7.2

HPLC conditions: Reverse phase C18 column, 4.6 x 250 mm; isocratic elution using methanol/water (75:25, v/v); flow rate 1.0 mL/min.; detector 360 nm

* After correction for laboratory blank.

Table 3.6-2

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS USING METHYLENE CHLORIDE EXTRACTION

Analyte	Retention Time (minutes)	MDL (µg/L)*
Formaldehyde	7.1	7.2
Acetaldehyde	8.6	171*

HPLC conditions: Reverse phase C18 column, 4.6 x 250 mm; isocratic elution using methanol/water (75:25, v/v): flow rate 1.0 mL/min.; detector 360 nm.

* These values include reagent blank concentrations of approximately 13 µg/L formaldehyde and 130 µg/L acetaldehyde.

these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks

3.6.3.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used This should be followed by detergent washing with hot water, and rinses with tap water and distilled water. It should then be drained, dried, and heated in a laboratory oven at 130°C for several hours before use. Solvent rinses with methanol may be substituted for the oven heating. After drying and cooling, glassware should be stored in a clean environment to prevent any accumulation of dust or other contaminants.

3.6.3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.6.3.2 Analysis for formaldehyde is especially complicated by its ubiquitous occurrence in the environment.

3.6.3.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the matrix being sampled. No interferences have been observed in the matrices studied as a result of using solid sorbent extraction as opposed to liquid extraction. If interferences occur in subsequent samples, some additional cleanup may be necessary.

3.6.3.4 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the HPLC conditions described allow for a resolution of the specific compounds covered by this method, other matrix components may interfere.

3.6.4 Apparatus and Materials

3.6.4.1 Reaction vessel - 250 ml Florence flask.

3.6.2.1.2 If compatible (i.e., multiple phases will not form on combination), the initial aqueous phase of the waste is added to the aqueous extract, and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume weighted average concentration.

3.6.2.1.3 A measured volume of aqueous sample or an appropriate amount of solids leachate is buffered to pH 5 and derivatized with 2,4-dinitrophenylhydrazine (DNPH), using either the solid sorbent or the methylene derivatization/extraction option. If the solid sorbent option is used, the derivative is extracted using solid sorbent cartridges, followed by elution with ethanol. If the methylene chloride option is used, the derivative is extracted with methylene chloride. The methylene chloride extracts are concentrated using the Kuderna-Danish (K-D) procedure and solvent exchanged into methanol prior to HPLC analysis. Liquid chromatographic conditions are described which permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

3.6.2.2 Stack Gas Samples Collected by Method 0011

3.6.2.2.1 The entire sample returned to the laboratory is extracted with methylene chloride and the methylene chloride extract is brought up to a known volume. An aliquot of the methylene chloride extract is solvent exchanged and concentrated or diluted as necessary.

3.6.2.2.2 Liquid chromatographic conditions are described that permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

3.6.3 Interferences

3.6.3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of

3.6.4.8.5 Strip-chart recorder compatible with detector - Use of a data system for measuring peak areas and retention times is recommended.

3.6.4.9 Glass fiber filter paper.

3.6.4.10 Solid sorbent cartridges - Packed with 500 mg C18 (Baker or equivalent).

3.6.4.11 Vacuum manifold - Capable of simultaneous extraction of up to 12 samples (Supelco or equivalent).

3.6.4.12 Sample reservoirs - 60 ml capacity (Supelco or equivalent).

3.6.4.13 Pipet - Capable of accurately delivering 0.10 ml solution (Pipetman or equivalent).

3.6.4.14 Water bath - Heated, with concentric ring cover, capable of temperature control ((±) 2°C). The bath should be used under a hood.

3.6.4.15 Volumetric Flasks - 250 or 500 ml.

3.6.5 Reagents

3.6.5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.6.5.2 Organic-free water - All references to water in this method refer to organic-free reagent water, as defined in Chapter I SW-846.

3.6.4.2 Separatory funnel - 205 ml, with Teflon stopcock

3.6.4.3 Kuderna-Danish (K-D) apparatus.

3.6.4.3.1 Concentrator tube - 10 ml graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

3.6.4.3.2 Evaporation flask - 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent

3.6.4.3.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

3.6.4.3.4 Snyder column - Two ball macro (Kontes K-569001-0219 or equivalent).

3.6.4.3.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

3.6.4.4 Vials - 10, 25 ml, glass with Teflon lined screw caps or crimp tops.

3.6.4.5 Boiling chips - Solvent extracted with methylene chloride approximately 10/40 mesh (silicon carbide or equivalent).

3.6.4.6 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

3.6.4.7 pH meter - Capable of measuring to the nearest 0.01 units

3.6.4.8 High performance liquid chromatograph (modular)

3.6.4.8.1 Pumping system - Isocratic, with constant flow control capable of 1.00 ml/min.

3.6.4.8.2 High pressure injection valve with 20 µL loop.

3.6.4.8.3 Column - 250 mm x 4.6 mm ID, 5 µm particle size, C18 (or equivalent).

3.6.4.8.4 Absorbance detector - 360 nm.

using 0.1 N HCl. The formaldehyde concentration is calculated using the following equation:

Concentration (mg/ml) = 30.03 x (N HCl) x (ml HCl) 25.0

where:

N HCl = Normality of HCl solution used

ml HCl = ml of standardized HCl solution used

30.03 = MW of formaldehyde

3.6.5.14.2 Stock formaldehyde and acetaldehyde - Prepare by adding 265 μL formalin and 0.1 g acetaldehyde to 90 ml of water and dilute to 100 ml. The concentration of acetaldehyde in this solution is 1.00 mg/ml. Calculate the concentration of formaldehyde in this solution using the results of the assay performed in Section 3.6.5.14.1.1.

3.6.5.14.3 Stock standard solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

3.6.5.15 Reaction Solutions

3.6.5.15.1 DNPH (1.00 μg/L) - Dissolve 142.9 mg of 70% (w/w) reagent in 100 ml absolute ethanol. Slight heating or sonication may be necessary to effect dissolution.

3.6.5.15.2 Acetate buffer (5 N) Prepare by neutralizing glacial acetic acid to pH 5 with 5 N NaOH solution. Dilute to standard volume with water.

3.6.5.15.3 Sodium chloride solution (saturated) Prepare by mixing of the reagent grade solid with water.

3.6.6 Sample Collection, Preservation, and Handling

3.6.6.1 See the introductory material to this Chapter, Organic Analytes, Section 4.1 of SW-846.

3.6.5.3 Methylene chloride, CH_2Cl_2 - HPLC grade or equivalent

3.6.5.4 Methanol, CH_3OH - HPLC grade or equivalent.

3.6.5.5 Ethanol (absolute), CH_3CH_2OH - HPLC grade or equivalent

3.6.5.6 2,4-Dinitrophenylhydrazine (DNPH) (70% (W/W)), $[2,4\text{-}(O_2N)_2C_6H_3]NHNH_2$, in organic-free reagent water.

3.6.5.7 Formalin (37.6 percent (w/w)), formaldehyde in organic-free reagent water.

3.6.5.8 Acetic acid (glacial), CH_3CO_2H.

3.6.5.9 Sodium hydroxide solutions NaOH, 1.0 N and 5 N.

3.6.5.10 Sodium chloride, NaCl.

3.6.5.11 Sodium sulfite solution, Na_2SO_3, 0.1 M.

3.6.5.12 Hydrochloric Acid, HCl, 0.1 N.

3.6.5.13 Extraction fluid - Dilute 64.3 ml of 1.0 N NaOH and 5.7 ml glacial acetic acid to 900 ml with organic-free reagent water. Dilute to 1 liter with organic-free reagent water. The pH should be 4.93 ± 0.02.

3.6.5.14 Stock standard solutions

3.6.5.14.1 Stock formaldehyde (approximately 1.00 mg/ml) - Prepare by diluting 265 µl formalin to 100 ml with organic-free reagent water.

3.6.5.14.1.1 Standardization of formaldehyde stock solution - Transfer a 25 ml aliquot of a 0.1 M Na_2SO_3 solution to a beaker and record the pH. Add a 25.0 ml aliquot of the formaldehyde stock solution (Section 3.6.5.14.1) and record the pH. Titrate this mixture back to the original pH for 18 hours. Filter the extract through glass fiber paper and store in sealed bottles at 4°C. Each ml of extract represents 0.050 g solid.

3.6.7.2 Cleanup and Separation

3.6.7.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. The cleanup procedures recommended in this method have been used for the analysis of various sample types. If particular circumstances demand the use of an alternative cleanup procedure, the analyst must determine the elution profile and demonstrate that the recovery of formaldehyde is no less then 85% of recoveries specified in Table 3.6-3. Recovery may be lower for samples which form emulsions.

3.6.7.2.2 If the sample is not clean, or the complexity is unknown, the entire sample should be centrifuged at 2500 rpm for 10 minutes. Decant the supernatant liquid from the centrifuge bottle, and filter through glass fiber filter paper into a container which can be tightly sealed.

3.6.7.3 Derivatization

3.6.7.3.1 For aqueous samples, measure a 50 to 100 ml aliquot of the sample. Quantitatively transfer the sample aliquot to the reaction vessel (Section 3.6.4.1).

3.6.7.3.2 For solid samples, 1 to 10 ml of leachate (Section 3.6.7.1) will usually be required. The amount used for a particular sample must be determined through preliminary experiments.

3.6.6.2 Environmental liquid and leachate samples must be refrigerated at 4°C, and must be derivatized within 5 days of sample collection and analyzed within 3 days of derivatization.

3.6.6.3 Stack gas samples collected by Method 0011 must be refrigerated at 4°C. It is recommended that samples be extracted within 30 days of collection and that extracts be analyzed within 30 days extraction.

3.6.7 Procedure

3.6.7.1 Extraction of Solid Samples

3.6.7.1.1 All solid samples should be homogeneous. When the sample is not dry, determine the dry weight of the sample, using a representative aliquot.

3.6.7.1.1.1 Determination of dry weight - In certain cases, sample results are desired based on a dry weight basis. When such data is desired, or required, a portion of sample for dry weight determination should be weighed out at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample.

3.6.7.1.1.2 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the % dry weight of the sample by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

3.6.7.1.2 Measure 25 g of solid into a 500 ml bottle with a Teflon lined screw cap or crimp top, and add 500 ml of extraction fluid (Section 3.6.5.13). Extract the solid by rotating the bottle at approximately 30 rpm

Note: For all reactions, the total volume of the aqueous layer should be adjusted to 100 ml with water.

3.6.7.3.3 Derivatization and extraction of the derivative can be accomplished using the solid sorbent (Section 3.6.7.3.4) or methylene chloride option (Section 3.6.7.3.5).

3.6.7.3.4 Solid Sorbent Option

3.6.7.3.4.1 Add 4 ml of acetate buffer and adjust the pH to 5.0 ± 0.1 with glacial acetic acid or 5 N NaOH. Add 6 ml of DNPH reagent, seal the container, and place on a wrist-action shaker for 30 minutes.

3.6.7.3.4.2 Assemble the vacuum manifold and connect to a water aspirator or vacuum pump. Assemble solid sorbent cartridges containing a minimum of 1.5 g of C18 sorbent, using connectors supplied by the manufacturer, and attach the sorbent train to the vacuum manifold. Condition each cartridge by passing 10 ml dilute acetate buffer (10 ml 5 N acetate buffer dissolved in 250 ml water) through the sorbent cartridge train.

3.6.7.3.4.3 Remove the reaction vessel from the shaker and add 10 ml saturated NaCl solution to the vessel.

3.6.7.3.4.4 Add the reaction solution to the sorbent train and apply a vacuum so that the solution is drawn through the cartridges at a rate of 3 to 5 ml/min. Release the vacuum after the solution has passed through the sorbent.

3.6.7.3.4.5 Elute each cartridge train with approximately 9 ml of absolute ethanol, directly into a 10 ml volumetric flask. Dilute the solution to volume with absolute ethanol, mixed thoroughly, and place in a tightly sealed vial until analyzed.

Table 3.6-3

SINGLE OPERATOR ACCURACY AND PRECISION USING SOLID SORBENT EXTRACTION

Analyte	Matrix Type	Average Percent Recovery	Standard Deviation Percent	Spike Range (µg/L)	Number of Analyses
Formaldehyde	Reagent Water	86	9.4	15-1430	19
	Final Effluent	90	11.0	46.8-1430	16
	Phenol formaldehyde Sludge	93	12.0	457-1430	15

3.6.7.3.5.5.1 Following K-D concentration of the methylene chloride extract to < 10 ml using the macro Snyder column, allow the apparatus to cool and drain for at least 10 minutes.

3.6.7.3.5.5.2 Momentarily remove the Snyder column, add 5 ml of the methanol, a [illegible] bead, or boiling chip, and attach the micro Snyder column. Concentrate the extract using 1 ml of methanol to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration. At the proper rate of distillation the balls of the column will actively chatter. [illegible] the chambers will not flood. When the apparent volume of liquid reaches [illegible], remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

3.6.7.3.5.5.3 Remove the Snyder column and rinse the flask and its lower joint with 1-2 ml of methanol and add to concentrator tube. A 5-ml syringe is recommended for this operation. Adjust the extract volume to 10 ml. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon-lined screw cap or crimp top. Proceed with liquid chromatographic analysis if further cleanup is not required.

3.6.7.4 **Extraction of Stack Gas Samples Collected by Method 0011**

3.6.7.4.1 Measure the aqueous volume of the sample prior to extraction (for moisture determination in case the volume was not measured in the field). Pour the sample into a separatory funnel and drain the methylene chloride into a volumetric flask.

3.6.7.4.2 Extract the aqueous solution with two or three aliquots of methylene chloride. Add the methylene chloride extracts to the volumetric flask.

3.6.7.3.5 Methylene Chloride Option

3.6.7.3.5.1 Add 5 ml of acetate buffer and adjust the pH to 5.0 ± 0.5 with glacial acetic acid or 5 N NaOH. Add 10 ml of DNPH reagent, seal the container, and place on a wrist-action shaker for 1 hour.

3.6.7.3.5.2 Extract the solution with three 20 ml portions of methylene chloride, using a 250 ml separatory funnel, and combine the methylene chloride layers. If an emulsion forms upon extraction, remove the entire emulsion and centrifuge at 2000 rpm for 10 minutes. Separate the layers and proceed with the next extraction.

3.6.7.3.5.3 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporator flask. Wash the K-D apparatus with 25 ml of extraction solvent to complete the quantitative transfer.

3.6.7.3.5.4 Add one to two clean boiling chips to the evaporative flask and attach a three ball Snyder column. Preset the Snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 10 ml, remove the K-D apparatus and allow it to drain and cool for a least 10 min.

3.6.7.3.5.5 Prior to liquid chromatographic analysis, the solvent must be exchanged to methanol. The analyst must ensure quantitative transfer of the extract concentrate. The exchange is performed as follows:

3.6.7.6.1.1.2 Process each calibration standard solution through the derivatization option used for sample processing (Section 3.6.7.3.4 or 3.6.7.3.5)

3.6.7.6.1.2 External standard calibration procedure

3.6.7.6.1.2.1 Analyze each derivatized calibration standard using the chromatographic conditions listed in Tables 3.6-1 and 3.6-2, and tabulate peak area against concentration injected. The results may be used to prepare calibration curves for formaldehyde and acetaldehyde.

3.6.7.6.1.2.2 The working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any analyte varies from the previously established responses by more the 10%, the test must be repeated using a fresh calibration standard after it is verified that the analytical system is in control. Alternatively, a new calibration curve may be prepared for that compound. If an autosampler is available, it is convenient to prepare a calibration curve daily by analyzing standards along with test samples.

3.6.7.7 Analysis

3.6.7.7.1 Analyze samples by HPLC, using conditions established in Section 3.6.7.6.1. Tables 3.6-1 and 3.6-2 list the retention times and MDLs that were obtained under these conditions. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements for Section 3.6.8.1 are met, or if the data are within the limits described in Tables 3.6-1 and 3.6-2.

3.6.7.7.2 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

3.6.7.4.3 Fill the volumetric flask to the line with methylene chloride. Mix well and remove an aliquot.

3.6.7.4.4 If high levels of formaldehyde are present, the extract can be diluted with mobile phase, otherwise the extract must be solvent exchanged as described in Section 3.6.7.5.3.3. If low levels of formaldehyde are present, the sample should be concentrated during the solvent exchange procedure.

3.6.7.5 Chromatographic Conditions

Column:	C18, 250 mm x 4.6 mm ID, 5 µm particle size
Mobile Phase:	methanol/water, 75:25 (v/v), isocratic
Flow Rate:	1.0 ml/min
UV Detector:	360 nm
Injection Volume:	20 µl

3.6.7.6 Calibration

3.6.7.6.1 Establish liquid chromatographic operating parameters to produce a retention time equivalent to that indicated in Table 3.6-1 for the solid sorbent options, or in Table 3.6-2 for methylene chloride option. Suggested chromatographic conditions are provided in Section 3.6.7.5. Prepare derivatized calibration standards according to the procedure in Section 3.6.7.6.1.1. Calibrate the chromatographic system using the external standard technique (Section 3.6.7.6.1.2).

3.6.7.6.1.1 Preparation of calibration standards

3.6.7.6.1.1.1 Prepare calibration standard solutions of formaldehyde and acetaldehyde in water from the stock standard (Section 3.6.5.14.2). Prepare these solutions at the following concentrations (in µg/ml) by serial dilution of the stock standard solution: 50, 20, 10. Prepare additional calibration standard solutions at the following concentrations, by dilution of the appropriate 50, 20, or 10 µg/ml standard: 5, 0.5, 2, 0.2, 1, 0.1.

$$\text{total } \mu g/ml = (RF)\,(\text{area of signal})\,(\text{concentration factor})$$

where:

$$\text{concentration factor} = \frac{\text{Final Volume of Extract}}{\text{Initial Extract Volume}}$$

3.6.8 Quality Control

3.6.8.1 Refer to Chapter One of SW-846 for guidance on quality control procedures.

3.6.9 Method Performance

3.6.9.1 The MDL concentrations listed in Table 3.6-1 were obtained using organic-free water and solid sorbent extraction. Similar results were achieved using a final effluent and sludge leachate. The MDL concentrations listed in Table 3.6-2 were obtained using organic-free water and methylene chloride extraction. Similar results were achieved using representative matrices.

3.6.9.2 This method has been tested for linearity of recovery from spiked organic-free water and has been demonstrated to be applicable over the range from 2 x MDL to 200 x MDL.

3.6.9.3 In a single laboratory evaluation using several spiked matrices, the average recoveries presented in Tables 3.6-3 and 3.6-4 were obtained using solid sorbent and methylene chloride extraction, respectively. The standard deviations of the percent recovery are also included in Tables 3.6-3 and 3.6-4.

3.6.9.4 A representative chromatogram is presented in Figure 3.6-1.

3.6.7.7.3 If the peak area exceeds the linear range of the calibration curve, a smaller sample volume should be used. Alternatively, the final solution may be diluted with ethanol and reanalyzed.

3.6.7.7.4 If the peak area measurement is prevented by the presence of observed interferences, further cleanup is required. However, none of the 3600 method series have been evaluated for this procedure.

3.6.7.8 Calculations

3.6.7.8.1 Calculate each response factor as follows (mean value based on 5 points):

$$RF = \frac{\text{concentration of standard}}{\text{area of the signal}}$$

$$\text{mean} = RF = \overline{RF} = \frac{(\sum_{i}^{5} RF_i)}{5}$$

3.6.7.8.2 Calculate the concentration of formaldehyde and acetaldehyde as follows:

$$\mu g/ml = (\overline{RF})\ (\text{area of signal})\ (\text{concentration factor})$$

where:

$$\text{concentration factor} = \frac{\text{Final volume of extract}}{\text{Initial sample (or leachate) volume}}$$

NOTE: For solid samples, a dilution factor must be included in the equation to account for the weight of the sample used.

3.6.7.8.3 Calculate the total weight of formaldehyde in the stack gas sample as follows:

Table 3.6-4

SINGLE OPERATOR ACCURACY AND PRECISION USING METHYLENE CHLORIDE EXTRACTION

Analyte	Matrix Type	Average Percent Recovery x	Standard Deviation Percent p	Spike Range (µg/L)	Number of Analyses
Formaldehyde	Reagent Water	91	2.5	50-1000	9
	Ground-water	92.5	8.2	50	6
	Liquids	69.6	16.3	250	12
Acetaldehyde	Reagent Water	60.3	3.2	50-1000	9
	Ground-water	63.6	10.9	50	12
	Liquids (2 types)	44.0	20.2	250	12
	Solids	58.4	2.7	0.10-1.0*	12

* Spike range in units of mg/g.

x = Average recovery expected for this method

p = Average standard deviation expected for this method.

3.6.10 References

1. Federal Register, 1986, 51, 40643-40652; November 7.

2. EPA Methods 6010, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency Washington, D.C. 20460.

METHOD 0011A

FORMALDEHYDE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

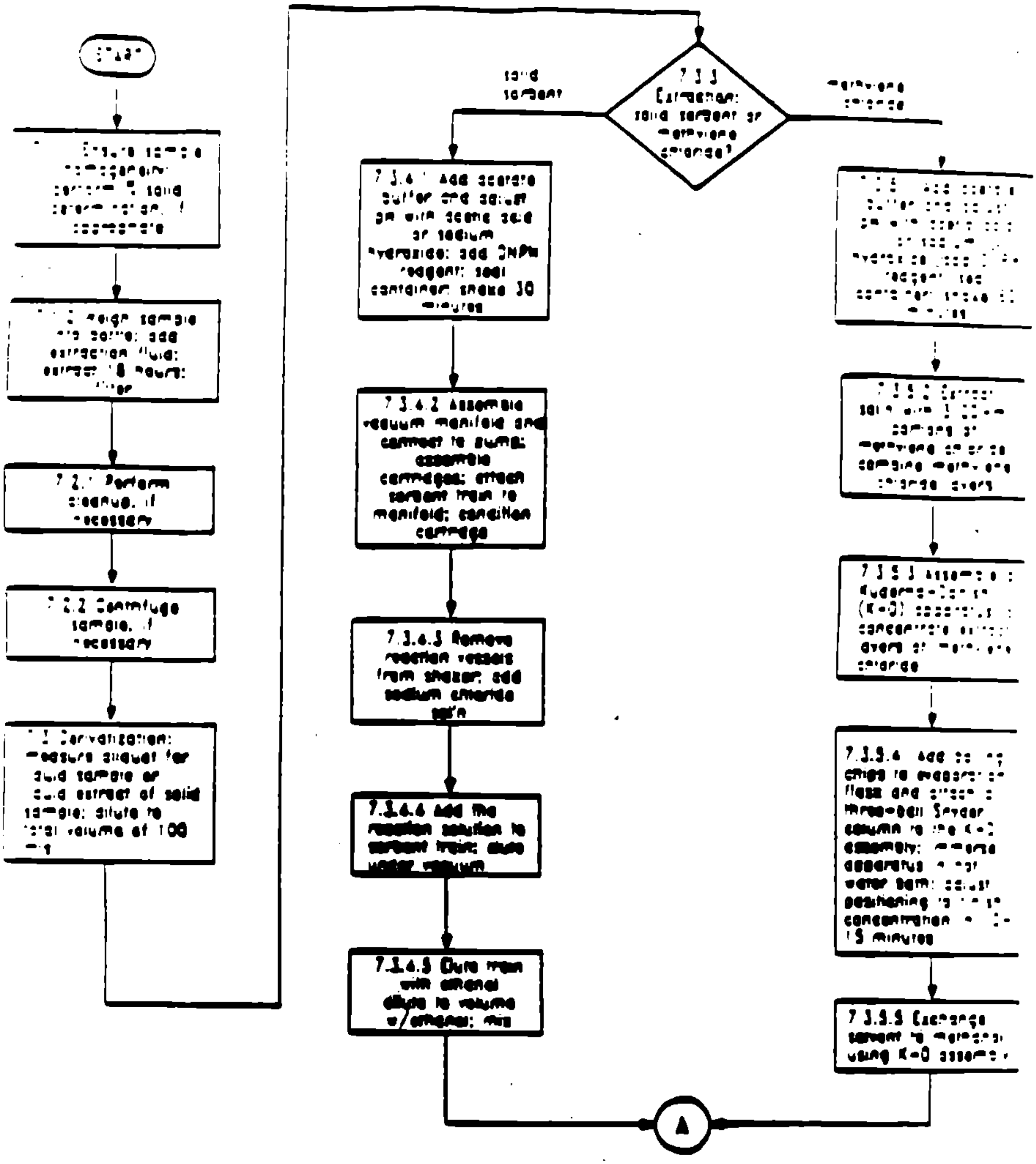
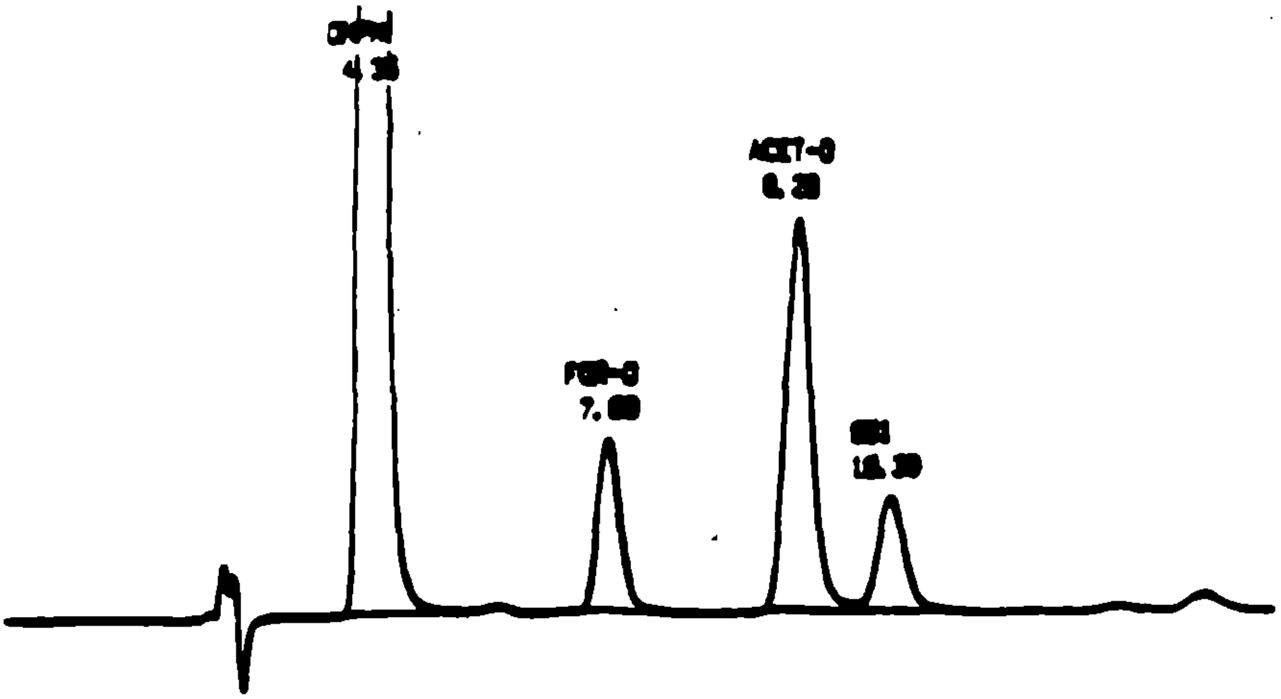
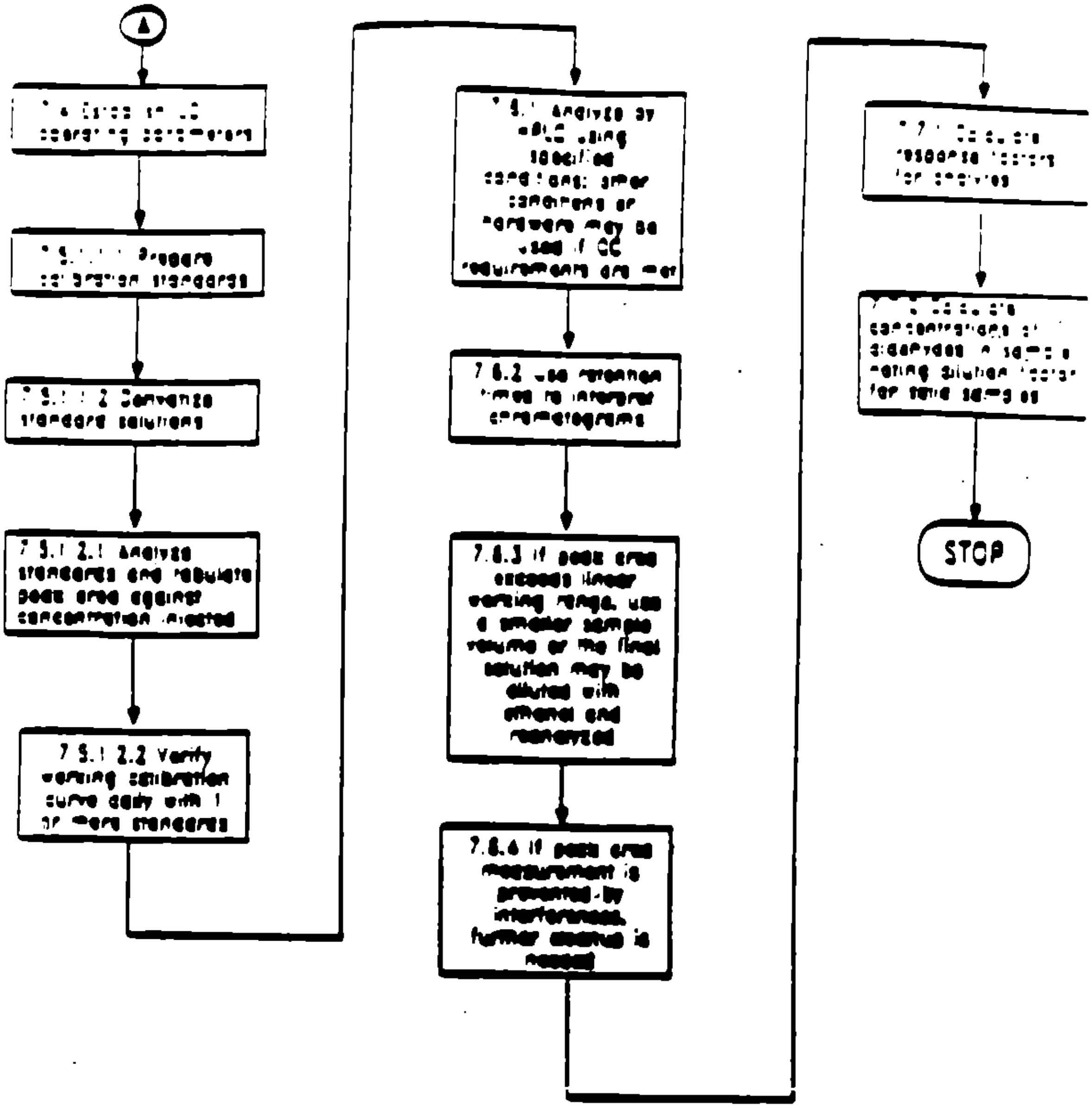


Figure 3.6-1

REPRESENTATIVE CHROMATOGRAM OF A 50 μg/L SOLUTION OF FORMALDEHYDE



FOR-D = Formaldehyde derivative
ACET-D = Acetaldehyde derivative


A
7.4 Establish LC operating parameters
7.5.1.1 Prepare calibration standards
7.5.1.1.2 Derivatize standard solutions
7.5.1.2.1 Analyze standards and tabulate peak area against concentration injected
7.5.1.2.2 Verify working calibration curve daily with 1 or more standards
7.6.1 Analyze by HPLC using specified conditions; other conditions or hardware may be used if QC requirements are met
7.6.2 Use retention times to interpret chromatograms
7.6.3 If peak area exceeds linear working range, use a smaller sample volume or the final solution may be diluted with ethanol and reanalyzed
7.6.4 If peak area measurement is prevented by interferences, further cleanup is needed
7.7.1 Calculate response factors for analytes
7.7.2 Calculate concentrations of aldehydes in sample using dilution factor for field samples
STOP

APPENDIX J.4

PAH

METHOD 0010

MODIFIED METHOD 5 SAMPLING TRAIN

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of semivolatile Principal Organic Hazardous Compounds (POHCs) from incineration systems (PHS, 1967). This method also may be used to determine particulate emission rates from stationary sources as per EPA Method 5 (see References at end of this method).

