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HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND

FINAL REPORT

DEVELOPMENT OF METHODOLOGY AND TECHNOLOGY FOR IDENTIFYING AND QUANTIFYING EMISSION PRODUCTS FROM OPEN BURNING AND OPEN DETONATION THERMAL TREATMENT METHODS.

FIELD TEST SERIES A, B, AND C

VOLUME 2, PART B

QUALITY ASSURANCE AND QUALITY CONTROL APPENDICES

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The report describes the quality assurance/quality control program conducted during 1990 field testing which supported the OB/OD Thermal Treatment Methods Study. The QA/QC program encompassed sample collection, preparation, storage, extraction, analytical instrument operation, data reduction, statistical data analyses and interpretation. Samples included atmosphere collected in evacuated stainless steel canisters, soil, and Teflon™-coated-glass-fiber media. The supercritical fluid chromatograph and gas chromatograph with mass spectrometers were the principal laboratory analytical instruments used during these field tests. Real-time instruments detected and quantified CO, CO_2, NO, NO_2, and NO_x. Near-real-time analyses were accomplished by use of a Teflon® bag in which samples of the plume were collected for analyses during testing operations. Sample-tracking was conducted using a system which permitted precise identification of individual specimens from collection through analyses or archiving. Analysis of atmosphere and soil samples spiked by the U.S. Environmental Protection Agency (EPA) reflected a degree of accuracy well within acceptable limits. (Continued on Reverse)

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supercritical fluid chromatography; SFC; demilitarization; munition dispersal; explosive dispersal; environment; BB; air building; U.S. Environmental Protection Agency; EPA; quality assurance; QA; quality control; QC; RDX; composition B; explosive D; M1 propellant; M6 propellant; single-base propellant; propellant manufacturing residue; air emissions; soil contamination.

19. (Cont'd)

Included in this report are the quality assurance program plan, test design plan, QA auditor reports, and reports of EPA-conducted audits. Almost all findings were highly satisfactory; most of those that suggested corrective action were minor and immediately resolved on-site.

FOREWORD

A test planning directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988[1]. A Technical Steering Committee Symposium was convened in July 1988. The requirement for identifying and quantifying emissions from the open detonation of explosives and open burning of propellants was discussed in detail by authorities from throughout the military, academic, and commercial communities. Conclusions and recommendations developed during the symposium are reported in proceedings of the symposium[2]. A series of TNT detonations and propellant burns were characterized in a BangBox (chamber) in December 1988 and January 1989 for the purpose of developing methodology and technology for large scale detonations and burns in the field. The field tests took place in 1989 and 1990 and are reported in two volumes.

Volume 1. A summary which describes the planning phase, the conduct of trials, sample analyses and results, and the conclusions and recommendations. It is useful for those who need only a quick review (executive summary) and those who need a detailed description of the conduct and results of the Field Tests Phases A, B, and C.

Volume 2, Part A. A stand-alone document which covers the quality assurance and quality control procedures, the blind spiking of samples, the on site challenges of equipment and personnel, the conclusions, and the recommendations.

Volume 2, Part B. The quality assurance (QA) program plan which was developed specifically to support phase "C" field testing. While directed to phase "C" testing, it also represents the procedures and techniques and QA philosophies which were used during OB/OD field testing phases "A" and "B" and is based on experience gained during these two earlier field tests.

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[1] Letter, AMSTE-TA-F, Headquarters, U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, 20 April 1988, subject: Test Planning Directive for Special Study of Open Burning/Open Detonation (OB/OD), Phase II, TECOM Project No 2-CO-210-000-017.

[2] *Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

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APPENDIX A. TEST DESIGN PLAN

Letters of instruction have been removed from the test design plan to avoid duplication of material contained in Appendix B, Letters of Instruction, of this volume.

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TEST DESIGN PLAN

FOR THE

IDENTIFICATION AND QUANTIFICATION OF

PRODUCTS AND RESIDUES

FROM THE OPEN BURNING/OPEN DETONATION

OF

TNT, COMPOSITION "B", EXPLOSIVE "D", RDX, AND PROPELLANTS

(FIFLD TEST PHASE "C")

Prepared By

U.S. ARMY ARMAMENT, MUNITIONS
AND CHEMICAL COMMAND

Demilitarization and Technology Branch
ROCK ISLAND, IL 61229-6000

APRIL 1990

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SECTION 1 INTRODUCTION

1.1 Objective.

The objective of this study is to identify and quantify the products and residues emitted to the air and to the soil from bulk 2,4,6-trinitrotoluene (TNT), Composition "B", Explosive "D", and RDX explosives, propellant manufacturing residue (PMR), and ammonium perchlorate (AP) during Open Burning/Open Detonation (OB/OD) operations. The data obtained from this study is needed to support environmental assessments and other documentation required under the Clean Air Act (CAA), Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), and other federal and state environmental statutes and regulations.

1.2 Test Concept

1.2.1 Subtests To Be Performed

1.2.1.1 Subtests. Subtests to be performed within the OB/OD Fixed Wing Aircraft (FWAC) Phase C test program are listed in Table 1 and include:

1.2.1.1.1 Open burning of PMR and AP.

1.2.1.1.2 Open detonation of bulk TNT, Composition "B", Explosive "D", and RDX explosives by conventional protocol.

1.2.1.1.3 Suspended detonation of bulk TNT.

1.2.1.1.4 Buried detonation of bulk TNT by conventional protocol.

1.2.1.2 Schedule.

Field testing will begin 30 July 1990 and will continue until this phase of the OB/OD test is completed.

1.2.2 Test and Evaluation Personnel Participation.

Testing, analysis, and reporting will be conducted by Dugway Proving Ground (DPG) personnel (including DPG contractors), and other contracted personnel.

1.2.3 Operations Security.

This test will not involve classified material, techniques, or information. Operations Security (OPSEC) sensitivity will be evaluated in accordance with (IAW) the provisions of Army Regulation (AR) 530-1 (Appendix D Reference 1).

Table 1 Field Test Phase C Tentative Test Matrix

Number	Type of Trial	Configuration	Material	Amount (lb)
1	OB[a]	Surface	Manufacturing Residue	7,300 x 2
2	OB	Surface	Ammonium Perchlorate	7,300 x 2
3	OD[b]	Surface	TNT	2,000 x 3
4	OD	Surface	TNT	2,000 x 3
5	OD	Surface	RDX	2,000 x 3
6	OD	Surface	RDX	2,000 x 3
7	OD	Surface	Explosive "D"	2,000 x 3
8	OD	Surface	Explosive "D"	2,000 x 3
9	OD	Surface	Composition "B"	2,000 x 3
10	OD	Surface	Composition "B"	2,000 x 3
11	OD	Suspended	TNT	2,000 x 3
12	OD	Suspended	TNT	2,000 x 3
13	OD	Buried	TNT	2,000 x 3
14	OD	Buried	TNT	2,000 x 3
15*	OD	Surface	RDX	2,000 x 3
16*	OD	Surface	Explosive "D"	2,000 x 3
17*	OD	Surface	Composition "B"	2,000 x 3
18*	OD	Surface	TNT	2,000 x 3

* For soil accumulation studies only; no aircraft sampling

[a] Open Burn

[b] Open Detonation

1.2.4 National Environmental Policy Act Process.

All testing is to be conducted within DPG boundaries and within the provisions of the DPG Installation Environmental Assessment. The Environmental Assessment for Open Burning/Open Detonation testing at DPG (February 1989) (Appendix D, Reference 2), citing the appropriate categorical exclusion, has been filed. Special test permits, if necessary will be obtained prior to initiation of testing. The proposed test program will be evaluated against the requirements of AR 200-2 (Appendix D, Reference 3).

1.2.5 Safety and Health.

Special consideration must be given to the quantities of explosives and propellants which will be burned or detonated during this test to ensure that personnel and physical assets are not endangered. Procedures delineated in applicable Depot Maintenance Work Requirements (DMWRs) should be followed when testing with conventional techniques. Army Materiel Command Regulation (AMC-R) 385-100 (Appendix D, Reference 4) applies when testing with techniques not covered by existing DMWRs or Standard Operating Procedures (SOPs). Special consideration must also be given to the FWAC sampling platform that is expected to encounter turbulence, low visibility, and airborne particulate and emissions.

1.2.6 Test Residue.

Should any test residue be classified as hazardous waste, it will be handled and disposed of IAW AR 420-47 (Appendix D, Reference 5), DPG Regulation 420-10 (Appendix D, Reference 6), and other existing regulations.

1.2.7 Statistical Design

1.2.7.1 The OB/OD Phase C program is designed to provide a minimum of two surface detonation trials of each explosive material TNT, RDX, Explosive "D", and Composition "B". Some additional configurations, suspended and buried, are planned for TNT with a minimum of two trials. The design and planned sampling will provide data to identify and quantify the amount of analytes produced. The number of data points from the airborne cloud will vary depending upon the number of passes through the cloud that can be accomplished on each detonation and the composition of extract to maximize detection of analytes. Soil sampling and fallout data will be collected on each detonation.

1.2.7.2 Only one trial set of each burn material, PMR and AP, are planned. The sampling planned will provide data to identify and quantify the amount of analyte produced.

1.3 Material Description

1.3.1 Burn Material.

The propellants will be obtained from various Naval sources and are indicative of current and future demilitarization assets.

1.3.2 Detonation Material.

These will be drawn from existing accountable stocks and will be representative of the overall demilitarization inventory.

1.3.3 Containers and Packaging Materials.

All packing material will be removed from the propellants and detonation materials. These containers and any other packaging materials will be evacuated from test areas prior to burning or detonation, or will be otherwise secured to ensure that none is consumed by the open burning or open detonation operations.

1.4 Sampling and Analysis

1.4.1 Target Analytes.

Gaseous and inorganic compounds and particulates targeted for analysis are shown in Table 2. Metals and elements targeted for analysis are shown in Table 3. Table 4 lists the volatile organics and Table 5 lists the semi-volatile organics targeted for analysis.

Table 2 Gaseous/Inorganic Compounds and Particulates Targeted for Analysis, OB/OD Field Test Phase C.

Analyte	Sampling/Analysis Technique
CO	Real-time instrument and 6 l canister
CO_2	Real-time instrument and 6 l canister
NO_x	Real-time analyzer
NO	Real-time analyzer
O_3	Real-time analyzer
Total Non-methane Hydrocarbon	GC-FID
Total Organic Hydrocarbon	Thermal optical
Total Inorganic Carbon	Thermal optical
Total Elemental Carbon	Thermal optical
Particulate, 0.15-0.30μm	ASASP-100X
Particulate, 2.0-27μm	FSSP-100X

Table 3 Metals/Elements Targeted for Analysis By PIXE, OB/OD Field Test Phase C.

- Antimony
- Arsenic
- Barium
- Cadmium
- Chromium
- Copper
- Lead
- Nickel

Table 4 Volatile Organic Combustion Products Targeted for Analysis. OB/OD Field Tests Phase C.

Methane
Cyclohexane
Acetylene
3-Methylhexane
i-Butane
Methylcyclohexane
n-Butane
Toluene
cis-2-Butene
3-Ethylhexane
1-Pentane
Ethylbenzene
Isoprene
Styrene
2-Methyl-2-butene
alpha-Pinene
4-Methyl-1-pentene
4-Ethyltoluene (para)
2-Methylpentane
2-Ethyltoluene (ortho)
1-Hexene
1,2,4-Trimethylbenzene & sec-Butylbenzene
d-Limonene
2,4-Dimethylpentane
Benzene
Ethylene
2,3-Dimethylpentane
Propene
n-Heptane
1,3-Butadiene
2,3,4-Trimethylpentane
2,2-Dimethylpropane
2-Methylheptane
i-Pentane
Ethylcyclohexane
n-Pentane
m-Xylene
cis-2-Pentene
n-Nonane
Cyclopentene
n-Propylbenzene
2,3-Dimethylbutane
1,3,5-Trimethylbenzene
2-Methyl-1-pentene
Mycrene
trans-2-Hexene
2-Methyl-2-pentene
Methylcyclopentane
n-Dodecane
Ethane
2-Methylhexane
Propane
2,2,3-Trimethylpentane
1-Butene
2,4-Dimethylhexane
trans-2-Butene
2,3-Dimethylhexane
3-Methyl-1-butene
n-Octane
2-Methyl-1-butene
p-Xylene
trans-2-Pentene
o-Xylene
2,2-Dimethylbutane
i-Propylbenzene
Cyclopentane
3-Ethyltoluene (meta)
3-Methylpentane
beta-Pinene
n-Hexane
n-Decane
cis-2-Hexene
n-Undecane

Table 5 Semivolatile Organic Combustion Products Targeted for Analysis, OB/OD Field Test PHASE C.

Semi-volatile Organics	Open Burning	Open Detonation
2,4-Dinitrotoluene	X	X
2,6-Dinitrotoluene	X	X
2,4,6-Trinitrotoluene	X	X
2-Nitronaphthalene	X	X
n-Nitrosodiphenylamine	X	X
2-Nitrodiphenylamine	X	
4-Nitrodiphenylamine	X	
Nitroglycerin	X	
1,3,5-Trinitrobenzene		X
1-Nitropyrene		X
Naphthalene	X	X
Benz[a]anthracene	X	X
Benzo[a]pyrene	X	X
Pyrene	X	
Phenol	X	X
Dibenzofuran	X	X
Diphenylamine	X	X
Diethyl Phthalate	X	

1.4.2 Sample Analysis. Samples will be analyzed as outlined in paragraphs 2.1.3.6, 2.1.4, and Appendix B of this volume.

SECTION 2 SUBTESTS

2.1 Open Burning

2.1.1 Objective.

Identify and quantify products and residues released into the atmosphere and soil during the open burning (OB) of propellants and propellant manufacturing residues (PMR).

2.1.2 Target Analytes

2.1.2.1 List of Analytes.

OB products that are targeted for detection and quantification are listed in Tables 2-5.

2.1.3 Test Procedures

2.1.3.1 Test Material

No explosives, other than small amounts of initiators, will be included in this subtest. The PMR will consist of a combination of NOISH-AA-2 and NOISH-AA-6 double-base propellants. Other propellants will be designated at a later date.

2.1.3.2 Propellant Configuration

2.1.3.2.1 Net propellant weight per burn, including test material and initiators, will be approximately 3300 kg (7300 lb), unless otherwise approved by the Project Officer (PO).

2.1.3.2.2 All propellants will be spread in steel pans 122 cm (4 ft) wide by 305 cm (10 ft) long, laid on the ground surface at the test site. Propellant depth will usually not exceed 7.5 cm (3 in), IAW DPG SOP DP-0000-G-002. In testing PMR, the reconfiguration of some of the material is such that the height may be more than 7.5 cm.

2.1.3.2.3 Each burn will be prepared IAW the applicable DMWR and/or SOP selected by the PO. Any variation from the selected SOP must be approved by the PO and be fully documented, including pictorial documentation.

2.1.3.2.4 The propellants will be distributed along the length of the pan. The depth of a propellant is not as important as the length.

2.1.3.2.5 Burn initiation will be made by two systems. The primary train will utilize approximately 6.8 kg (15 lb) of Type ABL casting propellant. A backup train will be made up from smokeless powder as in Phase "A" and Phase "B". Initiation will be made from both ends of the propellant train at the same time.

2.1.3.2.6 Test personnel will remove containers and any other packing materials from the test propellants. These materials will be evacuated from the test area prior to the OB, or otherwise secured to ensure they are not consumed by the OB of test material. Quality Assurance Specialist Ammunition Surveillance (QASAS) personnel will inspect all test material packaging to determine if any explosive contamination is present. Any explosive contaminated packaging will be turned over to the Hazardous Waste Coordinator at the Materiel Test Directorate (MTD) for proper disposition. Packing materials found free of explosive components will be disposed of as ordinary residue.

2.1.3.3 Test Matrix. There will be two burns with each propellant configured as outlined in Paragraph 2.1.3.2.1 and 2.1.3.2.2. (See Table 1.)

2.1.3.4 Open Burning Procedures.

OB procedures will parallel those used at depots involved in disposal of propellants. When variances are found between depot and test site procedures, the Program Manager (PM) will, upon request or recommendation of the PO, select the procedure(s) to be used. Experimental burning procedures may be tested only if approved by the PM. Once approved, they will be defined by an addendum to the Detailed Test Plan (DTP).

2.1.3.5 Meteorological Restrictions

2.1.3.5.1 Temperature: No limitation.

2.1.3.5.2 Relative humidity: No limitation.

2.1.3.5.3 Precipitation: None permitted. No thunderstorms within 10 km of test site.

2.1.3.5.4 Wind speed at 2 m: 0.0 to 8.0 m/s.

2.1.3.5.5 Wind direction: Dependent on grid design; testing will not be conducted if the wind is blowing toward Granite Mountain.

2.1.3.5.6 Wind shear: ± 45° of wind direction at 32 m through 2000 m height.

2.1.3.5.7 Visibility: Greater than 10 km.

2.1.3.5.8 Ambient light: Adequate for photographic coverage.

2.1.3.5.9 Atmospheric stability: Pasquill stability categories C or D. This may be waived by the PO on the advice of the Meteorologist-in-Charge.

2.1.3.5.10 Clearing index: At least 500 unless a variance is obtained from the State.

2.1.3.6 Air Sampling

A combination of sampling techniques will be used to collect samples for determination of the types and concentrations of volatile, semi-volatile, and particulate emissions generated from open burning. Analysis procedures will include subsequent laboratory assay as well as real-time and near-real-time analysis. Sampling devices will be sufficiently rugged to withstand the rigors of sampling and sampling platform environments and, to the maximum extent possible, be EPA-certified. Aircraft sampling will be performed as outlined in Appendix B of this volume, Aircraft Sampling Procedures.

2.1.3.6.1 Particulate Detectors and Samplers

a. Quartz fiber filter

Three separate quartz (Pallflex Type QAOT) filters, sampled from a single manifold.

b. Aerosol Probe

A probe to cover a range of 0.15- to 3.0-μm particulate diameters, PMS Active Scattering Aerosol Spectrometer Probe (ASASP-100X).

c. Aerosol Probe

A probe to cover a range of 2.0- to 47-μm diameter, PMS Forward Scattering Spectrometer Probe (FSSP-100X).

d. Integrating Nephelometer, MIE Instruments (Model RAM-1)

2.1.3.6.2 Gas Analyzers

a. Gas Filter Correlation CO_2 Analyzer, TECO Model 41H.

b. Gas Filter Correlation CO Analyzer, TECO Model 48.

c. UV Photometric O_3 Analyzer, TECO Model 49.

d. Chemiluminescent Nitrogen Oxides (NO_x) Analyzer, TECO, Model 92.

2.1.3.6.3 Volatile Organic Samplers

Electro-polished, stainless-steel, 6-L, evacuated canisters.

2.1.3.6.4 Metal Samplers

Pallflex quartz fiber filter, 203 mm x 254 mm. This is the same filter that is described in Paragraph 2.1.3.6.1.

2.1.3.6.5 Total Organic, Inorganic, and Elemental Carbon Samplers

A portion of each Quartz fiber filter will be analyzed for total organic (OC), inorganic (IC), and elemental (EC) carbon by thermal optical methods.

2.1.3.6.6 Ancillary Equipment

a. Pitot tube flow meter (1) for particulate filter.

b. Pumps (3) for aerosol filters.

c. 80-L Teflon^{TM} bag for air grab sampling.

2.1.3.7 Residue and Fallout Sampling

Residue and fallout samples will be collected and analyzed for each burn. The details of sample pan positioning, collection, and handling prior to delivery to the assay laboratory are outlined in Appendix C, Soil and Fallout Sampling. All samples will be individually collected and weighed.

2.1.3.7.1 Pan Residue

The residue from each pan will be weighed and stored in acid-washed bottles for assay and archiving.

2.1.3.7.2 "Sputter" Residue

"Sputter" residue samples are those located 1 m from the burn pans. These pan samples represent propelled grains of burned/burning propellant/residue that fall within 2 m of the burn pan. The residue from each pan will be weighed and stored in acid-washed bottles for assay and archiving.

2.1.3.7.3 Fallout Pans

Fallout pans are those located on the 6 and 12 m circles around the burn pans. The residue in each pan will be weighed and stored in acid-washed bottles for assay and archiving.

2.1.3.8 Data Requirements

The following information will be recorded on standardized data collection sheets to facilitate accurate recording, analysis, and reporting.

2.1.3.8.1 Burning Procedure

All procedures used will be thoroughly documented, to include a description and any modifications or adjustments made to those procedures outlined in the approved DTP. These changes will be documented as numbered changes to the DTP.

2.1.3.8.2 Burn Material

The material that is placed into each burn pan will be described by chemical content, lot number(s), source weight of material, and arrangement in the pan.

2.1.3.8.3 Meteorological Data

The following meteorological data will be measured and recorded by the Atmospheric Science Laboratory meteorological team at the test site.

a. Standard meteorological surface observations, taken every 15 min at 2 m.

(1) Wind speed, m/s.

(2) Wind direction.

(3) Temperature.

(4) Humidity.

(5) Barometric pressure.

(6) Cloud cover.

2.1.3.8.4 Tethersondes

Two tethersondes will be used to measure wind speed, wind direction, and temperature at 30 min intervals at 2000 m above ground level or as high as possible with prevailing winds. One tethersonde will be positioned at the grid and one at the CP. Tethersonde measurement at the grid will terminate at t-30 min for safety reasons, but measurement at the CP will continue at 30 min intervals to t+1 hr to the extent possible without interfering with aircraft operations.

2.1.3.8.5 Elevation of grid above mean sea level (±0.5 m).

2.1.3.8.6 Real-time flight data and chemical data with time identification.

2.1.3.8.7 Information to support assessment of the QA/QC program.

2.1.3.8.8 Altitude, speed, air temperature, and time in cloud of the FWAC.

2.1.3.8.9 Photography.

A combination of color video and color still photography will be used to document the burn.

a. Color video

Two color video cameras will be used during the OB trials of this subtest. These cameras will be positioned as determined by the PO to record the cloud produced by the OB trials, but will not be closer than 2275 m to the burn site.

(1) All camera positions will be surveyed and will be connected to the IRIG-timing device.

(2) Video cameras will be used to determine the cloud height versus time.

(3) Total documentation of the lenses used and distance to the burn will be recorded on

each video.

b. Color still photography.

(1) All OB subtests will be fully documented by color still photography.

(2) OB test material preparations, test equipment, and the burn events will be represented.

(3) Photographs of the aircraft sampling the plume will show cloud entry, exit, and in-cloud flight stages of the sampling pass.

(4) If any incidents occur, they will be photographically documented.

2.1.3.8.10 FWAC Sampling Data. See Appendix B of this volume.

2.1.3.8.11 Soil and Fallout Sampling Data. See Appendix B of this volume.

2.1.3.8.12 Chemical Assay. All chemical analysis results (field samples, standards, travel blanks, and internal controls) will be reported in units and formatted as outlined in the approved Quality Assurance (QA) Project Plan (QAPP).(Appendix C of this volume)

2.1.4 Analytical Procedures

2.1.4.1 Chemical analysis will be conducted by Alpine West Laboratory (AWL), Oregon

Graduate Center (OGC), Sunset Laboratories (SSL), Sandia National Laboratories (SNL), Brigham Young University (BYU) or by other laboratories as required. Procedures used will correspond with those delineated in Appendix B this volume. The laboratories should make every effort to process samples expeditiously. Samples that cannot be analyzed within 8 hrs of collection must be stored at -20°C until instruction as to their disposition is given by the PO or his representative.

2.1.4.1.1 All laboratories will be audited by QA/QC personnel.

2.1.4.1.2 Where applicable, the reference standard must be traceable to a National Institute of Standards and Technology (NIST) standard.

2.1.4.2 Inorganic Analysis (Metals)

As outlined in Table 3, proton-induced X-ray emission (PIXE) is the method to be used for metals analysis. The following metals will be scanned for: antimony, arsenic, barium, cadmium, chromium, copper, lead, and nickel. Procedures used will correspond with those delineated in Appendix B.

2.1.4.3 Semi-volatile and Volatile Organic Analysis

Post-trial organic compound analyses will be conducted by application of a variety of techniques, to include:

2.1.4.3.1 Gas Chromatography (GC) for volatile organics. (See Appendix B of this volume).

2.1.4.3.2 Gas Chromatography-Mass Spectrometry (GC-MS) for semi-volatile organics. (See Appendix B of this volume.)

2.1.4.3.3 Supercritical Fluid Chromatography-Mass Spectrometry (SFC-MS) semi-volatile organics. (See Appendix B of this volume.)

2.1.4.4 Total Organic, Inorganic, and Elemental Carbon Analysis

Thermal optical methods will be utilized to analyze portions of the quartz fiber filter for OC, IC, and EC.

2.1.4.5 Statistical Analysis.

2.1.4.5.1 All data will be tabulated and reported. Where sample replication and duplication permit, measure of central tendency will be reported; in other situations, the range may be used. Background samples of air and soil will be examined and used in corrections for all test data.

2.1.4.5.2 Chemical species of airborne gases and particulate will be reported as concentration per volume of air sampled and/or concentration per weight of particulate collected. The concentration data will be used in subsequent analyses along with the carbon concentration to determine emission factors. The emission factors will be used to determine the mass of species produced in the burn.

2.1.4.5.3 Particulate sample data will be used to determine the PM 2.5 (particle mass equal to or less than 2.5μm) and the PM 10.

2.1.4.5.4 The concentration of chemical species in the soil ejecta and fallout will be used in determining the expected amount of each species that are deposited in the soil around a burn site. The multiple detonations at a single site will provided data on the accumulation of chemical species that may occur.

2.2 Open Detonation (Surface/Buried)

2.2.1 Objective.

Identify and quantify CO_2, CO, NO, NO_x, O_3, and organic products, metals, and particulates released into the atmosphere and soil during the open detonation (OD) of TNT, Composition "B", Explosive "D", and RDX.

2.2.2 Target Analytes. Tables 2-5 apply.

2.2.3 Test Procedures

2.2.3.1 Test Material

Each detonation will consist of 907 kg (2000 lb) of bulk TNT, Composition "B", Explosive "D", or RDX. A small sample of each type of material will be retained for laboratory analysis.

2.2.3.2 Open Detonation Sites

Detonation sites will be selected by the DPG staff that have not been previously used for OD and have not been subjected to fallout from previous ODs, if possible. Each detonation point will be separated from others by at least 600 m to avoid fallout from airborne particulate.

2.2.3.3 Test Matrix

2.2.3.3.1 There will be nine detonations each of Composition "B", Explosive "D", and RDX: six multiple surface detonations at separate sites and three single surface detonations at the same site. Plume sampling by FWAC will be accomplished on all detonations except the three single surface, single site detonations. In this case, soil and fallout (pan) sampling will be accomplished. (See Appendix B.)

2.2.3.3.2 There will be twenty-one detonations of TNT: six multiple surface detonations, six multiple suspended detonations, and six multiple buried detonations, all at different sites; plus three single surface detonations at the same site. FWAC plume sampling will be carried out after all detonations except the three single surface detonations at the same site. In this case, soil and fallout sampling will be done on all detonations. (See Appendix B.)

2.2.3.3.3 Combined (composite) quartz fiber filter samples will collected on each group of three (multiple) detonations, thus enhancing the potential to be able to detect trace levels of semi-volatile organic combustion products.

2.2.3.4 Open Detonation Procedures

OD procedures will parallel those used at those depots and explosives/munitions manufacturing plants designated for disposal of explosives and munitions. When variance is found between test site and disposal site procedures, the PM will, upon request or recommendation of the PO, select the procedure(s) to be used. Buried detonations shall be at a depth of 4 feet, with a minimum cover of 2 feet of soil. (See Appendix D Reference 4)

2.2.3.5 Meteorological Restrictions. Paragraph 2.1.3.5 applies.

2.2.3.6 Air Sampling. Paragraph 2.1.3.6 applies.

2.2.3.7 Soil Sampling and Fallout Sampling. Soil sampling will consist of collection of a variety of pre-trial core samples and post-trial ejecta samples. The fallout sampling will be from pans placed on concentric circles. (See Appendix C for more detailed discussions.)

2.2.3.8 Data Requirements. The following information will be recorded on standardized data collection sheets to facilitate accurate recording, analysis, and reporting.

2.2.3.8.1 All OD procedures will be thoroughly documented, to include a description and any modifications or adjustments made to those procedures outlined in the approved DTP. These changes will be documented as numbered changes in the DTP.

2.2.3.8.2 Detonation Material. The explosive material used for each detonation will be described as to lot number, source, weight of material, placement on the ground (footprint, height, etc.), and the donor charge (material, position, and amount) used.

2.2.3.8.3 Other Data Requirements. See Paragraphs 2.1.3.8.3 through 2.1.3.8.12.

2.2.4 Analytical Procedures. Paragraph 2.1.4 applies.

2.3 Single Surface Detonations at Same Site.

2.3.1 Objective.

To identify and quantify organic products released into the soil as a result of multiple detonations at the same site, for each explosive type.

2.3.2 Target Analytes. Tables 2-5 apply.

2.3.3 Test Procedures.

2.3.3.1 Test Material. Paragraph 1.3.1 applies.

2.3.3.2 Open Detonations at the Same Site. The detonation sites will be as selected by DPG staff.

2.3.3.3 Test Matrix. Table 1 applies.

2.3.3.4 Test Procedures

The first detonation will be followed by ejecta soil sampling. The crater will then be filled with soil ejecta. A second detonation at the same site will be followed by ejecta soil sampling. The crater will then again be filled in with the soil ejecta. A third detonation with post-trial ejecta soil sampling will then be accomplished. The time between detonations will be as short as possible (limited to the time required to sample, fill the crater, and set up the new explosive charge). This routine will be followed for the other single surface detonation series for the other explosive types.

2.3.3.5 Meteorological Restrictions. Paragraph 2.1.3.5 applies.

2.3.3.6 Soil Sampling. Soil sampling will consist of pre-trial core samples and the post-trial ejecta samples. The pre-trial core samples will only be taken prior to the first detonation. There will be a post-trial core sample after each detonation. The ejecta soil sampling grid is as outlined in Appendix C.

2.3.3.7 Data Requirements. Paragraph 2.1.3.8 applies.

2.3.3.8 Analytical Procedures. Paragraph 2.1.4 applies, except for subparagraphs 2.1.4.2.

SECTION 3 - Appendices

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APPENDIX A - Test Criteria

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TEST CRITERIA

This test is being conducted to build combustion product databases for each of four (4) specific explosives and two propellants: TNT, Composition "B", Explosive "D", RDX and propellant manufacturing residue (PMR) (NOISH-AA-2 and NOISH-AA-6) and ammonium perchlorate (AP).

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APPENDIX B - Carbon Balance Technique

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CARBON BALANCE TECHNIQUE[1]

The carbon balance technique is based on two ideas. The first is that carbon can be used as a conservative chemical tracer for the products from a high explosive (HE) detonation, or for that matter, from an open burn. This is because neither a chemical detonation, nor a burn, nor the dispersal of the products of either, changes the total amount of carbon involved in the event. These processes only change the chemical form of the carbon and redistribute it in space. The second idea is that the cloud of combustion or detonation products is, to a reasonable approximation, homogeneous in relative composition. That is, although the absolute concentrations of gaseous and particulate products may vary by orders of magnitude across a cloud, their relative concentrations (the concentration ratios) are approximately the same throughout, independent of position within the cloud.

Based on these assumptions, one finds that the ratio of the concentration of any combustion or detonation product D_i in some sampling volume j to the concentration of all forms of carbon originating in the event in the same sampling volume is equal to the ratio of the average concentration of detonation product D_i in the whole cloud to the average concentration of all forms of carbon from the event in the whole cloud. This is expressed mathematically:

$$\frac{[D_{ij}]}{[C_j]} = \frac{\overline{[D_i]}}{\overline{[C]}} \qquad (1)$$

Here the $[C_j]$ indicates the concentration of carbon in all forms in the jth sampling volume, and the $\overline{[C]}$ indicates the concentration of carbon in all forms associated with the event averaged over the whole cloud. Next, we make use of the definition of average concentration over the cloud for both product D_i as well as for total carbon:

$$\frac{[D_{ij}]}{[C_j]} = \frac{D_{iT}/V_{ci}}{C_T/V_{ci}} = D_{iT}/C_T \qquad (2)$$

Here C_T is the total mass of all forms of carbon contained in the cloud originating in the event. Note that if combustion is complete, then C_T is equal to the total amount of carbon in the original HE or propellant. So, on the basis of measurements of the relative concentration of any detonation product D_i to the concentration of all forms of carbon from the event in some sampling volume of the cloud, one can calculate the total cloud content D_{iT} provided one knows

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[1] Extracted from "Measuring the Composition and Total Content of Explosively Generated Smoke Clouds", ZAK, B.D., Sandia National Laboratories, Albuquerque, New Mexico, July, 1988.

how much carbon was contained in the original mass detonated or burned:

$$D_{iT} = C_T \times \frac{[D_{ij}]}{[C_j]} \tag{3}$$

An emission factor EF for the ith detonation or combustion product is defined as:

$$EF_i = \frac{D_{iT}}{M} \tag{4}$$

Here, as before, D_{iT} is the total mass of the ith product emitted by the event into the cloud, and M is the total mass of the HE or propellant detonated or burned. To obtain the emission factor from the information provided by the carbon balance technique, one need only note that the total carbon mass in the HE or propellant is given by:

$$C_T = F_c \times M \tag{5}$$

Here F_c is the carbon fraction for the particular HE or propellant involved in the experiment. Substituting appropriately, one finds:

$$EF_i = \frac{[D_{ij}]}{[C_j]} \times \frac{[F_c \times M]}{M} = F_c \times \frac{[D_{ij}]}{[c_j]} \tag{6}$$

Note that as with other techniques, the assumptions on which this technique is based are only approximately correct; hence, the above equation is only approximate as well. Nevertheless, in actual use it has proven to be quite satisfactory as judged by the replicability of results. The chief difficulty one initially encounters when attempting to apply the technique is that it is not a simple matter to measure $[C_j]$, the concentration of all forms of carbon associated with the event in a sampling volume. The difficulty arises from the fact that the most abundant final combustion or detonation product is CO_2, and there is a natural background of CO_2 in the atmosphere of about 340 ppmV. One finds that for small amounts of HE or combustible material, it doesn't take long for the excess CO_2 in the cloud to dilute to the point that the CO_2 concentration in the cloud is indistinguishable from background. However, with state of the art techniques, one can measure CO_2 concentrations with about ± 2 ppmV uncertainty.

To illustrate the difficulty, we take the example of 100 pounds (45.4 kg) of pure TNT, with an equivalent atomic formula of $C_7H_5N_3O_6$. Taking into account the atomic weights of the constituents, we find that TNT is 37% carbon by weight. If a detonation of TNT was ideal, essentially all of the carbon would be in the form of CO_2. Thus, 16.8 kg of carbon would combine with 44.8 kg of oxygen to produce 61.6 kg of CO_2, or 1.4 kg moles. At STP, this amount of CO_2 occupies 31.4 m^3. We estimate that at one minute after detonation the cloud of detonation products occupies a volume of about 10^6 m^3. This is consistent with earlier OB/OD experience. Thus, ignoring minor temperature effects, the average concentration of CO_2 in the cloud of detonation products at 1 minute is calculated to be about 31 ppmV. Between two and three minutes, experience indicates that cloud volume will have increased an order of magnitude. Hence, the average concentration of CO_2 will have fallen to about 3 ppmV. At this concentration, the uncertainty in the difference between the average cloud concentration and the background concentration of CO_2 is almost equal to the average excess CO_2 concentration in the cloud. Thus, to use the carbon balance technique with good result on a 100-pound TNT detonation, one has between one and two minutes after the event to make the measurements. Thereafter, the uncertainty on the total cloud content of the species of concern becomes too large.

In practice, Sandia applies the carbon balance technique by using its DeHavilland Twin Otter STOL (Short Takeoff and Landing) instrumented aircraft to sample clouds and plumes. The aircraft has a 3-inch (7.62 cm) diameter sampling probe extending above and forward of the cockpit windshield. The probe transport line enters the top of the aircraft just aft of the cockpit through a gentle S-bend. Once inside the aircraft, the transport line expands to a 4-inch (10.2 cm) manifold which runs the length of the cabin to the baggage compartment at the rear. There the manifold connects to a 4-inch fast-acting pneumatic valve which in its normal position vents the probe flow out the side of the aircraft. The valve is actuated on entering a plume or cloud of interest, and returned to its normal position on emerging. When the valve is actuated, the flow is diverted through three quartz fiber filters to trap particulate material. Typical flow rates are on the order of 200 l/sec. An aliquot of the flow is diverted into an 80 l Tedlar^{TM} bag for the determination of gaseous species. Typically, three passes are made through the cloud and particulate and gases samples are composited on the quartz filter and 80 l bag respectively.

After the pass through the plume or cloud, the Tedlar^{TM} bag contains approximately 80 l of air drawn from the plume or cloud. As soon as the sample is captured, other valves and pumps are actuated to draw the sample into real-time gas analysis instrumentation, and also into a stainless steel canister for later laboratory analysis for CO_2, CO, and other volatile organics by gas chromatography. The real-time instrumentation always includes carbon dioxide and carbon monoxide monitors supplemented with other gaseous and particulate monitors according to the experiment being conducted. It takes about 2-5 minutes to pump the sampling bag empty. While the contents of the sampling bag are being pumped through the real-time instrumentation, the aircraft normally makes one or two more passes through the cloud or plume using its other real-time instrumentation such as particulate spectrometers and nephilometer for characterization. In this manner, the total particulate and gaseous carbon content of each composite sample is measured and can be used int he carbon balance method of determination of emission factors for any particular species of interest.

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APPENDIX C - Soil and Fallout Sampling

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SOIL AND FALLOUT SAMPLING

1. Program Background

Prompted by a growing inventory of excess and/or obsolete munitions and propellants and a concurrent expansion of environmental requirements and restrictions governing their disposal, the U.S. Army Armament, Munitions, and Chemical Command (AMCCOM) undertook a study to determine the environmental effects of open burning (OB) of propellants and open detonation (OD) of munitions and explosives. This effort has been titled the "Open Burning/Open Detonation (OB/OD) Study".

OB/OD techniques are of particular interest to ammunition logisticians because they are the fastest, least expensive, and perhaps the safest of all explosive/propellant disposal techniques. Technology surrounding OB/OD operations has now become routine, and ammunition specialists have mastered its use.

Some concerns primarily environmental, regarding OB/OD, have evolved over a period of years. The Clean Air Act (CAA), Clean Water Act (CWA), and Resource Conservation and Recovery Act (RCRA) are just some of the standards now being applied to military explosive disposal operations. The Department of Defense, in attempting to comply with environmental regulations, does not have a substantial supporting database on products of combustion from OB/OD operations. The OB/OD study is designed to address needs of the ammunition community for such data obtained through the use of a scientific test regimen that is acceptable to federal and state regulators.

2. Project Organization and Responsibilites

2.1. Test grids will be established by Lockheed Engineering and Sciences Company (LESC) at the base of Granite Mountain in the vicinity of West Downwind and Romeo Roads (Figure 1). Separate tests grids will be used for the open burn, surface detonations, buried detonations and suspended detonations to eliminate the possibility of cross contamination from one test to the next. All new detonation test grids will be located in areas free from previous explosive testing.

2.2. Soil sampling and fallout pan sampling will be performed by Lockheed personnel under the direction of the Lockheed Test Officer. Quality Assurance/Quality Control (QA/QC) supervision will be provided by a separate Lockheed QA/QC Officer to avoid any conflict of the data collection and to maintain the integrity of the collected samples. Sample responsibility will remain with the QA/QC division from the time the samples have been collected until they have been given to the appropriate laboratories for analysis. Soil processing prior to analysis will be done by the LESC Soil Sampling division under the direction of the LESC Test Officer. The LESC QA/QC division will monitor all soils processing. The LESC QA/QC Officer will report to the Quality Assurance Officer of Environmental Laboratories, Inc, who in turn, is responsible to the Program Manager or his Technical Steering Committee representative.

3. Objectives

The objective of this study is to identify and quantify the products and residues emitted to the air and to the soil from bulk TNT, Composition "B", Explosive "D", RDX explosives, certain double base propellants, and propellant manufacturing residue during open burning/open detonation (OB/OD) operations. ^Xxhe data obtained from this study is needed to support environmental assessments/other documentation, required under the Clean Air Act (CAA), Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), and other Federal/State environmental statutes and regulations.

4. Soil Sampling and Fallout Pan Procedures

4.1. Sample Handling Procedures.

4.1.1. Bottles used for storing soil samples will be acid washed prior to use and labels applied. Each washed and labeled bottle will be weighed to the nearest 0.01 grams before use and the tare weight will be recorded on the bottle label. This procedure will be followed for all samples collected.

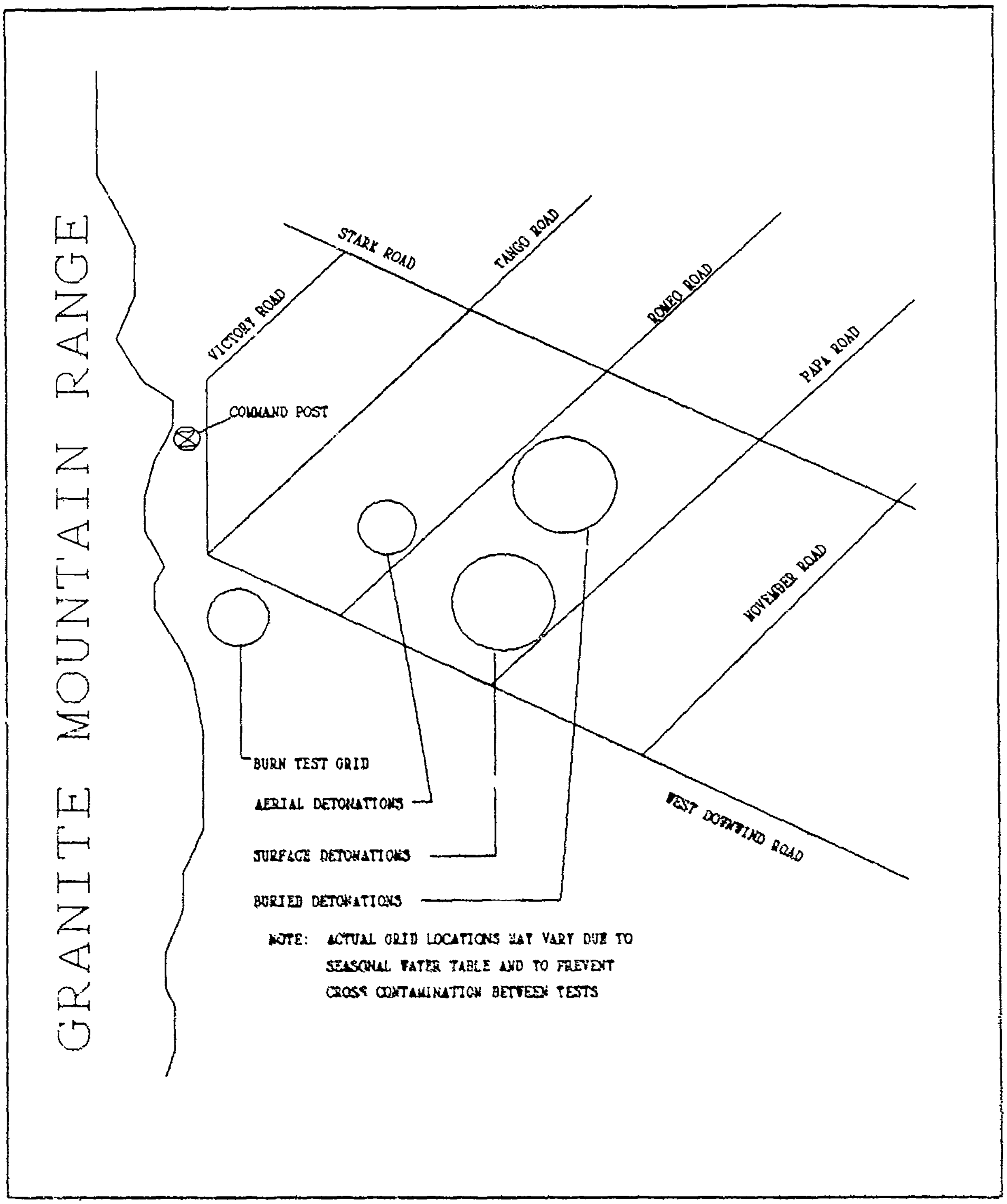
4.1.2. All bottle labels will use the following number system to identify all Soil, Fallout Pan and Sputter Pan samples taken during phase C.

All sample numbers will contain 9 groups (TNT-ODS-P-B-4-000-000- 1 of 4 - A).

group 1	group 2	group 3	group 4	group 5	group 6	group 7	group 8	group 9
TNT	ODS	P	B	4	000	000	1 of 4	A

Group 1 = Material Type
TNT = Trinitrotoluene
CMB = Composition "B"
EXD = Explosive "D"
RDX = RDX explosive
BDB = Burn Double Base
BAP = Burn Ammonium Perclorate
BMR = Burn Manufacturing Residu

Group 2 = Trial type
ODA = Open detonation - aerial
ODS = Open detonation - surface
ODB = Open detonation - buried
OOB = Open burn



NOTE: ACTUAL GRID LOCATIONS MAY VARY DUE TO SEASONAL WATER TABLE AND TO PREVENT CROSS CONTAMINATION BETWEEN TESTS

Figure 1. Test Grid Location

Group 3 = Trial Number
P = Pre test sample
O = ORI
1 = First test series
2 = Second test series
3 = Third test series

Group 4 = Detonation or Burn number within a trial
B = Background
O = ORI
1 thru 6 = Detonation or Burn site

Group 5 = Sample type
1 = Soil pretest core
2 = Soil ring - Aerial detonations
3 = Fallout Pan
4 = Soil - ejecta core
5 = Sputter Pan
6 = Burn Pan residue

Group 6 = Radius from grid center or distance from crater rim
050 = 50 meters from grid center
0R1 = 1 meter from the rim of a crater
01A = Detonation site 1 core A

Group 7 = Degrees from grid north
000 = Grid north
090 = 90 degrees from grid north
WHEN GROUP 5 IS 1, THEN
000 = unsegmented core B
072 = Top 72" of core A
168 = Bottom 96" of core A (from 72" - 168")
Note: Sample will be identified by the deepest point to which it is taken.

Group 8 = Number of sample bottles filled at a sampling site
1 of 4 = first sample bottle from a total of 4 samples taken

Group 9 = Duplicate sample identification
A = First sample at a duplicate location
B = Duplicate sample
Note: If duplicate sampling is not required then group 9 identification will not be used.

4.2. Duplicate Sampling

Duplicate soil samples will be collected on one detonation crater from each of the four explosive types. All twelve crater sampling points will be duplicated. The duplicate samples will be taken within .5 meters of the original location in soil that has not been physically or mechanically disturbed in taking the first sample. Identification is outlined in paragragh 4.1.2.

Duplicate pan samples will be collected on one detonation at each of the 24 sample locations of each of the explosive types. Minimal pan spacing is required. Sample collection procedures are outlined in LESC LOI No. 1.

5. Surface Multiple Detonation Test

The Phase C Field Test will not be conducted in the same location as the Phase "A" or "B" Field Test. Pretest core samples will be required at each detonation point.

5.1. Pretest Core Samples

Two cores will be taken within 1 meter of each other (designated "A" and "B")at the center of each of the surface detonation points. A configuration is shown in Figure 2 for accomplishing the surface detonations for one type of explosive material showing sites for two trials of three detonations each and an ORI detonation. All detonation points will be separated by a minumum of 600 meters. A 2½ inch split spoon core sampler will be used. The two cores taken at each site will be identified according to sample numbers in paragraph 4.1.2.

Core "A" for surface detonation sites will consist of three increments, 0 - 18", 18" - 54", and 54" - 84".

Core "B" will be one sample taken to a depth of 7 feet.

5.2. Post Test Soil Sampling. Only one site per 3 shot series will be sampled.

5.2.1. Ejecta Sampling ----Twelve soil samples of the ejecta material inside the crater and immediately adjacent to it will be taken using a 2" core sampler after the detonation series.

5.2.1.1. Sampling procedure. The 2" core sampler will be inserted vertically into the ejecta and with a twisting motion, pushed downward until the hard surface of the undisturbed soil is encountered. A narrow trench will then be dug along the side and to the same depth as the sampler. A 2½" or wider putty knife will be inserted under the core sampler to prevent loss of the material from the sampler as it is removed from the soil.

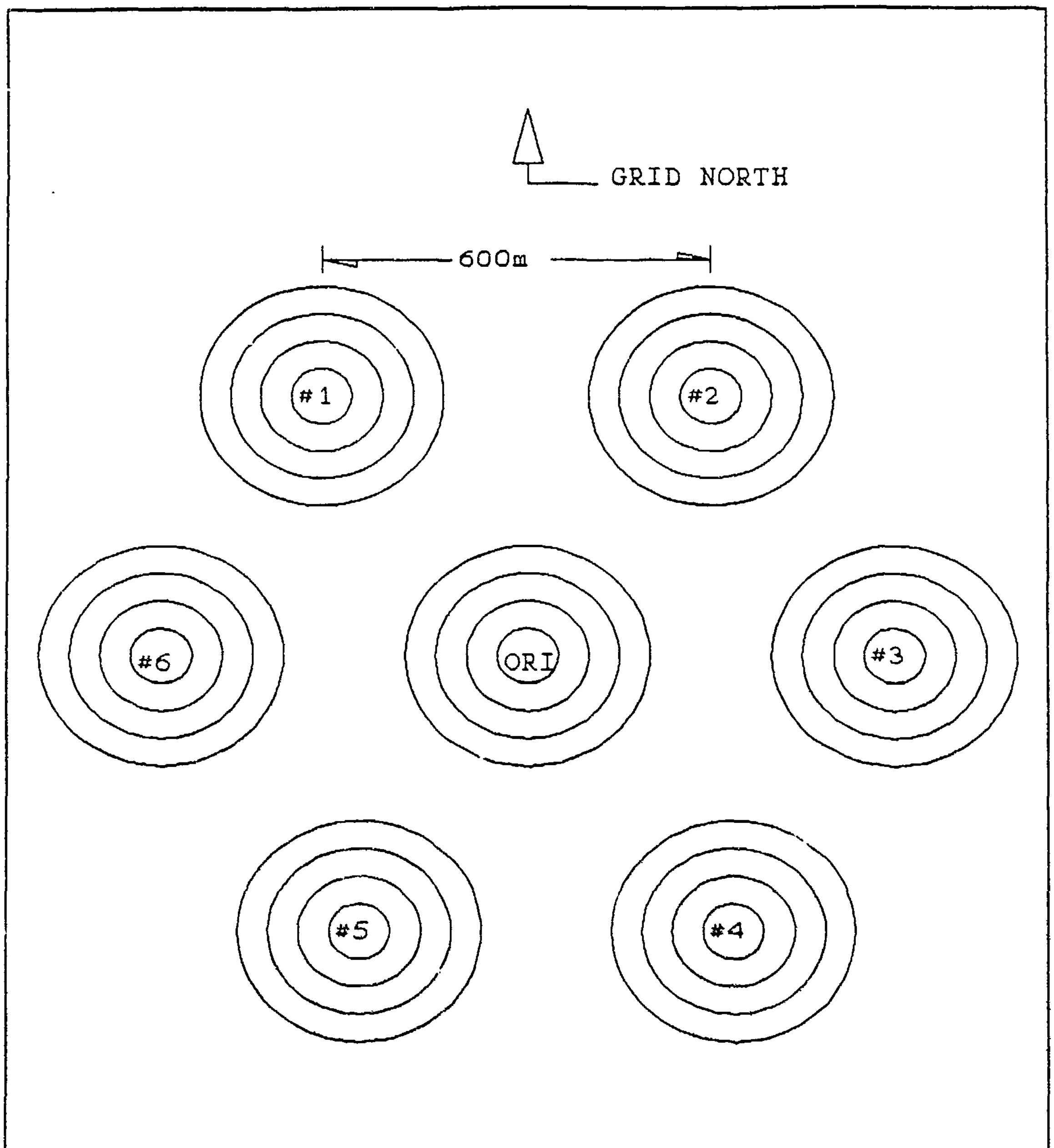


Figure 2. Surface Detonation Layout

5.2.1.2. Sampling Locations. The lowest point of the crater and the crater rim will be visibly determined by the Grid Sampling Supervisor. Samples of the detonation crater ejecta material will be taken as follows:

- 3 meters from the lowest visible point of crater on 4 diagonal lines established at 90° intervals from grid north (4 samples)
- 1 meter from the rim of the crater on the diagonal lines (4 samples)
- 4 meters from the rim of the crater on the diagonal lines (4 samples)

5.2.2. Fallout Pan Sampling ----Fallout pans will be placed out for collection of sample prior to the detonations. The pans have a permanent identification number and the dimensions of the pan have been recorded in a permanent file. This pan number along with the fallout weight are recorded in the field data pan sampling log.

Four concentric circles for pan sampling purposes will be placed around each surface detonation site (Figure 3). The circles will be at 50 m intervals, beginning at 50 meters out from the detonation point. Six fallout pans will be placed on each circle with a 60 degree spacing. There will be a 30 degree stagger of pans and soil sampling locations between circles as follows:

- 50 meter circle pans located at 0, 60, 120, 180, 240, and 300 degrees.
- 100 meter circle pans located at 30, 90, 150, 210, 270 and 330 degrees.
- 150 meter circle pans located at 0, 60, 120, 180, 240 and 300 degrees.
- 200 meter circle pans located at 30, 90, 150, 210, 270 and 300 degrees.

6. Propellant Burn Test

Fallout pans will be placed out for collection of samples prior to the burns. No pretest sampling will be taken. Two types of samples will be taken with the fallout pans. One series of pans will be placed to collect "sputter" of the propellant, while the other series will be concentric rings of pans to collect fallout.

