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AD-197 461  
VOL II

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HIGH-TEMPERATURE FLUID-WALL  
REACTOR TECHNOLOGY  
RESEARCH, TEST AND  
EVALUATION PERFORMED AT  
NAVAL CONSTRUCTION  
BATTALION CENTER,  
GULFPORT, MS, FOR THE USAF  
INSTALLATION/RESTORATION  
PROGRAM

R.W. HELSEL, R.W. THOMAS

EG&G IDAHO  
P.O. BOX 1625  
IDAHO FALLS ID 83415

JANUARY 1988

FINAL REPORT

JUNE 1985 - JULY 1985

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ENGINEERING AND SERVICES LABORATORY  
AIR FORCE ENGINEERING AND SERVICES CENTER  
TYNDALL AIR FORCE BASE, FLORIDA 32403

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REPORT DOCUMENTATION PAGE				Form Approved GSA No. 2706-108	
1. SECURITY CLASSIFICATION UNCLASSIFIED		16. RESTRICTIVE MARKINGS			
2. AUTHORING ORGANIZATION NAME(S)		3. DISTRIBUTION/AVAILABILITY STATEMENT Approved for Public Release Distribution Unlimited			
4. AUTHORING ORGANIZATION REPORT NUMBER		5. MONITORING ORGANIZATION REPORT NUMBER ESL-TR-87-06 Vol 11			
6a. NAME OF REPORTING ORGANIZATION EWG Idaho	6b. OFFICE SYMBOL (if applicable)	7a. NAME OF MONITORING ORGANIZATION			
7a. ADDRESS (City, State, and ZIP Code) Idaho Falls ID 83415		7b. ADDRESS (City, State, and ZIP Code)			
8a. NAME OF FUNDING SPONSORING ORGANIZATION (Air Force Engineering & Services Center)	8b. OFFICE SYMBOL (if applicable) RDW	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER			
10. ADDRESS (City, State, and ZIP Code) AFESC/RDW Randall AFB FL 32403-6001		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO	PROJECT NO	TASK NO	ORDER ACCESSION NO
11. TITLE (Include Security Classification) UNCLASSIFIED High-Temperature Fluid-Wall Reactor Technology Research, Test and Evaluation Performed at Naval Construction Battalion Center, Gulfport MS for the USAF Installation Restoration					
12. AUTHOR(S) R.W. Heibel and R.W. Thomas					
13a. TYPE OF REPORT Final	13b. TIME COVERED FROM Jun 85 to Jul 85	14. DATE OF REPORT (Year, Month, Day) January 1988	15. PAGE COUNT 143		
16. SUPPLEMENTARY NOTATION Availability of this report is specified on reverse of front cover.					
17. DISTRIBUTION STATEMENTS		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)			
17a. UNCLASSIFIED	17b. GROUP	Herbicide Orange Soil Sampling 2,4-D Pyrolysis			
17c. SUB GROUP		Dioxin 2,4,5 T			
		Analytical Methods 2,3,7,8-TCDD			
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The objective of this effort was to examine the feasibility of using a thermal-pyrolysis technology to treat Herbicide Orange (HO)-contaminated soil at the Naval Construction Battalion Center, (NCBC) Gulfport MS. A field demonstration of a pilot-scale unit provided the technical evaluation and cost estimates for comparison with other cleanup technologies. The J.M. Huber Advanced Electric Reactor (AER), a high-temperature fluid wall reactor, was used to successfully treat 1100 pounds of soil contaminated with Herbicide Orange and 2,3,7,8-tetrachlorobenzo-p-dioxin (TCDD). The AER process pyrolysis organics at a temperature of 3600 to 4100 degrees F. under a nitrogen atmosphere to prevent combustion. After AER testing was completed, samples of the NCBC feedstock and test-treated soil were sent to two laboratories for analysis. Based on two composite samples, analysis of feedstock showed 2,3,7,8-TCDD levels of 111 and 193 parts per billion (ppb). Concentrations in the treated soil for the sum of all dioxin/furan congeners was less than 1 ppb, the Air Force test criterion. The AER process demonstrated the					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED			
22a. NAME OF RESPONDER INDIVIDUAL JEFFREY J. SHORT, GS-12, DAF		22b. TELEPHONE (Include Area Code) (904) 283-2942	22c. OFFICE SYMBOL RDW		

No. 19 continued:

capability to treat dioxin-contaminated soil and may be considered for full-scale soil restoration at USAF MC sites. Sensitivity analyses of six variables (geographic location, soil quantity, electrical power prices, labor, capital equipment use charge and transportation) were performed to estimate cost for conditions other than those found at NCIC. The process may have application for treatment of more easily pyrolyzed organic compounds such as semivolatiles, pesticides, and polychlorinated biphenyls, as well as some inorganics.

This report is organized into two volumes: Volume I presents the final report on the performance of an advanced electric reactor for use in decontaminating soil containing Herbicide Orange. Volume II presents supplementary analytical data and historical information that supports the the research findings reported in the first volume.



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Unannounced	<input type="checkbox"/>
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This report was prepared for the Air Force in engineering and  
development, Aerospace Services Laboratory, Lyndon B. Johnson  
Florida, under Joint Order Number (JO ) 2107 9027. The principal  
contractor, JCS Idaho, Inc., is the prime contractor for the  
Department of Energy, Idaho National Engineering Laboratory. The  
major subcontractor for the project is the W. W. Huber, Corp.,  
Houston, Texas.

This report is contained in two volumes: Volume I presents the  
final report on the performance of an advanced electric reactor  
for the incineration of hazardous waste containing Herbicide Orange.  
Volume II presents complementary analytical data and historical  
information that supports and the research findings reported in  
Volume I.

Other contributors to this report include: W.A. Frop, A.S. Grey,  
R.L. Miller, R.D. Holland, R.D. Carvero, W.S. Williams, and  
B. Petersen.

This report has been reviewed by the Public Affairs Office (PAO)  
and is releasable to the National Technical Information Services  
(NTIS). It will be available to the general public,  
including foreign nationals.

This report has been reviewed and approved for publication.

*Terry L. Stoddart*

*James J. Walsh*

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## VOLUME II

### SECTION I INTRODUCTION

#### A. OBJECTIVE

The objective of this program is to demonstrate the feasibility of using a thermal-pyrolysis technology for soil cleanup and restoration of a Herbicide Orange (HO)-contaminated site at the Naval Construction Battalion Center (NCBC) at Gulfport, Mississippi. This program is under the sponsorship of the Air Force Engineering and Services Center (HQ AFESC), Tyndall Air Force Base, Florida. The objective is twofold:

1. Perform a field demonstration with a pilot-scale unit at the NCBC location using the Advanced Electric Reactor (AER) process owned by the J. M. Huber Corporation of Borger, Texas. The AER is a high-temperature fluid wall reactor.
2. Provide technical evaluation and cost estimates for full-scale cleanup/site restoration using the AER technology, which would provide information to compare this technology with others.

A specific goal of this technology testing was to reduce the total isomers of tetra, penta, and hexachlorodibenzo-p-dioxin and respective isomers of polychlorodibenzofuran to less than 1 part per billion (ppb). The overall soil treatment goal of the demonstration was to reduce the level of contaminants to criteria acceptable to Headquarters, U.S. Environmental Protection Agency (EPA) to facilitate the delisting of the soil under the auspices of the Resource Conservation and Recovery Act (RCRA) of 1976, as amended by the Hazardous and Solids Waste Amendments (HSWA) of 1984.

The AER field demonstration was one of two technologies selected for the Air Force Small-Scale Demonstration Program. Those technologies are being evaluated for decontamination treatment of former Department of



Defense (DOD) HO sites. The purpose of the research demonstrations is to provide actual field data on the feasibility of the technology so that scaleup and cost-effectiveness can be determined for future restoration efforts. The other small-scale technology undergoing research is a thermal desorption process being performed by the IT Corporation (ITC) at NCBC and Johnston Island (Pacific Ocean). Results of these tests appear in separate reports.

#### B. BACKGROUND

NCBC is a fenced, limited-access military installation. It is a land area of several square miles located approximately 2 miles from the Gulf of Mexico, and is approximately 20 feet above sea level.

Approximately 12 acres at NCBC served as an HO storage site. The storage site was stabilized with Portland cement approximately 30 years ago. The stabilized soil provided a hardened storage area for heavy supplies and equipment. Over the years, additional fill materials (shell, rock, soil, asphalt, and tar) were added to the storage area, providing a cover of up to several inches over the cement-stabilized soil. Through use, the contaminated site is now about 18 acres. During 1980, retention basins were constructed on the storage site to prevent migration of dioxin-contaminated soils offsite by surface runoff. Currently, the storage site within the fenced perimeter is a restricted area and is not used.

#### C. SCOPE

Volume I of this report presents the results of a pilot-scale pyrolysis process to treat NCBC soil contaminated by polychlorodibenzo-p-dioxins and polychlorodibenzofurans and presents a cost estimate for full-scale remedial action by this process. This volume presents appendices to document supporting data.

APPENDIX A

REQUEST FOR AND EPA AUTHORIZATION LETTERS FOR AIR FORCE  
ENVIRONMENTAL RESTORATION TECHNOLOGY, RESEARCH, AND TEST  
EVALUATION PROGRAM AT NCBC

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P.O. BOX 1625, IDAHO FALLS, IDAHO 83415

Appendix A, Exhibit 1

bcc: K. L. Falconer **KF**  
F. C. Fogarty  
T. H. Smith  
D. L. Uhl  
Central Files  
H. D. Williams File

March 14, 1985

Mr. Paul des Rosier  
Deputy Chairman, Dioxin Disposal Advisory Group  
Environmental Protection Agency  
401 M Street SW  
Washington, DC 20460

TRANSMITTAL OF TECHNICAL INFORMATION FOR USAF RESEARCH AND TEST EVALUATION  
-HDW-4-85

Dear Mr. des Rosier:

Based on previous discussions with the Dioxin Disposal Advisory Group (DDAG) and following the guidance provided by DDAG, EG&G Idaho, Inc. has prepared the attached document for review by DDAG. The document presents technical information concerning the Research Test and Evaluation activities of the United States Air Force (USAF) Environmental Restoration Program for former Herbicide Orange storage sites.

Captain T. L. Stoddart, USAF, Engineering Services Center, (HQ AFESC) has arranged for a presentation of this information to the DDAG in Washington, DC, on March 21, 1985 at 1000 hours. Representatives of EG&G Idaho, Inc. and its subcontractors, the IT Corporation and J.M. Huber Company, will be present to provide additional information or answer questions as they arise. Enclosed are eleven copies of the document for you to distribute at your discretion.

On behalf of the USAF Engineering Services Center and our subcontractors, we are pleased to present this information to you and will look forward to further discussions on March 21, 1985. If questions arise prior to that date, please contact me at FTS 583-1763 or K. L. Falconer at FTS 583-1559.

Very truly yours,

H. D. Williams  
Senior Program Specialist  
Hazardous Waste Program

March 14, 1985  
Mr. Paul des Rosier  
HDW4-85  
Page 2

ag

Enclosure:  
as Stated

cc: I. Aoki, DOE-ID  
M. Cook, EPA  
K. Kleveno, EPA  
J. McGraw, EPA  
T. L. Stoddart, Captain, USAF  
J. O. Zane, EG&G Idaho (w/o Enclosure)



Appendix A, Exhibit 2  
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

APR 25 1985

OFFICE OF  
SOLID WASTE AND EMERGENCY RESPONSE

Colonel Robert Boyer  
HQ AFESC/RD  
Tyndall Air Force Base, FL 32403

Dear Colonel Boyer:

We have reviewed your document entitled "Environmental Restoration Technology--Research and Test Evaluation," informing the Environmental Protection Agency (EPA) of your intent to treat soils contaminated with 2,3,7,8-tetra-chlorodibenzo-p-dioxin (2,3,7,8-TCDD). We understand that you wish to conduct a series of research tests on less than 3,700 pounds of soil (less than two tons, or two cubic yards) contaminated with approximately 200 ppb of 2,3,7,8-TCDD. The 2,3,7,8-TCDD-contaminated soil is located at the Naval Construction Battalion Center (NCRC), Gulfport, MS, the site of the research tests. We also understand that you plan to destroy the 2,3,7,8-TCDD in the soil by testing two treatment units for approximately three to five weeks. The two units are: 1) thermal pyrolysis using the Advanced Electric Reactor developed by the J. M. Huber Company, and 2) thermal desorption followed by ultraviolet light destruction, developed by the IT Corporation. Because the destruction tests are being conducted for research purposes, you have requested a waiver from notification under 40 CFR Part 775.

On March 21, 1985, the Dioxin Disposal Advisory Group (DDAG) met with the U.S. Air Force to discuss the details of the planned research and evaluation studies. As a result, the DDAG determined that the proposal involves potentially feasible technologies, that the technical, safety, and environmental factors have been adequately addressed, and that the research activities will provide useful information in the destruction of 2,3,7,8-TCDD-contaminated soils. Thus, a research waiver from the notification requirements under 40 CFR Part 775 is hereby granted to the U.S. Air Force (HQ AFESC) to conduct the research tests. The waiver is being granted since the quantity of soil is small, the equipment being used for the research is pilot scale, and the tests to be conducted are of short duration. If testing should continue beyond July 15, 1985, the effective date of the RCRA dioxin regulation (50 FR 1978-2006; January 14, 1985), the activities will be subject to the provisions of that rule.

Your research at Johnston Island, however, will occur after July 15, 1985. As such, it will be subject to the RCRA dioxin listing. As discussed with members of your staff, EPA is proceeding with the preparation of a research development and demonstration permit. If you have any questions, please feel free to contact Dr. Howard Fribush, Office of Solid Waste, on (202) 475-6678.

Sincerely yours,

*Jack W. McGraw*

Jack W. McGraw  
Acting Assistant Administrator

cc: Captain Terry Stoddart  
HQ AFESC/RDVW  
Tyndall Air Force Base

APPENDIX B

REQUEST FOR AND EPA AUTHORIZATION LETTERS  
FOR DIOXIN-CONTAMINATED WASTE DISPOSAL

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DEPARTMENT OF THE AIR FORCE  
HEADQUARTERS AIR FORCE ENGINEERING AND SERVICES CENTER  
TYNDALL AIR FORCE BASE, FL 32403

24 June 85

Reply to attn of: RDVW

subj: Herbicide Orange Waste Disposal; Letter of Transmittal

TO: Commanding Officer  
Naval Construction Battalion Center  
Gulfport, MS 39501  
code 470

1. Please find attached copies of documents pertaining to the disposal of Herbicide Orange contaminated wastes that resulted from HQ AFESC/RDVW research projects at the Naval Construction Battalion Center (NCBC). Tab #1 is the TOSCA notification submitted to the Environmental Protection Agency. Tab #2 is the Environmental Protection Agency letter authorizing the Air Force to dispose of the described waste.
2. The exact number of drums scheduled for disposal will be determined on the last day of packing. The total number of drums will be less than that stated in the permit application. The reduced number of drums resulted from waste compaction that reduced the total volume to be disposed. The description of the waste is accurate. the only variation is that the soltrol solvent described will be shipped as a solidified material. the solidification step was a requirement stipulated by Rollins inc.
3. The generation of the described waste material is a one time only action related to our research at NCBC. No additional disposal requirement is projected.
4. Should you have questions please contact me at (601)-864-0056.

Terry L. Stoddart, Capt, USAF, BSC  
Project Manager

attach  
tab #1 TOSCA letter  
tab #2 EPA authorization





DEPARTMENT OF THE AIR FORCE  
HEADQUARTERS AIR FORCE ENGINEERING AND SERVICES CENTER  
TYNDALL AIR FORCE BASE, FL 32403

APR 25 1985

REPLY TO  
ATTN OF RDVW

SUBJECT: Notification of Disposal of TCDD (Dioxin) Contaminated Waste from Air Force  
Environmental Restoration Activities

TO:  
Mr. Jack McGraw  
Acting Assistant Administrator for  
Pesticides and Toxic Substances  
U.S. Environmental Protection Agency  
Waterside Mall  
401 "M" Street, S.W.  
Washington D.C. 20460

1. The U.S. Air Force Installation Restoration Program is involved with two major research activities at former Herbicide Orange storage sites. The purpose of this application/notification is to provide for the disposal of dioxin contaminated personnel protection and sampling equipment generated during various phases of our two programs. Presently, surface and subsurface sampling is being conducted at the Naval Construction Battalion Center (NCBC), Gulfport MS and Eglin AFB (EAFB), Fort Walton Beach FL, to determine the profile and extent of contamination. Follow on phases involve testing soil decontamination technologies at NCBC.

2. Application for approval is made for disposal of TCDD contaminated waste under provisions of 40CFR, Part 775, 190(b). Disposal will be accomplished prior to 15 Jul 85.

a. Name and address of firm: HQ Air Force Engineering & Services Center  
Engineering and Services Laboratory  
(HQ AFESC/RD)  
Tyndall AFB FL 32403 6001  
ID No. FL 1570024124

b. Site 1: Naval Construction Battalion Center, Gulfport MS, ID  
#MS2170022626.

Site 2: Eglin AFB, Fort Walton Beach FL, ID# FL572024366.

c. Point of Contact: Capt Terry L. Stoddart  
HQ AFESC/RDVW  
Tyndall AFB FL 32403  
(904) 283-2942

d. A review of current analytical data indicates the maximum levels of 2,3,7,8 TCDD contamination in soils from NCBC and EAFB is 300 ppb. The average concentration in these soils ranges from 20-30 ppb. Based on these data we anticipate that the drummed waste will contain substantially lower concentrations of 2,3,7,8 TCDD. The soil is also contaminated with varying concentrations of 2,4,D and 2,4,5T, ID Nos. D016 and D077, respectively.

d. Quantity of Waste: The first phase of soil sampling at NCBC resulted in the generation of 27 drums of contaminated clothing. The subsurface sampling scheduled for NCBC in early May will generate another 22 drums. Similar activities at EAFB are anticipated to generate 22 drums of contaminated clothing. The follow on phase of the project, which involves testing of soil decontamination technologies, scheduled for Jun 85, will generate approximately 54 drums of contaminated clothing. Currently, no technology demonstrations are scheduled for Eglin AFB FL. One of the technologies scheduled for demonstration at NCBC will produce 75 gallons of dioxin-contaminated solvent. The solvent, Solitrol®, is a petroleum product manufactured by Phillips. The solvent has a flashpoint of 185°F, pH 7, and a copper strip corrosion of 1.0. It is anticipated that the dioxin contamination in the solvent will be less than 100 ppb. A total of 127 drums are scheduled for disposal. The waste to be disposed consists of 125 drums of contaminated chemical protective equipment and two drums of contaminated solvents.

e. All waste will be packaged, labeled, and transported in accordance with existing EPA, DOT, and state regulations. Wastes will be disposed by incineration at Rollins Environmental Services, Inc., Deer Park TX, EPA ID No. TXD0551141378.

f. Status of Waste: The 27 fiber drums of contaminated wastes are stored in a open-sided metal storage shed at NCBC. The shed has a concrete floor. It is surrounded by a 6 foot high chain link fence topped with barbed wire. The drums are stacked one high on wood pallets and covered with 6-mil plastic sheeting. This storage facility is located inside the contaminated area, which is surrounded by a fence and posted as a restricted area. Waste presently generated will be stored in a similar manner until pickup for transportation to Rollins Environmental Services, Inc., which is scheduled for the last week of Jun 85.

3. Your time and effort for consideration of this approval request is appreciated. If questions should arise, please contact Capt Terry Stoddart, Headquarters Air Force Engineering and Services Center, Engineering and Services Laboratory (HQ AFESC/RDV), Tyndall AFB FL 32403-6001; (904) 283-2942.

*James R. Van Orman*

JAMES R. VAN ORMAN  
Deputy Director of  
Engineering & Services Laboratory

cc: EG&G Idaho (Mr. Williams)  
U.S. EPA, Region 4  
Mr. DesRosien, EPA/ORD  
Mr. Kleveno, EPA/HRSD  
Mr. Cummins, EPA/OSWER  
325CES/DEEV  
AD/DEV  
NCBC/Code 470

Int cc AFESC/DEV

WH-562B/H. Fribush/ht/S242K/475-6726/05-23-85/02/ht/26

MAY 31 1985

Mr. James R. Van Orman  
Deputy Director of  
Engineering & Services Laboratory  
Department of the Air Force  
Headquarters Air Force Engineering and Services Center  
Tyndall Air Force Base, FL 32403


Dear Mr. Van Orman:

We have reviewed your letter of April 25, 1985, informing the Environmental Protection Agency (EPA) of your intent to dispose of waste materials contaminated with 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). We understand that you have about 125 drums of 2,3,7,8-TCDD-contaminated clothing and two drums of 2,3,7,8-TCDD-contaminated solvent. We also understand that you wish to dispose of this waste by incineration at Rollins Environmental Services, Deer Park, Texas.

The Agency's Dioxin Disposal Advisory Group (DDAG) has no objections to your planned disposal. We recommend that the incinerator be operated under the conditions that have demonstrated a destruction and removal efficiency (DRE) of 99.9999 percent for PCBs.

If you are unable to proceed with the planned disposal or if you choose an alternative method, please be advised that you are required to submit a new notification prior to disposing of 2,3,7,8-TCDD-contaminated waste materials. It should be noted that, after July 15, 1985, the effective date of the RCRA listing regulation (50 FR 1978-2006; January 14, 1985), which designates certain 2,3,7,8-TCDD-contaminated wastes as hazardous, you will be subject to the provisions of that rule. If you have any questions, please feel free to contact Dr. Howard Fribush, Office of Solid Waste, on (202) 475-6726.

Sincerely,

  
M. Jack W. McGraw  
Acting Assistant Administrator

APPENDIX C

PUBLIC NOTIFICATION AND LOCAL NEWS ARTICLES ON TECHNOLOGY,  
RESEARCH, AND TEST EVALUATION PROGRAM AT NCBC

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*Gulfport*  
*The Daily Herald*  
*FRI. May 24, 1985*

**PUBLIC NOTIFICATION**

Notice is hereby given of the public availability of a Toxic Substance Control Act (TOSCA) document titled "Environmental Restoration Technologies: Research Test and Evaluation" covering work to be conducted by the United States Air Force, at the Naval Construction Battalion Center, Gulfport, Mississippi. The document, available for review at the Gulfport-Harrison County Library, 21st Avenue, Gulfport, Mississippi, covers the scope, procedures, background and goals of research to be conducted by the Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall AFB, Florida. The research is aimed at discovering the most efficient, cost-effective method(s) of removing environmental contaminants from soil. For questions beyond the scope of this document, please contact the Directorate of Public Affairs, Air Force Engineering and Services Center, Tyndall AFB, Florida 32403, Telephone: (904) 232-6076, V-76,adv.23,4t.

# Permits for soil tests not needed

By TOM CHARLIER  
STAFF WRITER

The U.S. Air Force will not need Mississippi environmental permits to carry out testing next week on dioxin-contaminated soil at the Gulfport Seabee Center, state officials said.

The Pollution Control Permit Board, which was briefed Tuesday on work to be done at the base, will not require the Air Force, or its two contractors in the work, to undergo the normal permitting procedure, said Jack McMillan, who heads the Bureau of Pollution Control's solid-waste section.

The two companies selected to do the testing — J.M. Huber Co. of Atlanta, and the IT Corp. of Washington, D.C. — have begun delivering equipment to the base in preparation for the work, which is scheduled to take place June 5. The contractors will test experimental methods involving the use of heat and chemicals to remove dioxin from soil.

"Right now, we don't see any reason for issuing a permit, because it's just such a minute amount (of dioxin) they'll be dealing with," McMillan said.

The state, however, will require permits before any full-scale cleanup effort begins at the site, he added.

Sgt. Jim Denny, a spokesman for the Engineering and Services Laboratory at Tyndall Air Force Base, Fla., said the Air Force has provided the Bureau of Pollution Control with complete details of the work, which he said will not

See AGENT, Page A-2

1-7W  
502  
5/29/85

## Agent

Continued from Page A-11

pose any environmental threat. "Everything we're going to be doing is perfectly OK with them," he said.

The soil to be tested is on a sealed-off 12-acre portion of the base where thousands of drums of Agent Orange, a herbicide used to defoliate jungles during the Vietnam War, were stored from 1968 to 1977. The herbicide

contained dioxin, a contaminant produced during its manufacture, which remains in soil at the site as a result of leaks in the drums.

Dioxin has been shown to be extremely toxic in laboratory studies, with doses of as little as 5 parts per trillion producing cancerous tumors in test animals, according to some researchers. But the substance's effects on humans are not fully understood.

In soil samples taken at the Seabee Center, dioxin has been detected in concentrations of up to 200 to 300 parts per billion. According to the Centers for Dis-

ease Control in Atlanta, a concentration of 1 part per billion in soil is sufficient to warrant concern.

The testing is part of a \$1.7-million research and development program from which the Air Force will devise a plan for the cleanup of three dioxin-contaminated sites — the Seabee Center, Eglin Air Force Base, Fla., and Johnston Island, located west of Hawaii in the Pacific Ocean.