2.0 SUMMARY OF METHOD

2.1 Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high-efficiency glass- or quartz-fiber filter and a packed bed of porous polymeric adsorbent resin. The filter is used to collect organic-laden particulate materials and the porous polymeric resin to adsorb semivolatile organic species. Semivolatile species are defined as compounds with boiling points >100°C.

2.2 Comprehensive chemical analyses of the collected sample are conducted to determine the concentration and identity of the organic materials.

3.0 INTERFERENCES

3.1 Oxides of nitrogen (NO_x) are possible interferents in the determination of certain water-soluble compounds such as dioxane, phenol, and urethane; reaction of these compounds with NO_x in the presence of moisture will reduce their concentration. Other possibilities that could result in positive or negative bias are (1) stability of the compounds in methylene chloride, (2) the formation of water-soluble organic salts on the resin in the presence of moisture, and (3) the solvent extraction efficiency of water-soluble compounds from aqueous media. Use of two or more ions per compound for qualitative and quantitative analysis can overcome interference at one mass. These concerns should be addressed on a compound-by-compound basis before using this method.

4.0 APPARATUS AND MATERIALS

4.1 Sampling train:

4.1.1 A schematic of the sampling train used in this method is shown in Figure 1. This sampling train configuration is adapted from EPA Method 5 procedures, and, as such, the majority of the required equipment

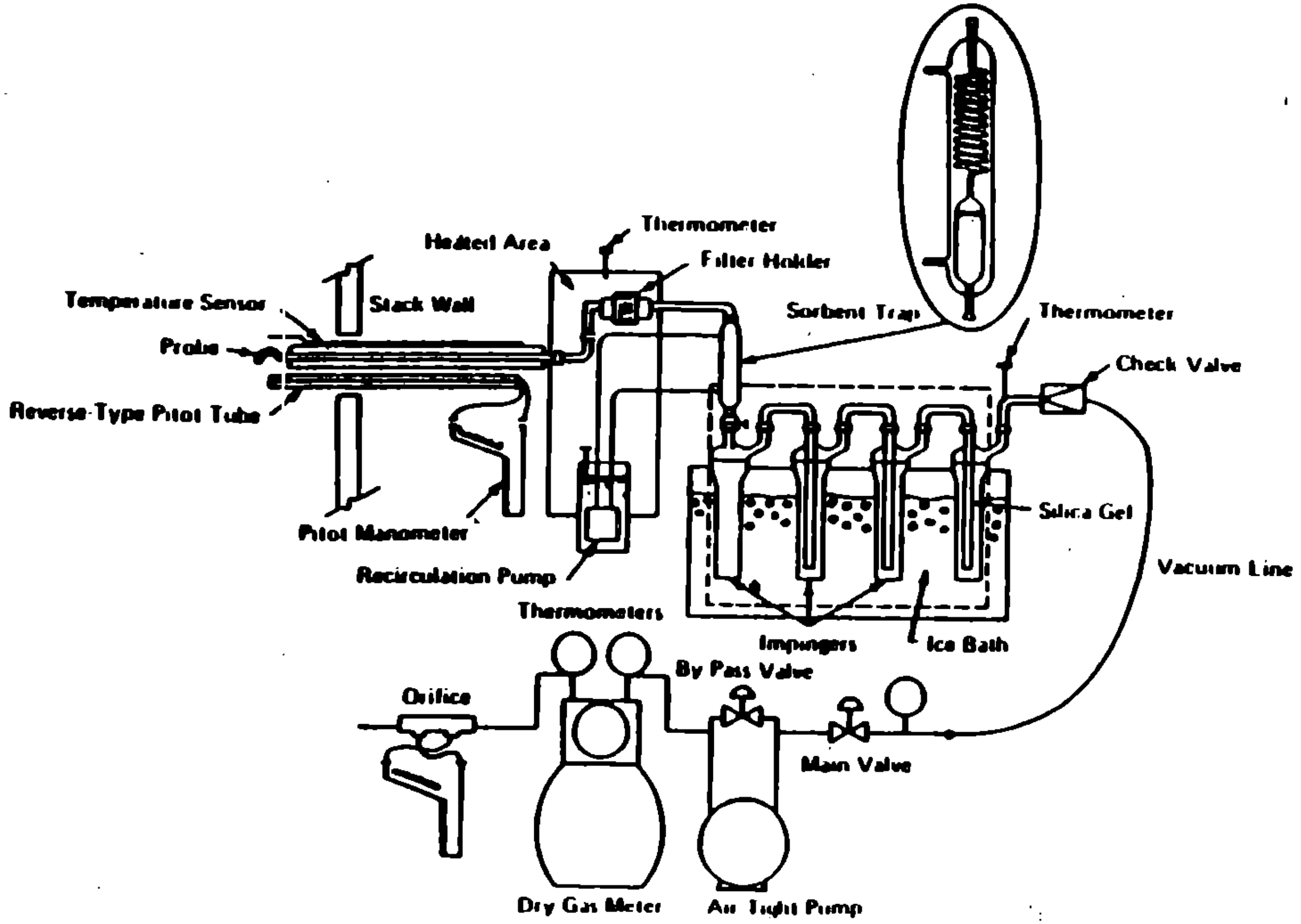


Figure 1. Modified Method 5 Sampling Train.

is identical to that used in EPA Method 5 determinations. The new components required are a condenser coil and a sorbent module, which are used to collect semivolatile organic materials that pass through the glass- or quartz-fiber filter in the gas phase.

4.1.2 Construction details for the basic train components are given in APTD-0581 (see Martin, 1971, in Section 13.0, References); commercial models of this equipment are also available. Specifications for the sorbent module are provided in the following subsections. Additionally, the following subsections list changes to APTD-0581 and identify allowable train configuration modifications.

4.1.3 Basic operating and maintenance procedures for the sampling train are described in APTD-0576 (see Rom, 1972, in Section 13.0, References). As correct usage is important in obtaining valid results, all users should refer to APTD-0576 and adopt the operating and maintenance procedures outlined therein unless otherwise specified. The sampling train consists of the components detailed below.

4.1.3.1 Probe nozzle: Stainless steel (316) or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant I.D. The nozzle shall be buttonhook or elbow design and constructed from seamless tubing (if made of stainless steel). Other construction materials may be considered for particular applications. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g., 0.32-1.27 cm (1/8-1/2 in.), or larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Paragraph 9.1.

4.1.3.2 Probe liner: Borosilicate or quartz-glass tubing with a heating system capable of maintaining a gas temperature of 120 ± 14°C (248 ± 25°F) at the exit end during sampling. (The tester may opt to operate the equipment at a temperature lower than that specified.) Because the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) are considered acceptable. Either borosilicate or quartz-glass probe liners may be used for stack temperatures up to about 480°C (900°F). Quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). (The softening temperature for borosilicate is 820°C (1508°F), and for quartz 1500°C (2732°F).) Water-cooling of the stainless steel sheath will be necessary at temperatures approaching and exceeding 500°C.

4.1.3.3 Pitot tube: Type S, as described in Section 2.1 of EPA Method 2, or other appropriate devices (Vollaro, 1976). The pitot tube shall be attached to the probe to allow constant monitoring of the stack-gas velocity. The impact (high-pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.

4.1.3.4 Differential pressure gauge: Inclined manometer or equivalent device as described in Section 2.2 of EPA Method 2. One manometer shall be used for velocity-head (ΔP) readings and the other for orifice differential pressure (ΔH) readings.

4.1.3.5 Filter holder: Borosilicate glass, with a glass frit filter support and a sealing gasket. The sealing gasket should be made of materials that will not introduce organic material into the gas stream at the temperature at which the filter holder will be maintained. The gasket shall be constructed of Teflon or materials of equal or better characteristics. The holder design shall provide a positive seal against leakage at any point along the filter circumference. The holder shall be attached immediately to the outlet of the cyclone or cyclone bypass.

4.1.3.6 Filter heating system: Any heating system capable of maintaining a temperature of 120 ± 14°C (248 ± 25°F) around the filter holder during sampling. Other temperatures may be appropriate for particular applications. Alternatively, the tester may opt to operate the equipment at temperatures other than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

4.1.3.7 Organic sampling module: This unit consists of three sections, including a gas-conditioning section, a sorbent trap, and a condensate knockout trap. The gas-conditioning system shall be capable of conditioning the gas leaving the back half of the filter holder to a temperature not exceeding 20°C (68°F). The sorbent trap shall be sized to contain approximately 20 g of porous polymeric resin (Rohm and Haas XAD-2 or equivalent) and shall be jacketed to maintain the internal gas temperature at 17 ± 3°C (62.5 ± 5.4°F). The most commonly used coolant is ice water from the impinger ice-water bath, constantly circulated through the outer jacket, using rubber or plastic tubing and a peristaltic pump. The sorbent trap should be outfitted with a glass well or depression, appropriately sized to accommodate a small thermocouple in the trap for monitoring the gas entry temperature. The condensate knockout trap shall be of sufficient size to collect the condensate following gas conditioning. The organic module components shall be oriented to direct the flow of condensate formed vertically downward from the conditioning section, through the adsorbent media, and into the condensate knockout trap. The knockout trap is usually similar in appearance to an empty impinger directly underneath the sorbent module; it may be oversized but should have a shortened center stem (at a minimum, one-half the length of the normal impinger stems) to collect a large volume of condensate without bubbling and overflowing into the impinger train. All surfaces of the organic module wetted by the gas sample shall be fabricated of borosilicate glass, Teflon, or other inert materials. Commercial versions of the complete organic module are not currently available, but may be assembled from commercially available laboratory glassware and a custom-fabricated sorbent trap. Details of two acceptable designs are shown in Figures 2 and 3 (the thermocouple well is shown in Figure 2).

4.1.3.8 Impinger train: To determine the stack-gas moisture content, four 500-mL impingers, connected in series with leak-free ground-glass joints, follow the knockout trap. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (1/2-in.) I.D. glass tube extending about 1.3 cm (1/2 in.) from the bottom of the outer cylinder. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of water or appropriate trapping solution. The third shall be empty or charged with a caustic solution, should the stack gas contain hydrochloric acid (HCl). The fourth shall contain a known weight of silica gel or equivalent desiccant.

4.1.3.9 Metering system: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment, as shown in Figure 1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry-gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems capable of maintaining sampling rates within 10% of isokineticity and of determining sample volumes to within 2% may be used. The metering system must be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates. Sampling trains using metering systems designed for flow rates higher than those described in APTD-0581 and APTD-0576 may be used, provided that the specifications of this method are met.

4.1.3.10 Barometer: Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30-m (100 ft) elevation increase (vice versa for elevation decrease).

4.1.3.11 Gas density determination equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.4 of EPA Method 2), and gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal.

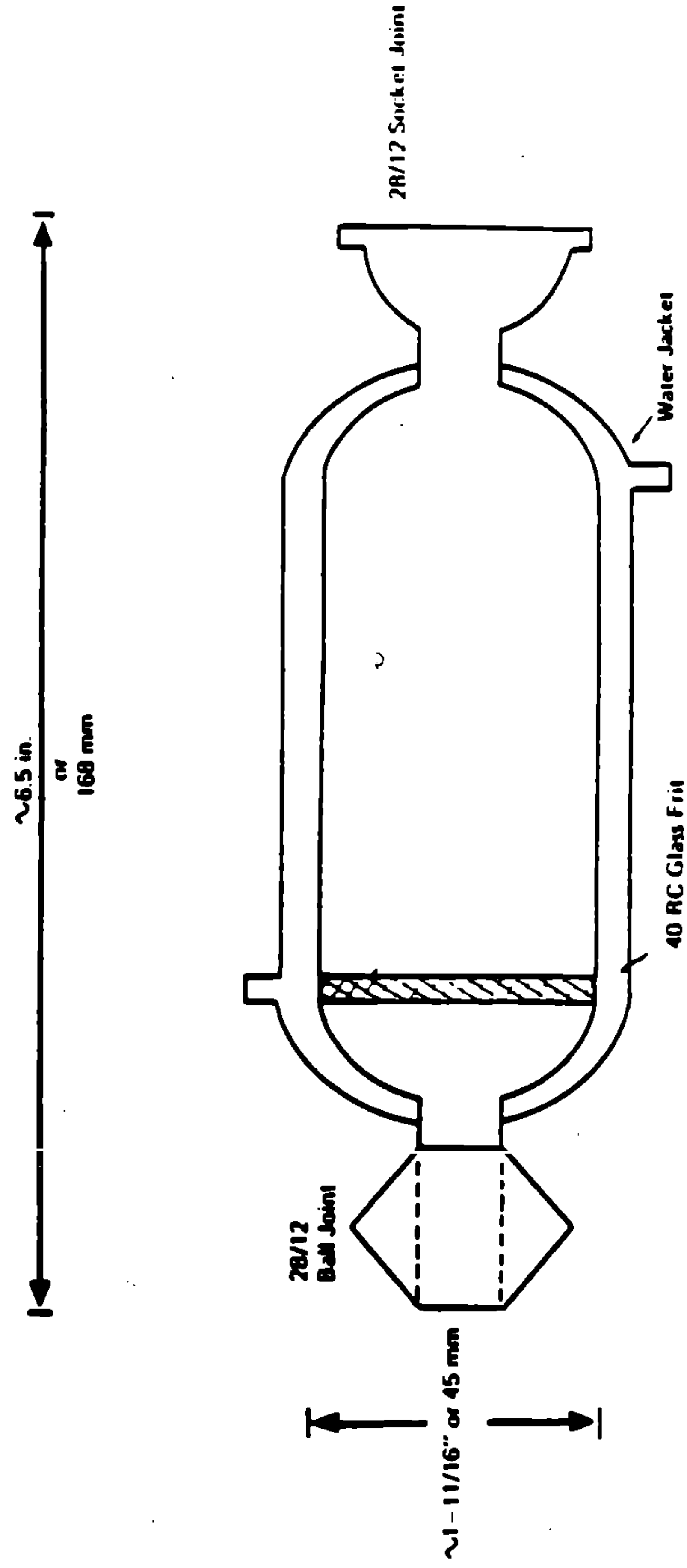


Figure 2. Adsorbent Sampling System.

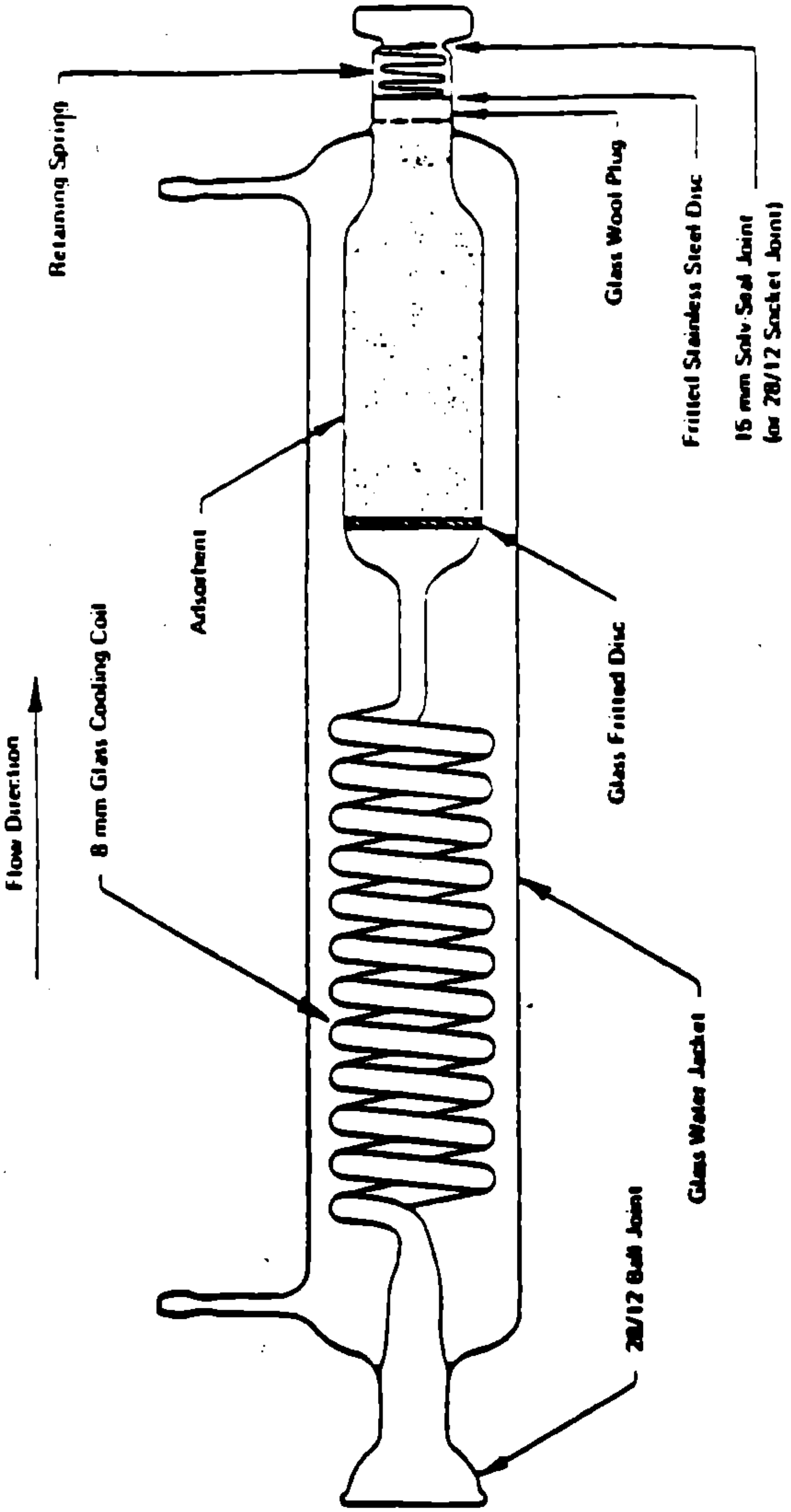


Figure 3. Adsorbent Sampling System.

Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see EPA Method 2, Figure 2-7). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

4.1.3.12 Calibration/field-preparation record: A permanently bound laboratory notebook, in which duplicate copies of data may be made as they are being recorded, is required for documenting and recording calibrations and preparation procedures (i.e., filter and silica gel tare weights, clean XAD-2, quality assurance/quality control check results, dry-gas meter, and thermocouple calibrations, etc.). The duplicate copies should be detachable and should be stored separately in the test program archives.

4.2 Sample Recovery:

4.2.1 **Probe liner:** Probe nozzle and organic module conditioning section brushes; nylon bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel, Teflon, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the organic module conditioning section.

4.2.2 **Wash bottles:** Three. Teflon or glass wash bottles are recommended; polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

4.2.3 **Glass sample storage containers:** Chemically resistant, borosilicate amber and clear glass bottles, 500-mL or 1,000-mL. Bottles should be tinted to prevent action of light on sample. Screw-cap liners shall be either Teflon or constructed so as to be leak-free and resistant to chemical attack by organic recovery solvents. Narrow-mouth glass bottles have been found to exhibit less tendency toward leakage.

4.2.4 **Petri dishes:** Glass, sealed around the circumference with wide (1-in.) Teflon tape, for storage and transport of filter samples.

4.2.5 **Graduated cylinder and/or balances:** To measure condensed water to the nearest 1 mL or 1 g. Graduated cylinders shall have subdivisions not >2 mL. Laboratory triple-beam balances capable of weighing to ±0.5 g or better are required.

4.2.6 **Plastic storage containers:** Screw-cap polypropylene or polyethylene containers to store silica gel.

4.2.7 **Funnel and rubber policeman:** To aid in transfer of silica gel to container (not necessary if silica gel is weighed in field).

4.2.8 Funnels: Glass, to aid in sample recovery.

4.3 Filters: Glass- or quartz-fiber filters, without organic binder, exhibiting at least 99.95% efficiency (<0.05% penetration) on 0.3-um dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Reeve Angel 934 AH or Schleicher and Schwell #3 filters work well under these conditions.

4.4 Crushed ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending on ambient air temperature.

4.5 Stopcock grease: Solvent-insoluble, heat-stable silicone grease. Use of silicone grease upstream of the module is not permitted, and amounts used on components located downstream of the organic module shall be minimized. Silicone grease usage is not necessary if screw-on connectors and Teflon sleeves or ground-glass joints are used.

4.6 Glass wool: Used to plug the unfritted end of the sorbent module. The glass-wool fiber should be solvent-extracted with methylene chloride in a Soxhlet extractor for 12 hr and air-dried prior to use.

5.0 REAGENTS

5.1 Adsorbent resin: Porous polymeric resin (XAD-2 or equivalent) is recommended. These resins shall be cleaned prior to their use for sample collection. Appendix A of this method should be consulted to determine appropriate precleaning procedure. For best results, resin used should not exhibit a blank of higher than 4 mg/kg of total chromatographable organics (TCO) (see Appendix B) prior to use. Once cleaned, resin should be stored in an airtight, wide-mouth amber glass container with a Teflon-lined cap or placed in one of the glass sorbent modules tightly sealed with Teflon film and elastic bands. The resin should be used within 4 wk of the preparation.

5.2 Silica gel: Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hr before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

5.3 Impinger solutions: Distilled organic-free water (Type II) shall be used, unless sampling is intended to quantify a particular inorganic gaseous species. If sampling is intended to quantify the concentration of additional species, the impinger solution of choice shall be subject to Administrator approval. This water should be prescreened for any compounds of interest. One hundred mL will be added to the specified impinger; the third impinger in the train may be charged with a basic solution (1 N sodium hydroxide or sodium acetate) to protect the sampling pump from acidic gases. Sodium acetate should be used when large sample volumes are anticipated because sodium hydroxide will react with carbon dioxide in aqueous media to form sodium carbonate, which may possibly plug the impinger.

5.4 Sample recovery reagents:

5.4.1 **Methylene chloride:** Distilled-in-glass grade is required for sample recovery and cleanup (see Note to 5.4.2 below).

5.4.2 **Methyl alcohol:** Distilled-in-glass grade is required for sample recovery and cleanup.

NOTE: Organic solvents from metal containers may have a high residue blank and should not be used. Sometimes suppliers transfer solvents from metal to glass bottles; thus blanks shall be run prior to field use and only solvents with low blank value (<0.001%) shall be used.

5.4.3 **Water:** Water (Type II) shall be used for rinsing the organic module and condenser component.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Because of complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

6.2 Laboratory preparation:

6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified.

6.2.2 Weigh several 200- to 300-g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

6.2.3 Check filters visually against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass Petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

6.2.4 Desiccate the filters at 20 ± 5.6°C (68 ± 10°F) and ambient pressure for at least 24 hr, and weigh at intervals of at least 6 hr to a constant weight (i.e., <0.5-mg change from previous weighing), recording results to the nearest 0.1 mg. During each weighing the filter must not be exposed for more than a 2-min period to the laboratory atmosphere and relative humidity above 50%. Alternatively (unless otherwise specified by the Administrator), the filters may be oven-dried at 105°C (220°F) for 2-3 hr, desiccated for 2 hr, and weighed.

6.3 Preliminary field determinations:

6.3.1 Select the sampling site and the minimum number of sampling points according to EPA Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. It is recommended that a leak-check of the pitot lines (see EPA Method 2, Section 3.1) be performed. Determine the stack-gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack-gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

6.3.2 Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of EPA Method 2).

6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

6.3.4 A minimum of 3 dscm (105.9 dscf) of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of POHCs from incineration systems. Additional sample volume shall be collected as necessitated by analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.0.

6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus one-half min.

6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-sample volumes. In these cases, the Administrator's approval must first be obtained.

6.4 Preparation of collection train:

6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.

6.4.2 Fill the sorbent trap section of the organic module with approximately 20 g of clean adsorbent resin. While filling, ensure that the trap packs uniformly, to eliminate the possibility of channeling. When freshly cleaned, many adsorbent resins carry a static charge, which will cause clinging to trap walls. This may be minimized by filling the trap in the presence of an antistatic device. Commercial antistatic devices include Model-204 and Model-210 manufactured by the 3M Company, St. Paul, Minnesota.

6.4.3 If an impinger train is used to collect moisture, place 100 mL of water in each of the first two impingers, leave the third impinger empty (or charge with caustic solution, as necessary), and transfer approximately 200-300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

6.4.4 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

6.4.5 When glass liners are used, install the selected nozzle using a Viton-A O-ring when stack temperatures are <260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connecting systems utilizing either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above, or by a leak-free direct mechanical connection. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

6.4.6 Set up the train as in Figure 1. During assembly, do not use any silicone grease on ground-glass joints that are located upstream of the organic module. A very light coating of silicone grease may be used on all ground-glass joints that are located downstream of the organic module, but it should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone-grease contamination. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and the filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack. The organic module condenser must be maintained at a temperature of 17 ± 3°C. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperature.

6.4.7 Place crushed ice around the impingers and the organic module condensate knockout.

6.4.8 Turn on the sorbent module and condenser coil coolant recirculating pump and begin monitoring the sorbent module gas entry temperature. Ensure proper sorbent module gas entry temperature before proceeding and again before any sampling is initiated. It is extremely important that the XAD-2 resin temperature never exceed 50°C (122°F), because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20°C (68°F) for efficient capture of the semivolatile species of interest.

6.4.9 Turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize.

6.5 Leak-check procedures

6.5.1 **Pre-test leak-check:**

6.5.1.1 Because the number of additional intercomponent connections in the Semi-VOST train (over the M5 Train) increases the possibility of leakage, a pre-test leak-check is required.

6.5.1.2 After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 381-mm Hg (15-in. Hg) vacuum.
(NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.)

6.5.1.3 If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first attaching a carbon-filled leak-check impinger (shown in Figure 4) to the inlet of the filter holder (cyclone, if applicable) and then plugging the inlet and pulling a 381-mm Hg (15-in. Hg) vacuum. (Again, a lower vacuum may be used, provided that it is not exceeded during the test.) Then, connect the probe to the train and leak-check at about 25-mm Hg (1-in. Hg) vacuum; alternatively, leak-check the probe with the rest of the sampling train in one step at 381-mm Hg (15-in. Hg) vacuum. Leakage rates in excess of 4% of the average sampling rate or >0.00057 m^3/min (0.02 cfm), whichever is less, are unacceptable.

6.5.1.4 The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with fine-adjust valve fully open and coarse-adjust valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do <u>not</u> reverse direction of the fine-adjust valve; this will cause water to back up into the organic module. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check, as shown below, and start over.

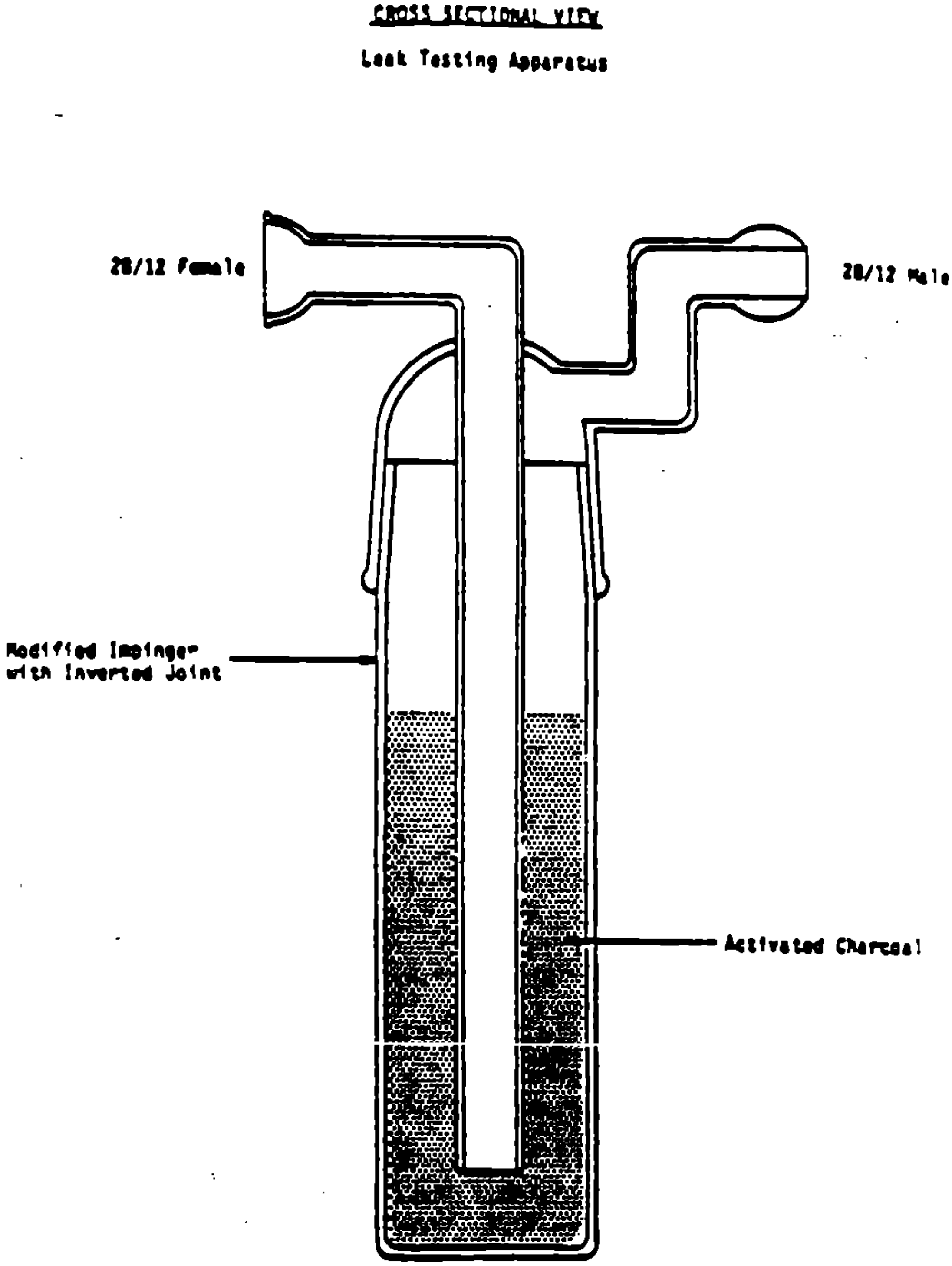


Figure 4. Leak-check impinger.

6.5.1.5 When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable). When the vacuum drops to 127 mm (5 in.) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed. This prevents the water in the impingers from being forced backward into the organic module and silica gel from being entrained backward into the third impinger.

6.5.2 Leak-checks during sampling run:

6.5.2.1 If, during the sampling run, a component (e.g., filter assembly, impinger, or sorbent trap) change becomes necessary, a leak-check shall be conducted immediately after the interruption of sampling and before the change is made. The leak-check shall be done according to the procedure outlined in Paragraph 6.5.1, except that it shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, the tester shall void the sampling run. (It should be noted that any "correction" of the sample volume by calculation by calculation reduces the integrity of the pollutant concentrations data generated and must be avoided.)

6.5.2.2 Immediately after a component change, and before sampling is reinitiated, a leak-check similar to a pre-test leak-check must also be conducted.

6.5.3 Post-test leak-check:

6.5.3.1 A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done with the same procedures as those with the pre-test leak-check, except that it shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate, correct the sample volume (as shown in the calculation section of this method), and consider the data obtained of questionable reliability, or void the sampling run.

6.6 Sampling-train operation:

6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, unless otherwise specified by the Administrator. Maintain a temperature around the filter of 120 ± 14°C (248 ± 25°F) and a gas temperature entering the sorbent trap at a maximum of 20°C (68°F).

6.6.2 For each run, record the data required on a data sheet such as the one shown in Figure 5. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5 at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity-head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

6.6.3 Clean the stack access ports prior to the test run to eliminate the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot-tube coefficient is 0.84 ± 0.02 and the stack-gas equivalent density (dry molecular weight) is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If the stack-gas molecular weight and the pitot-tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps (Shigehara, 1974) are taken to compensate for the deviations.

6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack, to prevent water from backing into the organic module. If necessary, the pump may be turned on with the coarse-adjust valve closed.

6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent unrepresentative dilution of the gas stream.