6.1. "Sputter" Pan Samples ---- Sputter pans (10) will be placed as shown in Figure 4 within 1 m of the burn pans. Pans will be covered upon placement, and remain covered until just prior to the burn. Procedures used for recovery of the samples are outlined in LESC LOI No. 1.

6.2. Fallout Pan Samples ----Fallout pans will be located on two concentric rings, 6 and 12 m from grid center. Four pans will be on the 6 meter ring and six pans will be located on the 12 meter ring. All pans will be located at 60 degree arc spacing with a 30 degree offset between rings

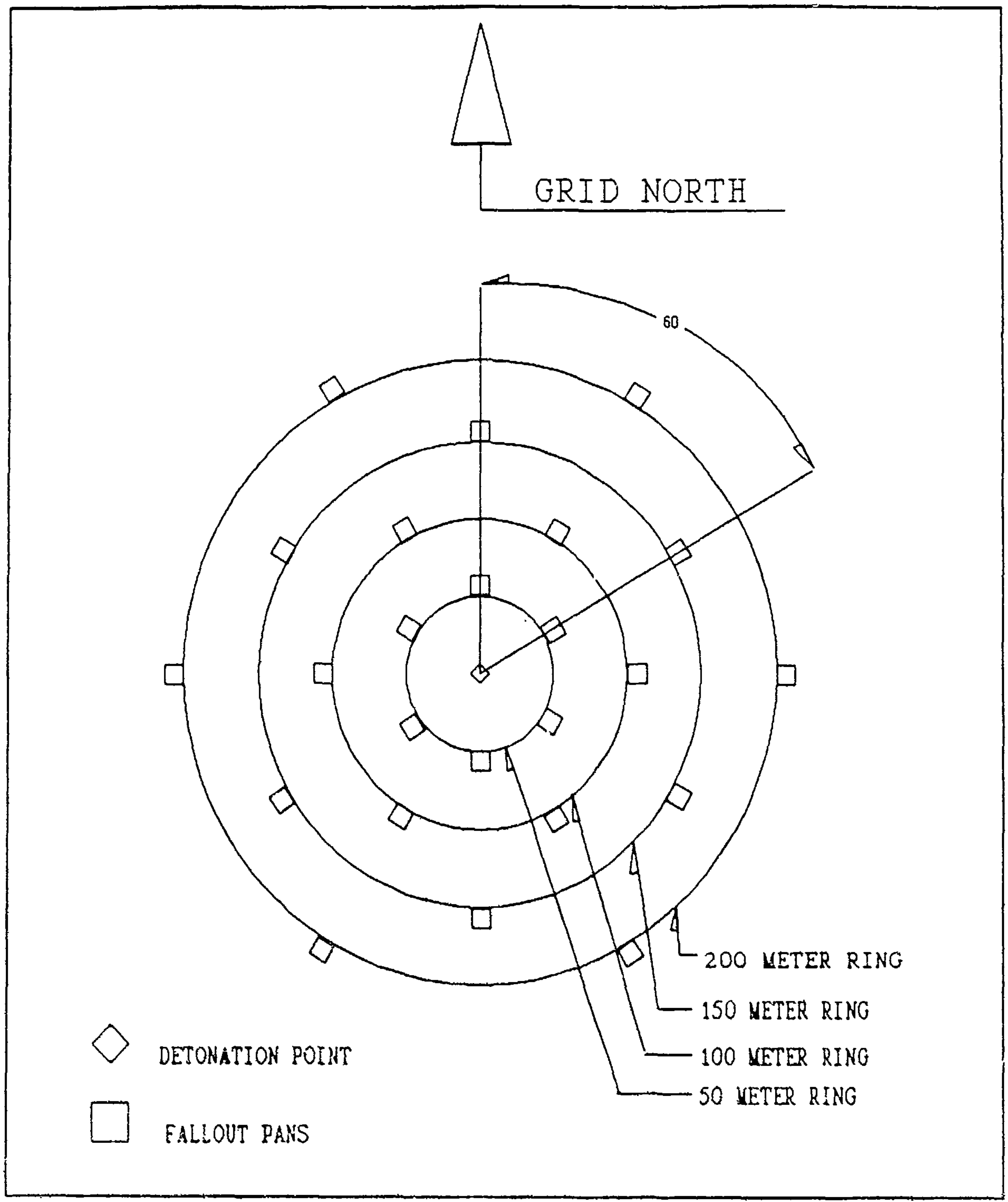


Figure 3. Fallout Pan Locations for Surface Detonations

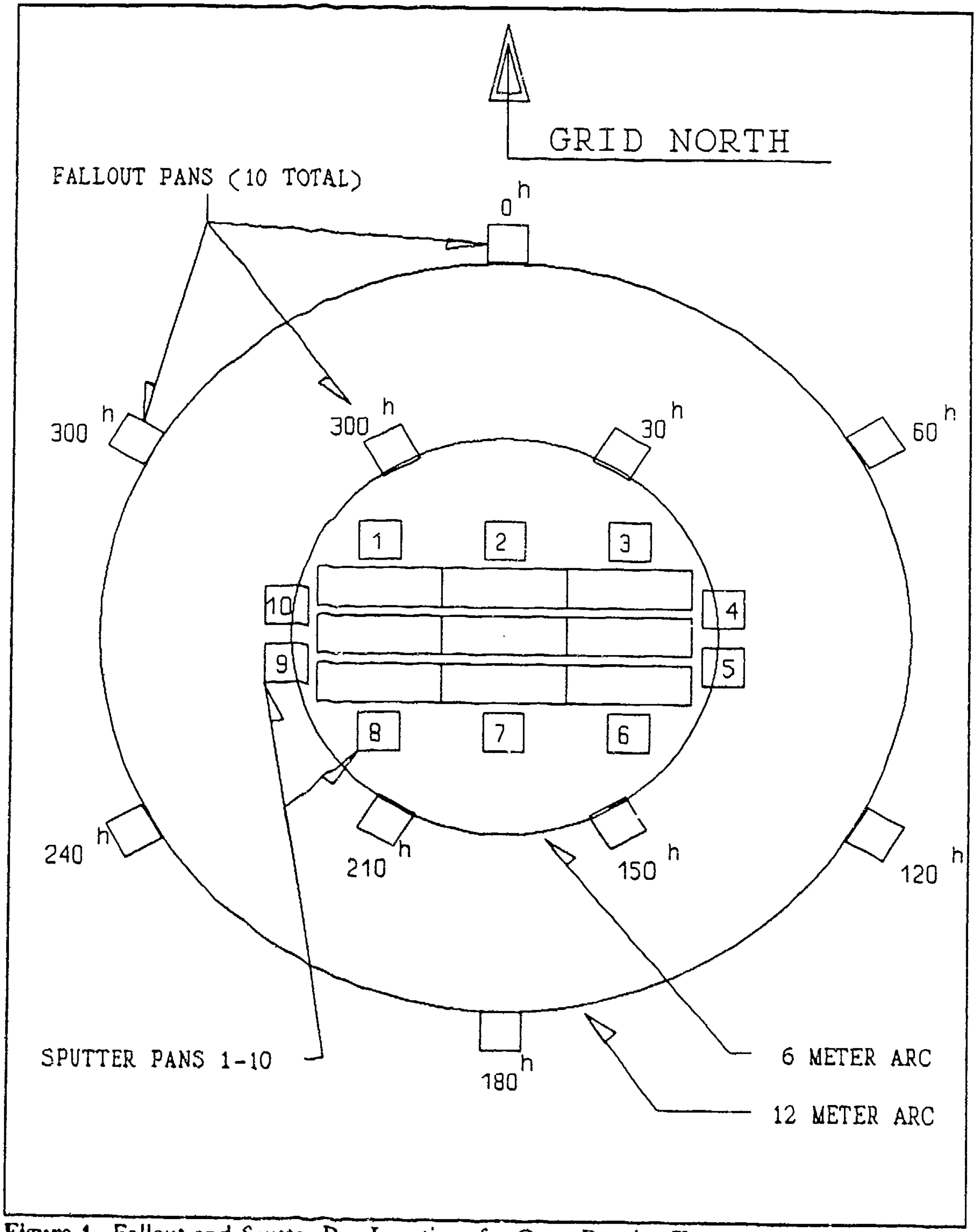


Figure 4. Fallout and Sputter Pan Locations for Open Burning Test

(Figure 4). Procedures used for recovery of the samples are outlined in LESC LOI No. 1.

6.3. Burn Pan Residue ----Residue from the propellants will be collected from the burn pans as soon as the pans are cool enough for safe collection after the burn. The residue will be swept up and collected into acid washed bottles with Teflon™ lined lids. Each burn pan residue will be weighed separately and held as a discrete sample. Two, one liter samples from each pan will be archived at -20° C at DPG. A one liter composite will be made from all burn pans and sent to Chemtech Laboratories located in Murray, Utah for analysis. All the residue remaining will be held until the determination on proper disposal is made by the PO.

7. Single Detonation Site With Multiple Detonations

7.1. Pretest Core Samples ----Two cores will be taken within 1 meter of each other (designated "A" and "B")at the center of each of the surface detonation points. All detonation points will be separated by a minumum of 600 meters. A 2½ inch split spoon core sampler will be used. The two cores taken at each site will be identified according to sample numbers in paragraph 4.1.2.

- Core "A" for surface detonation sites will consist of three increments, 0 - 18", 18" - 54", and 54" - 84".

- Core "B" will be one sample taken to a depth of 7 feet.

7.2. Post-test Ejecta Sampling ----Twelve soil samples of the ejecta material inside the crater and immediately adjacent to it will be taken using a 2" core sampler after every detonation.

7.2.1. Sampling procedure. The 2" core sampler will be inserted vertically into the ejecta and with a twisting motion, pushed downward until the hard surface of the undisturbed soil is encountered. A narrow trench will then be dug along the side and to the same depth as the sampler. A 2½" or wider putty knife will be inserted under the core sampler to prevent loss of the material from the sampler as it is removed from the soil.

7.2.2. Sampling Locations. The lowest point of the crater and the crater rim will be visibly determined by the Grid Sampling Supervisor. Samples of the detonation crater ejecta material will be taken as follows:

- 3 meters from the lowest visible point of the crater on 4 diagonal lines established at 90° intervals from grid north (4 samples)

- 1 meter from the rim of the crater on the diagonal lines (4 samples)

- 4 meters from the rim of the crater on the diagonal lines (4 samples)

8. Suspended Detonations

8.1. Pretest Background Soil Samples

The sampling area will be be divided into four quadrants each containing 5 samples. Quadrant #1 will start at grid north and continue clockwise 90°. Quadrants 2, 3, and 4 will continue in a clockwise rotation at 90° intervals. Beginning at grid center, radials will be established at 1, 2, 4, 8 and 16 meters from center. Each radial sampling position will be offset 45° (Figure 5). The soil sampling tool will consist of a ring 20 cm in diameter and 2.5 cm deep. The sampler will be pressed into the soil halfway, to a depth of 1.25 cm and all the soil within the ring will be removed with a stainless steel putty knife or scoop. The samples will be placed in acid washed bottles with Teflon lined lids.

- Five soil samples (20 samples total per site) will be taken from each of the four main quadrants.
- Each sample will be collected, weighed to the nearest 0.01 gram, and stored at -20° C at DPG in labeled, acid washed bottles with Teflon™ lids, as a discrete sample.

8.2. Post test Soil Sampling

Sampling positions will be located at the same locations as used for the pretest soil samples (20 samples per detonation). Samples will be taken as close to the original position as possible. Only one detonation site for each three shot series will be sampled.

9. Buried Detonations

9.1. Pretest Core Samples

Two cores will be taken within 1 meter of each other (designated "A" and "B")at the center of each of the buried detonation points. All detonation points will be separated by a minumum of 600 meters. A 2½ inch split spoon core sampler will be used. The two cores taken at each site will be identified according to sample numbers in paragraph 4.1.2.

- Core "A" will be taken in two sections. The top section will be taken to a depth of 54" (6 inches below the bottom of the burial pit). The bottom section will be taken from 54" to 90" (6 inches below the expected crater depth).
- Core "B" will be one sample taken to the same depth as Core "A".

9.2. Post Test Soil Sampling. Only one detonation site for each three shot series will be sampled.

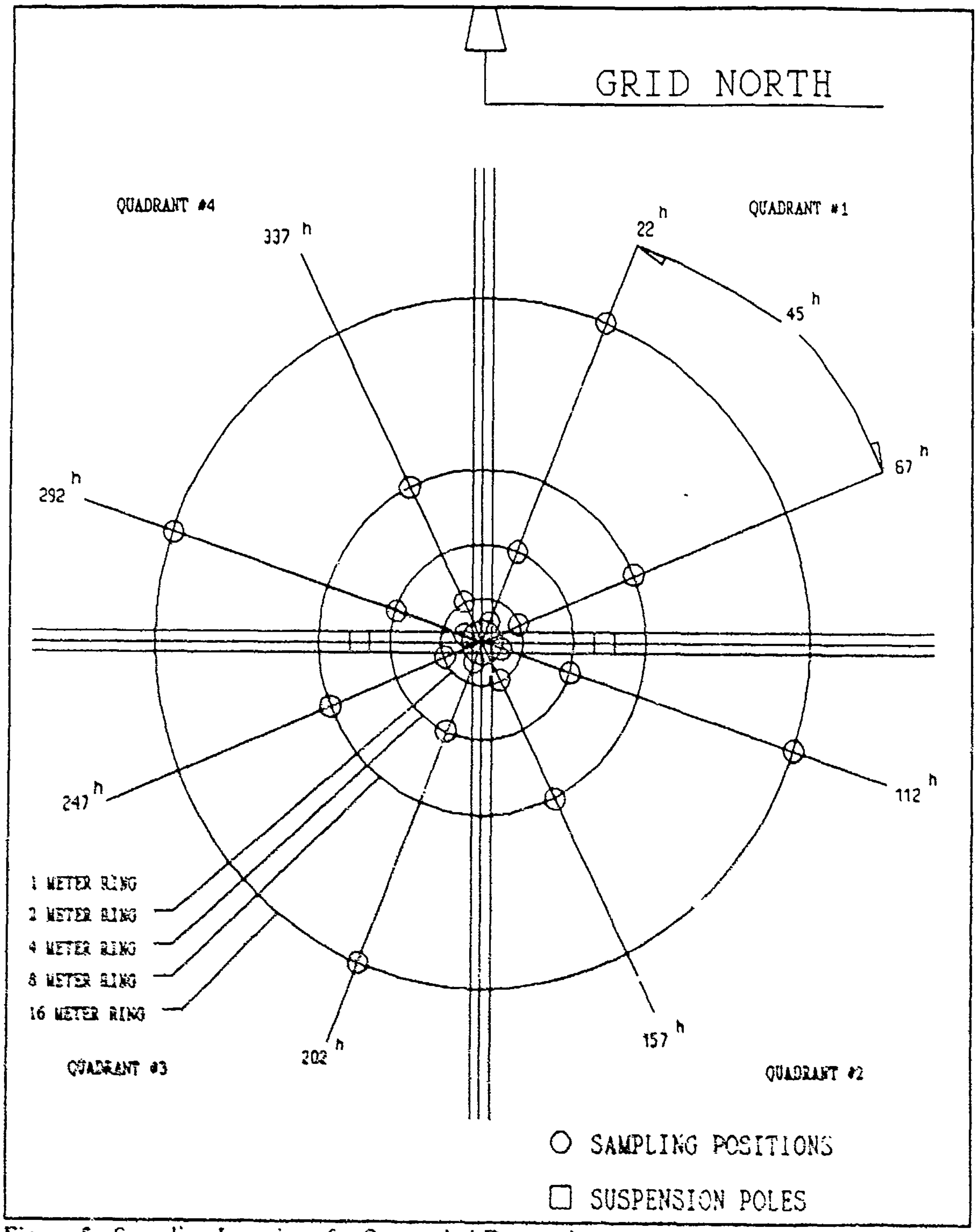


Figure 5. Sampling Locations for Suspended Detonations

9.2.1. Ejecta Sampling ----Twelve soil samples of the ejecta material inside the crater and immediately adjacent to it will be taken using a 2" core sampler after every detonation.

9.2.1.1. Sampling procedure. The 2" core sampler will be inserted vertically into the ejecta and with a twisting motion, pushed downward until the hard surface of the undisturbed soil is encountered. A narrow trench will then be dug along the side and to the same depth as the sampler. A 2½" or wider putty knife will be inserted under the core sampler to prevent loss of the material from the sampler as it is removed from the soil.

9.2.1.2. Sampling Locations. The lowest point of the crater and the crater rim will be visibly determined by the Grid Sampling Supervisor. Samples of the detonation crater ejecta material will be taken as follows:

- 3 meters from the lowest visible point of crater on 4 diagonal lines established at 90° intervals from grid north (4 samples)
- 1 meter from the rim of the crater on the diagonal lines (4 samples)
- 4 meters from the rim of the crater on the diagonal lines (4 samples)

9.2.2. Fallout Pan Sampling

Fallout pans will be placed out for collection of sample prior to the detonations (Figure 6). The pans have a permanent identification number and the dimensions of the pan have been recorded in a permanent file. This pan number along with the fallout weight are recorded in the field data pan sampling log.

9.2.2.1. Four concentric circles of fallout pans will be placed around each buried detonation site (see Figure 6). The circles will be at 50 m intervals, measured from the center of the detonation point. Six fallout pans will be placed on each circle with a 60 degree spacing. There will be a 30 degree stagger of pans between circles as follows:

- 50 meter circle pans located at 0, 60, 120, 180, 240 and 300 degrees.
- 100 meter circle pans located at 30, 90, 150, 210, 270 and 330 degrees.
- 150 meter circle pans located at 0, 60, 120, 180, 240 and 300 degrees.
- 200 meter circle pans located at 30, 90, 150, 210, 270 and 330 degrees.

After the ORI is conducted, it may be determined by the Program Manager or his representative, to eliminate any circle of sample pans that may be beyond the fallout pattern.

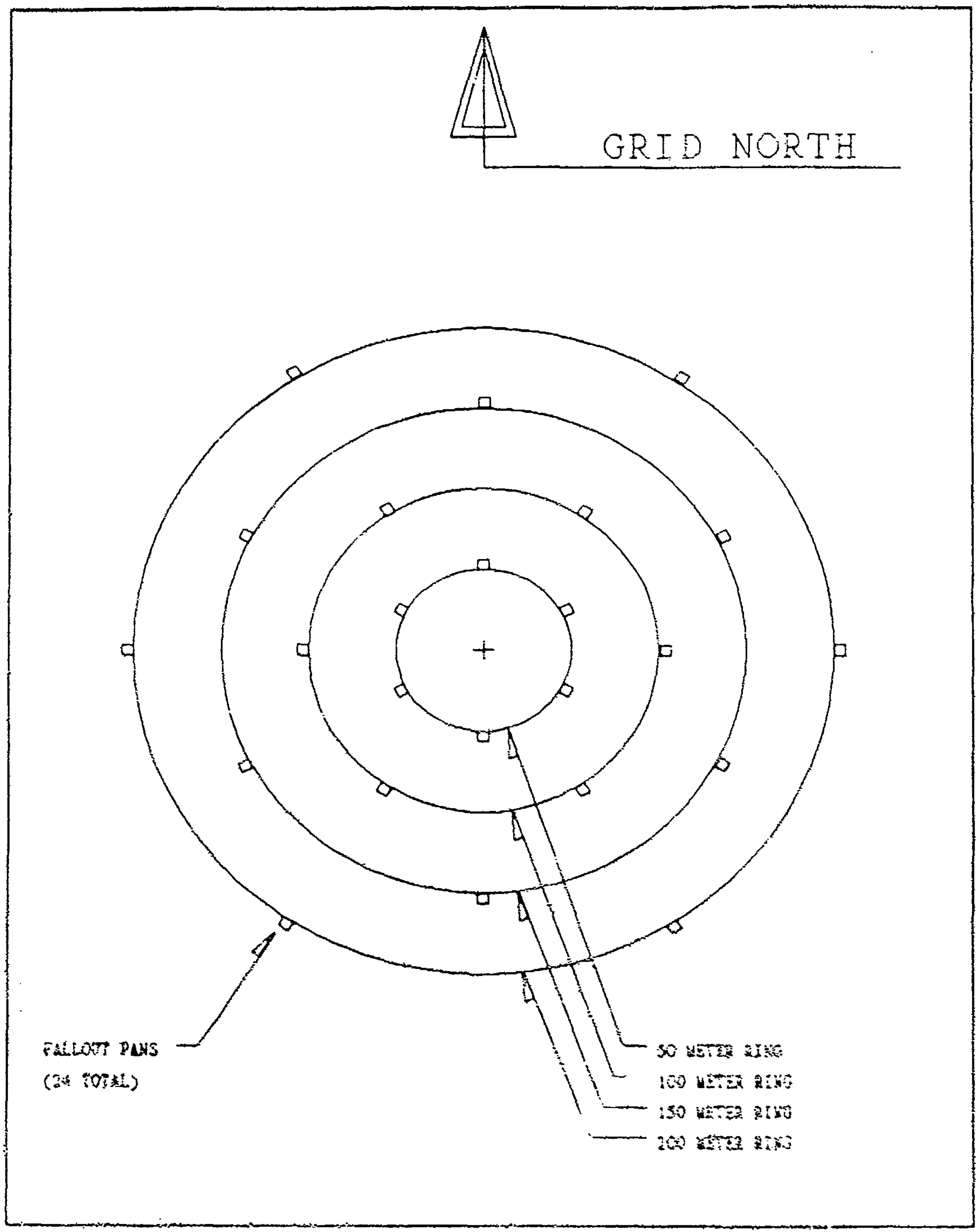


Figure 6. Fallout Pan Locations for Buried Detonations

APPENDIX D. REFERENCES

1. Headquarters, Department of the Army, Washington, D.C., Army Regulation (AR) 530-1, Operations Security (OPSEC), 15 October 1985.

2. U.S. Army Dugway Proving Ground, Dugway, Utah, Environmental Assessment for Open Burning/Open Detonation Test at U.S. Army Dugway Proving Ground, Dugway, Utah, TECOM Project Number 2-CO-210-000-017, 23 February 1989.

3. Headquarters, Department of the Army, Washington, D.C., AR 200-2, Environmental Effects of Army Actions, 16 November 1988.

4. Headquarters, U.S. Army Materiel Command, Alexandria, Virginia, AMC Regulation 385-100, Safety Manual, 1 August 1985.

5. Headquarters, Department of the Army, Washington, D.C., AR 420-47, Solid and Hazardous Waste Management, 1 January 1985.

6. U.S. Army Dugway Proving Ground, Dugway, Utah, DPG Regulation 420-10, Facilities Engineer Installation Hazardous Waste Management Plan, 29 June 1988.

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APPENDIX E. ABBREVIATIONS

AMC - U.S. Army Materiel Command

AR - Army Regulation

ASASP - active scattering aerosol spectrometer probe

AWL - Alpine West Laboratories

BYU - Brigham Young University

CAA - Clean Air Act

CP - Command Post

CWA - Clean Water Act

DMWR - depot maintenance work requirement

DPG - U.S. Army Dugway Proving Ground

DTP - Detailed Test Plan

EPA - U.S. Envirnomental Protection Agency

FID - flame ionization detector

FSSP - forward scattering spectrometer probe

FWAC - fixed-wing aircraft

GC-MS - gas chromatography-mass spectrometry

IAW - in accordance with

LOI - Letter of Instruction

MAAF - Michael Army Airfield

NC - nitrocellulose

NG - nitroglycerine

NIST - National Institute of Standards and Technology

NO_x - nitrogen oxides

OB - open burning

OB/OD - Open Burning/Open Detonation

OD - open detonation

OGC - Oregon Graduate Center

OPSEC - operations security

ORI - operational readiness inspection

PIXE - proton-induced x-ray emission

PM - program manager

PO - Project Officer

QA - quality assurance

QAPP - quality assurance project plan

QC - quality control

RCRA - Resource Conservation and Recovery Act

RDX - cyclotrimethylenetrinitramine

SFC-MS - supercritical fluid chromatography-mass spectrometry

SNL - Sandia National Laboratories

SSL - Sunset Laboratories

SOP - standing operating procedure

TA - triacetin

TNT - 2,4,6-trinitrotoluene

APPENDIX F. DISTRIBUTION LIST

Addressee	Copies
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Rock Island, IL
61299-6000 1

Sandia National Laboratories
ATTN: Mr Wayne Einfeld
DIV 6321
P.O. Box 5800
Albuquerque, NM 87111 4

APPENDIX B. LETTERS OF INSTRUCTION

During BangBox testing, letters of instruction (LOIs) were modified and revised to incorporate lessons learned. In some instances, no changes were necessary; in others, considerable revision occurred before testing concluded. The LOI's in this appendix reflect procedures used as the test ended and which were expected to be used during future tests.

SECTION 1 - [Soil] Sampling Handling Procedures (LESC)

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SAMPLE HANDLING PROCEDURES

FALLOUT AND SPUTTER PANS

1. A new pair of white cotton gloves must be worn by all personnel that will handle any samples or sampling equipment.

2. Set out covered clean pans as in grid diagram for appropriate test and stake down as required. The grid sampling supervisor will record each pan serial # and grid location on Sample Log sheet (enclosure 1). This same Sample Log sheet will be passed on to the QA/QC division for use when the samples are collected.

3. Load explosives or propellants.

4. Remove pan covers after explosives or propellants are set out and before EOD arms the event.

5. Arm explosive or propellant.

6. Burn or detonate explosive or propellant.

7. Reenter grid upon EOD approval.

8. Cover all pans prior to sample collection.

9. Uncover each pan when ready to remove sample material and perform the following:

    a. Compare label on jar with identification for trial and grid site and cross check with Pan Sample Log sheet that has been previously filled out.

    b. Visually inspect and document the contents of each pan.

    c. Remove any non fallout material (vegetation, rocks, etc.)

    d. Elevate pan to allow for contents to be swept into a sampler jar.

    e. Remove lid from tared jar.

    f. Place jar into compartment on the sample collection table.

    g. Place paper funnel into the hole in the top of the collection table to direct fallout material into jar.

h. Sweep fallout material from pan into jar with a new nylon brush.

i. Recap jar with the same Teflon lined lid.

j. Place filled sample jar beside the fallout pan.

k. Discard gloves, brushes, and paper funnel. Do not reuse.

l. The QA/QC person will retain the Sample Log sheet until all locations have been completed. The log sheets will then be turned over to the grid sampling supervisor and the labels rechecked when the samples are picked up by the QA/QC division.

10. Sample custody will remain with the QA/QC division until sample processing for analysis. Samples will then be checked out thru the QA/QC division.

11. Pick up clean white cotton gloves, brush, and paper funnel and move to the next sampling position. Repeat steps 9 a-l above until all positions have been completed.

ENCLOSURE 1 - Sample Log Sheet

		Trial #:
		Event:
		Date
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		
Pos. # (Az/M)	Pan #	Sample #
Comments: (Contents, What Removed, % Ash or Dust, etc)		

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SURFACE DETONATIONS

1. A new pair of white cotton gloves must be worn by all personnel that will handle any samples or sampling equipment. Prior to sampling, a sample log sheet will be filled out by the grid sampling supervisor. This sample log sheet will be turned over to the QA/QC division for monitoring during the sample collection process.

2. Enter grid upon approval of EOD.

3. Sample positions in the following order:

    a. Lowest visible point of the crater.

    b. One meter out on four diagonals from the lowest visible point of the crater.

    c. One meter out from the rim of the crater on the same diagonal lines.

    d. Four meters out from the rim of the crater on the same diagonal lines.

4. Insert the 2" core sampler vertically into the ejecta and with a twisting motion press it downward until it encounters hard undisturbed soil.

5. Using a shovel, dig a trench beside the sampler until the bottom of the sampler is reached.

6. Insert a 2 ½" (minimum) wide putty knife under the core sampler to prevent loss of soil when sampler is withdrawn for the soil.

7. Remove the sampler with the soil inside.

8. Remove sampler jar from the box and insure label on jar corresponds with position on the grid and the sample log sheet. Remove the lid from the jar.

9. Place the soil sample in the properly labeled, acid washed jar.

10. Replace Teflon lined lid on the jar.

11. Place the sealed sample jar beside the sampling position and discard gloves, sampler and putty knife. The sampler and putty knife will be blown clean with reagent grade nitrogen for reuse on the next sample or test.

12. Pick up a clean pair of cotton gloves, a core sampler and putty knife and move to the next sampling position. Repeat steps 4 thru 11 until all positions (13) have been sampled.

13. The sample log sheets will be turned over to the grid sampling supervisor and the labels rechecked when the samples are picked up by the QA/QC division.

14. Sample custody will remain with the QA/QC division until sample processing for analysis. Samples will then be checked out thru the QA/QC division.

SUSPENDED DETONATIONS

1. A new pair of white cotton gloves must be worn by all personnel that will handle any samples or sampling equipment. Prior to sampling, a sample log sheet will be filled out by the grid sampling supervisor. This sample log sheet will be turned over to the QA/QC division for monitoring during the sample collection process.

2. Enter grid upon approval from EOD.

3. Sample positions in the following order:

    a. All positions in the 16 meter ring.

    b. All positions in the 8 meter ring.

    c. All positions in the 4 meter ring.

    d. All positions in the 2 meter ring.

    e. All positions in the 1 meter ring.

4. Press soil ring completely into the soil.

5. Remove sample jar from box and insure label on jar corresponds with position on the grid and the sample log sheet. Remove the lid from the jar.

6. Insert a new paper funnel into the jar.

7. With the aid of a putty knife or scoop, remove all soil from the inside of the soil ring and place it in the sample jar.

8. Replace the Teflon lined lid on the jar.

9. Place the filled sample jar on the ground where the sample was taken.

10. Discard gloves, soil ring and putty knife or scoop. The soil ring and putty knife or scoop may be cleaned with reagent grade nitrogen and reused at the next sample or test.

11. Pick up a clean pair of cotton gloves, soil ring, putty knife or scoop and move to a new sampling position and repeat steps 3 thru 10 until all positions have been sampled.

13. The sample log sheets will be turned over to the grid sampling supervisor and the labels rechecked when the samples are picked up by the QA/QC division.

14. Sample custody will remain with the QA/QC division until sample processing for analysis. Samples will then be checked out thru the QA/QC division.

BURN PAN RESIDUE

1. Burn pan residue sampling will not commence until pans have sufficiently cooled for safety reasons.

2 The ash from each pan will be swept to one end with a corn broom, taking care not to walk in unswept area of the pan.

3. Put on a clean pair of cotton gloves.

4. Remove residue from the burn pans with a clean hand brush and dust pan and place it into properly labeled, acid washed bottles.

5. Clean broom, brush and dust pan with reagent grade nitrogen gas.

6 Move to the next pan and repeat steps 2 thru 5 above.

7. Sample custody will remain with the QA/QC division until sample processing for analysis. Samples will then be checked out thru the QA/QC division.

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SECTION 2 - Test Sample Processing for Soil and Ejecta Samples (LESC)

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Lockheed Eng. & Sciences Co.
OB/OD Field Test Phase C
LOI No. 2
1 March 1990

OPEN BURN/OPEN DETONATION

PHASE C

TEST SAMPLE PROCESSING FOR SOIL AND EJECTA SAMPLES

1. Remove sample from the freezer.
2. Thaw sample for 12 hours.
3. Clean cotton gloves will be worn by all personnel that could handle soil samples at any time.
4. Blow all sample processing equipment clean with reagent grade nitrogen.
5. Remove all metal pieces, stones and vegetation. This may be aided by passing the sample through a sieve column, however, removed material must be recorded and weighed prior to discard.
6. Pass sample thru sieve column.
    a. 3/8 inch (.375) mesh screen.
    b. #4 (.187) mesh screen.
7. Grind/crush all soils not passing a sieve column.
8. Pass ground soils through a sieve column.
9. Repeat steps 6 thru 8 until the total sample has been sieved.
10. Place all of sieved sample into mixer (6 or 12 quart as appropriate for sample volume). Homogenize for 2 minutes.
11. Weigh and record weight of homogenized sample.
12. Pass homogenized portion of the sample through a sample splitter and separate into two, approximately equal parts.

13. Weigh each portion and record.

14. Return one portion of the sample to the original container and change the label to reflect the weight change. Place the second portion of the sample into a separate container where it will become a part of the composited sample. If no compositing of sample is required place the second portion of sample in a properly labeled jar and go to Step 19.

15. All processing equipment will be blown clean between samples.

16. Repeat steps 5 thru 14 until all samples that will become part of the composited sample have been prepared and placed in the mixer.

17. Homogenize for two minutes.

18. Pass composited, homogenized sample through the splitter until it is reduced to a sample of approximately 700 grams.

19. Place approximately 500 grams of the sample in a properly labeled and weighed sample bottle for chemical assay, place the remainder of the composited sample into a properly labeled and weighed sample bottle for determining moisture content. In the event less than 500 grams is available, the entire sample will be used in the chemical assay.

20. Deliver all samples to the appropriate laboratory.

21. Return all unsieved and composited, but unused, portions of the samples to the freezer for storage (-20 degrees C).

SECTION 3 - Soil Sampling QA/QC (LESC)

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Lockheed Engineering & Sciences Co.
OB/OD Field Test Phase C
LOI No. 3
1 March 1990

Open Burn/Open Detonation
Soil Sampling QA/QC LOI

Project Organization and Responsibilities:

Soil sampling and fallout pan sampling will be performed by LESC personnel under the direction of the LESC test officer. Quality assurance/quality control will be provided by a separate LESC QA/QC officer to avoid any conflict of the data collection and to maintain the integrity of the collected samples. Sample responsibility will remain with the QA/QC division from the time the samples have been collected until they have been given to the appropriate laboratories for sample preparation (sieving, grinding, compositing, reweighing, etc.) and analysis. The LESC QA/QC officer will be under the direction of and will report to the Quality Assurance officer of ELI. Any time testing/sampling is in progress a QA/QC person will be present to observe and take notes on all test activities.

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1. Does the Label on me sample container correspond with the designated sample position.

2. Did all sampling personnel wear a clean pair of white cotton gloves for each sample location.

3. Were vehicles operated within 100 ft of sample positions.

4. Were any personnel observed smoking on the grid in vehicles transporting samples or in sample processing area.

5. Were samplers blown clean with reagent grade nitrogen between uses.

6. Were fallout/sputter pan lids removed from pans and placed in covered vehicles just prior to test.

7. Were all fallout pans and lids blown clean with reagent grade nitrogen between uses.

8. Were samples left at the sampling site at the completion of sampling.

9. Were the labels on the samples checked by a QA/QC supervisor prior to the samples being picked up for transport to the soils lab.

10. Were sample containers tare weighed.

11. Had the soil balance been calibrated within the past 6 months and proper certification attached to the instrument or posted.

YES	NO
1	
2	
3	
4	
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9	
10	
11	

* Any question answered with a * requires a written explanation.

12. Were certified reference weights used during the weighing of the samples (one for each 5 sample weights).

13. Were sample weights properly recorded on the sample label and in the log book.

14. Were sample audit trails traceable and samples properly preserved (freezer -20 C).

15. Were sampling crews knowledgeable of LOI and did they appear to know their respective jobs.

16. When sample positions required marking (ejecta samples at craters) was all required equipment available and personnel knowledgeable of procedures.

YES	NO
12	
13	
14	
15	
16	

* Any question answered with a * requires a written explanation.

SECTION 4 - Explosives and Propellant Emplacement (LESC)

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Lockheed Engineering & Sciences Co.
OB/OD Field Test Phase C
LOI No.4
1 March 1990

Open Burn/Open Detonation
Explosives/Propellant Emplacement LOI

1. Receipt of explosives and propellants:

a. Explosives and propellants will be receipt inspected in accordance with DPG SOP DP-0000-L-105.

b. Any explosive or propellant container that is leaking liquids will be marked for easy identification and the DPG Test Officer will be notified.

2. Emplacement of explosives:

a. Construct explosives containers of 18 gauge cold rolled steel.

b. Preplace explosive container on the test grid.

c. Place a 3/4 inch plywood walkway on the ground between the explosive's truck and the explosive's container.

d. Explosives will be emplaced by EOD personnel only.

e. After explosives are emplaced, sweep the walkway and place any spilled explosive that is recovered into the explosive container.

f Record the following data on each detonation:

(1) Test and trial number.

(2) Explosive type.

(3) Lot number(s)

(4) Explosive weight.

g. Obtain one sample of approximately S grams of explosive from each lot number and deliver to AWL for analysis.

3. Emplacement of propellants:

a. Place a 3/4 inch plywood walkway between the propellant truck and the burn pans.

b. Record the following data on each burn test:

(1) Test and trial number.

(2) Propellant type.

(3) Lot numbers(s).

(4) Propellant weight.

c. Obtain one sample of approximately 5 grams of propellant from each lot number and deliver to AWL for analysis.

d. Propellant will be emplaced by EOD personnel only.

e. After propellants are emplaced, sweep the walkway and place any spilled propellants that are recovered into the burn pans.

f. Describe the arrangement of propellants by type and lot number in the burn pans.

SECTION 5 - Site Selection for OB/OD Grid Complex (DPG)

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SITE SELECTION FOR OB/OD GRID COMPLEX

1. A remote testing area has been selected for the Open Burning/Open Detonation (OB/OD) grid complex at Dugway Proving Ground (DPG). This grid will be used for 1) limited scale field tests, and 2) detailed field tests.

2. Site selection criteria.

a. Clean area, one that has not been used and is not contaminated.

b. Area that provides for aircraft safety: an open flying area, free of mountainous terrain, power lines, poles, etc.

c. Existing control point (Tower Grid) that allows for visibility of test grid.

d. Isolated area so that blast effects are minimized.

e. Photo documentation accessibility, allowing for complete 360 degree camera coverage.

f. Area with predictable wind patterns.

g. Accessibility to grid by roads for vehicular traffic.

h. Relatively flat terrain.

i. Large complex with all grids on comparable terrain.

j. Small chance of cross contamination due to the large complex size.

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SECTION 6 - Filter Weighing Operating Procedure (LESC)

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SECTION 1. Purpose

This document describes the procedures used for weighing the (25 X 20 cm) Teflon coated glass fiber filters (Palliflex T60A20) used during Phase C of Open Burning/Open Detonation (OB/OD) at Dugway Proving Grounds (DPG), Utah.

SECTION 2. General Summary

2.1. Pre and post test filter weights are carried out to determine net weight gain after cloud sampling with the instrumented aircraft. Selected clean and loaded filters are also examined to determine weight gain or loss with time under controlled equilibration conditions.

2.2. Filters are used in sets of five for each flight. Three filters are used for sample (smoke or background) collection with the remaining **two serving** as field or control blanks. The initial weights of clean filters are determined no more than 36 hours before they are to be used for sampling. When not installed on the aircraft sampling manifold, all filters are stored in the weigh room. All filter sets to be used for sampling are weighed within 2 hours of the completion of the sampling flight. All filters are taken to the weigh room in the filter holders within 1/2 hour of the time the Sandia National Labs (SNL) aircraft lands at Michael Field and, after removal from the holders, are weighed immediately.

SECTION 3. Filter Type and Description

Palliflex T60A20, 25 X 20 cm, Teflon coated, glass fiber filters are used for the duration of Phase C, OB/OD tests.

SECTION 4. Equipment and Facilities

4.1. The following is provided by Sandia National Labs:

4.1.1. Mettler Balance, Model AE240 (Digital)

4.1.2. Air Pollution Filter Chamber for Balance

4.1.3. Balance Table (massive)

4.1.4. Hygrothermograph (temperature/relative humidity recorder) Operator

4.2. The following is provided by DPG/Lockheed:

4.2.1. Space in controlled environment room

4.2.2. Filter storage shelves

4.2.3. Small computer table

4.2.4. Air conditioner/heater unit

4.2.5. Dehumidifier

4.2.6. Air filtration unit

SECTION 5. Balance Calibration

The balance is calibrated with the internal weight at the beginning of each weighing session. A session is defined as any set of weight measurements that begins more than one hour after the last balance calibration.

SECTION 6. Reference Weights

A National Institutes of Standards and Technology (NIST) certified reference weight (2 grams) is to be weighed before and after each set of five filters. Reference weight observations are made to ensure that the balance is operating within the established range. Reference weight observations are acceptable if they are within plus or minus 1 mg of the expected value. The balance is tared (zeroed) before every weighing if the readout does not return to a 0.000 gram reading. A weighing consists of a zero observation and a stable weight reading of at least 10 seconds. If the balance does not stabilize or shows continual drift, the filter is to be repositioned in the balance, as occasionally a filter may unfold and touch a surrounding component of the balance causing an unstable reading.

SECTION 7. Filter Number Assignment

7.1. Each set of filters are assigned ELI numbers ending in I through 5 or 6 through 0. In the following procedure. 6 is equivalent to 1, 7 to 2, etc. There are 10 possible combinations of assignments of the two field blanks from the five filters in a set. The combinations are listed and assigned a number from I to 10 (0). Random digits are selected from a random number table until each number from O to 9 has been placed in the list. This list defines the order of selection of the field blank assignments. The sequence is repeated until all Phase C samples are collected. The assignments are:

Table 1 - Filter Number Assignment.

FILTER SET	FILTER BLANKS	FILTER SET	FILTER BLANK
1 - (11, 21)	1 & 4	6 - (16, 26)	3 & 4
2 - (12, 22)	4 & 5	7 - (17, 27)	2 & 3
3 - (13,. 23)	2 & 5	8 - (18, 28)	1 & 2
4 - (14, 24)	3 & 5	9 - (19, 29)	1 & 5
5 -(15, 25)	2 & 4	10 - (20, 30)	1 & 3

SECTION 8. Filter Handling, Folding and Storage

8.1. Filter Handling

Nylon gloves are to be worn during all filter handling procedures. Clean filters are stored in their original cardboard carton with the top set slightly off to the side to allow the filters to equilibrate at existing room conditions. The original plastic bag enclosing the clean filters is to be removed as well as the top 2 filters and the bottom 2 filters. Clean filters are removed from the top of the filter stack in descending order and are weighed flat in the balance's lower chamber. All filters are weighed, with the side that is impacted during sampling, facing up and not touching any surface. The stainless steel filter holders and neoprene gaskets are to be cleaned using cotton tissues and isopropanol prior to the loading of weighed filters.

8.2. Filter Folding

Used filters are folded in half and weighed in the balance's lower chamber for the first post test weighing. Final folding prior to storage is accomplished by re-opening the filter, folding the two short edges of the filter to the middle, rotating the filter 90 degrees, and folding the filter in half. This folding method positions all the particle loaded faces inside the folded filter and minimizes the loss of particulate matter during subsequent filter handling and reweighing. All filters, including those destined for cold storage and those for room temperature weight equilibration study, were folded in this manner.

8.3. Filter Storage

All folded filters are placed in a foil pouch following weighing. The pouch is constructed from a sheet of heavy duty aluminum foil folded in half, with the sides folded over approximately 1 cm to keep the filter from sliding around and to capture any particulate material that may inadvertently escape from the filter while inside the pouch. The foil pouch in turn is to be placed inside a manila envelope and placed on a filter storage shelf if the filter is selected for room temperature equilibration. During equilibration, both the foil pouch and manila envelope are propped open by an approximate 4 cm diameter aluminum foil ball to facilitate filter contact with the weigh room atmosphere. During loaded filter reweighing, the foil pouch is to be carefully checked for any material that might have escaped from the filter. Any material found in the foil folder is to be transferred on to the filter before weighing.

SECTION 9. Filter Equilibration

9.1. Control Filters

Three control filters are to be selected to monitor the environment and its effect on the filters. Control filters are to be weighed at least once a day. Each control filter is to be stored in a foil pouch, placed in a manila envelope, and both folder and envelope propped open with an aluminum foil ball. Control filters are not to be folded at any time.

9.2. Clean Filters

Clean filters are taken directly from the storage box that is left uncovered to allow filter contact the with weigh room atmosphere.

9.3. Used Filters

After the first post-sampling weighing of each filter set, one sample collection filter and one field blank filter are selected at random for further weight equilibrium study. The two sample filters are folded and placed in cold storage (dry ice or freezer). The field blank selected for cold storage is not folded prior to storage. The remaining sample filter (folded) and field blank filter (unfolded) are held at room conditions and periodically reweighed to determine how much, if any, weight is lost before the filters come to equilibrium with the weigh room atmosphere. After equilibration conditions are met, the two filters are to be put in cold storage. Equilibration (weight stability) of these filters is defined here as three successive filter weight observations that are within a 5 mg range. There must be at least an hour interval between any successive reweighing. If the first reweighing of the equilibration series is the first post-test determination, there must be at least a 1 2-hour interval between that weighing and the second of the three weighing.

SECTION 10. Data Recording

All weight data is read directly from the digital display of the balance when the "stable weight" indicator is lit and recorded onto preprinted forms. At the completion of the balance session, the forms are then taped into the weight notebook. Such additional information as date, time, relative humidity, temperature and balance operator are also recorded during each balance session. Completed hygrothermograph traces are to be inserted in the notebook as well. Additionally, an ELI collection report is to be filled out for each filter. The ELI sample number is to be written on the outside of the manila envelope in which the filter is stored. The original copy of the ELI collection report is clipped to the manila envelope and placed in cold storage with the filter. All filters are to be referenced by their respective ELI numbers.

SECTION 11. Filter Cutting Procedures for XRF Analysis

11.1. Processing Location

The filter cleaning area on the opposite side of the weigh room (from the balance) is to be used for the cutting operation.

11.2. Filter Handling and Cutting

Nylon gloves are to be worn during the procedure. The filters are removed from cold storage, unfolded, and placed on a isopropanol cleaned bench top surface with the loaded side facing upward. An approximate 50 X 100 mm section from the center of the filter is to be cut out with a razor blade.

11.3. Filter Folding

The cut out section is then single folded with particle loaded sides facing inward and placed in an aluminum foil pouch. The foil pouch is then placed in a manila envelope and labeled with an ELI number. The sample is then released to ELI personnel after the appropriate split sample collection reports are completed. The remaining filter section is refolded such that all particle loaded are not exposed. The remaining filter section is placed back in the aluminum pouch and manila envelope and returned to cold storage.

SECTION 12. WEIGH ROOM ENVIRONMENT ACCEPTABILITY CRITERIA

The temperature and relative humidity are to be observed and control points reset if the temperature and relative humidity are out of tolerance. A temperature is deemed acceptable if it is within the range of 67 to 74 degrees F. Similarly, relative humidity is acceptable if it is within the range of 25 to 55 percent.

13. Balance Performance Acceptability Criteria

The 2.000 gram reference weight is to be used on a daily basis to check the balance for stability and acceptable performance. The acceptance criteria for proper balance performance is: (1) A measurement of the reference weight that is within plus or minus 1 mg of the calibration value of the weight; and, (2) Balance stability (zero drift with draft shields closed) no more than plus or minus 1 mg with nothing on the weigh pan.

SECTION 7 - Burn Trials Processing Procedures (LESC)

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SECTION 1. Sputter Pan Procedure

1.1. Weigh each sample and record weight in the log book.

1.2. Composite all 10 sputter pan samples into one 500 ml sample bottle.

1.3. Weigh composite and record weight on bottle label and in log book.

1.4. Mark bottle label as a composite of appropriate position numbers.

1.5. Complete a new ELI form and record new ELI number in log book and on bottle label.

1.6. Place in freezer until shipped.

SECTION 2. Fallout Pans

2.1. Weigh each sample and record weight in log book.

2.2. Composite all 4 Fallout Pan Samples from the 6 meter ring into one 500 ml bottle.

2.3. Complete steps 1.c- f above.

2.4. Composite all 6 Fallout Pan Samples from the 12 meter ring into one 500 ml bottle.

2.5. Complete steps 1.c through f. above.

SECTION 3. Burn Pan Residue

3.1. Weigh each container of residue and record weights in log book

3.2. Composite all 3 containers into one

3.3. Label container as a composite of the appropriate locations and record weight on the label

3.4. Mix by shaking container for 30 seconds. e Let stand 3-5 minutes for dust to settle.

3.5. Remove 1 liter bottle of residue and send to CHEM TECH, Murray, Utah for hazardous waste determination.

3.6. Store in O/D Chem Lab until disposal.

SECTION 8 - Pretest Soil Sample (LESC)

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SECTION 1. Procedure

1.1. Transfer the soil from the soil pan into a tare weighed stainless steel bucket.

1.2. Weigh to the nearest gram on an OHAUS model 119S balance and record the weight in the log book.

1.3. Pass the soil thru a sleeve column:

1.3.1. 3/8 inch (.375) mesh screen

1.3.2. #4 (.187) mesh screen

1.4. Grind any soil not passing thru the sleeve column in a retch grinder.

1.5. Mix (homogenize) soil for 2 minutes in a hobart 12 quart mixer.

1.6. Split the soil sample

1.6.1. Split #1

1.6.1.1. Place 500 ± 5 grams of soil into a properly labeled sample bottle with a teflon lined lid, provided by AWL.

1.6.1.2. Record weight of soil on bottle label and in log book.

1.6.1.3. Place 200 ± 5 grams of soil into a properly labeled sample bottle with a teflon lined lid, provided by AWL.

1.6.1.4. Record weight of soil on bottle label and in log book.

1.6.1.5. Fill one properly labeled 1 liter sample bottle with soil. The bottle will be closed with a teflon lined lid. Both bottle and lid will be provided by AWL.

1.6.1.6. Record weight of soil on bottle label and in log book.

1.6.1.7. Complete a new ELI form for the 500 gram and 200 gram samples taken from split #1. The original ELI form will remain with the archive sample.

1.6.1.8. Record the ELI number on the proper bottle labels and in the log book.

1.6.1.9. Discard any remaining portion of split #1.

1.6.2. Hold until split #I has been processed. If sufficient soil samples are obtained from split #1, split #2 will be discarded.

1.6.3. If sufficient soil samples are not obtained form split #1, the difference will be made up from split #2 and **then any** remaining soil will be discarded.

1.7. Place samples into freezer until shipped.

SECTION 9 - Carbon Aerosol Analysis (SSL)

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SECTION 6. Carbon Aerosol Analysis

6.1. Carbon can exist in atmospheric aerosols primarily in three separate classifications:

6.1.1. As organic compounds,

6.1.2. As elemental ("soot") particles, or, more rarely,

6.1.3. As inorganic carbonate.

6.2. The ability to analyze the aerosol for carbon and properly speciate it into one of the above can be important for assigning sources of the aerosol.

6.3. The thermal-optical method of analysis as done at Sunset Laboratory was designed to properly speciate the carbon. This is done by optically correcting for pyrolytically produced elemental carbon from organics during the first part of the analysis. The presence of inorganic carbonate can be determined by accurate control of the thermal separation process.

6.4. The current specifications for this analysis are as follows:

6.4.1. Detection limit:

6.4.1.1. Organic Carbon - $\pm 0.3\ \mu g\ C/cm^2$

6.4.1.2. Elemental Carbon - $\pm 0.3\ \mu g\ C/cm^2$

6.4.2. Precision - ± 5 percent

6.4.3. Accuracy - ± 5 percent

6.4.4. The value for accuracy in speciation has been determined from analyzing model samples as well as comparison with other methods and combinations of other methods with this method.

6.4.5. The quality assurance procedure utilized at Sunset Laboratory was developed to insure the above criteria as much as possible. Samples received are stored in cold storage until analyzed as well as transported and archived under cool conditions.

6.5. To assure proper accuracy in quantification of carbon as well as proper speciation the following procedures are followed:

6.5.1. Each sample has an internal quantitating standard incorporated during the analysis. This serves as the value to quantitate the sample against by compensating for any variables such as gas flows, assuring that carbon is properly measured. Also, this serves as an indicator of problems which may arise such as sensitivity drops due to instrument malfunctions.

6.5.2. The internal standard consists of a sample of helium/methane gas of known mixture contained within a known volume sampling loop which is switched in-line with the analyzer ovens at the end of each analysis. Typically the mixture is about 5 percent methane (currently 5.26%) and the sample loop is l.O0 ml. The injected methane follows the same path as the sample, first being oxidized to carbon dioxide in the oxidizing oven and finally reduced to methane again in the methanator oven before being measured at the flame ionization detector.

6.5.3. External calibration standards are periodically run. These consist of known volumes of known concentrations of various aqueous organic compounds, such as potassium hydrogen phthalate, sucrose, or lactose. This serves as a check Or the accuracy of the quantification of carbon.

6.5.4. Because of the charring capability of sucrose it is currently used for most of these external standard analyses. Single-point checks are done at a frequency of about once for every 30 samples, while a multi-point calibration consisting of three different values is done at a frequency of about once very 100 samples. If any of the values are outside the precision range of ±5 percent, no samples are analyzed until corrective action has been taken.

6.5.5. Model compounds are analyzed which consist of various mixtures of organic and elemental carbons which are produced under conditions where at least one of the concentrations is known. This serves as a check on the ability to properly speciate the carbon into the organic and elemental fractions. This is only done on special occasions, such as during inter-laboratory comparison studies, when requested by certain contracting agencies, or at the beginning of important studies.

6.6. Another check which is done at a frequency of once for every 100 samples is analyzing a sample which has gone through a solvent extraction to remove most of the organic carbon. This process removes a large percent of the charring organics while usually not removing any of the elemental carbon particles. Thus, any interferences to proper speciation due to charring are minimized, allowing for what is hoped to be a more accurate value for elemental carbon

6.7. Duplicate analyses are done on approximately 10 percent of the samples. This is done as a measure of consistency in speciation of the organic/elemental fractions as well as to determine reproducibility of total quantity of carbon.

6.8. Instrument blanks are done at about the rate of one for every 30 samples. This serves the purpose of a "zero-chec", and is done in order to make sure detection limits are of the proper low magnitude.

6.9. Blind samples are submitted for analysis to serve as a crosscheck on precision and are done on a schedule determined by the submitting agency.

6.10. Filter blanks shall be analyzed in order to determine values which may be subtracted from the samples. These may be either static-field blanks (those taken to the field, but kept in the containers) or dynamic-field blanks (those placed in the sampling instruments, but not having ambient air drawn through them). These shall be sent at a frequency determined by the submitting agency.