The cleanup, designed to make the sites suitable for normal use, probably will not begin until late this year or early next year, Denny said.

Daily Herald 5/29/85

## Air Force's dioxin tests won't require state permits

By TOM CHARLIER  
Staff Writer

The U.S. Air Force will not need Mississippi environmental permits to carry out testing next week on dioxin-contaminated soil at the Gulfport Seabee Center, state officials said.

The Pollution Control Permit Board, which was briefed Tuesday on work to be done at the base, will not require the Air Force, or its two contractors in the work, to undergo the normal permitting procedure, said Jack McMillan, who heads the Bureau of Pollution Control's solid-waste section.

The two companies selected to do the testing — J. M. Huber Co. of Atlanta, and the IT Corp. of Washington, D.C. — have begun delivering equipment to the base in preparation for the work, which is scheduled to take place June 5. The contractors will test experimental methods involving the use of heat and chemicals to remove dioxin from soil.

"Right now, we don't see any reason for issuing a permit, because it's just such a minute amount (of dioxin) they'll be dealing with," McMillan said.

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The Sun 6/6/85

# Workers begin decontaminating dioxin-tainted soil at Seabee

By TOM CHARLIER  
STAFF WRITER

Technicians on Wednesday began processing dioxin-tainted soil at the Seabee Center in Gulfport in work that could lead to an eventual cleanup of a site where the herbicide Agent Orange was stored.

Specially-suited workers from IT Corp. of Knoxville, Tenn., were scheduled late last night to begin treating soil by an untested device designed to separate and destroy dioxin through the use of heat, infrared and ultraviolet light. The work had been rescheduled from afternoon to nighttime because of the heat.

Later this month, crews from another firm, J.M. Huber Co. of Borger, Texas, are slated to demonstrate an experimental process using a device known as an advanced electrical reactor. That process destroys dioxin with extremely high temperatures.

Both firms are under contract with the U.S. Air Force to process a total of 1,500 pounds of contaminated soil at the base. The work is part of a research effort headed by the Air Force, the U.S. Environmental Protection Agency and the U.S. Department of Energy to identify a "useful technology" to treat contaminated soil at three military installations — the Gulfport base, Eglin Air Force Base, Fla., and Johnston Island, in the Western Pacific Ocean.

With the tests, "we'll be able to determine whether or not it will be economically feasible to clean up the site," Air Force Maj. Jim Heaberg said.

It now costs between \$500 and \$1,000 a cubic yard to dispose of contaminated soil at high-tech incinerators throughout the country. Decontaminating the soil may offer a less-expensive alternative, the Air Force officials said.

IT Corp. of Knoxville will conduct work at Johnston Island, will monitor about 100,000 gallons of Agent Orange stored at the base. The work is expected to be completed by July 15.

"About 80 percent of the \$2.7 million earmarked for the research effort — which includes soil sampling and other monitoring already done at the three bases — will be spent in Gulfport. The higher costs are attributed to the problems posed by the unique soil conditions there, officials said.

"We're putting our money against the most difficult problem," said Wayne R. Mathis, an EPA engineer.

Heaberg said the surface of the storage site is composed of a mixture of asphalt, pea gravel and crushed shell.

Agent Orange, used to defoliate jungles during the Vietnam War, was stored at a 12-acre site on the Seabee Center between 1968 and 1977. The more than 540,000 gallons kept at the base in later years were among the 2.4 million gallons of the herbicide destroyed on a waste-burning site in the Pacific.

Dioxin, a byproduct of the manufacture of Agent Orange, has been shown to be extremely toxic in tests on laboratory animals. Soil samples at the former storage area have revealed dioxin concentrations of as high as 200 parts per billion — well above widely established safe levels.

The storage area is sealed off, and state and federal officials contend no significant contamination has been found off the site.

The testing at the Seabee Center must be completed by July 15. EPA and the Mississippi Bureau of Pollution Control have waived the usual hazardous-waste permit requirements for the testing until that date.

THE SUN 6-6-85



APPENDIX D

TEST PLAN FOR HUBER'S AER DEMONSTRATION TEST AT NCBC

TEST PLAN TO DEMONSTRATE HUBER'S  
ADVANCED ELECTRIC REACTOR ON  
DIOXIN CONTAMINATED SOILS AT  
GULFPORT, MISSISSIPPI



# HUBER TECHNOLOGY

J. M. HUBER CORPORATION

P. O. Box 2831

Borger, Texas 79008-2831

(806) 274-6331

TEST PLAN TO DEMONSTRATE HUBER'S  
ADVANCED ELECTRIC REACTOR ON  
DIOXIN CONTAMINATED SOILS AT  
GULFPORT, MISSISSIPPI

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## 1. INTRODUCTION

This test plan provides a detailed description of Huber's plans to conduct a test for the research and test evaluation of the Advanced Electric Reactor (AER) to treat soil contaminated with Herbicide Orange at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi. This test will be conducted under contract to EG&G Idaho, Inc., Idaho Falls, Idaho.

Huber will transport its mobile 3" AER to Gulfport, Mississippi, and process approximately 1,000 pounds of soil containing the toxic 2,3,7,8 tetrachlorodibenzo-p-dioxin (TCDD) at NCBC. The actual test will require about 30 to 40 hours of operation and be performed on June 24 and 25, 1985.

In September 1983, Huber embarked on an aggressive program to demonstrate the capability of the AER to decontaminate soil. This program includes the treatment of soils contaminated with HCB, PCB, carbon tetrachloride, octochlorodibenzo-p-dioxin, and TCDD. The Gulfport test represents a continuation of this demonstration process.

All of the tests conducted to date have been successful in destroying the contaminate being tested while protecting the environment.

These results clearly demonstrate the extremely high destruction capabilities of Huber's AER process and its intrinsic safety advantages over conventional treatment methods.

## 2. PROCESS DESCRIPTION

Huber's hazardous waste treatment process is based on the Advanced Electric Reactor (AER) shown in Figure 1.

The reactor employs a new technology to rapidly heat materials to temperatures in the range of 4000°F using intense thermal radiation in the near infrared region. The reactants, which can be gaseous, liquid, or solid form, are isolated from the reactor core walls by means of a gaseous blanket formed by flowing nitrogen radially inward through the porous core walls. Carbon electrodes are heated and in turn heat the reactor core to incandescent so that the heat transfer is accomplished by thermal radiative coupling from the core to the feed materials. The only feed streams to the reactor are the solid, liquid, or gaseous wastes and the blanket gas--nitrogen.

Destruction is accomplished by pyrolysis rather than oxidation; therefore, typical products and by-products produced by incineration such as carbon monoxide, carbon dioxide, and oxides of nitrogen are not formed in significant concentrations. The principal products of soil borne PCB destruction using the Huber process are hydrogen, chlorine, HCl, elemental carbon, and a granular, free-flowing, solid derived material.

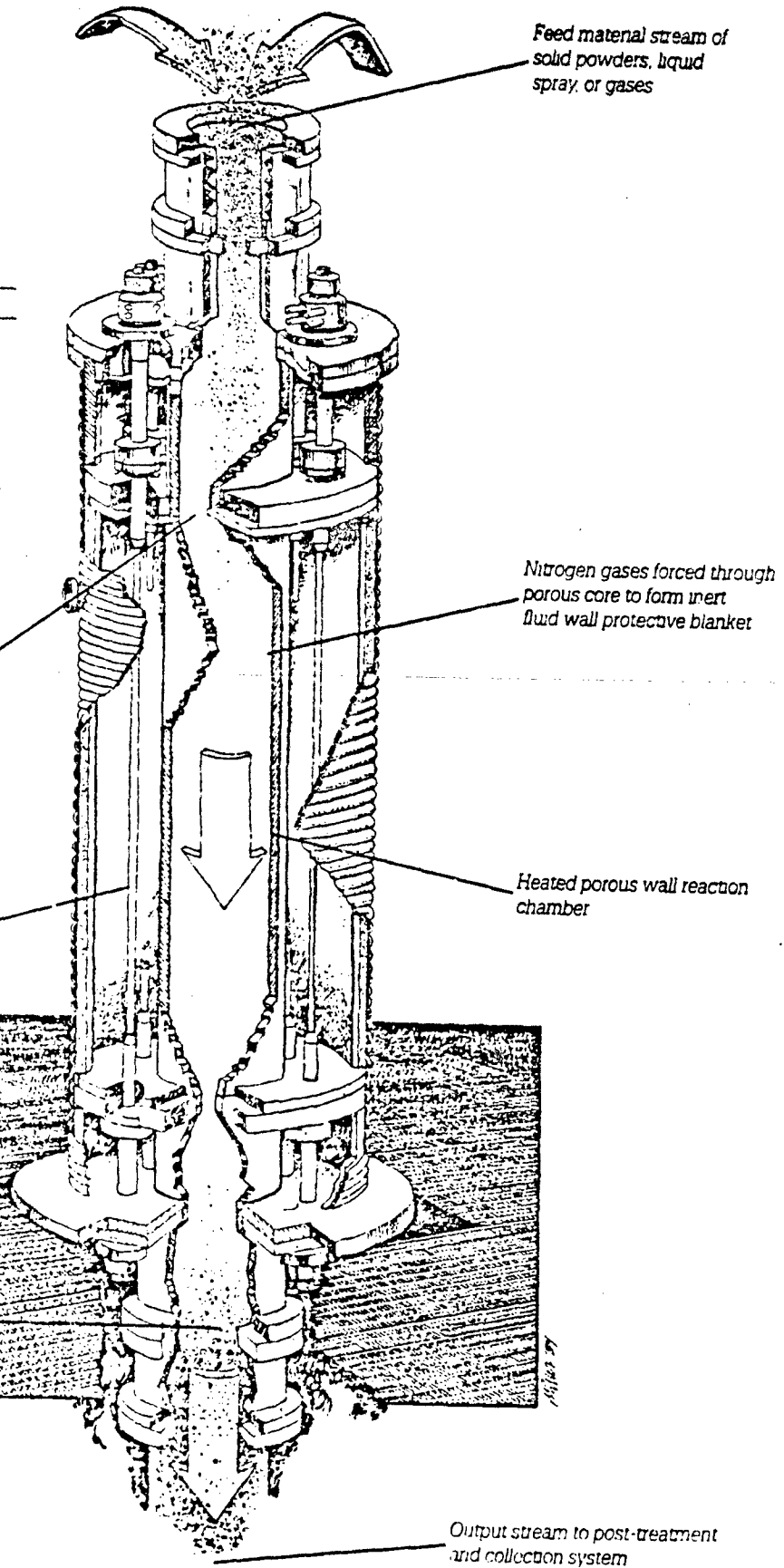
Figure 2 is a simplified process diagram of the HTG process as configured for solid hazardous waste destruction. The solid feed stream is introduced at the top of the reactor by means of a



FIGURE 1

**AER OPERATION**

The AER can process solid, liquid, or gaseous feed streams at temperatures approaching 5000°F. In this environment, most known solids sublime or melt, and chemical reactions occur at rates thousands of times faster than in conventional reactors. Feedstock solids must be in the form of free-flowing powders and liquids as discrete droplets.



Feed stream heated by thermal radiation from reaction chamber walls

Feed material stream of solid powders, liquid spray, or gases

Nitrogen gases forced through porous core to form inert fluid wall protective blanket

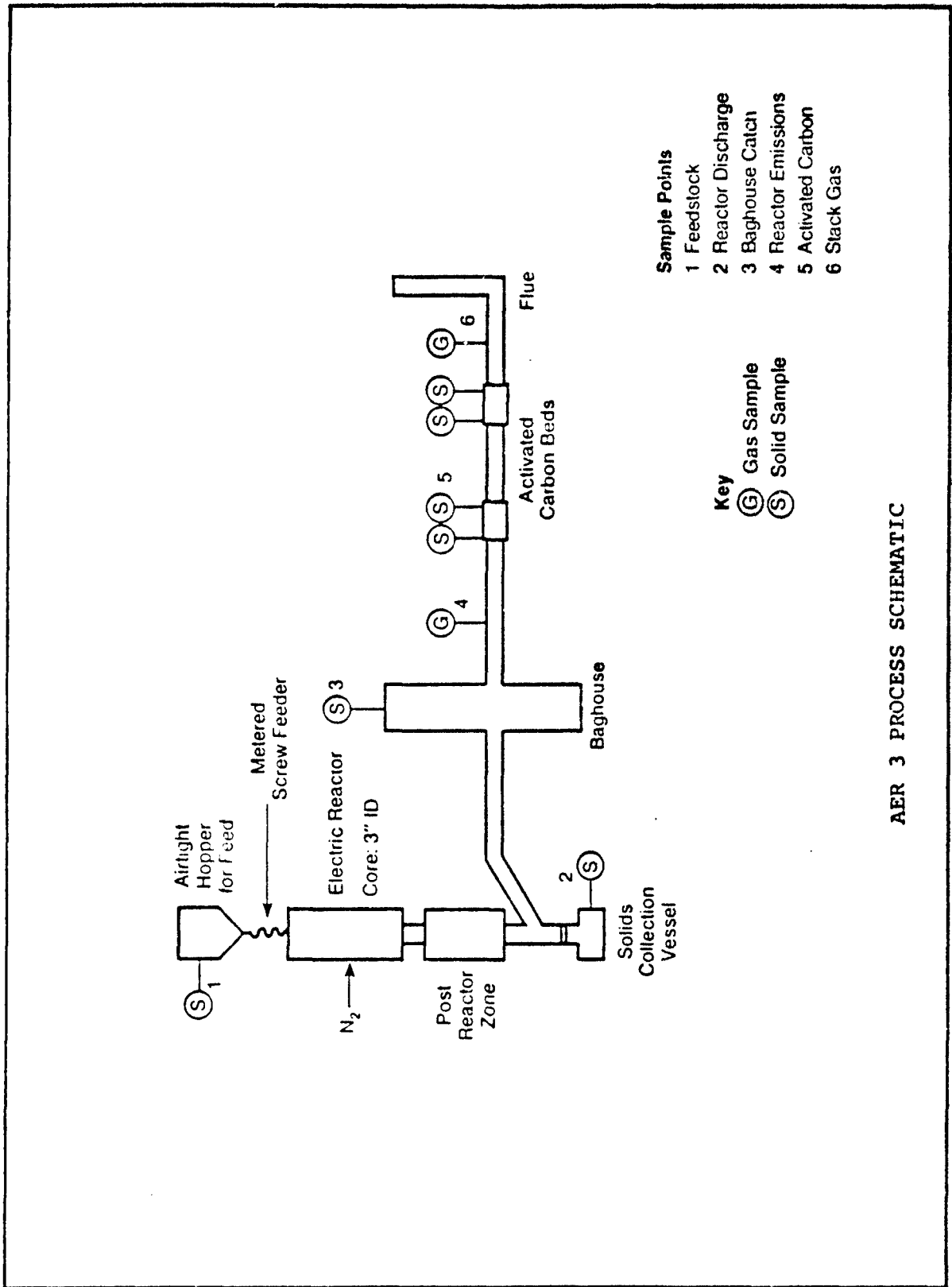
Heated porous wall reaction chamber

Electrodes radiatively heat reaction chamber walls

Treated material cools via radiation loss to unheated reactor and duct work walls

Output stream to post-treatment and collection system

FIGURE 2



metered screw feeder connecting the airtight feed hopper to the reactor. Nitrogen is introduced primarily at two points in the reactor annulus. Prior to entering the reactor, the nitrogen is preheated up to 1000°F using an electric circulation heater.

The solid feed passes through the reactor where pyrolysis occurs at temperatures between 3500° and 4500°F. After leaving the reactor, the product gas and waste solids pass through a post-reactor treatment zone (PRTZ).

The PRTZ provides for additional residence time but primarily cools the gas to less than 1000°F prior to downstream particulate cleanup.

Solids exiting the PRTZ are collected in a solids collection vessel which is sealed to the atmosphere. Any additional solids in the product gas are removed as the gases enter a bag house. Any residual organics and chlorine are removed by passing the product gas through activated carbon beds just upstream of the emission stack. The extremely small amount of process gas in the Huber system (150 scfm for a 25,000 ton per year plant) makes it economical to use absolute post reactor gas cleaning as is provided by the activated carbon beds. The organic, particulate, and chlorine free product gas composed almost entirely of nitrogen (some moisture) is then emitted to the atmosphere through the process stack.

For the Gulfport test, Huber will use the Toxbuster unit which is the same AER that was used for the on-site demonstration in Times Beach, Missouri. A schematic of the Toxbuster trailer is shown in Figures 3 and 4.

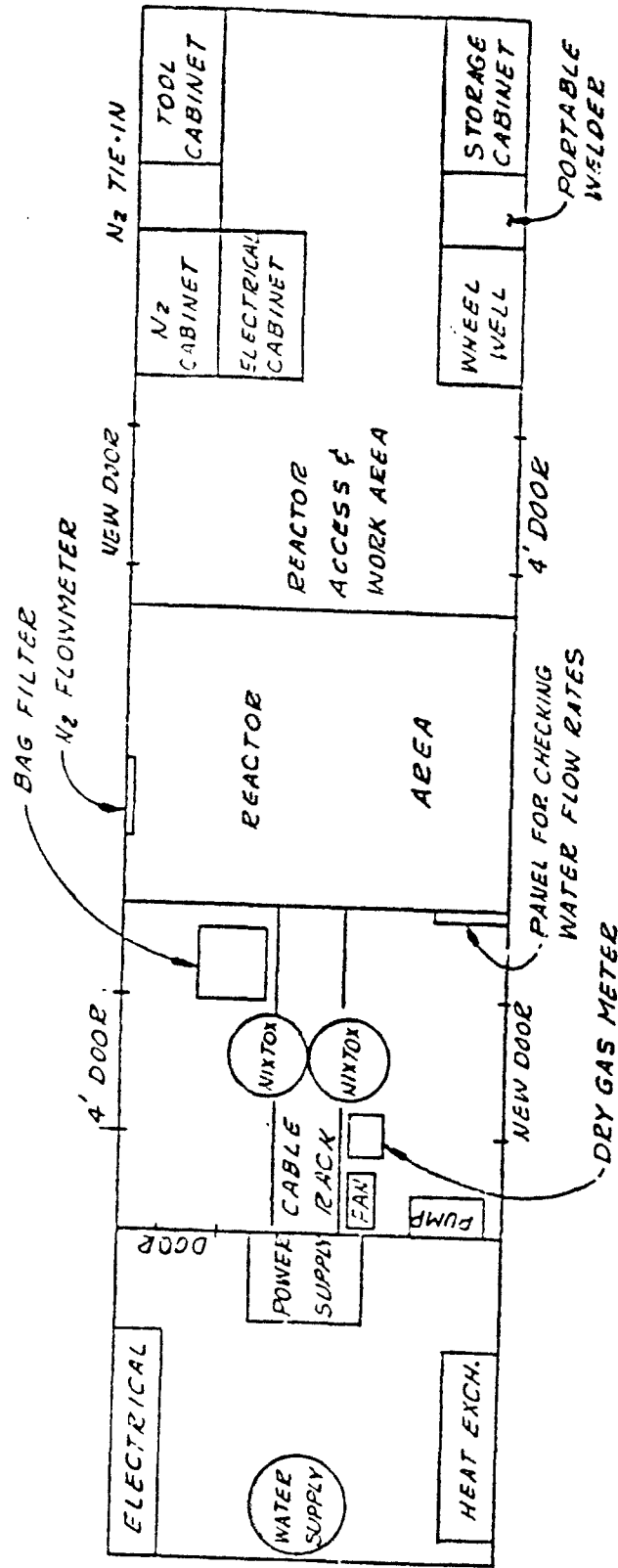
This unit, shown in Photographs 1, 2, 3, and 4, is Huber's smallest reactor and has an inside core diameter of three inches and a heated length of approximately three feet. This unit is installed in a covered truck/trailer to provide mobility and is used for proof-of-concept experiments and on-site demonstrations such as the Gulfport test.

#### SHUTDOWN PROCEDURES

Normal and emergency shutdown of the AER process is relatively simple. Normal and emergency shutdown consists of the following:

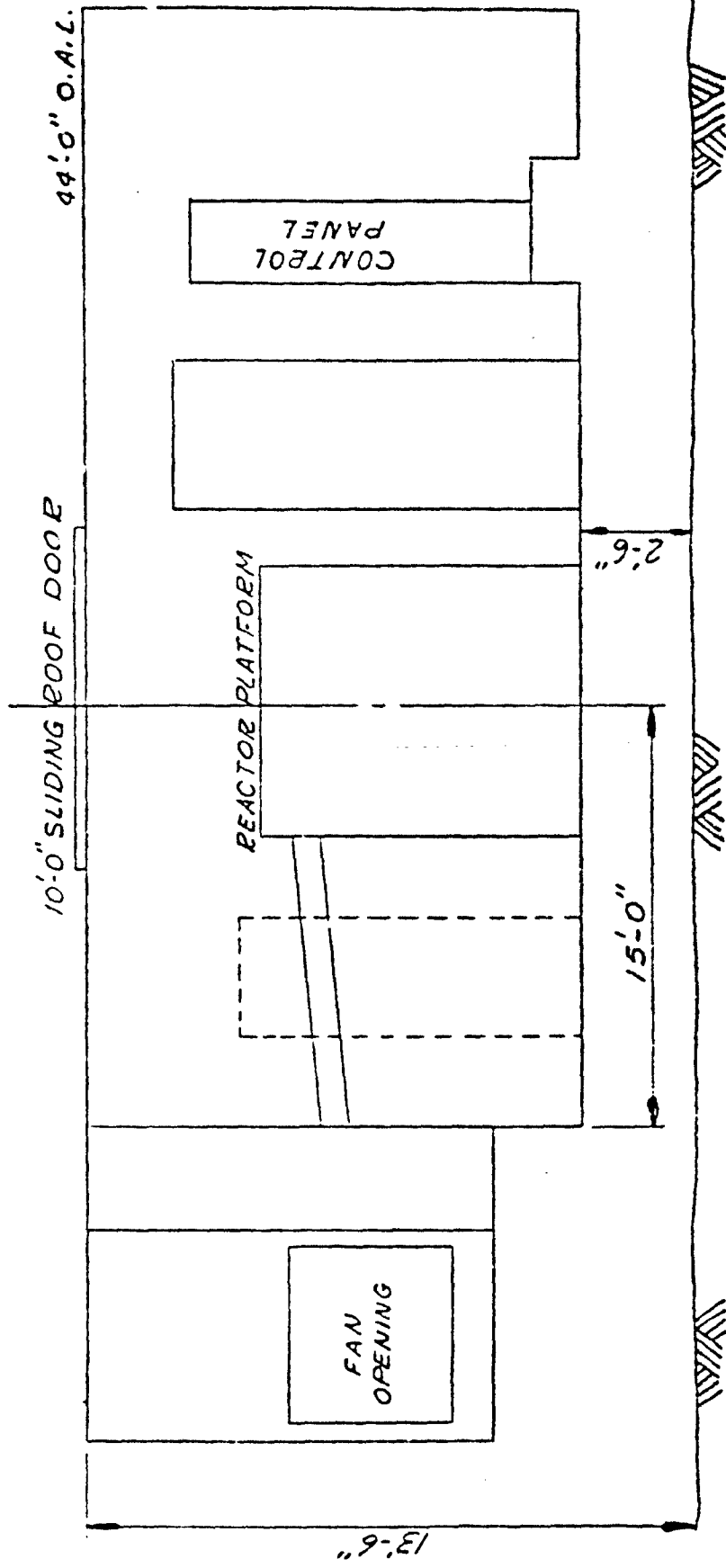
1. Turn off power to screw feeder.
2. Visually verify that no material is being fed to the reactor.
3. Close valve which isolates the screw feeder from the reactor.
4. Remove feed tube from reactor and place in a plastic bag (using Level C2 personnel protection).