6.6.6 Traverse the stack cross section, as required by EPA Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

6.6.7 During the test run, make periodic adjustments to keep the temperature around the filter holder and the organic module at the proper levels; add more ice and, if necessary, salt to maintain a temperature of <20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

Plant ____________________
Location ____________________
Operator ____________________
Date ____________________
Run No. ____________________
Sample Box No. ____________________
Meter Box No. ____________________
Meter $\Delta H_@$ ____________________
C Factor ____________________
Pitot Tube Coefficient C_p ____________________

Schematic of Stack Cross Section

Ambient Temperature ____________________
Barometric Pressure ____________________
Assumed Moisture % ____________________
Probe Length, m (ft) ____________________
Nozzle Identification No. ____________________
Average Calibrated Nozzle Diameter, cm (in) ____________________
Probe Heater Setting ____________________
Leak Rate, m^3/min, (cfm) ____________________
Probe Liner Material ____________________
Static Pressure, mm Hg (in. Hg) ____________________
Filter No. ____________________

Traverse Point Number	Sampling Time (θ) min.	Vacuum mm Hg (in. Hg)	Stack Temperature (T_s) °C(°F)	Velocity Head (ΔP_s) mm (in) H_2O	Pressure Differential Across Orifice Meter mm (H_2O) (in H_2O)	Gas Sample Volume m^3 (ft^3)	Gas Sample Temp. at Dry Gas Meter		Filter Holder Temperature °C(°F)	Temperature of Gas Entering Sorbent Trap °C(°F)	Temperature of Gas Leaving Condenser or Last Impinger
							Inlet °C(°F)	Outlet °C(°F)			
Total							Avg.	Avg.			
Average											

Figure 5. Particulate field data.

6.6.8 If the pressure drop across the filter or sorbent trap becomes too high, making isokinetic sampling difficult to maintain, the filter/sorbent trap may be replaced in the midst of a sample run. Using another complete filter holder/sorbent trap assembly is recommended, rather than attempting to change the filter and resin themselves. After a new filter/sorbent trap assembly is installed, conduct a leak-check. The total particulate weight shall include the summation of all filter assembly catches.

6.6.9 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

6.6.10 Note that when two or more trains are used, separate analysis of the front-half (if applicable) organic-module and impinger (if applicable) catches from each train shall be performed, unless identical nozzle sizes were used on all trains. In that case, the front-half catches from the individual trains may be combined (as may the impinger catches), and one analysis of front-half catch and one analysis of impinger catch may be performed.

6.6.11 At the end of the sample run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry-gas meter reading, and conduct a post-test leak-check. Also, leak-check the pitot lines as described in EPA Method 2. The lines must pass this leak-check in order to validate the velocity-head data.

6.6.12 Calculate percent isokineticity (see Section 10.8) to determine whether the run was valid or another test run should be made.

7.0 SAMPLE RECOVERY

7.1 Preparation:

7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling down because this will create a vacuum in the filter holder, drawing water from the impingers into the sorbent module.

7.1.2 Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the open outlet, being careful not to lose any condensate that might be present. Cap the filter inlet.

Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the organic module and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the organic module.

7.1.3 Cap the filter-holder outlet and the inlet to the organic module. Separate the sorbent trap section of the organic module from the condensate knockout trap and the gas-conditioning section. Cap all organic module openings. Disconnect the organic-module knockout trap from the impinger train inlet and cap both of these openings. Ground-glass stoppers, Teflon caps, or caps of other inert materials may be used to seal all openings.

7.1.4 Transfer the probe, the filter, the organic-module components, and the impinger/condenser assembly to the cleanup area. This area should be clean and protected from the weather to minimize sample contamination or loss.

7.1.5 Save a portion of all washing solutions (methanol/methylene chloride, Type II water) used for cleanup as a blank. Transfer 200 mL of each solution directly from the wash bottle being used and place each in a separate, prelabeled glass sample container.

7.1.6 Inspect the train prior to and during disassembly and note any abnormal conditions.

7.2 <u>Sample containers</u>:

7.2.1 **Container no. 1:** Carefully remove the filter from the filter holder and place it in its identified Petri dish container. Use a pair or pairs of tweezers to handle the filter. If it is necessary to fold the filter, ensure that the particulate cake is inside the fold. Carefully transfer to the Petri dish any particulate matter or filter fibers that adhere to the filter-holder gasket, using a dry nylon bristle brush or sharp-edged blade, or both. Label the container and seal with 1-in.-wide Teflon tape around the circumference of the lid.

7.2.2 **Container no. 2:** Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components first with methanol/methylene chloride (1:1 v/v) into a glass container. Distilled water may also be used. Retain a water and solvent blank and analyze in the same manner as with the samples. Perform rinses as follows:

7.2.2.1 Carefully remove the probe nozzle and clean the inside surface by rinsing with the solvent mixture (1:1 v/v methanol/-methylene chloride) from a wash bottle and brushing with a nylon bristle brush. Brush until the rinse shows no visible particles; then make a final rinse of the inside surface with the solvent mix. Brush and rinse the inside parts of the Swagelok fitting with the solvent mix in a similar way until no visible particles remain.

7.2.2.2 Have two people rinse the probe liner with the solvent mix by tilting and rotating the probe while squirting solvent into its upper end so that all inside surfaces will be wetted with solvent. Let the solvent drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container.

7.2.2.3 Follow the solvent rinse with a probe brush. Hold the probe in an inclined position and squirt solvent into the upper end while pushing the probe brush through the probe with a twisting action; place a sample container underneath the lower end of the probe and catch any solvent and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the solvent or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above-prescribed manner at least six times (metal probes have small crevices in which particulate matter can be entrapped). Rinse the brush with solvent and quantitatively collect these washings in the sample container. After the brushing, make a final solvent rinse of the probe as described above.

7.2.2.4 It is recommended that two people work together to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

7.2.2.5 Clean the inside of the front half of the filter holder and cyclone/cyclone flask, if used, by rubbing the surfaces with a nylon bristle brush and rinsing with methanol/methylene chloride (1:1 v/v) mixture. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone and cyclone flask (if applicable). Brush and rinse any particulate material adhering to the inner surfaces of these components into the front-half rinse sample. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents.

7.2.3 Container no. 3: The sorbent trap section of the organic module may be used as a sample transport container, or the spent resin may be transferred to a separate glass bottle for shipment. If the sorbent trap itself is used as the transport container, both ends should be sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon caps may be used. The sorbent trap should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If a separate bottle is used, the spent resin should be quantitatively transferred from the trap into the clean bottle. Resin that adheres to the walls of the trap should be recovered using a rubber policeman or spatula and added to this bottle.

7.2.4 Container no. 4: Measure the volume of condensate collected in the condensate knockout section of the organic module to within ±1 mL by using a graduated cylinder or by weighing to within ±0.5 g using a triple-beam balance. Record the volume or weight of liquid present and note any discoloration or film in the liquid catch. Transfer this liquid to a prelabeled glass sample container. Inspect the back half of the filter housing and the gas-conditioning section of the organic module. If condensate is observed, transfer it to a graduated or weighing bottle and measure the volume, as described above. Add this material to the condensate knockout-trap catch.

7.2.5 Container no. 5: All sampling train components located between the high-efficiency glass- or quartz-fiber filter and the first wet impinger or the final condenser system (including the heated Teflon line connecting the filter outlet to the condenser) should be thoroughly rinsed with methanol/methylene chloride (1:1 v/v) and the rinsings combined. This rinse shall be separated from the condensate. If the spent resin is transferred from the sorbent trap to a separate sample container for transport, the sorbent trap shall be thoroughly rinsed until all sample-wetted surfaces appear clean. Visible films should be removed by brushing. Whenever train components are brushed, the brush should be subsequently rinsed with solvent mixture and the rinsings added to this container.

7.2.6 Container no. 6: Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere strongly to the impinger wall. Because the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the container and its contents to 0.5 g or better.

7.3 Impinger water:

7.3.1 Make a notation of any color or film in the liquid catch. Measure the liquid in the first three impingers to within ±1 mL by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

7.3.2 Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Paragraph 4.1.3.7). Amber glass containers should be used for storage of impinger catch, if required.

7.3.3 If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

7.4 Sample preparation for shipment: Prior to shipment, recheck all sample containers to ensure that the caps are well secured. Seal the lids of all containers around the circumference with Teflon tape. Ship all liquid samples upright on ice and all particulate filters with the particulate catch facing upward. The particulate filters should be shipped unrefrigerated.

8.0 ANALYSIS

8.1 Sample preparation:

8.1.1 General: The preparation steps for all samples will result in a finite volume of concentrated solvent. The final sample volume (usually in the 1- to 10-mL range) is then subjected to analysis by GC/MS. All samples should be inspected and the appearance documented. All samples are to be spiked with surrogate standards as received from the field prior to any sample manipulations. The spike should be at a level equivalent to 10 times the MDL when the solvent is reduced in volume to the desired level (i.e., 10 mL). The spiking compounds should be the stable isotopically labeled analog of the compounds of interest or a compound that would exhibit properties similar to the compounds of interest, be easily chromatographed, and not interfere with the analysis of the compounds of interest. Suggested surrogate spiking compounds are: deuterated naphthalene, chrysene, phenol, nitrobenzene, chlorobenzene, toluene, and carbon-13-labeled pentachlorophenol.

8.1.2 Condensate: The "condensate" is the moisture collected in the first impinger following the XAD-2 module. Spike the condensate with the surrogate standards. The volume is measured and recorded and then transferred to a separatory funnel. The pH is to be adjusted to pH 2 with 6 N sulfuric acid, if necessary. The sample container and graduated cylinder are sequentially rinsed with three successive 10-mL aliquots of the extraction solvent and added to the separatory funnel. The ratio of solvent to aqueous sample should be maintained at 1:3. Extract the sample by vigorously shaking the separatory funnel for 5 min. After complete separation of the phases, remove the solvent and transfer to a Kuderna-Danish concentrator (K-D), filtering through a bed of precleaned, dry sodium sulfate. Repeat the extraction step two additional times. Adjust the pH to 11 with 6 N sodium hydroxide and reextract combining the acid and base extracts. Rinse the sodium sulfate into the K-D with fresh solvent and discard the desiccant. Add Teflon boiling chips and concentrate to 10 mL by reducing the volume to slightly less than 10 mL and then bringing to volume with fresh solvent. In order to achieve the necessary detection limit, the sample volume can be further reduced to 1 mL by using a micro column K-D or nitrogen blow-down. Should the sample start to exhibit precipitation, the concentration step should be stopped and the sample redissolved with fresh solvent taking the volume to some finite amount. After adding a standard (for the purpose of quantitation by GC/MS), the sample is ready for analysis, as discussed in Paragraph 8.2.

8.1.3 **Impinger:** Spike the sample with the surrogate standards; measure and record the volume and transfer to a separatory funnel. Proceed as described in Paragraph 8.1.2.

8.1.4 **XAD-2:** Spike the resin directly with the surrogate standards. Transfer the resin to the all-glass thimbles by the following procedure (care should be taken so as not to contaminate the thimble by touching it with anything other than tweezers or other solvent-rinsed mechanical holding devices). Suspend the XAD-2 module directly over the thimble. The glass frit of the module (see Figure 2) should be in the up position. The thimble is contained in a clean beaker, which will serve to catch the solvent rinses. Using a Teflon squeeze bottle, flush the XAD-2 into the thimble. Thoroughly rinse the glass module with solvent into the beaker containing the thimble. Add the XAD-2 glass-wool plug to the thimble. Cover the XAD-2 in the thimble with a precleaned glass-wool plug sufficient to prevent the resin from floating into the solvent reservoir of the extractor. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. If a question arises concerning the completeness of the extraction, a second extraction, without a spike, is advised. The thimble is placed in the extractor and the rinse solvent contained in the beaker is added to the solvent reservoir. Additional solvent is added to make the reservoir approximately two-thirds full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle 5-6 times per hr. Extract the resin for 16 hr. Transfer the solvent and three 10-mL rinses of the reservoir to a K-D and concentrate as described in Paragraph 8.1.2.

8.1.5 **Particulate filter (and cyclone catch):** If particulate loading is to be determined, weigh the filter (and cyclone catch, if applicable). The particulate filter (and cyclone catch, if applicable) is transferred to the glass thimble and extracted simultaneously with the XAD-2 resin.

8.1.6 **Train solvent rinses:** All train rinses (i.e., probe, impinger, filter housing) using the extraction solvent and methanol are returned to the laboratory as a single sample. If the rinses are contained in more than one container, the intended spike is divided equally among the containers proportioned from a single syringe volume. Transfer the rinse to a separatory funnel and add a sufficient amount of organic-free water so that the methylene chloride becomes immiscible and its volume no longer increases with the addition of more water. The extraction and concentration steps are then performed as described in Paragraph 8.1.2.

8.2 Sample analysis:

8.2.1 The primary analytical tool for the measurement of emissions from hazardous waste incinerators is GC/MS using fused-silica capillary GC columns, as described in Method 8270 in Chapter Four of this manual. Because of the nature of GC/MS instrumentation and the cost associated with sample analysis, prescreening of the sample extracts by gas chromatography/flame ionization detection (GC/FID) or with electron capture (GC/ECD) is encouraged. Information regarding the complexity and concentration level of a sample prior to GC/MS analysis can be of enormous help. This information can be obtained by using either capillary columns or less expensive packed columns. However, the FID screen should be performed with a column similar to that used with the GC/MS. Keep in mind that GC/FID has a slightly lower detection limit than GC/MS and, therefore, that the concentration of the sample can be adjusted either up or down prior to analysis by GC/MS.

8.2.2 The mass spectrometer will be operated in a full scan (40-450) mode for most of the analyses. The range for which data are acquired in a GC/MS run will be sufficiently broad to encompass the major ions, as listed in Chapter Four, Method 8270, for each of the designated POHCs in an incinerator effluent analysis.

8.2.3 For most purposes, electron ionization (EI) spectra will be collected because a majority of the POHCs give reasonable EI spectra. Also, EI spectra are compatible with the NBS Library of Mass Spectra and other mass spectral references, which aid in the identification process for other components in the incinerator process streams.

8.2.4 To clarify some identifications, chemical ionization (CI) spectra using either positive ions or negative ions will be used to elucidate molecular-weight information and simplify the fragmentation patterns of some compounds. In no case, however, should CI spectra alone be used for compound identification. Refer to Chapter Four, Method 8270, for complete descriptions of GC conditions, MS conditions, and quantitative and quantitative identification.

9.0 CALIBRATION

9.1 Probe nozzle: Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.[illegible] in.). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

9.2 Pitot tube: The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of EPA Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked, dented, or corroded and if it meets design and intercomponent spacing specifications.

9.3 Metering system:

9.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: Make a 10-min calibration run at 0.00057 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

9.3.2 After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). The vacuum shall be set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

9.3.3 Leak-check of metering system: That portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 6): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13-18 cm (5-7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks, if present, must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series shall be voided or calculations for test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

9.4 Probe heater: The probe-heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

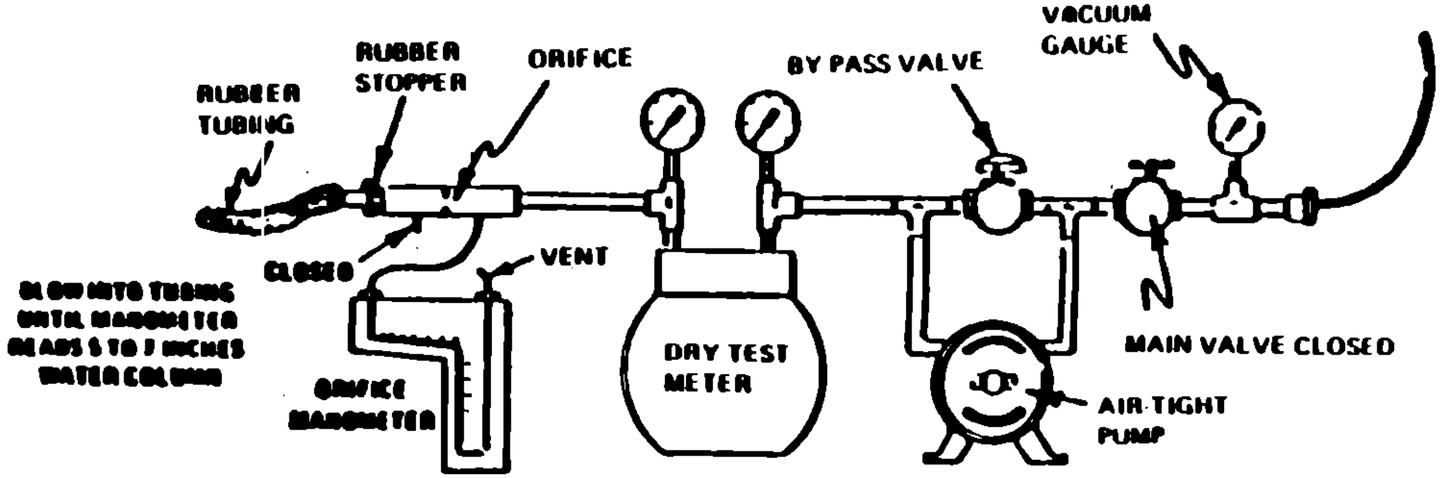


Figure 6. Leak check of meter box.

9.5 Temperature gauges: Each thermocouple must be permanently and uniquely marked on the casting; all mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change >1.5%.

9.5.1 **Impinger, organic module, and dry-gas meter thermocouples:** For the thermocouples used to measure the temperature of the gas leaving the impinger train and the XAD-2 resin bed, three-point calibration at ice-water, room-air, and boiling-water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2°C (3.6°F) with those of the absolute value of the reference thermometer.

9.5.2 **Probe and stack thermocouple:** For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice-water, boiling-water, and hot-oil-bath temperatures must be performed; it is recommended that room-air temperature be added, and that the thermometer and the thermocouple agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

9.6 Barometer: Adjust the barometer initially and before each test series to agree to within ±25 mm Hg (0.1 in. Hg) of the mercury barometer or the corrected barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

9.7 Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class-S standard weights; the weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

10.0 CALCULATIONS

10.1 Carry out calculations. Round off figures after the final calculation to the correct number of significant figures.

10.2 Nomenclature:

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_d = Type S pitot tube coefficient (nominally 0.84 ± 0.02), dimensionless.

I = Percent of isokinetic sampling.

L_a = Maximum acceptable leakage rate for a leak-check, either pre-test or following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak-check conducted prior to the "i^{th}" component change (i = 1, 2, 3...n) m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

M_d = Stack-gas dry molecular weight, g/g-mole (lb/lb-mole).

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack-gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m^3/K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).

T_m = Absolute average dry-gas meter temperature (see Figure 6), K (°R).

T_s = Absolute average stack-gas temperature (see Figure 6), K (°R).

T_{std} = Standard absolute temperature, 293K (528°R).

V_{lc} = Total volume of liquid collected in the organic module condensate knockout trap, the impingers, and silica gel, mL.

V_m = Volume of gas sample as measured by dry-gas meter, dscm (dscf).

$V_{m(std)}$ = Volume of gas sample measured by the dry-gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

V_s = Stack-gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

γ = Dry-gas-meter calibration factor, dimensionless.

ΔH = Average pressure differential across the orifice meter (see Figure 2), mm H_2O (in. H_2O).

ρ_w = Density of water, 0.9982 g/mL (0.002201 lb/mL).

θ = Total sampling time, min.

θ_1 = Sampling time interval from the beginning of a run until the first component change, min.

θ_i = Sampling time interval between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p = Sampling time interval from the final (nth) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

10.3 Average dry-gas-meter temperature and average orifice pressure drop: See data sheet (Figure 5, above).

10.4 Dry-gas volume: Correct the sample measured by the dry-gas meter to standard conditions (20°C, 760 mm Hg [68°F, 29.92 in. Hg]) by using Equation 1:

$$V_{m(std)} = V_m Y \frac{T_{std}}{T_m} \frac{P_{bar} + \Delta H/13.6}{P_{std}} = K_1 V_m Y \frac{P_{bar} + \Delta H/13.6}{T_m} \qquad (1)$$

where:

K_1 = 0.3858 K/mm Hg for metric units, or
K_1 = 17.64°R/in. Hg for English units.

It should be noted that Equation 1 can be used as written, unless the leakage rate observed during any of the mandatory leak-checks (i.e., the post-test leak-check or leak-checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 1 must be modified as follows:

a. Case I (no component changes made during sampling run): Replace V_m in Equation 1 with the expression:

$$V_m - (L_p - L_a)$$

b. Case II (one or more component changes made during the sampling run): Replace V_m in Equation 1 by the expression:

$$V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_1 or L_p) that exceed L_a.

10.5 Volume of water vapor:

$$V_{w(std)} = V_{lc} \frac{P_w}{M_w} \frac{RT_{std}}{P_{std}} = K_2 V_{lc} \qquad (2)$$

where:

K_2 = 0.001333 m^3/mL for metric units, or
K_2 = 0.04707 ft^3/mL for English units.

10.6 Moisture content:

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad (3)$$

NOTE: In saturated or water-droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 3) and a second from the assumption of saturated conditions. The lower of the two values of B_w shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note to Section 1.2 of Method 4. For the purposes of this method, the average stack-gas temperature from Figure 6 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ±1°C (2°F).

10.7 Conversion factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

10.8 Isokinetic variation:

10.8.1 **Calculation from raw data:**

$$I = \frac{100\ T_s[K_3F_{1c} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{60\theta V_s P_s A_n} \qquad (4)$$

where:

K_3 = 0.003454 mm Hg-m^3/mL-K for metric units, or
K_3 = 0.002669 in. Hg-ft^3/mL-°R for English units.

10.8.2 **Calculation for intermediate values:**

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(1-B_{ws})} \qquad (5)$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta(1-B_{ws})}$$

where:

K_4 = 4.320 for metric units, or
K_4 = 0.09450 for English units.

10.8.3 **Acceptable results:** If 90% $\leq$ I $\leq$ 110%, the results are acceptable. If the results are low in comparison with the standard and I is beyond the acceptable range, or if I is less than 90%, the Administrator may opt to accept the results.

10.9 To determine the minimum sample volume that shall be collected, the following sequence of calculations shall be used.

10.9.1 From prior analysis of the waste feed, the concentration of POHCs introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the maximum amount of POHC allowed to be present in the effluent. This may be expressed as:

$$\frac{(WF)\ (POHC_i\ conc)}{100}\ \frac{(100-\%DRE)}{100} = Max\ POHC_i\ Mass \qquad (6)$$

where:

WF = mass flow rate of waste feed per hr, g/hr (lb/hr).

$POHC_i$ = concentration of Principal Organic Hazardous Compound (wt %) introduced into the combustion process.

DRE = percent Destruction and Removal Efficiency required.

Max POHC = mass flow rate (g/hr [lb/hr]) of POHC emitted from the combustion source.

10.9.2 The average discharge concentration of the POHC in the effluent gas is determined by comparing the Max POHC with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary Method 1-4 determinations:

$$\frac{\text{Max POHC}_i \text{ Mass}}{DV_{eff(std)}} = \text{Max POHC}_i \text{ conc} \qquad (7)$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf).

$POHC_i$ conc = anticipated concentration of the POHC in the exhaust gas stream, g/dscm (lb/dscf).

10.9.3 In making this calculation, it is recommended that a safety margin of at least ten be included:

$$\frac{LDL_{POHC} \times 10}{POHC_{i\ conc}} = V_{TBC} \qquad (8)$$

where:

LDL_{POHC} = detectable amount of POHC in entire sampling train.
NOTE: The whole extract from an XAD-2 cartridge is seldom if ever, injected at once. Therefore, if aliquoting factors are involved, the LDL_{POHC} is not the same as the analytical (or column) detection limit.

V_{TBC} = minimum dry standard volume to be collected at dry-gas meter.

10.10 Concentration of any given POHC in the gaseous emissions of a combustion process:

1) Multiply the concentration of the POHC as determined in Method 8270 by the final concentration volume, typically 10 mL.

C_{POHC} (ug/mL) x sample volume (mL) = amount (ug) of POHC in sample (9) where:

C_{POHC} = concentration of POHC as analyzed by Method 8270.

2) Sum the amount of POHC found in all samples associated with a single train.

Total (ug) = XAD-2 (ug) + condensate (ug) + rinses (ug) + impinger (ug) (10)

3) Divide the total ug found by the volume of stack gas sampled (m^3).

(Total ug)/(train sample volume) = concentration of POHC (ug/m^3) (11)

11.0 QUALITY CONTROL

11.1 Sampling: See EPA Manual 600/4-77-027b for Method 5 quality control.

11.2 Analysis: The quality assurance program required for this study includes the analysis of field and method blanks, procedure validations, incorporation of stable labeled surrogate compounds, quantitation versus stable labeled internal standards, capillary column performance checks, and external performance tests. The surrogate spiking compounds selected for a particular analysis are used as primary indicators of the quality of the analytical data for a wide range of compounds and a variety of sample matrices. The assessment of combustion data, positive identification, and quantitation of the selected compounds are dependent on the integrity of the samples received and the precision and accuracy of the analytical methods employed. The quality assurance procedures for this method are designed to monitor the performance of the analytical method and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

11.2.1 Field Blanks: Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters, and resin cartridges. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling area, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual test train). The filter housing and probe of the blank train will be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train.

11.2.2 Method blanks: A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

11.2.3 Refer to Method 8270 for additional quality control considerations.

12.0 METHOD PERFORMANCE

12.1 Method performance evaluation: Evaluation of analytical procedures for a selected series of compounds must include the sample-preparation procedures and each associated analytical determination. The analytical procedures should be challenged by the test compounds spiked at appropriate levels and carried through the procedures.

12.2 Method detection limit: The overall method detection limits (lower and upper) must be determined on a compound-by-compound basis because different compounds may exhibit different collection, retention, and extraction efficiencies as well as instrumental minimum detection limit (MDL). The method detection limit must be quoted relative to a given sample volume. The upper limits for the method must be determined relative to compound retention volumes (breakthrough).

12.3 Method precision and bias: The overall method precision and bias must be determined on a compound-by-compound basis at a given concentration level. The method precision value would include a combined variability due to sampling, sample preparation, and instrumental analysis. The method bias would be dependent upon the collection, retention, and extraction efficiency of the train components. From evaluation studies to date using a dynamic spiking system, method biases of -13% and -16% have been determined for toluene and 1,1,2,2-tetrachloroethane, respectively. A precision of 19.9% was calculated from a field test data set representing seven degrees of freedom which resulted from a series of paired, unspiked Semivolatile Organic Sampling trains (Semi-VOST) sampling emissions from a hazardous waste incinerator.

13.0 REFERENCES

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METHOD 0010, APPENDIX A

PREPARATION OF XAD-2 SORBENT RESIN

1.0 SCOPE AND APPLICATION

1.1 XAD-2 resin as supplied by the manufacturer is impregnated with a bicarbonate solution to inhibit microbial growth during storage. Both the salt solution and any residual extractable monomer and polymer species must be removed before use. The resin is prepared by a series of water and organic extractions, followed by careful drying.

2.0 EXTRACTION

2.1 Method 1: The procedure may be carried out in a giant Soxhlet extractor. An all-glass thimble containing an extra-coarse frit is used for extraction of XAD-2. The frit is recessed 10-15 mm above a crenellated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass-wool plug and stainless steel screen because it floats on methylene chloride. This process involves sequential extraction in the following order.

Solvent	Procedure
Water	Initial rinse: Place resin in a beaker, rinse once with Type II water, and discard. Fill with water a second time, let stand overnight, and discard.
Water	Extract with H_2O for 8 hr.
Methyl alcohol	Extract for 22 hr.
Methylene chloride	Extract for 22 hr.
Methylene chloride (fresh)	Extract for 22 hr.

2.2 Method 2:

2.2.1 As an alternative to Soxhlet extraction, a continuous extractor has been fabricated for the extraction sequence. This extractor has been found to be acceptable. The particular canister used for the apparatus shown in Figure A-1 contains about 500 g of finished XAD-2. Any size may be constructed; the choice is dependent on the needs of the sampling programs. The XAD-2 is held under light spring tension between a pair of coarse and fine screens. Spacers under the bottom screen allow for even distribution of clean solvent. The three-necked flask should be of sufficient size (3-liter in this case) to hold solvent

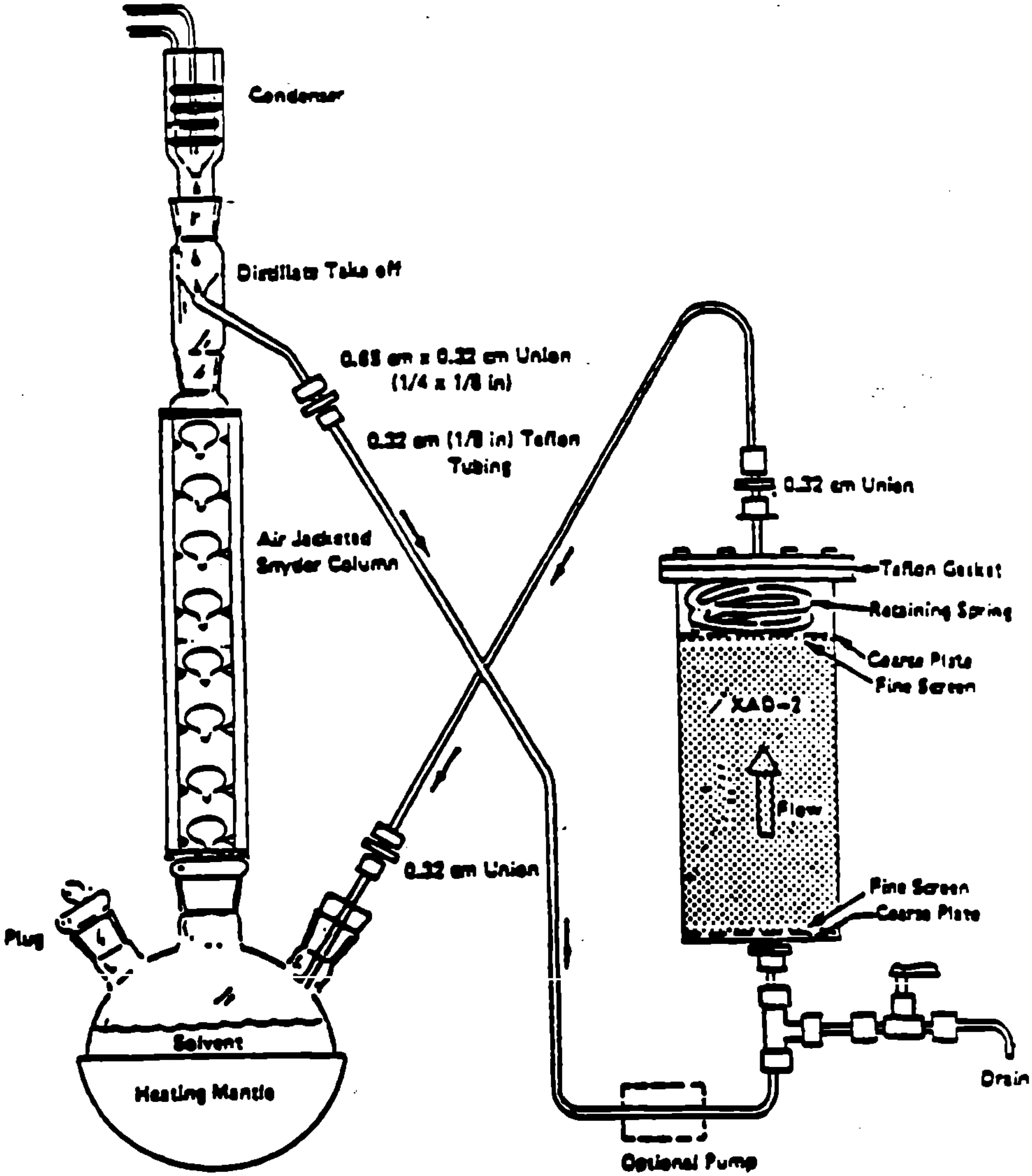

Figure A-1. XAD-2 cleanup extraction apparatus.

equal to twice the dead volume of the XAD-2 canister. Solvent is refluxed through the Snyder column, and the distillate is continuously cycled up through the XAD-2 for extraction and returned to the flask. The flow is maintained upward through the XAD-2 to allow maximum solvent contact and prevent channeling. A valve at the bottom of the canister allows removal of solvent from the canister between changes.

2.2.2 Experience has shown that it is very difficult to cycle sufficient water in this mode. Therefore the aqueous rinse is accomplished by simply flushing the canister with about 20 liters of distilled water. A small pump may be useful for pumping the water through the canister. The water extraction should be carried out at the rate of about 20-40 mL/min.

2.2.3 After draining the water, subsequent methyl alcohol and methylene chloride extractions are carried out using the refluxing apparatus. An overnight or 10- to 20-hr period is normally sufficient for each extraction.