6.11. Values for all quality assurance checks are to be submitted with each report for each sample set. This will include duplicate analyses, qualitative standards and instrument blanks.

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SECTION 10 - Aircraft Sampling Procedures (SNL)

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AIRCRAFT SAMPLING PROCEDURES

SECTION 1. Purpose

This document outlines all aspects of the gas and aerosol instrument calibration and sampling procedures that will be carried out as a part of the OBOD field test program at Dugway Proving Ground. All aspects of instrument setup, calibration, background and plume sampling are covered in this procedure.

SECTION 2. Test Description

A series of tests will be carried out at DPG that will involve the burning of propellant and detonation of explosive material. The SNL-instrumented aircraft will be used to penetrate the detonation or combustion cloud in order to collect both gaseous and aerosol samples such that a complete characterization of the plume can be done.

SECTION 3. Real-Time Instrument Calibration

3.1. NO_x monitor

Separate multi-point calibrations will be done on the NO and NO_x channels of the instrument using an NBS-traceable, EPA-certified standard NO test gas at the start and completion of the test series. This gas will be diluted to the working range of the instrument using a gas dilution system that includes mass flow controllers and a zero air supply. Five gas concentration levels will be generated and sampled with the instrument with the voltage response of the instrument determined for each concentration level. The sample gas will be delivered to the instrument through a teflon in-line filter installed on the instrument inlet. The conversion factors for the determination of the concentration levels from voltage output from the instrument will be determined by linear regression of the input concentration of the test gas against the measured voltage output for all test concentrations generated. The calibration response factors will be considered valid if the correlation coefficient (r^2) of the regression is greater than 0.98. All calibration data will be recorded in the instrument logbook.

Daily checks on instrument performance will be carried out using a zero gas and a NO span gas with a concentration in the normal working range of the instrument. In the event that the recorded instrument output during a span gas check deviates from the expected output by more than (+/-)

15 percent, the instrument will be checked and the problem remedied. Following any significant repairs on the instrument, a multipoint calibration will be carried out prior to placement of the instrument back in service. Results from all daily zero and span checks will be recorded in the instrument logbook.

3.2. CO Instrument

Multipoint calibrations comparable to those for the NO_x instrument will be performed on the CO instrument using a certified CO test gas and a gas dilution system.

Daily zero and span checks will be carried out using a zero gas source and a working range CO span gas. A (+/-) 15 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

3.3. CO_2 instrument

Multipoint calibrations comparable to those for the NO_x instrument will be performed on the CO_2 instrument using a certified CO_2 test gas and a gas dilution system.

Daily zero and span checks will be carried out using a zero gas source and a working range CO_2 span gas. A (+/-) 15 percent accuracy criteria will be applied to the span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

3.4. O_3 instrument

A multipoint calibration of the O_3 instrument will be conducted prior to the test series using a certified ozone transfer standard. The calibration gas will be passed through an in-line teflon filter connected to the inlet of the instrument.

Daily zero checks will be carried out on the instrument prior to each test. Due to the unavailability of a calibrated ozone source, daily span checks on this instrument will not be carried out. Results from all daily zero and span checks will be recorded in the instrument logbook.

3.5. Nephelometer

The integrating nephelometer will be calibrated with Freon-12 2 days prior to the start of the test series using Freon-12 response factors published in the literature. Daily zero checks will be carried out by filling the optical chamber of the instrument with particle-free air. Span checks will be performed using the built-in electronic span check feature included in the instrument. A (+/-) 15 percent accuracy criteria will be applied to the electronic span check results to determine acceptable instrument performance on a daily basis. Results from all daily zero and span checks will be recorded in the instrument logbook.

3.6. Aerosol probes (ASASP-100X and FSSP-100X)

As a result of the complex optical and electronic design of the SNL aerosol probes, they are sent to Droplet Technologies Inc. in Boulder, CO, which is a facility that specializes in the maintenance and repair of laser aerosol spectrometers. Here the probes are thoroughly checked out and evaluated by routine test procedures. A complete description of probe response is obtained form this firm following completion of the calibration. This information on probe performance under controlled laboratory conditions is used to determine probe response factors to be used in processing the field-collected data. Probe calibrations will be completed four weeks prior to the start of the test series. Calibration certificates detailing this periodic activity will kept on file.

A system check of each probe's performance will be carried out prior to the start of the test series using certified polystyrene latex particles of known size in an aerosol generation system. These tests will provide a quick measure of the overall performance of the probe. The probe response and performance will be judged acceptable if the probe classifies the test particles within two channels of the expected response channel.

SECTION 4. Aerosol Sampling System Calibration

4.1. Post-filter air flow meters

The pose-filter air flow meters will be used for qualitative flow indication only and will not be calibrated.

4.2. Transport pitot tube pressure sensor

A variable reluctance pressure transducer is used to measure the velocity pressure as measured by the pitot tube mounted on the centerline of the transport tube inside the aircraft. The voltage output of the transducer is measured at several points over its working range and an appropriate response factor determined by linear regression of the output voltages against the pressure inputs. Input pressure during these calibration tests is measured with an inclined manometer reserved exclusively for this use. Calibration of the pressure transducer will be completed one week prior to the start of the test series and the results recorded in the experiment logbook. Additional details concerning the calculation of response factors and sample volumes from the pitot tube sensor are given in Enclosure 1.

SECTION 5. Miscellaneous Equipment Calibration

5.1. Altitude pressure sensor

The piezo-ceramic pressure transducer used to measure pressure altitude during flight is on an annual calibration schedule at the SNL standard laboratory. The output of the sensor is characterized over its working range and an appropriate response factor determined by linear regression of the measured voltage output against the known pressure inputs. The calibration response factors will be considered acceptable if the correlation coefficient from the linear regression is in excess of 0.98.

5.2. Aircraft velocity pressure sensor

The piezo-ceramic pressure transducer used to measure velocity pressure at the external aircraft pitot tube during flight is on an annual calibration schedule at the SNL standards laboratory. The output of the sensor is characterized over its working range and an appropriate response factor determined by linear regression of the measured voltage output against the known pressure inputs. Regression results will be considered acceptable if the regression correlation coefficient is in excess of 0.98.

5.3. Temperature and dew point temperature sensors

The temperature and dew point sensors are on an annual calibration schedule at the SNL standards laboratory and are periodically checked against reference temperature and dew point temperature conditions. Response factors are changed to incorporate the results of this periodic check of sensor performance as necessary.

5.4. Data acquisition system (DAS) calibration

The performance of the data acquisition system will be checked against a voltage standard annually certified by the SNL standard laboratory one month prior to the onset of the test. The analog to digital (A/D) conversion unit in the data acquisition system will be checked at three points: approximately 10, 50, and 90 percent of the expected full scale voltage range of each data channel. Total system integrity will be checked by verification of proper voltage signals on the data files written by the system. Data acquisition system performance will be considered acceptable if voltmeter response is (+/-) 2 percent of the expected voltage check values.

SECTION 6. Filter Handling Procedures

6.1. Filter loading

Prior to loading the filters for a sampling mission, the filter housings will be cleaned with reagent grade isopropanol and tissue. Personnel loading the filters will wear clean cotton gloves to minimize handling contamination. The filters will be removed from their foil envelopes with stainless steel tweezers and placed on the backup screen. An isopropanol-wiped foam gasket is then placed on top of the filter, followed by the top half of the filter housing. The lock nuts are then snugged down such that good contact between the gasket and the two halves of the filter holders results. The filter number and filter unit position (forward, right, or left) are then recorded in the experiment logbook. Filter units are then installed on the aircraft manifold in the appropriate positions. The wires from the airflow sensors are plugged into their appropriate positions on the junction box in the rear compartment.

6.2. Filter unloading

The above sequence is followed in reverse order for filter unloading. Following removal of the top half of the filter unit, the filter is folded in half and then in half again in order to minimize the loss of particulate material from the surface of the filter. After the folded filter is inserted in its foil envelope, it is immediately placed in cold (dry ice) storage and held there until it is released to the analytical laboratory.

SECTION 7. General Sampling Description

7.1. Background sampling procedures

Background aerosol and gas samples will be collected during the initial flight of each test day. After take-off, the aircraft will be positioned in the vicinity of the test grid at an altitude of 1,000 feet above ground level (AGL) where a background aerosol sample will be collected over a period of 15 minutes. After zero and span checks, ambient level gas measurements will be made directly from the transport tube with the continuous monitors. The 80 l bag will also be filled with ambient air and sampled with the continuous monitors. Two 6 L canisters will also be filled during the background flight: the first directly from the tube, and a second from the teflon bag after it has been filled with ambient air. Following the collection of the background samples, the aircraft will land at Michael Army Air Field (MAAF) where the filters will be unloaded and stored and the aircraft immediately re-fueled for the next flight.

7.2. Plume sampling procedures

After clean filters have been loaded on the sampling manifold, the aircraft will take off and be positioned at 1,000 ft AGL in the vicinity of the test grid. Prior to plume sampling, zero and span checks will be make on the continuous monitors. The aircraft will then be positioned such that it can penetrate the detonation of combustion plume 2 minutes after ignition and the notice to detonate will be given to the ground crew. The aircraft will make its first plume penetration during which aerosol samples will be collected and an aliquot gas sample collected in the 80 l bag. A 6 L canister sample will also be collected over a 3-4 second interval directly from the transport tube as the aircraft transects the plume. A second and third plume penetration will be carried out in a similar manner. After three plume penetrations, the gas instrument inlet will be switched from the transport tube to the 80 l bag in order to measure the composite sample gas concentration from the plume penetrations. A 6 L canister sample will also be collected from the bag at this time. If the test involves a series of detonations, the 80 l bag will be emptied completely and the aircraft will again be positioned for plume penetration of the next detonation. Following completion of all plume penetrations and measurements of the composite gas samples, zero and span checks will be done on the continuous monitors. The aircraft will then return to MAAF where the filters will be unloaded and stored under dry ice conditions.

SECTION 8. Background Sampling Checklist

8.1. Pre-flight

Solvent-wash aircraft probe and transport tube with isopropanol
Check in-line gas sampling instrument filters
Warm up all continuous monitors using ground power unit
Check for proper range settings on all instruments
Load filters onto sampling manifold
Empty gas sampling bag

Check PMS set-up parameters and probe AC power
Load 6 L canisters
Load backup software for PMS and analog DAS
Load formatted 720KB and 1.2 MB backup disks

8.2. In-flight

Start analog DAS
Start PMS DAS
Start blowers to collect aerosol sample
Switch main valve to sample position
Check zero on all instruments for 3-5 min
Check span on all instruments for 3-5 min
Sample from transport tube with gas instruments for 5 min
Fill 6 L canister from transport tube; label and record in logbook
Fill gas sampling bag
Sample from bag with gas instruments for 5 min
Fill 6 L canister from sample bag; label and record in logbook
Close main sample valve and stop blowers

8.3. Post-flight

Remove filter units, unload filters, and store
Backup data files from analog and PMS DAS
Remove 6 L canisters and store

SECTION 9. Plume Sampling Checklist

9.1. Pre-flight

Warm up all continuous monitors using ground power unit
Check for proper range settings on all instruments
Load filters onto sampling manifold
Empty gas sampling bag
Check PMS set-up parameters
Load 6 L canisters
Load video tape and check video camera lens
Load still camera with data back, lenses, and film

9.2. In-flight (standing by)

Start Analog DAS (low rate)
Start PMS DAS (PMS on HOLD)
Start video system
Check zero on all instruments for 3-5 min
Check span on all instruments for 3-5 min
Flush bag at least two times and empty
Switch gas instruments to transport tube

Check for proper range setting on nephelometer and RAM-1
Install 6 L canister on transport tube sample port
Start new data file on analog DAS 5 minutes before shot time, t_0

9.3. In-flight (plume sample)

Approaching plume:

Switch analog data system to high rate 30 sec before plume entry
Place PMS system on SAMPLE 30 sec before plume entry

Enter plume:

Collect 6 L canister - label and record in logbook
Start blowers - Open main valve and bag valve

Exit plume:

Close main valve and bag valve - Stop blowers
Switch analog DAS to low rate
Place PMS system on HOLD

9.4. In-flight (after plume samples)

Stop video system
Switch gas instruments to bag and sample for at least 5 minutes
Collect 6 L canister sample from bag; label and record in logbook
Check zero on gas instruments for 3-5 min
Check span on gas instruments for 3-5 min
Close data file on analog an PMS DAS after gas readings complete

9.5. Post-flight

Remove filter units, unload filters, and store
Remove 6 L canisters and store
Backup data files from analog and PMS DAS
Remove, label and secure video tape

SECTION 10. Sample Analysis

10.1. Quartz fiber filters

The quartz fiber filters taken during background and plume sampling will be submitted to AWL for further analysis. The filters will first be weighed to determine aerosol weight collected. A 1- inch punch will be taken from the center of each filter and sent to SSL for carbon aerosol analysis. Solvent extraction will then be carried out on the filters. The extract will be analyzed by GC-MS and SFC-MS for trace level organics. A portion of the solvent extract and the filters will then be submitted to the PIXE lab at BYU for metals determination. A dilute nitric acid extraction will be done on the filters and a separate metals determination on both the filters and the solvent extract will be completed. Detailed analytical procedures are given for both laboratories in their respective Letters of Instruction.

10.2. 6 L Canisters

All 6 L canisters collected during the test will be submitted to the Oregon Graduate Center for analysis of CO, CO_2, H_2 and C_1 - C_{10} hydrocarbon species by gas chromatography. Complete details of this procedure are given in the OGC LOI.

ENCLOSURE 1

SECTION 1. Introduction

This enclosure describes the calibration and measurement methods used to determine the volume of air passing through each filter during sample collection.

SECTION 2. Transport Tube

Flow through the transport tube will be monitored continuously using a pitot tube and a variable reluctance pressure transducer. The pitot tube is positioned on the axis of the 3 3/4 inch diameter (internal) transport tube approximately 3 feet downstream of the probes supplying the nephelometer and OGC gas sampler and 9 inches upstream of the probe for the Teflon bag gas sampler. The flow in the transport tube is very turbulent (Reynolds number 50,000 or more) and we therefore assume the velocity profile in the tube is flat. The actual velocity at the pitot tube is calculated form the equation:

$$V = 1096\times\left[\frac{VP}{0.075F}\right]^{1/2}$$

where V = actual velocity, feet per minute; P = pitot velocity pressure, inches of water; 0.075 = air density, pounds per ft^3, at standard conditions; and F = density correction factor as given by:

$$F = \frac{amb\ press\ (mmHg)}{760} x \frac{298}{273.1 + amb\ temp\ (^\circ C)}$$

Because of the rapid transit through the transport tube, the ambient conditions measured outside the cabin are used in this calculation. The actual volumetric flow is calculated from the tube cross sectional area (0.0767 ft^2) and the actual V. The volumetric flow at standard conditions is obtained by multiplying the actual flow by the density correction factor. After combining all necessary conversion factors, the final equation becomes:

$$Q = 90.71\ \left[VP \times \frac{amb\ press\ (mmHg)}{273.1 + amb\ temp\ (^\circ C)} \right]^{1/2}$$

where Q is in standard liters per second. The pressure transducer is calibrated against an inclined manometer over the 0 - 2 inches water gauge (in WG) range used for the measurements. The offset and span controls are adjusted to provide numerical equivalency between output volts and inches of water.

SECTION 3. Hot Wire Anemometers

Flows through the three filters connected directly to the transport tube are measured using hot wire anemometers mounted at the filter housing exits. The amplified voltage output from the sensors is recorded and then converted to flow rate during data analysis.

The anemometers are calibrated in place in the filter housings over the anticipated flow rate range using a calibration system consisting of a 24-inch length of 1 1/2-inch diameter (internal) tubing and a miniature pitot tube with its tip located approximately 18 inches from the tube entrance. Air is drawn through the calibration system and then the filter holder with a HiVol sampler controlled with a Variac. the pitot velocity pressure is measured with the same transducer used for the transport tube measurements. The pitot and hot wire signals are recorded every 2 seconds after stable flow has been established at each calibration condition. The instantaneous standard flow rates are calculated using the tube area (0.0123 ft^2) and the same assumptions as for the transport tube. The equation, using the same units as for the transport tube is:

$$Q = 14.55\left[VP\,\frac{amb\ press\ (mmHg)}{273.1 + amb\ temp\ (^{\circ}C)}\right]^{1/2}$$

Polynomial regression equations are calculated for each set of paired flow rate and hot wire voltage data using nonlinear least squares software (TechGraphPad). A cubic equation is used unless an acceptable correlation ($r^2 > 0.99$) is obtained with a quadratic fit to the data.

SECTION 4. Flow Distribution Calculation

The flow through each of the filters is expected to be 1/3 of the total flow through the transport tube so long as all three filters are identical. The three hot wire anemometers permit verification of this and allow for apportioning the flow when different types of filters are used together. However, the hot wire flow values cannot be used directly because their response time for abrupt changes (2 - 3 seconds) is a significant part of the sample collection time (5 - 8 seconds). Therefore, the transport pitot tube will be used to determine the total volume of air that passes through the filters. The volume will be apportioned among the filters based on the last few seconds of the sample when the data indicate the post-filter flowmeter readings have stabilized.

SECTION 11 - VOC Collection Analysis System (OGC)

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Oregon Graduate Center
LOI - 1
REVISION NUMBER: 0
1 JUNE 1989

VOC Collection Analysis System
Adapted from EPA Compendium Method TO-14
R. A. Rasmussen

A. CANISTERS

1.1 Canister Construction

1.1.1 Type 304 stainless steel with 20 gage wall thickness used to fabricate stainless steel air sampling canisters. All welded seams welded metal-to-metal (no welding rod used) under Argon shield for T.I.G. weld, 100% penetration.

1.1.2 Sample sizes available: 850mL, 3.2L, 6L, 15L, and 32L. For simultaneous paired air sampling 3.2-L canisters are used.

1.1.3 All internal surfaces are electropolished using Molectrics, Inc. (Carson, CA), Type Power Kleen™ solution, commonly referred to as the SUMMAR process. This treatment leaves an enriched surface layer of chrome-nickel oxide that is designated as "passivated." After electropolishing, said surfaces are thoroughly washed with DI water and cleaned of any residual organics by a vacuum bake-out at 120 °C to 100 mTorr for several hours.

1.1.4 Valve configurations are typically a single Nupro™ SS-4H4 bellows stem valve. To ensure leak-tight construction to air sampling manifolds, the valves are fitted with Cajon™ VCRR 1/4-inch male connectors. These fittings use an expendable metal disc to provide vacuum-tight connections. Swagelok™ fittings are not acceptable for multiple use leaktight vacuum assemblies.

1.2 Canister Integrity

1.2.1 All canisters are hydrostatically tested to 20 Atm. (300 psig).

1.2.2 Recommended operational pressure range is -30 inches Hg to 2 Atm. (30 psig).

1.2.3 All containers are helium leak-tested to 1×10^{-9} cc/sec.

1.3 Canister Cleaning System

1.3.1 Vacuum pump (Alcatel, Hingham, MA, Model M2008A). Capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg (50 mTorr).

1.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning two-four canisters.

1.3.3 Shut-off valve(s) - five on-off Nupro™ SS4H4 valves.

1.3.4 Stainless steel vacuum gage (Varian, Palo Alto, CA, Model VH3) capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.

1.3.5 Cryogenic trap (2 required) - all glass, standard mechanical roughing pump type open tubular trap cooled with liquid nitrogen (-196 °C) to prevent contamination from back diffusion of oil from vacuum pump.

1.3.6 Stainless steel pressure gauges (2) (Span Instruments, Plano, TX) all SS, 0-345 kPa (0-50) psig) to monitor zero air pressure.

1.3.7 Stainless steel flow control valve, Nupro™ SS-4H4 - to regulate flow of zero air into canister(s).

1.3.8 Humidifier - pressurizable glass water bubbler containing high performance liquid chromatography (HPLC) grade deionized water or other system capable of providing moisture to the zero air supply.

1.3.9 Isothermal oven for heating canisters (0-150 °C), 16 x 24-inch I.D., special laboratory construction. Note: Very important that Nupro™ SS-4H4 valves remain outside of oven during bake-out procedure.

1.4 Calibration System and Manifold.

1.4.1 Calibration manifold - SS manifold, SS tubing internally passivated or electropolished. Sampling ports and internal baffles for flow disturbance to ensure proper mixing. System is designed to handle pressures up to 350 psig. Two-stage high purity regulators (Veriflow, Richmond, CT, Model IR501B-4-SSR) are used to reduce pressures from EPA audit cylinders.

1.4.2 Humidifier - 500-mL impinger flask containing HPLC grade deionized water.

1.4.3 Electronic mass flow controllers - ranges 0 to 5 L/min, 0 to 50 cm^3/min (Tylan Corporation, Carson, CA, Model FC260, or equivalent).

1.4.4 Telfon™ or SS in-line filter(s) for particulate control - 47-mm Teflon™ or 10-m-nominal SS discs, best source.

2. Standards, Reagents, and Materials

2.1 Gas cylinders of helium, hydrogen, nitrogen, argon/methane (5/95%), and zero air - ultrahigh purity grade, best source.

2.2 Gas calibration standards - cylinder(s) containing the following compounds of interest are available:

vinyl chloride
vinylidene chloride
1,1,2-trichloro-1,2,2-trifluoroethane
chloroform
1,2-dichloroethane
benzene
toluene
FreonR 12
methyl chloride
1,2-dichloro-1,1,2,2-tetrafluoroethane
methyl bromide
ethyl chloride
FreonR 11
dichloromethane
1,1-dichloroethane
cis-1,2-dichloroethylene
1,2-dichloropropane
1,1,2-trichloroethane
1,2-dibromoethane
tetrachloroethylene
chlorobenzene
benzyl chloride
hexachloro-1,3-butadiene
methyl chloroform
carbon tetrachloride
trichloroethylene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
o-xylene
m-xylene
p-xylene
styrene
1,1,2,2-tetrachloroethane
1,3,5-trimethylbenzene
1,2,4-trimethylbenzene
m-dichlorobenzene
o-dichlorobenzene
p-dichlorobenzene
1,2,4-trichlorobenzene

2.3 Primary reference standards are traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to an NBS/EPA-approved Certified Reference Material (CRM). For hydrocarbon speciation we use two different NBS SRM's: Benzene in N2, #1805 at 0.25 ppm; Propane in N2, #1665b at 3 ppm; Methane in Air, #1658a at 1 ppm and #1659 at 10 ppm. For carbon monoxide we use NBS SRM CO in Air #2612 at 10 ppm.

2.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders and system inlet line, to remove moisture and organic impurities from gas streams (built to own specifications using mol-sieve).

2.5 Deionized water - high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.

2.6 Hexane - for cleaning sampling system components, reagent grade, best source.

2.7 Methanol - for cleaning sampling system components, reagent grade, best source.

3. Sampling System

3.1 System Description

3.1.1 Pressurized Sampling (with Teflon™ diaphragm-type pump).

3.1.1.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control device to achieve a typical 10-30 psig final canister pressure.

3.1.1.2 In pressurized canister sampling the pump (Model FC1121, BRC, Hillsboro, OR) draws in ambient air from the sampling manifold to fill and pressurize the sample canister.

3.1.2 All Samplers

3.1.2.1 A flow control device (Veriflow, Richmond, CA, Model SS-423 OGC) is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled to about one atmosphere above ambient pressure over the desired sample period. For example, if a pair of 3.2L canisters are to be filled to 2 atmospheres (15 psig) absolute pressure in 3 hours, the flow rate can be calculated by:

Equation (5) Flow Rate Calculation

$$F = \frac{2 \times 6400}{3 \times 60} = \frac{71cm^2}{\mathrm{min}}$$

3.1.2.2 For automatic operation, the timer (Chrontrol, San Diego, CA, Model CD-4) is wired to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and close the valve when stopping the pump.

3.1.2.3 The use of the Skinner (Columbus, OH) Magnelatch™ (Model VTR 1211) valve avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton™ valve seat material. The Magnelatch™ valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods.

3.1.2.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period.

3.1.2.5 Prior to field use, each sampling system has passed a humid zero air certification (see TO-14, Section 12.2.2). All plumbing has been checked carefully for leaks. The canisters also have passed a humid zero air certification before use (see EPA-TO-14, Section 12.1).

3.2 Sampling Procedure

3.2.1 The sample canisters have been cleaned and tested according to the procedure in EPA-TO-14, Section 12.1.

3.2.2 The sample collection system is assembled and has met certification requirements as outlined in EPA-TO-14, Section 12.2.3. [Note: The sampling system should be contained in an appropriate enclosure if placed out-of-doors.]

3.2.3 To verify correct sample flow "practice" canisters are used in the sampling system. For the pump-driven system the practice canisters are not opened as the flow is measured at the outlet of the system. A mass flow meter is attached to the outlet line of the sampler and the vent. The valve is opened. The sampler pump is turned on and the reading of the mass flow meter or calibrated rotometer is compared to the flow rate specified: 71 mL/min. The valves should agree with +10%. If not, the sampler flow controller needs to be set to the desired setting.

3.2.4 The sampler is turned off. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.

3.2.5 An identification tag is attached to the canister. Canister serial number, sample number, location, date, time, and comments are recorded on the tag.

4. Analytical System

4.1 System Description

4.1.1 GC-FID System

4.1.1.1 The analytical system is composed of a gas chromatograph (HP5790, Avondale, PA) equipped with a capillary column and a flame ionization detector. In typical operation, sample air from pressurized canisters is vented to the analytical system from the canister at a flow rate of 80 cm^3/min. For analysis 500 cm^3/min of sample gas is used. Sub-ambient pressure canisters are connected directly to the inlet. The gas volume is measured via the increase in pressure in the fixed volume (4-L) vacuum flask. The sample gas stream is routed through a six-port chromatographic valve (Carle or Valco) and into the cryogenic trap. [Note: This represents a 6.2-minute sampling period at a rate of 80 cm^3/min.] The trap is 1/8-inch OD x 8 inches packed with 60-80 mesh glass beads and is cooled to -183 °C by immersion in LOX cryogen. VOCs are condensed on the trap surface while N_2, O_2, and other sample components are passed to the vacuum reservoir. After the organic compounds are concentrated, the valve is switched and the trap is heated. The re-volatilized compounds are transported by helium carrier gas at a rate of 4 cm^3/min to the head of a wide bore DB-1 capillary column (0.32 mm x 60 m). Since the column initial temperature is at -60 °C, the VOCs are cryofocussed on the head of the column. The oven temperature is programmed from -60 to 150 °C at 4 °C/min after an initial 2-minute hold. The VOCs in the carrier gas are chromatographically separated. The FID detector senses the presence of the carbon in the speciated VOCs, and the response is recorded by either a strip chart recorder or an electronic integrator.

4.1.1.2 Helium is used as the carrier gas (3 cm^3/min) to purge residual air from the trap at the end of the sampling phase and to carry the re-volatilized VOC through the wide GC column. Moisture and organic impurities are removed from the helium gas stream by a chemical purifier installed in the GC.

4.1.1.3 Gas scrubbers containing Drierite™ or silica gel and 5A molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: The purity of gas purifiers is checked prior to use.]

4.1.1.4 All lines should be kept as short as practical. All tubing used for the system should be chromatographic grade stainless steel connected with stainless steel fittings.

4.1.1.5 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates are set to obtain an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm^3/min; hydrogen, 30 cm^3/min; nitrogen, 30 cm^3/min; helium, 3 cm^3/min.

4.2 GC-FID Calibration

4.2.1 At the beginning of each day three analyses of a single point working standard (neohexane) are made. If the results fall within +2%, the average of the response is used to calibrate the successive analyses. If the values for the three initial neohexane analyses exceed +2%, a fourth and/or fifth analysis is performed. Neohexane as the daily calibration standard has been used in our laboratory for 14 years. The present values are directly traceable to our primary NBS SRM benzene and propane standards. The working standards are metered with and without humidified zero air, depending upon the analyses.

4.2.2 As an alternative, a multipoint humid static calibration (three levels plus zero humid air) is sometimes performed on the GC-FID system. During the humid static calibration analyses, three SUMMAR passivated canisters are filled each at a different concentration between 1 and 20 ppbv from the calibration manifold using a mass flow control arrangement. These calibration standards are each analyzed twice. The expected retention times are used to verify proper operation of the GC-FID system. A calibration response factor is determined for each analyte and used where appropriate. The computer calibration table is updated with this information.

4.2.3 Routine Calibration: The GC-FID system is calibrated daily with a one-point calibration. The system is calibrated either with the single point neohexane standard in a high pressure (2000 psig) tank procedure or with a low pressure (<40 psig) 6L SUMMAR passivated canister filled with humid calibration standards. After the single point calibration, the GC-FID analytical system is challenged with a humidified zero gas stream to ensure that the analytical system returns to specification (less than 0.2 ppbv of selective organics).

4.3 GC-FID System Performance Criteria

4.3.1 Humid Zero Air Certification

4.3.1.1 Before system calibration and sample analysis, the GC-FID analytical system is optimized.

4.3.1.2 Periodically the GC-FID system is challenged with humid zero air.

4.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable.

4.3.2 GC Retention Time Windows Determination.

4.3.2.1 For proper identification, the retention time windows must be established for each analyte.

4.3.2.2 To do this properly, the GC system must be within optimum operating conditions.

4.3.2.3 Three injections of a diluted auto exhaust standard containing all compounds for retention time window determination are made. [Note: The retention time window must be re-established for each analyte periodically or when drift in the R.T. is observed.]

4.3.2.4 The standard deviation of the three absolute retention times for each single component standard is calculated. The retention window is defined as the mean plus or minus three times the standard deviation of the individual retention times for each standard.

4.3.2.5 The retention time windows for each standard are determined on each GC column whenever a new GC column is installed or when major components of the GC are changed. The data are noted and retained in a notebook in the laboratory as part of user SOP and as a quality assurance check of the analytical system.

4.4 Analytical Procedures

4.4.1 Canister Receipt

4.4.1.1 The overall condition of each sample canister is observed. Each canister is expected to be received with an attached sample identification tag.

4.4.1.2 Each canister is recorded in the logbook. Noted on the identification tag are the date received and the initials of the recipient.

4.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the pressure (kPa, psig) is recorded. Final cylinder pressure is recorded on the canister sampling field data sheet.

4.5 GC-FID Analysis

4.5.1 The analytical system should be humid zero air certified and calibrated through working standards directly referenced to NBS SRMs.

4.5.2 Sixty minutes are required for each sample analysis: 15 minutes for system initialization and sample collection, 40 minutes for analysis, and 5 minutes for post-time, during which a report is printed.

4.5.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap at the end of the sampling phase and to carry the re-volatilized VOCs from the trap onto the GC column and into the FID. The hydrogen, burner air, and nitrogen flow rates are also checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system.

4.5.4 The sample canister is connected to the inlet of the GC-FID analytical system. The canister valve is opened and the canister flow is vented to flush the system prior to passing the sample through the freezeout loop into the receiving vacuum-volume reservoir. The VOCs are condensed in the trap.

4.5.5 The six-port valve is switched to the inject position, and the canister valve is closed.

4.5.6 The electronic integrator is started.

4.5.7 After the sample is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -60 °C, the VOCs are cryofocussed on the column. Then the oven temperature (programmed) increases and the VOCs elute from the column to the FID assembly.

4.5.8 The peaks eluting from the detectors are identified by retention time while peak areas are recorded in area counts.

4.5.9 The response factors are multiplied by the area counts for each peak to calculate mg/m^3 for the unknown sample.

4.5.10 Each canister is analyzed once. [Note: paired samples are collected and an equal aliquot from each is used in the analyses.]

5. Cleaning and Certification Program

5.1 Canister Cleaning and Certification

5.1.1 All canisters must be clean and free of any contaminants before sample collection.

5.1.2 All canisters are leak tested by pressurizing them to approximately 30 psig with zero air. The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than +2 psig over the 24-hour period. Alternately the canisters are tested with a He-leak MS system to 1 x 10-9 cc/sec.

5.1.3 A canister cleaning system is generally used to facilitate recycling of the cans. A cryogen (LN2) is added to the vacuum and zero air supply traps. The canister(s) are connected to a 2-position manifold. The manifold-vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The valve to the vacuum pump is opened after the manifold vent shut-off valve is closed. The canister(s) are evacuated to 100 mTorr for at least one hour.

5.1.4 The vacuum line is shut off and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 30 psig. A Bryon Instruments Model 25 (Raleigh, NC) zero gas generator system is used; the flow rate is limited to maintain optimum zero air quality.

5.1.5 At the end of cycle #1 the zero air shut-off valve is closed, and the canister(s) are allowed to vent down to atmospheric pressure through the manifold vent shut-off valve. The vent shut-off valve is closed. Steps 5.1.3 through 5.1.5 are repeated two additional times for a total of three evacuation/pressurization cycles for each set of canisters.

5.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 30 psig with humid zero air. The canister is then analyzed by a GC-FID or ECD analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) are not used.

5.1.7 The canister is reattached to the cleaning manifold and is then re-evacuated to <50 mTorr and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system, and the canister connection is capped with a brass Swagelok™ fitting. The canister is now ready for collection of an air sample. An identification tag is attached to the neck of each canister for field notes and chain-of-custody purposes.

5.1.8 As an option to the humid zero air cleaning procedures, the canisters are heated in an isothermal oven to 100 °C during Section 11.1.3 to ensure that lower molecular weight compounds (C2-C8) are not retained on the walls of the canister. For sampling heavier, more complex VOC mixture, the canisters should be heated to 120 °C. Once heated, the canisters are evacuated to 50 mTorr. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the GC-FID system. Any canister that has not tested clean (less than 0.2 ppbv of targeted compounds) are not used. Once tested clean, the canisters are re-evacuated to 50 mTorr and remain in the evacuated state until used. [Note: The Nupro SS-4H4 valves must be positioned outside of the oven; only the canister body is heated; otherwise, severe damage to the valve may occur.]

5.2 Sampling System Cleaning and Certification

5.2.1 Cleaning Sampling System Components

5.2.1.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50 °C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent only when needed.

5.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100 °C for 12 to 24 hours.

5.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

5.2.2 Humid Zero Air Certification

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv of targeted compounds) have occurred when challenged with the test gas stream.]

5.2.2.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero air.

5.2.2.2 The sampler is connected to the manifold and the humid zero air is passed through the system.

5.2.2.3 The humid zero gas stream passes through the sampling system to a GC-FID analytical system at 80 cm^3/min so that a 500 cm^3/min is pulled through the six-port valve and into the cryogenic trap. After the sample (500 mL) is reconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -60 °C, the VOCs are cryofocussed on the column. Then the oven temperature (programmed) increases and the VOCs begin to elute and are detected by the GC-FID. The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification test.

6. Performance Criteria and Quality Assurance

6.1 Standard Operating Procedures (SOPs)

6.1.1 The SOPs given in Sections 1-5 have described the following activities: (1) Manufacture, safety factor, assembly, calibration, leak check, and operation of specific sampling systems, and equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation of the analytical system for the specific equipment used; (4) canister storage and cleaning; and (5) data recording and processing.

6.1.2 Specific stepwise instructions have been provided in the SOPs and are available to and understood by the laboratory personnel conducting the work.

6.2 Method Relative Accuracy and Linearity

6.2.1 Accuracy is determined by measuring VOC standards from an NBS or EPA audit cylinder into a sampler. The contents are then analyzed for the components contained in the audit canister. Percent relative accuracy is calculated:

Where: Y = concentration of the targeted compound recovered from sampler.

X = concentration of VOC targeted compound in the NBS-SRM or EPA-CRM audit cylinders.

6.2.2 If the relative accuracy does not fall between 90 and 110 percent, the field sampler is not used. Historically, concentrations of the C2-C9 hydrocarbons are accurately measured with the FID. The detector is very linear over concentration ranges of 104 or more. All of the software for the Hewlett-Packard series of GC 5790's with 3390A data processors, GC 5890's with 3393A or 3396 recorder-integrators are designed to accommodate multilevel calibration entries, so the correct response factors are automatically calculated as well as concentrations in the range of ambient air analysis.

6.3 Quality Assurance

6.3.1 Sampling System

6.3.1.1 Pre- and post-sampling measurements with a mass flow meter or rotometer for flow verification of sampling system should be made periodically in the field.

6.3.1.2 Canisters are pressure tested to 30 psig +2 psig over a period of 24 hours or preferably He leak-tested to 1 x 10-9 He cc/sec.

6.3.1.3 All canisters are certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

6.3.1.4 All field sampling systems are certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

6.3.1.5 All field sampling systems have passed an initial humidified calibration gas certification [at VOC concentrations levels expected in the field (e.g., 0.5 to ppbv)] with a recovery of greater than 90%.

Equation (6) Percent Relative Accuracy

$$\%\ Relative\ Accuracy = \frac{X - y}{X} \times 100$$

6.4.1 GC-FID System Performance Criteria

6.4.1.1 The GC-FID analytical system, prior to analysis, is certified to be clean (less than 0.2 ppbv of targeted VOCs) through a humid zero air certification.

6.4.1.2 The GC-FID analytical system retention time windows for each analyte prior to sample analysis are verified when a new GC column or major components of the GC system are altered since the previous determination.

6.4.1.3 All calibration gases are traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM).

6.4.1.4 The retention time windows are re-established continuously throughout the course of the analytical period.

6.4.1.5 The long-term quality control results for measuring our neohexane working standard of 226 ppbv against two different NBS SRMs are given in Table 6.4.1.5. The precision of these intensive sets of analyses (usually 3 days are required to complete the tests) are very good: 1-4%. The data have been systematically obtained since 1985. Currently we intercalibrate our working standard against two NBS SRMs, benzene and propane, every six months. No difference or bias is observed for either NBS SRM used to calibrate the neohexane standard. The data suggest that the neohexane standard is very stable and is accurately referenced to the NBS SRMs. The NBS-SRMs concentrations and hydrocarbon species are very different from one another: 2.87 vs. 0.254 ppmv for propane and benzene respectively. This supports our contention that our neohexane standard is accurately referenced to the NBS-SRMs. We have been using neohexane as our daily working standard for 14 years. The prime reason it was selected is that it is one of the few hydrocarbons that can be used as an internal standard in an urban ambient air sample because it is not a product of auto exhaust. The lower detection limit of our GC-FID system using 500-mL air samples is 0.2 mg/m^3 for benzene. The precision of analysis for benzene at 3.0 mg/m^3 is +10% (2s). In clean air along the California coast observed benzene levels are 0.3 to 3mg/m^3, whereas at dirty sites 3-12 mg/m^3 values are measured. The lower detection limit of our GC-FID system of 0.1 to 0.2 mg/m^3 per compound for a 500-mL sample is determined more by the threshold limits of the HP electronic integrators than by the physical discernment of a peak. The lower detection limits for propane (C3), neohexane (C6), and benzene (C6 aromatic) are consistent within the analytical parameters used. Therefore, we believe that from C3 to C9 the lower detection limit for identified and unidentified hydrocarbons species is essentially the same: ~0.2 mg/m^3.

Primary Standards	Neohexane Working Standards			Percent	
	Given Value	Analysis	Assigned	Measured	Difference
	ppmv	Date		ppmv	ppmv
Benzene NBS-SR #1805 CAL 5679	0.254	Dec 1985	0.226	0.224	2
Benzene NBS-SR #1805 CAL 5679	0.254	Jun 1987	0.226	0.233	4
Benzene NBS-SR #1805 CAL 5679	0.254	Nov 1987	0.226	0.219	-3
Benzene NBS-SR #1805 CAL 5679	0.254	Apr 1988	0.226	0.221	-3
Propane NBS-SR #1665-B FF27623	2.87	Mar 1988	0.226	0.218	4
Benzene NBS-SRM #1805 CAL 5679	0.254	Oct 1988	0.226	0.222	±2
Propane NBS-SR #1665-B FF27623	2.87	Oct 1988	0.226	0.224	±1
Benzene NBS-SRM #1805 CAL 5679	0.254	Jun 1989	0.226	0.205	-9
Propane NBS-SRM #1665-B FF27623	2.87	Jun 1989	0.226	0.225	±1
Benzene NBS-SR #1805 CAL 5679	0.254	Oct 1989	0.226	0.226	0
Propane NBS-SRM #1665-B FF27623	2.87	Oct 1989	0.226	0.227	0
Benzene NBS-SRM #1805 CAL 5679	0.254	Apr 1990	0.226	0.226	0
Propane NBS-SRM #1665-B FF27263	2.87	Apr 1990	0.226	0.226	0

6.4.1.6 The absolute accuracy of our calibration standard is determined against the two NBS-SRM standards. The measured value for 7 determinations over three years is 0.224 ppmv +0.005, or +2.3%. This suggests that our neohexane standard is less than its assigned value of 0.226 ppmv by 0.002 ppm, or 0.9%. We do not believe this is a real difference.

SECTION 12 - General Laboratory Procedures (AWL)

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GENERAL LABORATORY PROCEDURES

A. Preparation and Cleaning of Glass Sample Storage Containers

Glass containers are washed with hot tap water and detergent, rinsed with distilled water, placed in a concentrated nitric/sulfuric (1/3) acid bath for 10 min, rinsed again with distilled water, and dried in an oven at 110°C for >8 h. The lids are lined with Teflon to prevent contamination of the sample.

B. Handling of Filters and Soils

Clean cotton (100%) gloves are used whenever handling soils or filters. In limited situations, Latex gloves may be used.

C. Labeling of Sample Storage Containers

Labels containing the date of use, ELI sample number, and other sample identification information are placed on glass storage containers immediately after sampling and return of the samples (e.g., soil samples, filters, etc.) to the storage containers. Likewise, similar information is recorded in a journal. Both the label and the journal record are signed and dated by AWL authorized personnel.

D. Storage of Filters, Soils, and Fall-out Pan Particulates

All soil and fall-out pan particulate samples are stored in acid-washed glass containers in a walk-in freezer (WIDB 629) at 5°C or in an AWL freezer (ESC 106) at -15°C prior to extraction. All filter samples are stored in their Teflon-lined envelopes in a freezer (ESC 106) at -15°C prior to extraction.

E. Storage of Acetonitrile Extracts

Glass vials (1-dram amber with Teflon-lined lids) containing the final extracts are stored at -15°C in a freezer.

F. Recording and Correcting Data

1. All data are recorded using a black permanent ink pen.
2. All dates are written in the order of day, month, year, the month being a three-letter abbreviation (i.e., 25 Nov 1988).

3. All times are stated according to a twenty-four hour clock with a colon separating the hour from the minute (i.e., 13:52).

4. All data sheets are signed and dated when completed and only after a check for completeness and correctness has been done.

5. All journals are signed and dated at the end of each day and at least once on each page.

6. A correction consists of the incorrect data crossed out with a single line such that it remains legible, the correct data written in, the date of correction, and the initials of the person making the correction.

7. Any reprocessing is considered new data and not a correction, and is handled as such.

SECTION 13 - Preparation, Handling, and Extraction of Quartz Fiber Filters (AWL)

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PREPARATION, HANDLING, AND EXTRACTION OF QUARTZ FIBER FILTERS

A. Preparation of Filters

1. Quartz fiber filters (20.3-cm x 25.4-cm rectangular) are purchased from Whatman. Cotton (100%) gloves are used for handling the filters.

2. The filters are placed on aluminum trays, fired in a muffle furnace at 650°C for 8 h, slowly cooled to room temperature, and individually stored in Teflon containers and marked with an I.D. number and weight.

B. Weighing of Filters

1. The fired filters are weighed both before and after sampling on an analytical balance to 0.01 mg. A minimum of 16 h should be allowed for initial equilibration before the first weighing.

2. Rectangular filters are placed back in the Teflon containers and labeled with the weight.

3. After sampling, a 1 in x 1 in square is cut from each filter and shipped to Sunset Laboratories for total carbon analysis. The filter is weighed on the analytical balance before and after cutting.

4. In order to ensure the accurate performance of the balance during weighing, the following are done:

   a. One calibration weighing each day.

   b. A reference weighing (same weight range) between every five samples.

   c. Weighing of control filters spread out over the weighing period.

C. Labeling of Filters

After sampling, the filters are returned to their labeled storage containers and given another label containing the date of use, ELI sample number, and other identification information. This information is also duplicated in the journal. The label and journal are signed by authorized AWL personnel.

D. Extraction of Filters After Sampling

1. Each filter is placed in a 60-mm X 180-mm cellulose thimble and extracted with 700 Ml of nanograde acetonitrile in a Soxhlet extractor (extra large, 1000-mL round bottom flask) as per LOI-5 for 6 h at a solvent temperature of 80°C.

2. The acetonitrile extract is concentrated to a volume approximately 1-2 mL using a rotary evaporator (50°C), LOI-6, transferred to a 1-dram amber vial (Teflon-lined cap), reduced to approximately 0.4 mL under N_2 purge, and brought to a volume of 0.4 mL by adding nanograde acetonitrile.

3. The samples are split using a glass micro pipette into two equal 0.2-mL parts, 20 μL of internal standard solution is added to one half (6 μg of 1-nitronaphthalene-d_7 and of 9-phenylanthracene), and the other half is placed in storage.

E. Storage of Acetonitrile Extracts

The 1-dram amber vials containing the final filter extracts are stored at -15°C in a freezer.

SECTION 14 - Extraction of Soil and Fall-Out Pan Particulates (AWL)

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EXTRACTION OF SOIL AND FALL-OUT PAN PARTICULATES

1. Approximately 1-400 grams of soil or fall-out pan residue are placed in a 60-mm x 180-mm cellulose extraction thimble.
2. 500 mL of nanograde acetonitrile is introduced into a 1000-mL round bottom flask.
3. A heating mantle connected to a variable auto transformer is used to heat the extraction flask to 80°C.
4. The soil is extracted using a Soxhlet extractor for 12 hours.
5. The extraction fluid is concentrated to a volume of 4-10 mL using a rotary evaporator.
6. The 4-10 mL of fluid is then passed though an alumina (oxide) column (3-5 cm in length) using acetonitrile as eluant.
7. The sample is concentrated to a volume of 0.4 mL and then placed in a 1-dram Teflon-sealed amber vial.
8. The extract is divided into two 0.2-mL portions and an internal standard (6 μg of 1-nitronaphthalene-d_7 and 6 μg of 9-phenylanthracene) is placed in one of the vials. Both are placed in storage at -15°C in a freezer.

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SECTION 15 - Analysis of Bulk Explosives and Propellants (AWL)

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ANALYSIS OF BULK EXPLOSIVES AND PROPELLANTS

A. Explosives samples

A small sample (< 1 g) of each explosives sample is collected and stored in a clean 1-dram glass bottle (Teflon-lined lid) until analyzed. Approximately 0.1 mg of the sample is dissolved in 5 mL of acetonitrile for analysis by SFC/MS and GC/MS. A sample (0.4 mL) of this solution is removed and split into two equal parts. To one half of this sample, 20 µL of internal standard is added (6 µg of 1-nitronaphthalene-d_7 and 6 µg of 9-phenyl-anthracene). Both halves are placed in storage at -15°C in a freezer.

B. Propellant Samples

Small samples of the propellants are collected and stored at -15°C. The samples are treated as in part A.

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SECTION 16 - Soxhlet Extractor Operation (AWL)

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SOXHLET EXTRACTOR OPERATION

A. The Soxhlet Extractor consists of three parts: extraction flask, Soxhlet, and condenser (Figure 1).

B. A 1000-mL (extra large) Soxhlet extractor is used with a 60-mm x 180-mm cellulose thimble to extract samples as follows:

1. 700 mL of solvent is placed in the 1000-mL extraction flask.
2. The extraction flask is placed in the heating mantle as shown in Figure 1.
3. The sample is placed in a clean thimble.
4. The Soxhlet extractor is assembled with the cellulose thimble in place.
5. The cold water to the condenser is turned on.
6. A variable auto transformer is connected to the heating mantle to control the temperature.
7. The heating mantle is heated to a temperature at which the solvent just boils and there is a turn-over time for the Soxhlet of approximately 10-12 mins.
8. After extracting for the required time, the power is switched off, and the extractor is allowed to cool.
9. After cooling, the thimbles are drained of any residual solvent which is added to the solvent in the extraction flask.
10. The sample is now ready for concentration.

C. After extraction, all Soxhlet parts are cleaned.

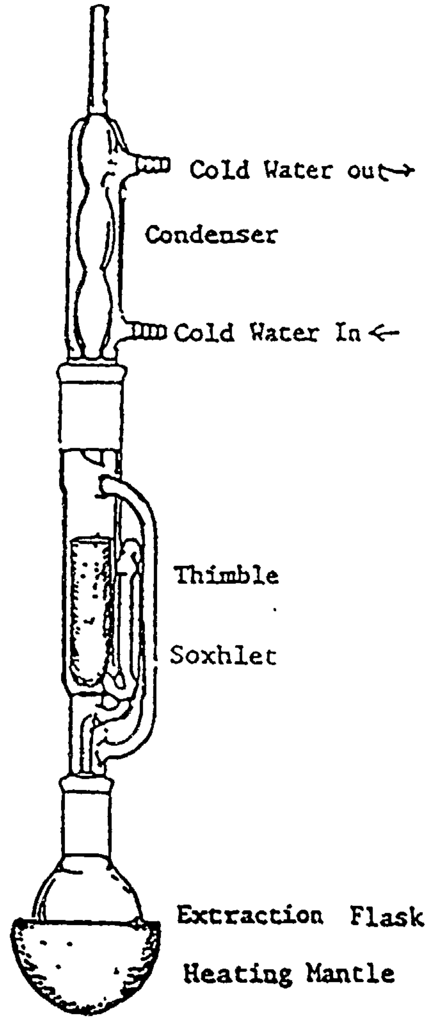

Figure 1. Soxhlet Extractor Set-up

Approved: ______________________ Received: ______________________
Study Director Quality Assurance Director

______________________ ______________________
Date Date

SECTION 17 - Rotary Evaporator Operation (AWL)

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ROTARY EVAPORATOR OPERATION

1. Turn on the water for vacuum cooling.
2. Turn the vacuum stopcock so that no vacuum is created (aligned with the hole in the condenser).
3. Attach the round bottom evaporating flask containing the sample to the exposed end of the vapor duct.
4. Lower the evaporating flask so that it is partially submerged in the water bath, which is maintained at or below 35°.
5. Adjust the rotation speed of the drive unit and add the vacuum by rotating the vacuum stopcock one quarter turn. Be certain that no bumping or foaming occurs.
6. When the sample has nearly evaporated (~1 mL left), turn off the vacuum and the drive unit. Remove the evaporating flask. Empty the receiving flask in the appropriate waste container.
7. Transfer the remaining solution to a 1-dram glass vial, rinse the round bottom flask with approximately 0.5 mL of solvent 3 times and transfer each rinse to the 1-dram vial.
8. Re-concentrate the sample to 0.4 mL by N_2 (g) purge.
9. Clean the exposed portion of the vapor duct by the following procedure:
    a. Turn the vacuum off and turn on the drive unit to a slow setting.
    b. Hold a 100-mL beaker of appropriate solvent under the rotating vapor duct. Be sure that all of the ground glass joint is wetted by the solvent.
    c. Lower the beaker of solvent until part of the opening of the vapor duct is above the surface of the solvent.
    d. Close the vacuum stopcock.
    e. Gradually raise and lower the beaker to allow the duct to suck up enough solvent to coat the end of the tube.

f. Partially open the stopcock valve; submerge the rotating vapor duct in the beaker of solvent and adjust the stopcock valve until the vapor duct fills with solvent (but does not overflow). Allow the solvent to remain in the rotating tube for at least 5 seconds. Close the stopcock and allow some of the solvent to overflow into the rotary evaporator receptacle.

g. Empty the receiving flask and cover the exposed portion of the vapor duct with aluminum foil.

SECTION 18 - Supercritica[illegible]d Chromatography/Mass Spectrometry (AWL)

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Alpine West
OBOD Field Test C
LOI-7
4 January 1991
Revision 5

SUPERCRITICAL FLUID CHROMATOGRAPHY/MASS SPECTROMETRY (SFC/MS)

This LOI describes the SFC/MS instrumentation and methodology for analysis of extracts of OB/OD samples. Preparation of extracts are described in separate LOIs. Three complementary SFC/MS analyses are performed on each extract. Selected-ion monitoring with negative ion chemical ionization (NICI/SIM) is used for the nitroaromatic target analytes. Selected-ion monitoring with positive ion chemical ionization (PICI/SIM) is used for the remaining target analytes. Full scan electron impact ionization (EI/MS) is used to identify nontarget analytes that may be of interest.

A. EI/MS

1. The instrument used for this method is a Lee Scientific Model 602/- Finnigan-MAT Incos 50.

2. The instrument is mass calibrated and tuned when needed using perfluorotributylamine (FC-43). Mass calibration is performed using software and recommended procedures provided by the manufacturer.

3. Chromatographic separation involves a Lee Scientific Model 600 SFC. A direct probe interface equipped with a heated frit restrictor delivers the eluent from the SFC to the MS. One microliter of sample is injected using a solvent venting technique. A 5% phenyl methylpolysiloxane stationary phase coated in a 0.050-mm i.d. fused silica capillary column is used for separation. The initial fluid density and temperature is a function of the sample solutes. Density programming is used for analysis, and all conditions (SFC and MS) are identical for all analyses.

4. A standard containing the target analytes of interest for the specific test is analyzed during each shift.

5. An analytical blank is analyzed every day that samples are analyzed.

6. Data acquisition consists of repetitive scanning from m/e 100 to m/e 500 with a cycle time of approximately 1 sec. Data acquisition begins with and continues throughout the chromatographic separation process.

7. Mass spectral interpretation is conducted for the most intense chromatographic peaks.

8. No quantitative analysis is performed on the EI/MS data.

B. PICI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/- Finnigan-MAT Incos 50.

2. The instrument is mass calibrated and tuned as described in A.2-A.3, except that tuning is conducted in the positive chemical ionization mode.

3. A standard of target analytes that respond in the positive ion mode, for each test, is analyzed daily.

4. A response curve is generated by analysis of the standards at 1.0, 10, 50, and 100 ng μL^{-1} of each compound. The internal standard is present in each standard at 20 ng μL^{-1}. The response curve consists of the response of a particular analyte relative to that of the internal standard, plotted as a function of analyte concentration. The purpose of the response curve is to provide quality assurance of response linearity. Quantitative calibration is performed using a standard solution analyzed at the beginning of each day samples are analyzed.