# AER 3" ELECTRIC REACTOR

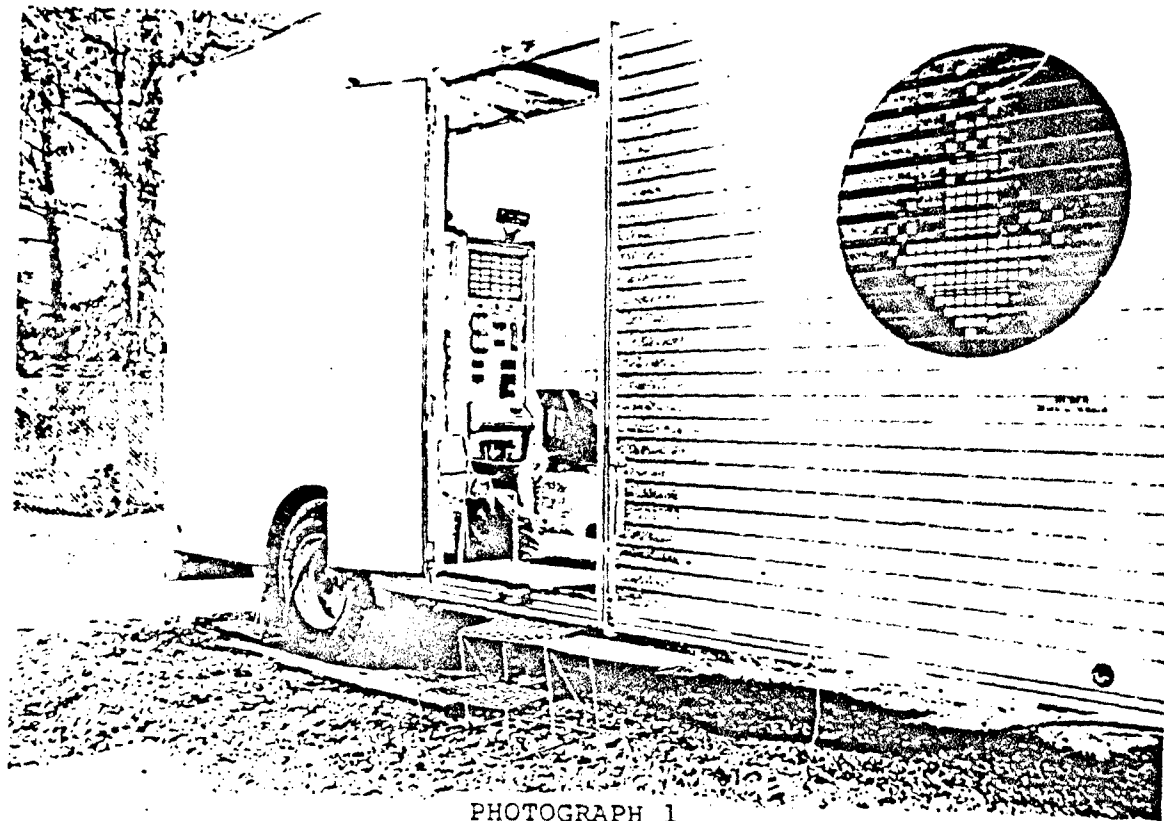


TOP VIEW  
EQUIPMENT LAYOUT

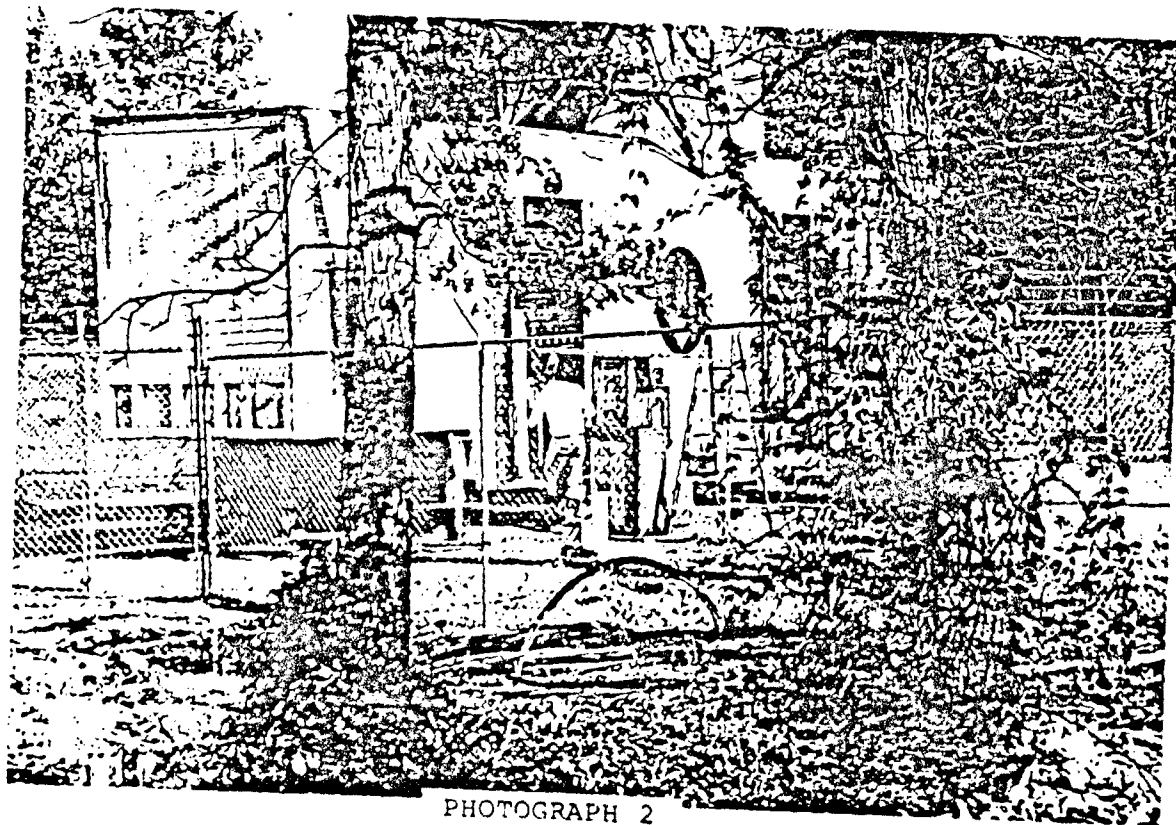
AER 3" ELECTRIC REACTOR



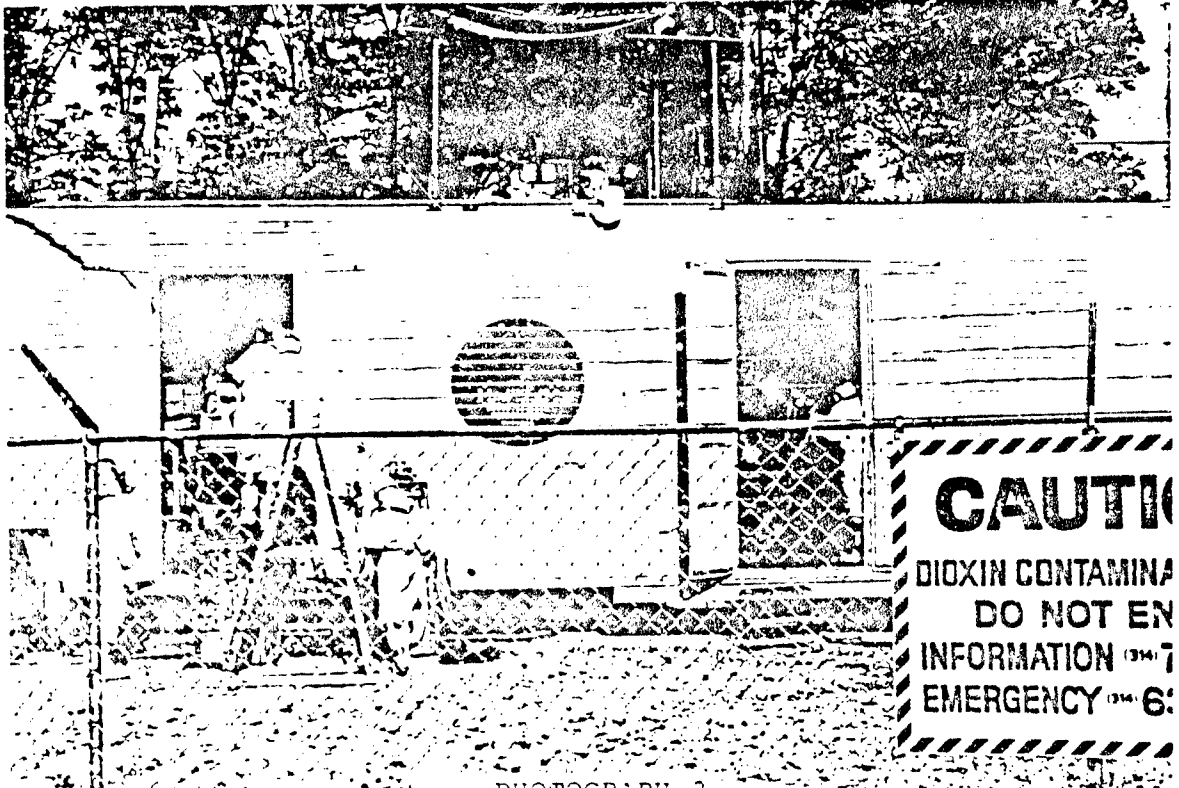
ELEVATION



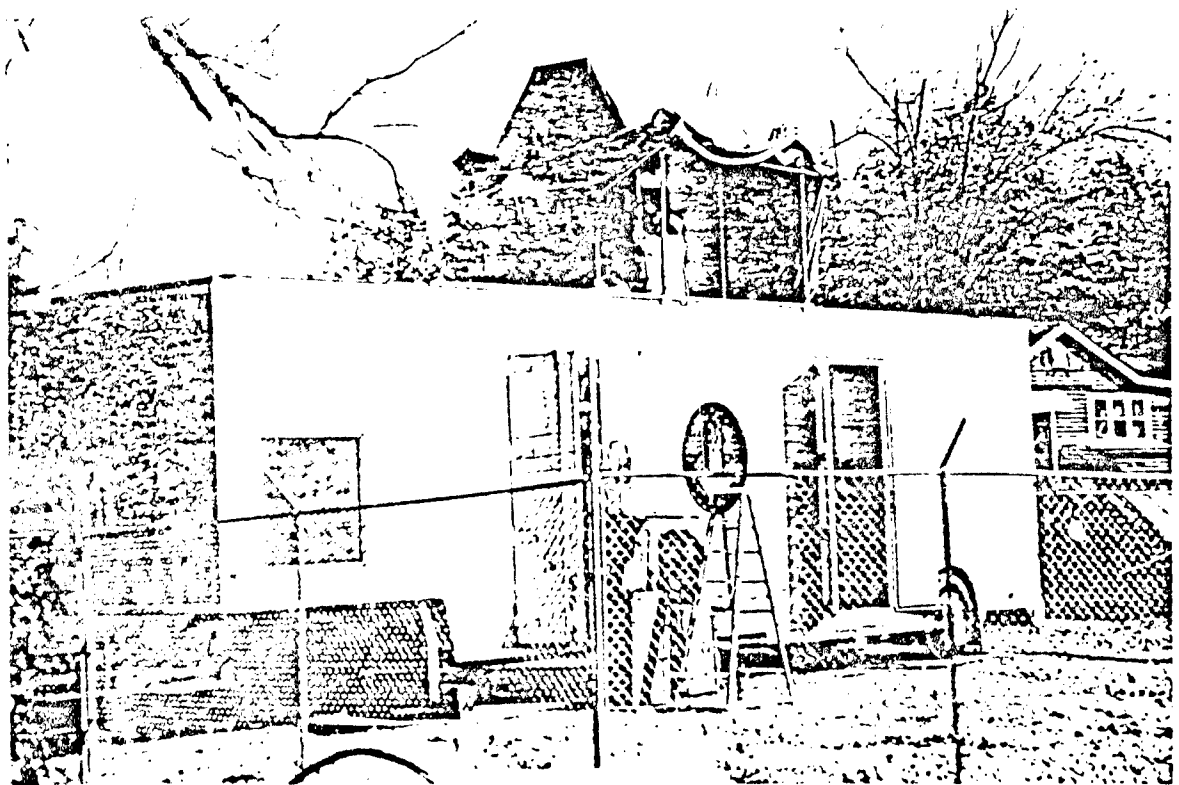
PHOTOGRAPH 1



PHOTOGRAPH 2



PHOTOGRAPH 3



PHOTOGRAPH 4



5. Turn off power to reactor.

The nitrogen purge remains on the reactor until the reactor temperature is about 200°F (normally this will take about three to four hours). After the reactor is cooled to less than 200°F, the nitrogen flow is stopped.

### 3. ACTIVITY SCHEDULE

The current schedule is based upon the Gulfport test being performed in June 1985. Figure 5 highlights the planned activities during June.

During the week of June 10, the Toxbuster will be packed in anticipation of transportation to Gulfport later in that week. Upon arrival at NCBC, the reactor will be located on Lot 42 near the electrical service pole which was installed to provide 480-volt, three-phase service to the Toxbuster. Because purified nitrogen is a necessary part of the AER process, a cryogenic nitrogen trailer will be placed adjacent to the AER. The locations of these units on Lot 42 are shown in Figure 6.

On June 17, the Toxbuster trailer will be leveled, power will be connected, and minor reactor assembly will be performed. Four Huber personnel will be on site to perform these tasks.

On June 18, the feed system will be installed and nitrogen will be hooked up to the trailer in anticipation of a shakedown test on June 19.

During the shakedown test, the Toxbuster will be brought up to approximately 4100°F to identify any problems that may have resulted from transportation of the system. If any repairs are required, they will be performed on June 20 along with other

JUNE 198

SUN	MON	TUE	WED	THUR	FRI	SAT	SUN
23	24	25	26	27	28	29	
• Set Feed Bin in Place 30	• Begin Gulfport Demo (0800) ↓ 5 or 6 8-Hour Shifts ↓	• ~ 18:00 to 24:00 Test Complete	• Begin Decontamination Procedures	• Equipment Disassembly and Loading	• Test Prep. • Pretest Meeting	• Equipment Arrives in Gulfport	
16	17	18	19	20	21	22	
	• Level Rx • Connect Power • Unpack • Rx Assembly	• Feed Sys. Setup • Hookup Nitrogen	• Shakedown Test	• Minor Repair and Test Prep.			
9	10	11	12	13	14	15	
	• Field Coordinator Arrives Gulfport						
2	3	4	5	6	7	8	

SUN MON TUE WED THUR FRI SAT SUN

May 198

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July 1985

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September 1985

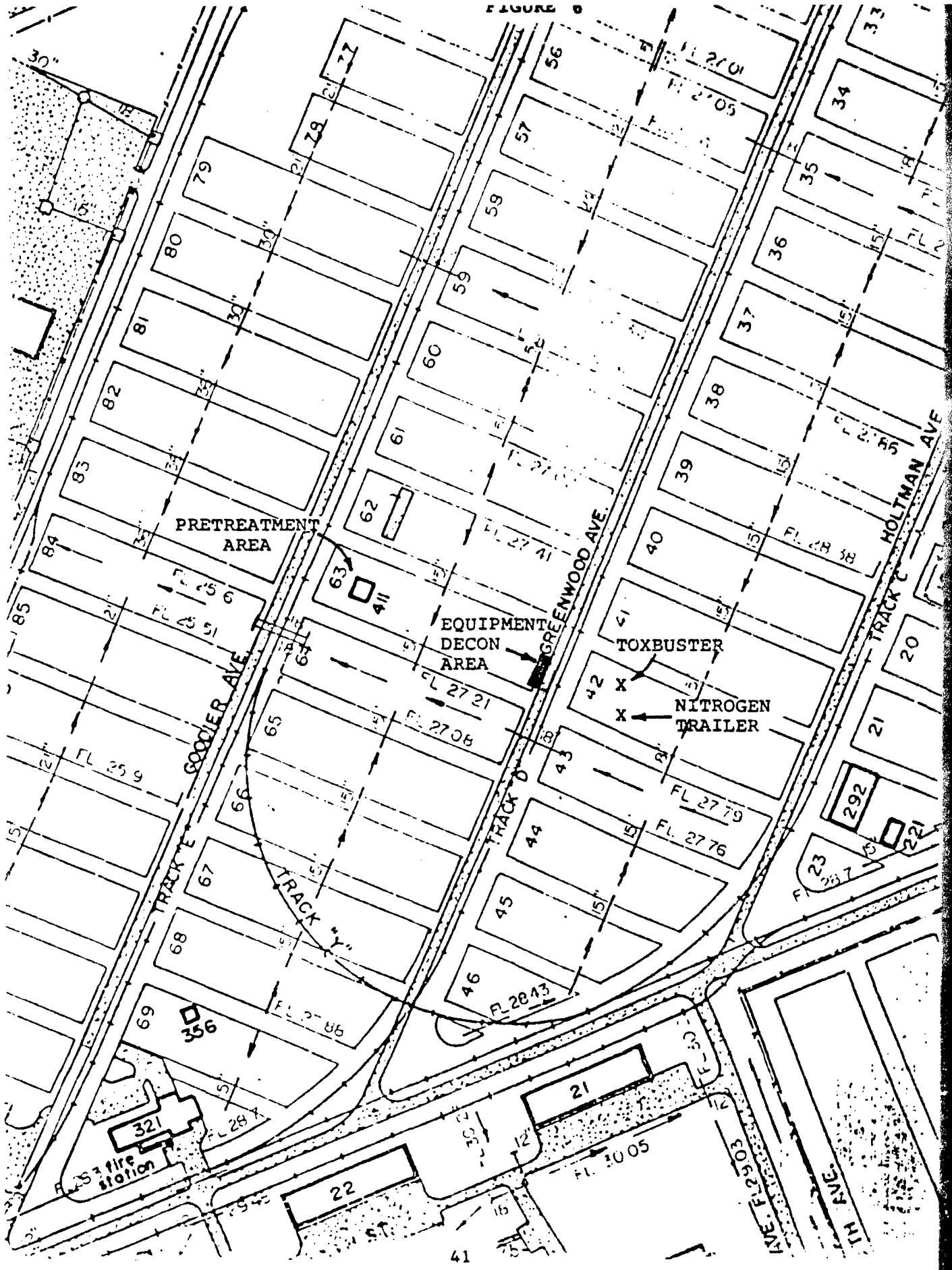
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October 1985

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PROPOSED GULFPORT DEMONSTRATION TEST PLAN

FIGURE 6



remaining test preparations. The remainder of the demonstration personnel will arrive at Gulfport on June 20.

On June 21, final test preparations will be performed. In addition, an overall pretest meeting will be held with all test participants. The agenda for this pretest meeting is shown in Table 1.

The coordination meeting is a necessary function used to ensure that all participants in the test are fully aware of their responsibilities. As shown on the agenda, this will include a brief review of the emergency procedures, health and safety procedures, and run plan. A checklist will also be given to each participant so that he can verify that all of his duties will be performed at the proper time.

Preparation of Gulfport soil to be used in the AER test is being done as a lower tier subcontract by IT Corporation. By June 22, IT Corporation will have the AER feed bin loaded with approximately 1,000 pounds of Gulfport soil that is free-flowing (about 1% moisture content) and -35 mesh.

On either June 22 or June 23, four individuals will be required to transport the filled feed bin to the AER for installation above the reactor. Prior to transporting the feed bin from the contaminated area, its exterior will be decontaminated. Transportation will require the use of the following personnel:

**TABLE 1**

**Agenda For Pretest Meeting**

1. Introduction and Overview
2. Review of Run Plan
3. Specific Participant Responsibilities
4. Health and Safety Review
5. Emergencies and Contingency Plans

1. A crane driver.
2. An individual to drive a pickup truck in which the feed bin will be transported.
3. Two operators in Level C1 protection equipment to install the feed bin on the reactor.

After the feed bin has been set in place above the reactor, the Toxbuster will be off limits (caution tape will be placed on all entrances) to individuals not wearing the following protection equipment (defined as Level C2 protection):

1. Standard safety equipment (i.e., hard hat, safety shoes, safety glasses).
2. Ventilated Tyvek coveralls.
3. Cotton gloves.
4. Half-face masks with organic, vapor, and Hepa filter cartridges.

The actual test will begin at 8 a.m. on June 24. Approximately three hours before the test is to begin, the lead operator and process engineer from Team 3 will bring the Toxbuster to operating conditions. The test is scheduled for completion during the afternoon or evening of June 25. Under normal circumstances, the

test should require about 30 to 40 hours to complete; however, unforeseen problems may cause the length of the test to be extended.

Decontamination procedures will begin on June 26. Towards the end of the same day, IT Corporation will take wipe samples for analytical verification that no dioxin is present on any of the AER equipment.

Verification of these tests is expected on Thursday, June 27, for final disassembly and loading of the Toxbuster equipment for its return to Borger on either June 28 or June 29.



#### 4. DETAILED RUN PLAN

Attachment 1 is a detailed run plan for the proposed test.

In the event of inclement weather or an emergency, operation of the Toxbuster will cease after emergency shutdown procedures (described in Section 2) are performed. The decision to stop operation of the Toxbuster will be made by the Field Coordinator.

If operation of the Toxbuster is stopped for any reason (i.e., weather problems, equipment breakdown, etc.), the continuation of the test will be determined by the EG&G on-site Project Director.

Attachment 2 summarizes the test conditions. As shown, approximately 1,000 pounds of soil contaminated with Herbicide Orange will be processed. Huber expects between 100-200 parts per billion of 2,3,7,8-tetrachlorodibenzo-dioxin (TCDD) in the feedstock. The concentration of TCDD and other products in the feedstock will be determined by the verification subcontractor.

For the proposed test, the following run conditions will be maintained:

<u>ITEM</u>	<u>VALUE</u>
Reactor Core Temperature °F	3500-4500
Nitrogen Rate, scfm	6-10
Feedstock Charge, Lbs	1,000
Feed Rate, Lb/Min	0.4-0.6
Estimated Bag Filter Catch, Lbs	10

## 5. PROJECT STAFFING

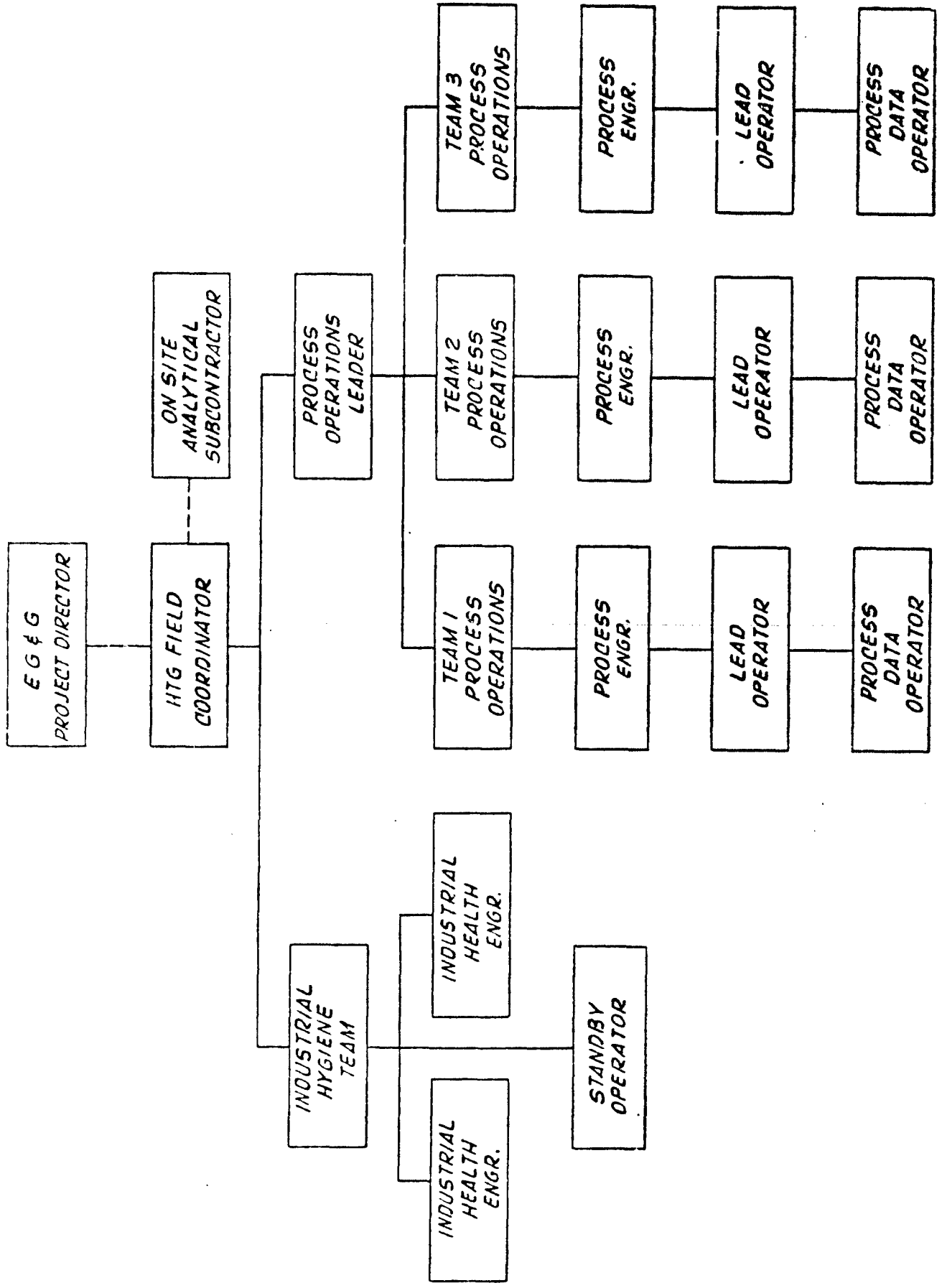
Figure 7 is a field team organizational chart for the personnel involved in the Gulfport test. As shown, this project organization requires six professionals and seven hourly personnel and consists of two groups under the general direction of the field coordinator.

The process operations team, under the direction of the process operations leader, is responsible for the operation of the AER and recording of all process operating parameters.

The industrial hygiene team is responsible for monitoring the activities of the process operations team and ensuring its safe operation.