2.2.4 All materials of construction are glass, Teflon, or stainless steel. Pumps, if used, should not contain extractable materials. Pumps are not used with methanol and methylene chloride.

3.0 DRYING

3.1 After evaluation of several methods of removing residual solvent, a fluidized-bed technique has proved to be the fastest and most reliable drying method.

3.2 A simple column with suitable retainers, as shown in Figure A-2, will serve as a satisfactory column. A 10.2-cm (4-in.) Pyrex pipe 0.6 m (2 ft) long will hold all of the XAD-2 from the extractor shown in Figure A-1 or the Soxhlet extractor, with sufficient space for fluidizing the bed while generating a minimum resin load at the exit of the column.

3.3 Method 1: The gas used to remove the solvent is the key to preserving the cleanliness of the XAD-2. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has routinely proved to be a reliable source of large volumes of gas free from organic contaminants. The liquid nitrogen cylinder is connected to the column by a length of precleaned 0.95-cm (3/8-in.) copper tubing, coiled to pass through a heat source. As nitrogen is bled from the cylinder, it is vaporized in the heat source and passes through the column. A convenient heat source is a water bath heated from a steam line. The final nitrogen temperature should only be warm to the touch and not over 40°C. Experience has shown that about 500 g of XAD-2 may be dried overnight by consuming a full 160-liter cylinder of liquid nitrogen.

3.4 Method 2: As a second choice, high-purity tank nitrogen may be used to dry the XAD-2. The high-purity nitrogen must first be passed through a bed

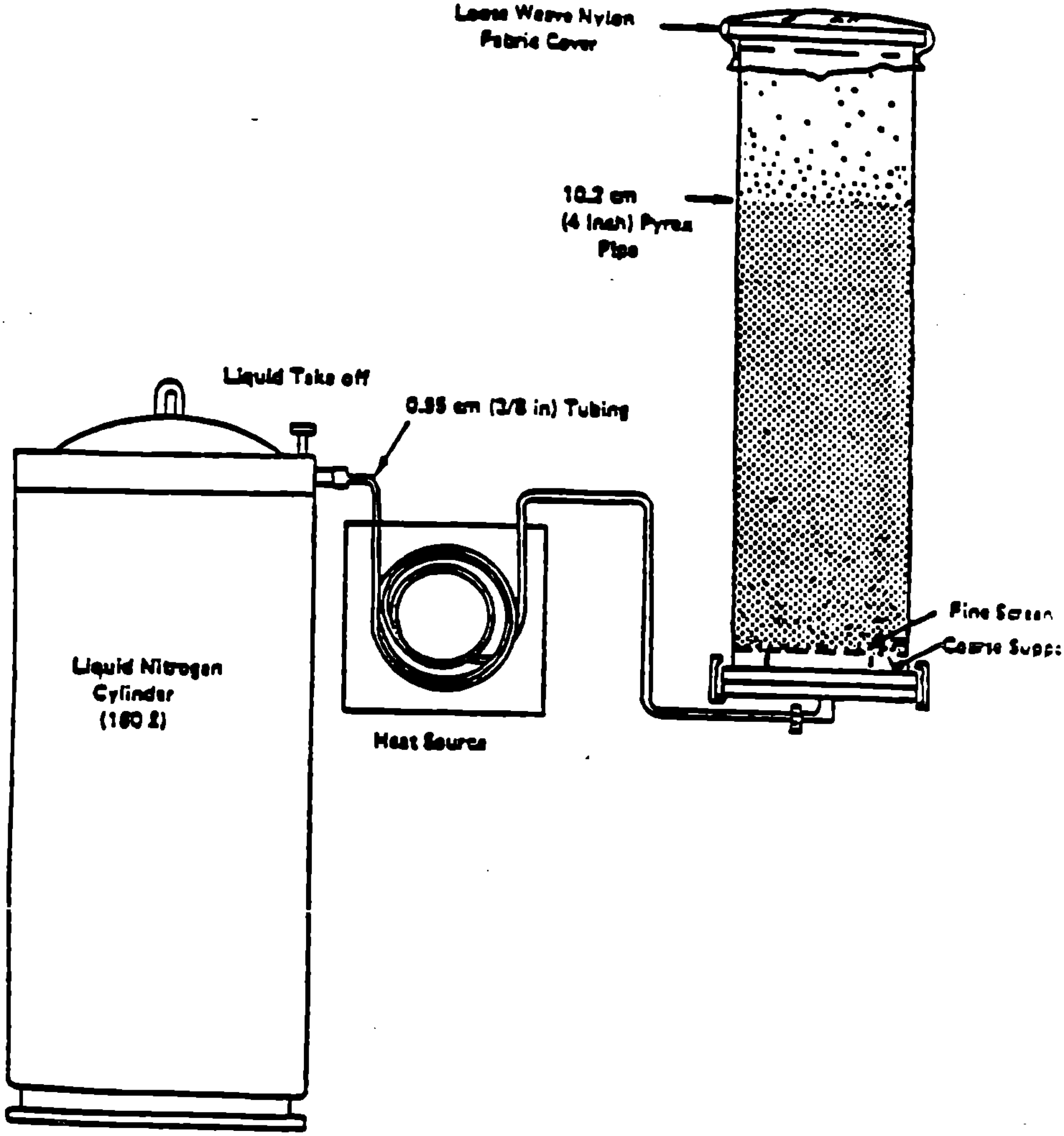

Figure A-2. XAD-2 fluidized-bed drying apparatus.

of activated charcoal approximately 150 mL in volume. With either type of drying method, the rate of flow should gently agitate the bed. Excessive fluidization may cause the particles to break up.

4.0 QUALITY CONTROL PROCEDURES

4.1 For both Methods 1 and 2, the quality control results <u>must</u> be reported for the batch. The batch must be reextracted if the residual extractable organics are >20 ug/mL by TCO analysis or the gravimetric residue is >0.5 mg/20 g XAD-2 extracted. (See also section 5.1, Method 0010.)

4.2 Four control procedures are used with the final XAD-2 to check for (1) residual methylene chloride, (2) extractable organics (TCO), (3) specific compounds of interest as determined by GC/MS, as described in Section 4.5 below, and (4) residue (GRAV).

4.3 <u>Procedure for residual methylene chloride</u>:

4.3.1 **Description:** A 1±0.1-g sample of dried resin is weighed into a small vial, 3 mL of toluene are added, and the vial is capped and well shaken. Five uL of toluene (now containing extracted methylene chloride) are injected into a gas chromatograph, and the resulting integrated area is compared with a reference standard. The reference solution consists of 2.5 uL of methylene chloride in 100 mL of toluene, simulating 100 ug of residual methylene chloride on the resin. The acceptable maximum content is 1,000 ug/g resin.

4.3.2 **Experimental:** The gas chromatograph conditions are as follows:

6-ft x 1/8-in. stainless steel column containing 10% OV-101 on 100/120 Supelcoport;

Helium carrier at 30 mL/min;

FID operated on 4 x 10^{-11} A/mV;

Injection port temperature: 250°C;

Detector temperature: 305°C;

Program: 30°C(4 min) <u>40°C/min</u> 250°C (hold); and

Program terminated at 1,000 sec.

4.4 <u>Procedure for residual extractable organics</u>:

4.4.1 **Description:** A 20±0.1-g sample of cleaned, dried resin is weighed into a precleaned alundum or cellulose thimble which is plugged with cleaned glass wool. (Note that 20 g of resin will fill a thimble, and the resin will float out unless well plugged.) The thimble containing the resin is extracted for 24 hr with 200-mL of pesticide- grade methylene chloride (Burdick and Jackson pesticide-grade or equivalent purity). The 200-mL extract is reduced in volume to 10-mL using a Kuderna-Danish concentrator and/or a nitrogen evaporation stream. Five uL of that solution are analyzed by gas chromatography using the TCO analysis procedure. The concentrated solution should not contain >20 ug/mL of TCO extracted from the XAD-2. This is equivalent to 10 ug/g of TCO in the XAD-2 and would correspond to 1.3 mg of TCO in the extract of the 130-g XAD-2 module. Care should be taken to correct the TCO data for a solvent blank prepared (200 mL reduced to 10 mL) in a similar manner.

4.4.2 **Experimental**: Use the TCO analysis conditions described in the revised Level 1 manual (EPA 600/7-78-201).

4.5 GC/MS Screen: The extract, as prepared in paragraph 4.4.1, is subjected to GC/MS analysis for each of the individual compounds of interest. The GC/MS procedure is described in Chapter Four, Method 8270. The extract is screened at the MDL of each compound. The presence of any compound at a concentration >25 ug/mL in the concentrated extract will require the XAD-2 to be recleaned by repeating the methylene chloride step.

4.6 Methodology for residual gravimetric determination: After the TCO value and GC/MS data are obtained for the resin batch by the above procedures, dry the remainder of the extract in a tared vessel. There must be <0.5 mg residue registered or the batch of resin will have to be extracted with fresh methylene chloride again until it meets this criterion. This level corresponds to 25 ug/g in the XAD-2, or about 3.25 mg in a resin charge of 130 g.

METHOD 0010, APPENDIX B

TOTAL CHROMATOGRAPHABLE ORGANIC MATERIAL ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 In this procedure, gas chromatography is used to determine the quantity of lower boiling hydrocarbons (boiling points between 90° and 300°C) in the concentrates of all organic solvent rinses, XAD-2 resin and LC fractions - when Method 1 is used (see References, Method 0010) - encountered in Level 1 environmental sample analyses. Data obtained using this procedure serve a twofold purpose. First, the total quantity of the lower boiling hydrocarbons in the sample is determined. Then whenever the hydrocarbon concentrations in the original concentrates exceed 75 ug/m^3, the chromatography results are reexamined to determine the amounts of individual species.

The extent of compound identification is limited to representing all materials as normal alkanes based upon comparison of boiling points. Thus the method is not qualitative. In a similar manner, the analysis is semiquantitative; calibrations are prepared using only one hydrocarbon. They are replicated but samples routinely are not.

1.2 Application: This procedure applies solely to the Level 1 C7-C16 gas chromatographic analysis of concentrates of organic extracts, neat liquids, and of LC fractions. Throughout the procedure, it is assumed the analyst has been given a properly prepared sample.

1.3 Sensitivity: The sensitivity of this procedure, defined as the slope of a plot of response versus concentration, is dependent on the instrument and must be verified regularly. TRW experience indicates the nominal range is of the order of 77 uV·V·sec·uL/ng of n-heptane and 79 uV·sec·ul/ng of n-hexadecane. The instrument is capable of perhaps one hundredfold greater sensitivity. The level specified here is sufficient for Level 1 analysis.

1.4 Detection limit: The detection limit of this procedure as written is 1.3 ng/uL for a 1 uL injection of n-decane. This limit is arbitrarily based on defining the minimum detectable response as 100 uv·sec. This is an easier operational definition than defining the minimum detection limit to be that amount of material which yields a signal twice the noise level.

1.5 Range: The range of the procedure will be concentrations of 1.3 ng/uL and greater.

1.6 Limitations

1.6.1 Reporting limitations: It should be noted that a typical environmental sample will contain compounds which: (a) will not elute in the specified boiling ranges and thus will not be reported, and/or (b) will not elute from the column at all and thus will not be reported. Consequently, the organic content of the sample as reported is a lower bound and should be regarded as such.

1.6.2 **Calibration limitations:** Quantitation is based on calibration with n-decane. Data should therefore be reported as, e.g., mg C8/m^3 as n-decane. Since response varies linearly with carbon number (over a wide range the assumption may involve a 20% error), it is clear that heptane (C7) detected in a sample and quantitated as decane will be overestimated. Likewise, hexadecane (C16) quantitated as decane will be underestimated. From previous data, it is estimated the error involved is on the order of 6-7%.

1.6.3 **Detection limitations:** The sensitivity of the flame ionization detector varies from compound to compound. However, n-alkanes have a greater response than other classes. Consequently, using an n-alkane as a calibrant and assuming equal responses of all other compounds tends to give low reported values.

2.0 SUMMARY OF METHOD

2.1 A mL aliquot of all 10-mL concentrates is disbursed for GC-TCO analysis. With boiling point-retention time and response-amount calibration curves, the data (peak retention times and peak areas) are interpreted by first summing peak areas in the ranges obtained from the boiling point-retention time calibration. Then, with the response-amount calibration curve, the area sums are converted to amounts of material in the reported boiling point ranges.

2.2 After the instrument is set up, the boiling point-retention time calibration is effected by injecting a mixture of n-C7 through n-C16 hydrocarbons and operating the standard temperature program. Response-quantity calibrations are accomplished by injecting n-decane in n-pentane standards and performing the standard temperature program.

2.3 Definitions

2.3.1 **GC:** Gas chromatography or gas chromatograph.

2.3.2 **C7-C16 n-alkanes:** Heptane through hexadecane.

2.3.3 **GCA temperature program:** 4 min isothermal at 60°C, 10°C/min from 60° to 220°C.

2.3.4 **TRM temperature program:** 5 min isothermal at room temperature, then program from 30°C to 250°C at 15°C/min.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: This procedure is intended for use on a Varian 1860 gas chromatograph, equipped with dual flame ionization detectors and a linear temperature programmer. Any equivalent instrument can be used provided that electrometer settings, etc., be changed appropriately.

4.2 Gases:

4.2.1 **Helium:** Minimum quality is reactor grade. A 4A or 13X molecular sieve drying tube is required. A filter must be placed between the trap and the instrument. The trap should be recharged after every third tank of helium.

4.2.2 **Air:** Zero grade is satisfactory.

4.2.3 **Hydrogen:** Zero grade.

4.3 Syringe: Syringes are Hamilton 701N, 10 uL, or equivalent.

4.4 Septa: Septa will be of such quality as to produce very low bleed during the temperature program. An appropriate septum is Supelco Microsep 138, which is Teflon-backed. If septum bleed cannot be reduced to a negligible level, it will be necessary to install septum swingers on the instrument.

4.5 Recorder: The recorder of this procedure must be capable of not less than 1 mV full-scale display, a 1-sec time constant and 0.5 in. per min chart rate.

4.6 Integrator: An integrator is required. Peak area measurement by hand is satisfactory but too time-consuming. If manual integration is required, the method of "height times width at half height" is used.

4.7 Columns:

4.7.1 **Preferred column:** 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-101 on 100/120 mesh Supelcoport.

4.7.2 **Alternate column:** 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-1 (or other silicon phase) on 100/120 mesh Supelcoport.

4.8 Syringe cleaner: Hamilton syringe cleaner or equivalent connected to a suitable vacuum source.

5.0 REAGENTS

5.1 Pentane: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for standards and for syringe cleaning.

5.2 Methylene chloride: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for syringe cleaning.

6.0 SAMPLING HANDLING AND PRESERVATION

6.1 The extracts are concentrated in a Kuderna-Danish evaporator to a volume less than 10 mL. The concentrate is then quantitatively transferred to a 10-mL volumetric flask and diluted to volume. A 1-mL aliquot is taken for both this analysis and possible subsequent GC/MS analysis and set aside in the sample bank. For each GC-TCO analysis, obtain the sample sufficiently in advance to allow it to warm to room temperature. For example, after one analysis is started, return that sample to the sample bank and take the next sample.

7.0 PROCEDURES

7.1 Setup and checkout: Each day, the operator will verify the following:

7.1.1 That supplies of carrier gas, air and hydrogen are sufficient, i.e., that each tank contains > 100 psig.

7.1.2 That, after replacement of any gas cylinder, all connections leading to the chromatograph have been leak-checked.

7.1.3 That the carrier gas flow rate is 30 ± 2 mL/min, the hydrogen flow rate is 30 ± 2 mL/min, and the air flow rate is 300 ± 20 mL/min.

7.1.4 That the electrometer is functioning properly.

7.1.5 That the recorder and integrator are functioning properly.

7.1.6 That the septa have been leak-checked (leak-checking is effected by placing the soap bubble flow meter inlet tube over the injection port adaptors), and that no septum will be used for more than 20 injections.

7.1.7 That the list of samples to be run is ready.

7.2 Retention time calibration:

7.2.1 To obtain the temperature ranges for reporting the results of the analyses, the chromatograph is given a normal boiling point-retention time calibration. The n-alkanes, their boiling points, and data reporting ranges are given in the table below:

	NBP,°C	Reporting Range,°C	Report As
n-heptane	98	90-110	C7
n-octane	126	110-140	C8
n-nonane	151	140-160	C9
n-decane	174	160-180	C10
n-undecane	194	180-200	C11
n-dodecane	214	200-220	C12
n-tridecane	234	220-240	C13
n-tetradecane	252	240-260	C14
n-pentadecane	270	260-280	C15
n-hexadecane	288	280-300	C16

7.2.2 **Preparation of standards:** Preparing a mixture of the C7-C16 alkanes is required. There are two approaches: (1) use of a standards kit (e.g., Polyscience Kit) containing bottles of mixtures of selected n-alkanes which may be combined to produce a C7-C16 standard; or (2) use of bottles of the individual C7-C16 alkanes from which accurately known volumes may be taken and combined to give a C7-C16 mixture.

7.2.3 **Procedure for retention time calibration:** This calibration is performed at the start of an analytical program; the mixture is chromatographed at the start of each day. To attain the required retention time precision, both the carrier gas flow rate and the temperature program specifications must be observed. Details of the procedure depend on the instrument being used. The general procedure is as follows:

7.2.3.1 Set the programmer upper limit at 250°C. If this setting does not produce a column temperature of 250°C, find the correct setting.

7.2.3.2 Set the programmer lower limit at 30°C.

7.2.3.3 Verify that the instrument and samples are at room temperature.

7.2.3.4 Inject 1 uL of the n-alkane mixture.

7.2.3.5 Start the integrator and recorder.

7.2.3.6 Allow the instrument to run isothermally at room temperature for five min.

7.2.3.7 Shut the oven door.

7.2.3.8 Change the mode to Automatic and start the temperature program.

7.2.3.9 Repeat Steps 1-9 a sufficient number of times so that the relative standard deviation of the retention times for each peak is <5%.

7.3 Response calibration:

7.3.1 For the purposes of a Level 1 analysis, response-quantity calibration with n-decane is adequate. A 10-uL volume of n-decane is injected into a tared 10 mL volumetric flask. The weight injected is obtained and the flask is diluted to the mark with n-pentane. This standard contains about 730 ng n-decane per uL n-pentane. The exact concentration depends on temperature, so that a weight is required. Two serial tenfold dilutions are made from this standard, giving standards at about 730, 73, and 7.3 ng n-decane per uL n-pentane, respectively.

7.3.2 **Procedure for response calibration:** This calibration is performed at the start of an analytical program and monthly thereafter. The most concentrated standard is injected once each day. Any change in calibration necessitates a full calibration with new standards. Standards are stored in the refrigerator locker and are made up monthly.

7.3.2.1 Verify that the instrument is set up properly.

7.3.2.2 Set electrometer at 1 x 10^{-10} A/mV.

7.3.2.3 Inject 1 uL of the highest concentration standard.

7.3.2.4 Run standard temperature program as specified above.

7.3.2.5 Clean syringe.

7.3.2.6 Make repeated injections of all three standards until the relative standard deviations of the areas of each standard are $\leq 5\%$.

7.4 Sample analysis procedure:

7.4.1 The following apparatus is required:

7.4.1.1 Gas chromatograph set up and working.

7.4.1.2 Recorder, integrator working.

7.4.1.3 Syringe and syringe cleaning apparatus.

7.4.1.4 Parameters: Electrometer setting is 1 x 10^{-10} A/mV; recorder is set at 0.5 in./min and 1 mV full-scale.

7.4.2 Steps in the procedure are:

7.4.2.1 Label chromatogram with the data, sample number, etc.

7.4.2.2 Inject sample.

7.4.2.3 Start integrator and recorder.

7.4.2.4 After isothermal operation for 5 min, begin temperature program.

7.4.2.5 Clean syringe.

7.4.2.6 Return sample; obtain new sample.

7.4.2.7 When analysis is finished, allow instrument to cool. Turn chromatogram and integrator output and data sheet over to data analyst.

7.5 Syringe cleaning procedure:

7.5.1 Remove plunger from syringe.

7.5.2 Insert syringe into cleaner; turn on aspirator.

7.5.3 Fill pipet with pentane; run pentane through syringe.

7.5.4 Repeat with methylene chloride from a separate pipet.

7.5.5 Flush plunger with pentane followed by methylene chloride.

7.5.6 Repeat with methylene chloride.

7.6 Sample analysis decision criterion: The data from the TCO analyses of organic extract and rinse concentrates are first used to calculate the total concentration of C7-C16 hydrocarbon-equivalents (Paragraph 7.7.3) in the sample with respect to the volume of air actually sampled, i.e., ug/m^3. On this basis, a decision is made both on whether to calculate the quantity of each n-alkane equivalent present and on which analytical procedural pathway will be followed. If the total organic content is great enough to warrant continuing the analysis -- >500 ug/m^3 -- a TCO of less than 75 ug/m^3 will require only LC fractionation and gravimetric determinations and IR spectra to be obtained on each fraction. If the TCO is greater than 75 ug/m^3, then the first seven LC fractions of each sample will be reanalyzed using this same gas chromatographic technique.

7.7 Calculations:

7.7.1 **Boiling Point - Retention Time Calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.1.1 Average the retention times and calculate relative standard deviations for each n-hydrocarbon.

7.7.1.2 Plot average retention times as abscissae versus normal boiling points as ordinates.

7.7.1.3 Draw in calibration curve.

7.7.1.4 Locate and record retention times corresonding to boiling ranges 90-100, 110-140, 140-160, 160-180, 180-200, 200-220, 220-240, 240-260, 260-280, 280-300°C.

7.7.2 **Response-amount calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.2.1 Average the area responses of each standard and calculate relative standard deviations.

7.7.2.2 Plot response (uv·sec) as ordinate versus ng/uL as abscissa.

7.7.2.3 Draw in the curve. Perform least squares regression and obtain slope (uV·sec·uL/ng).

7.7.3 **Total C7-C16 hydrocarbons analysis:** The required data for this calculation are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.3.1 Sum the areas of all peaks within the retention time range of interest.

7.7.3.2 Convert this area (uV·sec) to ng/uL by dividing by the weight response for n-decane (uV·sec.uL/ng).

7.7.3.3 Multiply this weight by the total concentrate volume (10 mL) to get the weight of the C7-C16 hydrocarbons in the sample.

7.7.3.4 Using the volume of gas sampled or the total weight of sample acquired, convert the result of Step 7.7.3.3 above to ug/m^3.

7.7.3.5 If the value of total C7-C16 hydrocarbons from Step 7.7.3.4 above exceeds 75 ug/m^3, calculate individual hydrocarbon concentrations in accordance with the instructions in Paragraph 7.7.5.5 below.

7.7.4 **Individual C7-C16 n-Alkane Equivalent Analysis:** The required data from the analyses are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.4.1 Sum the areas of peaks in the proper retention time ranges.

7.7.4.2 Convert areas (uV·sec) to ng/uL by dividing by the proper weight response (uV·sec·uL/ng).

7.7.4.3 Multiply each weight by total concentrate volume (10 mL) to get weight of species in each range of the sample.

7.7.4.4 Using the volume of gas sampled on the total weight of sample acquired, convert the result of Step 7.7.4.3 above to ug/m^3.

8.0 QUALITY CONTROL

8.1 Appropriate QC is found in the pertinent procedures throughout the method.

9.0 METHOD PERFORMANCE

9.1 Even relatively comprehensive error propagation analysis is beyond the scope of this procedure. With reasonable care, peak area reproducibility of a standard should be of the order of 1% RSD. The relative standard deviation of the sum of all peaks in a fairly complex waste might be of the order of 5-10%. Accuracy is more difficult to assess. With good analytical technique, accuracy and precision should be of the order of 10-20%.

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APPENDIX J.5

CEM AND GC

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METHOD 18—MEASUREMENT OF GASEOUS ORGANIC COMPOUND EMISSIONS BY GAS CHROMATOGRAPHY

Introduction

This method should not be attempted by persons unfamiliar with the performance characteristics of gas chromatography, nor by those persons who are unfamiliar with source sampling. Particular care should be exercised in the area of safety concerning choice of equipment and operation in potentially explosive atmospheres.

1 *Applicability and Principle*

1.1 Applicability. This method applies to the analysis of approximately 90 percent of the total gaseous organics emitted from an industrial source. It does not include techniques to identify and measure trace amounts of organic compounds, such as those found in building air and fugitive emission sources.

This method will not determine compounds that (1) are polymeric (high molecular weight), (2) can polymerize before analysis, or (3) have very low vapor pressures at stack or instrument conditions.

1.2 Principle.

The major organic components of a gas mixture are separated by gas chromatography (GC) and individually quantified by flame ionization, photoionization, electron capture, or other appropriate detection principles.

The retention times of each separated component are compared with those of known compounds under identical conditions. Therefore, the analyst confirms the identity and approximate concentrations of the organic emission components before hand. With this information, the analyst then prepares or purchases commercially available standard mixtures to calibrate the GC under conditions identical to those of the samples. The analyst also determines the need for sample dilution to avoid detector saturation, gas stream filtration to eliminate particulate matter, and prevention of moisture condensation.

2. *Range and Sensitivity*

2.1 Range. The range of this method is from about 1 part per million (ppm) to the upper limit governed by GC detector saturation or column overloading. The upper limit can be extended by diluting the stack gases with an inert gas or by using smaller gas sampling loops.

2.2 *Sensitivity*. The sensitivity limit for a compound is defined as the minimum detectable concentration of that compound, or the concentration that produces a signal-to-noise ratio of three to one. The minimum detectable concentration is determined during the presurvey calibration for each compound.

3 *Precision and Accuracy*

Gas chromatographic techniques typically provide a precision of 5 to 10 percent relative standard deviation (RSD), but an experienced GC operator with a reliable instrument can readily achieve 5 percent RSD. For this method, the following combined GC/operator values are required.

(a) Precision. Duplicate analyses are within 5 percent of their mean value.

(b) Accuracy. Analysis results of prepared audit samples are within 10 percent of preparation values.

4. *Interferences*

Resolution interferences that may occur can be eliminated by appropriate GC column and detector choice or by shifting the retention times through changes in the column flow rate and the use of temperature programming.

The analytical system is demonstrated to be essentially free from contaminants by periodically analyzing blanks that consist of hydrocarbon free air or nitrogen.

Sample cross-contamination that occurs when high-level and low-level samples or standards are analyzed alternately, is best

dealt with by thorough purging of the GC sample loop between samples.

To assure consistent detector response, calibration gases are contained in dry air. To adjust gaseous organic concentrations when water vapor is present in the sample, water vapor concentrations are determined for those samples, and a correction factor is applied.

5 *Presurvey and Presurvey Sampling.*

Perform a presurvey for each source to be tested. Refer to Figure 18-1. Some of the information can be collected from literature surveys and source personnel. Collect gas samples that can be analyzed to confirm the identities and approximate concentrations of the organic emissions.

5.1 Apparatus. This apparatus list also applies to Sections 6 and 7.

5.1.1 Teflon Tubing. (Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Diameter and length determined by connection requirements of cylinder regulators and the GC. Additional tubing is necessary to connect the GC sample loop to the sample.

5.1.2 Gas Chromatograph. GC with suitable detector, columns, temperature-controlled sample loop and valve assembly, and temperature programable oven, if necessary. The GC shall achieve sensitivity requirements for the compounds under study.

5.1.3 Pump. Capable of pumping 100 ml/min. For flushing sample loop.

5.1.4 *Flowmeters*. To measure flow rates.

5.1.5 Regulators. Used on gas cylinders for GC and for cylinder standards.

5.1.6 Recorder. Recorder with linear strip chart is minimum acceptable. Integrator (optional) is recommended.

5.1.7 *Syringes*. 0.5-ml, 1.0- and 10-microliter size, calibrated, maximum accuracy (gas tight), for preparing calibration standards. Other appropriate sizes can be used.

5.1.8 Tubing Fittings. To plumb GC and gas cylinders.

5.1.9 Septums. For syringe injections.

5.1.10 Glass Jars. If necessary, clean colored glass jars with Teflon-lined lids for condensate sample collection. Size depends on volume of condensate.

5.1.11 Soap Film Flow Meter. To determine flow rates.

5.1.12 Tedlar Bags. 10 and 50 liter capacity, for preparation of standards.

5.1.13 Dry Gas Meter with Temperature and Pressure Gauges. Accurate to ±2 percent, *for perparation of gas standards.*

5.1.14 Midget Impinger/Hot Plate Assembly. For preparation of gas standards.

5.1.15 Sample Flasks. For presurvey samples, must have gas-tight seals.

5.1.16 Adsorption Tubes. If necessary, blank tubes filled with necessary adsorbent (charcoal, Tenax, XAD-2, etc.) for presurvey samples.

5.1.17 Personnel Sampling Pump. Calibrated, for collecting adsorbent tube presurvey samples.

5.1.18 Dilution System. Calibrated, the dilution system is to be constructed following the specifications of an acceptable method.

5.1.19 *Sample Probes*. Pyrex or stainless steel, of sufficient length to reach centroid of stack, or a point no closer to the walls than 1 m.

5.1.20 Barometer. To measure barometric pressure.

5.2 Reagents.

5.2.1 Deionized Distilled Water.

5.2.2 Methylene Dichloride.

5.2.3 Calibration Gases. A series of standards prepared for every compound of interest.

5.2.4 Organic Compound Solutions. Pure (99.9 percent), or as pure as can reasonably be obtained, liquid samples of all the organic compounds needed to prepare calibration standards.

5.2.5 Extraction Solvents. For extraction of adsorbent tube samples in preparation for analysis.

5.2.6 Fuel. As recommended by the manufacturer for operation of the GC.

5.2.7 Carrier Gas. Hydrocarbon free, as recommended by the manufacturer for operation of the detector and compatability with the column.

5.2.8 Zero Gas. Hydrocarbon free air or nitrogen, to be used for dilutions, blank preparation, and standard preparation.

5.3 Sampling.

5.3.1 Collection of Samples with Glass Sampling Flasks. Presurvey samples can be collected in precleaned 250-ml double-ended glass sampling flasks. Teflon stopcocks, without grease, are preferred. Flasks should be cleaned as follows: Remove the stopcocks from both ends of the flasks, and wipe the parts to remove any grease. Clean the stopcocks, barrels, and receivers with methylene dichloride. Clean all glass ports with a soap solution, then rinse with tap and deionized distilled water. Place the flask in a cool glass annealing furnace and apply heat up to 500° C. Maintain at this temperature for 1 hour. After this time period, shut off and open the furnace to allow the flask to cool. Grease the stopcocks with stopcock grease and return them to the flask receivers. Purge the assembly with high-purity nitrogen for 2 to 5 minutes. *Close off the stopcocks* after purging to maintain a slight positive nitrogen pressure. Secure the stopcocks with tape.

Presurvey samples can be obtained either by drawing the gases into the previously evacuated flask or by drawing the gases into and purging the flask with a rubber suction bulb.

5.3.1.1 Evacuated Flask Procedure. Use a high vacuum pump to evacuate the flask to the capacity of the pump; then close off the stopcock leading to the pump. Attach a 6-mm outside diameter (OD) glass tee to the flask inlet with a short piece of Teflon tubing. Select a 6-mm OD borosilicate sampling probe, enlarged at one end to a 12-mm OD and of sufficient length to reach the centroid of the duct to be sampled. Insert a glass wool plug in the enlarged end of the probe to remove particulate matter. Attach the other end of the probe to the tee with a short piece of Teflon tubing. Connect a rubber suction bulb to the third leg of the tee. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and purge the probe with the rubber suction bulb. After the probe is completely purged and filled with duct gases, open the stopcock to the grab flask until the pressure in the flask reaches duct pressure. Close off the stopcock, and remove the probe from the duct. Remove the tee from the flask and tape the stopcocks to prevent leaks during shipment. Measure and record the duct temperature and pressure.

5.3.1.2 Purged Flask Procedure. Attach one end of the sampling flask to a rubber suction bulb. Attach the other end to a 6-mm OD glass probe as described in Section 5.3.1.1. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and apply suction with the bulb to completely purge the probe and flask. After the flask has been purged, close off the stopcock near the suction bulb, and then close the stopcock near the probe. Remove the probe from the duct, and disconnect both the probe and suction bulb. Tape the stopcocks to prevent leakage during shipment. Measure and record the duct temperature and pressure.