5. Response factors are established for each analyte (i) from analysis of a daily standard solution. The response factor (RF_i) is defined as follows:

$$RF_i = \frac{(A_i)\,(C_{IS})}{(A_{IS})\,(C_i)}$$

   where A_i and A_{IS} are the selected ion chromatographic peak areas of the analyte and internal standard, respectively. C_i and C_{IS} are the concentrations of the analyte and internal standard, respectively.

6. OB/OD samples and field controls are analyzed using the same instrumental conditions as the standard. Identification of target analytes are by retention time and response at the specific mass monitored for each analyte. The concentration of each analyte (C_i) as mass per unit volume of extract (eg. ng/mL) is defined as:

$$C_i = \frac{(A_i)\,(C_{IS})}{(A_{IS})\,(RF_i)}$$

   where the variables are functionally equivalent to those in B.5 but whose values are obtained from the analytical data of the OB/OD sample.

C. NICI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/-Finnigan-MAT Incos 50.

2. The instrument is mass calibrated and tuned as in B.2.

3. A standard of target analytes that respond in the negative ion mode, for each test, is analyzed daily.

4. The remainder of the analytical procedure is equivalent to that described in B.4-B.6.

D. Data Reporting and Archiving

1. The results from each EI/MS analysis consist of the following:

   (a) A total ion chromatogram annotated to indicate the peaks identified.

   (b) A background corrected mass spectrum for each identified compound.

   (c) A table consisting of a spectrum number and identification.

   Where possible, an order of magnitude estimation of concentration is included.

2. The results from each NICI/SIM/MS and PICI/SIM/MS consist of the following:

   (a) A single page reconstructed (total ion) chromatogram.

   (b) A single page mass chromatogram containing the quantification area for each analyte and internal standard.

   (c) A table containing the areas and response factors for each analyte and internal standard.

3. For each sample, a summary table is provided containing all target analytes found with final concentrations expressed as ng/sample.
4. Raw SFC/MS data are archived in disk image format on data cartridge tapes (DC 300 XL/P) at 10,000 ftpi.

E. Determination of Calibration Curves and instrument Detection Limits for SFC/MS

1. A stock solution of the target organic analytes in acetonitrile at a concentration of 100 ng μL^{-1} is prepared.

2. A logarithmic dilution of the stock solution (1 to 10 dilution) is made until the signal observed in the reconstructed total-ion chromatogram for the analytes in the mass spectrometer is less than three times the background signal.

3. The signal observed from the INCOS 50 data system is given in relative ion counts (RIC).

4. The point at which the RIC for the analytes is three times that of the background corresponds to the instrumental detection limits.

5. A graph of concentration versus observed peak area is made to construct a calibration curve for each analyte. This serves as a standard curve and is sometimes used to determine concentrations in unknown samples. This is accomplished by matching the observed signal to the corresponding concentration for that analyte. Where possible all quantitations are made from response curves and comparison to the internal standards.

6. Limits of quantification refer to the lowest concentration detectable above the background (analyte RIC at three times the background RIC).

7. All of the compounds on the target organic analyte list are tested in a like manner.

8. Steps 1 to 7 are used for both electron impact and chemical ionization.

SECTION 19 - Target Analytes for TNT Test (AWL)

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Target Organic Analytes for TNT test

1. 2,4-Dinitrotoluene
2. 2,6-Dinitrotoluene
3. 2,4,6-Trinitrotoluene
4. 2-Nitronaphthalene
5. N-Nitrosodiphenylamine
6. 1,3,5-Trinitrobenzene
7. 1-Nitropyrene
8. Naphthalene
9. Benz[*a*]anthracene
10. Benzo[*a*]pyrene
11. Pyrene
12. Phenol
13. Dibenzofuran
14. Diphenylamine

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SECTION 20 - Gas Chromatography/Mass Spectrometry

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GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

This LOI describes the GC/MS instrumentation and methodology for analysis of extracts of OB/OD samples. Preparation of extracts is described in a separate LOI. Three complementary GC/MS analyses are performed on each extract. Negative ion chemical ionization/selected ion monitoring (NICI/FS/MS) is used for the nitroaromatic target analytes. Positive ion chemical ionization/selected ion monitoring (PICI/FS/MS) is used for the remaining target analytes. Full scan electron impact ionization (EI/MS) is used to identify nontarget analytes that may be of interest.

A. PICI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/-Finnigan-MAT Incos 50 GC/MS operated in a conventional GC/MS mode.

2. Calibration, tuning, and sample analysis are as described in LOI-7, sections B.2-6.

B. NICI/SIM/MS

1. The instrument used for this method is a Lee Scientific Model 602/-Finnigan-MAT Incos 50 GC/MS operated in a conventional GC/MS mode.

2. Calibration, tuning, and sample analysis are as described in LOI-7, sections C.2-4.

C. EI/MS

1. The instrument used for this method is a Lee Scientific Model 602/-Finnigan-MAT Incos 50 operated in a conventional GC/MS mode.

2. Calibration, tuning, and sample analysis are as described in LOI-7, sections A.2, 4, 5, 7, and 8.

3. The chromatographic separation involves a Lee Scientific Model 600 GC. One microliter of sample is delivered by "cold" on-column injection. A 5% phenyl methylpolysiloxane stationary phase coated onto a 0.250 mm i.d. fused silica capillary column is used for separation. Samples are analyzed by temperature programming, and all conditions (GC and MS) are identical for all analyses.

4. Data acquisition consists of repetitive scanning from m/e 100 to m/e 500 with a cycle time of approximately 1 s. Data acquisition begins after the elution of the solvent peak and continues throughout the chromatographic separation process.

D. Data Reporting and Archiving

1. Data are reported and archived as described in LOI-7, sections D.1-3.

SECTION 21 - Quality Control Plan, Procedures for Accuracy, Precision, and Completeness (AWL)

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QUALITY CONTROL PLAN: PROCEDURES FOR ACCURACY, PRECISION AND COMPLETENESS

A. Weighing

1. All balances are calibrated at 6 month intervals by a Sartorius service engineer.
2. The analytical balance (Model 2434) is accurate to ± 0.00001 g, and the top-loading balance (Model E5500S) is accurate to ± 0.01 g.
3. All samples are weighed as per their corresponding LOI (LOI-2 for filters and LOI-3 for soils).

B. Extraction

1. Filters

During the extraction of real samples, 5-10% additional filter samples are introduced as blanks.

2. Soils and Particulates

a. Extraction efficiency of analytes from soil is calculated by the analysis of solutions from the extraction of 15 soils spiked with known concentrations of target analytes. Soils are spiked in triplicate at 0, 1, 10, 50, and 100 ppb (g/g) levels.

b. Completeness of extraction is measured by (1) re-extraction of the high spiked soils and (2) of a real test soil.

c. A minimum of 5 soil samples (spiked by the EPA) are extracted to determine the accuracy of the method.

d. During the extraction of real samples, approximately 5-10% additional soil samples are introduced as blanks.

C. Sample Storage

The temperature of the freezer used for storage is monitored daily.

D. SFC/MS and GC/MS

1. Instrument calibrations with FC-43 are run periodically and logged.

2. Analyte standard solutions are run daily, and peak areas relative to the internal standards are calculated. A deviation of over ± 30% will signify a dirty system and require cleaning.

3. Matrix blanks are run at the approximate frequency of 1 in every 10 field samples (≈10%).

4. A random number of samples will be run in duplicate weekly.

E. Sampling Handling

1. All samples are given an internal identification number.

2. All sample collection (ELI) sheets are checked against samples for completeness and correctness.

3. All samples are logged as to where they are stored and where they came from.

4. All samples have an AWL analysis sheet assigned (see attached).

ALPINE WEST LABORATORIES

SAMPLE ANALYSIS

SAMPLE DESCRIPTION: ______________________________

____

SAMPLE I.D. #: ______________________________

DATE RECEIVED: ______________________________

SAMPLE PREPARATION

EXTRACTED: ______________ DATE EXTRACTED: ______________

ANALYST: ______________________________

METHOD: ______________________________

TIME STARTED: ______________ TIME ENDED: ______________

SOLVENT: ______________________________

TEMPERATURE: ______________________________

CONCENTRATED: ______________ FINAL VOLUME: ______________

ANALYSIS

DATE ANALYZED: ______________________________

ANALYST: ______________________________

METHOD: ______________________________

RESULTS:

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SECTION 22 - Determination of Moisture Content in Soil Samples (AWL)

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DETERMINATION OF MOISTURE CONTENT IN SOIL SAMPLES

1. Approximately 200 grams of soil are placed in a tared 400-mL beaker.

2. The exact weight is recorded.

3. The beaker is covered with perforated aluminum foil.

4. The beaker is placed in an oven at 130°C for 24 h for the soil to dry.

5. The beaker is removed from the oven and cooled to room temperature.

6. The foil is removed, the beaker is weighed, and the weight is recorded.

7. The percent moisture is determined by

$$\%M = \frac{(\text{wet weight - dry weight})}{\text{dry weight}} \times 100$$

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SECTION 23 - Extraction Efficiency for Target Analytes From Soil Samples (AWL)

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Alpine West
OBOD Field Test C
LOI-11
4 January 1990
Revision 5

EXTRACTION EFFICIENCY FOR TARGET ANALYTES FROM SOIL SAMPLES

This experiment is designed to determine the extraction efficiency of the target analytes from Dugway soil. The target analytes are listed in LOI-7 of the Test Plan Field Test Phase C for the Study of OB/OD, July 1990. A qualified quality assurance person from ELI should be present to observe this experiment through completion.

A. Preparation of standard spiking solution

1. 10 mg of each standard will be weighed directly into individual sample vials. The Standards will be diluted with acetonitrile to a final concentration of ~10 mg/mL.
2. A 100 uL aliquot from each ~10 mg/mL standard solution will be transferred to a 25 mL volumetric flask and diluted with acetonitrile to a final concentration of 40 μg/mL for each target analyte. This final standard solution (40 μg/mL) will be used as the matrix spiking solution.

B. Preparation of spiked soil samples

1. Soil samples will be prepared in triplicate at four levels; 0.5 ppb, 1 ppb, 10 ppb, and 100 ppb.
2. 0.5 mL, 0.1 mL, 1 mL, or 10 mL of the spiking solution will be added to a 125 mL of acetone in a 1000 mL round bottom flask. 0.5 mL, 0.1 mL, 1.0 mL, and 10 mL of the spiking solution correspond to 0.5 ppb, 1 ppb, 10 ppb, and 100 ppb of each target analyte in 400 g of soil.
3. 400 g of soil will be weighed and added to the round bottom flask containing the acetone solution.
4. The soil will then be rotoevaporated until the solvent (acetone) is removed and the soil is dry.
5. The spiked soils and two blank soils will then be randomized and assigned unique numbers by the ELI observers so that the analyst can not determine the identity of the soils.
6. The soils will then be transferred to sample containers and refrigerated at -15°C until extraction.

C. Sample Extraction

1. All spiked soil samples and the 3 blanks (15 total) will be extracted following the procedure outlined in LOI-3 of the Test Plan Field Test Phase C for the Study of OB/OD, July 1990.
2. The 100 ppb spiked soils will be extracted a second time to determine if any residue from the target analytes are left after the first extraction.

D. Sample Analysis

1. All spiked and blank soil sample extracts will be analyzed for the target analytes following the procedure outlined in LOI-7 of the Test Plan Field Test Phase C for the Study of OB/OD, July 1990.

SECTION 24 - The Effect of Storage on Soil Samples from OB/OD

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July 8, 1990

THE EFFECT OF STORAGE ON SOIL SAMPLES FROM OB/OD

OBJECTIVE: To determine if prolonged storage has an adverse effect on the quantity of semi-volatiles found in the soil samples from OB/OD.

SOURCE OF SAMPLES: Ejecta from a TNT site used on the Phase C OB/OD test at DPG will be collected and bottled for this study. The site will be one that was used on the Phase C test with one detonation. The crater will be filled in a second TNT detonation will be made at this same site , the crater will again b e filled in, a third detonation will be made at this same site. After the third detonation a soil sample will be taken of the ejecta material large enough to make at least 24 500 g samples.

VARIABLES: Two

Method 1: extract immediately
Method 2: Store soil until assay is requested.

Holding Time 1: Immediate extraction and assay.
Holding Time 2: One month. (to be determined after review of the TSC)
Holding time 3: to be determined
Holding Time 4: to be determined

ANALYSIS: The analysis will be sequential with comparison of the two methods at each holding time. since the assay for the semi volatiles is expected to result in several compounds that will be tracked over time it is likely that compounds may have different retention rates in the storage media over time.

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SECTION 25 - Data Reduction and Analysis (ANDRULIS)

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DATA REDUCTION AND ANALYSIS - OUTLINE OF PROCEDURE

A. DATA RECEIVED

1. Results from: (1) individual sample assays (wt/vol, wt/area, etc.), (2) all blanks (laboratory method blanks, travel blanks, field blanks, etc.), (3) control standards (field standards, storage controls, sample preparation controls, laboratory QC standards, etc.), and (4) duplicate assays. All these results are to be presented in the chronological order in which the samples were analyzed, along with notations as to when instrument conditions were changed, maintenance performed, etc.

2. Daily calibration curves and all data used to generate the line of best fit. If the mean of several separate assays were used as the input data, furnish individual results. Describe the standard solutions, e.g., whether they include only a single compound or several compounds.

3. Algorithms (with constants) that are used in computing results in terms of engineering units from the measured units; e.g., wt/vol at standard conditions from millivolts.

4. Fixed wing aircraft (FWAC) real-time data recorded in 5-s intervals for a period of at least 5 min, for all indirectly sampled (samples from 80-L teflon tie bag) parameters, and data recorded at the high rate (5/s) for the direct sampling. Data will be furnished in a LOTUS 1-2-3 file with column and row identification, and with units.

5. Weights of quartz filters, fallout residue, burn pan residue, and soil samples will be furnished for each sample prior to compositing. All weights will be to an accuracy that is traceable to a certified weight standard.

6. Other aircraft data required, as a minimum: height, aircraft speed, and air temperatures during the background sampling and cloud sampling. All data will be identified by a time indicator that references the specific, recorded detonation or burn time.

7. All data produced from chemical analyses and other measurements. (The operator is not to arbitrarily delete readings.) Any data collector or other worker on the project is to identify any unusual incident and record special observations pertinent to sample collection, handling, storage, and analysis, and/or to the conduct of the test, associating the remarks with an individual sample whenever possible. Submit this information immediately upon completion of a task.

B. DATA ANALYSIS

1. Examine graphic output of real-time instruments after each trial, to determine if the trial was successful (based upon FWAC passage through the detonation or burn plume and its successful sampling with the aircraft sampling systems).

2. Verify the unit conversion algorithms by converting at least one non-zero value (in measured units) to engineering units.

3. Correct all analyte concentrations for measured background levels, as required.

4. Calculate the emission factor (using the carbon balance procedure) for each measured emission. Using these emission factors from each sampling point, determine the total mass of each element and chemical compound. (This method has previously been proven in tests at Sandia National Laboratories, and the method has been approved by the Open Burning/Open Detonation (OB/OD) Technical Steering Committee for use in determining the mass of the emittants from OB/OD.)

5. Compare the assay concentration results from the supercritical fluid chromatography/mass spectrometer analysis method with the results from the gas chromatography/mass spectrometer method. The comparison will use the results from the U.S. Environmental Protection Agency (EPA) report on spiked samples, the FWAC filter samples, and results of assay of internal laboratory standards and spiked samples.

6. Compare the concentrations of CO and CO_2 derived from the 6-L canister sampling system with the concentrations derived from the real-time fixed-wing-aircraft sampling system.

7. Calculate (as data permits) all components of variance, e.g., between filter variation within trial (weight and chemical analysis), between subsamples of the same extract when analyzed as separate samples on different days, between separately collected paired soil samples, etc. The amount of data available for these calculations will depend upon assets that the program manager can obligate to the program.

8. All results from these analyses/comparisons will be furnished the Program Manager and only upon his approval will additional distribution be made.

C. OUTLIER DATA

All available data and recorded operator comments will be considered prior to deleting a suspected outlier data point. If the comments do not provide a reasonable rationale for removing the datum, the laboratory worker or other data collector will be asked to review the information and make further remarks. All data points deleted by the statistical analyst will be documented, together with the reason for the deletion.

SECTION 26 - Procedures for Writing Letters of Instructions (ELI)

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Procedures for Writing Letters of Instruction (LOI)

1. Purpose

QA/QC practices require that all routine activities having an impact on data quality be fully documented. These day-to-day routine practices should be itemized in a clear, explicit, somewhat detailed, step-by-step manner so that most individuals could follow them in a uniform and consistent manner.

2. Scope

LOI should be prepared for all routine activities associated with the OB/OD project. Such activities may include:

Field or Laboratory Sampling
Field or Laboratory Analysis
Instrument or Method Calibrations
Preventative and Corrective Maintenance
Internal QC Procedures
Site Selection
Sample Preparation and Storage
Preparation and use of Spiked Samples
Instrument/Equipment Selection and use
Determination of Detection Limits and Limits of Quantification
Sample Handling and Transportation
Conduct Performance Audits
Data Reduction and Analysis

Once the LOI are written, they will be reviewed for adequacy by the QA Officer and Technical Coordinator, and kept in each Laboratory's files and made available during QA visits. LOI may also be changed or revised during the study to conform with the actual work effort. However, these changes should be clearly documented and the old LOI replaced with the new one (see document control section below).

3. Format

The format and outline of LOI may vary somewhat depending on the activity. However, all LOI should contain the following:

1. Title
2. Outline of procedures
3. References (if appropriate)
4. Document control block in upper right hand corner
5. Signature line for project officer and signature line for QA

4. Document Control

Each page of the LOI should contain a document control block in the upper right corner containing: LOI number, date of issue, revision number, and page number similar to that used for this document.

5. Where to Send LOI

A copy of each LOI should be kept in the originating laboratory and a signed copy should be sent to:

Dr. Gary M. Booth
Environmental Labs, Inc.
1125 South 550 East
Springville, Utah 84663

All LOI should be received by Nov 28, 1988. If you have any questions, you may call Gary Booth at (801) 378-2458.

Approved: ____________________ ____________________ Program Manager
Quality Assurance Unit

____________________ ____________________ Date

SECTION 27 - OB/OD Phase C QA/QC Forms

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LETTER OF INSTRUCTION

FOR

SAMPLE AND CUSTODY REPORT
AND
SHIPPING LIST DOCUMENT PREPARATION

OB/OD PHASE "C"

ENVIRONMENTAL LABS, INC.
3355 NORTH UNIVERSITY AVENUE, SUITE 275
PROVO, UTAH 84604

1.0 SAMPLE AND CUSTODY REPORT

1.1 GENERAL INSTRUCTIONS

1.1.1 Each sample and custody report (SCR) has a pre-printed four (4) digit number.

1.1.2 Each sample taken shall have a separate SCR completed.

1.1.3 If a sample is to be split into multiple samples, each fraction of the sample shall have a separate SCR completed. Each split from the original, and the original SCR would need to have the other sample numbers listed in section 3 of the SCR.

1.1.4 If a sample is to be composited from multiple samples, then a separate SCR shall be completed for the composited sample. The contributing sample numbers shall be listed in section 3 of the SCR. In the event that multiple fractions of a composite shall exist, then a separate SCR shall be completed for each portion of the sample.

1.1.5 The SCR consists of an original and three (3) copies. The distribution is as follows:

Original - Stays with sample until received at analysis laboratory. Once received at final destination and section 4 is completed, it is to be returned to ELI at the address on the form.

1st Copy - Retained by the final destination/analysis laboratory for their records.

2nd Copy - Retained by shipping facility for their records.

3rd Copy - To ELI QA personnel immediately after sections 1 and 2 are completed. This is to be done prior to sample departure from test site, with the exception of sample splitting or compositing done in the laboratory.

1.1.6 Date entries: All dates will be entered in the format of day of the month, the three letter abbreviation for the month, and the two digit abbreviation for the year. Example; 25 AUG 90. Entries such as 8/25/90 or 25/8/90 are not acceptable.

1.1.7 Time entries: These shall be entered based on the 24 hour clock. Example; 10:00AM = 1000, 10:00PM = 2200.

1.2 CHAIN OF CUSTODY

1.2.1 This section is to be completed for each person who has custody of the sample.

1.2.2 Each person who has custody should complete the first unused block of this section. Blank blocks should not be left in this section.

1.2.3 The entries for this section consist of signature, the person's affiliation (laboratory or company), and the date and time received (as specified in section 1.1.7).

1.2.4 When the sample has been received at it's final destination and is to be analyzed, the receiving laboratory should sign for receipt, and then place a diagonal line through the next block down. This will indicate final disposition with no further changes in custody. If the sample is being archived the preceding procedure will not be done, as it is possible the sample may have custody changes at a later time.

1.3 SECTION 1 - COLLECTION INFORMATION

1.3.1 In-house Sample Identifier: Enter any special in-house identification system utilized (i.e. tank number, filter number, etc.).

1.3.2 Sample Type/Description: Describe the sample type. Examples are 6L canister, quartz filter, etc.

1.3.3 Sample Source: Enter source. Examples are Velostat Bag, direct, burn pan, etc.. If collected on the ground give appropriate coordinates or designator sequence to allow locating on a map of the test site.

1.3.4 Other information is self-explanatory or referenced in the general instructions (section 1.1).

1.4 SECTION 2 - PRESERVATIVE INFORMATION: This section is self-explanatory or referenced in the general instructions (section 1.1).

1.5 SECTION 3 - SPLIT OR COMPOSITE SAMPLE INFORMATION: You will need to complete additional sample forms with all relevant information entered (sections 1.1.3 and 1.1.4).

1.6 SECTION 4 - SHIPPING INFORMATION

1.6.1 Shipping Date: As per section 1.1.6.

1.6.2 Time: As per section 1.1.7.

1.6.2 By: Signature of person shipping sample.

1.6.3 Shipping Method: Enter method of shipment such as Federal Express, UPS, Hand carried, etc.

1.6.4 Shipping List No.: When shipped, all sample report numbers in a given shipment will be logged on a shipping list form. Enter the number of the shipping list form from the upper right corner of that form.

1.7 SECTION 5 - FACILITY RECEIPT AND STORAGE INFORMATION

1.7.1 Receipt Date: As per section 1.1.6.

1.7.2 Time: As per section 1.1.7.

1.7.3 By: Signature of person receiving shipment.

1.7.4 Type of Storage and Location: Enter the type of storage utilized for the sample. Examples; room temperature cabinet, refrigerator at X°C, freezer at X°C.

1.8 SECTION 6 - DESTINATION LABORATORY/FACILITY INFORMATION

1.8.1 Check the appropriate box to indicate whether the sample is to be analyzed or held for backup/archival purposes.

1.8.2 If sample is to be analyzed then either check the box if no preparation is required or describe on lines provided the preparation done.

2.0 SHIPPING LIST DOCUMENT (Sample form on page 11)

2.1 GENERAL INSTRUCTIONS

2.1.1 Each shipping list (SL) has a pre-printed four (4) digit number.

2.1.2 All samples being shipped between laboratories/facilities shall have a SL completed.

2.1.3 The SL consists of an original and three (3) copies. The distribution is as follows:

Original - Stays with the shipment until received at destination. Once received and lines eleven (11)through fourteen (14) are completed, it is to be returned to ELI at the form return address at the top of the form.

1st Copy - Stays with shipment until received at destination. Once completed is retained by the receiving facility for their records.

2nd Copy - Upon completion of lines one (1) through ten (10) it is retained by the shipping facility for their records.

3rd Copy - To be given or mailed to ELI QA personnel after completion of lines one (1) through ten (10).

2.1.4 Date Entries: As specified in section 1.1.6.

2.1.5 Time Entries: As specified in section 1.1.7.

2.2 INDIVIDUAL ENTRIES BY LINE NUMBER

2.2.1 Line 1: Date of Shipment. Enter date of shipment in format as previously described (section 1.1.6).

2.2.2 Line 2: Preservation. Either check the none required box or describe the preservation done for the shipment.

2.2.3 Line 3: Type of Packing. Describe how the sample(s) are packed. Examples would be ice chest, cardboard box, or footlocker.

2.2.4 Line 4: Shipment Method. Describe shipping method. Examples are hand carried, Federal Express, UPS Overnight, etc.

2.2.5 Line 5: Sample reports included. Check whether the sample collection reports have been included for the samples being shipped.

2.2.6 Line 6: Sending Lab/Facility. Enter the name of the company, lab or facility doing the shipping.

2.2.7 Line 7: Destination Lab/Facility. Enter the name of company, lab or facility the shipment is destined for.

2.2.8 Line 8: Printed Name of Sender. Self explanatory.

2.2.9 Line 9: Signature of Sender. Self explanatory.

2.2.10 Line 10: Sample report numbers. List the ELI identification number from the upper right corner of each sample and custody report.

2.2.11 Line 11: Printed Name of Receiver. Self explanatory.

2.2.12 Line 12: Signature of Receiver. Self explanatory.

2.2.13 Line 13: Name of receiving lab. Enter the name of the receiving lab or facility.

2.2.14 Line 14: Date Shipment Received. Enter the date the shipment was received in the format specified (section 1.1.6).

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ENVIRONMENTAL LABS, INC OB/OD FIELD TEST SAMPLE & CUSTODY REPORT		RETURN TO: Clive D JORGENSEN OB/OD DATA MANAGEMENT - 534 WIBD-Dept of Zoology BYU, Provo, Utah 84602	NO. ELI Split/Composite sample? If yes, complete Section 3

CHAIN OF CUSTODY	SEC
Signature	1
Affiliation	
Date/Time Received	
Signature	2
Affiliation	
Date/Time Received	
Signature	3
Affiliation	
Date/Time Received	
Signature	
Affiliation	
Date/Time Received	
Signature	
Affiliation	
Date/Time Received	
Signature	4
Affiliation	
Date/Time Received	

COLLECTION INFORMATION

In-house sample identifier	Date collected
Sample type/description	Time collected Collected by (print)
Sample Source	Signature

PRESERVATION INFORMATION

Preservation required? If no skip to next section.	
Type of preservative	
Date/Time preserved	By

(Complete this section only for samples from same source).

SPLIT OR COMPOSITE SAMPLE INFORMATION

Split sample? Composite sample? Total samples

OTHER ELI NUMBERS	DESTINATION LABORATORY/FACILITY
ELI	
ELI	
ELI	
ELI	
ELI	
ELI	
ELI	
ELI	
ELI	

SHIPPING INFORMATION

Date/Time	By
Method	List No
Destination	

Continued on reverse side

Signature

Affiliation

Date/Time Received

Signature

Affiliation

Date/Time Received

Signature

Affiliation

Date/Time Received

Signature

Affiliation

Date/Time Received

Signature

Affiliation

Date/Time Received

Signature

Affiliation

Date/Time Received

Signature

Affiliation

5 FACILITY RECEIPT AND STORAGE INFORMATION

Date/Time	By
Type of storage	Location

6 DESTINATION LABORATORY/FACILITY

Sample to be analyzed?	Sample to be held as back-up/archive
If analyzed, preparation required?	None or describe ·

Field Quality Assurance Stamp	Data Management Stamp	Quality Assurance Stamp

NOTES/COMMENTS

ENVIRONMENTAL LABS, INC.

Shipping List For Samples

RETURN ADDRESS: Clive D. Jorgensen, OB/OD Data Management Department of Zoology - 534 WIDB, Brigham Young University Provo, Utah 84602	ELI No. Project Description: OB/OD

FIELD QUALITY ASSURANCE STAMP	1	Date of Shipment		
	2	Preservation	None Required	Describe
	3	Type of Packing		
	4	Shipment Method		
	5	Sample reports included	YES	NO
	6	Sending Laboratory/Facility		
	7	Destination Laboratory/Facility		
	8	Printed Name of Sender		
	9	Signature of Sender		
DATA MANAGEMENT STAMP	10	SAMPLE REPORT NUMBERS INCLUDED IN SHIPMENT		
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
		ELI	ELI	ELI
QUALITY ASSURANCE STAMP	11	Printed name of receiver		
	12	Signature of receiver		
	13	Name of receiving laboratory		
	14	Date shipment received		

Upon receipt at destination mail original to return address and retain first copy for your files.

CHEMICAL RECEIPT, STORAGE AND USE

RECEIVED:		
CHEMICAL SUBSTANCE		
FORMULATION		
QUANTITY		
BATCH OR LOT NUMBER		
FIELD SUPERVISOR	Signature:	Date:
CHEMICAL APPLICATOR	Signature:	Date:
CHEMICAL COORDINATOR	Signature:	Date:
QA SIGNATURE	Signature:	Date:
STORAGE:		
LOCATION OF STORAGE UNIT:		
BY:	Signature:	Date Stored:
USE:		
BY:	Signature:	Date Removed:
QUANTITY USED:		
DISPOSAL:		
QUANTITY REMAINING:		
BY:	SIGNATURE	Date Removed
FIELD SUPERVISOR:	Signature:	Date:
QA SIGNATURE:		Date:

No. ELI 128

Environmental Labs, Inc.

Collection Report

(1) Plot No

(2) Collection Date Day/Mo/Yr

Collection Description

Type of material in sample:

Field Quality Assurance Stamp

History of Sample (Date--Day/Mo/Yr, Time--am or pm)

Time Collected	Date	Time	Technician Signature
Time Preserved	Date	Time	Technician Signature

How Preserved

Time (un)-preserved 1	Date	Time	Technician Signature
Time (re)-preserved	Date	Time	Technician Signature
Time (un)-preserved-2	Date	Time	Technician Signature
Time (re)-preserved	Date	Time	Technician Signature

Where preserved

Data Management Stamp

Sample Preparation for Analysis

Description of Preparation-1:	Date	Technician Signature
Description of Preparation-2.	Date	Technician Signature

Delivered Prepared Sample for Analysis

Name of Analysis Agency:

Signature of Sending Person:	Signature	Date
Signature of Receiving Person:	Signature	Date

Quality Assurance Stamp

Analysis Requested.

Field Supervisor's Signature:

History of Official Sample	1. Sample # 2. Product

3. Laboratory

4. Date Received

5. Received By

6. Received From

7. Sent Via

8. Sample Condition

9. Condition of Seals

10. Sealed By

11. Date Sealed

12. Pieces Received

13. Place Stored

14. Assigned By

15. Assigned To

16. Delivered By

17. Date Delivered

18. Number Subs Received

19. Subs Analyzed

20. Date Seal Broken

21. Date Resealed

22. Resealed By

23. Place Stored

24. Date Jacket Sent Out

25. Remarks

OB/OD SITE VISIT WORKSHEET

Date of site visit (day-month-year): ______________________

QA personnel conducting site visit: ______________________

____

Name of site being visited: ______________________

Address of site being visited: ______________________

____

Name of internal QC Officer: ______________________

Phone number of internal QC: ______________________

LOI status on site (location, access, precision, agrees with actual methods etc. being employed).

____

____

____

____

Field/Lab Sampling: ______________________

____

Field/Lab Analysis: ______________________

____

Instrument/Method Calibration: ______________________

____

Preventive/Corrective Maintenance: ______________________

____

Internal QC Procedures: ______________________

____

Sample Preparation and Storage: ______________________

____

Preparation and Use of Spiked Samples: ____________________

____

Instrument/Equipment Selection and Use: ____________________

____

Determination of Detection Limits/Limits of Quantification:

____

____

Sample Handling and Transportation: ____________________

____

Data Reduction and Analysis: ____________________

____

Accomplish the following:

1. Logbooks next to instruments. With appropriate entries.
2. Obtain Name, address & phone numbers of all personnel working with study material. Check for changes at each visit. Have each person print name and sign same in log book for reference.
3. Obtain diagram of laboratory/building layout.
4. Check data (research journals). Pages intact. Entries in ink only?
5. Lab tracking forms, sample history forms being used?

Comment on the above items by number.

____

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APPENDIX C QUALITY ASSURANCE PROJECT PLAN

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A QA PROJECT PLAN FOR:
OB/OD FIELD TEST, PHASE "C"

APPROVAL: Program Manager original signed Date _____
MacDonald B. Johnson
QA Project Officer original signed Date_______
Gary M. Booth

Project Officer original signed Date_________
Kenneth Jones

Technical Coordinator original signed Date______
Cecil Eckard

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TABLE OF CONTENTS

Distribution of Approved QA Project Plan

1. MacDonald B. Johnson, Program Manager
2. Kenneth Jones, Project Officer
3. Raymond Rhodes, QA Advisor, EPA
4. Gary M. Booth, QA Project Officer, ELI
5. Wayne Einfeld, Coordinator, SNL
6. Kenneth Zahn, Andrulis Research Corporation
7. Cecil Eckard, Technical Coordinator, ARC
8. Chester Oszman, OSW, U.S. EPA
9. Reinhold Rasmussen, Oregon Graduate Center
10. Robert Cary, Sunset Laboratory
11. Robert Giauque, Lawrence Berkeley Laboratory
12. Milton L. Lee, Alpine West Laboratory
13. Wayne Ursenbach, Ursenbach Consulting
14. Daniel LaFleur, Naval Ordnance Station

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SECTION 1. Program Description

1.1. Current estimates of obsolete and/or unsafe explosive materials approach 200,000 short tons. The most appropriate way to dispose of most of these materials is by open burning (OB) and open detonation (OD). Because there is limited empirical data on the generation of particulates and criteria pollutants from OB/OD operations, a well-defined research program is critically needed.

1.2. To date, studies have been completed on a chamber (Bang Box) test, field test phase "A", and field test phase "B". The objective of this study is to identify and quantify the products and residues emitted to the air and to the soil from bulk Composition "B", Explosive "D", and RDX explosives, and certain propellants, and propellant manufacturing residue during OB/OD operations. The data obtained from this study is needed to support environmental assessments and environmental documentation required under the Clean Air Act, Resource Conservation and Recovery Act (RCRA), Clean Water Act, and other Federal/State environmental statutes and regulations. This technology will include instruments, sampling equipment and procedures used aboard a fixed-wing aircraft (FWAC) as well as soil sampling and analysis methods and QA/QC procedures. These tests, referred to as the OB/OD Field Test, Phase "C", will be conducted during August 1990 at Dugway Proving Ground (DPG) in Dugway, Utah.

1.3. This program brings together the expertise of scientists from Alpine West Laboratories (AWL), Sandia National Laboratories (SNL), Sunset Laboratories (SSL), PIXE Laboratory (PL), Lockheed Engineering and Science Co. (LESC), Dugway Proving Ground (DPG), and Andrulis Research Corporation (ARC). All laboratories have prepared Letters of Instruction (LOI) (Appendix I). Instructions for preparing LOI were sent out to all the laboratories during the 2nd week of November 1988 (Appendix II contains a copy of Instructions for Preparing LOI) by Environmental Labs Inc. (ELI). Many of these data collection and analyses require methods development and all analytical methods may not fall under U.S. E.P.A. (EPA) Preferred Methods. Technical QA support from the EPA[1] was provided by the Monitoring and Evaluation Branch of

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[1] Wherever EPA appears throughout this plan, it will refer to: U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Monitoring and Evaluation Branch, Research Triangle Park, NC 27711.

that Laboratory. Complete copies of the Test Design Plan may be obtained from Program Manager, Headquarters AMCCOM, Rock Island Arsenal, Rock Island, Illinois.

SECTION 2. Test Matrix

2.1. The test matrix was tentatively approved by the technical steering committee on 6 February 1990 (Table 1). All test will be conducted in August 1990. However, this schedule may be subject to change depending on the availability of the FWAC and other potential logistic difficulties associated with the project.

Table 1. Phase "C" Test Matrix - Open Air Trials[a]

Number	Type	Configuration	Material	Amount (lb)
1	OB[b]	Surface	Manufacturing Residue	5,000 x 2
2	OB	Surface	Manufacturing Residue	5,000 x 2
3	OD[c]	Surface	TNT	2,000 x 3
4	OD	Surface	TNT	2,000 x 3
5	OD	Surface	RDX	2,000 x 3
6	OD	Surface	RDX	2,000 x 3
7	OD	Surface	Explosive "D"	2,000 x 3
8	OD	Surface	Explosive "D"	2,000 x 3
9	OD	Surface	Composition "B"	2,000 x 3
10	OD	Surface	Composition "B"	2,000 x 3
11	OD	Suspended	TNT	2,000 x 3
12	OD	Surface	M6 Propellant	7,000 x 3
13	OD	Surface	M6 Propellant	7,000 x 3
14	OD	Surface	M6 Propellant	7,000 x 3
15	OD	Surface	M6 Propellant	7,000 x 3
16	OD	Surface	M1 Propellant	7,000 x 3
17	OD	Surface	M1 Propellant	7,000 x 3
18	OD	Surface	M1 Propellant	6,000 x 3

[a] From Detailed Test Plan (DTP) dated July 1990

[b] Open Burn

[c] Open Detonation

SECTION 3. Project Organization and Responsibilities

3.1. All personnel will come from ARC (test design coordination and report preparation), AWL (SFC analysis of exotics), SNL (FWAC air sampling), OGC (6-L tank analysis), SSL (filter carbon analysis), LESC (field QA), DPG (test site coordination), and PL (PIXE analysis). Other scientific consultants may be called on periodically as the project progresses. Quality Assurance will be done by personnel from ELI, LESC and EPA. The EPA Research Monitoring and Evaluation Branch will assist the QA effort by conducting performance audits of the continuous gaseous monitoring instruments, flow rates, and canister sampling.

SECTION 4. QA Objectives for Measuring Data Quality in Terms of Precision, Accuracy, Completeness, and Detection Limits.

4.1. Table 2 summarizes the precision, accuracy, completeness, and detection limit goals by Task. These data are part of the QC program for the laboratories which are described in more detail in Section 10. The values generated in this table were based on the results from the chamber (Bang Box) test, Field Test Phase "A", and Field Test Phase "B".

Table 2. Precision, accuracy, completeness, and detection limit objectives.

Task	Precision/Method	Accuracy/Method	Laboratory completeness[d]	Detection limits
1. Exotic organics particulates)	± 0-148 ng/sample duplicate samples (depends on analyte)[a]	± 50%/audit samples lab standards	90%	0.03-196 ng/mL (soil (depends on analyte)
2. Semi-volatiles (filters)	± 0-103 ng/sample duplicate samples (depends on analyte)[a]	± 50%/audit samples lab standards	90%	0.03-196 ng/mL (depends on analyte)
3. Metals (filters)	± 10%/duplicate samples	± 10%/NIST standards USGS standards	90%	0.1-10 ng/element (depends on analyte)
4. Particulate carbon (filters)	± 0.20-4.68 ng/sq. cm./ duplicate samples	± 1-4%/audit samples sucrose lab standards	90%	0.2 ug/cm^2
5. Volatiles (6-L canisters)	± 20% duplicate samples	± 15-20%/audit samples lab standards	90%	0.1-0.2 ppb
6 Real-time gases (FWAC)	± 15% / duplicates	± 15%/audit samples lab standards	90%	5.0 ppb-1.2 ppm[b]

[a]	DET SOIL	DET FILTER	BURN SOIL	BURN FILTER
2-Nitronaphthalene	0.6 (0.14)-2.59(0.0)*	24(1.4)	1.2(0.071)-2.6(0.07)	
2-4-Dinitrotoluene	0.6 (0.071)-34.7(0.14)	46(0.70)	0.29(0.028)-164(3.5)	
2,6-Dinitrotoluene	1.4(0.14)-11.4(0.35)	5.9(0.28)	131(5.7)-135(0.0)	
2,4,6-Trinitrotoluene	144(11.3)-787(32)	70(2.1)	178(3.5)-180(2.8)	
Naphthalene	612(11)-617(8.5)		2205(148)-2275(35)	
1,3,5-Trinitrobenzene	5.5(0.42)-5.8(0.14)		232(11)	

Phenol	NA[c]	NA[c]-2183(103)	78(0.71)-711(13)	78(0.71)-711(13)

[a] all quantities listed as: x ng/sample (Standard Deviation)

[b] CO_2 1.2 ppm, CO 0.1 ppm, O_3 5.0 ppb, NO_x 6.0 ppb, NO_2 6.0 ppb, NO 6.0 ppb.

[c] NA = Not analyzed.

[d] Laboratory completeness will be expressed as 2 ratios, 1 for collections and 1 for analyses.

SECTION 5. Test Procedures.

Details of the proposed OB/OD Field Test Phase "C" test procedures are outlined in the Test Design Plan (TDP). Of particular interest from a quality assurance standpoint is the collection of background soil samples for each test site and the collection of background air samples and data from background flights of the fixed wing aircraft for each test series.

5.1. Open Burning

5.1.1. A combination of sampling techniques will be used to determine gaseous emissions and particulate generated from open burning. These procedures will collect samples for subsequent laboratory assay and provide real-time/near-real-time analysis. Sampling devices will be sufficiently rugged to withstand the rigors of sampling and sampling platform environments and, to the maximum extent possible, be EPA-certified. Particulate detectors and samplers are described in the DTP.

5.1.2. Residue and fallout samples will be collected and analyzed for each burn. The details of sample pan position, collecting, and handling the samples prior to delivery to the assay laboratory are outlined in the LOI (Appendix B of this Volume). All samples will be individually collected and weighed. In addition, residue from each pan will be weighed and bottled for assay and archiving. "Kickout" pan residue samples are those located 1 m from the burn pans. These pan samples represent propelled parts of the manufacturing waste that fall very close to the burn pan (within 3 m on Phase "A"). The residue from each pan will be weighed and bottled for assay and archiving. Fallout pans are those located on the 6 and 12 m circles around the burn pans as specified in LOI. The residue in each pan will be weighed and bottled for assay and archiving.

5.1.3. Chemical analysis will be conducted by Alpine West Laboratory or other laboratories as required. Procedures used will correspond with those delineated in LOI prepared specifically for the OB/OD study. The laboratory should make every effort to process samples expeditiously. If it is projected that the samples cannot be analyzed within seven days of collection, procedures for preservation shall be implemented by the third day after collection. Preservation and deferred analysis must be approved by the PM. All participating laboratories will be audited by QA/QC personnel.

5.1.4. Particle Induced X-ray Emission (PIXE) is the procedure to be used for metal detection. The following metals will be scanned for: Antimony, Arsenic, Barium, Cadmium, Chromium, Copper, Lead, and Nickel.

5.1.5. Organic analyses will be conducted by post test analytical procedures. A combination of instruments will be used to conduct the analysis at separate laboratory facilities. Instruments to ge used include: Gas Chromatograph (GC), Gas Chromatograph/Mass Spectrometer (GC/MS), and Supercritical Fluid Chromatograph/Mass Spectrometer (SFC/MS),

5.1.6. Details of the test procedure for the FWAC are centered around instrument set-up, calibration, background, and plume sampling. Parameters to be measured by real-time instruments include CO_2, CO, NO_x, O_3, and particulate size distribution. Filters will be used to trap semi-volatile compounds, metals, and particulate carbon. Volatile compounds will be sampled from the plume using 6-L canisters.

5.2. Open Detonation

5.2.1. Detonation sites will be selected by DPG in an area virgin to detonation material. Each detonation point will be separated by at least 200 m to avoid fallout from airborne particulate.

5.2.2. There will be seven detonations, one ORI and six additional detonations. Independent sampling will be accomplished for each detonation except for the quartz filters on the aircraft. The quartz filters will be used for one detonation on the ORI and for the composite of three detonations on the tests. The depositing of particulate from the three detonations will provide a greater particulate mass for extraction and thus enhance detection of trace organics.

5.2.3. Open detonation procedures will parallel those used at those depots and explosives/munitions manufacturing plants designated for disposal of explosives and munitions.

When variance is found between these procedures, the PM will, upon request or recommendation of the project officer, select the procedure(s) to be tested.

5.2.4. Soil sampling will consist of pre-test core samples and post-test ejecta samples. The fallout sampling will be from pans placed concentric circles.

5.2.5. Multiple Detonations at the Same Site

5.2.5.1. The first TNT detonation will be followed by ejecta soil sampling. The crater will then be filled with soil ejecta. A second TNT detonation at the same site will be followed by ejecta soil sampling. The crater will then again be filled in with soil ejecta and a third detonation with ejecta soil sampling will be accomplished. The time between detonations will be as short as possible (time required to sample, fill crater and set the new explosive charge).

5.2.5.2. Soil sampling will consist of pretest core samples and the post test ejecta samples. The pretest core samples will only be taken prior to the first detonation. There will be a post test sample after each detonation. The ejecta soil sampling grid is described in DPG LOI.

5.2.5.3. Analytical procedures are the same as those described under the open detonation section.

SECTION 6. Test Samples

6.1. Sample Custody

6.1.1. Samples collected during Phase "C" will be assigned consecutive serial numbers based upon the type of sample. These consecutive serial numbers will be assigned to the samples in the field by the designated individuals. For the soil samples that would be the representative of LESC. For the FWAC samples, it will be the SNL representative. In addition, LESC has provided a sample numbering key to explain the numerical scheme to identify all soil, fallout pan, and sputter pan samples. This is referred to as an EPA number on the sample container labels.

6.1.2. Each sample shall have a sample report/custody form completed by the responsible party. The sample, and properly completed paperwork will be delivered to the ELI QA representative for shipment to the appropriate laboratory for analysis or further sample preparation (splitting, extraction, etc.). In the event of sample splitting the laboratory doing this shall prepare additional sample reports as delineated in the OB/OD sample report instructions.

6.2. Storage of Unused Samples and Unused Sample Extracts

All excess sample material not sent to the laboratories for analysis will be properly labeled and archived in a freezer at DPG. Sample material includes all particulate filters, soil, fallout, sputter pan, and residual materials. After extraction and analysis, all remaining individual extracted samples and unused extracts will be properly labeled and properly stored in a freezer at AWL or OGC. All of the above-mentioned materials will be kept properly stored in freezers until the program is completed, i.e. until the final report has been issued, or earlier if so directed by the program manager.

SECTION 7. Calibration Procedures

All continuous monitors for the appropriate gases will be calibrated according to the manufacturers' recommended procedures. Calibration curves for selected organic exotics and inorganics will also be prepared. Quality criteria for the appropriate calibration curves are outlined by each laboratory and recorded as part of their LOI. Depending on the laboratory and where appropriate, correlation coefficients and/or tables of residuals may accompany each set of data. The frequency of calibrations are noted as part of the LOI. In addition, technical instruments such as balances and chromatographs are calibrated periodically within specified time periods documented in LOI. The quality criteria for calibration and maintenance of the equipment will be maintained by the individual laboratory's QC program. Daily logbooks will be kept on all instrumentation.

SECTION 8. Data Management and Archiving of Raw Data

All of the data collected during the QC checks and the field investigation including all calibration data and logbook data, will be sent directly to the Data Management Center (DMC) at ELI. This includes all appropriate summary data to be used in the final report. This will include one complete set of data of all laboratory analyses, and a separate set of data listing only the QC/Audit samples analyzed by the laboratory. The DMC will be responsible for copying these data and sending the originals to a final QA storage area. Each data page will be stamped by the QA DMC and the QA records custodian for final storage. When the entire study by a given laboratory is completed and the data sent to ELI and stamped, copies of these data will be turned over to DPG Project Officer. The originals will be placed in the Quality Assurance Unit (QAU). Complete copies of the data will be eventually stored in the archiving facility at the Smith Family Living Center at Brigham Young University, or other appropriate facilities agreed upon by the Program Manager. It is also recommended that all data be sent to ELI on an IBM compatible disk. Magnetic tapes are also acceptable for storage of raw data; however, back-up discs of all raw data are strongly encouraged whenever possible along with the magnetic tapes.

SECTION 9. Data Analysis, Validation, and Reporting

9.1. Data analysis will be the responsibility of ARC. The data received from each laboratory will be received by ELI DMC and then sent to ARC. Upon receipt of the data, sample custody forms will be filled out and signed by both the QA and ARC personnel. Once the final report is completed, a QA team (composed of EPA and OB/OD QA personnel) will meet with ARC and spot check the raw data against that which is found in the final report and to evaluate the QA/QC data. Upon satisfactory completion of the QA audit and evaluation, a formal QA statement will be provided and placed in the final report.

SECTION 10. Internal Quality Control Checks, Audit Samples, and Frequency

10.1. Each laboratory has an internal QC program (Table 3). These QC programs will be documented in the form of LOI. Basically, these are a listing of the operational checks, the control limits for initiating corrective action, and the planned corrective action. Examples of items that are included:

10.1.1. Replicates
10.1.2. Spiked samples
10.1.3. Split samples
10.1.4. Control charts
10.1.5. Blanks
10.1.6. Internal standards
10.1.7. Quality control samples
10.1.8. Calibration standards and devices

Table 3. Quality Control , Audit Checks, and Criteria

Measurement	Type of procedures	Frequency	Criterion
Exotic organics (soil/particulates)	soil blank	approximately 10%	± 0-150 ng/sample
	duplicate soil samples 1 spiked, 1 not	once	± 50% of known conc.
	extraction efficiency (2x)	1 sample/test series	analyte recovery
	soil spikes (in-house)	3 at high conc.	± 50% of known conc.
		3 at low conc.	± 50% of known conc.
	soil spikes (EPA)	five	± 50% of known conc.
	temperature storage/ soil spikes	2 at beginning	± 50% of known conc.
		2 at mid-test	± 50% of known conc.
		2 at end	± 50% of known conc.
	temperature storage/ solvent spikes	2 at beginning	± 50% of known conc.
		2 at mid-test	± 50% of known conc.
		2 at end	± 50% of known conc.
	triplicate soil samples from same container (split samples)	once	± 0-150 ng/sample
	repeated aliquots from same soil extract	every sample	± 50% of known conc.
	standard solution of analytes	daily	± 3s from mean
	instrument calibration	daily	instrument specs
	calibration plots	once	$r^2 > 0.9$

Table 3. (continued)

Measurement	Type of procedures	Frequency	Criterion
semi-volatiles (filters)	weighing filter blanks	approximately 10%	± 3s
	weighing FWAC filter blanks	100%	± 3s
	filter spikes (in-house)	3 at high conc. 3 at low conc.	± 50% of known conc. ± 50% of known conc.
	extraction efficiency (2x)	1 filter/ test series	analyte recovery
metals (filters)	filter blanks	10%	± 10% of known conc.
	filter spikes (in-house)	3 at high conc. 3 at low conc.	± 10% of known conc. ± 10% of known conc.
	filter spikes (EPA)	once	± 10% of known conc.
	split samples (filters, solvent extracts, ash samples)	10%	± 10% of known conc.
particulate carbon	internal CH_4 standard	every sample	5 ± 5%
	sucrose spike (in-house)	3% (single-point) 1% (multi-point)	± 5% of known conc. ± 5% of known conc.
	duplicates	10%	± 5% of known conc.
	instrument blanks	3%	"zero-check"
	EPA spike	once	± 1-5%
	filter blanks	2	"background"

Table 3. (continued).

Measurement/Instrument	**Type of procedures**	**Frequency**	**Criterion**
volatiles (6-L canisters)	GC-FID calibration with neohexane	daily	± 2% of known conc.
	NIST VOC standard	once	15-20% of known conc.
	duplicate samples	10%	± 2% of known conc.
	EPA spike	several	± 5% of known conc.
real time gases	performance audit	once	± 15% of known conc.

10.1.9. Reagent checks

10.2. Of particular importance is the use of control charts or tables (with the data entered chronologically) which should be maintained on each appropriate unit of equipment for the entire project. These charts show quantitatively the degree of variation of an instrument through time, and they provide a permanent record of this variation. Table 2 lists the QC/audit checks and the criteria for acceptable performance limits. Details of these internal QC programs are provided in the attached LOI (Appendix B).

10.3. The sections below present summaries of the QC/audit procedures for each task in the Field Test Phase "B" study.

10.3.1. Exotic Organics (Soil and Particulates). Analysis of the exotic organics from soil and particulates will be under the direction of Dr. Milton L. Lee of AWL. A QC program is provided in an LOI in Appendix B and will be supervised by Dr. Karin Markides.

10.3.2. Soil blanks will be analyzed at the approximate frequency of 1 in every 10 field samples (10%). These samples will be analyzed in an identical manner to the field samples. The source of these blank samples will be from an uncontaminated site at DPG. Precision for the blanks will be reported.

10.3.3. Accuracy will be assessed using several techniques. Six soil samples will be spiked with a cocktail of analyte standards, 3 at a high concentration and 3 at a lower concentration. The results of these QC analyses must have a percentage recovery within ± 50% of the expected value. If these results fall outside the ± 50% range, then a technical review and evaluation of the data will be conducted and a decision will be made on the appropriate corrective action that is needed. It should be noted that percent recoveries will be reported but no adjustments will be made based on percent recoveries.

10.3.4. Also five external performance audit soil samples from AWL will be spiked by EPA with an analyte cocktail and sent to AWL for analysis. The results of these performance audit samples must show a percentage recovery within ± 50% of the expected value. Since the results of the

Phase A test showed ng levels of the various analytes, all in-house spiking experiments will be done at ng levels. If percent recoveries of the EPA QA performance audit samples are outside ± 50% of the expected value, then the sample results will be reviewed and evaluated and an appropriate corrective action will be taken. Duplicate test soil samples will be taken from the field. One of these will be analyzed and based on the organics found, the other sample will be spiked with the "found" compounds and extracted and analyzed. The results should show a percent recovery that is within ± 50% of the expected value. These experiments will also be technically reviewed if the results are outside these limits and an appropriate corrective action will be taken.

10.3.5. To evaluate extraction efficiency, one real sample from each test series (detonation or burn) will be extracted twice with acetonitrile. Based on the results, additional extractions of other samples may or may not be necessary. There will be no need to evaluate different extracting solvents since acetonitrile has already been demonstrated in the literature (Jenkins and Grant, 1987) to be the preferred solvent.