Because the test is to be performed around the clock, sufficient manpower is required for shift work. The industrial health engineers will work 12-hour shifts during the test.

Attachment 3 is a detailed description of the activities to be performed by all participants during the test.



FIELD TEAM ORGANIZATIONAL CHART

## 6. FEEDSTOCK DESCRIPTION AND PREPARATION

For proper operation, the AER requires a solid feedstock that has been properly sized and dried. Specifically, this requires solids with a particle distribution near 35 mesh and a moisture content of approximately 1% to ensure a free-flowing product.

IT Corporation has been retained as a lower tier subcontractor to Huber to perform feedstock preparation. Current plans require that IT Corporation complete all feedstock preparations by June 22 and load Huber's feed bin with approximately 1,000 pounds of a free-flowing, -35 mesh mixture of Herbicide Orange contaminated Gulfport soil.

Huber anticipates the following mixture will make up the 1,000 pounds of feedstock provided on June 22.

<u>DESCRIPTION</u>	<u>CONCENTRATION</u>
Concrete	21%
Sand/Gravel	45%
Shell	31%
Asphalt	1%
Tar	2%

## 7. DATA SAMPLING

During the test, Huber will be gathering information that will describe the operating parameters of the AER process. All sampling and analytical tests will be performed by the verification subcontractor. Huber anticipates a visit (in Borger, Texas) from the verification subcontractor to work out specific equipment needed for performing sampling in Gulfport. Huber will coordinate with the verification subcontractor for obtaining samples during the test. Huber anticipates this will include both solid and gaseous samples.

Attachment 4 is an example of the data sheets to be used by Huber personnel to record process data during the test.

The information obtained from these data sheets will be used along with the analytical data to establish overall destruction efficiencies. The information that will be documented as a result of using these forms includes the following:

- Total Amount of Soil Treated
- Instantaneous Soil Feed Rates
- Reactor Core Temperatures (via optical pyrometer)
- PPTZ Temperature (via thermocouple)

- Reactor Volts and Amps
- Nitrogen Flow Rates (using a rotameter)
- Reactor Power Requirement
- Reactor Pressures
- Total Gas Flow Rate Exiting Process (dry gas meter)

## 8. EQUIPMENT CHECK AND CALIBRATION

Several deliberate measures are used during the operation of the Toxbuster to maintain proper operation. As described in the previous section, several data forms have been used in previous tests for recording process operations parameters. These records were used to record the information in the Times Beach tests and were quite successful. Similar forms will be used during the NCBC tests.

Prior to the NCBC test, the entire AER process flow train will be leak tested using a soap solution. This test is used to verify that all connections are secure and that the entire system is sealed. The following equipment will also be calibrated according to standard calibration procedures:

- Rotameters (using Dry Gas Flow Meter)
- Optical Pyrometer (using portable pyrometer)
- Screw Feeder (by weight loss mechanism and batch testing using a clean surrogate at various settings as a function of time)
- Other Misc. Gauges

Redundancy is built into the AER design and data gathering to ensure proper AER operation. Proper operation of the reactor is



verified by (1) optical pyrometer readings; (2) reactor power consumption; (3) volt and amp readings; and (4) temperature of the treated material. In addition, the nitrogen flow rate is metered at two separate points within the process. The overall nitrogen flow is metered using a dry gas meter as it enters the trailer, and rotameters are used to regulate individual flows.

The flow of soil into the reactor is set by a metered screw feeder which is calibrated prior to all tests. Instantaneous soil feed rates are verified by an in-line weigh scale.

To ensure that all process operating parameters are recorded properly, redundancy is also built into the process data gathering. All process parameter readings are taken by two separate test participants. This procedure is used to guard against deficiencies in the recording of data which serves as a permanent record of the test. In the preparation of the final report, the Project Director (and Operating Team) will review the process operating data and present it in the summary of the test. Additionally, all exceptions to this Test Plan will be identified and discussed.

## 9. DECONTAMINATION

Decontamination procedures following the test are outlined in detail in the Industrial Health Manual and will only be described briefly.

Decontamination of the Toxbuster and associated equipment will be performed on June 26 and 27 according to the present run plan.

Upon completion of the test on June 25, the following feed equipment will be removed by personnel in Level C1 protection (a description of Level C1 personnel protection is included in Huber's Industrial Health Manual) and placed in the decontamination area (located inside the contaminated area):

- Feed Tube
- Feeder
- Feed Bin

This equipment will be decontaminated on either June 25 or 26, depending on how late in the day the test ends. All decontamination will be performed in Level C1 personnel protection. Decontamination will be performed just inside the contaminated zone using either steam cleaning or washing with a trisodium phosphate water solution. After cleaning, the equipment will be placed on plastic (in a buffer zone between the contaminated and

clean zone) and wipe sampled for decontamination verification. Until the results of the wipe sample are available, the equipment will remain wrapped in plastic in the buffer zone.

After the Toxbuster has been allowed to cool to ambient temperatures, the treated soil collection vessel will be unhooked from the AER process flow train, sampled by the verification subcontractor, sealed, weighed, and placed in the contaminated zone (or buffer zone). Removal of the collection vessel will be performed in Level C1 protection equipment.

Immediately after the collection vessel has been removed from the Toxbuster trailer, IT Corporation will enter the trailer and obtain wipe samples to verify that no TCDD is present. Wipe samples of the feed equipment will be taken immediately after decontamination procedures.

The results of the Toxbuster interior and feed equipment wipe samples taken on June 25 or 26 will be available on June 26 or 27 so that equipment disassembly and packaging can be performed.

All materials requiring disposal will be placed in fiber drums for disposal by EG&G. In addition, the two 55-gallon drums of activated carbon (Nixtox filters) will be removed from the AER process flow train and given to EG&G for disposal. All residual waters generated during decontamination procedures will be drained on the contaminated test site for evaporation.

## 10. FINAL REPORT

The current schedule requires that Huber prepare a draft final report which will be submitted to EG&G on September 3, 1985. After comments are received from EG&G by September 16, the final report will be issued September 30.

Contained in the final report will be the following items:

- Executive Summary
- Process Description
- Operating Parameters
- Review and Audit of Industrial Hygiene Plan
- On-Site Performance Summary
- Frequency of Sampling and Analytical Procedures Used
- Results and Conclusions

ATTACHMENT 1

Gulfport Run Plan

RESEARCH AND TEST EVALUATION

GULFPORT RUN PLAN

REVISION NO. 2

APRIL 15, 1985

TIME AND DATE

ACTION

6/22/85

Obtain Samples  
Record weights  
Preparation

The Process Team will weigh the filter bags and the feed bin and record these weights on the data sheets. The top and bottom of each Nixtox activated carbon drum will be sampled by the verification subcontractor. The feed bin, which will have been filled with about 1,000 pounds of soil by the pretreatment subcontractor (IT Corporation), will be installed over the reactor by test personnel in level "C1" protection. Feedstock samples will be taken by the verification subcontractor prior to feed bin installation. IT Corporation will take two pretest wipe samples. In addition, industrial hygiene monitoring equipment will be set up for sampling during the entire testing period. The equipment decontamination area will be set up for use on 6/26/85.

05:00 6/24/85

Reactor Warmup

The process engineer and lead operator from Team 3 will bring the reactor up to operating conditions by 08:00.

08:00 6/24/85

Begin Test  
Record process data

The reactor core will be maintained at between 3500 and 4500 F for the test. Ten to twenty minutes is allowed for Process Team 1 to optimize the feed rate and the nitrogen rate. The soil feed rate will begin at about .4 lb/m and be gradually increased to .6 lb/m by 08:30. This will provide some time for the system to equalize. The control data will be recorded every 15 minutes starting at 08:00 and continuing until shutdown.

08:50

Process gas sampling

The start of emission testing will be determined by the field coordinator. No further process changes will take place (other than emergency shutdown). Occasionally, the soil flow may be stopped (for less than 1 minute) to periodically clean the feed tube via a closed system in-line brush.

09:25

O<sub>2</sub>, CO<sub>2</sub>, CO, Stack  
Analyses

The emission testing team will be taking orsat and draeger tube samples at the stack. All other functions will continue.

The three process teams will operate the reactor on 8-hour shifts.

14:00 to 24:00 6/25/85

End of Test  
Samples

The field coordinator will determine the end of the test. The Process Team will remove contaminated equipment and place in decontamination area for cleaning. The feed bin will be weighed before it is decontaminated. The reactor and process components will be allowed to cool overnight. Reactor emissions samples will be taken by the verification subcontractor for later analysis. The industrial hygiene air filters will be turned over to the verification subcontractor for later analysis.

08:00 6/26/85

Samples

The treated solids collection vessel will be removed from the AER, sampled, sealed, weighed, and placed in the contaminated area. The Nixtox drums will be sampled. The bag filter vessel will be dismantled, sampled, and the filter bags will be weighed. All weights will be recorded by the process engineer and lead operator. Bag filter bags and other residue materials will be placed in a fiber drum for disposal.

10:00 6/26/85

Decontamination  
Samples

The Process Team will decontaminate the feed bin, feed hopper, and feed tube. IT Corporation will take wipe samples on the feed equipment, the "charge end" of the reactor, and the discharge end of the reactor. In addition, four other surfaces will be wipe tested. These wipe samples will be taken by the IT Corporation for immediate analysis to confirm decontamination of the Toxbuster.

13:00

Decontamination  
Analysis

The Process Team will decontaminate all other Toxbuster equipment under review of the industrial hygiene engineer.

The verification subcontractor will perform the process emissions analyses, activated carbon analyses, and air filters analyses. If any of these are positive for TCDD, the field coordinator will determine if any of the "Priority 3" samples need analysis.

ATTACHMENT 2

SUMMARY OF TEST CONDITIONS

1. Location: Naval Construction Battalion Center, Gulfport, Mississippi
2. Test to be conducted in cooperation with EG&G, Idaho Falls, Idaho.
3. Date: Late June, 1985 (nominally June 24, 1985)
4. Number of Tests: One (1)
5. Feed Rate: Between 0.4 and 0.6 lb/min
6. Test Duration: Approximately 30 to 40 hours
7. Dioxin Concentration: Approximately 100-200 ppb TCDD
8. Total Quantity of Soil to be Tested: Approximately 1,000 pounds
9. Total Quantity of Dioxin to be Destroyed: Approximately  $2 \times 10^{-4}$  pounds or 0.09 grams TCDD
10. Results Expected:
  - a. Greater than 99.9999% DRE or TCDD not detected in gas phase.
  - b. Greater than 99.9999% DRE or TCDD not detected in the solid phase.
11. Expected Reporting Date: September 30, 1985

In every previous demonstration test, the safety and hygiene practices which Huber implemented have adequately protected personnel, equipment, and the environment. Therefore, the procedures shown in the safety plan (also enclosed) have been tested and proven effective. Previous tests have verified that during actual operation, Huber's system is completely closed. Therefore, Level C1 protection will be utilized only when the system is open. As presently envisioned, an open system will be said to exist when preparing feed material, filling feed bins, changing the treated soil hopper, and inspecting equipment component internals.



ATTACHMENT 3

TEST PARTICIPANT DESCRIPTIONS

Job Title: Field Coordinator

Communications Equipment: Portable radio

Duties: The Field Coordinator has the overall responsibility to see that the test is conducted according to the written test plan. This includes coordinating the efforts of the Process Operations Leader and verification subcontractor. The Field Coordinator is also the primary contact between Huber and the Air Force, Navy, and EG&G. The Field Coordinator addresses any unforeseen problems and keeps everyone informed of test status and problems. The Field Coordinator will don Level "C1" personnel protection if needed during decontamination procedures. Otherwise, Level C2 personnel protection will be used.

Job Title: Industrial Hygiene Engineer (IHE)

Communications Equipment: Portable radio

Duties: The IHE has the authority to stop the run if he deems a safety hazard exists. The IHE cooperates with the Field Coordinator and process engineer to see that all personnel are properly clothed with protection equipment and that their jobs are carried out in a safe manner. The IHE periodically walks through the test site and notes any safety-related problems. During the test, Level C2 equipment will be used.

Job Title: Standby Operator (Runner)

Communications Equipment: None

Duties: The primary function of the Standby Operator is to act as a replacement for any of the operators in the event of an illness or injury. When in reserve, the Standby Operator assists the Industrial Hygiene Engineer or will handle miscellaneous duties. During the test, Level C2 equipment will be used.

Job Title: Process Engineers - Process Operations

Communications Equipment: Radio while at the top of the reactor

Duties: The Process Engineers rotate with the Lead Operator to monitor the process at the top of the reactor through the site glass. During the one-half intervals when not at the top of the reactor, the Process Engineer walks through the test site and monitors the equipment and the operators and lends assistance and direction as necessary. The Process Engineers are responsible for seeing that the operators carry out their job functions as specified in their respective assignment lists. The Process Engineers have complete responsibility for operation of the equipment, documentation of run data, and safety of the operation. The Process Engineers are expected to require Level "C1" uniforms when decontamination procedures are being performed. During the test, Level C2 protective equipment will be worn.

Job Title:       Lead Operator

Communications Equipment:       Portable radio

Duties:   The Lead Operator is responsible for the operation of the reactor and auxiliary equipment under the direction of the Process Engineer. He ensures that all process variables are maintained at prescribed levels and immediately notifies the Process Engineer if there are problems or process conditions vary outside of prescribed limits. The Lead Operator also keeps a log of all process changes and the time each change occurs. By monitoring the controls, he ensures that all equipment is operating properly. In the event of equipment problems, the Lead Operator relays instructions to other operators via radio under the direction of the Process Engineer. The Lead Operators are expected to require Level "C1" uniforms when decontamination procedures are being performed. During the test, Level C2 protective equipment will be worn.

Job Title: Process Data Operator

Communications Equipment: Portable radio

Duties: The Process Data Operator is responsible for real-time monitoring of the process equipment during the run. He takes periodic readings to obtain instantaneous measurements of the feed rate and other process parameters during the test. In addition, he checks for cooling water leaks, nitrogen gas leaks, etc. He immediately reports any conditions that might affect the safe operation of the equipment to the Process Engineer. Much of the data collected by the Process Data Operator is duplication to avoid holes in the overall data for proper documentation. During the test, Level C2 protective equipment will be worn.

**ATTACHMENT 4**

**Process Data Sheets**



DIOXIN DEMONSTRATION  
DATA SHEET

PROCESS LOG

REACTOR: \_\_\_\_\_

DATE: \_\_\_\_\_

TEST NO: \_\_\_\_\_

TEAM: \_\_\_\_\_

TIME	COMMENTS
_____	_____
_____	_____
_____	_____
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_____	_____

SIGNATURE (S) \_\_\_\_\_ 70 \_\_\_\_\_ DATE \_\_\_\_\_

MOBIO GULFPORT DENITRIFICATION DATA SHEET - CONTROL RECORDS

DATE: ____ / ____ / ____	ITEM	TIME								
	FEED DATA									
	Feeder Setting									
	Feeder Hopper Wt. (lbs)									
	Instantaneous Feed Rate (lb/m)									
	Total Soil Fed (lbs)									
	REACTOR DATA									
	Core Temp. Set Point °F									
	Millivolt Reading									
	Reactor Power, KW									
	Reactor Pressure, "H <sub>2</sub> O									
	NITROGEN FLOWS									
	Blanket Gas (Upper Plenum) %PSI									
	Sweep Gas (Lower Plenum) %PSI									
	Radiamatic Purge Gas									
	Sight Glass N <sub>2</sub> Purge ( )									
	Collection Vessel N <sub>2</sub> Purge (scfm)									
	Feed Tube N <sub>2</sub> ( )									
	Total N <sub>2</sub> scfm (rotameter)									
	Dry Gas Meter - N <sub>2</sub> Inlet									
	Dry Gas Meter - Flue Gas									
	PRTZ DATA									
	Upper PRTZ Temp. (T2) °F									
	Lower PRTZ Temp. (T3) °F									

NCBC GULFPORT DEMONSTRATION DATA SHEET - CONTROL RECORDS

Page \_\_\_\_ of \_\_\_\_

DATE: ____ / ____ / ____	TIME																			
Cooling Water Flows:		%/PSI																		
ØA		%/PSI																		
ØB		%/PSI																		
ØC		%/PSI																		
Reactor Body (Lower)		%/PSI																		
PRZ Body		%/PSI																		
Radiamatic		%/PSI																		
Cooling Water Temps:		°F																		
ØA		°F																		
ØB		°F																		
ØC		°F																		
Body (Lower)		°F																		
PRZ		°F																		
Radiamatic		°F																		
Upper Plenum		°F																		
Reactor Voltage:																				
ØA - ØB																				
ØA - ØC																				
ØB - ØC																				
Reactor Amps:																				
ØA																				
ØB																				
ØC																				

Signature(s): \_\_\_\_\_ Date \_\_\_\_\_

**NCBC GULFPORT DEMONSTRATION DATA SHEET**

**- PRETEST/POSTTEST CHECKLIST -**

New Filter Bags, Wt: \_\_\_\_\_  
 Filter Bags After Test, Wt: \_\_\_\_\_  
 Feed Bin Empty, Wt: \_\_\_\_\_  
 Feed Bin Full, Wt: \_\_\_\_\_  
 Feed Bin After Test, Wt: \_\_\_\_\_  
 Collection Vessel Empty, Wt: \_\_\_\_\_  
 Collection Vessel After Test, Wt: \_\_\_\_\_  
 Fiber Drums Filled After This Test: \_\_\_\_\_

**PRETEST/POSTTEST SAMPLING CHECKLIST**

<u>Location/Item</u>	<u>Time Sample Obtained</u>	<u>Individual's Initials Taking Custody</u>
1. Feed Bin	_____	_____
2. Pretest Wipe Samples	_____	_____
3. Nixtox Drums, Before Test	_____	_____
4. Nixtox Drums, After Test	_____	_____
5. Bag Filter Sample	_____	_____
6. Treated Soil Sample	_____	_____
7. Decon Wipe Samples	_____	_____
a. Feed Bin	_____	_____
b. Feed Hopper	_____	_____
c. Screw Feeder	_____	_____
d. Feed Tube	_____	_____
e. Inside Trailer	_____	_____

Comments

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Signature(s) \_\_\_\_\_

Date: \_\_\_\_\_

APPENDIX E

INDUSTRIAL HYGIENE, HEALTH, AND SAFETY  
PLANS SUPPORTING HUBER'S AER DEMONSTRATION  
TEST AT NCBC

INDUSTRIAL HYGIENE, HEALTH, AND SAFETY  
PLAN FOR THE TREATMENT OF SOIL  
CONTAINING HERBICIDE ORANGE USING  
THE ADVANCED ELECTRIC REACTOR AT THE  
NAVAL CONSTRUCTION BATTALION CENTER  
IN GULFPORT, MISSISSIPPI



## HUBER TECHNOLOGY

J. M. HUBER CORPORATION

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INDUSTRIAL HYGIENE, HEALTH, AND SAFETY  
PLAN FOR THE TREATMENT OF SOIL  
CONTAINING HERBICIDE ORANGE USING  
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NAVAL CONSTRUCTION BATTALION CENTER  
IN GULFPORT, MISSISSIPPI

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April 29, 1985

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## Introduction

The J. M. Huber Corporation has successfully and safely tested the use of a new technology for the degradation/destruction of dioxin in soil. This process, an Advanced Electric Reactor, has been utilized to destroy laboratory prepared octachlorodibenzo-p-dioxin in soil in two separate tests in Borger, Texas. Additionally, a highly successful and efficient test was recently completed in Times Beach, Missouri detoxifying tetrachlorodibenzo-p-dioxin contaminated soil. This technology has also been demonstrated to be highly effective in the destruction of polychlorinated biphenyls (PCB) and hexachlorobenzene (HCB) in soils and other organics such as  $CCl_4$  as liquids or on soils.

In every previous demonstration test, the safety and hygienic practices which Huber implemented adequately protected personnel, equipment, and the environment. Therefore, the procedures detailed in this safety plan have been tested and proven effective.

The following Industrial Hygiene, Health, and Safety Plan addresses the use of Huber's Advanced Electric Reactor for destruction of dioxin and other similar compounds (products contained in Herbicide Orange) in soil at the Naval Construction Battalion Center (NCBC) in Gulfport, Mississippi.

## MATERIAL CONTROLS

In order to minimize employees exposure to dioxin, the contaminated soils will be transferred from the ground to the pretreatment facility and then to the reactor in enclosed containers. These will be filled utilizing techniques which prohibit direct personal contact with the material and inhibit fugitive dust emissions.

## PROCESS CONTROLS

The destruction process is essentially a closed system due to the many safeguards Huber has built into the system. This includes the use of closed feed bins and hoppers for the decontaminated product and carefully fitted connections between the feed bins, reactor product bins, and the air pollution control system (bag filters and activated carbon beds). The closed Huber system also operates at very low pressures (about 1-2 inches of water) and thus offers little potential for hazards in the event of unforeseen operating problems

## TEST AREA CONTROL

The test area will be roped off using caution tape allowing an area sufficient to permit all of the operations which are part of the test to occur unhindered. Access to and departure from the site will be carefully controlled with specifically designated entrances and exits. Entrance into NCBC is controlled by Navy guards, and periodical patrols of the test site will be made by the Navy.

Two levels of protection, Levels C1 and C2, will be utilized at the job site. These levels are defined as follows:

### Level C1

Level C1 protection provides the following:

Personnel are to be qualitatively fitted with a full face air purifying respirator. The fit test procedures includes the use of isoamyl acetate or irritant smoke and will be administered by Huber's Industrial Hygienist prior to the test.

Combination NIOSH/MSHA approved pesticide or organic vapor/highly toxic particulate filter cartridges or canisters will be utilized. See Addendum B for care and cleaning of respirators.

Saran coated Tyvek coveralls with complete coverhood (Ref. 1) will normally be worn. Uncoated Tyvek coveralls may be used if appropriate.\*

VITON gloves - inner

NITRILE gloves or cotton gloves - outer

Rubber boots (decontaminatable) to pull over leather safety shoes or steel-toed rubber boots.

Disposable boots - Latex

Inner cotton garment, either coveralls or long underwear

Sleeves, pant cuffs and coverhoods of coveralls, will be attached to gloves, boots and coveralls, using duct tape.

- \* IF AMBIENT WORK STATIONS TEMPERATURE IS ABOVE 70 F AND HEAT BECOMES A PROBLEM, THE SARAN COATED TYVEKS MAY BE REPLACED BY PLAIN TYVEK IN ORDER TO PREVENT ACUTE HEAT STRESS. HUBER'S INDUSTRIAL HYGIENIST ON THE JOB WILL MAKE THIS DETERMINATION.

Level C2 protection includes the use of perforated Tyvek (or similar material) coveralls, rubber boots, cotton gloves, half face masks using organic vapor/highly toxic particulate cartridges (see Addendum B for care and cleaning of respirators), hard hats, and safety glasses.

The equipment will be initially placed on a clean, uncontaminated site situated near the test plot. Since the equipment will also be uncontaminated at this point, all set up and equipment installation will be considered clean and, thus, not require C1 or C2 protection. Standard safety equipment will be used during set up (i.e., hard hat, safety glasses and steel-toed boots).

Previous tests have verified that during actual operation, Huber's system is completely closed. Therefore, Level C1 protection will be utilized only when the system is open. Open systems include preparations of the feed material, filling the feed bin, mounting and unmounting the feed bin on the reactor, changing the solids collection vessel, and inspection of equipment component internals. During normal operation, whenever Level C1 equipment is not required, Level C2 equipment will be used.

Persons and equipment leaving the area will be decontaminated. Extensive equipment decontamination (i.e., steam cleaning) may be foregone if sampling (See Addendum C) shows less than 150 nanograms of dioxin per square meter wipe. The decontamination procedures are listed beginning on page 5.

### Personal Protection

#### Training

All project personnel will have been or will be trained regarding the hazards involved in handling Herbicide Orange contaminated soil, use and care of protective equipment, and the Industrial Hygiene and Safety Plan. An additional site specific training course will be provided for employees prior to initiation of site

work. Prior to the test, a Safety Meeting will be held to review procedures and key items of the Safety Plan.

#### HEAT STRESS

The use of Level C1 protective gear will increase the risk of heat stress. Because of this, a very stringent heat stress monitoring program will be instituted.

Workers in the exclusion zone will work in pairs. "Buddies" must be alert for signs of distress and will frequently check on each other. An additional employee assigned for decontamination will serve as a safety watch. The Safety Watch will be dressed in Level C1 when required. He will monitor the activities of on site persons, watching for signs of heat stress. The Safety Watch will have a buddy who remains in contact with him and is stationed in the non regulated zone.

A shower or hose will be readily available to enable rapid cool down of persons exposed to heat. Huber's Industrial Hygienist will supervise rapid cool-down should it be required.

A break area will be available to allow workers to cope with heat or cold conditions.

Depending on the ambient temperature, a work/rest regimen will be established to enable workers to adjust to heat (See Addendum A). At the discretion of Huber's Industrial Hygienist, work hours may be delayed until ambient temperatures are cooler.

Heat Stress Monitoring specifics are expounded upon in Reference 2.