5.3.2 Flexible Bag Procedure. Tedlar or aluminized Mylar bags can also be used to obtain the presurvey sample. Use new bags, and leak check them before field use. In addition, check the bag before use for contamination by filling it with nitrogen or air, and analyzing the gas by GC at high sensitivity. Experience indicates that it is desirable to allow the inert gas to remain in the bag about 24 hours or longer to check for desorption of organics from the bag. Follow the leak check and sample collection procedures given in Section 7.1.

5.3.3 Determination of Moisture Content. For combustion or water controlled processes, obtain the moisture content from plant personnel or by measurement during the presurvey. If the source is below 59° C, measure the wet bulb and dry bulb temperatures, and calculate the moisture content using a psychrometric chart. At higher temperatures, use Method 4 to determine the moisture content.

5.4 Determination of Static Pressure. Obtain the static pressure from the plant personnel or measurement. If a type S pitot tube and an inclined manometer are used, take care to align the pitot tube 90° from the direction of the flow. Disconnect one of the tubes to the manometer, and read the static pressure; note whether the reading is positive or negative.

5.5 Collection of Presurvey Samples with Adsorption Tube. Follow Section 7.4 for presurvey sampling.

6 *Analysis Development*

6.1 Selection of GC Parameters.

6.1.1 Column Choice. Based on the initial contact with plant personnel concerning the plant process and the anticipated emissions, choose a column that provides good resolution and rapid analysis time. The choice of an appropriate column can be aided by a literature search, contact with manufacturers of GC columns, and discussion with personnel at the emission source.

Most column manufacturers keep excellent records of their products. Their technical service departments may be able to recommend appropriate columns and detector type for separating the anticipated compounds, and they may be able to provide information on interferences, optimum operating conditions, and column limitations.

Plants with analytical laboratories may also be able to provide information on appropriate analytical procedures.

6.1.2 Preliminary GC Adjustment. Using the standards and column obtained in Section 6.1.1, perform initial tests to determine appropriate GC conditions that provide good resolution and minimum analysis time for the compounds of interest.

6.1.3 Preparation of Presurvey Samples. If the samples were collected on an adsorbent, extract the sample as recommended by the manufacturer for removal of the compounds with a solvent suitable to the type of GC analysis. Prepare other samples in an appropriate manner.

6.1.4 Presurvey Sample Analysis. Before analysis, heat the presurvey sample to the duct temperature to vaporize any condensed material. Analyze the samples by the GC procedure, and compare the retention times against those of the calibration samples that contain the components expected to be in the stream. If any compounds cannot be identified with certainty by this procedure, identify them by other means such as GC/mass spectroscopy (GC/MS) or GC/infrared techniques. A GC/MS system is recommended.

Use the GC conditions determined by the procedures of Section 6.1.2 for the first injection. Vary the GC parameters during subsequent injections to determine the optimum settings. Once the optimum settings have been determined, perform repeat injections of the sample to determine the retention time of each compound. To inject a sample, draw sample through the loop at a constant rate (100 ml/min for 30 seconds). Be careful not to pressurize the gas in the loop. Turn off the pump and allow the gas in the sample loop to come to ambient pressure. Activate the sample valve, and record injection time, loop temperature, column temperature, carrier flow rate, chart speed, and attenuator setting. Calculate the retention time of each peak using the distance from injection to the peak maximum divided by the chart speed. Retention times should be repeatable within 0.5 seconds.

If the concentrations are too high for appropriate detector response, a smaller sample loop or dilutions may be used for gas samples, and, for liquid samples, dilution with solvent is appropriate. Use the standard curves (Section 6.3) to obtain an estimate of the concentrations.

Identify all peaks by comparing the known retention times of compounds expected to be in the retention times of peaks in the sample. Identify any remaining unidentified peaks which have areas larger than 5 percent of the total using a GC/MS, or estimation of possible compounds by their retention times compared to known compounds, with confirmation by further GC analysis.

6.2 Calibration Standards. Prepare or obtain enough calibration standards so that there are three different concentrations of each organic compound expected to be measured in the source sample. For each organic compound, select those concentrations that bracket the concentrations expected in the source samples. A calibration standard may contain more than one organic compound. If available, commercial cylinder gases may be used if their concentrations have been certified by direct analysis.

If samples are collected in adsorbent tubes (charcoal, XAD-2, Tenax, etc.), prepare or obtain standards in the same solvent used for the sample extraction procedure. Refer to Section 7.4.3.

Verify the stability of all standards for the time periods they are used. If gas standards are prepared in the laboratory, use one or more of the following procedures.

6.2.1 Preparation of Standards from High Concentration Cylinder Standards. Obtain enough high concentration cylinder standards to represent all the organic compounds expected in the source samples.

Use these high concentration standards to prepare lower concentration standards by dilution, as shown by Figures 18-5 and 18-6.

To prepare the diluted calibration samples, calibrated rotameters are normally used to meter both the high concentration calibration gas and the diluent gas. Other types of flowmeters and commercially available dilution systems can also be used.

Calibrate each flowmeter before use by placing it between the diluent gas supply and suitably sized bubble meter, spirometer, or wet test meter. Record all data shown on Figure 18-4. While it is desirable to calibrate the cylinder gas flowmeter with cylinder gas, the available quantity and cost may preclude it. The error introduced by using the diluent gas for calibration is insignificant for gas mixtures of up to 1,000 to 2,000 ppm of each organic component.

Once the flowmeters are calibrated, connect the flowmeters to the calibration and diluent gas supplies using 6-mm Teflon tubing. Connect the outlet side of the flowmeters through a connector to a leak-free Tedlar bag as shown in Figure 18-5. (See Section 7.1 for bag leak check procedures.) Adjust the gas flow to provide the desired dilution, and fill the bag with sufficient gas for GC calibration. Be careful not to overfill and cause the bag to apply additional pressure on the dilution system. Record the flow rates of both flowmeters, and the laboratory temperature and atmospheric pressure. Calculate the concentration C_s in ppm of each organic in the diluted gas as follows:

$$C_s = \frac{10^6 (\bar{X} q_c)}{q_c + q_d} \qquad \text{Eq. 18-1}$$

where.

10^6 = Conversion to ppm.

$\bar{X}$ = Mole or volume fraction of the organic in the calibration gas to be diluted.

q_c = Flow rate of the calibration gas to be diluted.

q_d = Diluent gas flow rate.

Single stage dilutions should be used to prepare calibration mixtures up to about 1:20 dilution factor.

For greater dilutions, a double dilution system is recommended, as shown in Figure 18-6. Fill the Tedlar bag with the dilute gas from the second stage. Record the laboratory temperature, barometric pressure, and static pressure readings. Correct the flow reading for temperature and pressure. Calculate the concentration C_s in ppm of the organic in the final gas mixture as follows.

$$C_s = 10^6 \bar{X} \left(\frac{q_{c1}}{q_{c1} + q_{d1}}\right)\left(\frac{q_{c2}}{q_{c2} + q_{d2}}\right) \qquad \text{Eq. 18-2}$$

Where:

10^6 = Conversion to ppm.
$\bar{X}$ = Mole or volume fraction of the organic in the calibration gas to be diluted.
q_{c1} = Flow rate of the calibration gas to be diluted in stage 1.
q_{c2} = Flow rate of the calibration gas to be diluted in stage 2.
q_{d1} = Flow rate of diluent gas in stage 1.
q_{d2} = Flow rate of diluent gas in stage 2.

Further details of the calibration methods for flowmeters and the dilution system can be found in Citation 21 in the Bibliography.

6.2.2 *Preparation of Standards from Volatile Materials.* Record all data shown on Figure 18-3.

6.2.2.1 *Gas Injection Technique.* This procedure is applicable to organic compounds that exist entirely as a gas at ambient conditions. Evacuate a 10 liter Tedlar bag that has passed a leak-check (see Section 7.1), and meter in 5.0 liters of air or nitrogen through a dry gas meter that has been calibrated in a manner consistent with the procedure described in Section 5.1.1 of Method 5. While the bag is filling use a 0.5 ml syringe to inject a known quantity of "pure" gas of the organic compound through the wall of the bag, or through a septum-capped tee at the bag inlet. Withdraw the syringe needle, and immediately cover the resulting hole with a piece of masking tape. In a like manner, prepare dilutions having other concentrations. Prepare a minimum of three concentrations. Place each bag on a smooth surface, and alternately depress opposite sides of the bag 50 times to mix the gases Record the average meter temperature and pressure, the gas volume and the barometric pressure. Record the syringe temperature and pressure before injection.

Calculate each organic standard concentration C_s in ppm as follows:

$$C_s = \frac{G_v \times 10^6 \dfrac{293}{T_s} \dfrac{P_s}{760}}{V_m Y \dfrac{293}{T_m} \dfrac{P_m}{760} 1000} = \frac{G_v \times 10^3 \dfrac{P_s}{T_s} \dfrac{T_m}{P_m}}{V_m Y} \qquad \text{Eq. 18-3}$$

where:

G_v = Gas volume or organic compound injected, ml.
10^6 = Conversion to ppm.
P_s = Absolute pressure of syringe before injection, mm Hg.
T_s = Absolute temperature of syringe before injection, °K.
V_m = Gas volume indicated by dry gas meter, liters.
Y = Dry gas meter calibration factor, dimensionless.
P_m = Absolute pressure of dry gas meter, mm Hg.
T_m = Absolute temperature of dry gas meter, °K.
1000 = Conversion factor, ml/liter

6.2.2.2 *Liquid Injection Technique.* Use the equipment shown in Figure 18-9. Calibrate the dry gas meter as described in Section 6.2.2.1 with a wet test meter or a spirometer. Use a water manometer for the pressure gauge and glass, Teflon, brass, or stainless steel for all connections. Connect a valve to the inlet of the 50-liter Tedlar bag.

To prepare the standards, assemble the equipment as shown in Figure 18-9, and leak check the system. Completely evacuate the bag. Fill the bag with hydrocarbon free air, and evacuate the bag again. Close the inlet valve.

Turn on the hot plate, and allow the water to reach boiling. Connect the bag to the impinger outlet. Record the initial meter reading, open the bag inlet valve, and open the cylinder. Adjust the rate so that the bag will be completely filled in approximately 15 minutes. Record meter pressure and temperature, and local barometric pressure.

Allow the liquid organic to equilibrate to room temperature. Fill the 1.0 or 10 microliter syringe to the desired liquid volume with the organic. Place the syringe needle into the impinger inlet using the septum provided, and inject the liquid into the flowing air stream. Use a needle of sufficient length to permit injection of the liquid below the air inlet branch of the tee. Remove the syringe.

When the bag is filled, stop the pump, and close the bag inlet valve. Record the final meter reading, temperature, and pressure.

Disconnect the bag from the impinger outlet, and either set it aside for at least 1 hour, or massage the bag to insure complete mixing.

Measure the solvent liquid density at room temperature by accurately weighing a known volume of the material on an analytical balance to the nearest 1.0 milligram. A ground-glass stoppered 25-ml volumetric flask or a glass-stoppered specific gravity bottle is suitable for weighing. Calculate the result in terms of g/ml. As an alternative, literature values of the density of the liquid at 20 °C may be used.

Calculate each organic standard concentration C_s in ppm as follows:

$$C_s = \frac{\dfrac{L_v \rho}{M} (24.055 \times 10^6)}{V_m Y \dfrac{293}{T_m} \dfrac{P_m}{760} 1000} = 6.24 \times 10^4 \frac{L_v \rho T_m}{M V_m Y P_m} \qquad \text{Eq. 18-4}$$

where:

L_v = Liquid volume of organic injected, µl.
ρ = Liquid organic density as determined, g/ml.
M = Molecular weight of organic, g/g-mole.
24.055 = Ideal gas molar volume at 293 °K and 760 mm Hg, liters/g-mole.
10^6 = Conversion to ppm.
1000 = Conversion factor, µl/ml.

6.3 *Preparation of Calibration Curves.* Establish proper GC conditions, then flush the sampling loop for 30 seconds at a rate of 100 ml/min. Allow the sample loop pressure to equilibrate to atmospheric pressure, and activate the injection valve. Record the standard concentration, attenuator factor, injection time, chart speed, retention time, peak area, sample loop temperature, column temperature, and carrier gas flow rate. Repeat the standard injection until two consecutive injections give area counts within 5 percent of their average. The average value multiplied by the attenuator factor is then the calibration area value for the concentration.

Repeat this procedure for each standard. Prepare a graphical plot of concentration (C_s) versus the calibration area values. Perform a regression analysis, and draw the least squares line.

6.4 *Relative Response Factors.* The calibration curve generated from the standards for a single organic can usually be related to each of the individual GC response curves that are developed in the laboratory for all the compounds in the source. In the field, standards for that single organic can then be used to "calibrate" the GC for all the organics present. This procedure should first be confirmed in the laboratory by preparing and analyzing calibration standards containing multiple organic compounds.

6.5 *Quality Assurance for Laboratory Procedures.* Immediately after the preparation of the calibration curves and prior to the presurvey sample analysis, the analysis audit described in 40 CFR Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis," should be performed. The information required to document the analysis of the audit samples has been included on the example data sheets shown in Figures 18-3 and 18-7. The audit analyses should agree with the audit concentrations within 10 percent. When available, the tester may obtain audit cylinders by contacting: U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711. Audit cylinders obtained from a commercial gas manufacturer may be used provided that (a) the gas manufacturer certifies the audit cylinder in a manner similar to the procedure described in 40 CFR Part 61, Appendix B, Method 106, Section 5.2.3.1, and (b) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined as an analysis *performed by an individual other than the* individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis concentration is within 5 percent of the gas manufacturer's concentration

7 Final Sampling and Analysis Procedure

Considering safety (flame hazards) and the source conditions, select an appropriate sampling and analysis procedure (Section 7.1, 7.2, 7.3, or 7.4). In situations where a hydrogen flame is a hazard and no intrinsically safe GC is suitable, use the flexible bag collection technique or an adsorption technique. If the source temperature is below 100°C, and the organic concentrations are suitable for the detector to be used, use the direct interface method. If the source gases require dilution, use a dilution interface and either the bag sample or adsorption tubes. The choice between these two techniques will depend on the physical layout of the site, the source temperature, and the storage stability of the compounds if collected in the bag. Sample polar compounds by direct interfacing or dilution interfacing to prevent sample loss by adsorption on the bag.

7.1 Integrated Bag Sampling and Analysis.

7.1.1 Evacuated Container Sampling Procedure. In this procedure, the bags are filled by evacuating the rigid air-tight containers that hold the bags. Use a field sample data sheet as shown in Figure 18-10. Collect triplicate sample from each sample location.

7.1.1.1 Apparatus.

7.1.1.1.1 Probe. Stainless steel, Pyrex glass, or Teflon tubing probe, according to the duct temperature, with 6.4-mm OD Teflon tubing of sufficient length to connect to the sample bag. Use stainless steel or Teflon unions to connect probe and sample line.

7.1.1.1.2 Quick Connects. Male (2) and female (2) of stainless steel construction.

7.1.1.1.3 Needle Valve. To control gas flow.

7.1.1.1.4 Pump. Leakless Teflon-coated diaphragm-type pump or equivalent. To deliver at least 1 liter/min.

7.1.1.1.5 Charcoal Adsorption Tube. Tube filled with activated charcoal, with glass wool plugs at each end, to adsorb organic vapors.

7.1.1.1.6 Flowmeter. 0 to 500-ml flow range; with manufacturer's calibration curve.

7.1.1.2 Sampling Procedure. To obtain a sample, assemble the sample train as shown in Figure 18-9. Leak check both the bag and the container. Connect the vacuum line from the needle valve to the Teflon sample line from the probe. Place the end of the probe at the centroid of the stack, or at a point no closer to the walls than 1 m, and start the pump with the needle valve adjusted to yield a flow of 0.5 liter/minute. After allowing sufficient time to purge the line several times, connect the vacuum line to the bag, and evacuate until the rotameter indicates no flow. Then position the sample and vacuum lines for sampling, and begin the actual sampling, keeping the rate proportional to the stack velocity. As a precaution, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Record the source temperature, barometric pressure, ambient temperature, sampling flow rate, and initial and final sampling time on the data sheet shown in Figure 18-10. Protect the Tedlar bag and its container from sunlight. When possible, perform the analysis within 2 hours of sample collection.

7.1.2 Direct Pump Sampling Procedure. Follow 7.1.1, except place the pump and needle valve between the probe and the bag. Use a pump and needle valve constructed of stainless steel or some other material not affected by the stack gas. Leak check the system, and then purge with stack gas before the connecting to the previously evacuated bag.

7.1.3 Explosion Risk Area Bag Sampling Procedure. Follow 7.1.1 except replace the pump with another evacuated can (see Figure 18-9a). Use this method whenever there is a possibility of an explosion due to pumps, heated probes, or other flame producing equipment.

7.1.4 Other Modified Bag Sampling Procedures. In the event that condensation is observed in the bag while collecting the sample and a direct interface system cannot be used, heat the bag during collection, and maintain it at a suitably elevated temperature during all subsequent operations. (Note: Take care to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.) As an alternative, collect the sample gas, and simultaneously dilute it in the Tedlar bag.

In the first procedure, heat the box containing the sample bag to the source temperature, provided the components of the bag and the surrounding box can withstand this temperature. Then transport the bag as rapidly as possible to the analytical area while maintaining the heating, or cover the box with an insulating blanket. In the analytical area, keep the box heated to source temperature until analysis. Be sure that the method of heating the box and the control for the heating circuit are compatible with the safety restrictions required in each area.

To use the second procedure, prefill the Tedlar bag with a known quantity of inert gas. Meter the inert gas into the bag according to the procedure for the preparation of gas concentration standards of volatile liquid materials (Section 6.2.2.2), but eliminate the midget impinger section. Take the partly filled bag to the source, and meter the source gas into the bag through heated sampling lines and a heated flowmeter, or Teflon positive displacement pump. Verify the dilution factors periodically through dilution and analysis of gases of known concentration.

7.1.5 Analysis of Bag Samples.

7.1.5.1 Apparatus. Same as Section 5. A minimum of three gas standards are required.

7.1.5.2 Procedure. Establish proper GC operating conditions as described in Section 6.3, and record all data listed in Figure 18-7. Prepare the GC so that gas can be drawn through the sample valve. Flush the sample loop with gas from one of the three calibration mixtures, and activate the valve. Obtain at least two chromatograms for the mixture. The results are acceptable when the peak areas from two consecutive injections agree to within 5 percent of their average. If they do not, run additional analyses or correct the analytical techniques until this requirement is met. Then analyze the other two calibration mixtures in the same manner. Prepare a calibration curve as described in the same manner. Prepare a calibration curve as described in Section 6.3.

Analyze the source gas samples by connecting each bag to the sampling valve with a piece of Teflon tubing identified for that bag. Follow the specifications on replicate analyses specified for the calibration gases. Record the data listed in Figure 18-11. If certain items do not apply, use the notation "N.A." After all samples have been analyzed, repeat the analyses of the calibration gas mixtures, and generate a second calibration curve. Use an average of the two curves to determine the sample gas concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then report the final results by comparison to both calibration curves.

7.1.6 Determination of Bag Water Vapor Content. Measure and record the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.) If the bag has been maintained at an elevated temperature as described in Section 7.1.4, determine the stack gas water content by Method 4.

7.1.7 Quality Assurance. Immediately prior to the analysis of the stack gas samples, perform audit analyses as described in Section 6.5. The audit analyses must agree with the audit concentrations within 10 percent. If the results are acceptable, proceed with the analyses of the source samples. If they do not agree within 10 percent, then determine the reason for the discrepancy, and take corrective action before proceeding.

7.1.8 Emission Calculations. From the average calibration curve described in Section 7.1.5, select the value of C_s that corresponds to the peak area. Calculate the concentration C_c in ppm, dry basis, of each organic in the sample as follows:

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1-B_{ws})} \qquad \text{Eq. 18-5}$$

where:

C_s = Concentration of the organic from the calibration curve, ppm.

P_r = Reference pressure, the barometric pressure or absolute sample loop pressure recorded during calibration, mm Hg.

T_i = Sample loop temperature at the time of sample analysis, °K.

F_r = Relative response factor (if applicable, see Section 6.4).

P_i = Barometric or absolute sample loop pressure at time of sample analysis, mm Hg.

T_r = Reference temperature, the temperature of the sample loop recorded during calibration, °K.

B_{ws} = Water vapor content of the bag sample or stack gas, proportion by volume.

7.2 Direct Interface Sampling and Analysis Procedure. The direct interface procedure can be used provided that the moisture content of the gas does not interfere with the analysis procedure, the physical requirements of the equipment can be met at the site, and the source gas concentration is low enough that detector saturation is not a problem. Adhere to all safety requirements with this method.

7.2.1 Apparatus.

7.2.1.1 Probe. Constructed of stainless steel, Pyrex glass, or Teflon tubing as required by duct temperature, 6.4-mm OD, enlarged at duct end to contain glass wool plug. If necessary, heat the probe with heating tape or a special heating unit capable of maintaining duct temperature.

7.2.1.2 Sample Lines. 6.4-mm OD Teflon lines, heat-traced to prevent condensation of material.

7.2.1.3 Quick Connects. To connect sample line to gas sampling valve on GC instrument and to pump unit used to withdraw source gas. Use a quick connect or equivalent on the cylinder or bag containing calibration gas to allow connection of the calibration gas to the gas sampling valve.

7.2.1.4 Thermocouple Readout Device. Potentiometer or digital thermometer, to measure source temperature and probe temperature.

7.2.1.5 Heated Gas Sampling Valve. Of two-position, six-port design, to allow sample loop to be purged with source gas or to direct source gas into the GC instrument.

7.2.1.6 Needle Valve. To control gas sampling rate from the source.

7.2.1.7 Pump. Leakless Teflon coated diaphragm-type pump or equivalent, capable of at least 1 liter/minute sampling rate.

7.2.1.8 Flowmeter. Of suitable range to measure sampling rate.

7.2.1.9 Charcoal Adsorber. To adsorb organic vapor collected from the source to prevent exposure of personnel to source gas.

7.2.1.10 Gas Cylinders. Carrier gas (helium or nitrogen), and oxygen and hydrogen for a flame ionization detector (FID) if one is used.

7.2.1.11 Gas Chromatograph. Capable of being moved into the field, with detector, heated gas sampling valve, column required to complete separation of desired components, and option for temperature programming.

7.2.1.12 Recorder/Integrator. To record results.

7.2.2 Procedure. To obtain a sample, assemble the sampling system as shown in Figure 18-12. Make sure all connections are tight. Turn on the probe and sample line heaters. As the temperature of the probe and heated line approaches the source temperature as indicated on the thermocouple readout device, control the heating to maintain a temperature of 0 to 3°C above the source temperature. While the probe and heated line are being heated, disconnect the sample line from the gas sampling valve, and attach the line from the calibration gas mixture. Flush the sample loop with calibration gas and analyze a portion of that gas. Record the results. After the calibration gas sample has been flushed into the GC instrument, turn the gas sampling valve to flush position, then reconnect the probe sample line to the valve. Place the inlet of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and draw source gas into the probe, heated line, and sample loop. After thorough flushing, analyze the sample using the same conditions as for the calibration gas mixture. Repeat the analysis on an additional sample. Measure the peak areas for the two samples, and if they do not agree to within 5 percent of their mean value, analyze additional samples until two consecutive analyses meet this criteria. Record the data. After consistent results are obtained, remove the probe from the source and analyze a second calibration gas mixture. Record this calibration data and the other required data on the data sheet shown in Figure 18-11, deleting the dilution gas information.

(NOTE: Take care to draw all samples, calibration mixtures, and audits through the sample loop at the same pressure.)

7.2.3 Determination of Stack Gas Moisture Content. Use Method 4 to measure the stack gas moisture content.

7.2.4 Quality Assurance. Same as Section 7.1.7. Introduce the audit gases in the sample line immediately following the probe.

7.2.5 Emission Calculations. Same as Section 7.1.8.

7.3 Dilution Interface Sampling and Analysis Procedure. Source samples that contain a high concentration of organic materials may require dilution prior to analysis to prevent saturating the GC detector. The apparatus required for this direct interface procedure is basically the same as that described in the Section 7.2, except a dilution system is added between the heated sample line and the gas sampling valve. The apparatus is arranged so that either a 10:1 or 100:1 dilution of the source gas can be directed to the chromatograph. A pump of larger capacity is also required, and this pump must be heated and placed in the system between the sample line and the dilution apparatus.

7.3.1 Apparatus. The equipment required in addition to that specified for the direct interface system is as follows:

7.3.1.1 Sample Pump. Leakless Teflon-coated diaphragm-type that can withstand being heated to 120°C and deliver 1.5 liters/minute.

7.3.1.2 Dilution Pumps. Two Model A 150 Komhyr Teflon positive displacement type delivering 150 cc/minute, or equivalent. As an option, calibrated flowmeters can be used in conjunction with Teflon-coated diaphragm pumps.

7.3.1.3 Valves. Two Teflon three-way valves, suitable for connecting to 6.4 mm OD Teflon tubing.

7.3.1.4 Flowmeters. Two, for measurement of diluent gas, expected delivery flow rate to be 1,350 cc/min.

7.3.1.5 Diluent Gas with Cylinders and Regulators. Gas can be nitrogen or clean dry air, depending on the nature of the source gases.

7.3.1.6 Heated Box. Suitable for being heated to 120°C, to contain the three pumps, three-way valves, and associated connections. The box should be equipped with quick connect fittings to facilitate connection of: (1) The heated sample line from the probe, (2) the gas sampling valve, (3) the calibration gas mixtures, and (4) diluent gas lines. A schematic diagram of the components and connections is shown in Figure 18-13.

(NOTE: Care must be taken to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.)

The heated box shown in Figure 18-13 is designed to receive a heated line from the

probe. An optional design is to build a probe unit that attaches directly to the heated box. In this way, the heated box contains the controls for the probe heaters, or, if the box is placed against the duct being sampled, it may be possible to eliminate the probe heaters. In either case, a heated Teflon line is used to connect the heated box to the gas sampling valve on the chromatograph.

7.3.2 Procedure. Assemble the apparatus by connecting the heated box, shown in Figure 18-13, between the heated sample line from the probe and the gas sampling valve on the chromatograph. Vent the source gas from the gas sampling valve directly to the charcoal filter, eliminating the pump and rotameter. Heat the sample probe, sample line, and heated box. Insert the probe and source thermocouple to the centroid of the duct, or to a point no closer to the walls than 1 m. Measure the source temperature, and adjust all heating units to a temperature 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds. Verify the operation of the dilution system by analyzing a high concentration gas of known composition through either the 10:1 or 100:1 dilution stages, as appropriate. (If necessary, vary the flow of the diluent gas to obtain other dilution ratios.) Determine the concentration of the diluted calibration gas using the dilution factor and the calibration curves prepared in the laboratory. Record the pertinent data on the data sheet shown in Figure 18-11. If the data on the diluted calibration gas are not within 10 percent of the expected values, determine whether the chromatograph or the dilution system is in error, and correct it. Verify the GC operation using a low concentration standard by diverting the gas into the sample loop, bypassing the dilution system. If these analyses are not within acceptable limits, correct the dilution system to provide the desired dilution factors. Make this correction by diluting a high-concentration standard gas mixture to adjust the dilution ratio as required.

Once the dilution system and GC operations are satisfactory, proceed with the analysis of source gas, maintaining the same dilution settings as used for the standards. Repeat the analyses until two consecutive values do not vary by more than 5 percent from their mean value are obtained.

Repeat the analysis of the calibration gas mixtures to verify equipment operation. Analyze the two field audit samples using either the dilution system, or directly connect to the gas sampling valve as required. Record all data and report the results to the audit supervisor.

7.3.3 Determination of Stack Gas Moisture Content. Same as Section 7.2.3.

7.3.4 Quality Assurance. Same as Section 7.2.4.

7.3.5 Emission Calculations. Same as Section 7.2.5, with the dilution factor applied.

7.4 Adsorption Tube Procedure (Alternative Procedure). It is suggested that the tester refer to the National Institute of Occupational Safety and Health (NIOSH) method for the particular organics to be sampled. The principal interferent will be water vapor. If water vapor is present at concentrations above 3 percent, silica gel should be used in front of the charcoal. Where more than one compound is present in the emissions, then develop relative adsorptive capacity information.

7.4.1 Additional Apparatus. In addition to the equipment listed in the NIOSH method for the particular organic(s) to be sampled, the following items (or equivalent) are suggested.

7.4.1.1 Probe (Optional). Borosilicate glass or stainless steel, approximately 6-mm ID, with a heating system if water condensation is a problem, and a filter (either in-stack or out-stack heated to stack temperature) to remove particulate matter. In most instances, a plug of glass wool is a satisfactory filter.

7.4.1.2 Flexible Tubing. To connect probe to adsorption tubes. Use a material that exhibits minimal sample adsorption.

7.4.1.3 Leakless Sample Pump. Flow controlled, constant rate pump, with a set of limiting (sonic) orifices to provide pumping rates from approximately 10 to 100 cc/min.

7.4.1.4 Bubble-Tube Flowmeter. Volume accuracy within ±1 percent, to calibrate pump.

7.4.1.5 Stopwatch. To time sampling and pump rate calibration.

7.4.1.6 Adsorption Tubes. Similar to ones specified by NIOSH, except the amounts of adsorbent per primary/backup sections are 800/200 mg for charcoal tubes and 1040/260 mg for silica gel tubes. As an alternative, the tubes may contain a porous polymer adsorbent such as Tenax GC or XAD-2.

7.4.1.7 Barometer. Accurate to 5 mm Hg, to measure atmospheric pressure during sampling and pump calibration.

7.4.1.8 Rotameter. 0 to 100 cc/min, to detect changes in flow rate during sampling.

7.4.2 Sampling and Analysis. It is suggested that the tester follow the sampling and analysis portion of the respective NIOSH method section entitled "Procedure." Calibrate the pump and limiting orifice flow rate through adsorption tubes with the bubble tube flowmeter before sampling. The sample system can be operated as a "recirculating loop" for this operation. Record the ambient temperature and barometric pressure. Then, during sampling, use the ro tameter to verify that the pump and orifice sampling rate remains constant.

Use a sample probe, if required, to obtain the sample at the centroid of the duct, or at a point no closer to the walls than 1 m. Minimize the length of flexible tubing between the probe and adsorption tubes. Several adsorption tubes can be connected in series, if the extra adsorptive capacity is needed. Provide the gas sample to the sample system at a pressure sufficient for the limiting orifice to function as a sonic orifice. Record the total time and sample flow rate (or the number of pump strokes), the barometric pressure, and ambient temperature. Obtain a total sample volume commensurate with the expected concentration(s) of the volatile organic(s) present, and recommended sample loading factors (weight sample per weight adsorption media). Laboratory tests prior to actual sampling may be necessary to predetermine this volume. When more than one organic is present in the emissions, then develop relative adsorptive capacity information. If water vapor is present in the sample at concentrations above 2 to 3 percent, the adsorptive capacity may be severely reduced. Operate the gas chromatograph according to the manufacture's instructions. After establishing optimum conditions, verify and document these conditions during all operations. Analyze the audit samples (see Section 7.4.4.3), then the emission samples. Repeat the analysis of each sample until the relative deviation of two consecutive injections does not exceed 5 percent.

7.4.3 Standards and Calibration. The standards can be prepared according to the respective NIOSH method. Use a minimum of three different standards; select the concentrations to bracket the expected average sample concentration. Perform the calibration before and after each day's sample analyses. Prepare the calibration curve by using the least squares method.

7.4.4 Quality Assurance.

7.4.4.1 Determination of Desorption Efficiency. During the testing program, determine the desorption efficiency in the expected sample concentration range for each batch of adsorption media to be used. Use an internal standard. A minimum desorption efficiency of 50 percent shall be obtained. Repeat the desorption determination until the relative deviation of two consecutive determinations does not exceed 5 percent. Use the average desorption efficiency of these two consecutive determinations for the correction specified in Section 7.4.4.5. If the desorption efficiency of the compound(s) of interest is questionable under actual sampling conditions, use of the Method of Standard Additions may be helpful to determine this value.

7.4.4.2 Determination of Sample Collection Efficiency. For the source samples, analyze the primary and backup portions of the adsorption tubes separately. If the backup portion exceeds 10 percent of the total amount (primary and backup), repeat the sampling with a larger sampling portion.

7.4.4.3 Analysis Audit. Immediately before the sample analyses, analyze the two audits in accordance with Section 7.4.2. The analysis audit shall agree with the audit concentration within 10 percent.