10.3.6. Temperature storage of the analytes from soil/particulate material will be evaluated by spiking 6 soil/particulate samples with an analyte cocktail. Two samples will be analyzed at the beginning of the OB/OD project, 2 at mid-test, and 2 at the end of the test. All results should show a percent recovery that is within ± 50% of the expected value. These experiments will be reviewed if they fall outside this range and an appropriate corrective action will be taken. Six spiked acetonitrile solutions will be stored under identical conditions to the soil/particulate samples and evaluated in a similar manner. Control charts for temperature will be kept on the cooling/freezer units to record and evaluate temperature variation through time.

10.3.7. Precision from the test soil samples will be estimated by 2 different methods. First, repeated aliquots from the same soil extract will be injected into the SFC/MS (and a few GC/MS) system to get an estimate of the analytical error (imprecision). These values should be within 10% of each other. Secondly, 3 soil samples from the same sampling container will be extracted and analyzed separately for each test series. Precision for these soil samples should be similar to those in Table 2.

10.3.8. Quality control for the SFC/MS and GC/MS system will be conducted in 4 different ways.

10.3.8.1. A standard solution of analytes will be run daily and compared with the calibration plots to ensure adherence to the calibration plot. These values will be plotted on control charts chronologically. If the values deviate or drift significantly from the calibration value ± 3s, then the analyte solution and calibration will be redone.

10.3.8.2. Matrix blanks (obtained from the same area before testing) will be run approximately every 10 samples.

10.3.8.3. Instrument calibration will be conducted daily prior to analyzing samples.

10.3.8.4. Calibration plots for the target analytes will be prepared at the start of the study. From these plots, the detection limits will be calculated for each target analyte. These detection limits will be similar to those in Table 3.

10.3.9. Semi-volatiles (filters). Analysis of the semi-volatile organics from the filters of the FWAC will be by the same AWL scientists documented in the previous section. A QC program is outlined in the same LOI as for the soil/particulates (Appendix B).

10.3.9.1. The QC program for the filters is described for each of the operations of filter weighing, filter extraction, and filter storage. Discussions below follow in order of blanks, accuracy, precision, and temperature storage as they relate to the above operations.

10.3.9.2. Stored filter blanks will be weighed at the rate of approximately 1 in every 10 field test filters (10%). All filters which have been treated as true controls (e.g. "handling" filters on the FWAC or solvent-control spiked filters) will be weighed prior to using in the FWAC. Each blank filter will be weighed or extracted in the same manner as the test filters. As part of the QC weighing process, a certified National Institute for Standards and Technology (NIST) standard weight (about the same weight as a filter) will be weighed daily and the results plotted chronologically on a control chart to ensure correct operation of the analytical balance. A control chart will also be kept on the daily weights of the blank filters stored under the same conditions as the test filters. All filter weighings will be repeated at least 3 times (3 independent weighings) to get an estimate of precision. Precision estimates for the blank filters will be reported as standard deviations.

10.3.9.3. Accuracy for extraction of analytes from the filters will be assessed in the following manner. Three unexposed filters will be spiked with a high concentration of analytes and 3 with a lower concentration of analytes. All percent recovery results should be within ± 50% of the amount added. If these values fall outside these limits, then an appropriate corrective action will be needed.

10.3.9.4. Precision estimates of the filter weights will be obtained by weighing each series of test filters to get an idea of variation (precision) in collection. Also each filter (from one set) will be extracted separately to get some idea of variability due to extraction and analysis. In addition, 1 test filter from each test series will be extracted twice to evaluate extraction efficiency. Precision for these filter samples should be similar to those in Table 2.

10.4. Metals. Metals ranging from Al to Pb, excluding the rare earths, will be evaluated in filter samples from the FWAC and acetonitrile extracts. These data will be collected using PIXE analysis under the direction of Dr. Nolan Mangleson: Details of the PIXE QC program are given in the LOI (Appendix B). As part of the QC program, it may be useful to analyze old filters and extracts of filters from Field Test Phase A.

10.4.1. Unexposed blanks will be analyzed at the rate of 10% similar to that done at AWL. These samples will be analyzed in an identical manner to the test filters. Precision on the blanks will be ± 10% using duplicate filters. This precision level criterion is identical to that for the test filters.

10.4.2. Accuracy will be determined by spiking 3 replicate high and 3 replicate low concentrations of NIST standard solutions. The results of these QC samples must have percent recoveries that are within ±10% of the amount added. If the data do not come within these limits, then the experiment will be evaluated and an appropriate corrective action will be taken. If available, the second method of assessing accuracy may be by analyzing an EPA audit filter. These data must also come within the prescribed percent recovery of ± 10% of the amount added. NIST trace-element urban air particulate standards and USGS rock standards will be used as solid matrix standards for the ash samples.

10.4.3. Precision will be determined by repeated analysis on duplicate filter samples, acetonitrile extracts, and ash samples from burn pans.

10.4.4. Detection limit criteria will be 0.1 - 10 ng depending on the element.

10.5. Particulate Carbon (filters). Particulate carbon from the filters will be analyzed using a thermal-optical method at SSL. This method separates organic carbon from elemental carbon. This laboratory is under the direction of Robert Cary and represents a one-man operation. His QC program consists of a 7-step process. These are summarized below, and additional details are found in the LOI (Appendix B).

10.6. Volatiles (6-L Canisters). Volatile organics are trapped in stainless steel 6-L canisters mounted in the FWAC. Analysis of these volatile organics is under the direction of R.A. Rasmussen at OGC. A QC program is outlined in an LOI in Appendix B. The QC program consists primarily of GC-FID calibration on a daily basis using neohexane as the calibrant. Results are expected to fall within ± 2%; if not, then additional analyses will be conducted until the ± 2% is reproducible.

10.6.1. Accuracy will be assessed by analyzing VOC standards from NIST and audit cylinder from EPA. Percent recovery results should come within 15-20% of the amount added, or corrective action of repeat samples may be recommended. It will also be noted that 1 duplicate will be spiked to see if the spike is recovered.

10.6.2. Precision will be assessed by taking duplicate samples and recording expected results of ± 20%

10.6.3. Detection limits for C_3 to C_9 compounds will be 0.1-0.2 ug/m^3 depending on the compound.

10.7. Real-time gases (CO, CO_2, O_3, NO_x). All real-time gases will be conducted on equipment mounted within a FWAC. An LOI (Appendix B) contains extensive detail on gas and aerosol instrument calibration as part of their QC program. Real-time gas analysis is under the direction of Mr. Wayne Einfeld.

10.7.1. A performance audit will be performed on the majority of the instruments aboard the FWAC while at the Provo airport. The test will not continue until all instruments come within an accuracy of ± 15-20% from the known amounts of audit samples.

10.7.2. In addition to the performance audit, daily single point checks on the instruments performance will be carried out using certified gases and by completing multi-point calibration curves at least once. All of the accuracy data must fall within ± 15% of the known amount, or corrective repeat action must be completed.

10.7.3. Precision estimates from the same day should fall within ± 15%.

SECTION 11. Performance and System Audits

11.1. Personnel from ELI will visit AWL and other labs (except SNL which will be audited at DPG) at least once while analyses are being performed to monitor and assess the capability and performance of all instrumental and analysis systems, and assess the adherence to approved procedures. The Laboratory Director will be contacted prior to the visit. A Quality Assurance Unit Inspection Form (OB/OD Site Visit Worksheet and QA check-lists), such as shown in Appendix V, will be used by the QA auditor. These completed forms shall be used to write a formal letter to the Principal Investigator and a copy of this letter will be sent to the OB/OD Program Manager. A copy of the audit review will be retained in the DMC and the QA storage file.

11.2. In addition, an EPA Audit-Team will provide a Performance Audit on the real-time instruments from the FWAC at Provo airport at Rocky Mountain Helicopter. The measurements will include the parameters NO_x, NO_2, NO, CO, CO_2, 03, and sampler flow.

SECTION 12. Preventive Maintenance

Preventive maintenance tasks and schedules recommended by the manufacturers of the gas analyzers, chromatographs, mass spectrometers, and other technical equipment will be followed. Spare parts such as detector heads, septa, columns, and cylinder gases should be maintained on-hand during the project for daily checks and recalibrations. These procedures will be spelled-out in the LOI.

SECTION 13. Specific Procedures to be used to Routinely Assess Data Precision, and Accuracy

13.1. There are routine procedures used to assess the precision and accuracy of the measurement data. If appropriate, these procedures will include the equations to calculate precision and accuracy, and the methods used to gather data for the precision and accuracy calculations.

13.2. Examples of statistical procedures to be selected by the laboratories are central tendency and dispersion, measures of variability, significance tests, confidence limits, and testing for outliers. Adjustments for outlier removal will be under the direction of TSC.

13.3. Procedures for determining detection limits will be specified by the laboratories in their LOI.

SECTION 14. Quality Assurance Reports

14.1. A report, covering the activity for each laboratory, will be provided to Program Manager and Laboratory Manager. These reports may include:

- Assessment of measurement data accuracy, precision, detection limits, and completeness.
- Results of performance audits
- Results of system audits
- Significant QA/QC problems and recommended solutions

The ELI QA Unit will be responsible for preparing these reports. And, as already mentioned, a separate QA section will be provided in the final report which summarizes data quality information contained in the reports.

SECTION 15. Summary

The goal of this QA Project Plan is to provide the policies, objectives, functional activities, and specific QA/QC activities associated with the LOI and which are designed to achieve data quality. There are three objectives to this QA Plan: (1) to insure the quality of the field-collected data, (2) to insure an appropriate comparison of the SFC/MS and GC/MS procedures, (3) to provide QA/QC procedures for the overall study. An important part of the QC aspect of the QA Project Plan is for each of the laboratories to submit LOI to the QA Unit in order to establish written, documented procedures to be used by each laboratory. These LOI should establish the calibration procedures, frequencies, and the routine QC procedures. The flow of information will come from each parent laboratory to the Data Management Center to a QA storage file to DPG, then back to the QA Unit, and finally to the Program Manager. During the work effort, there will be a site visit to all laboratories to insure QA/QC compliance. SNL will have all of their equipment contained in the FWAC. Therefore, while the plane is at Provo airport, a Performance Audit will be completed in the field both before and after the project.

Glossary of Terms

Audit - A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system, or (2) system audits of a qualitative nature that consist of an on-site review of a laboratory's QA/QC system and physical facilities for sampling, calibration, and measurement.

Data Quality - The totality of features and characteristics of data that bear on their ability to satisfy a given purpose. The characteristics of major importance are mainly accuracy, precision, and completeness. These characteristics are defined as follows:

1. 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. Various measures of precision exist, depending upon the "prescribed similar conditions."

2. Accuracy - The degree of agreement of a measurement, X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio, X/T.

3. Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Data Validation - A systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

Performance Audits - Procedures used to determine quantitatively the accuracy of the total measurement system or component parts thereof.

Quality Assurance (QA) - The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Assurance Project Plan - An orderly assembly of detailed and specific procedures which delineates how data of known and accepted quality are produced for a specific project.

Quality Control (QC) - The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Often, QC procedures are referred to as those standards of performance that are checked within a laboratory.

Letters of Instruction (LOI) - Written documents which generally outline an operation, analysis or action whose mechanisms are generally prescribed and which are commonly accepted as the methods for performing certain routine or repetitive tasks. The LOI are more general than the Standard Operating Procedure (SOP).

Chain of Custody - A procedure for preserving the integrity of a sample or of data (e.g. a written record listing the location of the sample/data at all times).

References

Jenkins, Thomas F. and Clarence L. Grant. 1987. Comparison of extraction techniques for munitions residues in soil. Anal. Chem. 59: 1326-1331.

Rhodes, R. C. 1988. Quality Assurance Project Plan for the Integrated Air Cancer Project (IACP) - Study Roanoke, Virginia. EPA Contract Numbers 68-02-4550. The Quality Assurance Division Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27709.

Taylor, J. K. 1987. Quality Assurance of Chemical Measurements. Lewis Publishers, Inc.

U.S. Army Dugway Proving Ground. Sept. 1989. Detailed Test Plan Field Test Phase "B" for the Study of Open Burning/Open Detonation. Dugway, UT.

U.S. Environmental Protection Agency. Dec 1984. Quality Assurance Handbook for Air Pollution Measurement Systems: Volume I. Research Triangle Park, NC.

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APPENDIX D. QUALITY ASSURANCE AUDIT REPORTS

ENCLOSURE	TITLE
1.	Memorandum, 19 July 1989, Floyd W. McMullin, subject: Site Visit - AWL 17 July 1989
2.	Letter MD-77B, July 31, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed report of CO and CO_2 Audits
3.	Letter MD-77B, August 24, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment laboratory, Research Triangle Park, North Carolina, w/enclosed results of spiked soil samples.
4.	Letter, September 21, 1989, Alpine West Laboratories, Provo, Utah, w/enclosed results of spiked soil samples.
5.	Letter MD-77B, November 1, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.
6.	Memorandum, 20 January 1990, Floyd W. McMullin Jr, subject: Site visit to SSL on 17 January 1998 [sic].
7.	Memorandum, 20 January 1990, Floyd W. McMullin Jr, subject: Site visit to OGC on 18 January 1998 [sic].
8.	Memorandum, 22 January 1990 from Floyd W. McMullin, Jr., subject: Site Visit to SNL (AT Dugway) on 19 October 1989 [sic].
9.	Letter MD-77B, January 26, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed EPA audit report for [Phase B].
10.	Letter MD-77B, February 8, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.
11.	Memorandum, 09 February 1990, Todd D. Parrish, subject: Site Visit - AWL 09 February 1990.
12.	Memorandum, 9 February 1990, Floyd W. McMullin Jr., subject: AWL audit pertaining to EPA QAD deficiencies [sic].

13. Letter MD-77B, February 13, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed benzene audit report.

14. Memorandum, 22 February 1990, Todd Parrish, subject: Addendum to the site visit to AWL, 09 February 1990 [sic].

15. Letter MD-77B, March 13, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

16. Memorandum, Rhodes, 20 April 1990, U.S. Environmental Protection Agency, Atmospheric Research and exposure assessment Laboratory, Research Triangle Park, North Carolina.

17. Letter MD-77B, September 7, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

18. Letter, Raymond C. Rhodes, December 9, 1990, Raleigh, North Carolina.

19. Letter, February 19, 1991, Environmental Quality Assurance Management, Inc, Raleigh, North Carolina.

MEMORANDUM, 19 JULY 1989, FLOYD W. MCMULLIN, SUBJECT: SITE VISIT - AWL 17 JULY 1989.

ENCLOSURE 1

MEMORANDUM

TO: Environmental Labs Inc.

ATTN: Dr. Gary M. Booth

FROM: Floyd W. McMullin

DATE: 19 July 1989

RE: Site Visit - AWL 17 July 1989

ENVIRONM[illegible]BS INC.
FIELD OFFICE
RECEIVED
AUG 04 1989
BY
QUALITY ASSURANCE UNIT

This material is primarily that which is recorded in my logbook. The site visit followed EPA recommended guidelines.

LOI status on site: LOI being utilized are those as previously submitted and on file. The listing of current LOI are:

LOI 1 - 19 May 89 - General laboratory procedures
LOI 2 - 19 May 89 - Preparation and handling of XAD-2 resin traps, filters, and VOST trains.
LOI 3 - 19 May 89 - Rotary evaporator operation.
LOI 4 - 19 May 89 - Preparation and handling of quartz fiber filters.
LOI 5 - 19 May 89 - Preparation and handling of 32 litre tank extracts.
LOI 6 - 27 Jun 89 - Procedures of analysis of miscellaneous
LOI 7 - 26 May 89 - Supercritical Fluid Chromatography/Mass Spectrometry (SFC/MS) analysis of OB/OD samples.
LOI 8 - 26 May 89 - Gas Chromatography/Mass Spectrometry (GC/MS) analysis of OB/OD samples.
LOI 9 - 26 May 89 - Procedure for the extraction of adsorbed residues from soil.
LOI 10 - 26 May 89 - Procedures for the extraction and analysis of open burn samples.

It is noted that the above LOI have replaced the LOI of late 1988 and early 1989. However, the content and subject matter have been altered such that previously assigned LOI numbers may bear no relationship to the current ones. This may result in creating confusion.

The LOI in use are maintained in a 3-ring binder and are readily accessible for review. So far as I am able to determine the LOI are being followed as written.

Field/Lab Sampling: AWL is no longer supplying personnel for field sampling. Their work is now confined to the laboratory. Their current sampling techniques conform to the current LOI.

Lab Analysis: As specified in current LOI.

Instrument/Method Calibration: As described in LOI 7.

Preventive/Corrective Maintenance: As specified by equipment manufacturers.

DATA [illegible]
AUG 04 1989
REC. BY

Internal QC Procedures: Internal spikes are routinely introduced into extracted samples (LOI 9). These consist of 300 pg of 1-nitronapthalene-d_7 and 240 pg of 9-phenylanthracene.

Sample Preparation and Storage: Sample preparation is as per LOI. Extracted samples are stored in the lab at -20° C. It is noted that unprocessed samples are stored in another building at approximately 0° C. Additionally, access to the unprocessed samples is not controlled. The walk in freezer where they are stored is accessible to anyone in the building. Consideration should be given to obtaining some type of lockable container to keep these samples in, in the freezer.

Preparation and use of Spiked Samples: Currently spiked samples are prepared in-house and run every other day as part of the normal calibration equipment checks. No blind spikes are being analyzed at this time.

Instrument/Equipment selection and use: As specified in LOI.

Determination of detection limits/limits of quantification: Quantification is per sample by internal standards. This is specified in data for individual samples.

Sample handling and storage: Covered in previous section.

Data reduction and analysis: This is done by professional evaluation of the spectrographs and other accumulated information.

Miscellaneous:
1. A logbook is maintained next to the equipment. Utilized to record information pertaining to runs and data storage.
2. Only personnel working with study material at this time are Dr Christine Rouse and Dr. Milton Lee, both of AWL. Phone contact numbers are (801)378-4466 and (801)378-4338 respectively.
3. Laboratory journals are maintained utilizing standard lab notebooks which are bound and have numbered pages. All entries are in ink, and the journals appeared intact.

Floyd W. McMullin
Quality Assurance

Letter MD-77B, July 31, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed report of CO and CO_2 Audits.

ENCLOSURE 2

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

July 31, 1989

Mr. Dennis J. Morrison
Division 6321
Risk Assessment and Transportation
System Analysis
Saudia National Laboratories
P.O. Box 5800
Albuquerque, NM 87185

Dear Dennis:

Enclosed are the results of our recent CO and CO_2 audits. The agreement with our CO audit cylinders was quite good, but the CO_2 results could have been better. While we did not test the entire range of your equipment, I feel that the test points should have been more along the straight line regression. We will attempt to have a different set of CO_2 standards for our October audit at Dugway. This audit still had less than 15 percent error which is considered acceptable.

If you have any questions, please call me at 919-541-2205.

Sincerely,

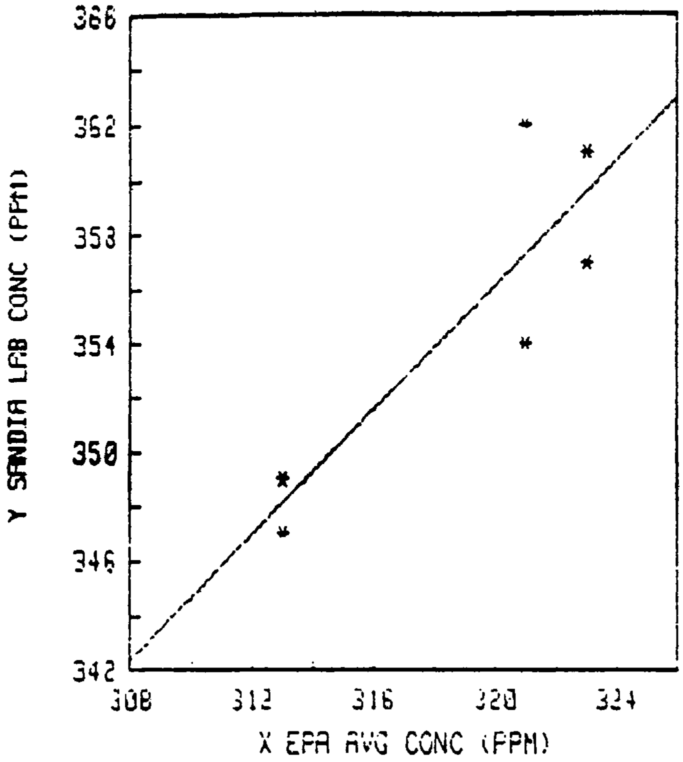
Bill

William F. Barnard
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Enclosure

cc: Wayne Einfeld, Saudia Labs
Don Johnson, U.S. Army
William J. Mitchell

SANDIA LAB CONC VS. EPA CONC

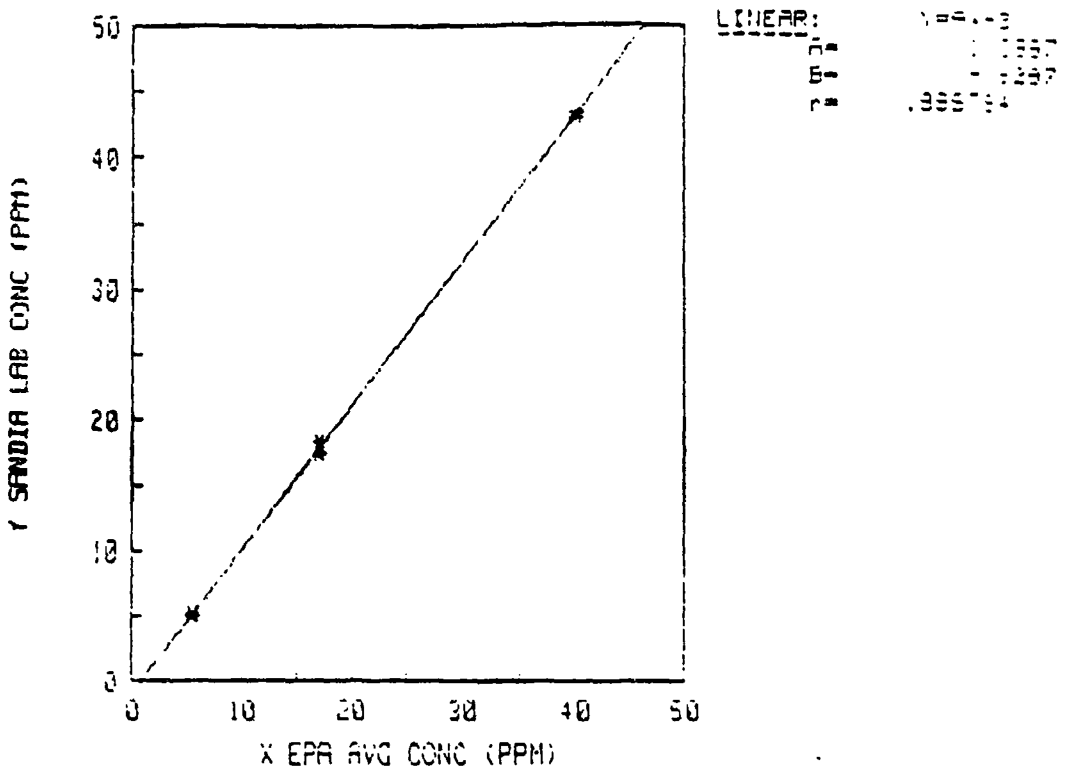


LINEAR: Y=AX+B
A= 1.1429
B= -9.5714
r= .877454

DATA:

No.	X	Y
1	313	349
2	313	347
3	323	357
4	321	354
5	321	362
6	323	361

SANDIA LAB CONC VS. EPA CONC



DATA:

No.	X	Y
1	5.41	5.27
2	5.41	5.00
3	40.03	43.00
4	17.05	17.40
5	17.05	18.40
6	40.03	43.40

Letter MD-77B, August 24, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment laboratory, Research Triangle Park, North Carolina, w/enclosed results of spiked soil samples.

ENCLOSURE 3

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

August 24, 1989

Mr. Mac Donald Johnson
c/o Commander
HQ-AMCCOM
AMSMC-DSM-D
Rock Island, IL 61299

Dear Don:

I received Alpine West Laboratories' (AWL) results for the soil samples we spiked with seven OBOD target compounds. The results were faxed to me on August 15, 1989. AWL analyzed the five soil samples using SFC-MS and stated that they were preparing to analyze the samples by GC/MS.

We placed known amounts of the seven OBOD target compounds on the soil samples in early December 1988 in the following way. First, each compound was weighed and placed in the same flask. The mixture was then dissolved in acetone to yield a solution containing between 18 and 29 micrograms per ml. An aliquot was then taken and placed in 75 to 125 ml of acetone. The entire volume of this latter solution was then added to a soil sample obtained at Dugway Proving Grounds. The soil/acetone slurry that resulted was placed on a rotary evaporator and the acetone removed by evaporation. After the acetone was removed, the soil sample was placed in a glass jar and sealed. Eight samples, four at one level and four at another level of OBOD target compounds, were spiked in this manner. Two other soil samples were spiked using only acetone to serve as blank or control samples.

AWL received two samples of one level, two of the other level and one control soil sample. Each sample was marked with a unique number so the analyst could not determine a spiked sample from the blank. Samples containing the same quantity of OBOD compounds were also given a unique number so that the analyst would not realize they were duplicates.

The results from AWL showed excellent recovery of the spikes using the SFC-MS technique. This technique shows a lot of promise and AWL is to be complemented on their proficiency with SFC-MS. The results also show that the samples were stable (as expected), since they were extracted and analyzed eight months after they were spiked. The precision obtained by AWL was also excellent as shown by their agreement on the duplicate samples.

AWL's results are shown in Tables 1 and 2. The true value (spiked value) and the % difference of the reported value from the true value are also presented. AWL did not detect any of the target compounds on the control (blank) sample.

I have not received any results yet from Batelle Columbus. Batelle received an identical set of samples. I'm assuming that Batelle will not submit results and therefore am releasing the true values to you.

Sincerely,

Bill Mitchell

William J. Mitchell, Ph.D.
Chief, Research and Monitoring
  Evaluation Branch
Quality Assurance Division (MD-77B)

Attachment

cc: H. Crist
    K. Caviston, NSI

Table 1. Results for Duplicate Samples (in Total Micrograms) for Level 1

	AWL Results			EPA True Value	% Diff
	4SA	1SA	AVG		
Phenol	7.0	7.0	7.0	7.2	-2.8%
N-nitrosodiphenylamine	5.6	5.6	5.6	5.5	1.8%
Benz-a-anthracene	5.8	5.8	5.8	5.8	0.0%
Benzo-a-pyrene	7.0	7.0	7.0	6.8	2.9%
Dibenz-a,h-anthracene	5.2	5.2	5.2	5.1	1.9%
2,4 dinitrophenol	4.5	4.6	4.6	4.6	0.0%
4-nitrophenol	5.6	5.6	5.6	5.5	1.8%

Table 2. Results for Duplicate Samples (in Total Micrograms) for Level 2

	AWL Results			EPA	
	45A	15A	Avg	True Value	% Diff
Phenol	36	35	35	36	-2.7%
N-nitrosodiphenylamine	28	28	28	28	0.0%
Benz-a-anthracene	29	28	29	29	0.0%
Benzo-a-pyrene	36	36	36	34	5.9%
Dibenz-a,h-anthracene	26	26	26	25	4.0%
2,4 dinitrophenol	23	22	23	23	0.0%
4-nitrophenol	28	28	28	28	0.0%

SAME AS EPA LETTER 1SA - LEVEL 1

SFC-MS of EPA Spiked Soil Sample 1SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	6.9	7.0	7.0	0.07
N-Nitrosodiphenylamine	5.6	5.7	5.6	0.07
Benz[a]anthracene	5.8	5.8	5.8	0.0
Benzo[a]pyrene	7.0	7.1	7.0	0.07
Dibenz[a,h]anthracene	5.1	5.2	5.2	0.07
2,4-Dinitrophenol	4.6	4.6	4.6	0.0
4-Nitrophenol	5.5	5.6	5.6	0.07

GC-MS of EPA Spiked Soil Sample 1SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	7.0	7.0	7.0	0.00
N-Nitrosodiphenylamine	5.6	5.6	5.6	0.00
Benz[a]anthracene	5.7	5.6	5.6	0.07
Benzo[a]pyrene	7.0	7.0	7.0	0.00
Dibenz[a,h]anthracene	5.1	5.1	5.1	0.00
2,4-Dinitrophenol	4.5	4.7	4.6	0.14
4-Nitrophenol	5.5	5.5	5.5	0.00

BEST AVAILABLE COPY

SAME AS EPA LETTER 1SA - LEVEL 1

SFC-MS of EPA Spiked Soil Sample 1SA

Compound	Amount (μg)			
	Trial 1	Trial 2	Average	Std. Dev.
Phenol	6.9	7.0	7.0	0.07
N-Nitrosodiphenylamine	5.6	5.7	5.6	0.07
Benz[a]anthracene	5.8	5.8	5.8	0.0
Benzo[a]pyrene	7.0	7.1	7.0	0.07
Dibenz[a,h]anthracene	5.1	5.2	5.2	0.07
2,4-Dinitrophenol	4.6	4.6	4.6	0.0
4-Nitrophenol	5.5	5.6	5.6	0.07

GC-MS of EPA Spiked Soil Sample 1SA

Compound	Amount (μg)			
	Trial 1	Trial 2	Average	Std. Dev.
Phenol	7.0	7.0	7.0	0.00
N-Nitrosodiphenylamine	5.6	5.6	5.6	0.00
Benz[a]anthracene	5.7	5.6	5.6	0.07
Benzo[a]pyrene	7.0	7.0	7.0	0.00
Dibenz[a,h]anthracene	5.1	5.1	5.1	0.00
2,4-Dinitrophenol	4.5	4.7	4.6	0.14
4-Nitrophenol	5.5	5.5	5.5	0.00

SAME AS EPA LETTER 4SA -LEVEL 2

SFC-MS of EPA Spiked Soil Sample 2SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	34	35	34	0.07
N-Nitrosodiphenylamine	23	28	25	0.0
Benz[a]anthracene	29	29	29	0.0
Benzo[a]pyrene	35	36	36	0.07
Dibenz[a,h]anthracene	25	26	26	0.0
2,4-Dinitrophenol	23	23	23	0.0
4-Nitrophenol	28	28	28	0.0

GC-MS of EPA Spiked Soil Sample 2SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	36	36	36	0.0
N-Nitrosodiphenylamine	28	28	28	0.0
Benz[a]anthracene	28	28	28	0.0
Benzo[a]pyrene	35	35	35	0.0
Dibenz[a,h]anthracene	25	26	25	0.0
2,4-Dinitrophenol	22	24	23	1.4
4-Nitrophenol	28	28	28	0.0

SFC-MS of EPA Spiked Soil Sample 3SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	.	.	.	.
N-Nitrosodiphenylamine	.	.	.	.
Benz[a]anthracene	.	.	.	.
Benzo[a]pyrene	.	.	.	.
Dibenz[a,h]anthracene	.	.	.	.
2,4-Dinitrophenol	.	.	.	.
4-Nitrophenol	.	.	.	.

GC-MS of EPA Spiked Soil Sample 3SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	.	.	.	.
N-Nitrosodiphenylamine	.	.	.	.
Benz[a]anthracene	.	.	.	.
Benzo[a]pyrene	.	.	.	.
Dibenz[a,h]anthracene	.	.	.	.
2,4-Dinitrophenol	.	.	.	.
4-Nitrophenol	.	.	.	.

SAME AS EPA LETTER 4SA-LEVEL 1

SFC-MS of EPA Spiked Soil Sample 4SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev
Phenol	7.0	7.0	7.0	0.0
N-Nitrosodiphenylamine	5.6	5.6	5.6	0.0
Benz[a]anthracene	5.7	5.8	5.8	0.07
Benzo[a]pyrene	7.1	7.0	7.0	0.07
Dibenz[a,h]anthracene	5.2	5.2	5.2	0.0
2,4-Dinitrophenol	4.5	4.5	4.5	0.0
4-Nitrophenol	5.6	5.6	5.6	0.0

GC-MS of EPA Spiked Soil Sample 4SA

	Amount (µg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	7.0	6.9	7.0	0.07
N-Nitrosodiphenylamine	5.5	5.7	5.6	0.14
Benz[a]anthracene	5.8	5.8	5.8	0.00
Benzo[a]pyrene	7.1	7.1	7.1	0.00
Dibenz[a,h]anthracene	5.1	5.1	5.1	0.00
2,4-Dinitrophenol	4.6	4.5	4.6	0.07
4-Nitrophenol	5.6	5.7	5.6	0.07

Letter, September 21, 1989, Alpine West Laboratories, Provo, Utah, w/enclosed results of spiked soil samples.

ENCLOSURE 4

ALPINE WEST LABORATORIES P.O. BOX 7521 UNIVERSITY STATION
PROVO, UTAH 84602

September 21, 1989

Dr. Bill Mitchell
U.S. Environmental Protection Agency
Performance Evaluation Branch
ERC Annex, MD-77B
Alexander Drive
Research Triangle Park, NC 27711

Dear Bill:

Enclosed are the GC-MS results of the spiked soil samples (1SA, 2SA, 3SA, 4SA, and 5SA). I have sent duplicate copies to Don Johnson.

Let me know if you have any questions.

Sincerely,

Milton

Milton L. Lee

CHEMICAL ANALYSIS: ENERGY • ENVIRONMENT • TOXICOLOGY • FORENSIC

GC-MS of EPA Spiked Soil Sample 1SA

Compound	Amount (μg)			
	Trial 1	Trial 2	Average	Std. Dev.
Phenol	7.0	7.0	7.0	0.00
N-Nitrosodiphenylamine	5.6	5.6	5.6	0.00
Benz[*a*]anthracene	5.7	5.6	5.6	0.07
Benzo[*a*]pyrene	7.0	7.0	7.0	0.00
Dibenz[*a*,*h*]anthracene	5.1	5.1	5.1	0.00
2,4-Dinitrophenol	4.5	4.7	4.6	0.14
4-Nitrophenol	5.5	5.5	5.5	0.00

GC-MS of EPA Spiked Soil Sample 2SA

Compound	Amount (μg)			
	Trial 1	Trial 2	Average	Std. Dev.
Phenol	36	36	36	0.0
N-Nitrosodiphenylamine	28	28	28	0.0
Benz[*a*]anthracene	28	28	28	0.0
Benzo[*a*]pyrene	35	35	35	0.0
Dibenz[*a*,*h*]anthracene	26	26	26	0.0
2,4-Dinitrophenol	22	24	23	1.4
4-Nitrophenol	28	28	28	0.0

GC-MS of EPA Spiked Soil Sample 3SA

	Amount (μg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	-	-	-	-
N-Nitrosodiphenylamine	-	-	-	-
Benz[a]anthracene	-	-	-	-
Benzo[*a*]pyrene	-	-	-	-
Dibenz[*a*,*h*]anthracene	-	-	-	-
2,4-Dinitrophenol	-	-	-	-
4-Nitrophenol	-	-	-	-

GC-MS of EPA Spiked Soil Sample 4SA

	Amount (μg)			
Compound	Trial 1	Trial 2	Average	Std. Dev.
Phenol	7.0	6.9	7.0	0.07
N-Nitrosodiphenylamine	5.5	5.7	5.6	0.14
Benz[*a*]anthracene	5.8	5.8	5.8	0.00
Benzo[*a*]pyrene	7.1	7.1	7.1	0.00
Dibenz[*a*,*h*]anthracene	5.1	5.1	5.1	0.00
2,4-Dinitrophenol	4.6	4.5	4.6	0.07
4-Nitrophenol	5.6	5.7	5.6	0.07

GC-MS of EPA Spiked Soil Sample 5SA

Compound	Amount (μg)			
	Trial 1	Trial 2	Average	Std. Dev.
Phenol	35	34	34	0.07
N-Nitrosodiphenylamine	27	28	28	0.07
Benz[*a*]anthracene	29	29	29	0.00
Benzo[*a*]pyrene	36	36	36	0.00
Dibenz[*a*,*h*]anthracene	24	25	24	0.07
2,4-Dinitrophenol	23	24	24	0.07
4-Nitrophenol	28	29	28	0.07

Letter MD-77B, November 1, 1989, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

ENCLOSURE 5

Nov 1 87 DD, M

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

November 1, 1989

Mr. Mac Donald Johnson
Commander Headquarters AMMCOM
AMSMC-DSM-D
Rock Island, IL 61299

Dear Don

This letter summarizes the results of the performance and systems audits my staff and I conducted on your OBOD project between December 1988 and June 1989. The individual reports issued on these audits are attached for your convenience in case you want more details on a specific audit.

In this letter, I attempted to integrate the results from all the audits to provide you an overview of the audit results. The following audits were conducted:

Bang box tests - Sandia Labs, Dec 1988/Feb 1989

- Systems audit
- Flowrate audits of samplers collecting integrated samples
- Accuracy audit of gas monitors(CO, CO_2, O_3, NO_2, SO_2)
- Extraction efficiency audit of semivolatiles from soil, XAD-2 and 32-liter canisters (tanks)
- Accuracy audit of VOC's by 6-liter canister

DPG Testing - June 1989

- Systems audit
- Accuracy audit of gas monitors (CO, CO_2)

The results of these audits were:

(A) Bang Box

1. Systems audit results. All the equipment was operating properly and the OD operations were being conducted in conformance to the Test Plan for the bang box test. It was recommended that Sandia Labs maintain a larger inventory of spare parts for their air monitoring equipment to avoid delays when field tests begin at DPG.

2. Flowrate audit results. All semi-VOST and particulate sampler flowrates were well within the acceptable range for these types of samplers. (Note - in the Test Plan the semi-VOST samplers were identified as VOST samplers, so the audit report followed this identification system.)

3. Accuracy audit of gas monitors. All monitors (CO_2, CO, NO_2, SO_2, O_3) were found to be operating well within the commonly accepted range for calibration accuracy for such monitors.

4. Efficiency of extraction of semivolatiles (target compounds) from soil and from XAD-2. Soil samples from DPG and the XAD-2 resins cleaned and packaged by Alpine West Laboratories (AWL) were spiked by us with known quantities of seven of the OBOD project's target compounds. The spiked samples were sent to AWL and to Batelle (BL) where they were to be extracted and analyzed. BL and AWL were the contract laboratories who were to extract and analyze the semi-VOST (XAD-2) and particulate samples from the bang box tests.

(a) Soil samples. Only AWL submitted results from the soil samples. Their extractions (by SFC/MS) were quantitative and their precision was excellent. Since the samples were extracted and analyzed eight months after spiking, the stability of these materials in soil samples is demonstrated.

(b) XAD-2 samples. Both BL and AWL found that the XAD-2 decomposed partially during the extraction and this made the efficiency of extraction tests unreliable. (Subsequent investigation showed that the XAD-2 had been heated too long during the pretest cleanup and oxidation of the resin had occurred.) We have voided the results from the XAD-2 audit.

5. Canister audits. Two 6-liter canisters and two 32-liter canisters (tanks) were spiked with known quantities of VOC (6 liter) and semivolatile (tanks) compounds.

(a) 6-liter canisters. These were sent to Oregon Graduate Center (OGC). These canisters contained seventeen VOC's commonly found in ambient air. The recoveries of these VOC's were similar to results found by other laboratories. For example, the higher the boiling point of the pure compound, the lower the recovery. Also, for most compounds, the precision achieved was good and the percent recovery was statistically the same for most compounds between canisters. For the most volatile VOC's, the difference between the reported and the spiked (expected) values were less than 1 ppb.

(b) 32-liter tanks. One tank was sent to AWL and the other was sent to BL. The two tanks had been spiked with the seven semi-VOC compounds used to spike the XAD-2 and the soil samples. The two laboratories also received an aliquot of the spiking solution although they did not know that it was the spiking solution. The results showed that none of the seven compounds was quantitatively recovered from the tank by AWL and BL. These results were used as the basis for discontinuing further work to develop the 32-liter tank sampler.

B. DPG ORI June 1989

1. Systems audit. The systems audit determined that the Sandia Labs airplane did not have an inventory of spare parts for the samplers. It was also

noted that there was no means to determine that the 6-liter canister was evacuated and that a sample was actually collected. Otherwise, the systems were operating properly and records and documentation were being adequately maintained.

2. Accuracy audit of gas monitors. Only CO and CO_2 monitors were on the airplane. The audit results showed that both monitors were calibrated within the accuracy range normally accepted for such analyzers. However, the CO_2 analyzer was close to being outside the acceptable range for slope--a situation that likely resulted because the zero had shifted between the last calibration and the audit. It was felt by the auditors that this shift would not adversely affect the CO_2 results for the June test because the CO_2 measured in the plume is corrected for the CO_2 measured outside the plume (background). In other words, the baseline shift would affect both the plume and background measurements to the same extent, so the difference (CO_2 from detonation or burn) would still be accurate.

In conclusion, our audits show us that the quality of the data gathered in your OBOD research program during the bang box tests and the DPG June 1989 pilot field tests will be adequate to meet your project's objectives. Your contract personnel and the DPG personnel obviously are committed to providing the DOD the high quality data that the environmental monitoring community is demanding to ensure that OBOD operations do not endanger the environment when done properly.

The performance and systems audits we conducted at DPG this month also support the above conclusions. I hope to have a complete audit report on the October DPG audits and the November laboratory audits to you by early December.

I hope the above information provides a summary of our QA efforts that meets your present needs. If you need additional information, please call.

Sincerely,

Bill Mitchell

William J. Mitchell, Ph.D.
Chief, Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Memorandum, 20 January 1990, Floyd W. McMullin Jr, subject: Site visit to SSL on 17 January 1998 [sic].

ENCLOSURE 6

MEMORANDUM

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd W. McMullin Jr

DATE: 20 January 1990

RE: Site visit to SSL on 17 January 1990

I met with Mr. Robert Cary of Sunset Lab to go over the OB/OD testing as relates to Phase B. At this point all samples (filters) submitted to him have been analyzed, and the data supplied to Mr. Wayne Einfeld of SNL as per his contract. He additionally gave me a printout of the results and sample listings which is attached for our review and files. His records agree with ours that he has received and analyzed 32 samples from phase B.

Mr. Cary is in the process of installing a new automated analysis system. This will necessitate another site visit when the next phase of testing and analysis is undertaken. Currently however, the equipment remains unchanged from that previously observed and evaluated on my previous visits of January and May 1989.

Generally my evaluation of this facility remains very good. Mr. Cary's techniques appear to be directed toward maintaining a very good internal quality control.

LOI STATUS ON SITE: This has been prepared previously and submitted during my visit of May 1989. The document is readily available to Mr. Cary at the lab.

FIELD/LAB SAMPLING: Sample for analysis consists of a 1x1 1/2 cm piece of the quartz glass filter to be analyzed. The actual section of the filter used is selected at random and cut from the rest of the filter utilizing a cutting punch device. Although this is selected at random Mr. Cary attempts to select an area of the filter which appears to have a consistent sample deposition.

FIELD/LAB ANALYSIS: After having manually placed the filter sample in the furnace and initiating the sequence on the control computer the rest of the analysis is fully automated under the computer's control. The basic sequence is that the sample is heated from room temperature through three different temperature levels (200, 400, and 700 degrees C approximately) while in an atmosphere of pure helium. During this time carbon compounds are monitored and the exact reflectivity (color) of the filter is monitored using a laser/photoelectric detector system. Sample is then cooled and oxygen is introduced. This results in burning off the carbon as carbon dioxide which is converted to methane utilizing a catalytic methanizer which is monitored/measured by a photoionization detector. The entire process allows a differentiation of carbon compounds versus elemental carbon in the sample. Output from the system is via a computer printer and consists of both numeric (for key values) and overall graphic representation.

INSTRUMENT/METHOD CALIBRATION: At least two methods to assure proper measurement and instrument calibration are utilized. The first method utilizes an automatic carbon insertion at the end of the analysis procedure for each sample. This value must read within a certain specified range or it indicates the unit is malfunctioning. The second method of calibration is to place a measured quantity of a carbon compound solution of a known concentration on a blank filter sample. This is then dried and subjected to analysis. As this sample contains a known quantity and concentration the amount of carbon may be predicted. If the sample fails to analyze within an acceptable error range (± 5%) all sections of the analysis equipment are checked/adjusted/ repaired/replaced, etc. as needed.

PREVENTIVE/CORRECTIVE MAINTENANCE: Has ongoing systems monitoring. In the event of problems immediate repairs or maintenance is performed.

INTERNAL QC PROCEDURES: Utilizes automatic carbon spikes completion of each sample as noted previously, and the spik filters as noted previously. He currently utilizes a sucrose solution because of the excellent pyrolysis characteristics of the material.

SAMPLE PREPARATION AND STORAGE: There is no preparation of the samples involved. All filters are initially prepared (cleaned) by AWL and they ship the used filters to him for analysis. The samples as received are stored in amber glass jars in a standard upright refrigerator.

PREPARATION AND USE OF SPIKED SAMPLES: See previous sections on calibration and QC procedures.

INSTRUMENT/EQUIPMENT SELECTION AND USE: Unit is a totally custom designed and built unit by R. Cary. Stated was developed over a number of years to its present state. Also made the comment that there was probably only one or two other comparable systems in the world.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION: Detection limit is 0.2 mcg/cm^2 ,Precision is ± 5% (see copy of methodology for deriving precision value attached).

SAMPLE HANDLING AND TRANSPORTATION: Sample are handled only by Robert Cary (Signature, etc. is in the logbook). Currently no transport of shipments is done by Sunset Labs.

DATA REDUCTION AN ANALYSIS: Automated computer analysis and printout at the time of the sample run. System utilizes custom software designed and developed by R. Cary.

NOTES ON OTHER ITEMS:

LOGBOOKS: There is no logbook for this piece of apparatus. Mr. Cary does not keep one as it makes and prints a calibration check with each sample run.

RESEARCH JOURNAL: Utilizes a numbered page ledger to enter each sample run. Each days entries are noted and are in ink. The journal I examined had no pages missing so far as I was able to determine.

LAB TRACKING FORMS: Does not currently utilize and in-house form. He does log each sample into a register upon receipt, on the same register he also notes the day ran, and the date results are sent out. He currently utilizes the collection report received with each sample for assignment of the sample number.

Floyd W. McMullin Jr.
Quality Assurance Officer

OB/OD SAMPLES
DECEMBER, 1989

Carbonate Carbon

Sample ID	OC,ug/sq cm	OC err	EC,ug/sq cm	EC err	CC,ug/sq cm	CC err
OBOD.2470	4.06	0.40	0.29	0.21	n.d.	
OBOD.2472	4.02	0.40	0.12	0.21	n.d.	
OBOD.2474	3.45	0.37	0.11	0.21	n.d.	
OBOD.2476	7.21	1.24	3.89	0.39	13.50	0.98
OBOD.2478	6.13	1.09	2.95	0.35	11.60	0.88
OBOD.2478RPT	6.97	1.17	3.18	0.36	12.50	0.93
OBOD.2480	6.23	0.97	2.97	0.35	9.20	0.76
OBOD.2480.ACID	9.00	0.65	1.49	0.27	n.d.	
OBOD.2482	4.73	0.44	0.06	0.20	n.d.	
OBOD.2484	5.01	0.45	0.09	0.20	n.d.	
OBOD.2486	4.95	0.45	0.08	0.20	n.d.	
OBOD.2488	5.33	0.47	0.00	0.20	n.d.	
OBOD.2490	5.46	0.90	3.10	0.36	8.60	0.73
OBOD.2492	6.54	1.06	4.01	0.40	10.60	0.83
OBOD.2494	5.58	0.89	3.19	0.36	8.20	0.71
OBOD.2496	5.51	0.48	0.09	0.20	n.d.	
OBOD.2498	5.28	0.46	0.16	0.21	n.d.	
OBOD.2500	5.17	0.46	0.10	0.21	n.d.	
OBOD.2502	4.40	0.42	0.16	0.21	n.d.	
OBOD.2504	4.70	0.44	0.24	0.21	n.d.	
OBOD.2506	4.18	0.41	0.18	0.21	n.d.	
OBOD.2508	4.27	0.41	0.26	0.21	n.d.	
OBOD.2510	4.27	0.41	0.25	0.21	n.d.	
OBOD.2512	4.10	0.40	0.26	0.21	n.d.	
OBOD.2514	3.58	0.38	0.15	0.21	n.d.	
OBOD.2516	6.26	0.51	0.15	0.21	n.d.	
OBOD.2518	4.70	0.52	0.64	0.23	1.80	0.39
OBOD.2520	3.90	0.46	0.44	0.22	1.40	0.37
OBOD.2522	4.50	0.49	0.65	0.23	1.30	
OBOD.2524	7.17	0.68	0.93	0.25	2.50	0.43
OBOD.2524.ACID	8.55	0.63	0.19	0.21	n.d.	
OBOD.2526	6.95	0.67	0.48	0.22	2.40	0.42
OBOD.2526.RPT	6.26	0.62	0.80	0.24	2.20	0.41
OBOD.2528	5.24	0.57	0.91	0.25	2.10	0.41
OBOD.2530	4.46	0.42	0.35	0.22	n.d.	
OBOD.2532	3.85	0.39	0.11	0.21	n.d.	
SUGAR.20UL.120	29.45	1.67	0.00	0.20	n.d.	
SUGAR.30UL.120	43.50	2.37	0.00	0.20	n.d.	
INST.BLNK.1208	0.01	0.20	0.00	0.20	n.d.	
INST.BNLK.1206	0.20	0.21	0.02	0.20	n.d.	

Received 20 JAN 1990

Sunset Laboratory
2017 19th Avenue
Forest Grove, Oregon 97116
(503) 357-5151
Robert A. Cary

UNCERTAINTIES ASSOCIATED WITH OC/EC ANALYSIS

The reported uncertainties for organic and elemental carbon analysis are derived from two sources: (1)the relative uncertainty, which has been determined from doing duplicate analysis on a fraction of samples analyzed; and (2)the detection limit, which has been doing a large number of instrument blank analyses.

The relative uncertainty is derived by calculating the relative percent variation from the mean of a sample which has been analyzed two times. The standard deviation of all the samples so analyzed has been found to be about 5%. It should be noted that this value is a reflection of all sources which may contribute to a difference in duplicate analyses, such as instrument analysis variations, sampling variations and deposit variations due to inhomogeneity of deposit density.

The detection limit has been determined by doing many instrumental blank analyses where an analysis is performed on a filter punch which is known to contain no carbon. The standard deviations of these analysis is found to be about 0.1 ug/sq cm of carbon. The uncertainty uses for the detection limit is twice this standard deviation, or 0.2 ug/sq cm.

The reported uncertainties are determined by a linear combination of the two values above, i.e., uncertainty = +-0.05(value) + 0.2. ug/sq cm carbon. Thus, at moderate to high levels, the uncertainty is determined mostly by the 5% variation and at low levels the detection limit is found to have a proportionally larger share in the uncertainty.

The above method can give an estimate of the precision of the analysis. However, determining the accuracy can not be done as precisely due to the fact that no standards exist for organic/elemental carbon aerosols. Attempts have been made to create such samples which would contain predictable amounts of each species and the results have been found to be within this 5% range. Also, interlaboratory/intermethod comparisons have been done indicating that the errors in accuracy are no greater than 5%.

Memorandum, 20 January 1990, Floyd W. McMullin Jr, subject: Site visit to OGC on 18 January 1998 [sic].

ENCLOSURE 7

MEMORANDUM

ENVIRONMENTAL LABS INC.
FIELD OFFICE
RECEIVED
JAN 22 1990
BY
QUALITY ASSURANCE UNIT

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd W. McMullin Jr.

DATE: 20 January 1990

RE: Site visit to OGC on 18 January 1990

I met with Dr. Reinhold Rasmussen and Mr. Robert Dalluge to go over the OB/OD Phase B testing and analysis.

LOI STATUS ON SITE: As OGC utilizes a standardized testing procedure they do not employ a specific LOI.

FIELD/LAB SAMPLING: All field sampling is done by SNL utilizing the FWAC testing platform. All samples for Phase B are 6 liter canisters which are evacuated. To obtain a sample it is connected to the appropriate apparatus, the valve opened, and it is allowed to fill to ambient pressure (0 psig). The valve is then closed and the threaded connector is capped.

During lab analysis the canister is connected to the appropriate analysis device, and a sample is pumped out of the container.

FIELD/LAB ANALYSIS: All analyses are accomplished utilizing one or more gas chromatographs. The types currently in use at OGC are Perkin Elmer 3920, Carle 211M, Hewlett Packard 5790A, and a Shimadzu GC-mini 2. A reduction gas detector manufactured by Trace Analytical, model RGD-2 is also used. Data from these devices is fed into a Hewlett Packard integrator for printout. The models currently in use are 3390A, 3396A, and 3388.

As of this date, all Phase B samples have been analyzed for CH_4, CO, and CO_2. The composite bag samples are in the process of having analysis for benzene.

INSTRUMENT/METHOD CALIBRATION: In all systems repeated calibration/standardization sample runs utilizing a test gas of known concentration is done. Generally this is done after every fourth sample run. This allows checking on an ongoing basis to see if the calibration is drifting.

PREVENTIVE/CORRECTIVE MAINTENANCE: As all the devices in use are of solid state construction no routine preventive maintenance is required. In the event of equipment failure the problem is found, corrected, the instrument recalibrated, and placed back in service.

INTERNAL QC PROCEDURES: Other than the routine calibration checks during sample runs I was not made aware of any additional methods employed.

SAMPLE PREPARATION AND STORAGE: As mentioned previously, after analysis the tanks are prepared for re-use so there is not long term storage of the samples.

PREPARATION AND USE OF SPIKED SAMPLES: As noted previously concentrations of a known amount are routinely run. Additionally they have received and analyzed an EPA audit sample. The sample was supposed to contain benzene. However, Dr. Rasmussen noted that while it did contain benzene, this showed as a minor peak in the chromatograph. They also detected alcohols, ketones, 2-butonone, and various other materials. He stated he felt the EPA methodology for sample preparation was poor to allow the degree of contamination they found in the sample they tested.