Prior to workers donning Level C1 personnel protective equipment, the following vital signs will be recorded by Huber's Industrial Hygienist when ambient temperatures are above 70 F.

- Weight
- Heart Rate
- Oral Temperature
- Blood Pressure

Heart rate, blood pressure and oral temperature will be monitored each time an individual dons Level C1 personnel protection when the employee's work site temperature exceeds 70 F. This monitoring will be used along with other criteria to determine rest time and further monitoring needs. The method of decision making for these incidences is shown in Addendum A. Medical data will be collected and interpreted by Huber's Industrial Hygienist.

Addendum A includes a discussion of different heat stresses, monitoring, and protection.

Persons using Level C1 equipment must replace body fluids. As workers doff Level C1 personnel protection they must be encouraged to drink 8 ounces of water, fruit juice or fruit juice flavored water. Commercial electrolyte drinks (for example Gatorade) may be used but the proper electrolyte balance can be achieved by following the above recommendations and by generously adding salt to meals.

#### MEDICAL MONITORING

The program in Addendum D will be used for medical monitoring of Huber personnel that use Level C1 protective equipment.

#### NOISE

A survey of the test area and trailer carrying the 3" reactor to identify any high noise sources was conducted. Hearing protection is not required unless employees work for more than 1-1/2 hours in the Heat Exchanger Room when the heat exchanger motors are operating.

#### DECONTAMINATION

A strictly controlled contamination reduction program has been established for both personnel and equipment. Guidelines are listed below.

Decontamination procedures will be arranged to generate as little excess liquid as possible. Decontamination solution may be a strong trisodium phosphate solution. The solution is prepared by mixing 0.4 pounds of trisodium phosphate into 1 gallon of water. As appropriate, steam cleaning may be used.

#### PERSONNEL

Because IT Corporation will already have an employee decontamination facility set up, Huber will utilize IT's facilities for personnel decontamination. A detailed description of IT's employee decontamination facility is contained in IT's Health and Safety Plan.

#### EQUIPMENT

Every effort will be made to reduce the need to decontaminate equipment. Small tools, sample containers, etc. will be decontaminated with a damp decon solution wipe off. The procedures used for large equipment decontamination are:

1. Purge with clean sand, when possible;
2. Scrape clean, and vacuum out all loose material;

3. Wash out with trisodium phosphate/water solution using sponge and scrub brush. Sponge rinse with clean water; or
4. Steam clean, if required.

Equipment decontamination will be performed on the contaminated site using either a trisodium phosphate solution or a steam generator. After decontamination the cleaned equipment will be placed in a buffer zone (adjacent to the contaminated area) until wipe testing verifies that the equipment is clean. While in the buffer zone, the equipment will be wrapped in plastic.

Huber's recommended acceptable level for contamination reduction on equipment is 150 nanograms of TCDD per square meter wipe sample.

## EMERGENCY RESPONSE PROCEDURES

### MEDICAL

Any worker suspected of being exposed to dioxin via inhalation, ingestion, or skin contact may be taken immediately, following decontamination, to the designated Medical Facility and will be examined. The need for medical attention will be determined on a case by case basis by Huber's on-site Industrial Hygienist and the J. M. Huber Project Director. Blood and urine samples may be collected and analyzed for appropriate parameters, namely liver function indicators and others as designated by the on-site Industrial Hygienist and the consulting physician.

### SPILL

Any spill or release of dioxin contaminated soil will require remedial action. In the event of a spill or other unanticipated occurrence while test personnel are in Level C2 equipment, the feeder will be turned off and the Toxbuster will be vacated. After donning Level C1 equipment, corrective actions will begin. Small spills will immediately be vacuumed up (using a hazardous waste vacuum cleaner equipped with a HEPA filter) and contaminated surfaces will be washed with soap and water. Large spills, which require more extensive decontamination, will be evaluated by on-site management and appropriate actions followed as the situation directs. Shovels, sand, absorbents, etc., will be maintained on-site to accommodate these situations.

### FIRE

Personnel are trained in use of portable fire extinguishers. These will be maintained on site. The location and use of portable fire extinguishers in the Toxbuster will be reviewed prior to Huber's endurance/preparation run in May. Additional fire reporting, response, and evacuation procedures at the NCBC will be followed. A fire will require use of SCBA for personnel protection.

### SPECIAL SAFETY CONSIDERATIONS

#### Heat From the Reactors

In addition to ambient heat, there will also be heat generated by the reactors. This will add to the heat burden on the workers, making heat stress monitoring all the more essential when Level C1 equipment is used.

Workers must be carefully observed and monitored.

Some of the reactor associated equipment is also a hot object contact hazard. Because workers may have impaired visibility and

hampered movement due to the personal protection, it is important to remind them of this hazard.

#### FALLS

The reactor must be accessed and attended by ladder and elevated platforms. Safeguards are built into the system following applicable OSHA guidelines, but the use of the personal protection equipment will make these areas and access to them more hazardous. Therefore, workers will be advised of the specific areas where extra precautions are required.

#### ELECTRICAL

High voltage electrical components are safely guarded. However, workers will be reminded of these potential hazards. If there is a need for electrical service during the test, a special protection protocol will be worked out by the Industrial Health Engineer, the project director, and the operators so the electrician will be protected from exposure, but will still be able to work safely with the electrical gear.

#### INDUSTRIAL HYGIENE MONITORING

There are no formally established Industrial Hygiene Monitoring procedures yet developed for workers potentially exposed to Herbicide Orange containing dioxins. IT Corporation is responsible for the equipment to be used for the air monitoring program. Huber recommends the use of Industrial Hygiene Monitoring (i.e., personnel and area sampling) to determine airborne indications of potential exposure. Addendum C contains the recommended Industrial Hygiene Monitoring equipment and frequency of sampling. IT Corporation samplers will be placed in the work area by the Huber Project Director or someone he designates and will be placed in locations or on persons most likely to be exposed to dioxin (i.e., near feed bin at top of reactor). Analysis of samples should be a modification of the procedure used for stack sample analysis and wipe sample analysis.



### References

1. A proposal to provide support for a Hazardous Waste Treatment Demonstration, prepared for J. M. Huber Corporation, Borger, Texas, Proposal No. P-84-1828, Site Safety Plan, submitted jointly by Roy F. Weston, Inc., West Chester, PA, and York Research Consultants, Denver, CO, 1984.
2. The Industrial Environment-its Evaluation and Control, U. S. Department of Health, Education, and Welfare, 1973.
3. WESTON-SPER Health and Safety Training Manual Roy F. Weston, Inc., in association with Jacobs Engineering Group Inc., Tetra Tech, Inc., and ICF Incorporated, 1982.

Addendum A

CLASSIFICATION, MEDICAL ASPECTS, AND  
PREVENTION OF HEAT ILLNESS

Introduction

Weather conditions are a serious consideration in planning and conducting site operations. Humid weather can cause physical discomfort, loss of efficiency, personal injury, and in the extreme, can be life threatening. The worker is highly susceptible to these effects due to the wearing of protective clothing which decreases natural body ventilation and hence cooling. There are six generally recognized types of heat illness briefly described in decreasing order of severity below.

Heatstroke and Heat Hyperpyrexia

Clinical Features: Heatstroke: (1) hot dry skin: red, mottled, or cyanotic skin; (2) high rising core temperature, 104.9 F and over; (3) brain disorders: mental confusion, loss of consciousness, convulsions, or coma, as core temperature continues to rise. Fatal, if treatment delayed.

Heat hyperpyrexia: a milder form; core temperature lower; less severe brain disorders; some sweating.

Underlying  
Physiological  
Disturbance:

Heatstroke: failure of the central drive for sweating (cause unknown) leading to loss of evaporative cooling and an uncontrolled accelerating rise in core temperature.

Heat hyperpyrexia: partial rather than complete failure of sweating.

Predisposing  
Factors:

(1) sustained exertion in heat by unacclimatized workers, (2) obesity and lack of physical fitness, (3) recent alcohol intake, (4) dehydration, (5) individual susceptibility, (6) chronic cardiovascular disease in the elderly.

Treatment:

Heatstroke: immediate and rapid cooling by immersion in chilled water with massage, or by wrapping in wet sheet with vigorous fanning with cool dry air. Avoid overcooling. Treat shock if present. Obtain emergency medical care.

Heat hyperpyrexia: less drastic cooling required if sweating still present and core

temperature 104.9 F. Obtain emergency medical care.

Prevention: Medical screening of workers. Selection based on health and physical fitness. Acclimatization for 8 to 14 days by graded work and heat exposure. Monitoring workers during sustained work in severe heat.

#### Heat Exhaustion

Clinical Features: (1) Fatigue, nausea, headache, giddiness; (2) skin clammy and moist, complexion pale, muddy, or with hectic flush; (3) may faint on standing, with rapid thready pulse and low blood pressure; (4) oral temperature normal or low, but rectal temperature usually elevated (37.5 to 38.5 C). Water-restriction type: urine volume small, highly concentrated. Salt-restriction type: urine less concentrated, chlorides less than 3 milligrams per liter.

Underlying Physiological Disturbance: (1) dehydration from deficiency of water and/or salt intake; (2) depletion of circulating blood volume; (3) circulatory strain from competing demands for blood flow to skin and to active muscles.

Predisposing Factors: (1) sustained exertion in heat; (2) lack of acclimatization; (3) failure to replace water and/or salt lost in sweat.

Treatment: Remove to cooler environment. Administer salted fluids by mouth or give intravenous infusions of normal saline (0.9 percent) if patient is unconscious or vomiting. Keep at rest until urine volume and salt content indicate that salt and water balances have been restored. Obtain emergency medical care.

Prevention: Acclimatize workers using a breaking in schedule for 1 or 2 weeks. Supplement dietary salt only during acclimatization. Ample drinking water to be available at all times and to be taken frequently during work day.

Clinical Features: Painful spasms of muscles used during work (arms, legs, or abdominal). Onset during or after work hours.

Underlying Physiological Disturbance:	Loss of body salt in sweat. Water intake dilutes electrolytes. Water enters muscles, causing spasms.
Predisposing Factors:	(1) heavy sweating during hot work; (2) drinking large volumes of water without replacing salt loss.
Treatment:	Salted liquids by mouth, or more prompt relief by intravenous infusion, massage the cramped muscles. Obtain medical care as required.
Prevention:	Adequate salt intake with meals. In unacclimatized men, provide salted (0.1 percent) drinking water.

#### Heat Syncope

Clinical Features:	Fainting while standing erect and immobile in heat.
Underlying Physiological Disturbance:	Pooling of blood in dilated vessels of skin and lower parts of the body.
Predisposing Factors:	Lack of acclimatization.
Treatment:	Remove to cooler area. Recovery prompt and complete.
Prevention:	Acclimatization. Intermittent activity to assist venous return to heart.

#### Heat Rash

Clinical Features:	Profuse tiny raised red vesicles (blister like) on affected areas. Pricking sensations during heat exposure.
Underlying Physiological Disturbance:	Plugging of sweat gland ducts with retention of sweat and inflammatory reaction.
Predisposing factors:	Unrelieved exposure to humid heat with skin continuously wet with unevaporated sweat.
Treatment:	Mild drying lotions. Skin cleanliness to prevent infection. Obtain medical care as required.

Prevention: Cooled sleeping quarters to allow skin to dry between heat exposure.

Heat Fatigue-Transient

Clinical Features: Impaired performance of skilled sensorimotor, mental, or vigilance tasks in heat.

Underlying Physiological Disturbance: Discomfort and physiological strain.

Predisposing: Performance decrement greater in unacclimatized and unskilled men.

Treatment: Not indicated unless accompanied by other heat illness.

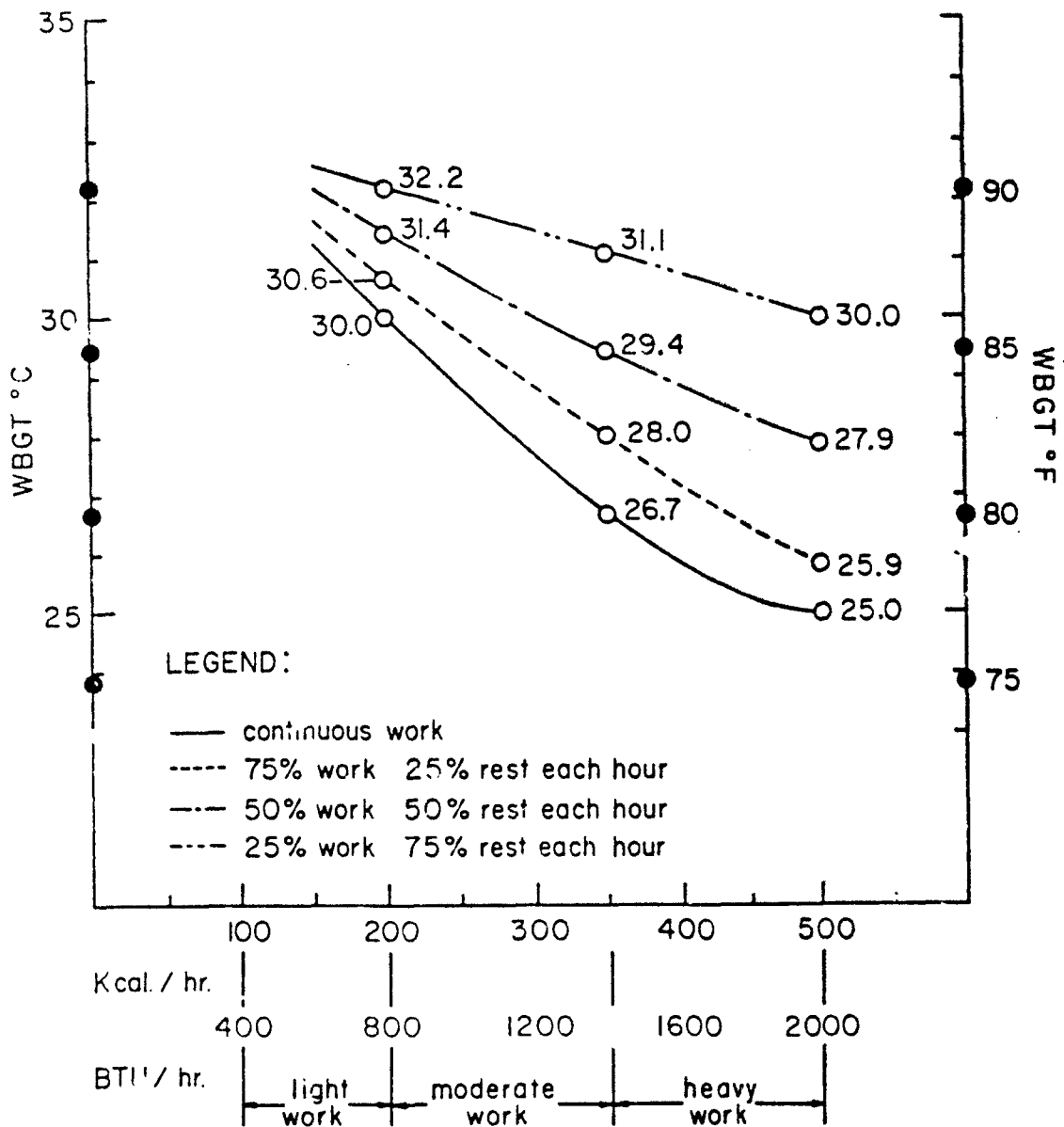
Prevention: Acclimatization and training for work in the heat.

Prevention

Heat illness is not 100% preventable; however, by using physically and medically fit workers (no colds, sunburns, etc.), careful planning, work distribution, and personnel monitoring, the likelihood of heat illness can be drastically reduced. A first planning step is to consider the weather conditions and level of protective clothing.

Employees required to use Level C1 protection equipment will be monitored for heat stress by the Wet Bulb Globe Temperature Index (WBGT) technique. This method will require the use of a heat stress monitoring device, such as the Wibget Heat Stress Monitor (Renter Stokes).

The WBGT shall be compared to the Threshold Limit Values (TLV) outlined in the ACGIH TLV's Manual (see Figure 1 on the following page), and a work-rest regimen established, as necessary, according to the WBGT obtained. Because workers will be wearing impermeable Level C1 protective clothing, 5 C must be subtracted from the TLV's for heat stress.



American Conference of Governmental Industrial Hygienists: Cincinnati, Ohio, 1971.

Figure 1 Permissible Heat Exposure Threshold Limit Value.

Addendum B



## CARE AND CLEANING OF RESPIRATORS

### I. GENERAL REQUIREMENTS

Any organization using respirators on a routine basis should have a program for their care and cleaning. The purpose of a program is to assure that all respirators are maintained at their original effectiveness. If they are not, in any way, their protection factors may be voided. Ideally one person in an organization is trained to inspect, clean, repair, and store respirators.

The program should be based on the number and types of respirators, working conditions, and hazards involved. In general, the program should include:

- Inspection
- Cleaning and Disinfection
- Repair
- Storage

### II. INSPECTION

Inspect respirators after each use. Inspect a respirator that is kept ready for emergency use monthly to assure it will perform satisfactorily.

On air-purifying respirators, thoroughly check all connections for gaskets and "O" rings and for proper tightness. Check the condition of the face piece and all its parts, connecting air tube, and headbands. Inspect rubber or elastomer parts for pliability and signs of deterioration.

Maintain a record for each respirator inspection, including date, inspector, and any unusual conditions or findings.

### III. CLEANING AND DISINFECTION

Collect respirators at a central location. Brief employees required to wear respirators on the respirator program and assure them that they will always receive a clean and sanitized respirator. Such assurances can boost morale. Clean and disinfect respirators as follows:

- Remove all cartridges, canisters, and filters, plus gaskets or seals not affixed to their seats.
- Remove elastic headbands.
- Remove exhalation cover.
- Remove speaking diaphragm or speaking diaphragm-exhalation valve assembly.

-Remove inhalation valves.

-Wash face piece and breathing tube in cleaner/sanitizer powder mixed with warm water, preferably at 120 to 140 F. Wash components separately from the face mask, as necessary. Remove heavy soil from surfaces with a hand brush.

-Remove all parts from the wash water and rinse twice in clean warm water.

-Air dry parts in a designated clean area.

-Wipe face pieces, valves, and seats with a damp lint-free cloth to remove any remaining soap or other foreign materials.

NOTE: Most respirator manufacturers market their own cleaners/sanitizers as dry mixtures of a bactericidal agent and a mild detergent. One-ounce packets for individual use and bulk packages for quantity use are usually available.

#### IV. REPAIRS

Only a trained person with proper tools and replacement parts should work on respirators. No one should ever attempt to replace components or to make adjustments or repairs beyond the manufacturer's recommendations. It may be necessary to send high-pressure-side components of SCBA's to an authorized facility for repairs. Huber intends to have spare equipment on hand to use in the event of component failure. Repair of respirators will be performed at the factory. In the event that an emergency repair is required, a CIH will make the repairs as follows:

-Disassemble and hand clean the pressure-demand and exhalation valve assembly (SCBA's only). Exercise care to avoid damage to the rubber diaphragm.

-Replace all faulty or questionable parts or assemblies. Use parts only specifically designed for the particular respirator.

-Reassemble the entire respirator and visually inspect the completed assembly.

-Insert new filters, cartridges, or canisters, as required. Make sure that gaskets or seals are in place and tightly sealed.

#### V. STORAGE

Follow manufacturers' storage instructions, which are always

furnished with new respirators or affixed to the lid of the carrying case. In addition, these general instructions may be helpful:

- After respirators have been inspected, cleaned, and repaired, store them so to protect against dust, excessive moisture, damaging chemicals, extreme temperatures and direct sunlight.
- Do not store respirators in clothes lockers, bench drawers, or tool boxes. Place them in wall compartments at work stations or in a work area designated for emergency equipment. Store them in the original carton or carrying case.
- Draw clean respirators from storage for each use. Each unit can be sealed in a plastic bag, placed in a separate box, and tagged for immediate use.

Addendum C

J. M. HUBER, INC.  
Industrial Hygiene Sample Plan  
DIOXIN DESTRUCTION TESTS

## INTRODUCTION

Huber's required industrial hygiene test plan is designed to give a representative, but thorough testing of both airborne concentrations of dioxin and any surface contamination which may occur during the testing.

## SAMPLE PROCEDURES

Huber recommends that air samples be collected by using personnel or area samplers with a sampling rate of at least 3.0 liters per minute. Any dioxin should be collected on a glass fiber filter. Because TCDD has such a low vapor pressure, very little gaseous phase TCDD is expected. For this reason, sorbent tube samples should be obtained by placing XAD-2 sorbent tubes as a back-up to the glass fiber filter. A sampling time of 7 hours is needed to get the required 1260 liters. The lower detection limit for 1260 liters of air is 0.4 nanograms per cubic meter.

Pretest wipe samples should be collected on the day prior to the Site Test. Wipe area is defined as one square meter of surface area. In instances where the area is less than one square meter, the area of the equipment being tested will be adequate (i.e., wipe down the entire piece of equipment).

The table on the following page indicates the suggested number of samples to be taken and their location. The nature of the samples taken, their location and sampling frequency will be determined by Huber's on-site Industrial Hygienist.

SUGGESTED INDUSTRIAL  
HYGIENE SAMPLING PLAN

	<u>PRETEST</u>	<u>TEST</u>	<u>DECON</u>
A. Feed System			3
B. Charge end of reactor		1	
C. Discharge end of reactor 1		1	
D. Air pollution control system- inside first elbow		1	
E. Air pollution control system- <sup>2</sup> discharge		1	
F. Flat surface near reactor charge		1	
G. Flat surface in discharge area <sup>3</sup>		1	
H. Additional sample		1	
- Pretest wipe sample	1		
- Pretest wipe sample	1		
 <u>Air Samples</u>			
A. Hi-vol at control panel		1	
B. Hi-vol at trailer door		1	
C. Personnel at reactor feed		1	
D. Personnel at reactor discharge		1	

Footnotes:

1. Analyze C only if D or F positive.
2. Analyze F only if E and G positive.
3. Analyze H only if G positive.
4. A Process Engineer's respirator filter (picked at random) should be analyzed for the presence of dioxin as he has the greatest potential for exposure.

Addendum D



J. M. HUBER, INC.

Medical Monitoring Program

## Heat Stress Monitoring

For monitoring the body's recuperative ability to excess heat, one or more of the following techniques should be used as a screening mechanism. Monitoring of personnel wearing impervious clothing should commence when the ambient temperature is 70 F or above. Frequency of monitoring should increase as the ambient temperature increases or as slow recovery rates are indicated. When temperatures exceed 85 F, workers should be monitored for heat stress after every work period. The parameters to be monitored, and the frequency of monitoring, will be determined by Huber's Industrial Hygienist.

1. Heart rate (HR) should be measured by the radial pulse for 30 seconds as early as possible in the resting period. The HR at the beginning of the rest period should not exceed 110 beats per minute. If the HR is higher, the next work period should be shortened by 10 minutes (or 33%), while the length of the rest period stays the same. If the pulse rate is 100 beats per minute at the beginning of the next rest period, the following work cycle should be shortened by 33%.
2. Body temperature should be measured orally with a clinical thermometer as early as possible in the resting period. Oral temperature (OT) at the beginning of the rest period should not exceed 99 F. If it does, the next work period should be shortened by 10 minutes (or 33%), while the length of the rest period stays the same. However, if the OT exceeds 99.7 F at the beginning of the next period, the following work cycle should be further shortened by 33%. OT should be measured again at the end of the rest period to make sure that it has dropped below 99 F.
3. Body water loss (BWL) due to sweating should be measured by weighing the worker before and after wearing Level C1 personnel protection. The clothing worn should be similar at both weighings; preferably the worker should be nude. The scale should be accurate to plus or minus 1/4 lb. BWL should not exceed 1.5% of the total body weight. If it does, the worker should be instructed to increase his daily intake of fluids by the weight lost. Ideally, body fluids should be maintained at a constant level during the work day. This requires replacement of salt lost in sweat as well.
4. Good hygienic standards must be maintained by frequent change of clothing and daily showering. Clothing should be permitted to dry during rest periods. Persons who notice skin problems should immediately consult medical personnel.

D. Effects of Heat Stress

If the body's physiological processes fail to maintain a normal body temperature because of excessive heat, a number of physical reactions can occur ranging from mild (such as fatigue, irritability, anxiety, and decreased concentration, dexterity, or movement) to fatal. Standard reference books should be consulted for specific treatment or qualified medical aid sought.

APPENDIX F

MODIFIED EPA CARCINOGEN ASSESSMENT GROUP'S LIST  
FOR HERBICIDE ORANGE CONTAMINATION

APPENDIX F  
MODIFIED EPA CARCINOGEN ASSESSMENT  
GROUP'S LIST FOR HERBICIDE  
ORANGE CONTAMINATION

A copy of the draft "The Carcinogen Assessment Group's List of Carcinogens, July 14, 1980," dated 1984, was obtained from EPA (Headquarters) to review for compounds which could be associated with Herbicide Orange (HO). The Carcinogen Assessment Group's (CAG) list consists of chemicals for which there is substantial evidence of carcinogenicity.