7.4.4.4 Pump Leak Checks and Volume Flow Rate Checks. Perform both of these checks immediately after sampling with all sampling train components in place. Perform all leak checks according to the manufacturer's instructions, and record the results. Use the bubble-tube flowmeter to measure the pump volume flow rate with the orifice used in the test sampling, and the result. If it has changed by more than 5 but less than 20 percent, calculate an average flow rate for the test. If the flow rate has changed by more than 20 percent, recalibrate the pump and repeat the sampling.

7.4.4.5 Calculations. All calculations can be performed according to the respective NIOSH method. Correct all sample volumes to standard conditions. If a sample dilution system has been used, multiply the results by the appropriate dilution ratio. Correct all results by dividing by the desorption efficiency (decimal value). Report results as ppm by volume, dry basis.

7.5 Reporting of Results. At the completion of the field analysis portion of the study, ensure that the data sheets shown in Figure 18-11 have been completed. Summarize this data on the data sheets shown in Figure 18-15.

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I. Name of Company _______________ Date _______

Address _______________

____

Contacts _______________ Phone _______

_______________ _______

Process to be sampled _______________

____

____

Duct or vent to be sampled _______________

____

II. Process description _______________

____

____

____

____

Raw material _______________

____

____

Products _______________

____

____

Operating cycle

Check: Batch _______ Continuous _______ Cyclic _______

Timing of batch or cycle _______________

Best time to test _______________

Figure 18-1. Preliminary survey data sheet.

III. Sampling site

A. Description

Site description _______________

Duct shape and size _______________

Material _______________

Wall thickness _______________ inches

Upstream distance _______ inches _______ diameter

Downstream distance _______ inches _______ diameter

Size of port _______________

Size of access area _______________

Hazards _______________ Ambient temp. _______ °F

B. Properties of gas stream

Temperature _____ °C _____ °F, Data source _______

Velocity _______, Data source _______

Static pressure ___ inches H_2O, Data source _______

Moisture content _______ %, Data source _______

Particulate content _______, Data source _______

Gaseous components

N_2 _______ % Hydrocarbons _____ ppm

O_2 _______ % _______ _____

CO _______ % _______ _____

CO_2 _______ % _______ _____

SO_2 _______ % _______ _____

Hydrocarbon components

_______ _______ ppm

_______ _______ ppm

_______ _______ ppm

_______ _______ ppm

_______ _______ ppm

_______ _______ ppm

Figure 18-1 (continued). Preliminary survey data sheet.

C. Sampling considerations

Location to set up GC ____________________

____

Special hazards to be considered ____________________

____

Power available at duct ____________________

Power available for GC ____________________

Plant safety requirements ____________________

____

Vehicle traffic rules ____________________

____

Plant entry requirements ____________________

____

Security agreements ____________________

____

Potential problems ____________________

____

D. Site diagrams. (Attach additional sheets if required).

Figure 18-1 (continued). Preliminary survey data sheet.

Components to be analyzed	Expected concentration

Suggested chromatographic column ____________________

____

Column flow rate ____ml/min Head pressure____ mm Hg

Column temperature:

Isothermal ________°C

Programmed from ____°C to ____°C at ____°C/min

Injection port/sample loop temperature ______°C

Detector temperature ______°C

Detector flow rates: Hydrogen _____ml/min.

head pressure____ mm Hg

Air/Oxygen _____ml/min.

head pressure____ mm Hg

Chart speed ________ inches/minute

Compound data:

Compound	Retention time	Attenuation

Figure 18-2. Chromatographic conditions data sheet.

Preparation of Standards in Tedlar Bags
and Calibration Curve

	Standards Mixture #1	Standards Mixture #2	Standards Mixture #3
Standards Preparation Data:			
Organic: ____			
Bag number or identification			
Dry gas meter calibration factor			
Final meter reading (liters)			
Initial meter reading (liters)			
Metered volume (liters)			
Average meter temperature (°K)			
Average meter pressure, gauge (mm Hg)			
Average atmospheric pressure (mm Hg)			
Average meter pressure, absolute (mm Hg)			
Syringe temperature (°K) (Section 6.2.2.1)			
Syringe pressure, absolute (mm Hg) (Section 6.2.2.1)			
Volume of gas in syringe (ml) (Section 6.2.2.1)			
Density of liquid organic (g/ml) (Section 6.2.2.2)			
Volume of liquid in syringe (µl) (Section 6.2.2.2)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Carrier gas flow rate (ml/min)			
Column temperature			
Initial (°C)			
Rate change (°C/min)			
Final (°C)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24-hr clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Organic retention time (min)			
Attenuation factor			
Peak height (mm)			
Peak area (mm²)			
Peak area x attenuation factor (mm²)			
Calculated concentration (ppm) (Equation 18-3 or 18-4)			

Plot peak area x attenuation factor against calculated concentration to obtain calibration curve.

Figure 18-3. Standards prepared in Tedlar bags and calibration curve.

Flowmeter Calibration

Flowmeter number or identification ____________

Flowmeter type ____________

Calibration device (x): Bubble meter ___ Spirometer ___ Wet test meter ___

Readings at laboratory conditions:

Laboratory temperature (T_{lab}) ______ °K

Laboratory barometric pressure (P_{lab}) ______ mm Hg

Flow data:

Flowmeter reading (as marked)	Flowmeter temp. (°K)	Flowmeter pressure (absolute)	Calibration device Time (min)	Calibration device gas volume[a]	Calibration device flow rate[b]

a = Volume of gas measured by calibration device, corrected to standard conditions (liters).

b = Calibration device gas volume/time.

Plot flowmeter reading against flow rate (standard conditions), and draw a smooth curve. If the flowmeter being calibrated is a rotameter or other flow device that is viscosity dependent, it may be necessary to generate a "family" of calibration curves that cover the operating pressure and temperature ranges of the flowmeter.

While the following technique should be verified before application, it may be possible to calculate flow rate readings for rotameters at standard conditions Q_{std} as follows:

$$Q_{std} = Q_{lab}\left(\frac{760 \times T_{lab}}{P_{lab} \times 293}\right)^{1/2}$$

Flow rate (laboratory conditions)	Flow rate (standard conditions)

Figure 18-4. Flowmeter calibration.

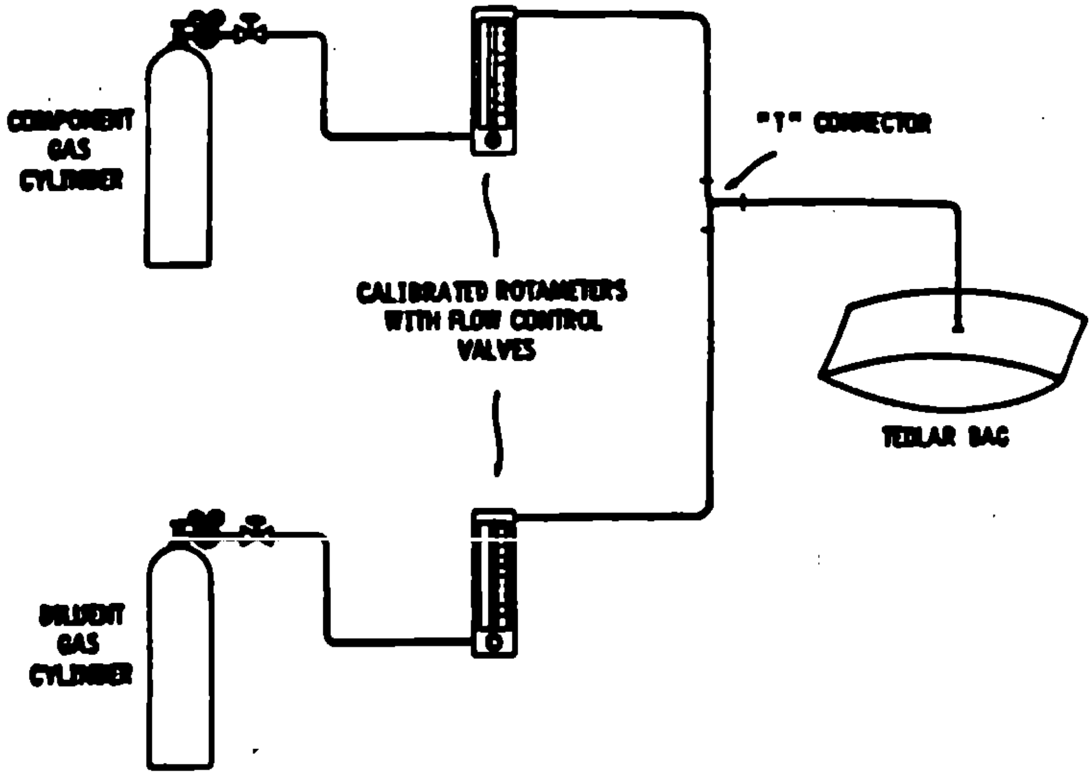

Figure 18-5. Single-stage calibration gas dilution system.

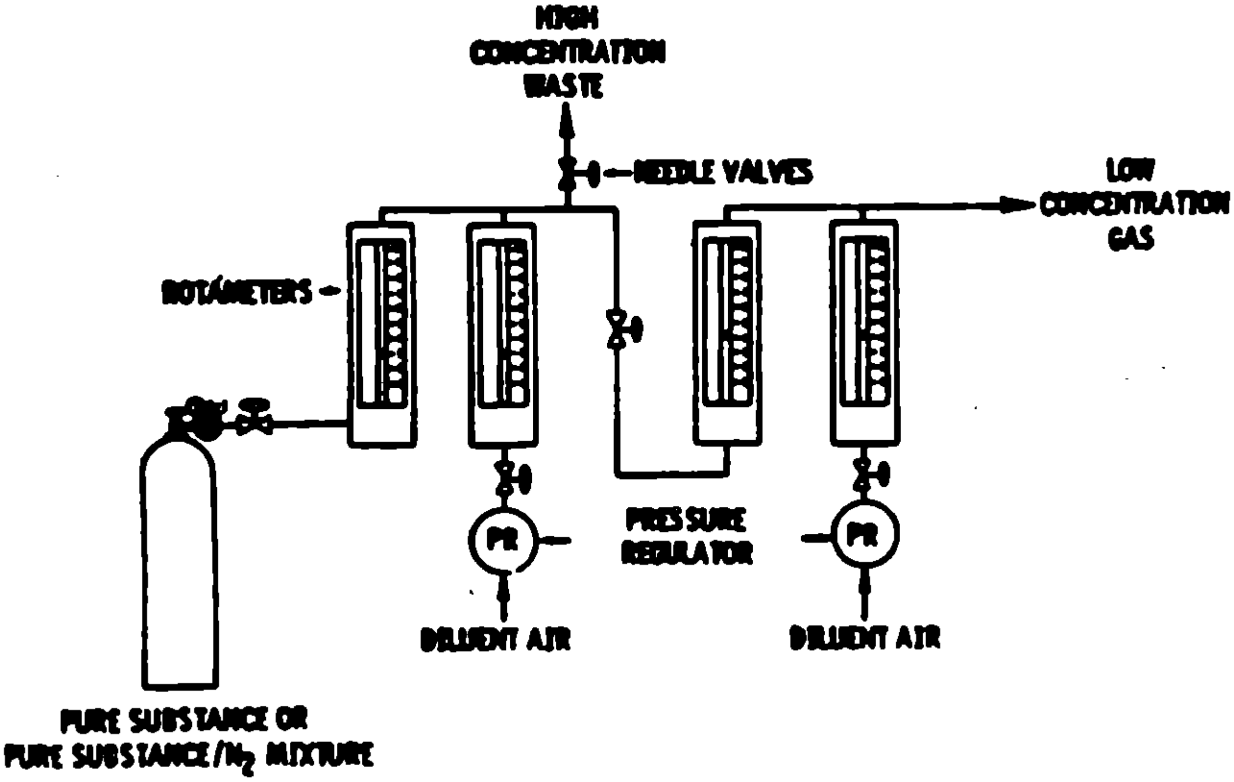

Figure 18-6. Two-stage dilution apparatus.

Preparation of Standards by Dilution of Cylinder Standard

Cylinder standard: Organic ________ Certified concentration ______ ppm

Standards Preparation Data: Date ________

Stage 1	Mixture 1	Mixture 2	Mixture 3
Standard gas flowmeter reading			
Diluent gas flowmeter reading			
Laboratory temperature (°K)			
Barometric pressure (mm Hg)			
Flowmeter gage pressure (mm Hg)			
Flow rate cylinder gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions (ml/min)			
Calculated concentration (ppm)			
Stage 2 (if used)			
Standard gas flowmeter reading			
Diluent gas flowmeter reading			
Flow rate stage 1 gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions (ml/min)			
Calculated concentration (ppm)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Carrier gas flow rate (ml/min)			
Column temperature:			
Initial (°C)			
Program rate (°C/min)			
Final (°C)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24-hr clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Retention time (min)			
Attenuation factor			
Peak area (mm²)			
Peak area x attenuation factor			

Plot peak area x attenuation factor against calculated concentration to obtain calibration curve.

Figure 18-7. Standards prepared by dilution of cylinder standard.

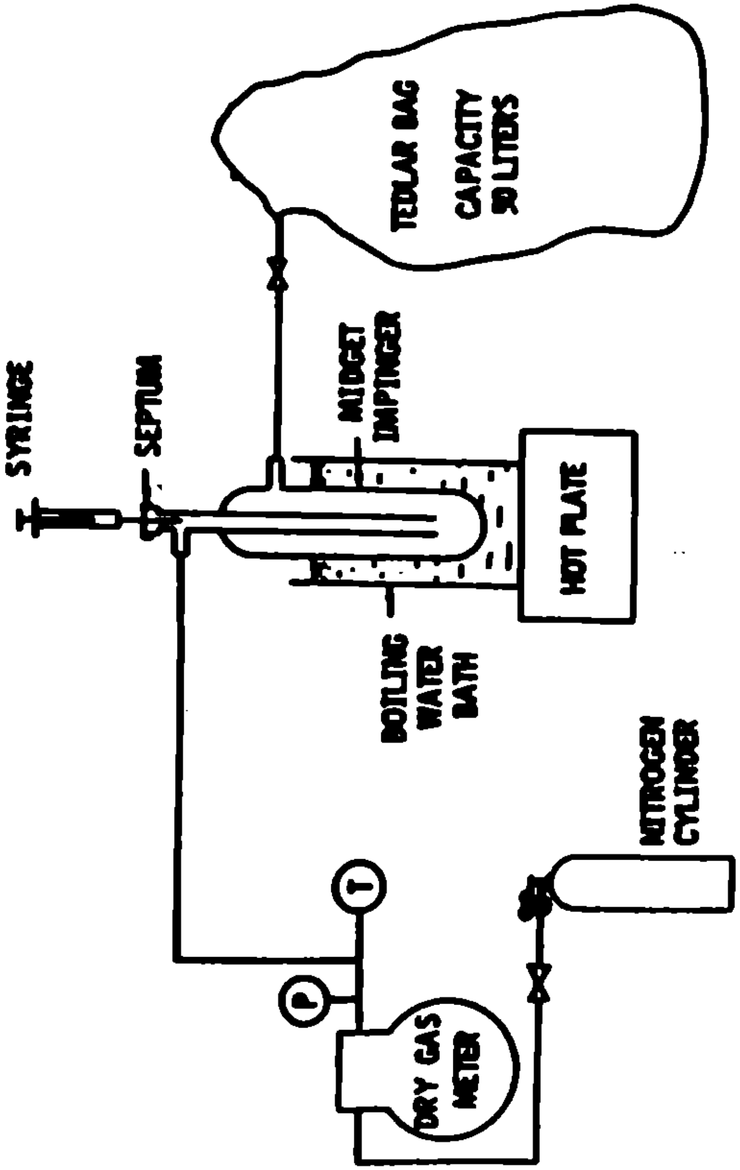

Figure 18-8. Apparatus for preparation of liquid materials.

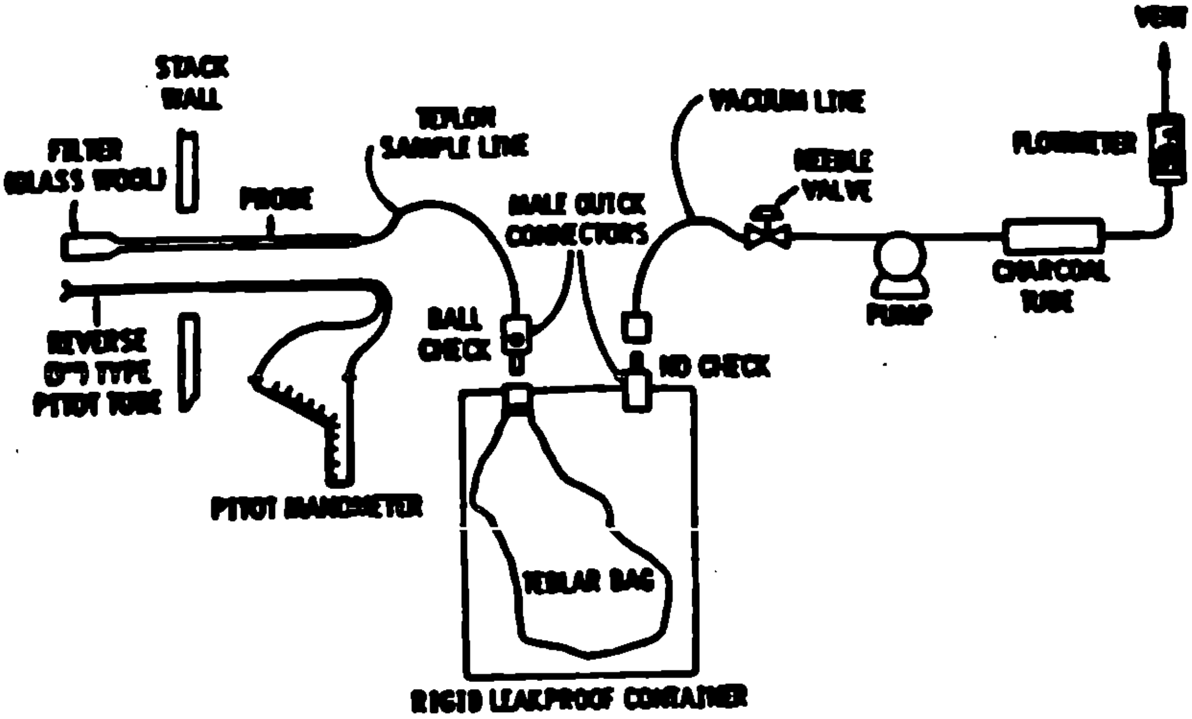

Figure 18-9. Integrated bag sampling train.

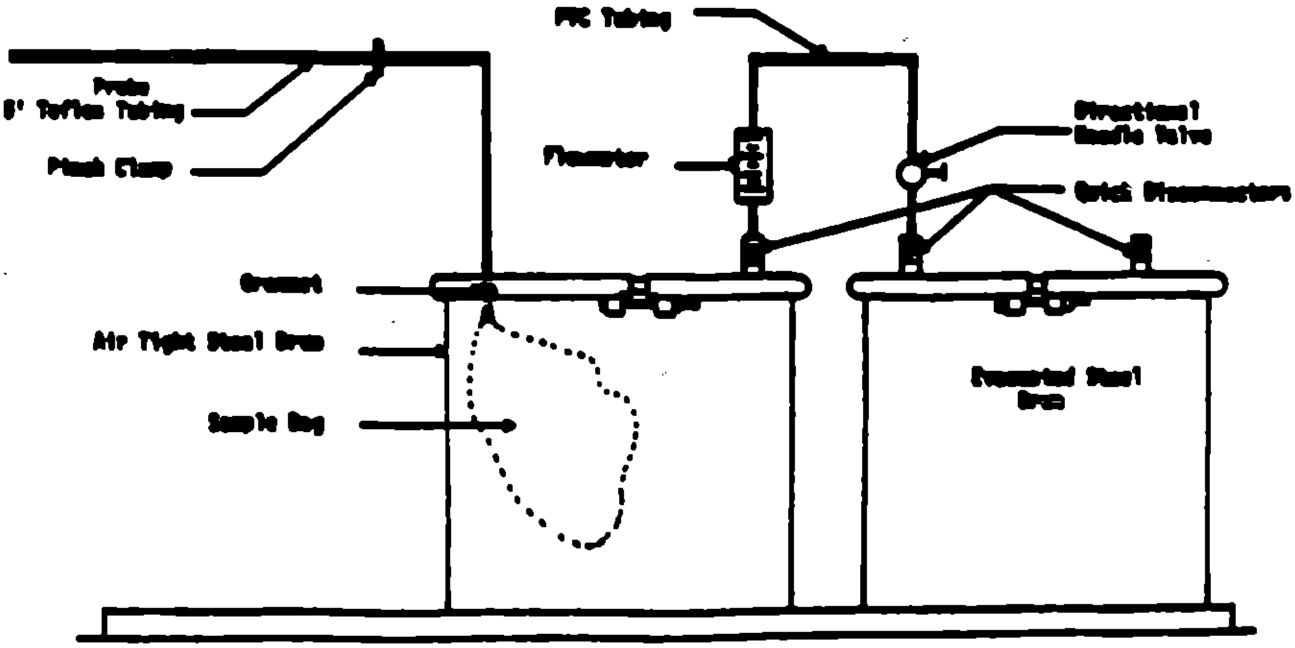

Figure 18-9a. Explosion risk gas sampling method.

Plant__________________ Date__________

Site______________________________

	Sample 1	Sample 2	Sample 3
Source temperature (°C)	______	______	______
Barometric pressure (mm Hg)	______	______	______
Ambient temperature (°C)	______	______	______
Sample flow rate (appr.)	______	______	______
Bag number	______	______	______
Start time	______	______	______
Finish time	______	______	______

Figure 18-10. Field sample data sheet - Tedlar bag collection method.

Plant__________________ Date__________

Location______________________________

1. General information

Source temperature (°C) ______

Probe temperature (°C) ______

Ambient temperature (°C) ______

Atmospheric pressure (mm) ______

Source pressure (″Hg) ______

Absolute source pressure (mm) ______

Sampling rate (liter/min) ______

Sample loop volume (ml) ______

Sample loop temperature (°C) ______

Columnar temperature:

Initial (°C)/time (min) ______

Program rate (°C/min) ______

Final (°C)/ time (min) ______

Carrier gas flow rate (ml/min) ______

Detector temperature (°C) ______

Injection time (24-hour basis) ______

Chart speed (mm/min) ______

Dilution gas flow rate (ml/min) ______

Dilution Gas used (symbol) ______

Dilution ratio ______

Figure 18-11. Field analysis data sheets.

2. Field Analysis Data - Calibration Gas

Run No.__________ Time__________

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Run No.__________ Time__________

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Run No.__________ Time__________

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Figure 18-11 (continued). Field analysis data sheets.

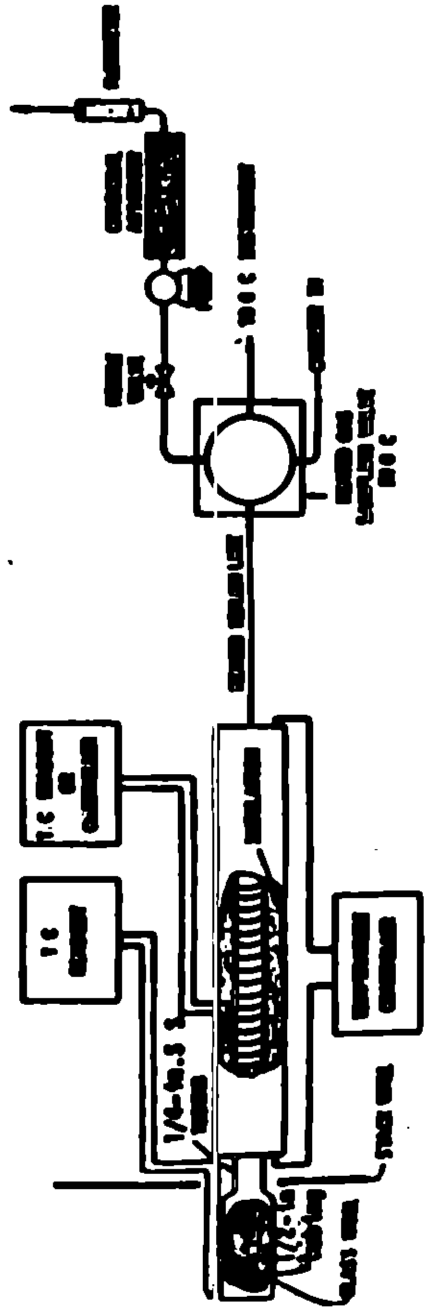

Figure 18-12. Direct interface sampling system.

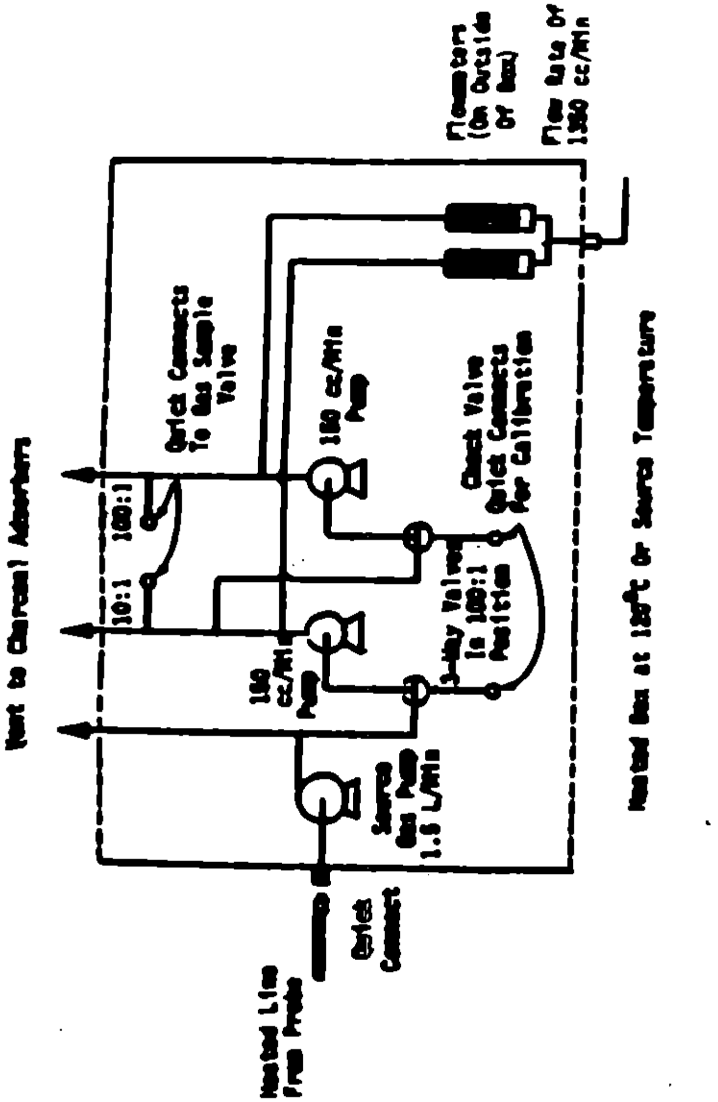

Figure 18-13. Schematic diagram of the heated box required for dilution of sample gas.

GASEOUS ORGANIC SAMPLING AND ANALYSIS CHECK LIST

(Respond with initials or number as appropriate)

		Date
1. Presurvey data:		
A. Grab sample collected	☐	
B. Grab sample analyzed for composition	☐	
Method GC	☐	☐
GC/MS	☐	☐
Other	☐	☐
C. GC-FID analysis performed	☐	
2. Laboratory calibration data:		
A. Calibration curves prepared	☐	
Number of components	☐	☐
Number of concentrations/component (3 required)	☐	☐
B. Audit samples (optional):		
Analysis completed	☐	
Verified for concentration	☐	
OK obtained for field work	☐	
3. Sampling procedures:		
A. Method:		
Bag sample	☐	☐
Direct interface	☐	☐
Dilution interface	☐	☐
B. Number of samples collected	☐	
4. Field analysis:		
A. Total hydrocarbon analysis performed	☐	
B. Calibration curve prepared	☐	
Number of components	☐	☐
Number of concentrations per component (3 required)	☐	☐

Figure 18-14. Sampling and analysis check.

GASEOUS ORGANIC SAMPLING AND ANALYSIS DATA

Plant____________________

Date____________________

Location____________________

	Source sample 1	Source sample 2	Source sample 3
1. General information			
Source temperature (°C)			
Probe temperature (°C)			
Ambient temperature (°C)			
Atmospheric pressure (mm Hg)			
Source pressure (mm Hg)			
Sampling rate (ml/min)			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Sample collection time (24-hr basis)			
Column temperature:			
Initial (°C)			
Program rate (°C/min)			
Final (°C)			
Carrier gas flow rate (ml/min)			
Detector temperature (°C)			
Chart speed (cm/min)			
Dilution gas flow rate (ml/min)			
Diluent gas used (symbol)			
Dilution ratio			

Performed by (signature):____________________

Date:____________________

Figure 18-14. Sampling and analysis sheet.

EPA METHOD 3A

particular caution. Calibration will be affected by variation in stack gas temperature, pressure, compressibility, and molecular weight. Use the procedure in Section 3.3.1. Record all the measuring device parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

4. Calibration

4.1 Flow Rate Measuring Device. Use the procedure in Method 2A, Section 4, and apply the same performance standards. Calibrate the measuring device with the principal stack gas to be measured (e.g., air, nitrogen) against a standard reference meter. A calibrated dry gas meter is an acceptable reference meter. Ideally, calibrate the measuring device in the field with the actual gas to be measured. For measuring devices that have a volume rate readout, calculate the measuring device calibration coefficient, Y_m, for each run as follows:

$$Y_m = \frac{(Q_r)(T_r)P_{bar}}{(Q_m)(T_m)(P_{bar} + P_g)} \qquad \text{Eq. 2D-1}$$

where:

Q_r = reference meter flow rate reading, m³/min (ft³/min).

Q_m = measuring device flow rate reading, m³/min (ft³/min).

T_r = reference meter average absolute temperature, °K (°R).

T_m = measuring device average absolute temperature, °K (°R).

P_{bar} = barometric pressure, mm Hg (in. Hg).

P_g = measuring device average static pressure, mm Hg (in. Hg).

For measuring devices that do not have a readout as flow rate, refer to the manufacturer's instructions to calculate the Q_m corresponding to each Q_r.

4.2 Temperature Gauge. Use the procedure and specifications in Method 2A, Section 4.2. Perform the calibration at a temperature that approximates field test conditions.

4.3 Barometer. Calibrate the barometer to be used in the field test with a mercury barometer prior to the field test.

5. Gas Flow Rate Calculation

Calculate the stack gas flow rate, Q_s, as follows:

$$Q_s = K_1 Y_m Q_m \frac{(P_{bar} + P_g)}{T_m} \qquad \text{Eq. 2D-2}$$

Where:

K_1 = 0.3858 for international system of units (SI); 17.64 for English units.

6. Bibliography

1. Spink, L.K. Principles and Practice of Flowmeter Engineering. The Foxboro Company. Foxboro, MA. 1967.

2. Benedict, Robert P. Fundamentals of Temperature, Pressure, and Flow Measurements. John Wiley and Sons, Inc. New York, NY. 1969.

3. Orifice Metering of Natural Gas. American Gas Association. Arlington, VA. Report No. 3. March 1978. 88 p.

METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO_2), percent oxygen (O_2), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be

made, either an Orsat or a Fyrite[1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO_2 and O_2 concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds *other than CO_2, O_2, CO, and nitrogen (N_2)* are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency.