INSTRUMENT/EQUIPMENT SELECTION AND USE: Instrumentation in use is as noted in the Field/Lab Analysis section. It is outside my expertise to judge whether or not the actual operation and applications are correct.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION: The limits of detection are 0.1 to 0.2 ppb. The accuracy is ± 15 -20%. Precision is determined by doing multiple analysis of the same sample to determine a standard deviation. This is then divided by the mean of the deviation, and multiplied by 100 to obtain a relative standard deviation. The value they have arrived at is ± 5%.

SAMPLE HANDLING AND TRANSPORTATION: Samples are handled by only four people (Signatures are in logbook). They are Dr. Reinhold Rasmussen, Robert Dalluge, Don Stearns, and Bob Watkins. All transport of samples is through Federal Express.

DATA REDUCTION AN ANALYSIS: For the type of analysis performed there is no apparent specialized analysis of results required.

NOTES ON OTHER ITEMS:

LOGBOOKS: Logbooks for individual pieces of equipment are not kept. Calibration runs are kept with the analyzation runs done at the same time.

LAB DIAGRAM: Not obtained.

RESEARCH JOURNAL: Those I have seen appeared to be intact, with all entries in ink.

LAB TRACKING FORMS: So far as I can determine no forms of any type are used to track the cylinders in-house. Prior to shipment out in the field all cylinders numbers are logged. When used in the field the data on each tank (number, date, time, etc.) is recorded in the research journal and in the portable computer utilized by SNL. Upon return to the lab they are processed (analyzed) as soon as practicable. The information used is that of the tank number and the information on the sample form. They also check to assure that all tanks sent out are received back at OGC. As noted previously, following analysis the tanks are cleaned for re-use. This effectively negates there being any "sample" to track, store, etcetera after completion on analysis.

MISCELLANEOUS:

1. Dr. Rasmussen noted that they did an analysis on the standard gases (CO and CO_2) in use by SNL. The results have been furnished to Wayne Einfeld.

2. Howard Crist of EPA conducted an audit of OGC. Dr. Rasmussen noted that his report did not contain the date of the visit, so they would have to search through their records to determine the date.

3. While checking sample information I found that OGC had received and analyzed four (4) more samples than what we knew about and completed sample reports on. These were found to be sample from the second burn done of 25 October 1989. These tanks carried the numbers VJ 111, 112, 113, and 114. They are noted in the summary sheet attached to this report. Sample forms will be completed on these and they will be added to the sample master list.

OVERALL ASSESSMENT: Based on their practice of doing repeated controls interspersed during sample analysis I feel that their quality control is good.

Other than getting them to write down their actual analysis process (step by step) so others could have a better understanding of what they do and how they do it, I have no other recommendations at this time.

Floyd W. McMullin Jr.
Quality Assurance Officer

12-Jan-90

OGC 6L CANISTER SAMPLE LIST (CONTINUED)

OGC #	SAMPLE DATE	TIME	TEST TYPE	PORT
VJ119	25-OCT-89	1000	BURN1/PASS1	TUBE
VJ121	25-OCT-89	1000	BURN1/PASS2	TUBE
VJ122	25-OCT-89	1000	BURN1/PASS3	TUBE
VJ120	25-OCT-89	1000	BURN1/COMP	BAG
VJ111	25-OCT-89	1030	BURN2/PASS1	TUBE
VJ113	25-OCT-89	1030	BURN2/PASS2	TUBE
VJ112	25-OCT-89	1030	BURN2/PASS3	TUBE
VJ114	25-OCT-89	1030	BURN2/COMP	BAG
VJ131	25-OCT-89	1100	OSTNT/PASS1	TUBE
VJ132	25-OCT-89	1100	OSTNT/PASS2	TUBE
VJ133	25-OCT-89	1100	OSTNT/PASS3	TUBE
VJ134	25-OCT-89	1100	OSTNT/COMP	BAG
VJ152	25-OCT-89	1300	BKGND	TUBE
VJ151	25-OCT-89	1300	BKGND	BAG
VJ153	31-OCT-89	0930	BKGND	TUBE
VJ154	31-OCT-89	0930	BKGND	BAG
VJ155	31-OCT-89	1100	S1TNT/SHOT1/PASS1	TUBE
VJ157	31-OCT-89	1100	S1TNT/SHOT1/PASS2	TUBE
VJ158	31-OCT-89	1100	S1TNT/SHOT1/PASS3	TUBE
VJ128	31-OCT-89	1100	S1TNT/SHOT1/COMP	BAG
SDA078	31-OCT-89	1100	S1TNT/SHOT2/PASS1	TUBE
SDA079	31-OCT-89	1100	S1TNT/SHOT2/PASS2	TUBE
SDA080	31-OCT-89	1100	S1TNT/SHOT2/PASS3	TUBE
SDA081	31-OCT-89	1100	S1TNT/SHOT2/COMP	BAG
SDA074	31-OCT-89	1100	S1TNT/SHOT3/PASS1	TUBE
SDA075	31-OCT-89	1100	S1TNT/SHOT3/PASS2	TUBE
SDA076	31-OCT-89	1100	S1TNT/SHOT3/PASS3	TUBE
SDA077	31-OCT-89	1100	S1TNT/SHOT3/COMP	BAG
VJ147	31-OCT-89	1500	S2TNT/SHOT1/PASS1	TUBE
VJ148	31-OCT-89	1500	S2TNT/SHOT1/PASS2	TUBE
VJ149	31-OCT-89	1500	S2TNT/SHOT1/COMP	BAG
SDA058	31-OCT-89	1500	S2TNT/SHOT2/PASS1	TUBE
VJ150	31-OCT-89	1500	S2TNT/SHOT2/PASS2	TUBE
VJ159	31-OCT-89	1500	S2TNT/SHOT2/COMP	BAG
VJ160	31-OCT-89	1500	S2TNT/SHOT3/PASS1	TUBE
VJ161	31-OCT-89	1500	S2TNT/SHOT3/COMP	BAG

NO SAMPLE REPORTS DONE ON THESE 1-18-90

Reports now completed.

NOTES:

OGTNT = GROUND ORI TNT
G1TNT = FIRST GROUND TNT 3 SHOT SERIES
G2TNT = SECOND GROUND TNT 3 SHOT SERIES

OB/OD FIELD TEST October 1989

R.A. Rasmussen
11-20-89

Item	SN	Date	Time	Type	Port	CH4 ppbv	CO ppbv	CO2* ppmv
1	VJ102	16-Oct-89	1230	Bkgnd sample	Tube	1782	106	356
2	VJ101	16-Oct-89	1230	Bkgnd sample	Bag	1784	118	355
3	VJ099	16-Oct-89	1445	TNT ORI Pass 1	Tube	1927	4622	520
4	VJ100	16-Oct-89	1445	TNT ORI	Composite bag	1885	2636	449
5	VJ096	17-Oct-89	1130	Background	Tube	1780	108	351
6	VJ095	17-Oct-89	1130	Background	Bag	1782	109	356
7	VJ097	17-Oct-89	1330	Shot 1/Pass 1/TNT	Tube	2209	9803	496
8	VJ098	17-Oct-89	1330	TNT Shot 1/Pass 2/	Tube	1892	2805	388
9	VJ107	17-Oct-89	1330	TNT/Shot 1/Pass 3/	Tube	1854	2247	390
10	VJ109	17-Oct-89	1330	TNT/Shot 1/	Bag composite	1940	4186	416
11	SDA040	17-Oct-89	1330	TNT/Shot 2/Pass 1/	Tube	2097	9948	545
12	VJ108	17-Oct-89	1330	TNT/Shot 2/Pass 2/	Tube	1810	1308	382
13	VJ110	17-Oct-89	1330	TNT/Shot 2/Pass 3/	Tube	1848	2458	392
14	SDA038	17-Oct-89	1330	TNT/Shot 2/	Bag composite	1876	3409	410
15	SDA039	17-Oct-89	1330	TNT/Shot 3/Pass 1/	Tube	1916	5156	449
16	SDA037	17-Oct-89	1330	TNT/Shot 3/Pass 2/	Tube	1832	2800	420
17	VJ104	17-Oct-89	1330	TNT/Shot 3/Pass 3/	Tube	1793	208	354
18	VJ105	17-Oct-89	1330	TNT/Shot 3/	Bag composite	1837	2569	409
19	VJ103	18-Oct-89	0900	Background	Tube	1761	127	356
20	VJ106	18-Oct-89	0900	Background	Bag	1762	122	355

* CO2 by TCD

OB/OD FIELD TEST October 1989

R.A. Rasmussen
11-20-89

Item	SN	Date	Time	Type	Port	CH4 ppbv	CO ppbv	CO2* ppmv
21	VJ135	18-Oct-89	1100	TNT/Shot 1/Pass 1/	Tube	2054 2060	9793 --	609
22	VJ136	18-Oct-89	1100	TNT/Shot 1/Pass 2/	Tube	1847	2633	411
23	VJ137	18-Oct-89	1100	TNT/Shot 1/Pass 3/	Tube	1810	1220	376
24	VJ138	18-Oct-89	1100	TNT/Shot 1/	Bag #1	1856	3223	419
25	VJ124	18-Oct-89	1100	TNT/Shot 1/	Bag composite #2 (replicate)	1860	3066	423
26	VJ123	18-Oct-89	1100	TNT/Shot 2/Pass 1/	Tube	2126	9474	488
27	VJ125	18-Oct-89	1100	TNT/Shot 2/Pass 2/	Tube	1914	3421	413
28	VJ126	18-Oct-89	1100	TNT/Shot 2/Pass 3/	Tube	1800	753	359
29	VJ140	18-Oct-89	1100	TNT/Shot 3/	Bag composite	1908	3347	401
30	VJ139	18-Oct-89	1100	TNT/Shot 3/Pass 1/	Tube	2527 2672	22431 --	645
31	VJ141	18-Oct-89	1100	TNT/Shot 3/Pass 2/	Tube	1861	2067	388
32	VJ142	18-Oct-89	1100	TNT/Shot 3/Pass 3/	Tube	1917	3486	409
33	VJ144	18-Oct-89	1100	TNT/Shot 3/	Bag composite	2016	6162	437
34	VJ127	18-Oct-89	1100	TNT/Shot 3/	Bag composite #2 (replicate)	2026	6144	432
35	VJ146	19-Oct-89	1300	Background	Tube	1767	102	356
36	VJ145	19-Oct-89	1300	Background	Bag	1765	106	355
37	VJ115	19-Oct-89	1400	Burn/ORI/Pass 1/	Tube	1763	173	582
38	VJ116	19-Oct-89	1430	Burn/ORI/Pass 2/	Tube	1761	148	442
39	VJ117	19-Oct-89	1430	Burn/ORI/Pass 3/	Tube	1767	116	388

* CO2 by TCD

OB/OD FIELD TEST October 1989

R.A. Rasmussen
11-20-89

Item	SN	Date	Time	Type	Port	CH4 ppbv	CO ppbv	CO2* ppmv
40	VJ118	19-Oct-89	1430	Burn ORI	Bag composite	1770	157	448
41	VJ119	25-Oct-89	1000	Burn/Pass 1/	Tube	1762	122	355
42	VJ121	25-Oct-89	1000	Burn/Pass 2/	Tube	1766	114	435
43	VJ122	25-Oct-89	1000	Burn/Pass 3/	Tube	1766	123	403
44	VF111	25-Oct-89	1030	Burn2/Pass 1	Tube	1766	132	550
45	VF113	25-Oct-89	1030	Burn2/Pass 2	Tube	1715	185	380
46	VF112	25-Oct-89	1030	Burn2/Pass 3	Tube	1766	134	458
47	VF114	25-Oct-89	1030	Burn2/Comp	Bag	1762	130	502
48	VJ120	25-Oct-89	1000	Burn	Bag composite	1768	115	464
49	VJ131	25-Oct-89	1100	TNT(SUS)ORI/Pass 1/	Tube	1772	447	431
50	VJ132	25-Oct-89	1100	TNT(SUS)ORI/Pass 2/	Tube	1776	438	448
51	VF133	25-Oct-89	1100	TNT(SUS)ORI/Pass 3/	Tube	1771	257	393
52	VJ134	25-Oct-89	1100	TNT(SUS)ORI/	Bag composite	1789	433	410
53	VJ152	25-Oct-89	1300	Background	Tube	1775	94	354
54	VJ151	25-Oct-89	1300	Background	Bag	1771	97	359
55	VJ153	31-Oct-89	930	Background	Tube	1797	121	365
56	VJ154	31-Oct-89	930	Background	Bag	1802	118	364
57	VJ155	31-Oct-89	1100	Shot 1/Pass 1/	Tube	1793	660	448
58	VF157	31-Oct-89	1100	Shot 1/Pass 2/	Tube	1795	520	414
59	VF158	31-Oct-89	1100	Shot 1/Pass 3/	Tube	1787	308	388

CO2 by TCD

OB/OD FIELD TEST October 1989

R.A. Rasmussen
11-20-89

Item	SN	Date	Time	Type	Port	CH4 ppbv	CO ppbv	CO2* ppmv
60	VF128	31-Oct-89	1100	Shot 1/	Bag	1786	347	399
61	SDA078	31-Oct-89	1100	Shot 2/Pass 1/	Tube	1804	631	416
62	SDA079	31-Oct-89	1100	Shot 2/Pass 2/	Tube	1804	572	420
63	SDA080	31-Oct-89	1100	Shot 2/Pass 3/	Tube	1788	378	401
64	SDA081	31-Oct-89	1100	Shot 2/	Bag	1797	198	409
65	SDA074	31-Oct-89	1100	Shot 3/Pass 1/	Tube	1787	1379	593
66	SDA075	31-Oct-89	1100	Shot 3/Pass 2/	Tube	1796	390	411
67	SDA076	31-Oct-89	1100	Shot 3/Pass 3/	Tube	1780	127	361
68	SDA077	31-Oct-89	1100	Shot 3/	Bag	1782	457	437
69	VJ160	31-Oct-89	1500	Test 2/Shot 3/Pass 1/	Tube	1878	2913	612
70	VJ161	31-Oct-89	1500	Test 2/Shot 2/	Bag	1807	1018	448
71	VJ147	31-Oct-89	1500	Test 2/Shot 1/Pass 1/	Tube	1777	2450	684
72	VJ148	31-Oct-89	1500	Test 2/Shot 1/Pass 2/	Tube	1777	966	487
73	VJ149	31-Oct-89	1500	Test 2/Shot 1/	Bag	1776	1309	537
74	SDA058	31-Oct-89	1500	Test 2/Shot 2/Pass 1/	Tube	1849	858	483
75	VJ150	31-Oct-89	1500	Test 2/Shot 2/Pass 2/	Tube	1783	710	443
76	VJ159	31-Oct-89	1500	Test 2/Shot 2/	Bag	1792	735	457

Memorandum, 22 January 1990 from Floyd W. McMullin, Jr., subject: Site Visit to SNL (AT Dugway) on 19 October 1989 [sic].

ENCLOSURE 8

MEMORANDUM

ENVIRONMENTAL LABS INC.
FIELD OFFICE
RECEIVED
JAN 22 1990
BY
QUALITY ASSURANCE UNIT

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd W. McMullin Jr.

DATE: 22 January 1990

RE: Site visit to SNL (At Dugway) on 19 October 1989

A site visit of SNL was conducted while operating on site at the Dugway Proving Ground. At that time SNL personnel were operating out of trailer 48, a mobile facility from the applied atmospheric research division of SNL.

Two worksheets/checklists were utilized during this visit. The OB/OD site visit worksheet (pages 168 & 169 of QAPP Rev 3 dtd 3 Oct 89), and the Quality Assurance Practices Checklist (pages 170 & 171 of QAPP Rev 3 dtd 3 Oct 89). The original lists are filed with the site visit documents in ELI's QA files.

Name of internal QC Officer: Wayne Einfeld

LOI status on site (location, access, precision, agrees with actual methods etc. being employed): LOI available. Agrees in most respects with actual methods being employed. Some variation from LOI due to demands of testing in a field situation.

Field/Lab Sampling: Real time data for gases, particle sizing, etc. is stored electronically.

Field/Lab Analysis: Realtime data may be analyzed to some extent utilizing available computer systems. Filters to AWL (on dry ice) for storage and analysis. Six liter canisters to OGC for analysis.

Instrument/Method Calibration: All instruments recently calibrated and checked by EPA audit team.

Preventive/Corrective Maintenance: All instruments zerced and span checked each day. Corrective maintenance as per instrument technical manuals as needed.

Internal QC Procedures: All instruments daily calibration, zero and span checks.

Sample Preparation and Storage: All samples prepared by two people to check process employed. Checklist utilized in this process.

Preparation and Use of Spiked Samples: Not applicable in field situation. However, known value gases (CO & CO_2), and known particle size materials routinely utilized in checking instrumentation.

Instrument/Equipment Selection and Use: Based on needs and equipment durability and performance.

Determination of Detection Limits/Limits of Quantification:
This information already included in previous documentation.

Sample Handling and Transportation: Handling as per LOI. Filters flown to Provo to ELI representative for delivery to AWL. Six liter canisters delivered to representative of ELI on site for shipment to OGC.

Data Reduction and Analysis: Various, based on analysis required.
Miscellaneous Items:

1. Logbooks are available on site for all instruments. Entries are appropriate and in ink. Individual researchers also maintain personal research journals.

2. Name, address, and phone numbers of all personnel working with study materials has not changed from that filed from previous site visit. Those personnel and information are:

Wayne Einfeld	(505)844-4143	SNL Div 6321
Brian Mokler	(505)299-7610	SNL Div 6321 (Contract person)
Dennis Morrison	(505)844-3376	SNL Div 6321

3. Individual researchers maintain personal journals. Those I examined appeared intact, with entries in ink.

4. Lab tracking forms are not applicable in this setting. No physical samples are retained by the laboratory for analysis.

QUALITY ASSURANCE PRACTICES CHECKLIST

1. Administrative Information
   Name of Laboratory SNL - Applied Atmospheric Research
   Organizational Chart
   Scope of Capabilities
   Accreditations, recognitions, organizational memberships

		Yes	No	Exp.
2.	Human Resources			
2.1	Roster of personnel available	X		
2.2	Resumes of key personnel available	X		
2.3	Qualifications of all key persons			

	commensurate with responsibilities	X		
2.4	Work load commensurate with staffing	X		
2.5	Staff morale high	X		
3.	Physical Resources			
3.1	Facilities adequate for all services offered	X		
3.2	Laboratory environment adequate	X		
3.3	Adequate space for all operations	X		
3.4	Adequate housekeeping	X		
3.5	Adequate safety/inspections	X		
4.	Equipment			
4.1	Equipment list maintained	X		
4.2	Equipment used in specific tests identifiable _X_			
4.3	Equipment manuals filed systematically: Where: Equipment trailer	X		
4.4	All equipment serviceable and in calibration _X_			
4.5	Calibration status of all equipment indicated: _X_ How: Equipment logbooks			
4.6	List special facilities and opinion of their adequacy FWAC - Adequate to task and test design.			
5.	Quality Assurance System			
5.1	QA policy statement on file	X		
5.2	QA Officer with assigned duties	X		
5.3	All employees aware of their QA responsibilities	X		
5.4	QA Manual on file Adequacy: On File at SNL.	X		
5.5	Written procedures used for each test	X		
5.6	SOPs for all recurring tests (Methods Manual)	X		
5.7	Staff competent in test procedures	X		
5.8	Methods validated before use: How: Earlier experimentation	X		
5.9	System to qualify test operators	N/A		
5.10	Reagent control practiced	N/A		
5.11	Demonstration of statistical control: How: Span instrument with control charts.	X		
5.12	GLPs, GMPs on file and followed	X		
5.13	System of corrective actions	X		

6.	Control Charts			
6.1	QA policy requires use of control charts	_X_		
6.2	Control charts used to monitor system	_X_	___	___
6.3	Control charts used to assign confidence limits	_X_		
6.4	List of current control charts on file: Number on list: All gas instruments.	_X_	___	___
6.5	Control charts available for inspection by clients _X_	___	___	
7.	Reference Materials			
7.1	QA policy requires use of RMs	_X_	___	___
7.2	Internal reference materials listed and used	_X_	___	___
7.3	All reference materials inventoried	_X_	___	___
7.4	Reference materials used with control charts and limits	_X_	___	___
8.	Records			
8.1	Systematic format(s) for data	_X_	___	___
8.2	Lab notebooks kept systematically	_X_	___	___
8.3	Charts/readouts referenced to notebooks	_X_	___	___
8.4	Records periodically reviewed for adequacy _X_	___	___	
8.5	All records will pass critical inspection _X_	___	___	
9.	Reports			
9.1	Reports reviewed before release	_X_	___	___
9.2	Reports reference all important supporting data	_X_	___	___
9.3	Statistically supported limits of uncertainty _X_	___	___	
9.4	System for reports/records/data retrieval _X_	___	___	
10.	Sample Management			
10.1	Sample management system used	_X_	___	___
10.2	Computerized sample management	_X_	___	___
10.3	Sample preparation facilities adequate	_X_	___	___
10.4	Sample storage adequate	_X_	___	___
11.	Audits			
11.1	QA system management system used Frequency____________	_?_	___	___
11.2	External QA system audits	_X_	___	
11.3	Performance audits on regular basis	_X_	___	

11.4 Corrective actions taken as result of audits _X_ ___ ___

11.5 Records kept of corrective actions _X_ ___ ___

Floyd W. McMullin Jr.
Quality Assurance Officer

Letter MD-77B, January 26, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed EPA audit report for [Phase B].

ENCLOSURE 9

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

January 26, 1990

Mr. MacDonald Johnson
31 Potter Street
Salt Lake City, UT 84113

Dear Don:

Attached is the final EPA Audit Report for Phase B DOD/AMCCOM OB/OD Project (October-December 1989).

If you have any questions, please call me at 919-541-2365.

Sincerely,

Linda F. Porter
Acting Chief, Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Attachment

Memorandum, 9 February 1990, Floyd W. McMullin Jr., subject: AWL audit pertaining to EPA QAD deficiencies [sic].

ENCLOSURE 12

Audit Report on Phase B of the DOD/AMCCOM OB/OD Project

(October-December 1989)

by

W. Mitchell
L. Porter
E. Hunike
R. Rhodes
L. Smith
J. Bowen
O. Dowler
H. Crist

US EPA
AREAL/QAD (MD-77B)
RTP, NC 27711

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1.0 INTRODUCTION

The Department of Defense (DOD) is evaluating the environmental safety of disposing of munitions and propellants by burning (OB) or detonating (OD) them. OB and OD are the traditional means used for disposing of unsafe and also of surplus munitions and propellants in most countries. Recently, the US Environmental Protection Agency (EPA) and state agencies questioned the environmental safety of these operations and requested that DOD show that these activities neither harm the environment nor endanger human health.

A DOD effort (OB/OD) to answer the questions raised by EPA and the States is being done by the Demil and Technology Branch, Armament Munitions and Chemical Command (AMCCOM) located at Rock Island Arsenal, Rock Island, IL. The AMCCOM project manager is Mr. MacDonald Johnson. The field testing, however, is being done at Dugway Proving Ground (DPG), Dugway, UT, under the direction of the Material Test Directorate, Test Management Division (TMD), Artillery and Hazards Branch. Mr. Ken Jones and Mr. John Woffinden are the TMD project officers. Capt. David Coxson is the DPG test officer.

The latest phase of the DOD's OB/OD project (Phase B) was conducted at DPG from October 16 to November 1, 1989. Prior to these tests, AMCCOM conducted testing at Sandia National Laboratories (SNL), Albuquerque, NM, and at DPG. The SNL tests involved detonating 0.5 lb blocks of TNT in a hemispherical building (bangbox) and measuring the pollutants generated. The bangbox, which had a volume of approximately 950 m^3, was used because it held the products of the detonation so that they could be sampled and studied as a function of time. Through these bangbox tests, which were conducted from November 1988 into February 1989, AMCCOM was able to evaluate candidate sampling methods for their effectiveness in measuring the air pollutants resulting from OD operations. An analogous study, which involved burning M30 propellant in the bangbox, was also done to evaluate the effectiveness of the candidate methods for measuring the air pollutants resulting from OB operations.

The test methods selected for use in monitoring the field tests were then subjected to pilot testing at DPG in June 1989 (Phase A). These tests involved two separate burns of 7040 lbs of M30 propellant and five detonations of 1980 lbs of bulk TNT. During each burn and detonation, SNL sampled the resulting plume for volatile organic compounds (VOC) and for particulate using equipment contained in their deHaviland Twin Otter aircraft. The aircraft was operated under the direction of Mr. Wayne Einfeld of SNL. The Phase A tests at DPG confirmed that the candidate air pollution measurement methods selected, based on the bangbox tests, would be suitable for measuring OB and OD plumes under field conditions.

Samples from the bangbox tests and the Phase A tests were also used to develop improved analytical methods for the semivolatile organic materials (semi-VOC) found in the soil and the airborne particulate that result from OB and OD operations. This work was done at Alpine West Laboratories (AWL), Provo, UT, under the direction of Dr. Milton Lee.

AWL is the contract analytical laboratory for the ash, particulate and soil sample analyses from the OB and OD operations. AWL was able to show that Supercritical Fluid Chromatography (SFC)/Mass Spectrometry (MS) provided equal or better analytical sensitivity than GC/MS for the semi-VOC in the soil and particulate.

The first full-scale field tests (Phase B, October 16 to November 1, 1989) were divided into four categories: (1) surface OD; (2) suspended OD; (3) sequential OD; and (4) OB in pans. The surface OD operations involved detonating 2132 lbs of bulk TNT at ground level and the suspended OD operations involved detonating 2000 lbs of bulk TNT in a bucket approximately 35 ft above the ground. Seven surface OD and seven suspended OD tests were done with only one detonation done per grid site. The first test of each type was designated as the ORI (Operational Readiness Inspection). Its purpose was to familiarize field personnel with the procedures they would be doing during the actual field tests and to permit project management personnel to confirm that these procedures would be carried out properly. Soil and air samples were collected for each detonation; three detonations comprised one full test.

The OB tests involved burning three separate burns (6652 lbs., 6598 lbs., and 6614 lbs.) of a combination of AA-6, AA-2, MK-76, and MK-6JATO Navy propellant in burn pans. The first burn was designated as the ORI and the other two burns each comprised a full test. Soil, ash and air samples were collected from each burn.

The last activity--sequential OD--involved detonating three (3599 lbs., 3768 lbs., and 3579 lbs.) quantities of reclaimed TNT in sequence at the same grid site. The crater formed from the previous detonation was filled in and the next detonation set off over the filled-in crater. The objective of this last type of OD activity was to determine the cumulative impact of OD operations on soil pollution. In the surface and suspended OD tests, each detonation was done at a new site to provide as much replication as possible between detonations and to avoid cross contamination. Only soil samples were taken in the sequential OD tests.

The surface and suspended OD plumes and the OB plumes were sampled by SNL using their Twin Otter aircraft under the direction of Mr. Wayne Einfeld. The aircraft was stored overnight at the Provo Municipal Airport, but between detonations/burns it landed at Michael Army Airfield, DPG, where the samples were recovered. The aircraft collected: particulate samples using quartz fiber filters mounted in high volume sampler housings (three filters and housings per test to provide a sampling rate of approximately 200 m^3 min^{-1}); VOC's in stainless steel (SS) canisters and air samples in an 80 L Teflon bag. The Teflon bag's contents were subsequently analyzed for CO, CO_2, O_3 and NO/NO_2 using ambient air monitors installed in the aircraft. The monitors used were: Teco 48 (CO), Teco 41H (CO_2), Teco 49-100 (O_3) and CSI 1600 (NO/NO_2). The aircraft was also equipped with sensors for temperature, dewpoint, altitude, airspeed and real time measurement of aerosol particle size and concentration.

The canister samples were sent to Oregon Graduate Center (OGC), Beaverton, OR, for analysis for VOC's. Dr. Rei Rasmussen of OGC is responsible for these analyses. OGC is also responsible for measuring the CO and CO_2 content of each canister. These latter values are used to calculate the total pounds of each VOC released to the atmosphere from each OD and OB activity using the carbon balance technique.

The filter samples were sent to AWL for weight gain determination and subsequent analysis for semi-VOC by SFC/MS (with spot checks by GC/MS). AWL also cut a 1 square inch portion from each filter and sent it to Sunset Laboratories for particulate carbon analysis. A cut of the solvent extract and the entire extracted filter are both sent to Dr. Mangelson (BYU) for PIXE (particle-induced x-ray emission) analysis for metals.

AWL is also to receive: (1) soil samples collected from the craters and the fallout pans dispersed around the detonation sites; (2) the ash residue in the burn pans; (3) materials thrown from the burn pans and collected by the pans and trays that surrounded the burn trays; (4) samples of the blackened soil; and (5) samples of the tar paper and inhibitor residues. These samples have not been received by AWL as of January 11, 1990.

The soil and ash residue samples from all Phase B activities (surface OD, suspended OD, sequential OD and propellant OB) were collected by personnel from Lockheed Environmental Services (LESC). LESC personnel were responsible for all the ground support services needed by the Phase B test program with the exception of the munition and propellant set up/placement and ignition itself. LESC personnel, under the general direction of Jim Stephens, set out the test grid, put the fallout and sputter pans and trays in the position required by the test design, recovered and weighed the soil and ash samples, and stored the samples until they could be transported to AWL. The field operations were carried out under the supervision of Monty Law of LESC.

Environmental Laboratories Inc. (ELI), Provo, UT, has overall QA responsibility for the Phase B OB/OD project. ELI personnel, under the direction of Dr. Gary Booth, were responsible for documenting that all project Letters of Instructions (LOI's) were adhered to by the OB/OD participants. They were responsible for documenting instances where deviations from the LOI's occurred and for recommending corrective action. They were also responsible for ensuring that GLP (good laboratory practices) and GFP (good field practices) were followed throughout Phase B.

Personnel from the QA Division (QAD) of the EPA's Atmospheric Research and Exposure Assessment Laboratory (AREAL), Research Triangle Park, NC, conducted performance and systems audits on the OB/OD Phase B field tests and the laboratories (OGC, AWL) processing the samples. QAD has been participating in the OB/OD project at the request of the AMCCOM project manager (MacDonald Johnson) and the EPA's Office of Solid Waste (OSW). QAD provides external QA, performance samples and advice on QA matters.

Weather and operational delays caused the initial schedule to not be met. The schedule that finally was achieved was:

10/16	Surface OD	One detonation (ORI) 2,132 lbs. block TNT Air and soil samples taken.
10/17	Surface OD	Detonation trial (surface Series I) Three each of 2,132 lb. block TNT Air and soil samples taken.
10/18	Surface OD	Detonation trial (surface Series II) Three each of 2,132 lb. block TNT Air and soil samples taken.
10/19	OB	Burn ORI 6,606 lb. Navy propellants Air and residue samples taken.
10/25	OB	Burn trials (two separate burns sampled) 6,542 lb. Navy propellants 6,312 lb. Navy propellants Air and residue samples taken.
10/25	Suspended OD	Suspended detonation ORI 2,000 LB. block TNT
10/30	Sequential OD	Surface single detonation #1 3,599 lb. reclaimed TNT Double soil samples taken.
10/31	Suspended OD	Suspended detonation on trial Series I Three each x 2000 lb. block TNT Double soil samples taken. Air samples taken.
11/1	Sequential OD	Suspended detonation trial Series II 3 each x 2000 lb. block TNT Air and soil samples taken. Surface single detonation # 2 3,768 lb. reclaimed TNT Soil samples taken. Surface single detonation #3 3,579 lb. reclaimed TNT Soil samples taken.

QAD personnel were present for all surface OD tests (October 16-18), all OB tests, and for the suspended OD-ORI. QAD personnel also audited the SNL aircraft before the tests began and audited AWL during and after the Phase B field tests. OGC was audited in November.

2.0 AUDITS CONDUCTED BY QAD/AREAL

2.1 Aircraft Audits (Pretest)

Jack Bowen and Bud Dowler conducted performance and systems audits on the aircraft on October 13-15, 1989, while it was located at the Provo, UT, airport. The accuracy of the flow measurement portion of the aircraft's particulate sampler and the calibration accuracy of the CO, CO_2, O_3 and NO monitors were the primary focus of the performance audits. The operation of the nephelometers, data acquisition system, instrument operating procedures, certification dates of the calibration standards, and the operational readiness of the canister and Teflon bag sampler collection systems were the focus of the systems audits.

2.2 Systems Audits on DPG Operations

Elizabeth Hunike, Lisa Smith, Linda Porter, Raymond (Rocky) Rhodes and Bill Mitchell of QAD conducted systems audits on the DPG field activities. Systems audits were conducted on the aircraft during sample recovery at the DPG airfield, on the LESC field personnel setting out the explosives/propellants/fallout trays/sputter pans and recovering the soil samples and the ash samples; and the LESC personnel weighing these samples. Bill Mitchell was at DPG from October 16-17, Linda Porter from October 17-20, Rocky Rhodes from October 23-27 and Lisa Smith and Elizabeth Hunike from October 16-26.

2.3 Systems Audits on Alpine West Laboratories (AWL)

On October 19, E. Hunike, L. Porter and L. Smith visited AWL to assess the conformance of AWL personnel to the LOI's of the project and particularly to determine how the filter samples were being conditioned. On October 24, E. Hunike, L. Smith and R. Rhodes again visited AWL to see if corrective action had been taken concerning problems with the filter conditioning. L. Porter and L. Smith also visited this laboratory on December 5-6 to assess their conformance to the LOI's for filter extraction, filter conditioning and filter handling.

2.4 Systems Audit on Oregon Graduate Center (OGC)

Howard Crist of QAD conducted a systems audit on OGC in Beaverton, OR, on November 7, 1989, to assess OGC's conformance to their OB/OD LOI.

2.5 Performance Evaluation Samples

On November 17, 1989, NSI Environmental Services Inc., an on-site contractor for QAD, placed known quantities of VOC compounds at the ppb level in six stainless steel canisters and sent these canisters to OGC for analysis. These six canisters had been cleaned by OGC and sent to NSI for spiking.

On November 16, 1989, NSI sent five soil samples spiked with known μg quantities of some OB/OD target semivolatile compounds to AWL. NSI had received these soil samples on October 13 from AWL laboratories in acid washed jars and returned the samples to AWL in the same jars. The soil samples had been collected at DPG from an area where OB/OD operations had not been conducted according to DPG records. The materials were placed on the soil using the slurry/rotovac evaporation technique used previously (bangbox tests).

OGC and AWL will analyze these samples and return the results to QAD for comparison to the spiked values. The results will then be sent to the project manager, MacDonald Johnson.

3.0 AUDITING PROCEDURES

3.1 Performance Audits on Aircraft

The accuracy of the particulate sampler's flow was checked using a standard (L-type) pitot tube that had been borrowed from another EPA unit. The check was accomplished as follows. First, a 0.6 m extension was installed on the entrance to the aircraft's sampling probe and the standard pitot tube was inserted through a 1 cm hole located approximately 0.25 m from the inlet of the extension. The particulate sampler was then started and the flow read by the aircraft's pitot tube was measured and compared to the flow measured by the EPA pitot tube. (The EPA pitot tube flow measurement used the pressure drop across the pitot tube as measured by an electronic manometer.) When a relatively large difference was detected an additional 1 m extension was added. This additional extension had no effect on the results. (Subsequent QAD tests showed that the borrowed pitot tube had not been properly constructed, i.e., there were small burrs on the static pressure ports.)

The CO, CO_2 and O_3 monitors were audited using a QAD developed dynamic dilution system and compressed gas cylinders containing CO, CO_2 and SO_2. The audit consisted of challenging each monitor with known concentrations of O_3, CO_2 and CO, and comparing the monitors' results to the known concentration. A linear regression was used for this comparison. The QAD system can be used to calibrate monitors as well as audit them. Because the SNL NO calibrator was broken at the time of the audit, it was not possible for SNL to calibrate the NO analyzer. Instead, QAD used its system to calibrate the monitor. Thus, it was not possible for QAD to audit the NO monitor because the same system would have been used for the audit as was used to calibrate it.

3.2 Systems Audits

The OB/OD project's LOI's, QA project plan (QAPP) and work plan (WP) were used as the primary source of information for these audits. QAD's experience and knowledge of good laboratory practices (GLP) also served as a source of information for these audits. Mostly these audits involved observing the OB/OD project personnel as they carried out their assigned duties and comparing their activity to the section of the LOI, QAPP or WP that applied. (A systems audit form was used by each QAD auditor. This form was constructed using the information contained in the written documents associated with the project.) The observations of project personnel were supplemented with reviews of the logbooks and forms used by the project's personnel and by asking questions of these personnel to assess their knowledge of the project's objectives and procedures that they were responsible for carrying out.

4.0 AUDIT RESULTS

4.1 Performance Audit on Aircraft

The CO, CO_2 and O_3 monitors gave results that were within the target criteria (QAPP) of ±10% of the true value. (As mentioned in Section 3.1, the NO instrument could not be audited.) Originally, the EPA pitot tube gave a flow reading 15% higher than the aircraft's pitot tube. A subsequent check of the EPA pitot tube's calibration using both QAD's rootsmeter and also a QAD L-type (standard) pitot tube determined that the EPA tube used in the audit was in error by approximately -15%. When this correction was made the aircraft's pitot tube and the EPA pitot tube agreed within 2.7%. (As mentioned earlier, the EPA pitot tube used had been borrowed from another EPA unit. It likely had never been used before and, when inspected by QAD personnel after the audit, was found to have burrs around the static pressure orifices. This pitot tube was used because it was considerably shorter than the QAD pitot tube and thus was easier to ship to the site.)

The actual audit results obtained were:

Monitor	Slope	Intercept	Corr Coeff
Teco 41H - CO_2	0.938	21.3	0.9999
Teco 48 - CO	1.06	0.4	0.9980
Teco 49 - O_3	0.983	1.2	0.9997

The individual data points are presented in Appendix A (CO, CO_2, O_3 Monitor Audit Results). The QAPP specified that all these monitors have a slope (based on the linear regression) between 0.90

and 1.10. All three monitors met this specification and the specification for intercept.

Because SNL had no information on the stability of O_3, NO and NO_2 in the Teflon bag used to collect these gases, SNL and QAD personnel attempted to check the stability of these gases in the Teflon bag. Only the stability of NO was checked, however, because of time limitations. For the NO check, the QAD audit system was used to fill the bag with 30 L of air containing 500 ppb NO. The NO concentration was then measured using the CSI 1600 at 18, 21, 24, 27 and 30 min after the bag had been filled. No decay was observed. SNL agreed to evaluate the stability of NO_2 and O_3 if time permitted during the test schedule or after the aircraft had returned to Albuquerque and submit the results to QAD and ELI. QAD has not received this information to date. These stability checks should be done and reported, if they have not already been done.

4.2 Systems Audits

4.2.1 Aircraft

The condition of the gas monitors and conversations with SNL personnel at the beginning of the audit indicated that the monitors were not checked out before they were placed on the aircraft, nor were they checked out before the aircraft left Albuquerque. The CSI 1600 monitor had a defective output board and one had to be sent from SNL in Albuquerque. Further, it was found that the SNL NO/NO_2 calibrator had a broken mixing chamber and a new one needed to be sent from SNL in Albuquerque. Since neither the board nor the chamber had arrived by October 15, QAD offered to calibrate the CSI 1600 using the QAD audit equipment. This calibration was done by measuring the CSI 1600's output at a point before the defective board. (We were later told that the output board arrived on October 16, 1989.)

It was also found that the SNL data acquisition system would not accept O_3 values greater than 100 ppb. However, this problem should not affect the OB/OD test results because the monitor's digital readout meter could be read easily and because the O_3 levels likely to be encountered during the testing should be less than 100 ppb.

The system that was to be used by SNL to check that the stainless steel canisters were evacuated before use and to indicate that a sample was being collected (decrease in vacuum) had not been shipped in time to be checked out by the QAD audit team. Auditors were told that it would arrive tomorrow or the day after tomorrow over the period October 13 to October 19. The actual date it arrived is unknown to QAD personnel since we never saw it.

One of the recommendations of an earlier QAD audit (bangbox, February 1989) was that SNL develop a spare parts inventory, particularly before any OB/OD field testing begins. We want to reemphasize this earlier recommendation.

Otherwise, the aircraft equipment was in good shape. The data acquisition system (DAS), aerosol probes, and temperature and dew point sensors had been calibrated within the last year. The altitude and velocity sensors had not been calibrated within the last year, but were scheduled for calibration in November 1989. (The QAPP stated that all these instruments would have been calibrated within the last year.) The records and documentation were found to be adequately maintained and the SNL personnel were knowledgeable about the purpose of the OB/OD tests and were proficient in carrying out their responsibilities. More details on the systems audit of the aircraft are given in Appendix B (Systems Audit Results at DPG, pp 1-4).

It did appear to the auditors that the sample labelling and tracking scheme was cumbersome and subject to transcription errors. A simpler system should be developed, if possible.

4.2.2 DPG

In general, the LESC personnel were proficient in their duties. They were cognizant of the importance of avoiding sample contamination, ensuring that samples were properly labelled and completing all data and report sheets fully. QAD personnel did observe some minor deviations from the LOI's; but, in our opinion, none of the deviations should affect data quality. When deviations from the LOI's were brought to the attention of the appropriate LESC person, corrective action was taken immediately. It was apparent that LESC's management (Jim Stephens, Monty Law) had spent considerable effort in training their field personnel. Mr. Law should also be commended for the efforts he took to ensure that all samples were recovered and properly labelled. He was observed to be working 12-15 hr each day during the period when surface detonations were being done.

More details on the systems audit are presented in Appendix B (Systems Audit Results at DPG, pp 4-14).

4.2.3 OGC

The QAD auditor concluded that OGC will produce valid OBOD data of documentable quality through their careful calibration and canister cleaning procedures. OGC has dedicated a GC/FID and an operator (Bob Watkins) to the analysis of the OB/OD canister samples. Eight canisters are analyzed per day and a one-point calibration check is performed daily using a neohexane standard 226 ppb, Scott Specialty Gases, Plumsteadville, PA). Three injections are made and the agreement obtained with the GC/FID's calibration curve is usually within 3%. Every six months the neohexane standard is intercompared to benzene and propane SRM's from NIST (formerly NBS). The GC/FID system is exceptionally stable with little daily drift. A humidified zero air sample is also run daily to check for system cleanliness. (The GC/FID system is subjected to a full-scale calibration every few months.)

The samples are analyzed by taking a 500 ml aliquot and condensing the organics in a trap cooled with liquid oxygen. The trap is then heated to revolatilize the organics so they can be passed into the GC/FID using helium as the carrier gas. Compounds are identified by their retention times and the data are then given to Dr. Rasmussen who checks the data for reasonableness. If necessary, Dr. Rasmussen orders other confirmatory tests (such as GC/MS), but this is rarely necessary according to Dr. Rasmussen. No statistical tests are done on the data.

The analytical procedure used by OGC is adapted from Method TO-14 in the USEPA guidance document "Compendium of Methods for the Determination of Toxic Organics in Ambient Air."

After the analysis is completed to Dr. Rasmussen's satisfaction, the canister is cleaned by evacuating it to a vacuum pressure of 50 millitorr while maintaining the canister at 100 to 125°C. After cleaning, approximately 2% of the canisters are checked for contamination. After cleaning, all canisters are evacuated and stored for 3 to 5 days; the vacuum in each canister is then checked to ensure the canister is leak free.

4.2.4 AWL

On October 19, the QAD audit team observed that particulate had been lost from the quartz filters used in the surface OD tests. This particulate had been transferred to the Teflon sheets between which the filter had been stored in the envelope. Dr. Christine Rouse of AWL was aware of the situation and was open to suggestions on how to correct the problem. These suggestions included:

1) recovering the particulate from the Teflon sheets and placing it back onto the filter or into a tared beaker;

2) folding the filters differently at DPG;

3) maintaining the folded filter in a vertical position and in a press during shipment; and

4) placing the filter in a rack during sample conditioning such that it would open into a "V" shape.

The auditors also suggested that the thermometer in the freezer that would store the filter and soil extracts at -20°C should be able to read -20°C (its lowest graduation was -10°C). There were no extracts in the freezer at the time. It was also suggested that the % RH and room temperature be recorded when the filters were weighed since the balance was located in a room that could not maintain conditions of temperature and % RH recommended for filter weighing. (These EPA environmental conditions are guidelines; they are not mandatory requirements!) AWL responded to these suggestions by monitoring temperature and % RH in the room where the balances were located and the filters conditioned using real-time continuous recorders over the time of the project. AWL has reported that the recordings show that the recommended conditions were maintained. QAD requests copies of the recorder charts for the period October 14-November 14, 1989. The audit team was told that the balance being used was recently calibrated (October 13, 1989) using NIST traceable weights, but there was no written record available for the auditors to review. The last certified calibration, based on a tag on the balance, was December 18, 1987. At a later date, Mr. Lon Que Adams of the BYU Instrument Shop stated that AWL's balances had been calibrated prior to the filter weighings.

A second audit was done at AWL on October 24, 1989. At this time, it was observed that the QC filters had all been weighed at the same time, and it was uncertain as to how many weighings had been done on each filter. (These filters were to provide an assessment of the impact that changes in the balance room's % RH and temperature had on the filter weights over the time that the filters were being conditioned). A set of filter tare weights was given to the auditors with the statement that the QC filters had been weighed twice. However, inspection of the data the next day showed that the second weighing had yielded values identical to the first weighing. When contacted about this, Dr. Rouse said a mistake had been made and that she would send a corrected data sheet. A corrected data sheet was finally received January 11, 1990, with a statement that the filters were weighed in a filter holder using a mechanical balance. AWL reported that three repetitive weights, one after another, were taken and that 10 hours was required to weigh the full set of filters with this procedure. In their response, AWL also stated that every fifth filter was reweighed "four days after the first weighing." QAD notes that the AWL data provided indicates that the first weighing was on Saturday, October 14, and the second on Monday, October 16, _two_ days later.

It was also observed at the second audit that the logbook on filter weights was not clear as to the date, analyst, % RH and temperature. Thus, it was not possible to inspect the filter weights obtained over time. It was recommended to AWL and to ELI that the documentation for the filter weighings be improved. It was also suggested that a small fan be operated in the balance room to assure uniform temperature and humidity throughout the room and to thus hasten and maintain equilibrium conditions. It was suggested that the fan should be placed away from the filters, aimed at the wall, and turned off during filter weighing. QAD also suggested that the filters be placed in a "V" open position rather than lying flat.

A third audit was done on December 5 and 6 to assess what corrective action had been taken. The information promised to QAD on the corrected QC filter weights had not been received at this time, despite being promised. The auditors found that the filter logbook was still not easy to review. The filter weighing books were found to be insufficiently documented. There were many notations and weights entered, but the auditors could not easily follow the sequential weighings for any specific filter. There were some summary sheets of filter weights available, but there were some discrepancies between these summary tables and the weights in the logbook. Possibly these discrepancies were false, i.e., due to the auditor's difficulty in following the sequence of weighings recorded in the logbook rather than to an error on the part of AWL. AWL has since decided to change their recording procedure in the logbook; they plan to record filters by sample in tha future. It was also learned that AWL had switched balances at some point during the filter weighings. From a visual scan of the tabulated data, it looked as if the second balance differed by approximately 10 mg from the first. Further, all filters had been extracted so additional weighings were not possible. (The one square inch cutting had been done and sent to Sunset Labs before the extraction.)

Because the option to reweigh the filters was not available, the auditors decided to assess how the 10 mg difference between the two balances might affect the filter results. This assessment was accomplished on December 7 (after the auditors had returned to RTP, NC) using photocopies of pages in the filter weighing logbook. This assessment showed that a 10 mg error would have little impact on the surface OD filters since these filters collected between 155 and 290 mg (based on the weights for the first balance only!). However, the impact on the OB and suspended OD tests could be significant since the filters for these tests collected less than 10 mg (OB) and 15-25 mg (suspended OD), respectively. It was also noted in doing this assessment that the background particulate samples collected prior to each test series had lost between 5 and 12 mg. The reason for this is not known, but it is very likely that fibers are lost from the filters during sampling or handling since the filters are heated at 650°C for 8 hr before use (and thus could become brittle). To be conservative, all exposed filter weights should be increased by 10 mg when the OB/OD results are tabulated.

The auditors also learned that the particulate lost from the filters during transport had been shaken off the Teflon sheets onto the appropriate filter. However, there was no documentation of when this was done. AWL reported that particulate lost from the filters during transport was put back on the filter at arrival as the filters were laid out for conditioning within an estimated ten minutes from the time and date that they were signed off for receiving. The auditors were also told that the particulate on filters collected after October 19 had not been lost because the changes needed to avoid particulate loss during transport had been done. Also, when filters had lost particulate during shipment, the lab noted this on the data sheet that accompanied the filter. Inspection of the data sheets showed that only the earlier data sheets noted particulate loss.

Also, one of the logbook pages (p. 25), dated November 29, 1989, contains the statement, "all quantitation values for samples (except F.B.) must be multiplied by 2." The log provided no explanation or reason for this. AWL later reported that quantitation of samples that were combined from two ELI samples had to be multiplied by two in order to obtain actual concentration.

The situation with the filters was found to be unsatisfactory and immediate corrective action was deemed a necessity. For example, NIST weights should be used to determine which balance is accurate and the filter weights from the first balance only should be used if the filters had come to a constant weight before the balance switch was made. QAD suggested that AWL conduct an intercomparison study between the two balances used for filter weighing. AWL reported at a later date that the two balances had been correlated to each other at a single test weight, and it was found that they were both accurate to a tenth of a mg.

The auditors were surprised that ELI had not conducted any follow-up audits (to the October 19 and October 24 QAD audits) to ensure that effective corrective action had been taken. (The μg of semi-VOC per gram of particulate released as a part of OB and OD operations is of high interest to EPA and to the States since this particulate can be inhaled and retained by living beings far away from the OB/OD grounds.)

Other findings of the December 5 and 6 audit of the AWL laboratory were:

1) The % RH and temperature in the weighing room remained quite stable over the period mid-October to the last week of November. For example, the % RH ranged from 15 to 25% over this time period and the temperature ranged from 78 to 83°C.

2) In contrast to the filter weight situation, the SFC/MS work and the sample extraction efforts conducted to date were well documented. Calibration standards are made for each OB/OD target compound and calibration checks are done daily on the MS. Every three months a Finnigan representative (Finnigan manufactured the MS) performs a maintenance check on the MS system.

3) The MS system automatically rejects any data where the QC sample result differs from the calibration curve by more than 10% and an internal standard is run daily. Further, the MS response factor obtained for each OB/OD target compound is plotted each time such a value is taken and compared to the historical response factor for that analyte. (The acceptance criteria is 10%.) If either of

the above 10% limits is exceeded, recalibration of the MS system is required.

4) The MS logbook was very complete and seemed to contain all the data needed to track a sample from extraction to analysis.

5) The extraction of the filters was well documented. The five QC filters were extracted along with the filter samples and every sixth filter extracted was a blank.

6) The thermometer in the freezer in which the extracts are stored had been changed to one that was readable to -20°C before the samples were stored in the freezer.

4.3 Performance Evaluation Sample Results

The canisters containing known quantities of VOC's and the soil samples containing the target semi-VOC compounds have been received by OGC and AWL, respectively. The analytical results are expected from OGC by the end of December and from AWL by mid-February 1990.

5.0 RECOMMENDATIONS

1) More frequent audits of AWL should be done by ELI, the OB/OD contractor with overall responsibility for QA.

2) The OB/OD project manager, or his AMCCOM representative, should be at the field test site (DPG) when the tests are being done, until at least they become a routine procedure.

3) Project personnel should ensure that spare parts are available for all equipment on the aircraft for which equipment failure will cause a significant delay in the test schedule.

4) The aircraft and its air sampling equipment should be thoroughly checked out and calibrated before it leaves SNL.

5) Better record keeping of the filter weights and the weighing system is needed and the difference between the balances needs to be investigated.

6) Procedures for conditioning, homogenizing, and blending the soil and pan samples and for removing debris and vegetation, etc. need to be fully developed before these samples are extracted and analyzed.

Appendix A

CO_2, CO, O_3, NO, NO_x Monitors and Flow Audit Results

CO2-CO AUDIT RESULTS
TWIN OTTER AIRCRAFT
PROVO, UTAH   OCTOBER 14, 1989

Model S/N	Range ppm	Audit Conc. ppm	Analyzer Response ppm	Difference ppm	Difference percent	Regression-Response on Audit Conc. Y=mX+b Slope	Intercept	Corr. Coeff.
TECO/41-H	[CO2]	355	353	-2	-0.6			
		455	450	-5	-1.0	0.9378	21.35	0.9999
		655	635	-20	-3.0			
TECO/48	[CO]	0.0	0.0	---	---	1.059	0.395	0.9980
		37.0	39.0	2.0	5.0			
		15.0	18.0	3.0	20.0			
		6.0	6.0	0.0	0.0			

O3 AUDIT RESULTS
TWIN OTTER AIRCRAFT
PROVO, UTAH OCTOBER 15, 1989

Model S/N	Range ppb	Audit Conc. ppb	Analyzer Response ppb	Difference		Regression-Response on Audit Conc. Y=mX+b		
				ppb	percent	Slope	Intercept	Corr. Coeff.
TECO	0-1000*	0.0	0.0	---	---			
49-100		25.0	27.0	2.0	8.0	0.983	1.189	0.9997
		50.0	51.0	1.0	2.0			
		75.0	75.0	0.0	0.0			
		95.0	94.0	-1.0	-1.0			

* AIRCRAFT DATA SYSTEM WAS OVER RANGE AT CONCENTRATIONS ABOVE 100 PPB. INSTRUMENT DISPLAY WOULD READ CONCENTRATIONS ABOVE 100 PPB.

NO-NO2-NOX CALIBRATION RESULTS
TWIN OTTER AIRCRAFT
PROVO, UTAH OCTOBER 15, 1989

Model S/N	Range ppb	Audit Conc. ppb	Analyzer Response ppb	Difference		Regression-Response on Audit Conc. Y=mX+b		
				ppb	percent	Slope	Intercept	Corr. Coeff.
CSI	0-5	0.0	0.0					
1600	[NO]	0.50	0.50			1.00	0	1.00
		0.76	0.76					
		1.02	1.02					
	[NOX]	0.0	0.0			1.001	-0.003	0.9999
		0.50	0.49					
		0.76	0.76					
		1.02	1.02					

NOTE: ANALYZER WAS CALIBRATED USING EPA GAS DILUTION SYSTEM AND [NO] STANDARD.