Due to the generality of the CAG list, many of the listed chemicals are not applicable. Based on a screening review by Chemical Sciences personnel at EG&G Idaho, Inc., creosote and identified chemicals that are also on the EPA Priority Pollutant List (PPL) were determined as applicable for analytical testing of the treated NCBC soil to support the soil restoration evaluation. The PPL modified for HO is shown in Appendix G.

APPENDIX G

MODIFIED PRIORITY POLLUTANT LIST  
FOR HERBICIDE ORANGE CONTAMINATION

APPENDIX G  
MODIFIED PRIORITY POLLUTANT LIST  
FOR HERBICIDE ORANGE CONTAMINATION

The Priority Pollutant List (PPL) referenced in Section 307.(a)(1) of the Federal Water Pollution Control Act as amended by the Clean Water Act of 1977 consisted of 129 toxic pollutants or combinations of pollutants. This list was subsequently reduced to 126 by formal deletions presented in the Federal Register (di- and tri-chlorofluoromethanes, January 8, 1981, 46 FR 2266; and bis-(chloromethyl) ether, February 4, 1981, 46 FR 10723).

Based on a review of this list by Chemical Sciences personnel at EG&G Idaho, Inc., the list of 125 priority pollutants in Table G-1 were evaluated as necessary for laboratory analysis of the treated soil to support soil restoration evaluation. The one chemical not included is 1,2-diphenylhydrazine (PPL #37) because its reactivity rules out presence of the chemical because of the time since the HO storage area was used.

The list is organized into six groups; purgeable organics, base/neutral extractable organic compounds, acid extractable organic compounds, pesticides/PCBs, metals, and miscellaneous. The PPL number and chemical analysis serial (CAS) number are included for cross reference.

TABLE G-1. MODIFIED PPL FOR HO CONTAMINATION

Priority Pollutant Number	Name	CAS Number
<u>Purgeable Organics (28)</u>		
	Acrolein	107-02-8
	Acrylonitrile	107-13-1
4V	Benzene	71-43-2
6V	Carbon tetrachloromethane (carbon tetrachloride)	56-23-5
7V	Chlorobenzene	108-90-7
10V	1,2-Dichloroethane	107-06-2
11V	1,1,1-Trichloroethane	71-55-6
13V	1,1-Dichloroethane	75-34-3
14V	1,1,2-Trichloroethane	79-00-5
15V	1,1,2,2-Trichloroethane	79-34-5
16V	Chloroethane (ethyl chloride)	75-00-3
19V	2-Chloroethyl vinyl ether	100-75-8
23V	Trichloromethane (chloroform)	67-66-3
29V	1,1-Dichloroethene	75-35-4
30V	Trans-1,2-dichloroethene	156-60-5
32V	1,2-Dichloropropane	78-87-5
33V	1,3-Dichloropropane	10061-01-05
38V	Ethyl benzene	100-41-4
44V	Dichloromethane (methylene chloride)	75-09-2
44V	Chloromethane (methyl chloride)	
46V	Bromomethane (methyl bromide)	
47V	Tribromomethane (bromoform)	75-25-2
48V	Bromodichloromethane	75-27-4
51V	Dibromochloromethane	124-48-1
85V	Tetrachloroethene	127-18-4
86V	Tuolene	108-88-3
87V	Trichloroethene	79-01-6
88V	Chloroethene (vinyl chloride)	75-01-4
<u>Base/Neutral Extractable Organic Compounds (45)</u>		
1B	Acenaphthene	83-32-9
5B	Benzidine	92-87-5
8B	1,2,4-Trichlorobenzene	120-82-1
9B	Hexachlorobenzene	118-74-1



TABLE G-1. MODIFIED PPL FOR HC CONTAMINATION (CONTINUED)

Priority Pollutant Number	Name	CAS Number
12B	Hexachloroethane	67-72-1
18B	bis (2-Chloroethyl) ether	111-44-4
20B	2-Chloronaphthalene	91-50-1
25B	1,2-Dichlorobenzene	95-58-1
26B	1,3-Dichlorobenzene	541-73-1
27B	1,4-Dichlorobenzene	106-46-7
28B	3,3'-Dichlorobenzidine	91-94-1
33B	2,4-Dinitrotoluene	131-11-3
36B	2,6-Dinitrotoluene	606-20-3
39B	Fluoranthene	206-44-0
40B	4-Chlorophenyl phenyl ether	7005-72-3
41B	4-Bromophenyl phenyl ether	101-55-3
42B	bis (2-Chloroisopropyl) ether	108-60-1
43B	bis (2-Chloroethoxy) methane	111-91-1
52B	Hexachlorobutadiene	87-68-3
53B	Hexachlorocyclopentadiene	77-47-4
54B	Isophorone	78-59-1
55B	Naphthalene	91-20-3
56B	Nitrobenzene	
62B	Diphenyl nitrosamine (N-nitrosodiphenylamine)	62-75-9
63B	Di-n-propyl nitrosamine (N-Nitrosodi-n-propylamine)	621-64-7
66B	bis (2-Ethylhexyl) phthalate	117-81-7
67B	Benzyl butyl phthalate	85-68-7
68B	Di-n-butyl phthalate	84-74-2
69B	Di-n-octyl phthalate	117-84-0
70B	Diethyl phthalate	84-66-2
71B	Dimethyl phthalate	131-11-3
72B	Benzo(a)anthracene	56-55-3
73B	Benzo(a)pyrene	50-32-8
74B	Benzo(b)fluoranthene	205-99-2
75B	Benzo(k)fluoranthene	207-08-9
76B	Chrysene	218-01-9

TABLE G-1. MODIFIED PPL FOR HQ CONTAMINATION (CONTINUED)

Priority Pollutant Number	Name	CAS Number
77B	Acenaphthylene	208-96-8
78B	Anthracene	120-12-7
79B	Benzo(g,n,i)perylene	141-24-2
80B	Fluorene	86-73-7
81B	Phenathrene	81-01-8
82B	Dibenzo(a)anthracene	53-70-3
83B	Indeno(1,2,3-c,d)pyrene	193-39-5
84B	Pyrene	129-00-0
	Dimethyl nitrosamine (N-nitrosodimethylamine)	86-30-6
<u>Acid Extractable Organic Compounds (11)</u>		
21A	2,4,5-Trichlorophenol	88-06-2
22A	4-Chloro-3-methylphenol (p-Chloro-m-cresol)	59-50-7
24A	2-Chlorophenol	95-57-8
31A	2,4-Dichlorophenol	120-83-2
34A	2,4-Dimethylphenol	105-67-9
57A	2-Nitrophenol	88-75-2
58A	4-Nitrophenol	100-02-7
59A	2,4-Dinitrophenol	51-28-5
60A	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)	534-52-1
64A	Tetrachlorophenol	87-86-5
65A	Phenol	108-95-1
<u>Pesticides/PCBs (26)</u>		
	Aldrin	309-00-2
	Alpha-BHC	319-84-6
	Beta-BHC	319-85-7
	Gamma-BHC (Lindane)	58-89-9
	Delta-BHC	319-86-8
	Chlordane	57-74-9
	4,4'-DDD	72-55-8
	4,4'-DDE	72-54-8
	4,4'-DDT	50-29-3

TABLE G-1. MODIFIED PPL FOR HO CONTAMINATION (CONCLUDED)

Priority Pollutant Number	Name	CAS Number
	Dieldrin	60-57-1
	Endosulfan I	959-98-8
	Endosulfan II	33212-65-9
	Endosulfan sulfate	1031-07-8
	Endrin	72-20-8
	Endrin aldehyde	7421-93-4
	Heptachlor	76-44-8
	Heptachlor epoxide	1024-57-3
	Toxaphene	800-35-2
	PCB-1016	12674-11-2
	PCB-1221	1104-28-2
	PCB-1232	11141-16-5
	PCB-1242	53469-21-9
	PCB-1248	12672-29-6
	PCB-1254	11097-69-1
	PCB-1260	11096-82-5

Metals (13)

Antimony  
Arsenic  
Beryllium  
Cadmium  
Chromium

Copper  
Lead  
Mercury  
Nickel  
Selenium

Silver  
Thallium  
Zinc

Miscellaneous (1)

Total Cyanides

APPENDIX H

MODIFIED LIST OF COMPOUNDS  
INDIGENOUS TO HERBICIDE ORANGE

APPENDIX H  
MODIFIED LIST OF COMPOUNDS  
INDIGENOUS TO HERBICIDE ORANGE

Before the Herbicide Orange (HO) stored at the NCBC was destroyed, samples were taken from drums to determine the chemical composition of HO by each manufacturing source. Analytical results were presented in Aerospace Research Laboratories Report AD-A011 597, Analytical Methodology for Herbicide Orange. Volume 1: Determination of Chemical Composition, May 1975. While a number of chemicals were common to each manufacturer's HO, some chemicals of low concentration were found unique to a single manufacturer. Based on a review of the report data by Chemical Sciences personnel of EG&G Idaho, Inc., a list of chemicals (Table H-1) was prepared as a spectrum of possible HO chemical constituents in the treated NCBC soil that should be analytically tested for the soil restoration evaluation.

TABLE H-1. MODIFIED SUMMARY OF COMPOSITION OF HERBICIDE ORANGE

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CHEMICAL COMPOUND

2,4-Dichlorophenol  
2,4,6-Trichlorophenol  
Trichloroanisole  
Dichloro-Methoxyanisole

Butoxydichlorophenol  
Butoxytrichlorophenol  
Butyl-monochlorophenoxyacetate  
Butyl-dichlorophenoxyacetate

Butyl-trichlorophenoxyacetate  
Butyl-methoxy-dichlorophenoxyacetate  
Octyl-dichlorophenoxyacetate  
Octyl-dichlorophenoxypropionate

Octyl-trichlorophenoxyacetate  
1,1-Dibutoxy-2-trichlorophenoxyethane  
Octyl-methoxy-dichlorophenoxyacetate  
Butyl-bis-dichlorophenoxyacetate

Butyl ester of bis trichlorophenoxyacetic acid  
Butyl ester of trichlorophenoxy-(methoxy-dichlorophenoxy)-acetic acid  
2,4-Dichlorophenoxyacetic acid (free acid)  
2,4,5-Trichlorophenoxyacetic acid (free acid)

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APPENDIX I

ITC 2,3,7,8-TCDD ANALYTICAL RESULTS FOR AER TREATED SOIL  
AND BAGHOUSE FILTER SAMPLES



IT CORPORATION

AUG 12 1985

August 6, 1985

Mr. D. Derrington  
J.M. Huber Corp.  
P. O. Box 2831  
Borger, TX 79008

Dear Mr. Derrington,

Attached, please find the final report for the two soil samples received in our laboratory 6/15/85. These results were reported to the site verbally 6/20/85. Please feel free to contact me regarding questions concerning this data.

Sincerely,

Dennis M. Catalano  
Dioxin Laboratory Manager

njc

*xc: DBD 9-12-85 jpb*

Regional Office

IT Corporation • 312 Directors Drive • Knoxville, Tennessee 37923 • 615-690-3211



## HUBER REPORT

### Summary of Method

Two soil samples were received for the analysis of 2,3,7,8-TCDD. The samples and blank were spiked prior to extraction with an internal standard/surrogate solution containing 50 ng  $^{13}\text{C}$ -2,3,7,8-TCDD and 10 ng  $^{37}\text{Cl}$ -2,3,7,8-TCDD. The samples were soxhlet extracted, and cleaned up using the EPA reference method described in "The Determination of 2,3,7,8-TCDD From Soil and Sediment," revised September 1983. Extracts were analyzed by GC/MS operating in the selected ion monitoring mode for enhanced sensitivity.

### Sample Preparation

Thirty grams of one soil and 6 grams of the bag fines were weighed into separate jars. The samples were spiked with the internal standard/surrogate mixture and allowed to stand overnight for equilibration. The samples were pretreated with 100 ml 1 N HCl for 1 hour and subsequently filtered through a Buchner Funnel and air dried overnight, followed by a 16 hour soxhlet extraction with Benzene. Thirty grams of  $\text{NaSO}_4$  served as the blank. The resulting extracts were filtered into a KD Flask and the volume was reduced to approximately 10 ml and subsequently subjected to liquid column chromatography cleanup.

### Sample Cleanup

To aid in the removal of chemical interferences, the sample and blanks were cleaned up using dual column chromatography consisting of an acid-modified silica gel column followed by a neutral alumina column. Detailed descriptions of these cleanup techniques can be found in Option "A" of the EPA reference stated in the summary section. Final extracts were concentrated to near dryness and raised to 50  $\mu\text{l}$  with 11 ng  $^{13}\text{C}$ -2,3,7,8-TCDF which was used as an internal standard.

### GC/MS Analysis and Results

#### (1) Isomer specific 2,3,7,8-TCDD

The sample extracts were analyzed using HRGC/LRMS scanning in the selected ion monitoring mode for enhanced sensitivity. The column used for this

isomer specific analysis was a 60 m SP 2330 fused silica column. Before acquisition of the samples, a seven isomer performance mixture containing the six most closely eluting TCDD isomers to 2,3,7,8-TCDD was run. In addition, a five point calibration plot was run in triplicate. The mean response factor obtained from the fifteen point calibration was used for all subsequent calculations. The shift standard, analyzed on the same day as the samples, produced an acceptable response factor within 10% of the fifteen point. Percent recovery is reported by comparing  $^{13}\text{C}$ -TCDD to  $^{13}\text{C}$ -TCDF. Accuracy of the method is obtained by the recovery of  $^{37}\text{Cl}$ -TCDD versus  $^{13}\text{C}$ -TCDD.

The results shown in Table 1 are reported in ppb. A detection limit is calculated from 2.5 times the signal in the area of the elution of  $^{13}\text{C}$ -TCDD whenever a sample contains no detectable TCDD.

Table 1. Isomer Specific Results

Client #	Sample ID	IT #	Date	Time	2,3,7,8-TCDD (ppb)	Surrogate (% Accuracy)	% Recovery
Huber Run 1 (bag fines AER)		J2609	6/20/85	17:40	0.75	94%	90%
HU-NCBC-R1-02 (treated soil AER)		J2610	6/20/85	18:18	ND(0.045)	89%	93%
Reagent Blank		TBLK 381	6/17/85	13:48	ND(0.097)	91%	87%

APPENDIX J

HUBER FUEL GAS ANALYSIS DATA SHEETS

INJECT TIME 25 11:39:12

.10111

1.00110

1.72

3.02

4.157

4.51

5.00111

6.00001

7.00111

8.00110

11.50000

FIXED GAS ANALYSIS DURING  
ACTUAL TEST  
6-23-85  
(14:20)

FLUE GAS ANALYSIS

25 11:39:12

FILE 1 METHOD 5. RUN 790 INDEX 4

ANALYST: R. G. RYAN

NAME	CONC	RT	AREA DC	RF	PRT
AR	0.041	1.51	166 01	4204.	1.
N2	94.505	1.72	332000 01	3637.579	1.179
CO	1.217	3.02	4252 01	3614.865	2.
CO2	4.157	4.51	14929 01	3712.4	2.907

TOTALS 100. 352147

UNNORMALIZED TOTAL 96.727

FILE 1 METHOD 5. RUN 790 INDEX 4

NAME CONC BTU Sp. Gr.

N2	0.	0.	0.
O2	0.	0.	0.
AR	0.041	0.	0.0006
N2	94.505	0.	0.9140
CH4	0.	0.	0.
CO	1.217	3.9	0.0110
CO2	4.157	0.	0.0632
TOTAL	100.		

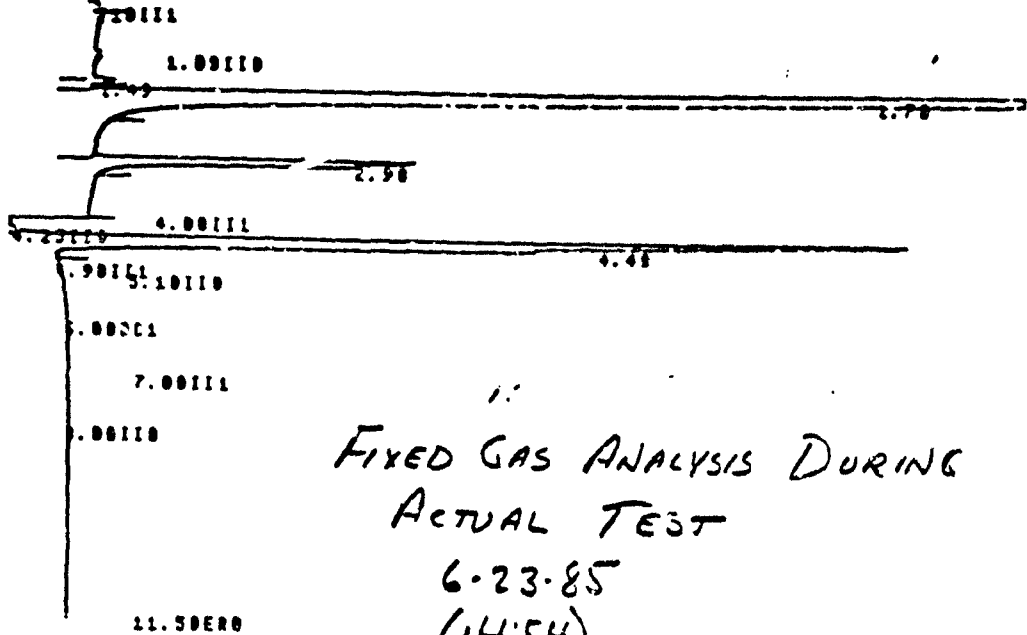
GROSS BTU/CU. FT.	DRY @ 14.696	3.90657
SPECIFIC GRAVITY	DRY @ 14.696	0.990327
GROSS BTU/CU. FT.	DRY @ 14.650	3.8944596
SPECIFIC GRAVITY	DRY @ 14.650	0.987257
GROSS BTU/CU. FT.	SAT @ 14.650	3.8304107
SPECIFIC GRAVITY	SAT @ 14.650	0.9730504

67

131

KA 91# GAS Tre B.F. AER-3 H:54 6-23-85

INJECT TIRE 26 00154150



FUE GAS ANALYSIS 26 00154150

FILE 1 METHOD 5. RUN 000 INDEX 1

ANALYST: R. G. RYAN

NAME	CONC	RT	AREA BC	RF	RRT
AR	9.037	1.49	147 01	4145.	2.
N2	94.721	1.7	329254 01	3619.225	1.141
CO	1.171	2.98	4040 01	1596.03	2.
CO2	4.071	4.48	14312 01	3656.	3.007

TOTALS 100. 347761

UNNORMALIZED TOTAL 96.15

FILE 1 METHOD 5. RUN 000 INDEX 1

NAME	CONC	BTU	Sp. Gr.
N2	0.	0.	0.
O2	0.	0.	0.
AR	9.037	0.	0.0005
N2	94.721	0.	0.9161
CH4	0.	0.	0.
CO	1.171	3.8	0.0113
CO2	4.071	0.	0.0619
TOTAL	100.		

GROSS BTU/CU. FT.	DRY @ 14.696	3.75891	68
SPECIFIC GRAVITY	DRY @ 14.696	0.9090355	
GROSS BTU/CU. FT.	DRY @ 14.650	3.7472574	
SPECIFIC GRAVITY	DRY @ 14.650	0.9067671	
GROSS BTU/CU. FT.	SAT @ 14.650	3.6913347	
SPECIFIC GRAVITY	SAT @ 14.650	0.9725676	

KX OFF GAS Pre D.F. ASK-3 16:05 6-23-85

INJECT TIME 25 11:25:10

.20111

.00110

1.72

3.01

.00111

4.36

.20110-90111

6.00001

.00111

0.00110

11.50000

FIXED GAS ANALYSIS DURING  
ACTUAL TEST  
6-23-85  
(16:05)

FLUE GAS ANALYSIS

25 11:25:10

FILE 1 METHOD 5. RUN 797 INDEX 3

ANALYST: R. G. RYAN

NAME	CONC	RT	AREA BC	RF	RRT
AR	0.034	1.51	141 01	4204.	1.
N2	94.788	1.72	342549 01	1637.579	1.139
CO	1.181	3.01	4240 01	1614.865	1.992
CO2	3.997	4.5	14743 01	3712.4	2.98
TOTALS	100.		361673		
UNNORMALIZED TOTAL		99.347			

FILE 1 METHOD 5. RUN 797 INDEX 3

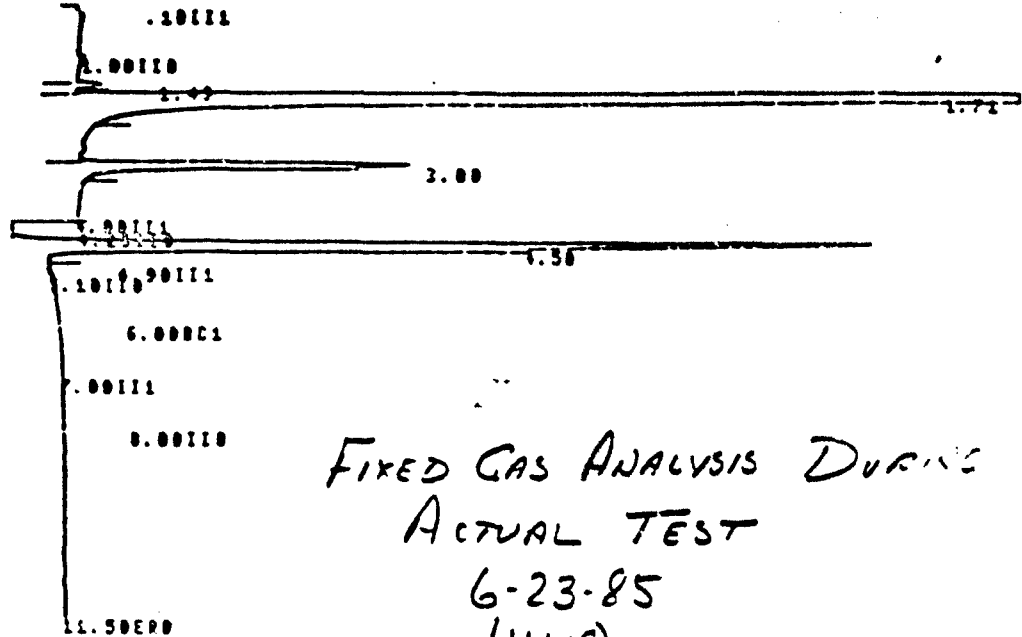
NAME	CONC	BTU	Sp. Gr.
N2	0.	0.	0.
O2	0.	0.	0.
AR	0.034	0.	0.0005
N2	94.788	0.	0.9168
CH4	0.	0.	0.
CO	1.181	3.0	0.0114
CO2	3.997	0.	0.0687
TOTAL	100.		

GROSS BTU/CU. FT.	DRY @ 14.696	3.79181
SPECIFIC GRAVITY	DRY @ 14.696	0.9894145
GROSS BTU/CU. FT.	DPY @ 14.650	3.7792579
SPECIFIC GRAVITY	DRY @ 14.650	0.9863473
GROSS BTU/CU. FT.	SAT @ 14.650	3.7248747
SPECIFIC GRAVITY	SAT @ 14.650	0.9721538

69

K2 off GAS Pre B.F AER-3 16:09 6-23-85

INJECT TIME 25 11:00:11



FIXED GAS ANALYSIS DURING  
ACTUAL TEST  
6-23-85  
(16:09)

FLUE GAS ANALYSIS 25 11:00:11  
FILE 1 METHOD 5. RUN 796 INDEX 2  
ANALYST: R. G. RYAN

NAME	CONC	RT	AREA BC	RF	RRT
N2	0.045	1.49	186 01	4204.	1.
N2	94.976	1.71	343077 01	3637.579	1.148
CO	1.166	3.	4193 01	2614.665	2.013
CO2	2.813	4.5	14050 01	3712.4	3.62
TOTALS	100.		361504		
UNNORMALIZED TOTAL		99.303			

NAME	CONC	BTU	Sp. Gr.
N2	0.	0.	0.
O2	0.	0.	0.
AR	0.045	0.	0.0006
N2	94.976	0.	0.9186
CH4	0.	0.	0.
CO	1.166	3.7	0.0113
CO2	2.813	0.	0.0579
TOTAL	100.		

GROSS BTU/CU. FT.	DRY @ 14.696	3.74286
SPECIFIC GRAVITY	DRY @ 14.696	0.9824426
GROSS BTU/CU. FT.	DRY @ 14.650	3.7312571
SPECIFIC GRAVITY	DRY @ 14.650	0.9853794
GROSS BTU/CU. FT.	SAT @ 14.650	2.6775647
SPECIFIC GRAVITY	SAT @ 14.650	0.9711999

70



APPENDIX K

IT CORPORATION INDUSTRIAL HYGIENE REPORT FOR ACTIVITIES  
SUPPORTING AER3 NCRB TESTS



AUG 26 1985

August 20, 1985

Mr. Darrell Derrington  
J. M. Huber Corporation  
P.O. Box 2831  
Borger, Texas 7900-2831

Dear Darrell:

Please find attached a table which gives results of industrial hygiene monitoring for 2,3,7,8 TCDD on June 22, 1985 during the test run of the AER at Gulfport, Mississippi. As can be seen, both employee exposure results were below detectable levels, which shows that the protection outlined in the Health and Safety Plan and worn during on-site activities was appropriate. Also, the area sample was below detectable levels which indicates that there were no fugitive emissions of 2,3,7,8 TCDD from the system.