2. Apparatus

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

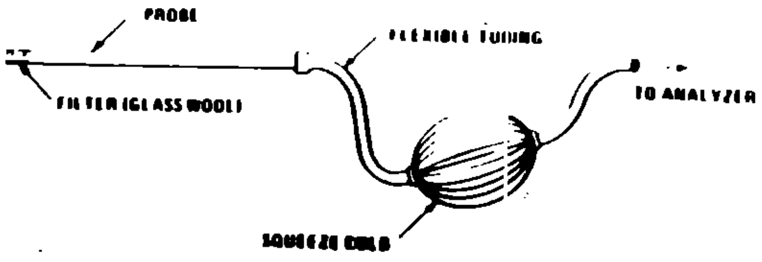

Figure 3.1 Grab sampling train

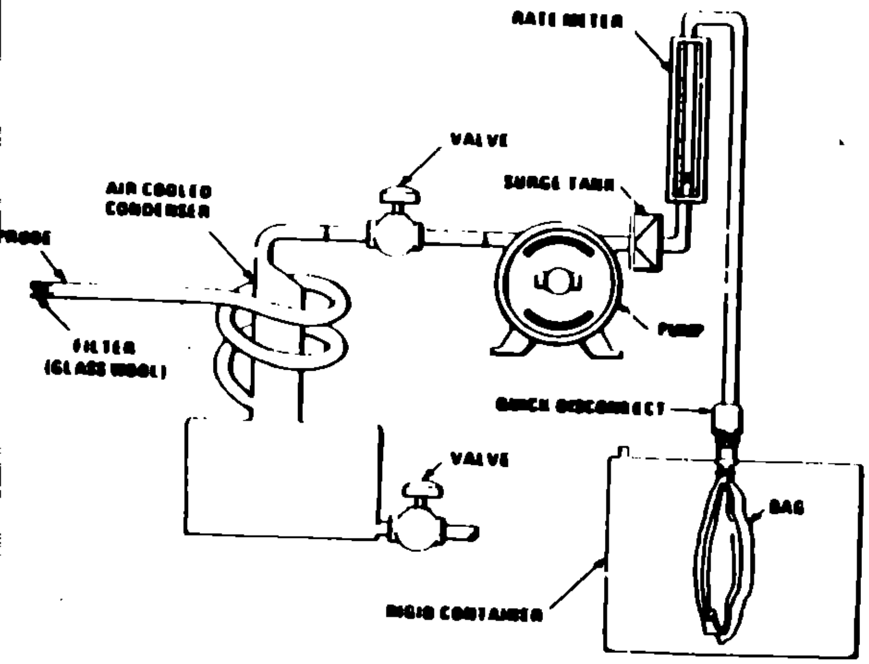

Figure 3.2 Integrated gas sampling train

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2, CO_2, CO, and N_2, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ±2 percent of the selected flow rate. A flow rate range of 500 to 1000 cm³/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater that 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. *Dry Molecular Weight Determination*

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3.1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leaked-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3.2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft³) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak check described in Section 5 be performed before this determination; however, the check is optional.

Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.6 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.6, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4. *Emission Rate Correction Factor or Excess Air Determination*

NOTE: A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

FIGURE 3-3.—SAMPLING RATE DATA

Time	Traverse pt.	Q lpm	% dev.*
Average			

*% Dev. = $(Q - Q_{avg})/Q_{avg} \times 100$ (Must be ≤ |10%|)

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis.

NOTE: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 4.4 be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of a least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft³) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three of four) should be make between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more that (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before an after the analysis.

NOTE: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 4.4.1 be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4.4 Quality Control Procedures.

4.4.1 Data Validation When Both CO_2 and O_2 Are Measured. Although in most instances, only CO_2 or O_2 measurement is required, it is recommended that both CO_2 and O_2 be measured to provide a check on the quality of the data. The following quality control procedure is suggested.

NOTE: Since the method for validating the CO_2 and O_2 analyses is based on combustion of organic and fossil fuels and dilution of the gas stream with air, this method does not apply to sources that (1) remove CO_2 or O_2 through processes other than combustion, (2) add O_2 (e.g., oxygen enrichment) and N_2 in proportions different from that of air, (3) add CO_2 (e.g., cement or lime kilns), or (4) have no fuel factor, F_o, values obtainable (e.g., extremely variable waste mixtures). This method validates the measured proportions of CO_2 and O_2 for the fuel type, but the method does not detect sample dilution resulting from leaks during or after sample collection. The method is applicable for samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO_2 added or removed from the gas stream is not significant in relation to the total CO_2 concentration. The CO_2 concentrations from other types of scrubbers using only water or basic slurry can be significantly affected and would render the F_o check minimally useful.

4.4.1.1 Calculate a fuel factor, F_o, using the following equation:

$$F_o = \frac{20.9 - \%O_2}{\%CO_2}$$

Eq. 3-3

Where:

$\%O_2$ = Percent O_2 by volume (dry basis).
$\%CO_2$ = Percent CO_2 by volume (dry basis).
20.9 = Percent O_2 by volume in ambient air.

If CO is present in quantities measurable by this method, adjust the O_2 and CO_2 values before performing the calculation for F_o as follows:

$$\%CO_2(adj) = \%CO_2 + \%CO$$

$$\%O_2(adj) = \%O_2 - 0.5\ \%CO$$

Where:

%CO = Percent CO by volume (dry basis).

4.4.1.2 Compare the calculated F_o factor with the expected F_o values. The following table may be used in establishing acceptable ranges for the expected F_o if the fuel being burned is known. When fuels are burned in combination, calculate the combined fuel F_d and F_c factors (as defined in Method 19) according to the procedure in Method 19 Section 5.2.3. Then calculate the F_o factor as follows:

$$F_o = \frac{0.209\ F_d}{F_c}$$

Eq. 3-4

Fuel type	F_o range
Coal:	
Anthracite and lignite	1.016-1.130
Bituminous	1.083-1.230
Oil:	
Distillate	1.260-1.413
Residual	1.210-1.370
Gas:	
Natural	1.600-1.836
Propane	1.434-1.586
Butane	1.405-1.553
Wood	1.000-1.120
Wood bark	1.003-1.130

Calculated F_o values beyond the acceptable ranges shown in this table should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ±12 percent is appropriate for the F_o factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate, i.e., if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time-consuming and costly.

5. *Leak-Check Procedure for Orsat Analyzers*

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.3 Record the meniscus position.

5.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

5.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. *Calculations*

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).
%EA = Percent excess air.
$\%CO_2$ = Percent CO_2 by volume (dry basis).
$\%O_2$ = Percent O_2 by volume (dry basis).
%CO = Percent CO by volume (dry basis).
$\%N_2$ = Percent N_2 by volume (dry basis).
0.264 = Ratio of O_2 to N_2 in air, v/v.
0.280 = Molecular weight of N_2 or CO, divided by 100.
0.320 = Molecular weight of O_2 divided by 100.
0.440 = Molecular weight of CO_2 divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of percent O_2, CO, and N_2 (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA = \frac{\%O_2 - 0.5\%\ CO}{0.264\ \%N_2 - (\%O_2 - 0.5\ \%CO)}$$

Eq. 3-1

NOTE: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO)$$

Eq. 3-2

NOTE: The above equation does not consider argon in air (about 0.9 percent, molecular weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. *Bibliography*

1. Altshuller, A. P. *Storage of Gases and Vapors in Plastic Bags.* International Journal of Air and Water Pollution. 6:75-81. 1963.

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4. Mitchell, W. J. and M. R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association 26:491-495. May 1976.

5. Shigehara, R. T., R. M. Neulicht, and W. S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. 4(2):21-26. August, 1976.

METHOD 3A—DETERMINATION OF OXYGEN AND CARBON DIOXIDE CONCENTRATIONS IN EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1. *Applicability and Principle*

1.1 Applicability. This method is applicable to the determination of oxygen (O_2) and carbon dioxide (CO_2) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A sample is continuously extracted from the effluent stream; a portion of the sample stream is conveyed to an instrumental analyzer(s) for determination of O_2 and CO_2 concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

Same as Method 6C, Sections 2.1 and 2.2, except that the span of the monitoring system shall be selected such that the average O_2 or CO_2 concentration is not less than 20 percent of the span.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of the O_2 or CO_2 concentration. The measurement system consists of the same major subsystems as defined in Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, Response Time, and Calibration Curve. Same as Method 6C, Sections 3.3 through 3.8, and 3.10.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. *Measurement System Performance Specifications*

Same as Method 6C, Sections 4.1 through 4.4.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for O_2 or CO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe. A leak-free probe, of sufficient length to traverse the sample points.

5.1.2 Sample Line. Tubing, to transport the sample gas from the probe to the moisture removal system. A heated sample line is not required for systems that measure the O_2 or CO_2 concentration on a dry basis, or transport dry gases.

5.1.3 Sample Transport Line, Calibration Valve Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.3 through 5.1.9, and 5.1.11, except that the requirements to use stainless steel, Teflon, and nonreactive glass filters do not apply.

5.1.4 Gas Analyzer. An analyzer to determine continuously the O_2 or CO_2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g.,

Method 3 - Gas Analysis for the Determination of Dry Molecular Weight

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability.

1.1.1 This method is applicable for determining carbon dioxide (CO_2) and oxygen (O_2) concentrations and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, carbon monoxide (CO), and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

1.1.2 Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight; and (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency (EPA).

1.1.3 **Note.** Mention of trade names or specific products does not constitute endorsement by EPA.

1.2 **Principle.** A gas sample is extracted from a stack by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2, percent O_2, and if necessary, for percent CO. For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.

2. APPARATUS

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 **Probe.** Stainless steel or borosilicate glass tubing equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials, inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions, may be used for the probe. Examples of such materials are aluminum, copper, quartz glass, and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. Same as in Section 2.1.1.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser no greater than 250 ml that will not remove O_2, CO_2, CO, and N_2, to remove excess moisture which would interfere with the operation of the pump and flowmeter.

2.2.3 Valve. A needle valve, to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. A rotameter, or equivalent rate meter, capable of measuring flow rate to within 2 percent of the selected flow rate. A flow rate range of 500 to 1000 cc/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run. A capacity in the range of 55 to 90 liters is suggested. To leak check the bag, connect it to a water manometer, and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm (2 to 4 in.) H_2O and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.), for the flexible bag leak check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm (30 in.) Hg, for the sampling train leak check.

2.3 Analysis. An Orsat or Fyrite type combustion gas analyzer. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

3. SINGLE-POINT, GRAB SAMPLING AND ANALYTICAL PROCEDURE

3.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight. If an Orsat analyzer is used, it is recommended that the analyzer be leak checked by following the procedure in Section 6; however, the leak check is optional.

3.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer, and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 7.2.

3.4 Repeat the sampling, analysis, and calculation procedures until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

4. SINGLE-POINT, INTEGRATED SAMPLING AND ANALYTICAL PROCEDURE

4.1 The sampling point in the duct shall be located as specified in Section 3.1.

4.2 Leak check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just before sampling, leak check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag, and make sure that all connections are tight.

4.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

4.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that Orsat leak check described in Section 6, be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 7.2.

4.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

5. MULTI-POINT, INTEGRATED SAMPLING AND ANALYTICAL PROCEDURE

5.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

5.2 Follow the procedures outlined in Sections 4.2 through 4.5, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

6. LEAK-CHECK PROCEDURE FOR ORSAT ANALYZER

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak checked on site before the flue gas sample is introduced into it. The procedure for leak checking an Orsat analyzer is as follows:

6.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.

6.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.

6.3 Record the meniscus position.

6.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

6.5 For the Orsat analyzer to pass the leak check, two conditions must be met:

6.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

6.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

6.6 If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease leaking stopcocks. Replace leaking rubber connections. After the analyzer is reassembled, repeat the leak-check procedure.

7. CALCULATIONS

7.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).

$\%CO_2$ = Percent CO_2 by volume, dry basis.

$\%O_2$ = Percent O_2 by volume, dry basis.

%CO = Percent CO by volume, dry basis.

$\%N_2$ = Percent N_2 by volume, dry basis.

0.280 = Molecular weight of N_2 or CO, divided by 100.

0.320 = Molecular weight of O_2 divided by 100.

0.440 = Molecular weight of CO_2 divided by 100.

7.2 **Dry Molecular Weight.** Use Equation 3-1 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO) \qquad \text{Eq. 3-1}$$

<u>Note:</u> The above equation does not consider argon in air (about 0.9 percent, molecular weight of 39.9). A negative error of about 0.4 percent is introduced. The tester may choose to include argon in the analysis using procedures subject to approval of the Administrator.

8. BIBLIOGRAPHY

1. Altshuller, A.P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. 6:75-81. 1963.

2. Conner, William D. and J.S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. 25:291-297. 1964.

3. Burrell Manual for Gas Analysts, Seventh edition. Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, PA. 15219. 1951.

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5. Shigehara, R.T., R.M. Neulicht, and W.S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. 4(2):21-26. August 1976.

Time	Traverse pt.	Q, liter/min	% dev.[a]
Average			

[a] % dev. = $(Q - Q_{avg})/Q_{avg} \times 100$ (Must be $\leq|10\%|$)

Figure 3-3. Sampling rate data.

EPA METHOD 10

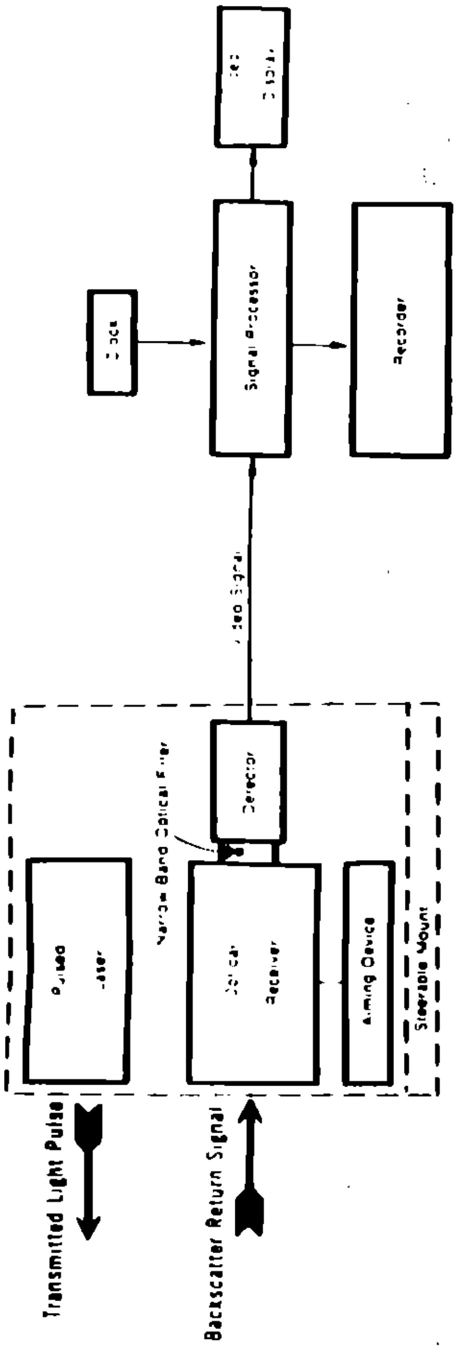

Figure AM1-VII. Functional Block Diagram of a Basic Lidar System

4.2 Performance Evaluation Tests. The owner of a lidar system shall subject such a lidar system to the performance verification tests described in Section 3, prior to first use of this method. The annual calibration shall be performed for three separate, complete runs and the results of each should be recorded. The requirements of Section 3.3.1 must be fulfilled for each of the three runs.

Once the conditions of the annual calibration are fulfilled the lidar shall be subjected to the routine verification for three separate complete runs. The requirements of Section 3.3.2 must be fulfilled for each of the three runs and the results should be recorded. The Administrator may request that the results of the performance evaluation be submitted for review.

5. *References*

5.1 The Use of Lidar for Emissions Source Opacity Determination, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA-330/1-79-003-R, Arthur W. Dybdahl, current edition (NTIS No. PB81-246662).

5.2 Field Evaluation of Mobile Lidar for the Measurement of Smoke Plume Opacity, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA/NEIC-TS-128, February 1976.

5.3 Remote Measurement of Smoke Plume Transmittance Using Lidar, C. S. Cook, G. W. Bethke, W. D. Conner (EPA/RTP). Applied Optics 11, pg 1742. August 1972.

5.4 Lidar Studies of Stack Plumes in Rural and Urban Environments, EPA-650/4-73-002, October 1973.

5.5 American National Standard for the Safe Use of Lasers ANSI Z 136.1-176, March 8, 1976.

5.6 U.S. Army Technical Manual TB MED 279, Control of Hazards to Health from Laser Radiation, February 1969.

5.7 Laser Institute of America Laser Safety Manual, 4th Edition.

5.8 U.S. Department of Health, Education and Welfare, Regulations for the Administration and Enforcement of the Radiation Control for Health and Safety Act of 1968, January 1976.

5.9 Laser Safety Handbook, Alex Mallow, Leon Chabot, Van Nostrand Reinhold Co., 1978.

METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 Applicability. This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and Sensitivity*

2.1 Range. 0 to 1,000 ppm.

2.2 Sensitivity. Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences*

Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H_2O) and carbon dioxide (CO_2) are 3.5 percent H_2O per 7 ppm CO and 10 percent CO_2 per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H_2O per 25 ppm CO and 10 percent CO_2 per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and Accuracy*

4.1 Precision. The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 Accuracy. The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus*

5.1 Continuous Sample (Figure 10-1).

5.1.1 Probe. Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2 Integrated Sample (Figure 10-2).

5.2.1 Probe. Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2.3 Valve. Needle valve, or equivalent, to to adjust flow rate.

5.2.4 Pump. Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 Rate Meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min (0.035 cfm).

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

5.2.6 Flexible Bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft³). Leak test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 Pitot Tube. Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 Analysis (Figure 10-3).

5.3.1 Carbon Monoxide Analyzer. Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 Drying Tube. To contain approximately 200 g of silica gel.

5.3.3 Calibration Gas. Refer to section 6.1.

5.3.4 Filter. As recommended by NDIR manufacturer.

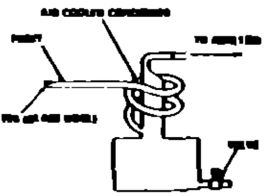

Figure 10-1. Continuous sampling train.

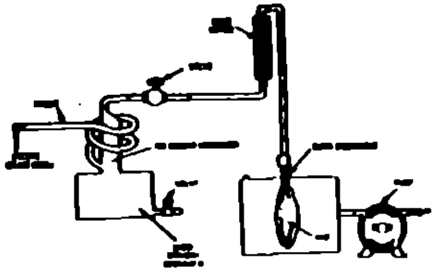

Figure 10-2. Integrated gas-sampling train.

5.3.5 CO_2 Removal Tube. To contain approximately 500 g of ascarite.

5.3.6 Ice Water Bath. For ascarite and silica gel tubes.

5.3.7 Valve. Needle valve, or equivalent, to adjust flow rate.

5.3.8 Rate Meter. Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min (0.035 cfm) through NDIR.

5.3.9 Recorder (*optional*). To provide permanent record of NDIR readings.

6. *Reagents*

6.1 Calibration Gases. Known concentration of CO in nitrogen (N_2) for instrument span, prepurified grade of N_2 for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

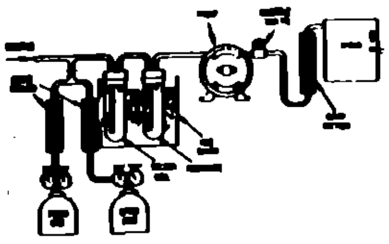

Figure 10-3. Analytical equipment.

6.2 Silica Gel. Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 Ascarite. Commercially available.

7. *Procedure*

7.1 Sampling.

7.1.1 Continuous Sampling. Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See section 7.2 and 8). CO_2 content of the gas may be determined by using the Method 3 integrated sample procedure, or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 Integrated Sampling. Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO_2 content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.2 CO Analysis. Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in section 8. Purge analyzer with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration*

Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of 1 hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO_2 removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1—FIELD DATA

	Comments
Location	
Test	
Date	
Operator	

Clock time	Rotameter setting, liters per minute (cubic feet per minute)

9. *Calculation*

Concentration of carbon monoxide. Calculate the concentration of carbon monoxide in the stack using Equation 10-1.

$$C_{CO\,stack} = C_{CO\,NDIR}(1 - F_{CO_2}) \qquad \text{Eq. 10-1}$$

Where:

$C_{CO\,stack}$ = Concentration of CO in stack, ppm by volume (dry basis).

$C_{CO\,NDIR}$ = Concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

F_{CO_2} = Volume fraction of CO_2 in sample, i.e., percent CO_2 from Orsat analysis divided by 100

10. *Alternative Procedures*

10.1 Interference Trap. The sample conditioning system described in Method 10A, sections 2.1.2 and 4.2, may be used as an alternative to the silica gel and ascarite traps.

11. *Bibliography*

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11.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, PA.

11.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, CA. October 1967.

11.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, NJ.

11.6 UNOR Infrared Gas Analyzers, Bendix Corp., Ronceverte, WV

ADDENDA

A. PERFORMANCE SPECIFICATIONS FOR NDIR CARBON MONOXIDE ANALYZERS

Range (minimum)	0-1000 ppm
Output (minimum)	0-10mV
Minimum detectable sensitivity	20 ppm
Rise time, 90 percent (maximum)	30 seconds
Fall time, 90 percent (maximum)	30 seconds
Zero drift (maximum)	10% in 8 hours
Span drift (maximum)	10% in 8 hours
Precision (minimum)	±2% of full scale
Noise (maximum)	±1% of full scale
Linearity (maximum deviation)	2% of full scale
Interference rejection ratio	CO_2—1000 to 1, H_2O—500 to 1

B. *Definitions of Performance Specifications.*

Range—The minimum and maximum measurement limits.

Output—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

Full scale—The maximum measuring limit for a given range.

Minimum detectable sensitivity—The smallest amount of input concentration that can be detected as the concentration approaches zero.

Accuracy—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

Time to 90 percent response—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

Rise Time (90 percent)—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

Fall Time (90 percent)—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

Zero Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

Span Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

Precision—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

Noise—Spontaneous deviations from a mean output not caused by input concentration changes.

Linearity—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

METHOD 10A—DETERMINATION OF CARBON MONOXIDE EMISSIONS IN CERTIFYING CONTINUOUS EMISSION MONITORING SYSTEMS AT PETROLEUM REFINERIES

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of carbon monoxide (CO) at petroleum refineries. This method serves as the reference method in the relative accuracy test for nondispersive infrared (NDIR) CO continuous emission monitoring systems (CEMS's) that are required to be installed in petroleum refineries on fluid catalytic cracking unit catalyst regenerators [40 CFR Part 60.105(a)(2)].

1.2 Principle. An integrated gas sample is extracted from the stack, passed through an alkaline permanganate solution to remove sulfur and nitrogen oxides, and collected in a Tedlar bag. The CO concentration in the sample is measured spectrophotometrically using the reaction of CO with p-sulfaminobenzoic acid.

1.3. Range and Sensitivity.

1.3.1 Range. Approximately 3 to 1800 ppm CO. Samples having concentrations below 400 ppm are analyzed at 425 nm, and samples having concentrations above 400 ppm are analyzed at 600 nm.

1.3.2 Sensitivity. The detection limit is 3 ppm based on three times the standard deviation of the mean reagent blank values.

1.4 Interferences. Sulfur oxides, nitric oxide, and other acid gases interfere with the colorimetric reaction. They are removed by passing the sampled gas through an alkaline potassium permanganate scrubbing solution. Carbon dioxide (CO_2) does not interfere, but, because it is removed by the scrubbing solution, its concentration must be measured independently and an appropriate volume correction made to the sampled gas.

1.5 Precision, Accuracy, and Stability.

1.5.1 Precision. The estimated intralaboratory standard deviation of the method is 3 percent of the mean for gas samples analyzed in duplicate in the concentration range of 39 to 412 ppm. The interlaboratory precision has not been established.

1.5.2 Accuracy. The method contains no significant biases when compared to an NDIR analyzer calibrated with National Bureau of Standards (NBS) standards.

1.5.3 Stability. The individual components of the colorimetric reagent are stable for at least 1 month. The colorimetric reagent must be used within 2 days after preparation to avoid excessive blank correction. The samples in the Tedlar[1] bag should be stable for at least 1 month if the bags are leak-free.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 10A-1, and component parts are discussed below:

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[1] Mention of trade names or commercial products in this publication does not constitute the endorsement or recommendation for use by the Environmental Protection Agency.

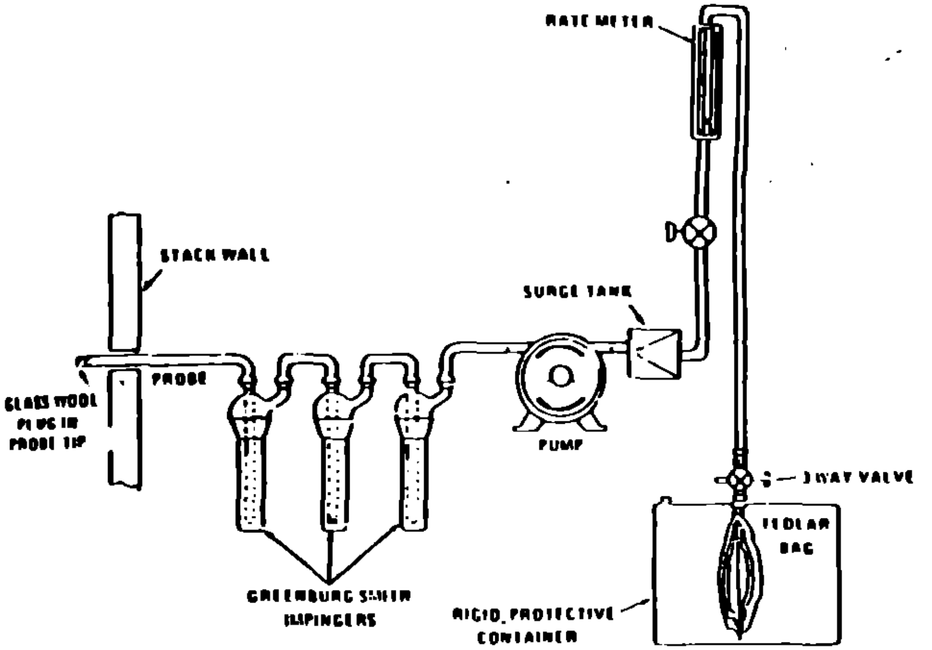

Figure 10A-1. Sampling train.

2.1.1 Probe. Stainless steel, sheathed Pyrex glass, or equivalent, equipped with a glass wool plug to remove particulate matter.

2.1.2 Sample Conditioning System. Three Greenburg-Smith impingers connected in series with leak-free connections.

2.1.3 Pump. Leak-free pump with stainless steel and Teflon parts to transport sample at a flow rate of 300 ml/min to the flexible bag.

2.1.4 Surge Tank. Installed between the pump and the rate meter to eliminate the pulsation effect of the pump on the rate meter.

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The sampling train is assembled as shown in Figure 6A-1, except the isopropanol bubbler is not included. The probe must be heated to a temperature sufficient to prevent water condensation and must include a filter (either in-stack, out-of-stack, or both) to prevent particulate entrainment in the peroxide impingers. The electric supply for the probe heat should be continuous and separate from the timed operation of the sample pump.

Adjust the timer-switch to operate in the "on" position from 2 to 4 minutes on a 2-hour repeating cycle or other cycle specified in the applicable regulation. Other timer sequences may be used with the restriction that the total sample volume collected is between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

Add cold water to the tank until the impingers and bubblers are covered at least two-thirds of their length. The impingers and bubbler tank must be covered and protected from intense heat and direct sunlight. If freezing conditions exist, the impinger solution and the water bath must be protected.

NOTE: Sampling may be conducted continuously if a low flow-rate sample pump (20 to 40 ml/min for the reagent volumes described in this method) is used. Then the timer-switch is not necessary. In addition, if the sample pump is designed for constant rate sampling, the rate meter may be deleted. The total gas volume collected should be between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

4.1.2 *Leak-Check Procedure.* The leak-check procedure is the same as described in Method 6, Section 4.1.2.

4.1.3 Sample Collection. Record the initial dry gas meter reading. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the first impinger (or filter), and start the timer and the sample pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Assure that the timer is operating as intended, i.e., in the "on" position for the desired period and the cycle repeats as required.

During the 24-hour sampling period, record the dry gas meter temperature one time between 9:00 a.m. and 11:00 a.m., and the barometric pressure.

At the conclusion of the run, turn off the timer and the sample pump, remove the probe from the stack, and record the final gas meter volume reading. Conduct a leak check as described in Section 4.1.2. If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for leakage. Repeat the steps in this section (4.1.3) for successive runs.

4.2 Sample Recovery. The procedures for sample recovery (moisture measurement, peroxide solution, and CO_2 absorber) are the same as in Method 6A, Section 4.2.

4.3 Sample Analysis. Analysis of the peroxide impinger solutions is the same as in Method 6, Section 4.3.

4.4 Quality Assurance (QA) Audit Samples. Only when this method is used for compliance determinations, obtain an audit sample set as directed in Section 3.3.6 of Method 6. Analyze the audit samples at least once for every 30 days of sample collection, and report the results as directed in Section 4.4 of Method 6. The analyst performing the sample analyses shall perform the audit analyses. If more than one analyst performed the sample analyses during the 30-day sampling period, each analyst shall perform the audit analyses and all audit results shall be reported. Acceptance criteria for the audit results are the same as in Method 6.

5. *Calibration*

5.1 Metering System.

5.1.1 Initial Calibration. The initial calibration for the volume metering system is the same as for Method 6, Section 5.1.1.

5.1.2 Periodic Calibration Check. After 30 days of operation of the test train, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (1) The leak check is not to be conducted, (2) three or more revolutions of the dry gas meter must be used, and (3) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor determined in Section 5.1.1, then the dry gas meter volumes obtained during the test series are acceptable and use of the train can continue. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1; and for the calculations for the preceding 30 days of data, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run. Use the latest calibration factor for succeeding tests.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers initially and at 30-day intervals.

5.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instructions.

5.4 Barometer. Calibrate against a mercury barometer initially and at 30-day intervals.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

6. *Calculations*

The nomenclature and calculation procedures are the same as in Method 6A with the following exceptions:

P_{bar} = Initial barometric pressure for the test period, mm Hg.

T_m = Absolute meter temperature for the test period, °K.

7. *Emission Rate Procedure*

The emission rate procedure is the same as described in Method 6A, Section 7, except that the timer is needed and is operated as described in this method. Only when this method is used for compliance determinations, perform the QA audit analyses as described in Section 4.4.

8. *Bibliography*

The bibliography is the same as described in Method 6A, section 7, with the addition of the following:

8.1 Butler, Frank E; J.E. Knoll, J.C. Suggs, M.R. Midgett, and W. Mason. The Collaborative Test of Method 6B: Twenty-Four-Hour Analysis of SO_2 and CO_2. JAPCA, Vol. 33, No. 10, October 1983.

METHOD 6C—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1. *Applicability and Principle*

1.1 *Applicability.* This method is applicable to the determination of sulfur dioxide (SO_2) concentrations in controlled and uncontrolled emissions from stationary sources only when specified within the regulations.

1.2 Principle. A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental analyzer for determination of SO_2 gas concentration using an ultraviolet (UV), nondispersive infrared (NDIR), or fluorescence analyzer. Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

2.1 *Analytical Range.* The analytical range is determined by the instrumental design. For this method, a portion of the analytical range is selected by choosing the span of the monitoring system. The span of the monitoring system shall be selected such that the pollutant gas concentration equivalent to the emission standard is not less than 30 percent of the span. If at any time during a run the measured gas concentration exceeds the span, the run shall be considered invalid.

2.2 Sensitivity. The minimum detectable limit depends on the analytical range, span, and signal-to-noise ratio of the measurement system. For a well designed system, the minimum detectable limit should be less than 2 percent of the span.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface. That portion of a system used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the analyzers from the effects of the stack effluent.

3.1.2 Gas Analyzer. That portion of the system that senses the gas to be measured and generates an output proportional to its concentration.

3.1.3 Data Recorder. A strip chart recorder, analog computer, or digital recorder for recording measurement data from the analyzer output.

3.2 Span. The upper limit of the gas concentration measurement range displayed on the data recorder.

3.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

3.4 Analyzer Calibration Error. The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the calibration gas when the calibration gas is introduced directly to the analyzer.

3.5 Sampling System Bias. The difference between the gas concentrations exhibited by the measurement system when a known concentration gas is introduced at the outlet of the sampling probe and when the same gas is introduced directly to the analyzer.

3.6 Zero Drift. The difference in the measurement system output reading from the initial calibration response at the zero concentration level after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

3.7 Calibration Drift. The difference in the measurement system output reading from the initial calibration response at a mid-range calibration value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

3.8 Response Time. The amount of time required for the measurement system to display 95 percent of a step change in gas concentration on the data recorder.

3.9 Interference Check. A method for detecting analytical interferences and excessive biases through direct comparison of gas concentrations provided by the measurement system and by a modified Method 6 procedure. For this check, the modified

Method 6 samples are acquired at the sample by-pass discharge vent.

3.10 Calibration Curve. A graph or other systematic method of establishing the relationship between the analyzer response and the actual gas concentration introduced to the analyzer.