OB/OD FLOW AUDIT RESULTS
AIRCRAFT TRANSPORT TUBE
PROVO, UTAH OCTOBER 15, 1989

Sampler I.D. Date	Sampler Flow SCFM	Audit Flow SCFM	Percent Difference
HIGH VOLUME SAMPLER WITH FILTERS HAVING 6 1/4 INCH SQUARE HOLES.	172	177	2.9
HIGH VOLUME SAMPLERS WITH NO FILTERS.	296	304	2.7

* All flows compared at 25deg C. and 760mmHg

Appendix B

Systems Audit Results at DPG

(Each EPA/QAD auditor at DPG used the same audit form. The completed audit form in this appendix is an integration of all forms completed by QAD auditors.)

EPA/QAD/RTP NC.

OB/OD FIELD STUDY SYSTEMS AUDIT
DUGWAY PROVING GROUND, UTAH

Audited By: E. HUNIKE, L. SMITH, L. PORTER, R. RHODES, W. MITCHELL   Dates: Oct 16-26, 1989

Test Description: SURFACE AND SUSPENDED OPEN DETONATIONS AND SURFACE BURNS

A. AIRCRAFT SYSTEMS AUDIT

General

1. Do personnel appear to be familiar with the procedure?

   Yes

2. Do they seem to have an understanding of the principles involved with the procedure or is it all done by rote?

   They do seem to understand based on the review of their logbooks and their proficiency

3. Is a copy of the SOP/LOI available on location?

   Yes - for gaseous monitors. Not sure if the LOI's for the particle collector/sizers were there

4. Is the SOP/LOI being followed?

   Yes, to the extent we could tell. There were some minor deviations from the written LOI's but these deviations had been approved by the proj. offic

Logbooks

1. Are CO, CO_2, NO_x, O_3, and nephelometer zero and span checks being done daily and entered into logbook?

   Yes, based on a review of the logbooks. Reviews were done on Oct. 17 and 18.

2. If span value for (1) is > ±15%, is corrective action taken?

   We did not observe any instance when the span value exceeded ±15%.

Data Acquisition System

1. Is a zero and span of CO, NOx, CO_2, O_3 monitors done before and after plume is sampled?

Yes

Filter Handling (Loading/Unloading)

1. Were the filter housings cleaned with isopropanol? What grade was used? Was it done thoroughly?

Housings are cleaned before the first flight of the day using #2 grade isopropanol.

2. Did personnel wear gloves while handling the filter? What type? (Cotton gloves are not acceptable.)

Cotton gloves were worn on October 16, 1989, plastic gloves thereafter.

3. Was the filter handled ONLY with tweezers?

Yes.

4. Was the foam gasket cleaned with isopropanol before the filter is put in the filter holder?

Gasket was cleaned before the filter was installed for the first flight of the day. It was not cleaned again until the next day.

5. When assembled, is the filter holder tightly sealed against the gasket?

Yes.

6. How are the filters numbered or labeled?

Filters are not numbered or labelled. They are kept in marked envelopes that have the filter ID written on the outside.

7. Are the filter holders/units labeled as to position in the aircraft manifold?

Yes, they are labelled A, B and C. The filters are given the appropriate letter designation when they are installed. This letter designation is written on the filter storage envelop and in the aircraft's log book.

8. Are the filter holders installed in the correct position in the manifold?

They were properly installed when checked on Oct 16, 17.

9. Are adequate records kept (e.g., filter #, filter holder position, etc.)? Are these kept in a logbook?

Records are kept in a logbook. They seem to be adequate.

10. Are the air flow sensor wires plugged into their appropriate places on the junction box? Is this checked after the installation of the filter holders?

Sensors were properly plugged in when checked on Oct 16, 17.

11. During filter unloading, are steps 1-10 followed in reverse order?

They were were checked on Oct 16, 17.

12. Are steps taken to protect the filter from contamination? What are they?

Yes. Filters are recovered one at a time and exposure to the air is limited to only a few minutes during loading and unloading.

13. Is the filter folded properly according to the SOP/LOI (folded twice) and put in the foil container?

Filter folding procedure was changed after LOI was written for Phase 6 Tests. Filter folding procedure was changed also during Phase B to minimize particle losses

14. Is any particulate lost during filter removal and folding?

No particulate was lost during filter removal and folding, however some was found to come off filter during shipment to lab (AEWL). See notes section.

15. Is the filter put in dry ice storage? When?

No dry ice was available on Oct 16, 17 or 18 at time aircraft was audited.

16. Do personnel use good laboratory techniques in this procedure (i.e, gloves worn during filter handling; tweezers used; avoidance of contamination of filter; filter removed from filter holder in a clean environment)?

Yes to all questions.

17. Are filter ID numbers on the filter and on the foil container? Is all sample information written on the label and/or form?

Filters are not labelled. The manila envelope in which they are stored in labelled with a filter ID number and the filter's position and test in which used.

18. Are forms being completed properly?

Yes, when checked on Oct 16, 17.

Canisters

1. Are canisters properly attached to sys~em (plumbing)?

Yes, when checked on Oct. 16, 17.

2. Are canisters properly labeled?

Yes, when checked on Oct. 16, 17.

3. Are the correct number being taken per flight?

LOI says one canister per pass but Wayne Einfeld (SNL) said that only one could be taken per flight because of time and space restrictions.

4. Are forms being completed correctly and fully?

Yes, when checked on Oct 16, 17.

5. Are canisters checked for leaks before use? If so, how done.

The leak check system had not been received as of October 19, 1989. Wayne Einfeld said that it arrived on October 20, 1989 one was used thereafter. Hopefully ELI's records will confirm this.

B. SOIL/ASH SAMPLES

General For All Types (Comments Cover Period Oct 16-26, 1989)

1. Did all personnel have on clean cotton gloves when handling soil/fallout samples and equipment?

   Yes, each time checked between Oct 16-26.

2. Was there smoking at test grids or in vehicles transporting samples or while processing samples?

   NO instances observed between Oct 16-26.

3. Were vehicles operated within 100 feet of soil samples?

   Only one instance observed and pan was covered at that time.

4. Were fallout pan cov blown clean after each test with a laboratory quality compressed ogen gas?

   Covers were blown clean using N_2 prior to each test.

5. Were fallout pan covers removed from grids and placed in covered vehicles prior to test?

   Covers were placed uncovered in pick up trucks.

6. Was a QA person present while all soil samples and/or fallout samples taken?

   LEMSCO person responsible for QA was present each time checked

7. Were fallout pans or sputter pans cleaned between tests? How was this done?

   Pans were blown clean with N_2

8. Are sample trays for fallout or sputter (burn) properly labeled and positioned?

   Yes, however, during burns the stakes and paper labels were charred. Trays had to be repositioned with respect to LOI for burns. See notes section.

9. How are the pans and positions labeled?

Stakes mark position and pans are inscribed with a permanent ID number and also have a label that identifies the pan's position with respect to the stake

10. Do personnel have data sheets for all activities?

Yes. One instance was observed where the data sheet did not have the pan ID numbers written on it. This was quickly corrected when the auditor asked why.

11. Do they have a copy of the SOP/LOI for their activities on site?

Yes. There are 3 people to a team. Two collect samples and move pan into position and third collects information needed to complete the form.

Ejecta Material (Surface Detonation)

1. Are the samples taken following procedures outlined in the SOP/LOI?

Yes.

2. Are the personnel familiar with the sampling procedure?

Yes.

3. Are they knowledgeable of the reasons/principles for doing the procedure or is it done by rote?

Yes.

4. Are precautions taken to avoid sample contamination? What are they?

Yes. All personnel wear gloves when collecting sample or moving pans and they change gloves after each sample.

5. Are samples recovered in the right order (from outside to center of crater)?

Yes.

6. Is any sample lost during the collection procedure?

Some sample was lost during recovery due to wind. However, losses were very small when observed and the sample recovery teams did their best to protect samples from the wind during recovery.

7. How many samples were collected?

LOI - surface OD - 24 trays, 13 crater samples.
Other surface OD - 12 trays, 13 crater samples.

8. Are they taken from the locations called for in the SOP/LOI?

Yes.

9. Was one (1) sample taken in the center of the crater?

Yes.

10. How was the center of the crater determined?

At first it was estimated to be the point where the explosive set, but after Oct 18 the deepest point was selected as the center.

11. Were four (4) samples taken 3 meters from the center of the crater (based on LOI)?

Yes.

12. Were four (4) samples taken 1 meter from the rim of the crater (based on LOI)?

Yes.

13. Were four (4) samples taken 4 meters from the rim of the crater (based on LOI)?

Yes.

14. Did the personnel make sure the sample consisted only of the loose ejecta material by following the LOI?

Yes to the extent possible.

15. How were these samples labeled?

Samples were labelled with preprinted, paper labels. The labels showed the date, test type, position, ELI and EPA #'s and the collection laboratories' name. Information could also be added concerning the gross weight, tare weight and net weight of the sample.

16. How is the sample integrity assured (e.g., labeled correctly; no mix-ups; no contamination, etc.)?

Once sample is in the jar, the jar is sealed and left next to the pan/sample position. A second person comes to check that the label is correct and completed fully and then picks up the sam

17. How is the sample position reflected in the labeling process? Is it clear? Is it adequate to ensure the samples can't be mixed up or mis-identified?

yes, sample position is reflected on the the label as per the LOI.

Fallout Pan/Burn Pan Procedures

1. Do pans have permanent ID number? Were dimensions of pans measured and recorded?

Yes - all pans had a permanent ID imprinted on th sides and dimensions were recorded. Some were 1 m^2 an others 1 yd^2.

2. Were pans staked to the ground with 4 metal stakes?

Four stakes were used during OD test and 2 stakes we used for the OB tests.

3. Are sample recovery procedures outlined in LOI being followed (i.e., proper order of recovery; attempt made to keep vehicle dust from getting into pan; funnel being used; trays cleaned before reuse)?

yes.

4. Are personnel familiar with the sampling procedure?

yes.

5. Are they knowledgeable about the reasons/principles for doing the procedure or is it done by rote?

yes.

6. Are precautions taken to avoid sample contamination? What are they?

Yes. Clean brushes, gloves, funnels and jars are used at each pan location.

7. Was fallout material brushed into acid washed bottle using a clean nylon bristle brush and a stainless steel or glass funnel? Or scooped out and then brushed out?

A paper funnel was used since metal and steel funnels were not available.

8. Were bottles sealed with Teflon lined lids?

Yes

9. Was brush blown out after every pan sample with lab quality compressed nitrogen gas? Was scoop cleaned?

Scoops were not used. Brushes were blown clean with N_2 (in a cylinder). Note - bristles from the brush that fell into the jar were not removed.

10. Were sample bottles labeled with correct ID number as on fallout pans?

Yes, each time checked on Oct. 17, 18 and 25.

11. Is any sample lost during sample recovery (transfer to weighted bottle)?

A minor amount was lost due to wind at times.

12. How many samples were collected?

All required by LOI.

13. Was sample custody form completed?

Yes.

14. Is someone checking the jars at the tray (after they have been filled) for correct labeling and identification of the sample?

Yes.

15. Has balance used to tare jars been calibrated recently?

Yes.

16. What methods are being used to obtain QC and precision data on weighing of jars?

A standard weight (50 or 1500 g) was weighed afte every 5th sample jar and the weight was recorded.

17. Are soil/tray samples being properly stored?

Yes, as far as auditors could tell.

18. Do personnel involved in soil sample (crater) and tray sample recovery know their duties and responsibilities?

Yes.

19. Are sputter pans (for burns) within 1 meter of the burn pans?

Yes.

20. Are fallout pans (for burns) located properly? How are they located?

Yes. They are located according to the LOI.

21. Is the residue from the burn pan collected according to the SOP/LOI?

The LOI specified waiting 4 hr after burn, but pans w found to be cool enough after 2 hr. Thus, samples wer recovered after 2 hr.

22. Are all samples stored under proper conditions as outlined in the SOP/LOI?

Yes, as far as auditors could tell.

Suspended Detonation Sampling

1. Are sample recovery procedures outlined in LOI being followed?

Yes

2. Are personnel familiar with the sampling procedure?

Yes.

3. Are they knowledgeable about the reasons/principles for doing the procedure or is it done by rote?

Yes.

4. Are precautions taken to avoid sample contamination? What are they?

Yes.

5. Is soil sampling tool a ring 20 cm in diameter and 2.5 cm deep?

Yes

6. Was soil within ring removed with Teflon covered plastic scoop?

A wide metal spatula was used because a Teflon covered scoop could not be found.

7. Were samples put in acid washed bottles with Teflon lined lids?

Yes.

8. Were samples labeled properly?

Yes.

9. Were five (5) soil samples taken from each main quadrant?

Yes.

10. Was any sample lost during sample recovery?

No instance of sample loss was observed.

11. Was sample custody form completed?

Yes.

C. EXPLOSIVES

1. Have explosives being used been verified before blow/burn?

Yes, when checked on Oct 16 and 18.

2. Are explosives being placed in proper orientation (stacking) for blows?

Yes, when checked on Oct. 16 and 18.

3. Is schedule being followed as to type of blow/burn?

No. Aircraft problems and weather condition caused unavoidable delays. All tests were accomplished.

D. VIDEO

1. Are cameras properly placed?

Yes, as far as auditors could tell.

2. Are they being activated on schedule?

Yes, as far as auditors could tell.

Special Notes

1. Filter Handling Procedures

In the pilot test (DPG, June 1989), each filter was sent to the field in aluminum foil and after use the filter was folded twice and sealed in the aluminum foil. In Phase B, however, each filter was placed between Teflon sheets and stored in a manila-type mailing envelope during shipment to the test site. After use, each filter was folded once, placed between the Teflon sheets and returned to the shipping envelope.

When the laboratory (AWL) handling the filters was audited on October 19, 1989, it was observed that some particulate had come off the filter during transport. It was suggested that the filters should be folded twice and then kept in a vertical position and under pressure during shipment. We do not have any documentation that this change was made. Hopefully, ELI records, AWL records or SNL records will indicate if and when these changes were made.

It was also suggested that the filters should be conditioned in the vertical position rather than lying flat and that the particulate that had come off the filters be recovered and placed back on the appropriate filter. We were told (December 5, 1989) that this (particulate recovery) was done, but there was no documentation of when or how.

2. OB Sputter and Fallout Pan Repositioning

The original diagram/LOI indicated that it was possible to locate a fallout pan at each end of the burn tray at a point 5 m from the center of the middle tray. However, when the pans were being positioned, it was found that sputter pans were already located at the positions where these two fallout pans would be located. Therefore, the fallout pans at 0° and 180° on the 6 m diameter were eliminated. The revised diagram is attached.

3. Weight Loss by Jars Due to Low Humidity at DPG

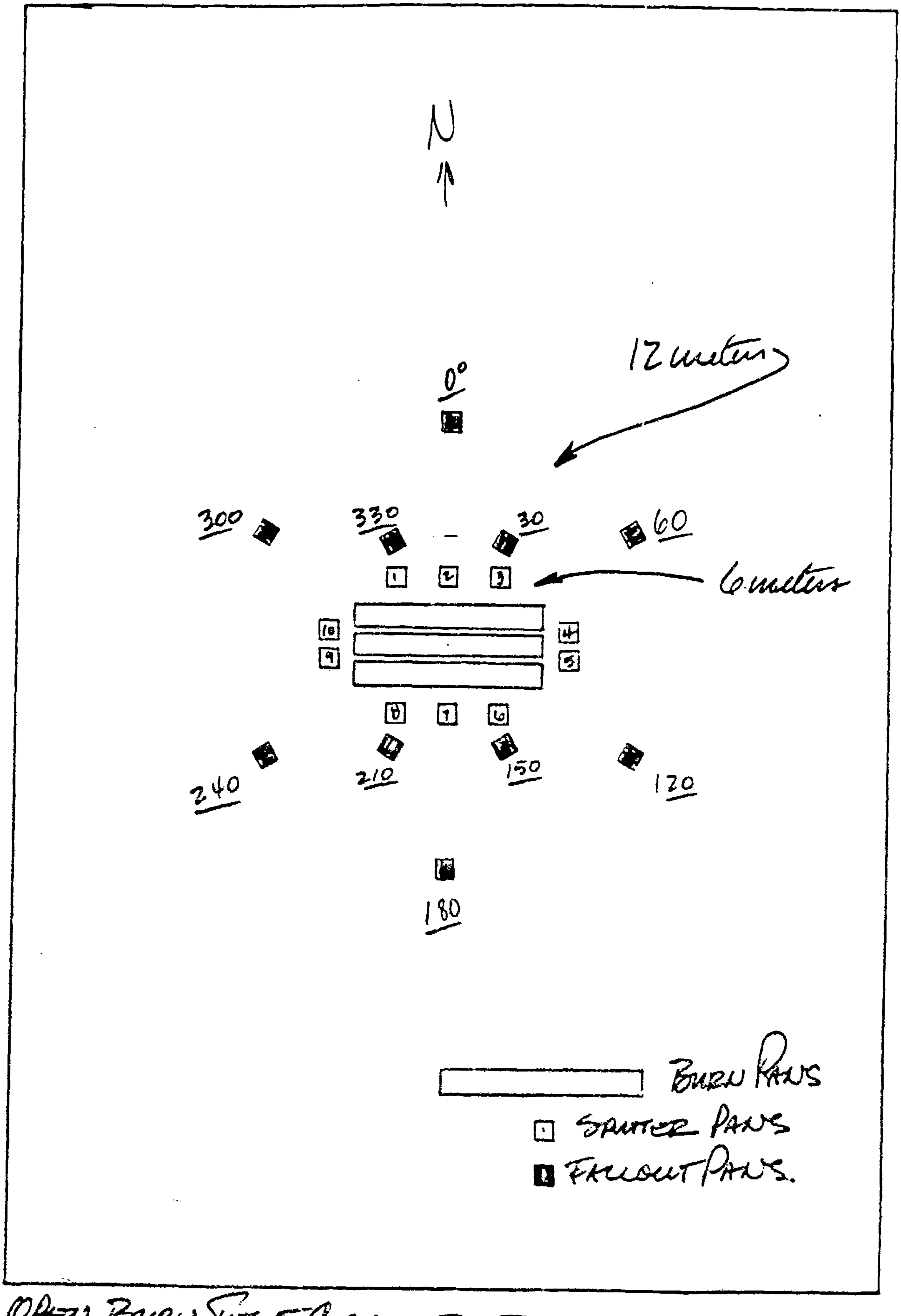
It was observed by Monty Law and Linda Porter that jars shipped to DPG, but not used, had lost weight. Although the weight loss was small (0.5 g), it might be prudent to condition the jars in the same environmental conditions before and after use.

4. Removing Vegetation from Soil and Tray Samples

It is obvious that vegetation should be removed from the soil and tray samples before they are extracted. However, the procedure to do this does not seem to have been agreed upon (e.g., sieving the sample, picking it out with tweezers). Obviously, this should be decided before the soil, fallout and ash samples are extracted.

5. Correcting for Soil Moisture Content

Like the vegetation removal situation, there does not seem to be agreement on how to determine (or even if there is a need to determine) the moisture content of the soil. A decision should be made quickly. Certainly the samples to be extracted should not be used to determine the moisture in the sample, but, if a decision is not made soon, this might accidentally occur.



OPEN BURN TEST GRND. I & II

Letter MD-77B, February 8, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

ENCLOSURE 10

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

February 8, 1990

Mr. MacDonald Johnson, AMCCOM
31 Potter Street
Salt Lake City, UT 84113

Dear Don:

As you requested, I reviewed the ELI QA/QC report for the bang box tests that I received yesterday. In my opinion, the report is poorly written and organized; it is not focused, is incomplete and has many grammatical problems. The sentence structures in many parts are terrible. I definitely would not release it in its present condition. A major rewrite is needed.

The report closely resembles the draft Rocky and I reviewed a long time ago (September 1989?). I was surprised that our comments and suggestions were ignored, particularly since Rocky spent hours going over the report's deficiencies with Gary. The bang box tests were completed by February 1989. Why has it taken so long to prepare a report? The test design was very simple and the QC and QA activities very straightforward.

I have written many comments on the enclosed copy of the report. Few are complimentary. It is obvious that ELI personnel do not understand the characteristics and operation of the SNL monitors for gases and particles.

I believe the concept of completeness used in the report is erroneous in relation to the OB/OD program. I think it needs to be redefined.

Some other points about the report:

(1) Normally, a QA/QC report presents a short description of the test/experimental design.

(2) Normally, a table follows the page on which it was cited unless the report is very short.

(3) Some of the pages in the body of the report are not numbered. Simple carelessness!

(4) Appendices should have numbered pages, particularly since there are more than 125 pages of appendices.

(5) The number of appendices seems excessive. How many of them are really necessary to understand the report? A two-page description of the test design might let you eliminate the appendices.

(6) The report seems incomplete to me. It assumes the reader knows all there is to know about the test design and how it was done. This is a major deficiency.

I suggest you find a technical editor for the report.

Sincerely,

William J. Mitchell
Chief, Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Memorandum, 09 February 1990, Todd D. Parrish, subject: Site Visit - AWL 09 February 1990.

ENCLOSURE 11

MEMORANDUM

ENVIRONMENTAL LABS INC.
FIELD OFFICE
RECEIVED
15 FEB 1990
BY Ma
QUALITY ASSURANCE UNIT

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Todd D. Parrish

DATE: 09 February 1990

RE: Site Visit - AWL 09 February 1990

This purpose of this memorandum is to discuss the findings of my site visit of Alpine West Laboratory (AWL). The site visit was conducted according to the EPA's recommended guidelines, Environmental Labs' visit worksheet, and the Quality Assurance Practice Checklist.

LOI STATUS

LOI being used for the Phase B Field Tests of the project are in ELI's files and are identical to those being used at AWL. The listing of current LOI is as follows:

LOI 1 - 01 OCT 89 - General Laboratory Procedures
LOI 2 - 01 OCT 89 - Preparation, Handling, and Extraction of Quartz Fiber Filters
LOI 3 - 01 OCT 89 - Extraction of Soil and Fall-Out Pan Particulates
LOI 4 - 01 OCT 89 - Analysis of Bulk Explosives and Propellants
LOI 5 - 01 OCT 89 - Soxhlet Extractor Operation
LOI 6 - 01 OCT 89 - Rotary Evaporator Operation
LOI 7 - 01 OCT 89 - Supercritical Fluid Chromatography / Mass Spectrometry (SFC/MS)
LOI 8 - 01 OCT 89 - Gas Chromatography / Mass Spectrometry (GC/MS)
LOI 9 - 01 OCT 89 - Quality Control Plan: Procedures for Accuracy, Precision, and Completeness
LOI 10 - 19 JAN 90 - Determination of Moisture Content in Soil Samples

These are the LOI that are being utilized specifically for the Field Tests. These LOI are being followed as far as I can determine, with the exception that not all weights are being measured in triplicate, and samples are not analyzed in duplicate. Many aspects of the LOI have not yet been utilized at this early stage of the analysis (i.e. soil extraction, soil blanks, and EPA soil audit). The LOI are located in a 3-ring binder next to Chris Rouse's office, and are accessible for review.

FIELD/LAB SAMPLING

AWL no longer is involved in the collection of samples. During Phase B, Sandia National Laboratory delivered the filter samples and Lockheed has been responsible for delivery of soil samples. Both of these two laboratories have delivered samples under the direction of ELI.

LAB ANALYSIS

LOI located in the facility spell-out the analytical procedures. The following analytical procedures are being utilized at AWL.

SFC/MS

Electron Impact/MS - Data collected, but no quantitative analysis is performed on the EI/MS data.

Positive Ion/MS - Analyte concentrations are derived from this data for all samples.

Negative Ion/MS - Analyte concentrations are derived from this data for all samples.

GC/MS

NICI/FS/MS - These analyses are performed on all samples, with the exception of extraction blanks. This is used for nitroaromatic target analytes.

PICI/FS/MS - These analyses are performed on all samples, with the exception of extraction blanks. This is used for all other target analytes.

EI/MS - This method is used to identify non-target analytes that may be of interest to the OB/OD study. It is not used for quantitative analysis.

As mentioned previously, the filter samples are not being analyzed in duplicate. Instead, the three filters from each test were extracted separately, then 0.5 mL of filter 1 is composited with 0.5 mL of filter 2. This is analyzed. Also, 0.5 mL of filter 2 is composited with 0.5 mL of filter 3. This is analyzed. AWL will compare the results of the two analyses in order to determine precision.

INSTRUMENT CALIBRATION

The instrument is calibrated daily using perfluorotributylamine (FC-43). This is the first activity of each day. This is described very clearly in LOI 7 and 8. Calibration curves have not been derived as of yet, but will be done in the future.

PREVENTATIVE/CORRECTIVE MAINTENANCE

The mass spectrometer is tuned every six months by a Finnigan specialist who performs primary maintenance. Chris Rouse retains copies of the maintenance performed on the machine. The SFC is repaired when a component fails (i.e. the pump was just replaced). The columns are replaced every three months when analyzing filters and every two months when analyzing soils.

INTERNAL QUALITY CONTROL

Karin Markedis is specifically in charge of quality control at AWL. They introduce an internal standard at the beginning of the day with other control standards. The internal standard for NI/MS is 1-Nitronaphthalene (300 pg μL^{-1}), and the internal standard for the PI/MS is 9-Phenylanthracene (240 pg μL^{-1}). Other quality control procedures are listed in LOI 9. The filter extraction efficiency experiment has been done, but the results have not been compiled yet. Also extraction filter blanks were introduced for each field test. This equals about one extraction filter per every 4-5 samples. The filter EPA audit spike has not yet been analyzed. The soil EPA audit spike will be analyzed with the other soil samples. The freezer study has also not been performed yet. Calibration curves have not yet been performed, but will be done in the future. As mentioned before, AWL does not weigh all samples three times, a minority of the samples are only weighed once (i.e. filters after being extracted).

SAMPLE PREPARATION AND STORAGE

Sample preparation is done according to the LOI. Chris utilizes two freezers, one that contains storage samples, and one that contains working samples. Both freezers have restricted access. Temperature is monitored in both freezers, and the temperature log is updated daily.

PREPARATION AND USE OF SPIKED SAMPLES

The spiked samples are prepared by AWL and are introduced daily for analysis. This is detailed in LOI 7 and 9.

INSTRUMENT/EQUIPMENT SELECTION AND USE

This is according to the LOI.

DETERMINATION OF DETECTION LIMITS/LIMITS OF QUANTIFICATION

A detection limit is determined for each target analyte. This is determined to be three-times the background noise. These limits will be determined when the calibration curves are completed. This is included in LOI 7.

SAMPLE HANDLING AND STORAGE

This is discussed above. They also utilize an in-house tracking form which is compared to the ELI collection report. (LOI 9)

DATA REDUCTION AND ANALYSIS

This is performed by Chris Rouse, and involves an evaluation of spectrographs and other information. The computer disks that contain the data are located in a fire-proof filing cabinet that is locked.

MISCELLANEOUS

1. A logbook is maintained next to the equipment. It is utilized to record information pertaining to runs and data storage.
2. The only personnel working with study material at this time are Dr. Christine Rouse and Dr. Milton Lee.
3. Laboratory journals are maintained using standard lab notebook procedures. All entries are dated, in ink, and intact.

MEMORANDUM

ENVIRONMENTAL LABS INC
FIELD OFFICE
RECEIVED
15 FEB 1990
BY
QUALITY ASSURANCE UNIT

TO: Environmental Labs, Inc.

ATTN: Dr. Gary Booth

FROM: Floyd W. McMullin Jr.

DATE: 9 February 1990

RE: AWL audit pertaining to EPA QAD deficiencies

Prior to conducting the audit visit to AWL I completed a review of the EPA "Audit Report on Phase B of the DOD/AMCCOM OBOD Project" (Draft), AWL response to draft report dated 11 January 1990, and the EPA "Audit Report for Phase B DOD/AMCCOM OB/OD Project" (Final). Audit was conducted with Dr. Christine Rouse of AWL.

1. <u>FILTER PARTICULATE LOSS:</u> QAD observed on 19 Oct that particulate had been lost from the quartz filters used in the surface OD test.

    1) QAD suggestions included

        a. recovering the particulate from the teflon sheets and placing it back onto the filter or into a tared beaker,

        b. folding the filter differently at DPG,

        c. maintaining the folded filter in a vertical position and in a press during shipment, and

        d. placing the filter in a rack during sample conditioning such that it would open into a "V" shape.

CORRECTION: AWL did recover particulate from the teflon sheets and placed it back on filter. The method of folding the filters at DPG by SNL personnel was changed to decrease the possibility of particulate loss from the filter. This involved folding over the two side edges prior to placement back in the teflon sheets after sampling. Filters were maintained in a vertical position, and in a press during shipment after the problem was pointed out. It is noted however that the only time the particulate loss seems to be a problem is during the surface detonations.

AUDIT: All suggested improvements listed above except "d" were instituted. AWL does not anticipate instituting this suggestion in the future.

2. FREEZER THERMOMETER: QAD suggested that the thermometer in the freezer for filter and soil extracts be replaced with a unit that will register to at least -20°C (lowest

on unit was -10°C).

CORRECTION: AWL reports that the thermometer has been replaced.

AUDIT: Thermometers for both freezers, which will be utilized for OB/OD samples have thermometers which will register to -30°C. The freezer temerature is monitored daily, while a graph of the temperatures is prepared and on a monthly basis entered into a logbook maintained in the lab.

3. %RH AND ROOM TEMPERATURE: It is suggested that the %RH and temperature be recorded when the filters were weighed since the balance was located in a room that could not maintain conditions of temperature and %RH recommended for filter weighing.

CORRECTION: AWL reports that temperature and %RH were monitored using real time continuous monitors over the time of the project. AWL further reports that these recordings show that the recommended conditions were maintained.

AUDIT: Recording charts are entered into a logbook maintained in the lab. These are available for review as needed.

4. BALANCE CALIBRATION: Audit team was told that the balance had been recently calibrated using NIST traceable weights. No written record of this calibration was available. The most recent calibration according to the equipment tag was 18 Dec 87.

CORRECTION: AWL has supplied a balance calibration report showing calibrations for balances in use as September 89 (1612) and August 89 (2434). The report states that the class S weights were calibrated August 11, 1975. It also states they are obtaining a new set of class S weights (100g to 1mg). The new weights will have annual calibration checks through the manufacturer.

AUDIT: For future use the lab needs to utilized currently calibrated class S weights since the last calibration of the weights in 1975, their accuracy is questionable.

5. FILTER WEIGHING: It was noted that QC filter weighings had all been done on the same day, and it was uncertain how many weighings each filter had. It was noted that AWL supplied filter data list and stated each QC filter was reweighed 4 days after initial weighing but data list showed they were reweighed 2 days after initial weighing.

CORRECTION: AWL reported that filters were weighed in a filter holder utilizing a mechanical balance. Each filter had 3 repetitive weights done during the initial weighing. Problems with drifting balance weight were noted during the weighing and found that folding the filters would make more accurate weights. AWL also reports that a set of 5 QC filters were weighed repetitively during a 30 day period (14 Oct - 14 Nov)

AUDIT: They determined that actual reweighing was 2 days after the first. Determined that all filters were weighed folded due to the drifting balance weight problem.

6. FILTER WEIGHING LOG BOOK: It was observed that the logbook on filter weights was not clear as to date, analyst, %RH and temperature. It was recommended that documentation for filter weighings be improved.

CORRECTION: AWL states will alter recording procedure in the logbook. They plan to record filters by sample in the future.

AUDIT: Although AWL states they will set up logbooks by sample in the future for ease in reading, both they and myself feel this is somewhat of a departure from GLP which calls for logbooks to be chronologically organized to provide continuous documentation as to what has been done. We recommend contact with EPA QAD personnel for their input as to methodologies for recording to allow easier auditing, but at the same time maintain GLPs.

7. CHANGING BALANCES DURING WEIGHING: It was determined that AWL had switched balances during the weighing of the filters. QAD stated it looked as if the second balance differed by approximately 10mg from the first. QAD recommended that all exposed filter weights should be increased by a minimum of 10mg when the results are tabulated.

CORRECTION: AWL performed a comparison for the weights obtained on the QC filters on the mechanical balance (16 Oct - 26 Oct) and the weights obtained on the electronic balance (8 Nov - 14 Nov) with acceptable variation in the weights noted. Additionally, they have performed a balance correlation and found that they both are accurate to a tenth of a mg (in balance report, done at a single weight of 5g).

AUDIT: They recommend that if multiple balances are utilized in the future that full range correlations be done to determine if variance is a constant value or alters as a percentage of the weight.

8. FILTER PARTICULATE LOSS DURING TRANSPORT: Auditors state that AWL reports that particulate lost from filters during transport had been shaken back onto the filter. AWL reports the material was placed back onto the filter at arrival as the filters were laid out for conditioning within an estimated 10 minutes of arrival. AWL stated that when filter had lost particulate during transport, the lab noted this on the data sheet that

accompanied the filter. QAD states that inspection of the data sheets showed that only the earlier data sheets noted particulate loss.

<u>CORRECTION:</u> See number one item corrections. This removed the problem after 19 October 1989. Data sheets on file at AWL note which samples were effected by this problem.

<u>AUDIT:</u> They checked data sheets and verified that particulate loss was noted (upper right corner of form).

9. <u>FAN IN THE BALANCE ROOM:</u> QAD personnel suggested that a small fan be operated in the balance room to assure uniform temperature and humidity throughout the room and to thus hasten and maintain equilibrium conditions.

<u>CORRECTION:</u> AWL did not implement this suggestion. In their response to the EPA Dr. Lee states, "The suggestion to install a small fan in the balance room was not followed because this would influence the temperature and humidity in the room, make the balance unstable, and provide a risk for blowing particles off the conditioning filters. The filters were conditioned in a flat position to minimize the risk of moving particles."

<u>AUDIT:</u> No action nor followup on this item.

In summary I would say that it appears that AWL has instituted the changes recommended by the EPA QAD personnel with the exceptions of placing the filter in a rack during sample conditioning (Item #1), and installation of a fan in the balance room (Item #9).

Floyd W. McMullin Jr.
Quality Assurance Officer

Letter MD-77B, February 13, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, w/enclosed benzene audit report.

ENCLOSURE 13

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

February 13, 1990

Dr. R. A. Rasmussen
Oregon Graduate Institute of
Science and Technology
19600 N.W. Von Neumann Dr.
Beaverton, Oregon 97006-1999

Dear Rai:

Six canisters were sent to you in November 1989 as audit samples for the OB/OD Project. These canisters contained selected volatile organic compounds at low parts per billion concentrations. All of the samples were humidified and two of the canisters were blank samples containing only zero air.

Table 1 lists the results of your analyses of the samples for benzene.

Your results compare very well with our values for the audit canisters. The only other volatile organics supposed to be in the canisters were carbon tetrachloride, chloroform, tetrachloroethylene and vinyl chloride at concentrations from 1 ppb-5 ppb. Any other compounds that may be present as impurities should be at extremely low or trace concentrations.

Please let me know if you have any questions regarding the results.

Sincerely yours,

Howard Crist

Howard Crist
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Enclosure

cc: Don Johnson
W. Mitchell
K. Caviston
Gary Booth

Table 1 Results of Analyses of OB/OD Audit Samples for Benzene

	Benzene, ppbv				
Sample (S/N)	Spiked	Reported		Difference, %	
041 (blank)	0	< 0.2	< 0.2	-	-
042	0.9	0.9	0.8	0	-11
043 (blank)	0	< 0.2	< 0.2	-	-
044	1.0	1.2	1.1	20	10
045	2.5	2.9	2.6	16	4.0
046	3.0	3.4	3.2	13	6.7

Memorandum, 22 February 1990, Todd Parrish, subject: Addendum to the site visit to AWL, 09 February 1990 [sic].

ENCLOSURE 14

ENVIRONMENTAL LABS, INC.
MEMORANDUM

TO: Dr. Gary Booth

FROM: Todd Parrish

DATE: 22 February 1990

RE: Addendum to the site visit to AWL - 09 February 1990

On 22 February 1990, ELI was made aware of a mistake in its 09 February 1990 audit of AWL. The problem is located in the last paragraph of the Lab Analysis section. The paragraph should be as follows:

As mentioned previously, the filter samples are not being analyzed in duplicate. Instead, the three filters from each test were extracted separately, then 0.5 mL of each extract is stored separately. 0.25 mL of filter A extract is composited with 0.25 mL of filter B extract. This composite is now sample 1. 0.25 mL of filter B extract is composited with 0.25 mL filter C extract. This composite is now sample 2. Each of these samples is analyzed. AWL will compare the results of the two analyses in order to determine precision.

The origin of the mistake is two-fold. The first being that when the procedure was being explained to the auditor, there was no clear mention of any precise measurements. The procedure was described as "taking half of the sample and compositing it with half of another." There was no mention of 0.5 mL being stored or 0.25 being composited. The second origin can be related to the failure of the auditor to probe into the matter in more depth. The procedure should have been looked into greater depth and the laboratory personnel questioned to a greater extent.

Todd Parrish
Quality Assurance

ENVIRONMENTAL LABS INC.
FIELD OFFICE
RECEIVED
22 FEB 1990
BY TP
QUALITY ASSURANCE UNIT

Letter MD-77B, March 13, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

ENCLOSURE 15

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

March 13, 1990

Dr. R. A. Rasmussen
Oregon Graduate Institute
of Science and Technology
19600 NW Von Neumann Dr.
Beaverton, OR 97006-1999

Dear Rai:

The concentrations of the chlorinated hydrocarbons in the OB/OD canisters were not analyzed accurately since benzene was the only target compound in the audit gas. When these mixtures were prepared we did not know that you would be interested in analyzing the other VOCs in the audit gas.

Sincerely,

Howard

Howard Crist
Research and Monitoring
Evaluation Branch
Quality Assurance Division (MD-77B)

Memorandum, Rhodes, 20 April 1990, U.S. Environmental Protection Agency, Atmospheric Research and exposure assessment Laboratory, Research Triangle Park, North Carolina.

ENCLOSURE 16

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

DATE: April 20, 1990

SUBJECT: Report of Trip to Salt Lake City and Provo, Utah to Review and Discuss Items Concerning the OB/OD Program, Feb. 20-23, 1990

R C Rhodes

FROM: R. C. Rhodes, Quality Assurance Specialist

TO: W. J. Mitchell, Chief, Research Monitoring and Evaluation Branch

Ellen Streib, Linda Porter, Lisa Smith, and myself visited Dugway Proving Ground (DPG), Alpine West Laboratory (AWL), and Environmental Laboratories, Incorporated (ELI) to review and discuss items concerning the OB/OD Program on February 20-23, 1990. We arrived at Salt Lake City around noon, Tuesday February 20. A brief schedule of events follows:

Afternoon, Feb. 20

Linda and Ellen visited Dugway Proving Ground to review the handling and storage of soil samples by Lockheed personnel.

Lisa and myself visited Alpine West to review their analytical procedures and their filter handling procedures. Near the end of the visit Lisa began feeling sick as if coming down with the flu. After returning to the hotel in Salt Lake City, she remained in her room the next day, and then returned to North Carolina on Thursday with Linda and Ellen.

Wednesday February 21

Linda, Ellen, and I visited AWL and ELI. We met Monte Law, of Lockheed there to review the procedures and equipment used for grinding, mixing, and splitting the soil samples. This equipment was located in the Agronomy Department of BYU in the same building as Dr. Gary Booth of ELI. Later in the morning we reviewed some of the records at ELI. In the afternoon, from 1:00 to 3:00, we reviewed the PIXE procedures and equipment in a nearby building. Dr. Nolan Mangleson reviewed the principles of the PIXE and explained the operation of the equipment. From 3:00 to 5:00 I sat in on a meeting between Don Johnson and Gary Booth concerning the "final" ELI report on the Bang Box phase of the testing. Also present were Duane Long of Andrulis Research, and Gary's personnel, Floyd McMullin and Todd Parrish.
Duane had rewritten the ELI report, revising it considerably

based on my previous comments on the second draft of the ELI report and on Duane's critique as well. A copy of my original comments are enclosed. Linda and Ellen, at 3:00 pm returned to Salt Lake City to check on Lisa.

Thursday, February 22

I reviewed the latest ELI report on the Bang Box Phase of testing, prepared by Duane, and made further recommendations for improvement.

Additional Detailed Notes Follow:

Confusion on the Duplicate Analyses of Filters by AWL. The unusual and complicated method used by AWL in preparing two duplicate analyses from three separate filter extracts was explained by Dr. Christine Rouse and Dr. Milton Lee. Since all three filters sampled the same air in the cloud (albeit the three samplers ran at slightly different flow rates), they were in effect triplicate samples. To prepare the "duplicates" (with the objective of reducing the number of analyses), equal portions of the extracts from filters A and B were combined to form one sample for analysis, and equal portions of the extracts from filters B and C were combined to form the "duplicate" sample for analysis.

Confusion of the Supplier of Filters Analyzed by Dr. Mangleson on the PIXE. Dr. Mangleson had analyzed a number of blank filters for trace elements and had found them to be "quite dirty". In particular, the iron, copper, barium, and phosphorous seemed to be contaminants even though the filters had been fired by Dr. Christine Rouse to remove impurities. It was determined that the filters were procured by Wayne Einfeld and were from Pallflex since the filter had no filter numbers stamped on the filters. Whatman filters supplied by EPA/RTP, with filter numbers such as Q714369-Q7143477, were used in the Bang Box test and Phase A tests. According to Floyd McMullin's notebook the above numbered filters were used on the "big gulp" test at the Bang Box, February 15, 1990. Pallflex filters were used in Phase B tests. After returning to RTP I sent to Dr. Mangleson copies of the trace metal analyses of past Whatman quartz- and glass-fiber filters.

Loss of Canister Sample in June 1989 Phase B Tests. In June no canister sample was obtained due to loss of vacuum. The plumbing was to be changed to include a vacuum gage to confirm integrity of vacuum before sampling and to determine changes in vacuum during sampling. The gage was to be received Oct. 17 at Provo? by Wayne Einfeld during the test series. Floyd McMullin stated that the equipment ws received and he personally observed it when he was on the plane on Oct. 19.

Grinding, Weighing, Mixing, and Splitting. Grinding of the soil samples is done until all of the material passes a USA Standard Testing #4 sieve. Prior to grinding, the samples at DPG were removed from the freezer to thaw for 12 hours. The samples would be at BYU for grinding, weighing, mixing, and splitting between 8 am and 4 pm, and then returned to the DPG freezer. The Mettler balance at BYU used to weigh the samples was last calibrated on 10 Oct. '89, and to be next calibrated on 12 Apr. '90 according to the calibration sticker. At each daily use the balance is checked with 500 gram and 1000 gram weights, the balance having a maximum capacity of 1600 grams. Each sample bottle holds approximately 500 grams. The mixing time, at slow speed, was two minutes.

Completeness and Organization of Records at ELI. I had previously had a copy of Volumes I and II of the QA Handbook for Air Pollution Measurement Systems mailed to Dr. Gary Booth for his use on OB/OD. In particular I desired that he have the filter handling procedures in the section for the TSP sampler in Volume II. In reviewing the copy of Volume II that he had received, the section on TSP was missing. On Tuesday evening I borrowed a copy of Volume II from Rolf Roebbling of the Utah Air Monitoring Office to take to ELI on Wednesday.

Much of the records at ELI are kept on the computer. Copies of the directories of the OB/OD files are attached. The main directories are

TEXTFILE/OBOD
TEXTFILE/OBOD/ARC (Andrulis Research)
TEXTFILE/OBOD/AWL
TEXTFILE/OBOD/BCD
TEXTFILE/OBOD/BUDGETS
TEXTFILE/OBOD/ELIPLAN
TEXTFILE/OBOD/LBL
TEXTFILE/OBOD/MEMOS
TEXTFILE/OBOD/MISC
TEXTFILE/OBOD/OGC
TEXTFILE/OBOD/PIXE
TEXTFILE/OBOD/QAPP
TEXTFILE/OBOD/REPORTS
TEXTFILE/OBOD/SNL
TEXTFILE/OBOD/SSL

Hard copy of the files are maintained in an orderly fashion in metal file cabinets.

We requested a listing of the various visits and trips made by the ELI personnel for the OB/OD project. Such a list was prepared as is included herewith.

Also included herewith are copies of ELI site visits to

AWL on 9 Feb. 1990 by Todd Parrish
PIXE lab on 14 Feb. 1990 by Todd Parrish and Floyd McMullin

Temperature/Humidity Records at AWL for the Filter Conditioning and Weighing Room. When visiting AWL on Tuesday, Feb. 20, we requested to see and did observe all of the temperature/humidity hard copy original charts that were used during the conditioning and weighing of the filters. The records showed that the temperature was maintained within plus or minus 5 degrees F. and the humidity, within plus or minus 5 percent relative humidity.

```
02/21/90  10:28              Directory C:\TEXTFILE\OBOD\*.*
Free Mem:   472144    Disk Space Free: 24315904, Used:          0     Files:  16

. <CURRENT>      <DIR>                             3 .. <PARENT>      <DIR>
ARC        .     <DIR>    02/21/90 09:58  3 AWL        .     <DIR>    02/21/90 08:50
BCD        .     <DIR>    02/21/90 08:51  3 BUDGETS .     <DIR>    02/21/90 09:24
ELIPLAN .     <DIR>    02/21/90 08:51  3 LBL        .     <DIR>    02/21/90 08:51
MEMOS    .     <DIR>    02/21/90 08:52  3 MISC      .     <DIR>    02/21/90 08:51
OGC        .     <DIR>    02/21/90 08:52  3 PIXE      .     <DIR>    02/21/90 08:52
QAPP      .     <DIR>    02/21/90 10:08  3 REPORTS .     <DIR>    02/21/90 08:52
SNL        .     <DIR>    02/21/90 08:53  3 SSL        .     <DIR>    02/21/90 08:53




1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:29              Directory C:\TEXTFILE\OBOD\ARC\*.*
Free Mem:   472144    Disk Space Free: 24315904, Used:     20428     Files:  13

. <CURRENT>     <DIR>                             3 .. <PARENT>     <DIR>
ANDRULIS.          1057  03/22/89 10:25  3 AWL-DATA.LTR        800  07/31/89 12:10
BUDGET   .KEN       672  12/09/88 15:19  3 C-ECKARD.LTR       1025  08/04/89 12:35
D-B08-04.L89       1026  08/04/89 12:38  3 GEB-EXP .LTR       3049  12/01/89 12:17
KHZ04-12.L89        898  04/12/89 09:01  3 KHZ10-07.L88       3290  10/07/88 14:55
KHZ11-04.L88       3554  11/11/88 15:44  3 KHZ12-09.L88       2189  12/09/88 15:27
MLL-EXP .LTR       2868  12/01/89 12:03




1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:29              Directory C:\TEXTFILE\OBOD\AWL\*.*
Free Mem:    472144      Disk Space Free: 24315904, Used:     29480     Files:   7

. <CURRENT>     <DIR>                        3  .. <PARENT>     <DIR>
ALPINE   .LEE      1365  03/15/89 15:43  3  AWL07-17.M89       5397  07/19/89 12:46
AWLAUDIT.DOC       7124  02/08/90 12:36  3  AWLREV   .M89      3028  07/17/89 12:27
EPA02-09.M90      12566  02/20/90 11:42









1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:30              Directory C:\TEXTFILE\OBOD\BCD\*.*
Free Mem:    472144      Disk Space Free: 24315904, Used:     74461     Files:   8

. <CURRENT>     <DIR>                        3  .. <PARENT>     <DIR>
BCDFINAL.MEM       6371  07/13/89 14:34  3  BCDPART1.M89       6371  07/13/89 14:33
BCDPART2.M89       2820  07/13/89 15:03  3  OBDATA   .TAB     37894  05/06/89 22:30
OBOD1C   .LOI      6105  08/09/89 09:48  3  OBOD2C   .LOI     14900  08/09/89 09:53









1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:30            Directory C:\TEXTFILE\OBOD\BUDGETS\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:     27995     Files:  8

. <CURRENT>     <DIR>                       3  .. <PARENT>     <DIR>
BUDGET   .          4795  08/12/88 15:43  3  BUDGET-O.BOD       5716  01/06/89 10:58
BUDGTSAN.DIA        4795  08/12/88 15:43  3  OBOD     .         7855  05/08/89 19:18
QABUDGT .BB         1493  04/11/89 12:06  3  QABUDGT .BOX       3341  04/11/89 12:20
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:31            Directory C:\TEXTFILE\OBOD\ELIPLAN\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:    244079     Files.  7

. <CURRENT>     <DIR>                       3  .. <PARENT>     <DIR>
MASTERP .LAN       36769  11/10/89 14:57  3  OUTLINE .PLN      46176  11/10/89 14:58
QA-PLAN .BOX       46026  07/05/89 09:03  3  QAPLANFI.ELD      85688  01/13/90 10:55
TECHNIC .PLN       29420  04/11/89 13:34
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:31            Directory C:\TEXTFILE\OBOD\LBL\*.*
Free Mem:   472144     Disk Space Free: 24315904, Used:      9953     Files:   3

. <CURRENT>     <DIR>                     3 .. <PARENT>     <DIR>
LBL01-31.M89      9953  02/01/89 23:08
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:32            Directory C:\TEXTFILE\OBOD\MEMOS\*.*
Free Mem:   472144     Disk Space Free: 24315904, Used:     21027     Files:   6

. <CURRENT>     <DIR>                     3 .. <PARENT>     <DIR>
EPA      .        1244  09/16/89 18:46    3 PHASE-A .M90     4302  02/09/90 14:17
REPORT   .        2057  09/15/89 15:15    3 SCHEDULE.M90    13424  02/21/90 08:42
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:32          Directory C:\TEXTFILE\OBOD\MISC\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:    579850     Files:  26

. <CURRENT>      <DIR>                          3 .. <PARENT>     <DIR>
ADDRESS .LST      2029  02/20/90 14:18  3 ADRESS  .QA      2570  12/29/88 12:08
CUSTODY .DOC      1673  09/30/89 14:16  3 DEB01-18.L89     2209  01/18/89 17:07
HSB11-21.L88       922  11/19/88 14:28  3 J-K10-07.L88     2207  10/07/88 11:25
LOI     .         1460  11/09/88 15:27  3 MBJ01-18.L89     1597  01/18/89 14:22
MBJ01-25.L89      1562  01/25/89 16:48  3 MBJ02-28.L89     1049  02/28/89 15:11
MBJ09-19.L88       704  09/19/88 14:49  3 MERGE   .        1977  11/11/88 15:50
OBOD-QA .LTR      2002  02/20/90 14:31  3 PERSONAL.QA     23283  09/28/89 11:03
PHASEB  .TBL    501358  02/14/90 10:16  3 QA      .        4620  08/13/88 12:29
QAL12-21.L88      9188  12/29/88 12:08  3 R-RHODES.LTR     1103  10/25/88 13:50
SAMPLE  .INS      4940  10/13/89 11:16  3 SHIPMENT.INS     2609  10/03/89 12:14
SITEFORM.DOC      4612  12/20/88 21:52  3 SWITCHIN.M88      989  11/22/88 10:23
T-PARISH.LTR      1313  12/22/88 11:27  3 TABLE   .        3874  11/10/89 12:55




1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:33          Directory C:\TEXTFILE\OBOD\OGC\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:     24607     Files:   5

. <CURRENT>      <DIR>                          3 .. <PARENT>     <DIR>
OGC01-12.M90      3801  01/12/90 14:19  3 OGC01-18.M90     7806  01/20/90 12:54
OGC01-24.M89     13000  01/20/90 11:09




1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:33              Directory C:\TEXTFILE\OBOD\PIXE\*.*
Free Mem:   472144     Disk Space Free: 24315904, Used:      7978     Files:   3

. <CURRENT>    <DIR>                       3  .. <PARENT>    <DIR>
PIX02-14.M90     7978  02/20/90 12:50










1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:34              Directory C:\TEXTFILE\OBOD\QAPP\*.*
Free Mem:   472144     Disk Space Free: 24315904, Used:    211713     Files:   6

. <CURRENT>    <DIR>                       3  .. <PARENT>    <DIR>
MASTERP .LAN    36769  11/10/89 14:45  3  OUTLINE .PLN    46981  11/10/89 12:24
PRELIMIN.PLN    41270  04/07/89 15:13  3  QAPLANFI.ELD    86693  11/02/89 15:44










1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6    F3 for Help)
```

```
02/21/90  10:35             Directory C:\TEXTFILE\OBOD\REPORTS\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:    515273     Files:  19

. <CURRENT>     <DIR>                          3  .. <PARENT>     <DIR>
BBFINAL .RPT    96090  02/15/90 09:33  3  CONTENTS.        4152  02/02/90 11:42
FINAL   .       89681  02/02/90 11:41  3  FINISH  .       51254  01/29/90 16:15
INT05-89.MEM     5537  07/03/89 11:29  3  MONTHLY .APR     3834  08/21/89 14:36
MONTHLY .DEC     5978  08/21/89 14:27  3  MONTHLY .FEB     6401  08/21/89 14:25
MONTHLY .JAN     5741  08/21/89 14:32  3  MONTHLY .MAR     3947  08/21/89 14:34
OBJECTIV.       31469  01/27/90 11:10  3  OBOD-B  .RPT    88493  02/09/90 10:23
PRECISIO.        9525  01/27/90 12:22  3  QA-FINAL.TWO    30005  09/30/89 14:47
QARPRTBB.D89     5376  05/16/89 16:05  3  REV     .       32208  01/27/90 15:20
TABLES  .       45582  01/31/90 15:52
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:35             Directory C:\TEXTFILE\OBOD\SNL\*.*
Free Mem:    472144     Disk Space Free: 24315904, Used:     35731     Files:   8

. <CURRENT>     <DIR>                          3  .. <PARENT>     <DIR>
SNL01-03.M89     2386  12/20/88 22:23  3  SNL02-21.LTR     2726  02/20/90 13:41
SNL08-03.M89     4050  08/04/89 13:08  3  SNL10-19.M89    12445  01/22/90 13:41
SNL12-16.M88    12049  01/11/89 16:04  3  W-EINFLD.LTR     2075  03/15/89 16:17
```

```
1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