The following is a general outline of the health and safety items on this job:

1. Heat stress readings using the WBGT methods, supplemental with blood pressure, pulse, and oral temperature readings, were taken throughout the test run. The WBGT readings ranged from 84°F-90°F. Appropriate breaks were scheduled and employees took these breaks. No evidence of a heat stress related illness, by observations or physical monitoring, was noted.
2. During the actual run the health and safety plan was followed without exception. CI protection was worn when any internal activity was done on the AER, employee decontamination procedures were followed, and other general safety guidelines were practiced.

Regional Office

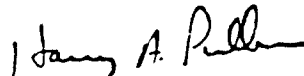
IT Corporation • 312 Directors Drive • Knoxville, Tennessee 37923 • 615-690-3211

Page 2  
Mr. Darrell Derrington  
August 20, 1985

3. The decontamination plan for equipment was not followed. Due to this, there was potential for contamination of other equipment that had been decontaminated, contamination of clean areas, and undue potential exposure for employees. Several adjustments have to be made in order to assure that the equipment was properly decontaminated.

If you have any questions, please call me.

Sincerely,

  
Harry A. Pullum, CIH  
Manager, Health and Safety

jn

INDUSTRIAL HYGIENE SAMPLES  
Huber AER Test Run

Sample Number	Location	Date Sampled	Picograms (pg) TCDD Per Sample	Volume Sampled, Cubic Meters	Results, Picograms Per Cubic Meter
J4029	Area sample - Huber Trailer inside door (reactor discharge door) opposite control panel	6/22/85	None detected at 630 pg	13.08	Less than 48.2
J4030	Personnel Sample - James McMahan - Operator at Reactor Discharge	6/22/85	None detected at 600 pg	2.81	Less than 213.5
J4031	Personnel Sample Dale Schramm AER Operator at Reactor Feed Area	6/22/85	None detected at 750 pg	2.73	Less than 274.7
J4032	Field Blank	6/22/85	None detected at 1300 pg	-	-

Lab: IAS-Knoxville  
 Case No.: AIC-100-1  
 Batch/shipment No.: GWT/Port IH

Report Date: 8/14/85  
 Column: 6UM 512330

Sample No.	Extra Cleanup	mg/sample	ICUB Reas.	DL	Instr.	GC/MS Analysis		Time	Rel. Ion Abund.		PPB Surrogate		Relative Ion Abundance		(15 X Comm)
						Date	DL		320/322	332/334	Meas.	% Acc'y	322	328*	
J4012	Yes	1.3	ND	1.3	4500	6/03/85	12:48	0.83	0.87	347878	959808	1172710	96		
J4013	Yes	0.35	ND	0.35	4500	6/03/85	12:54	0.87	0.83	311856	71216	87072	62		
J4014	Yes	1.3	ND	1.3	4500	6/14/85	13:25	0.83	0.81	357160	965088	1175930	96		
J4015	Yes	1.7	ND	1.7	4500	6/03/85	13:33	0.81	0.85	201888	696672	858304	63		
J4016	Yes	0.76	ND	0.76	4500	6/03/85	14:02	0.85	0.86	315640	908203	1105350	87		
J4017	Yes	1.0	ND	1.0	4500	6/04/85	14:47	0.86	0.82	292704	88208	102992	67		
J4018	Yes	0.61	ND	0.61	4500	6/03/85	15:54	0.67	0.79	302089	807904	907040	81		
J4019	Yes	1.2	ND	1.2	4500	6/03/85	15:16	0.85	0.83	357187	947704	1149310	78		
J4020	Yes	0.95	ND	0.95	4500	6/04/85	14:37	0.83	0.80	371312	984840	1205570	88		
J4021	Yes	1.2	ND	1.2	4500	6/21/85	11:18	0.85	0.85	205346	737096	896680	72		
J4022	Yes	0.82	ND	0.82	4500	6/21/85	12:29	0.85	0.82	287735	772018	895659	75		
J4023	Yes	1.2	ND	1.2	4500	6/21/85	11:05	0.85	0.82	392480	963414	117990	83		
J4024	Yes	5.9	ND	5.9	4500	6/21/85	13:11	0.85	0.84	278180	701341	858445	78		
J4025	Yes	0.58	ND	0.58	4500	6/21/85	13:47	0.86	0.84	269591	698760	849682	86		
J4026	Yes	0.12	ND	0.12	4500	6/21/85	14:37	0.82	0.82	327886	783904	964752	88		
J4027	Yes	0.63	ND	0.63	4500	6/21/85	10:29	0.84	0.84	337296	92248	110772	91		
J4028	Yes	0.60	ND	0.60	4500	6/27/85	19:22	0.80	0.81	265051	666296	820320	86		
J4029	Yes	0.75	ND	0.75	4500	6/27/85	23:01	0.81	0.81	217864	545776	673072	84		
J4030	Yes	1.3	ND	1.3	4500	6/27/85	18:56	0.81	0.81	301376	764377	941600	79		
J4031	Yes	1.7	ND	1.7	4500	6/27/85	19:46	0.78	0.78	355552	904448	1113810	91		
J4032	Yes	1.6	ND	1.6	4500	6/30/85	21:21	0.81	0.81	176104	418503	524728	61		
Tulank 351	Yes	1.3	ND	1.3	4500	6/04/85	10:43	0.80	0.80	296144	789136	966608	69		
Tulank 352	Yes	1.6	ND	1.6	4500	6/03/85	12:01	0.80	0.80	395526	1059350	1293060	88		
Tulank 369	Yes	1.3	ND	1.3	4500	6/23/85	20:05	0.84	0.84	230152	579984	713090	83		

\*Corrected for contribution by native ICUB; 0.9% of m/z 322 subtracted.

Recovery of spike (13C-ICUB) on IH tube = 51%.

Recovery of spike (13C-ICUB) on IH tube = 67%.

Recovery of spike (13C-ICUB) on IH tube = 60%.

- MB - Reagent Blank
- P - Partial Scan/Confirmatory Analysis
- N - Native ICUB Spike
- D - Duplicate/Fortified Field Blank
- FB - Field Blank
- ND - Not Detected
- DL - Detection Limited

D357:UC-AF-12

APPENDIX L

MEDICAL EXAMINATION CERTIFICATION FOR AER OPERATING PERSONNEL

# J. M. Huber Corporation

P. O. Box 2831

Borger, Texas 79007

☎ 061 274-8331  
TELEX 73-8458

September 6, 1985

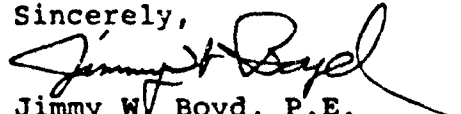
Harry D. Williams  
EG&G Idaho, Inc.  
P. O. Box 1625  
Idaho Falls, Idaho 83415

Dear Harry:

Enclosed is a letter from Dr. Richard Rehm certifying that there were no significant medical changes in the Huber personnel who participated in the Gulfport demonstration at the Naval Command Battalion Center Site. As the letter indicates, he found no evidence of any toxic material exposure or ill effects from this operation.

I trust that this will be sufficient information for your records. Should you have any questions, please feel free to contact me.

Sincerely,



Jimmy W. Boyd, P.E.  
Manager Environmental Compliance

pjh

Enclosure

xc: Jack Clem  
EG&G File

AMARILLO INDUSTRIAL HEALTH CENTER  
2400 LINE AVENUE  
AMARILLO, TEXAS 79106

ASSOCIATES  
PETER G. FAGAN, M.D.  
RICHARD D. REHM, M.D.

TELEPHONE  
AC 808-372-1135

September 3, 1985

Mr. Dick Shaw  
J. M. Huber Corp.  
P.O. Box 2831  
Borger, TX 79007

Dear Mr. Shaw:

I have completed reviewing the laboratory reports, histories, and photographs of those Huber employees who were involved in the Gulf port operation. Comparing these results to those of the initial examinations, I do not find any significant medical changes. There is no evidence of any toxic material exposure or any ill effects from this operation.

Once again, we enjoy the opportunity to work with Huber and its employees. Thank you.

Sincerely,



Richard D. Rehm, M.D.

RR/kd

CC: Darrell Derrington



APPENDIX M

ITC WIPE SAMPLING PROCEDURES

APPENDIX M  
ITC WIPE SAMPLING PROCEDURES

The standard operating procedures established for collecting wet wipe samples are as follows:

a. Materials and Apparatus

- 3 inch x 3 inch sterile cotton gauze pads, individually wrapped
- Sample bottles, glass with Teflon-lined caps
- Hexane (pesticide grade)
- Distilled water
- Glass graduated cylinder
- Sample labels
- Sample log and chain-of-custody records
- Indelible ink pen
- Ruler or square area guide
- Tape or other marking material to outline wipe area

b. Select area for collecting a series of wet wipe samples for a matrix type. Ensure surface area is sufficient to collect all required samples.

c. Mark the location of the wipe on the item(s).

d. To collect a wipe sample, use the following procedure:

- Put on a clean pair of disposable gloves
- Remove a pad from its individually wrapped package
- Hold pad in hand
- Soak pad with 8 ml of hexane using the graduated cylinder to measure volume. Fill cylinder from laboratory squeeze bottle.

- e. Sample an area by applying pressure to the pad, then drawing it across the area in both directions, ensuring that the entire area is well contacted.
- f. Upon completion of the wet wipe sample, carefully fold the pad over at least twice, being careful not to touch the contaminated side of the wipe pad, and place in labeled sample collection bottle. Bottles should be temporarily stored in plastic bags until all samples have been collected.
- g. The sampling person in charge of field data should ensure the following information is accurately recorded:
  - Sample description/item description
  - Sample date and time
  - Area date and time
  - Area sampled
  - Observations/problems, if pertinent
  - Names of sampling personnel
- h. Change gloves after taking each sample.
- i. Upon removal of samples from the site, a Chain-of-Custody form shall be established for the samples. The Chain-of-Custody will act as a transmittal form from sampling personnel to laboratory personnel and will be signed at this time to document that samples are properly delivered and received by appropriate staff members.

APPENDIX N

COPIES OF UNIFORM HAZARDOUS WASTE SHIPMENT FORMS FOR  
SHIPMENTS THAT INCLUDED DIOXIN-CONTAMINATED WASTES  
FROM AER3 NCBC TESTS

	<u>Page</u>
Exhibit 1. Waste Agreement Letter .....	151
Exhibit 2. Uniform Hazardous Waste Manifest, June 6, 1985 .....	154
Exhibit 3. Uniform Hazardous Waste Manifest, June 28, 1985 .....	156



# ecology and environment, inc.

195 SUGG ROAD, P.O. BOX D, BUFFALO, NEW YORK 14225, TEL. 716-632-4491, TELEX 91-9183

International Specialists in the Environment

May 22, 1985

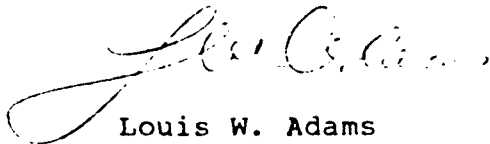
Naval Construction Battalion Center  
Code Orange  
Gulfport, Mississippi 39501  
ATTN: Harry Williams  
864-0056

Dear Mr. Williams:

Ecology and Environment, Inc. (E & E) has been conducting sampling at the Naval Construction Battalion Center and Eglin Air Force Base for dioxin under a contract to EG&G Idaho, Inc. During this sampling, sampling wastes consisting of protective clothing, sampling equipment (spoons, drills, aluminum trays, etc.), and drill borings (soil) have been generated. The sampling wastes were packaged in fiber drum containers and left on the respective sites. These wastes are now being processed for disposal by Rollins Environmental Services, Inc.

Paragraph 6 of Rollins form 101-81 (attached) presents a list of chemicals unacceptable to Rollins. None of the chemicals listed in paragraph 6 are in the fiber drums filled by E & E. It is further noted that the requirement for steel drums has been changed by Rollins to fiber drums.

Sincerely,



Louis W. Adams  
Project Manager

Copy available to D.L.  
Please fully legible copy



# Rollins Environmental Services (TX) Inc.

P.O. Box 808, Deer Park, Texas 77636 (713) 678-6001

101-81

RES Ref. No. \_\_\_\_\_

This letter, upon receipt by Rollins Environmental Services (TX) Inc. ("RES"), of your acceptance, shall be the agreement between RES and \_\_\_\_\_ ("Company") with respect to Waste (defined below), term, price and representations

**WARRANTY.** RES. To comply with all existing laws, ordinances and regulations of the United States and of any state, county, township or another subdivision thereof, or other governmental agency which may be applicable to the removal of Waste. RES shall obtain all permits, licenses and other forms of documentation required in order to comply with such laws and regulations.

**RES INDEMNIFICATION.** Following loading and departure from Company's plant, if RES provides transportation or, following delivery f.o.b. RES' facility, if Company provides transportation, Company shall be relieved of responsibility and RES shall become solely responsible for any and all loss, damage or injury to persons or property and RES shall indemnify and hold Company harmless from any and all liability, damages, costs, claims, demands, and expenses of whatever type or nature, including, but not limited to, pollution or other damage, which shall be caused by, arise out of, or in any manner be connected with the Waste, except as provided in COMPANY INDEMNIFICATION below.

**COMPANY WARRANTIES.** Company represents and warrants that the Waste loaded and removed under this Agreement shall be the Waste defined on Schedule "A", attached hereto and made a part hereof, and has been thoroughly characterized on the waste data sheet submitted to RES. Company agrees to prepare and execute RES' waste data sheet for each shipment of Waste. If the Waste is packaged, Company warrants that such Waste shall be prepared for shipment and packaged in containers specified by the then current and applicable regulations of the United States Department of Transportation, Environmental Protection Agency or any successors thereof and/or any state, municipal and/or Federal agency having jurisdiction, as the case may be. Company shall be responsible for properly packaged Waste on RES' trailers if RES is providing transportation.

**COMPANY INDEMNIFICATION.** Company will indemnify and hold harmless RES from any and all loss, damages, including damage or undue wear and tear to equipment, claims, suits, or costs which shall arise or grow out of any injury to any person or persons or any property (including the person or property of Company or its employees) caused by or resulting in any way from Company's failure to comply with Company's Warranty concerning the Waste. Company shall be responsible for and indemnify RES against any and all liability, damages, costs, claims, demands, and expenses of whatever type or nature resulting from the acts and/or omissions of Company and/or its employees, until departure of RES vehicles from Company's plant, if RES provides transportation or, if Company provides transportation, until delivery f.o.b. RES' facility.

1. **TERMINATION.** Either party to the right of either party to terminate this Agreement at any time upon thirty (30) days prior written notice. This Agreement shall automatically terminate on \_\_\_\_\_

2. **PAYMENT.** RES shall invoice Company for the hauling and treatment of Waste at the rates and terms set forth on Schedule "A" attached hereto and made part hereof. RES shall add an amount equal to one and one-half percent (1½%) of the maximum legally permissible amount on invoices which remain unpaid for more than thirty (30) days after date of invoice. Like charges may be made for each subsequent thirty (30) day period that such invoice remains unpaid.

3. **RES REJECTION.** Company understands and agrees that RES, upon notice to Company, has the absolute and unqualified right to reject any shipment of Waste which does not conform to the description of Schedule "A" (the "Waste Data Sheet") supplied by Company to RES. After any such rejection, RES will, with Company's assistance and approval, pursue all other reasonable means of disposal. If the Waste is rejected, Company shall be obligated (a) to pay the cost of transportation to RES' facility if such transportation was performed by RES, and (b) to pay the cost of return transportation from RES' facility to Company's premises (Company having the right to select the carrier) and (c) to pay all other reasonable charges incurred by RES with the prior consent of Company.

4. **TITLE.** Following loading and departure from Company's plant, if RES provides transportation or, following delivery f.o.b. RES' facility, if Company provides transportation, Company shall be relieved of title responsibility and risk of loss for the Waste, and RES shall take title, responsibility and risk of loss. However, title, risk of loss and all other incidents of ownership to non-conforming Waste shall be deemed to revert in the Company at the time revocation of acceptance is communicated to Company and RES shall be responsible for its own negligence or willful acts.

**FORCE MAJEURE.** Delays or failure of either party in the performance of its required obligations shall be excused if caused by circumstances beyond the reasonable control of the party affected, including but not limited to, acts of God, strikes, labor holiday, fire, flood, windstorm, explosion, riot, war, sabotage, action or request of governmental authority, accident, inability to obtain material, equipment or transportation, provided that a prompt notice of such delay is given and the parties shall be diligent in attempting to remove such cause(s)



those specified below:

2-acetylaminofluorene, Chemical Abstracts Service Registry No. 62759	1%
alpha-naphthylamine, Chemical Abstracts Service Registry No. 134327	1%
4-aminodiphenyl, Chemical Abstracts Service Registry No. 92671	0.1%
4-aminodiphenylamine, Chemical Abstracts Registry No. 92875	0.1%
beta-naphthylamine, Chemical Abstracts Service Registry No. 91598	0.1%
beta-propiolactone, Chemical Abstracts Service Registry No. 57578	1%
bis-chloromethyl ether, Chemical Abstracts Service Registry No. 542881	0.1%
3,3'-dichlorobenzidine, Chemical Abstracts Service Registry No. 91941, and its salts	1%
4-dimethylaminoazobenzene, Chemical Abstracts Service Registry No. 60117	1%
ethyleneimine, Chemical Abstracts Service Registry No. 151564	1%
methyl chloromethyl ether, Chemical Abstracts Service Registry No. 107302	0.1%
4,4'-methylene bis (2-chloroaniline), Chemical Abstracts Service Registry No. 101144	1%
4-nitrobiphenyl, Chemical Abstracts Service Registry No. 92933	0.1%
N-nitrosodimethylamine, Chemical Abstracts Service Registry No. 62759	1%
polychlorinated biphenyls	0.005%

Additions may be made by RES to the foregoing list of substances from time to time, such additions by RES becoming effective and binding after three days' written notice to Company.

Company agrees that all Waste containing asbestos (including actinolite, amosite, anthophyllite, chrysotile, crocidolite, and tremolite) fibers longer than 5 micrometers detectable by phase contrast microscopy shall be subject to the following conditions:

- a. The presence of asbestos in the Waste shall be clearly noted on RES' waste data sheet.
- b. Waste shall be packaged in closed steel drums bearing a label which conforms with 29 CFR 1910.1001.

Company further represents and warrants that, to the best of its knowledge, Waste does not contain vinyl chloride monomer in a liquid or gaseous form except as specified on RES' waste data sheet.

All previous representations, including but not limited to, proposal(s), purchase order(s) and/or invoice(s), either written or oral are hereby annulled and superseded. No modification shall be binding unless in writing and executed by RES and Company.

Please indicate your agreement to the above recitals by executing and returning a copy of this letter.

ACCEPTED this 10 day of June, 19 85

HQ AFESC / RDVW ("Company")

BY Terry L. Stoddart

Address Capt USAF RSC  
Project Manager

ROLLINS ENVIRONMENTAL SERVICES (TX) INC. ("RES")

BY: \_\_\_\_\_

Address: P.O. Box 609  
Deer Park, Texas 77536

Copy available to DTIC does not  
permit fully legible reproduction



*[Handwritten signature or scribble]*

print or type (Form designed for use on elite (12-pitch) typewriter.)

Form Approved OMB No. 2000-0404. Expires 7-31-88

<b>UNIFORM HAZARDOUS WASTE MANIFEST</b>	1. Generator's US EPA ID No. MS 2170022626	Manifest Document No. EG1050-3	2. Page 1 of	Information in the shaded areas is not required by Federal law.
---	---	-----------------------------------	--------------	---

3. Generator's Name and Mailing Address United States Navy Naval Construction Battalion Center Gulfport, MS 39501		A. State Manifest Document Number	
4. Generator's Phone (601) 865-2484		B. State Generator's ID 99928	
5. Transporter 1 Company Name Tri State Motor Transit Co., Inc.		C. State Transporters ID	
6. US EPA ID Number MOD 095038998		D. Transporter's Phone 800-641-7528	
7. Transporter 2 Company Name		E. State Transporter's ID	
8. US EPA ID Number		F. Transporter's Phone	
9. Designated Facility Name and Site Address Rollins Environmental Services 2027 Battleground Road Deer Park, Texas 71536		G. State Facility's ID	
10. US EPA ID Number TXD 055141378		H. Facility's Phone (713) 479-6001	

GENERATOR	11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	12. Containers		13. Total Quantity	14. Unit Wt/Vol	15. Waste No.
		No.	Type			
a	X Hazardous Waste Solid N.O.S. ORM-E NA9189	40	DF DM TS	3,113	PTTS	D017
b						
c						

J. Additional Descriptions for Materials Listed Above	K. Handling Codes for Wastes Listed Above
---	---

17. Special Handling Instructions and Additional Information  
 Disposable clothing and/or sampling equipment contained in the drums may be contaminated with 2,4,5-trichlorophenol or its pesticide derivative (TCDD). Do not open or reuse containers. Handle with care.

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packaged, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national governmental regulations.

Printed/Typed Name CLUFF, JAMES H.	Signature <i>James H. Cluff</i>	Date Month Day Year 6 26 85
Transporter 1 Acknowledgement of Receipt of Materials	Signature <i>H. Staudt</i>	Date Month Day Year 6 26 85
Printed/Typed Name TRI-STATE H. STAUDT	Signature	Date Month Day Year
Transporter 2 Acknowledgement of Receipt of Materials	Signature	Date Month Day Year

19. Discrepancy Indication Space

20. Facility Owner or Operator Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.		Date
Printed/Typed Name 154	Signature	Month Day Year

GENERATOR  
TRANSPORTER  
FACILITY



*State, Motor*

*USMC, East MS*

INVENTORY NUMBER

16 CONTRACT NUMBER

ITEM	DESCRIPTION OF MATERIAL	UNIT	QUANTITY RELEASED
1.	<p><i>Herz - Waste</i></p> <p><i>Gen VAN</i></p> <p><i>excorted to East Gate and Reported NCB</i>  <i>0955 hr 6/26/85</i>  <i>T. Stoddart</i></p>	1	T/C

SHIPMENT NUMBER	9A. "X" TYPE OF SHIPMENT <input type="checkbox"/> PARTIAL <input checked="" type="checkbox"/> FINAL	10. TIME LOADED	11. VEHICLE LICENSE NO.
12. RELEASED BY (Signature of PDO or Authorized Representative) <i>Black</i>		13. TITLE OF AUTHORIZED DISPOSAL REPRESENTATIVE <i>Traffic BS</i>	
4. SIGNATURE OF PURCHASER OR AGENT <i>T. Stoddart Capt USAF</i>		15. DATE PROPERTY IS RELEASED <i>6/26/85</i>	
6. TIME SHIPMENT LEAVES INSTALLATION		17. BENTRY'S INITIALS	

TO BE COMPLETED BY SECURITY PERSONNEL ONLY

3. Generator's Name and Mailing Address <b>United States Navy Naval Construction Battalion Center ATTN CODE 470 Gulfport, MS 39501 Generator's Phone (601) 865-2684</b>		A. State Manifest Document Number
6. US EPA ID Number <b>MO0 09038990</b>		B. State Generator's ID <b>84928</b>
7. Transporter 1 Company Name <b>Tri State Motor Transit Co., Inc.</b>		C. State Transporter's ID
8. US EPA ID Number		D. Transporter's Phone <b>800-841-7800</b>
9. Designated Facility Name and Site Address <b>Rollins Environmental Services 2027 Battleground Road Deer Park, Texas 71526</b>		E. State Transporter's ID
10. US EPA ID Number <b>TXD 055141378</b>		F. Transporter's Phone
		G. State Facility's ID
		H. Facility's Phone <b>(713) 679-0201</b>

11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	12. Containers		13. Total Quantity	14. Unit Wt/Vol	15. Waste No.
	No.	Type			
<b>X</b> Hazardous Waste Solid H.O.S. CM-E RA9189	31	DF	6,265	P	8017

J. Additional Descriptions for Materials Listed Above	K. Handling Codes for Wastes Listed Above
---	---

15. Special Handling Instructions and Additional Information  
**Disposable clothing and/or sampling equipment contained in the drums may be contaminated with 2,4,5-trichlorophenol or its pesticide derivative (TCDD). Do not open or reuse containers. Handle with care.**

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national governmental regulations.

Printed/Typed Name <b>CLIFF, JAMES H</b>	Signature <i>[Signature]</i>	Date Month Day Year <b>6 24 85</b>
---	---------------------------------	--

17. Transporter 1 Acknowledgement of Receipt of Materials	Date
Printed/Typed Name	Month Day Year
Signature	

18. Transporter 2 Acknowledgement of Receipt of Materials	Date
Printed/Typed Name	Month Day Year
Signature	

19. Discrepancy Indication Space

20. Facility Owner or Operator Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.	Date
Printed/Typed Name	Month Day Year
Signature	

Copy available to DTIC does not permit fully legible reproduction

GENERATOR COPY

SHIPPER'S NUMBER  
 1000

NAVY  
 M.S.