4. *Measurement System Performance Specifications*

4.1 Analyzer Calibration Error. Less than ±2 percent of the span for the zero, mid-range, and high-range calibration gases.

4.2 Sampling System Bias. Less than ±5 percent of the span for the zero, and mid- or high-range calibration gases.

4.3 Zero Drift. Less than ±3 percent of the span over the period of each run.

4.4 Calibration Drift. Less than ±3 percent of the span over the period of each run.

4.5 Interference Check. Less than ±7 percent of the modified Method 6 result for each run.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for SO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1. The essential components of the measurement system are described below:

5.1.1 Sample Probe. Glass, stainless steel, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation.

5.1.2 Sample Line. Heated (sufficient to prevent condensation) stainless steel or Teflon tubing, to transport the sample gas to the moisture removal system.

5.1.3 Sample Transport Lines. Stainless steel or Teflon tubing, to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

5.1.4 Calibration Valve Assembly. A three-way valve assembly, or equivalent, for blocking the sample gas flow and introducing calibration gases to the measurement system at the outlet of the sampling probe when in the calibration mode.

5.1.5 Moisture Removal System. A refrigerator-type condenser or similar device (e.g., permeation dryer), to remove condensate continuously from the sample gas while maintaining minimal contact between the condensate and the sample gas. The moisture removal system is not necessary for analyzers that can measure gas concentrations on a wet basis; for these analyzers, (1) heat the sample line and all interface components up to the inlet of the analyzer sufficiently to prevent condensation, and (2) determine the moisture content and correct the measured gas concentrations to a dry basis using appropriate methods, subject to the approval of the Administrator. The determination of sample moisture content is not necessary for pollutant analyzers that measure concentrations on a wet basis when (1) a wet basis CO_2 analyzer operated according to Method 3A is used to obtain simultaneous measurements, and (2) the pollutant/CO_2 measurements are used to determine emissions in units of the standard.

5.1.6 Particulate Filter. An in-stack or heated (sufficient to prevent water condensation) out-of-stack filter. The filter shall be borosilicate or quartz glass wool, or glass fiber mat. Additional filters at the inlet or outlet of the moisture removal system and inlet of the analyzer may be used to prevent accumulation of particulate material in the measurement system and extend the useful life of the components. All filters shall be fabricated of materials that are nonreactive to the gas being sampled.

5.1.7 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The pump may be constructed of any material that is nonreactive to the gas being sampled.

5.1.8 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent.

(NOTE: The tester may elect to install a back-pressure regulator to maintain the sample gas manifold at a constant pressure in order to protect the analyzer(s) from overpressurization, and to minimize the need for flow rate adjustments.)

5.1.9 Sample Gas Manifold. A sample gas manifold, to divert a portion of the sample gas stream to the analyzer, and the remainder to the by-pass discharge vent. The sample gas manifold should also include provisions for introducing calibration gases directly to the analyzer. The manifold may be constructed of any material that is nonreactive to the gas being sampled.

5.1.10 Gas Analyzer. A UV or NDIR absorption or fluorescence analyzer, to determine continuously the SO_2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

(NOTE: Housing the analyzer(s) in a clean, thermally-stable, vibration-free environment will minimize drift in the analyzer calibration.)

5.1.11 Data Recorder. A strip chart recorder, analog computer, or digital recorder, for recording measurement data. The data recorder resolution (i.e., readability) shall be 0.5 percent of span. Alternatively, a digital or analog meter having a resolution of 0.5 percent of span may be used to obtain the analyzer responses and the readings may be recorded manually. If this alternative is used, the readings shall be obtained at equally spaced intervals over the duration of the sampling run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be obtained. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be obtained.

5.2 Method 6 Apparatus and Reagents. The apparatus and reagents described in Method 6, and shown by the schematic of the sampling train in Figure 6C-2, to conduct the interference check.

5.3 SO_2 Calibration Gases. The calibration gases for the gas analyzer shall be SO_2 in N_2 or SO_2 in air. Alternatively, SO_2/CO_2, SO_2/O_2, or $SO_2/CO_2/O_2$ gas mixtures in N_2 may be used. For fluorescence-based analyzers, the O_2 and CO_2 concentrations of the calibration gases as introduced to the analyzer shall be within 1 percent (absolute) O_2 and 1 percent (absolute) CO_2 of the O_2 and Co_2 concentrations of the effluent samples as introduced to the analyzer. Alternatively, for fluorescence-based analyzers, use calibration blends of SO_2 in air and the nomographs provided by the vendor to determine the quenching correction factor (the effluent O_2 and CO_2 concentrations must be known). Use three calibration gases as specified below:

5.3.1 High-Range Gas. Concentration equivalent to 80 to 90 percent of the span.

5.3.2 Mid-Range Gas. Concentration equivalent to 40 to 60 percent of the span.

5.3.3 Zero Gas. Concentration of less than 0.25 percent of the span. Purified ambient air may be used for the zero gas by passing air through a charcoal filter, or through one or more impingers containing a solution of 3 percent H_2O_2.

6. *Measurement System Performance Test Procedures*

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. There are two alternatives for establishing the concentrations of calibration gases. Alternative Number 1 is preferred.

6.1.1 Alternative Number 1. Use of calibration gases that are analyzed following the Environmental Protection Agency Traceability Protocol Number 1 (see Citation 1 in the Bibliography). Obtain a certification from the gas manufacturer that Protocol Number 1 was followed.

6.1.2 Alternative Number 2. Use of calibration gases not prepared according to Protocol Number 1. If this alternative is chosen, obtain gas mixtures with a manufacturer's tolerance not to exceed ±2 percent of the tag value. Within 6 months before the emission test, analyze each of the calibration gases in triplicate using Method 6. Citation 2 in the Bibliography describes procedures and techniques that may be used for this analysis. Record the results on a data sheet (example is shown in Figure 6C-3). Each of the individual SO_2 analytical results for each calibration gas shall be within 5 percent (or 5 ppm, whichever is greater) of the triplicate set average; otherwise, discard the entire set, and repeat the triplicate analyses. If the average of the triplicate analyses is within 5 percent of the calibration gas manufacturer's cylinder tag value, use the tag value; otherwise, conduct at least three additional analyses until the results of six consecutive runs agree with 5 percent (or 5 ppm, whichever is greater) of their average. Then use this average for the cylinder value.

6.2 Measurement System Preparation. Assemble the measurement system by following the manufacturer's written instructions for preparing and preconditioning the gas analyzer and, as applicable, the other system components. Introduce the calibration gases in any sequence, and make all necessary adjustments to calibrate the analyzer and the data recorder. Adjust system components to achieve correct sampling rates.

6.3 Analyzer Calibration Error. Conduct the analyzer calibration error check by introducing calibration gases to the measurement system at any point upstream of the gas analyzer as follows:

6.3.1 After the measurement system has been prepared for use, introduce the zero, mid-range, and high-range gases to the analyzer. During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate at the analyzer. Record the analyzer responses to each calibration gas on a form similar to Figure 6C-4.

NOTE: A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

6.3.2 The analyzer calibration error check shall be considered invalid if the gas concentration displayed by the analyzer exceeds ±2 percent of the span for any of the calibration gases. If an invalid calibration is exhibited, take corrective action, and repeat the analyzer calibration error check until acceptable performance is achieved.

6.4 Sampling System Bias Check. Perform the sampling system bias check by introducing calibration gases at the calibration valve installed at the outlet of the sampling probe. A zero gas and either the mid-range or high-range gas, whichever most closely approximates the effluent concentrations, shall be used for this check as follows:

6.4.1 Introduce the upscale calibration gas, and record the gas concentration displayed by the analyzer on a form similar to Figure 6C-5. Then introduce zero gas, and record the gas concentration displayed by the analyzer. During the sampling system bias check, operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to achieve proper calibration gas flow rates at the analyzer. Alternately introduce the zero and upscale gases until a stable response is achieved. The tester shall determine the measurement system response time by observing the times required to achieve a stable response for both the zero and upscale gases. Note the longer of the two times as the response time.

6.4.2 The sampling system bias check shall be considered invalid if the difference between the gas concentrations displayed by the measurement system for the analyzer calibration error check and for the sampling system bias check exceeds ±5 percent of the span for either the zero or upscale calibration gas. If an invalid calibration is exhibited, take corrective action, and repeat the sampling system bias check until acceptable performance is achieved. If adjustment to the analyzer is required, first repeat the analyzer calibration error check, then repeat the sampling system bias check.

7. *Emission Test Procedure*

7.1 *Selection of Sampling Site and Sampling Points.* Select a measurement site and sampling points using the same criteria that are applicable to Method 6.

7.2 Interference Check Preparation. For each individual analyzer, conduct an interference check for at least three runs during the initial field test on a particular source category. Retain the results, and report them with each test performed on that source category.

If an interference check is being performed, assemble the modified Method 6 train (flow control valve, two midget impingers containing 3 percent H_2O_2, and dry gas meter) as shown in Figure 6C-2. Install the sampling train to obtain a sample at the measurement system sample by-pass discharge vent. Record the initial dry gas meter reading.

7.3 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as for Method 6 plus twice the system response time. For each run, use only those measurements obtained after twice response time of the measurement system has elapsed, to determine the average effluent concentration. If an interference check is being performed, open the flow control valve on the modified Method 6 train concurrent with the initiation of the sampling period, and adjust the flow to 1 liter per minute (±10 percent).

(*Note:* If a pump is not used in the modified Method 6 train, caution should be exercised in adjusting the flow rate since overpressurization of the impingers may cause leakage in the impinger train, resulting in positively biased results).

7.4 Zero and Calibration Drift Tests. Immediately preceding and following each run, or if adjustments are necessary for the measurement system during the run, repeat the sampling system bias check procedure described in *Section 6.4* (Make no adjustments to the measurement system until after the drift checks are completed.) Record and analyzer's responses on a form similar to Figure 6C-5.

7.4.1 If either the zero or upscale calibration value exceeds the sampling system bias specification, then the run is considered invalid. Repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before repeating the run.

7.4.2 If both the zero and upscale calibration values are within the sampling system bias specification, then use the average of the initial and final bias check values to calculate the gas concentration for the run. If *the zero or upscale calibration drift value* exceeds the drift limits, based on the difference between the sampling system bias check responses immediately before and after the run, repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before conducting additional runs.

7.5 Interference Check (if performed). After completing the run, record the final dry gas meter reading, meter temperature, and barometric pressure. Recover and analyze the contents of the midget impingers, and determine the SO_2 gas concentration using the procedures of Method 6. (It is not necessary to analyze EPA performance audit samples for Method 6.) Determine the average gas concentration exhibited by the analyzer for the run. If the gas concentrations provided by the analyzer and the modified Method 6 differ by more than 7 percent of the modified Method 6 result, the run is invalidated.

8. *Emission Calculation*

The average gas effluent concentration is determined from the average gas concentration displayed by the gas analyzer, and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with *Section 7.4. The average gas concentration* displayed by the analyzer may be determined by integration of the area under the curve for chart recorders, or by averaging all of the effluent measurements. Alternatively, the average may be calculated from measurements recorded at equally spaced intervals over the entire duration of the run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, *whichever is less restrictive, shall be used.* For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be used. Calculate the effluent gas concentration using Equation 6C-1.

$$C_{gas} = (\bar{C} - C_o)\frac{C_{ma}}{C_m - C_o} \qquad \text{Eq. 6C-1}$$

Where:

C_{gas} = Effluent gas concentration, dry basis, ppm.

$\bar{C}$ = Average gas concentration indicated by gas analyzer, dry basis, ppm.

C_o = Average of initial and final system calibration bias check responses for the zero gas, ppm.

C_m = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.

C_{ma} = Actual concentration of the upscale calibration gas, ppm.

9. *Bibliography*

1. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors. Protocol Number 1. U.S. Environmental Protection Agency, Quality Assurance Division. Research Triangle Park, NC. June 1978.

2. Westlin, Peter R. and J. W. Brown. Methods for Collecting and Analyzing Gas Cylinder Samples. Source Evaluation Society Newsletter. 3(3):5-15. September 1978.

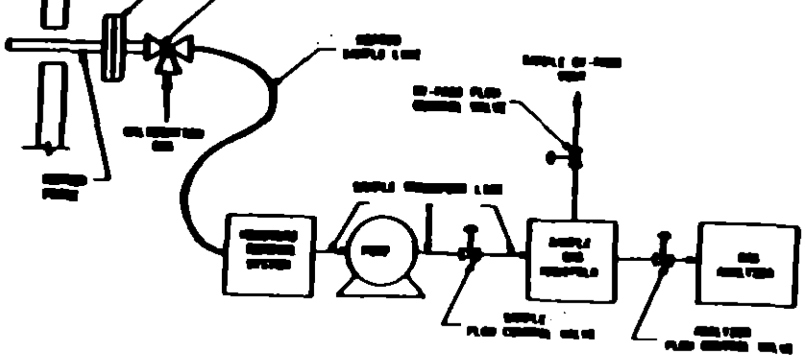

Figure 6C-1. Measurement system schematic.

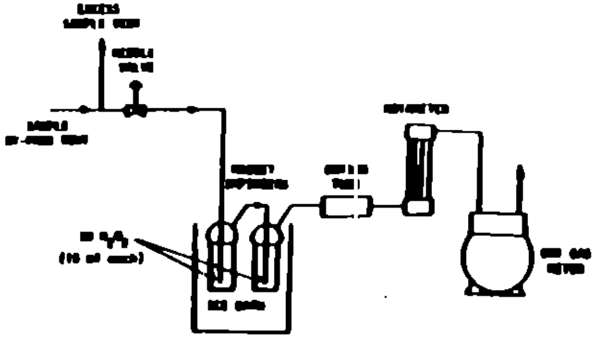

Figure 6C-2. Interference check sampling train.

FIGURE 6C-3—ANALYSIS OF CALIBRATION GASES

Date ______________________ Analytic method used ______________

	Gas concentration (indicate units)		
	Zero ª	Mid-range ᵇ	High-range ᶜ
Sample run			
1			
2			
3			
Average			
Maximum percent deviation			

ª Average must be less than 0.25 percent of span

ᵇ Average must be 50 to 60 percent of span

ᶜ Average must be 80 to 90 percent of span

FIGURE 6C-4—ANALYZER CALIBRATION DATA

Source identification: ______________

Test personnel: ______________

Date: ______________

Analyzer calibration data for sampling runs: ______________

Span: ______________

	Cylinder value (indicate units)	Analyzer calibration response (indicate units)	Absolute difference (indicate units)	Difference (percent of span)
Zero gas				
Mid-range gas				
High-range gas				

FIGURE 6C-5—SYSTEM CALIBRATION BIAS AND DRIFT DATA

Source identification: ______________ Run number ______________

Test personnel: ______________ Span: ______________

Date: ______________

	Analyzer calibration response	Initial values		Final values		Drift (percent of span)
		System calibration response	System cal bias (percent of span)	System calibration response	System cal bias (percent of span)	
Zero gas						
Upscale gas						

$$\text{System Calibration Bias} = \frac{\text{System Cal Response} - \text{Analyzer Cal Response}}{\text{Span}} \times 100$$

$$\text{Drift} = \frac{\text{Final System Cal. Response} - \text{Initial System Cal. Response}}{\text{Span}} \times 100$$

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO_2) per dry standard cubic meter, without having to dilute the sample.

2. *Apparatus*

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a

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values versus concentration in μg NO_3^-/ml. Do not force the curve through zero. Draw a smooth curve through the points. The curve should be linear. With the linear curve, use linear regression to determine the calibration equation.

6. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Same as in Method 7C, Section 6.1.

6.2 Total μg NO_2 Per Sample.

$$m = (S - B) \times 250 \times \frac{1000}{50} \times \frac{46.01}{62.01} = 3710\ (S - B) \qquad \text{Eq. 7D-1}$$

Where:

m = Mass of NO_x, as NO_2, in sample, μg.
S = Analysis of sample, μg NO_3^-/ml.
B = Analysis of blank, μg NO_3^-/ml.
250 = Volume of prepared sample, ml.
46.01 = Molecular weight of NO_2.
62.01 = Molecular weight of NO_3^-.
1000 = Total volume of $KMnO_4$ solution, ml.
50 = Aliquot $KMnO_4$/NaOH solution, ml.

6.3 Sample Concentration.

$$C = K_1 \frac{m}{V_{m(std)}}$$

Where:

C = Concentration of NO_x as NO_2, dry basis, mg/dscm.
K_1 = 10^{-3} mg/μg.
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

6.4 Conversion Factors.

1.0 ppm NO = 1.247 mg NO/m³ at STP.
1.0 ppm NO_2 = 1.912 mg NO_2/m³ at STP.
1 ft³ = 2.832×10^{-2} m³.
1000 mg = 1 g.

7. *Quality Control*

Quality control procedures are specified in Sections 4.1.2 (flow rate accuracy) and 4.5 (audit analysis accuracy) of Method 7C.

8. *Bibliography*

1. Margeson, J.H., W.J. Mitchell, J.C. Suggs, and M.R. Midgett. Integrated Sampling and Analysis Methods for Determining NO_x Emissions at Electric Utility Plants. U.S. Environmental Protection Agency, Research Triangle Park, NC. Journal of the Air Pollution Control Association. 32:1210-1215. 1982.

2. Memorandum and attachment from J.H. Margeson, Source Branch, Quality Assurance Division, Environmental Monitoring Systems Laboratory, to The Record, EPA. March 30, 1983. NH_3 Interference in Methods 7C and 7D.

3. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III—Stationary Source Specific Methods. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-600/4-77-027b. August 1977.

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METHOD 7E—DETERMINATION OF NITROGEN OXIDES EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1. *Applicability and Principle*

1.1 Applicability. This method is applicable to the determination of nitrogen oxides (NO_x) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental chemiluminescent analyzer for determination of NO_x concentration. Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

Same as Method 6C, Sections 2.1 and 2.2.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of NO_x concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface, Gas Analyzer, and Data Recorder. Same as Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.1.2 NO_2 to NO Converter. A device that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO).

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, and Response Time. Same as Method 6C, Sections 3.2 through 3.8.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. *Measurement System Performance Specifications*

Same as Method 6C, Sections 4.1 through 4.4.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for NO_x that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe, Sample Line, Calibration Valve Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.1 through 5.1.9, and 5.1.11.

5.1.2 NO_2 to NO Converter. That portion of the system that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO). An NO_2 to NO converter is not necessary if data are presented to demonstrate that the NO_2 portion of the exhaust gas is less than 5 percent of the total NO_x concentration.

5.1.3 NO_x Analyzer. An analyzer based on the principles of chemiluminescence, to determine continuously the NO_x concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

5.2 NO_x Calibration Gases. The calibration gases for the NO_x analyzer shall be NO in N_2. Three calibration gases, as specified in Sections 5.3.1 through 5.3.3, of Method 6C, shall be used. Ambient air may be used for the zero gas.

6. *Measurement System Performance Test Procedures*

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 7, and change all 5 percent performance values to 10 percent (or 10 ppm, whichever is greater).

6.2 Interference Response. Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response (e.g., changes in the gas detector). Conduct the interference response in accordance with Section 5.4 of Method 20.

6.3 Measurement System Preparation, Analyzer Calibration Error, and Sample System Bias Check. Follow Sections 6.2 through 6.4 of Method 6C.

6.4 NO_2 to NO Conversion Efficiency. Unless data are presented to demonstrate that the NO_2 concentration within the sample stream is not greater than 5 percent of the NO_x concentration, conduct an NO_2 to NO conversion efficiency test in accordance with Section 5.6 of Method 20.

7. *Emission Test Procedure*

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 7.

7.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the system calibration drift test. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as the total time required to perform a run using Method 7, plus twice the system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed, to determine the average effluent concentration.

7.3 Zero and Calibration Drift Test. Follow Section 7.4 of Method 6C.

8. *Emission Calculation*

Follow Section 8 of Method 6C.

9. *Bibliography*

Same as bibliography of Method 6C.

METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03×10^{-7} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74×10^{-7} lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³)

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high-level calibration gas at the calibration valve assembly. Adjust the analyzer output to the appropriate levels, if necessary. Calculate the predicted response for the low-level and mid-level gases based on a linear response line between the zero and high-level responses. Then introduce low-level and mid-level calibration gases successively to the measurement system. Record the analyzer responses for low-level and mid-level calibration gases and determine the differences between the measurement system responses and the predicted responses. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and must be replaced or repaired prior to testing. No adjustments to the measurement system shall be conducted after the calibration and before the drift check (Section 7.3). If adjustments are necessary before the completion of the test series, perform the drift checks prior to the required adjustments and repeat the calibration following the adjustments. If multiple electronic ranges are to be used, each additional range must be checked with a mid-level calibration gas to verify the multiplication factor.

6.5 Response Time Test. Introduce zero gas into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. Record the time from the concentration change to the measurement system response equivalent to 95 percent of the step change. Repeat the test three times and average the results.

7. *Emission Measurement Test Procedure*

7.1 Organic Measurement. Begin sampling at the start of the test period, recording time and any required process information as appropriate. In particular, note on the recording chart periods of process interruption or cyclic operation.

7.2 Drift Determination. Immediately following the completion of the test period and hourly during the test period, reintroduce the zero and mid-level calibration gases, one at a time, to the measurement system at the calibration valve assembly. (Make no adjustments to the measurement system until after both the zero and calibration drift checks are made.) Record the analyzer response. If the drift values exceed the specified limits, invalidate the test results preceding the check and repeat the test following corrections to the measurement system. Alternatively, recalibrate the test measurement system as in Section 6.4 and report the results using both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

8. *Organic Concentration Calculations*

Determine the average organic concentration in terms of ppmv as propane or other calibration gas. The average shall be determined by the integration of the output recording over the period specified in the applicable regulation.

If results are required in terms of ppmv as carbon, adjust measured concentrations using Equation 25A-1.

$$C_c = K\,C_{meas} \qquad \text{Eq. 25A-1}$$

Where:

C_c = Organic concentration as carbon, ppmv.

C_{meas} = Organic concentration as measured, ppmv.

K = Carbon equivalent correction factor,

K = 2 for ethane.

K = 3 for propane.

K = 4 for butane.

K = Appropriate response factor for other organic calibration gases.

9. *Bibliography*

9.1 Measurement of Volatile Organic Compounds—Guideline Series. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. EPA-450/2-78-041. June 1978. p. 46-54.

9.2 Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol No. 1). U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory. Research Triangle Park, NC. June 1978.

9.3 Gasoline Vapor Emission Laboratory Evaluation—Part 2. U.S. Environmental Protection Agency. Office of Air Quality Planning and Standards. Research Triangle Park, NC. EMB Report No. 75-GAS-6. August 1975.

METHOD 25B—DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A NONDISPERSIVE INFRARED ANALYZER

1. *Applicability and Principle*

1.1 Applicability. This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes. (Other organic materials may be measured using the general procedure in this method, the appropriate calibration gas, and an analyzer set to the appropriate absorption band.) The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

1.2 Principle. A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a nondispersive infrared analyzer (NDIR). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

2. *Definitions*

The terms and definitions are the same as for Method 25A.

3. *Apparatus*

The apparatus is the same as for Method 25A with the exception of the following:

3.1 Organic Concentration Analyzer. A nondispersive infrared analyzer designed to measure alkane organics and capable of meeting or exceeding the specifications in this method.

4. *Calibration Gases*

The calibration gases are the same as required for Method 25A, Section 4. No fuel gas is required for an NDIR.

5. *Measurement System Performance Specifications*

5.1 Zero Drift. Less than ±3 percent of the span value.

5.2 Calibration Drift. Less than ±3 percent of the span value.

5.3 Calibration Error. Less than ±5 percent of the calibration gas value.

6. *Pretest Preparations*

6.1 Selection of Sampling Site. Same as in Method 25A, Section 6.1.

6.2 Location of Sample Probe. Same as in Method 25A, Section 6.2.

6.3 Measurement System Preparation. Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing the sample interface and the organic analyzer. Make the system operable.

6.4 Calibration Error Test. Same as in Method 25A, Section 6.4.

6.5 Response Time Test Procedure. Same as in Method 25A, Section 6.5.

7. *Emission Measurement Test Procedure*

Proceed with t[illegible] emission measurement immediately up[illegible]tisfactory completion of the calibration.

7.1 Organic Measurement. Same as in Method 25A, Section 7.1.

7.2 Drift Determination. Same as in Method 25A, Section 7.2.

8. *Organic Concentration Calculations*

The calculations are the same as in Method 25A, Section 8.

9. *Bibliography*

The bibliography is the same as in Method 25A, Section 9.

METHOD 27—DETERMINATION OF VAPOR TIGHTNESS OF GASOLINE DELIVERY TANK USING PRESSURE-VACUUM TEST

1. *Applicability and Principle*

1.1 Applicability. This method is applicable for the determination of vapor tightness of a gasoline delivery tank which is equipped with vapor collection equipment.

1.2 Principle. Pressure and vacuum are applied alternately to the compartments of a gasoline delivery tank and the change in pressure or vacuum is recorded after a specified period of time.

2. *Definitions and Nomenclature*

2.1 Gasoline. Any petroleum distillate or petroleum distillate/alcohol blend having a Reid vapor pressure of 27.6 kilopascals or greater which is used as a fuel for internal combustion engines.

2.2 Delivery Tank. Any container, including associated pipes and fittings, that is attached to or forms a part of any truck, trailer, or railcar used for the transport of gasoline.

2.3 Compartment. A liquid-tight division of a delivery tank.

2.4 Delivery Tank Vapor Collection Equipment. Any piping, hoses, and devices on the delivery tank used to collect and route gasoline vapors either from the tank to a bulk terminal vapor control system or from a bulk plant or service station into the tank.

2.5 Time Period of the Pressure or Vacuum Test (t). The time period of the test, as specified in the appropriate regulation, during which the change in pressure or vacuum is monitored, in minutes.

2.6 Initial Pressure (P_i). The pressure applied to the delivery tank at the beginning of the static pressure test, as specified in the appropriate regulation, in mm H_2O.

2.7 Initial Vacuum (V_i). The vacuum applied to the delivery tank at the beginning of the static vacuum test, as specified in the appropriate regulation, in mm H_2O.

2.8 Allowable Pressure Change (Δp). The allowable amount of decrease in pressure during the static pressure test, within the time period t, as specified in the appropriate regulation, in mm H_2O.

2.9 Allowable Vacuum Change (Δv). The allowable amount of decrease in vacuum during the static vacuum test, within the time period t, as specified in the appropriate regulation, in mm H_2O.

3. *Apparatus*

3.1 Pressure Source. Pump or compressed gas cylinder of air or inert gas sufficient to pressurize the delivery tank to 500 mm H_2O above atmospheric pressure.

3.2 Regulator. Low pressure regulator for controlling pressurization of the delivery tank.

3.3 Vacuum Source. Vacuum pump capable of evacuating the delivery tank to 250 mm H_2O below atmospheric pressure.

3.4 Pressure-Vacuum Supply Hose.

3.5 Manometer. Liquid manometer, or equivalent instrument, capable of measuring up to 500 mm H_2O gauge pressure with ±2.5 mm H_2O precision.

3.6 Pressure-Vacuum Relief Valves. The test apparatus shall be equipped with an in-line pressure-vacuum relief valve set to acti-

METHOD 25A—DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A FLAME IONIZATION ANALYZER

1. *Applicability and Principle*

1.1 **Applicability.** This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

1.2 **Principle.** A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

2. *Definitions*

2.1 **Measurement System.** The total equipment required for the determination of the gas concentration. The system consists of the following major subsystems:

2.1.1 **Sample Interface.** That portion of the system that is used for one or more of the following: sample acquisition, sample transportation, sample conditioning, or protection of the analyzer from the effects of the stack effluent.

2.1.2 **Organic Analyzer.** That portion of the system that senses organic concentration and generates an output proportional to the gas concentration.

2.2 **Span Value.** The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations. The span value is established in the applicable regulation and is usually 1.5 to 2.5 times the applicable emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100 percent of the recorder scale.

2.3 **Calibration Gas.** A known concentration of a gas in an appropriate diluent gas.

2.4 **Zero Drift.** The difference in the measurement system response to a zero level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

2.5 **Calibration Drift.** The difference in the measurement system response to a mid-level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair or adjustment took place.

2.6 **Response Time.** The time interval from a step change in pollutant concentration at the inlet to the emission measurement system to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.

2.7 **Calibration Error.** The difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.

3. *Apparatus*

A schematic of an acceptable measurement system is shown in Figure 25A-1. The essential components of the measurement system are described below:

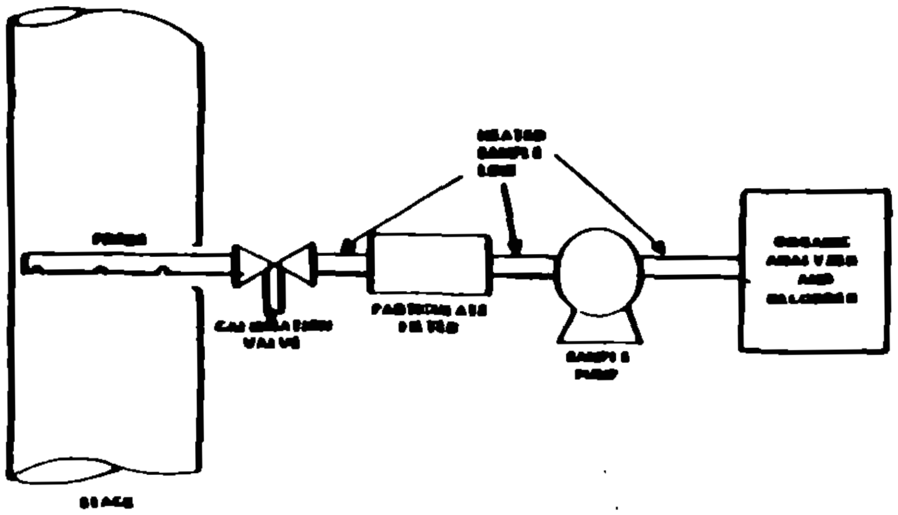

Figure 25A-1. Organic Concentration Measurement System.

3.1 **Organic Concentration Analyzer.** A flame ionization analyzer (FIA) capable of meeting or exceeding the specifications in this method.

3.2 **Sample Probe.** Stainless steel, or equivalent, three-hole rake type. Sample holes shall be 4 mm in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

3.3 **Sample Line.** Stainless steel or Teflon* tubing to transport the sample gas to the analyzer. The sample line should be heated, if necessary, to prevent condensation in the line.

3.4 **Calibration Valve Assembly.** A three-way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are applicable.

3.5 **Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter should be heated to prevent any condensation.

3.6 **Recorder.** A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute. Note: This method is often applied in highly explosive areas. Caution and care should be exercised in choice of equipment and installation.

4. *Calibration and Other Gases*

Gases used for calibrations, fuel, and combustion air (if required) are contained in compressed gas cylinders. Preparation of calibration gases shall be done according to the procedure in Protocol No. 1, listed in Reference 9.2. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval of the Administrator.

Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value. Organic compounds other than propane can be used following the above guidelines and making the appropriate corrections for response factor.

4.1 **Fuel.** A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

4.2 **Zero Gas.** High purity air with less than 0.1 parts per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

4.3 **Low-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value.

4.4 **Mid-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value.

4.5 **High-level Calibration Gas.** An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value.

5. *Measurement System Performance Specifications*

5.1 **Zero Drift.** Less than ±3 percent of the span value.

5.2 **Calibration Drift.** Less than ±3 percent of span value.

5.3 **Calibration Error.** Less than ±5 percent of the calibration gas value.

6. *Pretest Preparations*

6.1 **Selection of Sampling Site.** The location of the sampling site is generally specified by the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. The sample port shall be located at least 1.5 meters or 2 equivalent diameters upstream of the gas discharge to the atmosphere.

6.2 **Location of Sample Probe.** Install the sample probe so that the probe is centrally located in the stack, pipe, or duct and is sealed tightly at the stack port connection.

6.3 **Measurement System Preparation.** Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing the sample interface and the organic analyzer. Make the system operable.

FIA equipment can be calibrated for almost any range of total organics concentrations. For high concentrations of organics (>1.0 percent by volume as propane) modifications to most commonly available analyzers are necessary. One accepted method of equipment modification is to decrease the size of the sample to the analyzer through the use of a smaller diameter sample capillary. Direct and continuous measurement of organic concentration is a necessary consideration when determining any modification design.

6.4 **Calibration Error Test.** Immediately prior to the test series, (within 2 hours of the start of the test) introduce zero gas and

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* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.