```
02/21/90  10:35            Directory C:\TEXTFILE\OBOD\SSL\*.*
Free Mem:   472144    Disk Space Free: 24315904, Used:    19316     Files:   6

. <CURRENT>     <DIR>                  3  .. <PARENT>     <DIR>
R-CARY  .LTR      1174  01/09/89 12:00  3  SSL01-17.M90     7436  01/20/90 11:43
SSL01-23.M89      7256  01/24/89 19:37  3  SSL05-09.M89     3450  05/12/89 08:11







1 *Mark; 2 Delete; 3 Move/Rename; 4 Select Files; 5 Lock; 6 Look; (F7 to Exit,
7 Other Dir; 8 Copy; 9 Word Srch; F2 Name Srch; F5 Find Files: 6   F3 for Help)
```

ENVIRONMENTAL LABS, INC.
MEMORANDUM

TO: Ms. Lisa Smith
EPA, QAD

FROM: Floyd W. McMullin Jr.
Environmental Labs

DATE: 21 February 1990

RE: ELI Personnel schedules during OB/OD

DATE	LOCATION	PERSON	PURPOSE
21 Nov 88	Dugway Proving Gnd, UT	Todd Parrish	Soil Samples (EPA)
30 Nov 88	Sandia National Labs	Gary Booth Todd Parrish Floyd McMullin	Bang Box Tests
1 Dec 88	Sandia National Labs	Gary Booth Todd Parrish Floyd McMullin	Bang Box Tests
2 Dec 88	Sandia National Labs	Gary Booth Todd Parrish Floyd McMullin	Bang Box Tests
3 Dec 88	Sandia National Labs	Gary Booth Todd Parrish Floyd McMullin	Bang Box Tests
5 Dec 88	Sandia National Labs	Todd Parrish Floyd McMullin	Bang Box Tests
6 Dec 88	Sandia National Labs	Todd Parrish Floyd McMullin	Bang Box Tests
7 Dec 88	Sandia National Labs	Todd Parrish Floyd McMullin	Bang Box Tests
16 Dec 88	Sandia National Labs	Floyd McMullin	Site Audit-Bang Box
28 Dec 88	BCD, OH	Todd Parrish	Site Audit-Bang Box
29 Dec 88	BCD, OH	Todd Parrish	Site Audit-Bang Box
5 Jan 89	Andrulis Research, SLC	Gary Booth	Meeting
23 Jan 89	Sunset Lab, OR	Floyd McMullin	Site Audit-Bang Box
24 Jan 89	Oreg. Grad. Center	Floyd McMullin	Site Audit-Bang Box
25 Jan 89	AWL, UT	Todd Parrish	Site Audit-Bang Box

30 Jan 89	Sandia National Labs	Gary Booth Todd Parrish	Bang Box Tests
31 Jan 89	LBL, CA	Floyd McMullin	Site Audit-Bang Box
1 Feb 89	Sandia National Labs	Todd Parrish	Bang Box Tests
2 Feb 89	Sandia National Labs	Todd Parrish	Bang Box Tests
3 Feb 89	Sandia National Labs	Todd Parrish	Bang Box Tests
6 Feb 89	Sandia National Labs	Gary Booth Floyd McMullin	Bang Box Tests
7 Feb 89	Sandia National Labs	Gary Booth Floyd McMullin	Bang Box Tests
8 Feb 89	Sandia National Labs	Floyd McMullin	Bang Box Tests
9 Feb 89	Sandia National Labs	Floyd McMullin	Bang Box Tests
15 Feb 89	Sandia National Labs	Floyd McMullin	Bang Box Tests
16 Feb 89	Sandia National Labs	Floyd McMullin	Bang Box Tests
20 Feb 89	Andrulis Research, SLC	Floyd McMullin	Meeting
27 Mar 89	Salt Lake City	Gary Booth	Meeting
9 May 89	Sunset Lab, OR	Floyd McMullin	Site Audit-Bang Box
15 May 89	Andrulis Research, SLC	Gary Booth	Meeting
16 May 89	Andrulis Research, SLC	Gary Booth	Meeting
17 May 89	Raleigh, NC	Gary Booth	Meeting
18 May 89	EPA, Raleigh, NC	Gary Booth	Meeting
19 May 89	EPA, Raleigh, NC	Gary Booth	Meeting
30 Jun 89	Andrulis Research, SLC	Gary Booth	Meeting
6 Jul 89	Andrulis Research, SLC	Gary Booth	Meeting
17 Jul 89	AWL, UT	Floyd McMullin	Site Audit-Bang Box
18 Sep 89	Dugway Proving Gnd, UT	Gary Booth	Meeting
20 Sep 89	Andrulis Research, SLC	Gary Booth	Meeting
21 Sep 89	Andrulis Research, SLC	Gary Booth	Meeting
28 Sep 89	Andrulis Research, SLC	Gary Booth	Meeting
4 Oct 89	Dugway Proving Gnd, UT	Gary Booth	Phase B Samples
5 Oct 89	Dugway Proving Gnd, UT	Gary Booth	Phase B Samples
10 Oct 89	Andrulis Research, SLC	Gary Booth	Meeting
13 Oct 89	SNL FWAC, Provo, UT	Floyd McMullin	EPA System Audit
14 Oct 89	SNL FWAC, Provo, UT	Todd Parrish	EPA System Audit
16 Oct 89	Dugway Proving Gnd, UT	Gary Booth Floyd McMullin	Phase B
16 Oct 89	Andrulis Research, SLC	Gary Booth	Meeting
17 Oct 89	Dugway Proving Gnd, UT	Gary Booth Floyd McMullin	Phase B
18 Oct 89	SNL at DPG, UT	Floyd McMullin	Site Audit-Phase B
18 Oct 89	Dugway Proving Gnd, UT	Gary Booth Floyd McMullin	Phase B

19 Oct 89	Dugway Proving Gnd, UT	Gary Booth Todd Parrish Floyd McMullin	Phase B Phase B
23 Oct 89	Dugway Proving Gnd, UT	Gary Booth Floyd McMullin	Phase B
24 Oct 89	AWL	Gary Booth	Phase B
25 Oct 89	Dugway Proving Gnd, UT	Gary Booth	Phase B
26 Oct 89	Dugway Proving Gnd, UT	Gary Booth Todd Parrish Floyd McMullin	Phase B
27 Oct 89	Andrulis Research, SLC	Gary Booth	Meeting
30 Oct 89	Dugway Proving Gnd, UT	Gary Booth	Phase B
31 Oct 89	Dugway Proving Gnd, UT	Gary Booth	Phase B
6 Nov 89	Andrulis Research, SLC	Gary Booth	Phase B
7 Nov 89	Andrulis Research, SLC	Gary Booth	Phase B
8 Nov 89	Andrulis Research, SLC	Gary Booth	Phase B
9 Nov 89	Andrulis Research, SLC	Gary Booth	Phase B
10 Nov 89	Andrulis Research, SLC	Gary Booth	Phase B
13 Nov 89	Aberdeen, MD	Gary Booth	Phase A & B
14 Nov 89	Aberdeen, MD	Gary Booth	Phase A & B
4 Dec 89	Andrulis Research, SLC	Gary Booth	Meeting
12 Jan 90	Andrulis Research, SLC	Gary Booth	Meeting
17 Jan 90	Sunset Lab, OR	Floyd McMullin	Site Audit-Phase B
18 Jan 90	Oreg. Grad. Center	Floyd McMullin	Site Audit-Phase B
19 Jan 90	Sunset Lab, OR	Floyd McMullin	Site Audit-Phase B
9 Feb 90	AWL, UT	Todd Parrish	Site Audit-Phase B
9 Feb 90	AWL, UT	Floyd McMullin	Audit-EPA items
14 Feb 90	PIXE, UT	Todd Parrish Floyd McMullin	Site Audit-Phase B

This listing does not contain the schedule for ELI personnel on site at Dugway Proving Ground during the Phase A testing which occurred during June 1989. The ELI employee who worked on that portion was not available. Consequently I was unable to obtain his log book at this time. If needed I will be able to furnish this information at a later time.

Letter MD-77B, September 7, 1990, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina.

ENCLOSURE 17

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

September 7, 1990

Mr. MacDonald Johnson
c/o Commander
HQ-AMCCOM
AMSMC-DSM-D
Rock Island, IL 61299

Dear Don,

This is an interim summary of the audit results from the Quality Assurance Division's (QAD's) performance and systems audits of the OB/OD Phase C field tests. The audits covered the test period August 6-27, 1990 and included audits of the aircraft, field tests, and laboratory. The audit team consisted of W. Mitchell, W. Barnard, J. Bowen, A. Hines, E. Hunike, and L. Porter. A final report will be issued after the audits of Alpine West Laboratory and Oregon Graduate Center are completed.

Because the OB/OD project is large and uses the services of a number of different contractors, the audit results will be divided according to contractor activities and addressed as follows:

1. Lockheed Environmental Services (LESC)
2. Sandia National Laboratories (SNL)
3. Alpine West Laboratory (AWL)
4. Environmental Laboratories Inc. (ELI)

LOCKHEED ENVIRONMENTAL SERVICES (LESC)

LESC personnel are responsible for all the ground support services needed by the Phase C test program with the exception of the set up/placement and ignition of the munitions and propellant. LESC personnel under the supervision of Jim Stephens and Monty Law (1) set out the test grid; (2) put the fallout and sputter pans and trays in the position required by the test design; (3) recover, weigh, and composite the soil and ash samples, and (4) store the samples until they are transported to AWL.

During QAD's audit of Phase C, most of the activities listed above were observed. As in the previous phases of the OB/OD project, LESC is doing an excellent job. The field staff appear to be conscientious and knowledgeable. They are careful to minimize sample loss and to avoid sample contamination in a situation that does not lend itself to easy, routine sample collection. One example of LESC's ingenuity and care in sample collection was the use of tray covers as wind breakers to help prevent sample loss during the transfer from collection tray to labeled sample bottles.

Since LESC uses students for most of their collection activities, employee turn-over could cause potential problems because of the constant influx of new, untrained people. However, LESC has done a commendable job of minimizing the potential problem with excellent employee training and by assigning inexperienced employees to work with experienced employees.

Procedures for weighing and compositing the soil, pan, and tray samples were observed. LESC staff performing the procedures appeared to be conscientious and knowledgeable. Gloves were worn by all but one of the personnel handling the collected field samples. This was immediately corrected when it was brought to Monty's attention. The staff appear to be aware of the importance of maintaining the integrity of the sample and will quickly correct anyone who might do something that could affect this integrity.

The compositing procedure itself has some unavoidable problem areas which were discussed with Monty Law. These were:

1. The loss of some of the extremely fine sample dust during sample compositing. This is an innate problem when transferring dry solids during the compositing of samples. It was recommended to Monty that he try to minimize this by keeping the number of transfers to a minimum (e.g., transferring ash pan samples directly to a clean weighed bottle rather than to one of the field sample bottles and then to the clean weighed bottle). The overall controlling factor is the care taken by the individual staff members to minimize this sample loss during sample transfer.

2. The loss of weight by field sample bottles from sitting in the heat at the test site. Field sample bottles containing very small amounts of sample often give a negative sample weight. This appears to be due to weight loss in the sample bottle during the test. Possible ways to eliminate this problem are:

a. Condition bottles in a controlled environment before doing the pre- and post-test weights.

b. Label bottles and allow them to condition in a field-type environment before doing the pre-test weight.

c. Use field blank bottles to determine weight loss. If this is a constant value, it could be applied as a correction factor.

Currently, LESC is removing the sample from field bottles showing a negative weight and weighing the sample separately. This does solve the weight loss problem for those samples having a negative weight, but does not deal with those samples having a very low but positive weight.

3. The room used to composite samples contains a lot of airborne dust during the clean-up of the compositing equipment (sifters, grinders, mixers, etc.). This type clean-up should never be done while samples are open and exposed to the air and LESC personnel should wear simple disposable filter masks during this procedure to minimize inhaled dust. It should be noted that during the audit of LESC all sample bottles were covered when the compositing equipment was being cleaned and the comment is made only as a reminder.

SANDIA NATIONAL LABORATORIES (SNL)

SNL samples the OB and OD plumes using a Twin Otter aircraft under the direction of Mr. Wayne Einfeld. The aircraft is stored overnight at the Provo Municipal Airport, but between detonations/burns it lands at Michael Army Airfield, Dugway, where the samples are recovered. The aircraft collects: particle samples using Teflon coated filters mounted in high volume sampler housings; VOC's in stainless steel (SS) canisters and air samples in an 80 L Teflon bag. The Teflon bag's contents are analyzed for CO, CO_2, O_3 and NO/NO_2 using ambient air monitors installed in the aircraft. The aircraft is also equipped with sensors for temperature, dewpoint, altitude, airspeed and real time measurement of aerosol particle size and concentration.

System and performance audits were performed on the aircraft throughout the week of August 6, 1990. Usually, these audits are performed at the beginning of the testing phase to ensure the equipment is functioning properly. However, this time the auditors found numerous equipment failures which resulted in SNL having to repair equipment and/or get it operating properly during the time field tests were occurring. Although in field testing such problems inevitably occur, the type and number of problems encountered by SNL in each phase of OB/OD raises serious questions about SNL's capability to adequately support the OB/OD project. It is recommended that OB/OD review the manner in which SNL is supporting the project. SNL appears to be operating in an independent manner in accordance to their own timetable. The basis for these observations is delineated below.

The OB/OD activities performed by SNL fall into two categories, aircraft operations and filter weighing.

1. Aircraft Operations

A. Audits (8/6-10/90)

Numerous problems were found during QAD's system and performance audits of the aircraft's sampling equipment. As stated above, these problems were more numerous then those generally encountered in field testing. QAD audited the plane August 6-10, 1990. A brief summary of the audit results follows.

1. The CO analyzer was audited on the evening of 8/6/90 following that day's tests. It was found to be operating correctly.

2. The CO_2 analyzer was audited on 8/7/90 and was found to be operating correctly.

3. The transport tube flow was audited on 8/10/90 and found to be within acceptable agreement with the expected flow. The flow was within +/- 10% of the expected value.

4. The ozone analyzer was down 8/6/90 through 8/8/90. SNL found that a misaligned pin connector on one of the pc boards was causing the problem. SNL repaired the ozone analyzer the evening of 8/8/90 and calibrated it the morning of 8/9/90 before that day's tests began. QAD audited the ozone monitor on 8/10/90. The results of this audit were in good agreement with EPA's audit values. To audit the whole data collection system, SNL ozone values were taken from the computer output.

5. The NOX analyzer was not functioning properly. When it was audited on the evening of 8/7/90, the NOX analyzer was found to be reporting concentration values that were less than half of the expected values. One of the sample line fittings inside the analyzer was leaking. The leak was repaired on 8/8/90 and the analyzer was audited that same evening. The results of this audit were in good agreement with the expected values.

The problems listed above are not new to SNL. Similar problems were encountered during QAD's last audit of the aircraft's monitoring equipment.

Other equipment problems included failure of the air intake valve on two occasions. The first failure occurred 8/7/90 and resulted in the cancellation of the test in progress and the invalidation of all gas results. The valve to the sample bag stuck partially open, thus diluting the mixture in the bag. Although the valve was repaired, it failed again the following week.

Heat appears to be a problem. On Monday evening, 8/6/90, SNL personnel removed the tops from all the analyzers as they thought the heat (>100 deg.) was seriously affecting the performance of the analyzers and the on-board computer. SNL personnel mentioned that the on-board computer had been having noise problems with 6 of the 12 input lines registering more noise than normal. Small muffin fans were installed in an attempt to cool the electronics. The extreme temperatures to which the equipment and personnel are being subjected is viewed by the auditors to be detrimental to both equipment and personnel. During one of the tests in which a QAD auditor went on the aircraft, the temperature inside the aircraft felt hotter than it did on the tarmac at Dugway, which had been measured at 102 degrees. Conducting OB/OD tests when temperatures are less extreme would alleviate this problem.

The auditors feel that more time should be spent in checking out the aircraft monitoring equipment prior to the beginning of the field testing. It appears to take 3-7 days to get the equipment functioning properly once the aircraft is at the field test site.

The audit team strongly recommends that OB/OD find a way to improve SNL's support of the project or else look for alternatives to ensure that the project's needs will be met in a cost-efficient and timely manner. The problems that have precipitated this recommendation are viewed as ones that have had and, if unresolved, will continue to have a very disruptive and negative effect on the OB/OD project as a whole. These problems include:

1. The lack of back-up equipment and spare parts for the aircraft's monitoring equipment. Every time there is an equipment failure the OB/OD project loses time and data. How detrimental this is to the project is dependent on which data is lost and for how long. Considering how frequently SNL experiences equipment failures, it appears to be of utmost importance that adequate spare parts and back-up monitoring equipment be readily available. To lose data, cancel tests, or keep large groups of technical people waiting to perform their assigned tasks because of equipment failure is not cost effective and wastes resources (human and monetary). OB/OD needs to find a way to resolve this problem soon. (NOTE: QAD has made this recommendation <u>many</u> times: the Bang Box test, Phase A and Phase B.)

2. At best, there is a continuing problem with communications between SNL and the rest of the OB/OD project staff. SNL's independent operation coupled with an apparent unwillingness to help resolve problems and communicate openly has caused delays in the test schedule. Examples of this include: (1) SNL's stating they were going back to refuel the plane on 8/7/90 and would be back in 45 to 55 minutes. In fact they were experiencing problems with the sampling valve; they did not refuel. This left all DPG, QAD, ELI, and LESC personnel waiting at the site in temperatures exceeding 100 degrees F. Two hours later the test was cancelled for the day because of the valve problem. (2) QAD was told by ELI that the standard procedure for the aircraft testing included

radioing the ELI staff member, who routinely flies with the SNL sampling crew, when it is time for the aircraft to leave for the test site. This procedure enables ELI to work on necessary reports, etc. between tests. During routine auditing of the phase C tests, QAD auditors had the opportunity to observe activities at the Michael Airfield. The auditors noted the arrival of the ELI staff member and waited for the aircraft to leave. It was apparent that the aircraft was ready to return to the test site but it was not deployed. The aircraft continued to sit at the tarmac for an excessively long time. Finally, Wayne Einfeld arrived on a bike and the crew immediately hurried to the aircraft and it took off. Occurrences like this raise real questions concerning SNL's integrity.

3. OB/OD and SNL need to find a way to more effectively use the pilot's flight time. Currently, most of this time is wasted sitting on the ground. Since the time starts when the aircraft leaves Provo, it would help to keep the aircraft closer to the actual test site. Other possibilities would be to have Wayne Einfeld communicate with the other OB/OD staff via radio or phone for the 0700 meeting and delay the aircraft's departure until closer to the actual test time. The delay of a test burn because of pilot time is not cost effective. Especially when so little of the flight time is actually time spent in the air.

4. During the first Navy burn on 8/14/90 the aircraft appeared to miss the mass of the plume. The aircraft appeared to be passing under most of the plume instead of thru it. Perhaps the videos could verify whether this was actually the case.

B. Audits (8/27/90)

SNL had promised to determine the stability of NO_2 in the Teflon sample bag. This test was to have been done before Phase C began. The test was not done. SNL needs to do stability testing on the compounds analyzed for in the sample bag. The need for this was emphasized by the auditors when they did a second performance audit of the aircraft on 8/27/90. During this audit the stability of NO_2 was checked and an attempt was made to check the stability of ozone. The stability of the ozone could not be checked because the TECO 49PS would not operate properly under the small back pressure produced when the sample line was connected to the Teflon bag. The results of the stability check for NO_2 after 6 minutes in the Teflon bag indicated about a 20% loss of NO_2. The test was performed twice. Concentrations of 125 ppb were introduced into the bag and analyzed after 6 minutes. The analyzed concentrations were found to be 96 and 100.7 ppb, respectively. The results of this test indicate that further work needs to be done to assess the stability of the compounds in the Teflon bag especially some of the more reactive ones like ozone. (NOTE: The sample collected in the Teflon bag is analyzed within 10 minutes of collection.)

The results of the 8/27/90 performance audit conducted by QAD on the aircraft showed that all the monitors were operating within the target criteria of +/- 10% of the true value. QAD performed one point checks of these monitors. The percent difference for the monitors was found to range between -6.0 and +3.7 percent.

The auditors reported that the valve that had failed twice in earlier tests in Phase C had been replaced with a new unit and were told that further failures should not occur. However, once again it should be stressed that replacement parts for critical components need to be readily available.

2. Balance Room

The balance room is poorly set up. The space is cramped and the sample compositing activities in the adjoining room greatly increase the likelihood of dust contamination. The room contains a "particle buster" but its location does not provide any protection for the area in which the filters are loaded and unloaded.

The balance was originally connected to a lap top computer so that the sample weights could be processed automatically. However, the computer software did not work and the weighing had to be done manually.

The balance room operator was not as familiar with the procedures as he should have been. He did not understand that he was to put the samples on dry ice as soon as they were weighed and he did not know why he was doing it. Communication was apparently a problem. For example, after having the correct way to condition the filters explained to him on two different occasions, the filters were found to still be too tightly sealed to allow adequate equilibration. The filter conditioning problem was addressed a third time during the audit of the filter unloading procedures but the auditor has no confidence that the problem has been resolved. Data comparing the results of the tightly closed filters with those left open to the atmosphere were promised to the auditor and were to have been sent to QAD by ELI. These data have not been received.

The OB/OD project needs to have a well documented, carefully planned and implemented sample test series in which the filters are fully conditioned to a constant weight prior to sampling and then fully conditioned again to a constant weight after sampling to provide a documented, scientific basis for the filter conditioning and weighing procedures. This has not been accomplished to date and there is no excuse for this.

The audit of the filter loading/unloading and weighing procedures was hampered by the small space allotted for this activity and by a seeming unwillingness on SNL's part to provide opportunity to do an adequate audit. Most of the comments on the filter procedures are based on silent observation with minimal discussion after the fact or when no activities were in progress.

SNL appeared to follow the LOI for the unloading of filters from the filter holders and the subsequent clean-up of the filter holder with isopropanol. However, it was impossible to ascertain if reagent grade isopropanol was used as it was kept in a plastic wash bottle. It was also impossible to ascertain how adequately the filter holders were cleaned as they were not readily available for inspection by the auditor. However, from the auditor's vantage point the filter holders appeared to be wiped lightly with isopropanol-soaked "kimwipes". It should be noted that during the observation of the unloading of the filters from the 8/14/90 yellow D test that the folded filter from sample holder A had a pair of tweezers dropped on it and a cloud of dust came off the filter.

SNL appears to be keeping records of the pre- and post-filter weights in a logbook. These logbooks appear to be kept in a neat and orderly fashion. It was not possible to determine how adequate the documentation in the logbook was due to its unavailability for careful review. The afternoon the filter procedures were audited, SNL was in a hurry to return to Provo (perhaps because of pilot flight time) and the logbook and related information were taken with them.

The consequences of making changes in the filter conditioning and weighing operations do not appear to be carefully thought out or planned before they are implemented. For example, at the last minute it was decided to switch from storing the filters in aluminum foil to storing them between Teflon sheets. The use of Teflon sheets caused problems in phase B with sample particle loss; so why the sudden switch to a problem procedure?
These random, undocumented and unexplainable changes need to be eliminated. There is a real need for a firm commitment to a carefully planned, fully documented and faithfully adhered to set of procedures.

3. Summary

It is the general impression of the auditors that SNL does not want to communicate openly about its activities. It is also felt that perhaps SNL was not aware of EPA's purpose for being present and that this may have contributed to their seemingly uncooperative attitude. Whatever, it is apparent to the auditors that SNL needs to improve its support of the OB/OD project and needs to address the problems raised above.

Heat also seems to be a problem that needs to be addressed. The extreme temperatures in the aircraft cabin raise questions concerning the monitors' ability to function correctly. These temperatures also have a negative impact on SNL personnel. Perhaps OB/OD should seriously consider the possibility of leaving the monitoring equipment on the ground in a controlled environment. The aircraft could then be used to take bag samples which would be subsequently analyzed on the ground. The stability of the sample would have to be well documented for this type of analysis to be done. This is another reason to document the stability of the gases in the Teflon bag.

ALPINE WEST LABORATORY (AWL)

AWL is responsible for the analysis of the soil and ash samples collected from the craters, fallout pans, sputter pans, burn trays, etc. AWL receives the composited samples for analysis from LESC. These samples are to be delivered within 72 hours after the test. AWL is also responsible for the analysis of the filter samples.

QAD attempted to audit AWL on 8/16/90. The audit had been coordinated through ELI and the auditors had made it very clear that the audit was to be done when AWL was actually doing sample extraction. The auditors were told that AWL would be extracting samples on both 8/15/90 and 8/16/90. The auditors notified AWL thru ELI that they would be there on 8/16/90. However, when they arrived, they were told that the extractions had been finished the previous night and that AWL had expected them the previous day. Thus, one of the primary reasons for the audit was not accomplished.

A second major reason for the audit was to go over the logbooks with Christine Rouse. The auditors wanted to review the raw data she used to calculate the extraction efficiencies for target analytes from soil. This information, which had been given to QAD personnel on 8/7/90, reported recoveries for five samples over a concentration range from 0.001 to 10.0 ng/sample. The percent recoveries and standard deviations remained essentially constant across the whole concentration range. The occurrence of this is highly unlikely as variability around a sample inevitably increases as you approach the detection limit of the instrument. The auditors had planned to review this raw data to gain more insight into the actual design and execution of the test.

The auditors also wanted to obtain the list of detection limits for the OB/OD target analytes that Milton Lee had promised to send to Howard Crist. This list was needed to help QAD try to determine the source of the differences between AWL's results and QAD's spiked values for soil samples. Review of the raw data for the analysis of these samples had also been a high priority item for this audit. It had been determined by QAD's contractor on 8/10/90 that some if not all of the spiked compounds were indeed in the spiked soil samples and based on AWL's stated detection limits, they were at a concentration where they should have been detected. Unfortunately, AWL had not detected most of the spiked compounds, and for those detected, had reported concentrations that were significantly different from the spiked concentrations.

The auditors were unable to review this data because the analyst, Christine Rouse, was scheduled to leave in the next 5 minutes to catch a plane. When it was suggested that someone else could go over the data with the auditors, she said no one else would be able to understand the logbook. The auditors felt that was probably true based on the condition of the previous AWL logbooks that had been examined during past audits of AWL. (One of the QAD auditor's saw Ms. Rouse approximately 20 to 25 minutes later still at BYU!)

Since none of the primary reasons for the audit could be carried out, the auditors agreed to review the requested data at a later date. Ed Lee showed us the sample filtration and roto-vap procedures that were being done and Bill, AWL's new QA officer, reviewed the OB/OD QA/QC check list he had developed.

Overall, the audit of AWL was unproductive and disappointing. It is the auditors' opinion that future audits of AWL not be conducted unless there is reasonable assurance of the availability of both the data and AWL staff, and that actual extractions will be in progress.

ENVIRONMENTAL LABORATORIES INC. (ELI)

ELI has overall QA responsibility for Phase C of the OB/OD project. Although ELI was not directly audited, the auditors felt that comment should be made concerning the need for ELI personnel to set an example for other OB/OD project personnel in the manner in which they, themselves, adhere to "good" QA practices and to the OB/OD LOI's. It was noticed on several occasions that this was not the case. QA personnel of all people should know better than to enter the vicinity of the crater before LESC personnel have marked the crater sample sites. Also, they should know better then to enter the burn pan test site before the sample trays have been covered. And most certainly they should know better than to lift the lids on the sample trays to make photographs of the tray's contents. Although ELI personnel should and do know better, they were observed doing all three things in violation of the OB/OD LOI's. This type of thing has got to be eliminated. How can you correct or report deviations from correct procedure, if you yourself deviate from following the correct procedure?

CONCLUSIONS

A project the size of the OB/OD project absolutely must have the project officer available on site during the actual field test. It takes someone with authority, i.e. the project officer, to direct, correct, and ensure the smooth effective operation of the testing phase of the project. Field testing has enough innate problems without adding more caused by insufficient cooperation and coordination between the various project groups. This is the responsibility of the project officer and it cannot be easily assigned to or assumed by someone else.

If you have any questions concerning this interim report please contact me at FTS 689-2365.

Sincerely,

Linda F. Porter
Chemist, Research Monitoring and Evaluation Branch
Quality Assurance Division (MD-77B)

Letter, Raymond C. Rhodes, December 9, 1990, Raleigh, North Carolina.

ENCLOSURE 18

December 9, 1990

Mr. MacDonald B. Johnson
HQ-AMCCOM
AMSMC-DSM-D
Rock Island Arsenal
Rock Island, IL 61299

Dear Don:

This letter constitutes a response to the interim audit report presented by Ms. Linda F. Porter, Research Monitoring and Evaluation Branch, Atmospheric Research and Exposure Assesment Laboratory, Environmental Protection Agency, Research Triangle Park, NC in her letter to you of September 7, 1990. Earlier I had thought of waiting until EPA's final report is submitted. However, since their final report will not be available until after the the results of the spiked canisters are received from OGC and the spiked soil samples are recieved from AWL, I am submitting my comments on the interim report now in case a consolidated response is being prepared and for possible use in the draft OB/OD report being prepared.

My general comments are that I agree with most of the findings of the EPA audit team, but have several corrections and additional comments that I would like to make. These comments follow under the general headings used in the September 7 report.

Lockheed Environmental Services (LESC)

I agree that the LESC personnel appeared to be well trained and conscientious in performing their work. In fact, on one occasion one of the field sampling crew very appropriately "requested" the DPG Project Officer, their Quality Assurance Officer, and another vehicle driver to turn off the motors of their vehicles when they had left the motors (and air conditioners) running when parked on a field road near several detonation sites just prior to sampling.

The EPA report refers to Jim Stephens and Monte Law as supervisors, when the Quality Assurance Project Plan indicates that Monte Law was the LESC Quality Assurance Officer. During the first few days of testing both Stephens and Law were present in the field carrying out their respective responsibilities. However, during most of the test periods only Monte Law was present and appeared to be the supervisor and the QA Officer. It is my opinion that he wore two hats quite effectively, and the change allowed Stephens to pursue other work. This situation was made possible by the complete and detailed work of Floyd McMullin of ELI in recording and tracking all field samples, a responsibility that I would normally consider being that of LESC. In addition, Floyd assisted SNL in transporting in his personal automobile the filter adaptors to and from the aircraft at Michael Field and the filter weighing-soil handling building. In my opinion, these shifts of responsibilities did actually improve the effectiveness (and maybe? the efficiencies) of the operations, because of the particular individuals involved. However, the assumed responsibilities were not exactly as defined in the QAPP.

I quite agree with EPA's comment concerning the dustiness of the soil handling room, as one would expect. I believe that the technicians did take proper precautions in handling the samples to minimize any effects of the dust. However, I think that the entire room -- floor, equipment, tables, etc. -- could have been thoroughly cleaned more frequently to minimize the amount of dust present, particularly because of the weighing room being in the same building and the arrangement requiring one to walk through the soil handling room to get to the weighing room.

Sandia National Laboratories (SNL)

I agree that additional time should have scheduled for all of the aircraft equipment to be thoroughly and completely checked out, calibrated, and put through a trial run before the tests were to begin. In every instance, I believe, when the SNL crew and aircraft came from Albuquerque to Utah for tests, there were problems and delays in getting it completely operational as planned. Additionally, they often incurred delays because of lack of spare instruments and parts. This, in spite of a strong recommendation by EPA during SNL's first tests at DPG that adequate spare parts and instruments should be readily available.

At the top of page 6 of the interim report, EPA states that an ELI member "routinely flies with the SNL sampling crew". This statement is not true for Phase "C". An ELI person did sometimes fly with the crew of previous phases, but never during Phase "C".

Yes, the SNL flight operations always seemed to be at the mercy of the pilots, because of the pilot's "flight time" constraints. When I asked the SNL crew how the "flight time" was defined, the response was that "not even a group of Philadelphia lawyers could agree". And so, the pilot's word was always gospel. With scheduling being such an important consideration for the OB/OD field tests involving the aircraft, it should have behooved the SNL supervisor to know very precisely how "flight time" is defined.

I also agree that on several instances, the aircraft seemed to miss the center mass of the cloud. I have several photographs to indicate this -- the first and second passes for the third M1 propellant burn on September 5, 1990. Videos from the aircraft and from the DPG ground camera may confirm or negate these "amateur" photos. But it was most important that the sampling of the cloud from the M1 burns provide a maximum catch of particulate and gases to enhance the detection of any pollutants. I understand that the aircraft need not hit the center of the cloud because of the carbon balance principle -- but enough material must be caught in the sample to measure!

When will or has further work been completed to accurately and precisely determine the stability of the gases in the Teflon bag? This was a KEY ITEM that was to be checked in the Bang Box tests. It's late in the day to make this determination! If the 20% loss is valid for these gases, then an appropriate upward adjustment of <u>25%</u> should be considered in the data obtained.

Mention has been made previously of the likelihood of dust contamination of the weighing room from the adjacent soil handling room. And as an afterthought, an appropriate mat for wiping shoes should have been placed just outside the door to the weighing room, or an entrance air-lock (vestibule) built between the soil handling room and the weighing room. I also agree that the weighing room was too small for optimum operation. There was hardly room for more than two persons to move around. Further, the makeshift clear plastic curtain used to separate the filter loading and unloading operation from the weighing operation also restricted access and movement in the room. The curtain was installed to prevent the currents of air from the air conditioner/heater, the dehumidifier, and the electronic air filter from affecting the balance located on the other side of the curtain. Although this separation of the two areas would seem to inhibit the control of temperature and humidity in the weighing area, the temperature and humidity records indicate that such was not the case.

The "particle buster" referred to in the EPA report is apparently the German-manufactured soil pulverizer, which fortunately, because of dust generation, was seldom needed.

In the second paragraph under the Balance Room heading, the statement that "the weighing had to be done manually" could be misinterpreted. The handling of the filters is always done manually. It was only the recording of the weights that had to be done manually because of failure of the connected computer software (or hardware?). Because of the problem, the computer was disconnected from the balance.

The above-mentioned problem with the computer as well as other problems encountered with the weighing operation is still another instance where the SNL personnel did not have adequate time to "prove-in" the operations prior to the beginning of actual test operations. Other problems were the static electricity effects on the weighing when using the folded thick Teflon sheets, in which to handle and store the exposed filters. Aluminum foil was then substituted in place of the Teflon sheets originally planned. Another problem that was encountered was the use of a humidifier in the small room to assist in controlling the humidity. The normal running capacity of the humidifier was too great and caused excessive fluctuations in humidity. The solution for adequate humidity control was to aim at controlling the humidity at a much lower level, 25 to 55%, and to control the humidity by the operation of the air conditioner and <u>de</u>humidifier. And, as previously mentioned, the clear plastic curtain had to be "jury rigged" to preclude the effects of air currents on the balance.

Although the SNL crew were rather efficient and resourceful in solving the problems mentioned above, they had not solved all of the problems before the first test. (Actually, when the EPA audit team was present, they had not yet settled on a consistent procedure. At the time, I personally was permitting the SNL personnel to solve their problems before auditing the operation.)

Alpine West Laboratory (AWL)

Nothing much can be added to the auditors' comments. Hopefully, their critical problems can be solved. I, personally, have never had a opportunity to audit AWL under routine operational conditions. Because of their problems with the analyses of the two series of spiked soil samples, they were continually involved in investigating their analytical process to solve their problems and optimize the process. I do very much agree with the decision that was made to halt all analyses by AWL until they provided objective evidence of adequate corrective action. I do plan to prepare a report on the combined results of the first three series of EPA-spiked soil samples.

Environmental Laboratories Inc. (ELI)

I believe that the noted observations made by the EPA auditors were somewhat strained in order to make some comments. Good judgment has to be exercized by an auditor in knowing when it might be appropriate to intentionally challenge the rules to observe the action of the operating personnel. In several of the incidences mentioned, the operating personnel very appropriately warned and halted the movement by the ELI personnel, and in the other, extreme care was taken to prevent any deleterious effect of taking the photograph. An adequate audit cannot be made just by observing from a distance and by asking questions. In many cases, the very act of asking questions of persons involved in actual operations is a disrupting influence that might adversely affect the procedure being followed.

Should you have any questions concerning these comments, please call.

Very truly yours,

Rocky

Raymond C. Rhodes

cc: Andrulis Research Corp., Cecil Eckard

Letter, February 19, 1991, Environmental Quality Assurance Management, Inc, Raleigh, North Carolina.

ENCLOSURE 19

EQAMI
Environmental Quality Assurance Management, Inc.

February 19, 1991

Mr. MacDonald B. Johnson, Program Manager
Open Burning/Open Detonation
HQ-AMCCOM
AMSMC-DSM-D
Rock Island Arsenal
Rock Island, IL 61299

Dear Mr. MacDonald:

As requested by telephone with you and Mr. Douglass Bacon of Andrulis Research Corporation on Friday, February 15, 1991, I have prepared and have attached hereto a brief report summarizing the activities of Environmental Laboratory Incorporated (ELI) during Phase C of the OB/OD program. A more comprehensive report by ELI would have been prepared in the normal course of events following the field and laboratory activities for Phase C. However, any such efforts that may have been underway ceased when funding to ELI was terminated. Nevertheless, the major results of ELI activities, including any quality system audits, were included in the comprehensive quality assurance report for all phases (Bang Box, A, B, and C) that I prepared under contract to ARC. These ELI activities were either described explicitly or implied in the recommendations of the report. Some of these activities were also detailed in the letter of December 9, 1990 from R. C. Rhodes to MacDonald Johnson, included *in toto* in an Appendix of the comprehensive report.

If you have any questions concerning the attached report, please call.

Sincerely,

R C Rhodes

Raymond C. Rhodes, President

cc: Mr. Douglass Bacon, ARC

6704 Winding Trail Road • Raleigh, North Carolina 27612 • (919) 848-0242

ELI ACTIVITIES DURING PHASE C OF OB/OD

The activities of ELI during Phase C of OB/OD were performed by four persons:

1. Dr. Gary Booth, President of ELI
2. Raymond C. Rhodes, Quality Assurance Consultant, EQAM
3. Floyd W. McMullin, Jr., Quality Assurance Officer
4. Todd D. Parrish, Quality Assurance Officer

Dr. Gary Booth, President of ELI, was the Quality Assurance Project Officer for the OB/OD program throughout all phases -- BangBox, A, B, and C. He was also a member of the Technical Steering Committee (TSC). In both capacities, he provided the necessary interface between the technical and Quality Assurance considerations and concerns for OB/OD. He directed (a) the preparation of the Quality Assurnce Project Plan (QAPP) for the various phases, (b) the Quality Assurance coordination with other operational organizations, and (c) the conducting of on-site quality systems audits of the various field and laboratory operational organizations and facilities.

Raymond C. (Rocky) Rhodes, during the BangBox, A, and B phases, was a Quality Assurance Specialist for the Environmental Protection Agency (EPA). As such, he provided Quality Assurance guidance and oversight for these early phases of OB/OD. Following retirement from EPA, he was employed by ELI to play a key role in the QA activities for Phase C. As such, he participated in the QA planning and the preparation of the QAPP for Phase C. During the conduct of Phase C, he attended various initial and daily planning meetings , observed the field test operations, the filter weighing operations, and the soil weighing and handling operations at Dugway Proving Ground (DPG). Limited observations were made of the Alpine West Laboratory (AWL) operations because during the entire months of August and September, 1990 the AWL personnel were involved in investigational efforts to more fully develop and refine their methods to extract and analyze OB/OD soil samples. Subsequent to the termination of ELI activities, R. C. Rhodes was engaged by ARC to prepare a final and comprehensive QA report for all the phases of the OB/OD effort.

Floyd W. McMullin, Jr., Quality Assurance Officer, was involved in all the phases of OB/OD. During Phase C, he was involved in the preparation of the QAPP. He observed the pre-test soil sampling in the field test site areas at DPG, the filter weighing and soil handling facilities at DPG, and pre-test records of the continuous pollutant gas analysers on the fixed wing aircraft (FWAC). Whereas on previous phases, he flew with the crew in the FWAC to observe operations, he flew on only one flight (August 14, 1990, a Yellow D test) during Phase C. McMullin's major efforts during Phase C involved the assignment of sample numbers and the distribution of sample forms to the field operational groups, tracking of samples to laboratories for analysis, and the maintaining and distribution of a complete computer sample listing for Phase C. He also assisted in the transportation of test filters between the FWAC at Michael Field at DPG and the filter weighing facility at DPG. Although these sample number assignment and sample tracking activities are very necessary and important activities for the project, in a strict sense they are not QA activities. The development of the sample numbering system was a joint effort of the technical and QA personnel and are a major QA concern. The basic sample tracking and chain-of-custody procedures, developed previously by ELI for earlier phases, was used during Phase C.

Todd Parrish was involved only with some sample inventory activities at, and sample shipping from, the ELI facilities at Provo, UT.

The dates of the activities of Rhodes, McMullin, and Parrish, as indicated from their notebook records are indicated in the following table.

The major findings of ELI personnel during activities and systems audits of Phase C are included in the final Quality Assurance report as observations (Section 3.1.1 for Sandia National Laboratory and Section 3.1.8 for Lockheed Engineering and Sciences Corporation), and as implied by the content of the Summary and Conclusions (Section 5) and Recommendations (Section 6).

During Phase C, the conducting of performance audits was delegated from ELI to EPA because of EPA's unique experience and capabilities. Performance audits, in the form of spiked canisters, were conducted for the Oregon Graduate Center for the analysis of volatile organic compounds. Performance audits, in the form of spiked soil samples, were conducted for the Alpine West Laboratory for the analysis of semi-volatile organic compounds. The results of these performance audits are to be reported by EPA.

No on-site systems audits of other participating laboratories -- OGC, SSL, and LBL -- were conducted during Phase C. It was decided by Quality Assurance personnel and the Program Manager that the information gained form previous audits was sufficient to be carried over and applicable to Phase C.

SCHEDULE OF ACTIVITIES BY ELI PERSONNEL

JUNE

Tue 19		McMullin	Parrish
Sat 24			Parrish

JULY

Tue 3	Rhodes		
Thu 5	Rhodes	McMullin	
Fri 6	Rhodes	McMullin	
Mon 9	Rhodes		
Tue 10	Rhodes		
Mon 16	Rhodes		
Tue 17		McMullin	
Fri 20		McMullin	
Mon 23		McMullin	
Thu 26	Rhodes		
Mon 30	Rhodes	McMullin	
Tue 31		McMullin	

AUGUST

Wed 1	Rhodes	McMullin	
Thu 2	Rhodes		
Fri 3	Rhodes		
Mon 6	Rhodes	McMullin	
Tue 7	Rhodes	McMullin	
Wed 8	Rhodes	McMullin	
Thu 9	Rhodes	McMullin	Parrish
Fri 10	Rhodes		Parrish
Sun 12		McMullin	
Mon 13	Rhodes	McMullin	
Tue 14	Rhodes	McMullin	Parrish
Wed 15	Rhodes	McMullin	Parrish
Thu 16	Rhodes	McMullin	Parrish
Fri 17	Rhodes		Parrish
Sat 18	Rhodes	McMullin	
Mon 20	Rhodes	McMullin	
Tue 21	Rhodes	McMullin	
Wed 22	Rhodes	McMullin	Parrish
Thu 23	Rhodes	McMullin	
Mon 27	Rhodes	McMullin	
Tue 28	Rhodes	McMullin	
Wed 29	Rhodes	McMullin	
Thu 30	Rhodes	McMullin	
Fri 31	Rhodes		

SEPTEMBER

Tue	4	Rhodes	McMullin	
Wed	5	Rhodes	McMullin	
Thu	6	Rhodes	McMullin	
Fri	7	Rhodes		
Sun	9			Parrish
Mon	10	Rhodes		
Tue	11	Rhodes		
Wed	12	Rhodes		Parrish
Thu	13	Rhodes		
Fri	14	Rhodes		
Sat	15	Rhodes		
Mon	17	Rhodes		
Tue	18	Rhodes	McMullin	
Wed	19	Rhodes	McMullin	
Thu	20	Rhodes		
Fri	21	Rhodes	McMullin	
Mon	24	Rhodes		Parrish

APPENDIX E. CONSOLIDATED ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AEHA	U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland
AFB	Air Force Base
AMC	U.S. Army Materiel Command, Alexandria, Virginia
AMCCOM	U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois
amino-PAH	aminopolycyclic aromatic hydrocarbons
ANOVA	analysis of variance
AP	ammonium perchlorate
APS	aerodynamic particle sizer
ASASP	active scattering aerosol spectrometer probe
AWL	Alpine West Laboratories, Provo, Utah
BB	BangBox
BCD	Battelle Columbus Division, Columbus, Ohio
BD	target analyte not found in concentrations above detection limits
BYU	Brigham Young University, Provo, Utah
CAA	Clean Air Act
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
CI-SIM	chemical ionization, selective-ion monitoring
CSI	Columbia Scientific Instruments
C•V	concentration times cloud volume method
CWA	Clean Water Act
DMC	Data Management Center
DMPS	differential mobility particle sizer
DoD	Department of Defense
DPG	U.S. Army Dugway Proving Ground, Dugway, Utah
EC	electron capture *or* elemental carbon
ECD	electron capture detector
EDAX	energy-dispersive X-ray analysis
EER	Energy and Environmental Research Corporation, Irvine, California

EF	emission factor(s)
EI	electron impact
EI-MS	mass spectrometer used in the electron impact ionization mode
EI/MS	electron impact ionization/ mass spectrometry
EIS	environmental impact statement
ELI	Environmental Labs, Incorporated, Provo, Utah
EOD	explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
EPO	Environmental Protection Office, U.S. Army Dugway Proving Ground, Dugway, Utah
ER	expansion ratio
FID	flame ionization detector
FSSP	forward scattering spectrometer probe
FTIR	Fourier Transform Infrared Spectrometry
FWAC	fixed-wing aircraft
GC	gas chromatograph(y)
GC-ECD	gas chromatography with an electron capture detector
GC-FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography-mass spectrometry
GLP	good laboratory practices
HE	high explosive
HMX	octamethylenehexanitramine
HNBB	hexanitrobibenzyl
HRGC/HRMS	combined capillary column gas chromatography/high resolution mass spectrometry
HS	high-speed
LASD	Los Angeles Sheriff Department
LBL	Lawrence Berkeley Laboratory, Berkeley, California
LC	liquid chromatography
LOD	limit of detection
LOI	letter(s) of instruction
NO_x	nitrogen oxide (s)

MR	multiple range
MRI	Midwest Research Institute, Kansas City, Kansas
MS	mass spectrometry (or mass spectrometer)
MSA	Mine Safety and Appliance Company
NA	not targeted for analysis *or* not applicable
NASA	National Aeronautical and Space Administration
NATICH	National Air Toxics Information Clearinghouse
NBS-SRM	National Bureau of Standards (now NIST)- Standard Reference Material
ND	no data *or* detection limit not determined
NEPA	National Environmental Policy Act
NF	not found in the sample matrix *or* not determined
NIST	National Institute of Science and Technology
nitro-PAH	nitropolycyclic aromatic hydrocarbons
NIOSH	National Institute for Occupational Safety and Health
NOSIH	Naval Ordnance Station, Indian Head, Maryland
NO_x	nitrogen oxides
NS	not sampled
OB	open burning
OB/OD	open burning/open detonation
OC	organic carbon
OD	open detonation
OGC	Oregon Graduate Center, Beaverton, Oregon
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbons
PANH	polycyclic aromatic nitrogen heterocycles
PAOH	polycyclic aromatic oxygen heterocycles
PCDD	polychlorinated dibenzodioxins
PCDF	polychlorinated dibenzofurans
PETN	pentaerythritol tetranitrate
PEP	propellants, explosives, and pryotechnics
PIC	products of incomplete combustion
PICI/SIM	Positive ion chemical ionization/selective ion monitoring

PID	photoionization detector
PIP	product improvement program
PM	program manager
PMS	Particle Measuring Systems, Inc.
PUF	polyurethane foam
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QAA	quality assurance agency
QAPP	quality assurance project plan
QAU	quality assurance unit
RCRA	Resource Conservation and Recovery Act
RDX	hexamethylenetrinitramine
REMB	Research Monitoring and Evaluation Branch of USEPA
RFD	Reno (Nevada) Fire Department
RIC	relative ion count
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina
SDPDA	Special Defense Property Disposal Account
SEM	scanning electron microscope/microscopy
SFC	supercritical fluid chromatography
SFC/MS	supercritical fluid chromatography/mass spectrometry
SF_6	sulfur hexafluoride
SIM	selected-ion monitoring (or selective-ion monitoring)
SNL	Sandia National Laboratories, Albuquerque, New Mexico
SOP	standing operating procedures
SS	stainless steel
SSC	stainless steel canister
SSL	Sunset Laboratory, Forest Grove, Oregon
STEL	short-term exposure limit
STP	standard temperature and pressure (25°C and 760 torr)
TCD	thermal conductivity detector

TDP	test design plan
TEAD	U.S. Army Tooele Army Depot, Tooele, Utah
TECO	Thermo Electron Instruments (Company)
TECOM	U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland
THC	total hydrocarbon
TLV	threshold limit values
TNT	2,4,6-trinitrotoluene
TSC	technical steering committee
TSP	total suspended particulate
TWA	time-weighted average
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland
UV	ultraviolet
VOC	volatile organic compounds
VOST	semivolatile organic sampling train
VSDM	Volume Source Diffusion Model
XRF	X-ray fluorescence *or* X-ray fluorescence spectrometer

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APPENDIX F. REFERENCES

(1) U.S. Armament, Munitions and Chemical Command, Rock Island, Illinois, Final Report, Development of Methodology and Technology for Identifying and Quantifying Emission Products from Open Burning/Open Detonation Thermal Treatment Methods,BangBox Test Series, 1 October 1991.

(2) Letter, Environmental Labs, Inc., Provo, Utah, 30 June 1990, subject: Interim Report, QA/QC Program for OB/OD Phase 'A', June 1989.

(3) U.S. Environmental Protection Agency, Quality Assurance Materials Bank: Analytical Reference Standards, Las Vegas, Nevada, 1988.

(4) Rhodes, R.C., "Limits of Uncertainty of Environmental Measurements (Using as an Example, the Dichotomous Particulate Sampler) Translations", 44th Annual Quality Congress, ASQC, San Francisco, California, May 14-16, 1990.

(5) Kanare, H.M., Writing the Laboratory Notebook, American Chemical Society, Washington, 1985

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APPENDIX G. BIBLIOGRAPHY

(1) Arter, D.R., *Quality Audits for Improved Performance*, Milwaukee, Wisconsin, 1989.

(2) Dux, J.P., *Handbook of Quality Assurance for the Analytical Chemistry Laboratory*, 2nd ed., Van Nostrand Reinhold, New York, New York, 1990.

(3) Booth, Gary, *A QA Project Plan for: OB/OD Field Test, Phase "B"*, Environmental Labs, Inc., Provo, Utah, 22 September 1989.

(4) Holland, J.C. and S.F. Henry, "Humidity Effects on Weighing Glass-fiber and Quartz-fiber Filters," Northrop Services Inc., Environmental Sciences, Research Triangle Park, North Carolina.

(5) Memorandum, undated, MacDonald B. Johnson, subject: Interim Report for Field Test Phase "A" of the Open Burning/Open Detonation Study.

(6) Notebook (Parrish), Environmental Labs, Inc., Provo, Utah, period: 4 December 1989 thru 9 September 1990.

(7) Logbook (Parrish), Environmental Labs, Inc., Provo, Utah, period: 21 November 1988 thru 22 February 1990.

(8) Notebook (McMullin), Environmental Labs, Inc., Provo, Utah, 6 July 1990.

(9) Logbook (McMullin), Environmental Labs, Inc., Provo, Utah, period: 30 November 1988 thru 9 July 1990.

(10) Logbook (McMullin), Environmental Labs, Inc., Provo, Utah, period: 19 June 1990 thru 2' September 1990.

(11) Letter MD-77B, U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, June 12, 1990.

(12) Logbook (Division 6321), Sandia National Laboratories, Albuquerque, New Mexico, purpose: Balance Log for Weighing Filters for OB/OD Phase "C", July thru September 1990.

(13) Logbook (Apple), Sandia National Laboratories, Albuquerque, New Mexico, purpose: Bang Box Testing, August 21-24, 1990.

(14) Sayle, A.J., Management Audits: The Assessment of Quality Management Systems, 2nd ed., Quality Press, Milwaukee, Wisconsin, 1988.

(15) Taylor, J.K., Quality Assurance of Chemical Measurements, Lewis Publishers, Chelsea, Michigan, 1987.

(16) Taylor, J.K., Statistical Techniques for Data Analysis, Lewis Publishers, Chelsea, Michigan, 1990.

(17) U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, Detailed Test Plan, Open Burning/Open Detonation, Field Trials Phase "C", TECOM Project No. 2-CO-210-000-017, 12 July 1990.

(18) Thresh, J.L., How to Plan, Conduct, and Benefit From Effective Quality Audits, MGI Management Institute, Harrison, New York, 1984.

(19) U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois, Test Design Plan for the Identification and Quantification of Products and Residues from the Open Burning/Open Detonation of TNT, Composition "B", Explosive "D", RDX, and Propellants (Field Test Phase "C"), April 1990.

(20) U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, "List of Designated Reference and Equivalent Methods", April 12, 1988.

(21) U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment

Laboratory, Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I, Principles; Volume II, Ambient Air Methods; Volume V, Meteorological Measurements, Research Triangle Park, North Carolina,

(22) U.S. Environmental Protection Agency, Analytical Reference Standards: Federal Supplemental Index, Las Vegas, Nevada, 1987.

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