DATE SHIPPED 8-25

ORIGINATING CARRIER  
 CONSIGNEE TO (Mail or street address of consignee - for purposes of notification only)  
 HOLLANDS - F. NAVY'S MATERIAL SERVICE  
 DESTINATION STATE COUNTY  
 DELIVERY ADDRESS (To be filled in only when shipper desires and governing tariffs provide for delivery thereat)  
 DIRECTING CARRIER (ST DELIVERING CARRIER TRACTOR NO. TRAILER NO.)

EXECUTIVE OFFICES  
 PO BOX 113, JOPLIN, MO  
 Subject to Section 7 of and of applicable bill of lading, if shipment is to be delivered to consignee without return or consignor, the consignor shall the following statement: The carrier shall not make del of this shipment without paying freight and all other lawful due

NUMBER PACKAGES	KIND OF PACKAGE, DESCRIPTION OF ARTICLES, SPECIAL MARKS, AND EXCEPTIONS	WEIGHT (Subject to Correction)	CLASS OR RATE	CR. COL.	(Signature of consignor)
1	...	...	...	...	...
...	...	...	...	...	...

If charges are to be prepaid or stamp here "To be Prepaid"  
 Rec'd \$  
 apply in prepayment of the due on the property described hereon  
 Agent or Cashier  
 Per  
 (The signature here authorizes only the amount prepaid)

LENGTH	WIDTH	HEIGHT	TOTAL FEET OF TRAILER SPACE OCCUPIED
...	...	...	...

UNLESS A GREATER VALUE IS DECLARED, THE SHIPPER HEREBY RELEASES THE VALUE TO \$5000.00 PER TON OF 2000 POUNDS FOR EACH ARTICLE THIS IS TO CERTIFY THAT THE ABOVE NAMED MATERIALS ARE PROPERLY CLASSIFIED, DESCRIBED, PACKAGED, MARKED, AND LABELED, AND ARE IN PROPER CONDITION FOR TRANSPORTATION, ACCORDING TO THE APPLICABLE REGULATION OF THE DEPARTMENT OF TRANSPORTATION

UNLESS OTHERWISE NOTED VEHICLE CONTAINS HAZARDOUS MATERIALS PROPERLY PLACARDED IN ACCORDANCE WITH 49CFR172.506.

ARRIVED AT SHIPPER	DATE	TIME	PREARRANGED SCHEDULE	DATE	TIME	LOADING STARTED	DATE	TIME
...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...

1ST TIME CARGO CHANGED CUSTODY			2ND TIME CARGO CHANGED CUSTODY			4TH TIME CARGO CHANGED CUSTODY		
DATE	TIME	TRACTOR NO.	DATE	TIME	TRACTOR NO.	DATE	TIME	TRACTOR NO.
...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...

ARRIVED AT DESTINATION	DATE	TIME	PREARRANGED SCHEDULE	DATE	TIME	UNLOADING STARTED	DATE	TIME
...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...

RECEIVED THE ABOVE DESCRIBED PROPERTY (The reverse of this page is blank.)

APPENDIX 0

ECOLOGY AND ENVIRONMENT, INC. SAMPLING PROTOCOL  
FOR AER3 NCBC TEST

## APPENDIX O

### SAMPLING PROTOCOL

A bound sampling logbook will be individually assigned to each site. The logbook will be kept by the on-site sampling team. Field data sheets will also be used. At a minimum, the following information will be kept in the logbook and on the field data sheets:

- o Site name
- o Demonstration project name
- o Test run number
- o Sample test point number
- o Sample number
- o Date and time sampled
- o Ambient air temperature
- o General weather conditions
- o Name of sampler
- o Name of laboratory performing the analysis
- o Date sample was shipped
- o Airbill number.

To enhance decontamination, a small plastic bag will be placed around the outside of the sample bottles and held in place by a rubber band so that any sample spilled during the collection or compositing process will not contaminate the outside of the jar. After sample collection, the sample bottles will be decontaminated by first removing and discarding the rubber band and outer plastic bag. The sample bottle will be washed with clean water, dried, and placed in a clean bag; then the bag will be sealed.

Soil samples will be collected in disposable aluminum trays, using stainless steel scoops or spoons. Enough soil will be collected to fill two 16-ounce wide-mouth jars. Pretreated soils will be sampled as they are fed into the furnace feed bins. Treated soil exits the furnace at extremely high temperatures and will be sampled after it has cooled to

ambient air temperature. All soil samples will be composites of at least five aliquots. They will be sieved into the wide-mouth jars through 10-mm screen and the jars will be sealed with aluminum lined caps.

Representative carbon filter bed samples will be collected during the equipment purging and cleaning cycle. They will be placed in two 16-ounce wide-mouth jars and sealed with aluminum-lined caps.

APPENDIX P

PACKING LISTS FOR HUBER TEST-RELATED SAMPLES BEING  
SHIPPED TO ANALYTICAL LABORATORIES

	<u>Page</u>
Exhibit 1. Cooler No. 5 .....	165
Exhibit 2. Cooler No. 7 .....	168
Exhibit 3. Cooler No. 11 .....	172
Exhibit 4. Cooler No. 12 .....	176



PACKING LIST

Cooler No.: NCBC #5  
 Date Shipped: 6/17/85  
 Federal Express Airbill No.: 353-895-964

TO: California Analytical Laboratories, Inc.  
 2544 Industrial Blvd.  
 West Sacramento, CA 95691

FROM: USAF Sampling & Analytical Program  
 Environmental Restoration & Technology  
 Research & Test Evaluation Project

EG&G Idaho, Inc.  
 Code Orange/USAF Project Trailer  
 Naval Construction Battalion Center  
 Gulfport, MS 39501  
 601/864-0056

Ref: RFP # C85-130686

Quant. Sample # / Description

-----

1	IT-NCBC-R1-04 / IT PHOTOLYSIS RUN 1 SOLVENT AFTER TREATMENT
1	IT-NCBC-R2-03 / IT PHOTOLYSIS RUN 2 SOLVENT BEFORE TREATMENT
1	IT-NCBC-R2-09A / IT PHOTOLYSIS RUN 2 REAR PART OF 1st GAS CARBON
1	IT-NCBC-R2-27 / IT STACK TEST IN-LINE PARTICULATE FILTER
1	IT-NCBC-R2-28 / IT STACK TEST FIELD BLANK PARTICULATE FILTER
1	IT-NCBC-R2-35 / IT STACK TEST KOH (IMPINGER #3)
1	IT-NCBC-R2-36 / IT STACK TEST KOH FIELD BLANK
1	EE-NCBC-R1-01 / HI-VOL SAMPLER #1, RUN 1 OFF-SITE CONTROL FILTER
1	EE-NCBC-R1-02 / HI-VOL SAMPLER #2, RUN 1 ON-SITE CONTROL FILTER
1	EE-NCBC-R1-03 / HI-VOL SAMPLER #3, RUN 1 ON-SITE DOWNWIND FILTER
1	EE-NCBC-R1-04 / HI-VOL SAMPLER #4, RUN 1 OFF-SITE DOWNWIND FILTER

-----

11 TOTAL SAMPLES

Sample # IT-NCBC-R1-04 taken 6/7/85 at 0430. 1, 1/2 gallon jug. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
 DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans DL < = 0.1



Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organic indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Suspended solids content.

Sample # IT-NCBC-R2-03 taken 6/14/85 at 1800. 1, 16 oz jar. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Sample # IT-NCBC-R2-09A taken 6/13/85 at 1950. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above test result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Total amount of carbon.

Sample #'s IT-NCBC-R2-27 and IT-NCBC-R2-28 taken between 2300 on 6/12/85 and 0500 on 6/13/85, 1, 16 oz jar each. 1, petri dish each. No preservatives. IT-NCBC-R2-27 has an initial tare of 0.2533 g. IT-NCBC-R2-28 has an initial tare of 0.2543 g.

Analyze for: Particulate loading DL < = 0.0001 gr/scf.

Sample #'s IT-NCBC-R2-35 and IT-NCBC-R2-36 taken between 2300 on 6/12/85 and 0500 on 6/13/85. 1, 16 oz jar each. No preservatives.

Analyze for: Hydrogen chloride DL < = 1.0 ppm.

Sample #'s EE-NCBC-R1-01, EE-NCBC-R1-02, EE-NCBC-R1-03, and EE-NCBC-R1-04. Each contains a plastic bag with a particulate filter in a folder. Initial tares are listed below:

EE-NCBC-R1-01 (filter # 1131): 3.03360 g.  
EE-NCBC-R1-02 (filter # 1132): 3.02484 g.  
EE-NCBC-R1-03 (filter # 1133): 2.98913 g.  
EE-NCBC-R1-04 (filter # 1134): 3.03537 g.

Volumes of air passing through each filter are as follows:

EE-NCBC-R1-01: 4845.99 cu m  
EE-NCBC-R1-02: 4635.54 cu m (estimate)  
EE-NCBC-R1-03: 4845.99 cu m  
EE-NCBC-R1-04: 2877.06 cu m

Analyze for: Total suspended particulates.

2,3,7,8-TCDD DL < = 0.1 ppb.

PACKING LIST

Cooler No.: NCBC #7  
 Date Shipped: 6/17/85  
 Federal Express Airbill No.: 353-895-964

TO: California Analytical Laboratories, Inc.  
 2544 Industrial Blvd.  
 West Sacramento, CA 95691

FROM: USAF Sampling & Analytical Program  
 Environmental Restoration & Technology  
 Research & Test Evaluation Project

EG&S Idaho, Inc.  
 Code Orange/USAF Project Trailer  
 Naval Construction Battalion Center  
 Gulfport, MS 39501  
 601/C64-0056

Ref: RFP # C85-130686

Quant. Sample # / Description

1	HU-NCBC-R1-02 / HUBER RUN 1 SOIL AFTER TREATMENT
1	IT-NCBC-R2-02 / IT RUN 2 SOIL AFTER TREATMENT
1	IT-NCBC-R2-09 / IT RUN 2 FRONT PART OF 1st GAS CARBON BED
1	HU-NCBC-R1-09 / HUBER RUN 1 FIRST GAS CARBON DRUM
1	HU-NCBC-R1-09A / HUBER RUN 1 SECOND (DOWNSTREAM) GAS CARBON DRUM
1	IT-NCBC-R3-02 / IT RUN 3 SOIL AFTER TREATMENT
-----	
6	TOTAL SAMPLES

Sample HU-NCBC-R1-02 taken 7/14/85 at 0220. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
 DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
 DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # IT-NCBC-R2-02 taken 6/13/85 at 0624. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # IT-NCBC-R2-09 taken 6/13/85 at 1950. Contains 2, 16 oz jars. No preservative

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Sample # HU-NCBC-R1-09 taken 6/14/85 at 0315. Contains 2, 16 oz bottles. No preservative

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Hydrogen chloride DL < = 1 ppm.

Nitrogen oxide DL < = 1 ppm.

Sample # HU-NCBC-R1-09A taken 6/14/85 at 0315. Contains 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

Total amount of carbon present.

Hydrogen chloride DL < = 1 ppm.

Nitrogen oxide DL < = 1 ppm.

Sample # IT-NCBC-R3-02 taken 6/14/85 at 0330. Contains 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chlorodibenzo-p-dioxins  
DL < = 0.1 ppb.

Total isomers of tetra-, penta-, & hexa-chloro-dibenzofurans  
DL < = 0.1 ppb.

Modified CAG list DL < = 10 ppb.

Modified PPL list DL < = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) DL < = 10 ppb.

If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Packing List

Cooler No. 11

Date Shipped: 6/28/85

Federal Express Airbill No.: 353 895 942

TO: California Analytical Laboratories, Inc.  
2544 Industrial Blvd.  
West Sacramento, CA 95631

FROM: USAF Sampling & Analytic Program  
Environmental Restoration & Technology  
Research & Test Evaluation Project

EG&G Idaho, Inc.  
Code Orange/USAF Project Trailer  
Naval Construction Battalion Center  
Gulfport, MS 39501  
601/964-0056

Ref: RFP # C85-130-130686

Quant. Sample # / Description

1	HU-NCBC-R1-01 / SOIL BEFORE DESTRUCTION PROC. (HUBER RUN #1)
1	HU-NCBC-R1-01 / SOIL BEFORE DESTRUCTION PROC. (HUBER RUN #2)
1	HU-NCBC-R2-02 / SOIL AFTER DESTRUCTION PROC. (HUBER RUN #2)
1	HU-NCBC-R2-09 / HUBER RUN #2 FIRST GAS DRUM
1	HU-NCBC-R2-09A / HUBER RUN #2 SECOND GAS DRUM
1	HU-NCBC-R2-03 / HUBER RUN #2 BAGHOUSE PARTICULATE
1	EE-NCBC-R2-01 / HI VOL. SAMPLER #1, RUN #2 OFF-SITE CONTROL
1	EE-NCBC-R2-02 / HI VOL. SAMPLER #2, RUN #2 ON-SITE CONTROL
1	EE-NCBC-R2-03 / HI VOL. SAMPLER #3, RUN #2 ON-SITE DOWNWIND
1	EE-NCBC-R2-04 / HI VOL. SAMPLER #4, RUN #2 OFF-SITE DOWNWIND
1	EE-NCBC-R3-01 / HI VOL. SAMPLER #1, RUN #3 OFF-SITE CONTROL
1	EE-NCBC-R3-02 / HI VOL. SAMPLER #2, RUN #3 ON-SITE CONTROL
1	EE-NCBC-R3-03 / HI VOL. SAMPLER #3, RUN #3 ON-SITE DOWNWIND
1	EE-NCBC-R3-04 / HI VOL. SAMPLER #4, RUN #3 OFF-SITE DOWNWIND

14 TOTAL SAMPLES

Sample HU-NCBC-R1-01 taken 6/12/85 at 2135. 2, 16 oz jars. No preservatives.

Sample HU-NCBC-R2-01 taken 6/21/85 at 0130. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chloro-dibenzo p-dioxins, to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to DL < or = 0.1 ppb.

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) to LD, or = 10 ppb.

Sample HU-NCBC-R2-02 taken 6/24/85 at 1910. 2, 16 oz jars. No preservatives.

Sample HU-NCBC-R2-03 taken 6/24/85 at 2020. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chloro-dibenzo p-dioxins to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to DL < or = 0.1 ppb.

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) to a DL < or = 10 ppb.



If the above tests result in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), then perform an EP Toxicity Test.

Sample # No's EE-NCBC-R2-01, EE-NCBC-R2-02, EE-NCBC-R2-03, EE-NCBC-R2-04, EE-NCBC-R3-01, EE-NCBC-R3-02, EE-NCBC-R3-03, EE-NCBC-R3-04, each consist of a particulate filter in a folder in a plastic bag. Initial tares are listed below.

Sample No.	Filter No.	Initial Tare	Volume Sampled
EE-NCBC-R2-01	1135	3.01042	4,547.10 <sup>a</sup>
EE-NCBC-R2-02	1136	3.01013	4,637.07 (est.)
EE-NCBC-R2-03	1137	2.92856	4,547.10 <sup>a</sup>
EE-NCBC-R2-04	1138	2.96794	4,721.23 <sup>b</sup>
EE-NCBC-R3-01	1139	2.96849	1,761.05
EE-NCBC-R3-02	1140	2.95780	1,596.16 (est.)
EE-NCBC-R3-03	1141	2.97475	1,761.05
EE-NCBC-R3-04	1143	1.97590	1,828.50

- a. Later found in error; corrected value is 4562.
- b. Later found in error; corrected value is 4735.

Analyze Above Samples For:

Total suspended particulates  
2,3,7,8-TCDD to DL < or =0.1 ppb.

Sample HU-NCBC-R2-09 taken 6/24/85 at 1940. 2, 16 oz jars. No preservatives.

Sample HU-NCBC-R2-09A taken 6/24/85 at 1900. 2, 16 oz jars. No preservatives.

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chlorodibenzo-p-dioxins. To DL < or = 0.1 ppb..

Total isomers of tetra-, penta-, and hexa-dibenzofurans to DL < or = 0.1 ppb.

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppb.

Total amount of carbon present.

Hydrogen chloride to DL < or = 1 ppm.

Nitrogen oxide to DL < or = 1 ppm.

PACKING LIST

Cooler No. 12  
Date Shipped: 6/28/85  
Federal Express Airbill No. 353-895-920

TO: Battelle Columbus Laboratories, Inc.  
Attn: Dr. David Miller  
505 King Ave.  
Columbus, OH 43201

FROM: USAF Sampling & Analytic Program  
Environmental Restoration & Technology  
Research & Test Evaluation Project

EG&G Idaho, Inc.  
Code Orange / USAF Project Trailer  
Naval Construction Battalion Center  
Gulfport, MS 39501  
601/864-0056

Quant.	Sample # / Description
1	HU-NCBC-R2-02/HUBER RUN #2, SOIL AFTER TREATMENT
1	HU-NCBC-R1-01/HUBER RUN #1, SOIL BEFORE TREATMENT
1	IT-NCBC-R2-04/IT RUN #2 SOLVENT AFTER PHOTOLYSIS
3	TOTAL SAMPLES

Analyze for: 2,3,7,8-TCDD to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-chloro-dibenzo-p-dioxins to DL < or = 0.1 ppb.

Total isomers of tetra-, penta-, and hexa-dibenzofurans to DL < or = 0.1 ppb.

Modified CAG list to DL < or = 10 ppb.

Modified PPL list to DL < or = 1 ppm.

Organics indigenous to herbicide orange (Appendix D) to DL < or = 10 ppb.

In addition:

Sample HU-NCBC-R2-02 taken 6/24/85 at 1910. 2, 16 oz jars. No preservatives. Requires that if any of the above tests results in concentrations greater than those limits set for any of the contaminants in RCRA Sec. 261.24, Table I (EP Toxicity), an EP Toxicity Test should be run.

Sample IT-NCRC-R2-04 taken 6/15/85 at 0315. 1, 80 oz amber jug. Non preserved. Requires suspended solids analyses.

Sample HU-NCBC-R1-01 taken 6/12/85 at 2135. 2, 16 oz jars. Non-preserved. Requires no additional analyses.

APPENDIX Q

REVIEW/EVALUATION OF ANALYTICAL RESULTS FOR HUBER PROCESS  
VERIFICATION SAMPLES

APPENDIX Q  
REVIEW/EVALUATION OF THE ANALYTICAL RESULTS  
FOR HUBER PROCESS VERIFICATION SAMPLES

Chemical Sciences of EG&G Idaho, Inc., had the responsibility for reviewing and evaluating all analytical data from the J. M. Huber Company process technology demonstration at the NCBC site. California Analytical Laboratories, Inc., (CAL) was selected as the subcontract laboratory for analysis of all process verification samples collected during the Huber demonstration. These samples were shipped to CAL from the NCBC site in several separate batches. Samples were received at the laboratory on June 15, June 21, and June 29, 1985.

The analytical results were transmitted by CAL in several submittals. The various submittals were as follows:

<u>Item</u>	<u>Date of Submittal</u>	<u>Description</u>
1	9/10/85	Preliminary reports on polychlorinated dibenzo-p-dioxins and dibenzofurans
2	1/21/86	Data summaries and information on analytical protocols
3	1/31/86	Results of sample submitted for EP Toxicity Test
4	3/11/86	Additional data and information plus clarification of data summaries. (This information was provided in response to requests made by EG&G Idaho during meetings with CAL on March 4 and 5, 1986.)
5	3/21/86	Additional information including a revised inorganics analysis data package, some reanalysis results for specific samples, and additional supporting information. (This information was also provided in response to EG&G Idaho requests made during the March 4 and 5, 1986, meetings with CAL.)
6	4/22/86	Results of the reanalysis of five samples for semivolatile organics.

All data submitted by CAL were included in the EG&G Idaho review/evaluation process.

After the samples were submitted to CAL, 2 to 4 months elapsed before any analyses were performed. Thus, sample holding times as dictated by EPA were exceeded by wide margins. Other problems with various portions of the CAL data are documented and discussed in detail in this appendix. Furthermore, at the time of review, there were no universally accepted data review protocols for the polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). Therefore, because of the problems and limitations of the CAL data and also the absence, in some cases, of applicable data review protocols to validate the results, EG&G Idaho considered it inappropriate to attempt to validate the results in the strict sense used by the EPA. Instead, Chemical Sciences reviewed and evaluated the results to determine if the appropriate analytical protocols were used and applied correctly, if the various calculations were correct, and if the results were consistent and logical.

Results of the EG&G Idaho review/evaluation process are presented in the following sections. Discussion has been broken down by the class of analysis performed, i.e., inorganics, volatile organics.

#### INORGANIC RESULTS

Samples from the Huber demonstration project were submitted to California Analytical Laboratory for inorganic element analysis, including cyanide, on June 21 and 29, 1985. The methods used for analysis were taken from the U.S. EPA Contract Laboratory Program (CLP) protocols. The specific instrumental techniques used are specified on the data reporting sheets.

On January 21, 1986, analytical results for inorganic elements were submitted to EG&G Idaho for review. A review of the data showed them to be in error. It appears that on the summary sheets dated January 21, 1986, the duplicate results, percent recovery, spiked sample results and relative

percent difference (RPD) values obtained on October 18, 1985, were used. In each case, however, sample results obtained on a different run were used. Therefore, none of the calculations were correct.

The above errors were pointed out to the contract laboratory, and corrected data sheets were prepared on March 19, 1986. All calculations on the new data sheets were correct. From the data submitted, it appears that proper analytical procedures were used.

As stated previously, the samples for inorganic analysis were submitted to the contract laboratory on June 21 and 29, 1985. These samples were analyzed on October 18, 1985. During this time, approximately 5 months, the samples were stored at ambient temperature and without preservatives.

The CLP protocol states that samples for cyanide analysis are to be stored at 4°C and the maximum holding time is 14 days. For mercury, the maximum holding time is 30 days. For all other metals, the maximum holding time is 6 months. It also states that for mercury and all other metals, the pH of the sample is to be adjusted to 2, with nitric acid for preservation purposes. In the protocol, no differentiation is made between liquid and solid samples.

Although the values for metals and cyanide cannot be validated, they can be used as a general guide for evaluating their fate in the Huber process.

Another area of concern is spike recovery values. Each sample was spiked with a known amount of each element being determined. The percent recovery of the spiked elements gives a measure of the extraction efficiency. The percent recovery of each spiked element should fall within 75 to 125 percent of the amount added to the sample. A review of the data shows that 22 spike recovery results, representing 36.5 percent of the values reported, were outside this target window. Twenty-one of the values were low, ranging from 0 to 74 percent spike recovery. The 0 percent



recovery result was for antimony on sample HU-NCBC-R2-09. The high result was for lead on sample HU-NCBC-R2-01. This spike recovery value was reported as 148 percent. CAL's position is that these questionable spike recovery values are normal and are completely acceptable to EPA. EG&G Idaho contacted the organization performing review of inorganic analytical results for EPA and was informed that currently no action is taken if the spike recovery values are outside the stated limits. However, EG&G Idaho feels that it is indicative of questionable analytical techniques.

#### VOLATILE ORGANIC COMPOUNDS

Various soil and carbon filter samples were analyzed for volatile organic compounds. The analytical procedures were taken from the CLP protocol and based on EPA Method 624.

The samples submitted to CAL were received by that laboratory on June 21 and 29, 1985. The samples were stored at ambient temperature and extracted on September 16, 1985, approximately 3 months after receipt. In addition, the samples for volatile organic analysis were taken about 3 weeks after the containers had been opened to take samples for semivolatile organic components.

CLP protocol states that samples for volatile organic analysis must be protected from the light and refrigerated at 4°C from the time of receipt until they are extracted. The extraction and analysis are to be done within 10 days of sample receipt.

CLP protocol further states that a 4- or 5-point calibration curve is to be prepared for each instrument used in the analysis. This calibration curve is necessary to determine the linearity of response for that instrument. No evidence could be found that any calibration curve had been prepared.

Based on all of the above, results obtained for volatile organic components are not considered valid.

#### SEMIVOLATILE ORGANIC COMPOUNDS

The semivolatile organic compounds were analyzed using the CLP protocol, which is based on EPA Method 625. The samples submitted to CAL were received at that laboratory on June 21 and 29, 1985. The extraction for semivolatile organic components was performed on August 27, 1985, approximately 2 months after receipt of the samples.

The CLP protocol states that samples for organic analysis must be protected from light and refrigerated at 4°C from the time of receipt until extracted. Solid samples must be extracted within 10 days of receipt and the extract analyzed within 40 days of extraction. The samples in question were stored at ambient temperature for 2 months prior to extraction.

As discussed with the volatile organics, preparation of a 4- or 5-point calibration curve for the analytical instrumentation used is not evident.

The analysis contract required that semivolatile organic compounds be analyzed to 1 ppm. It appears that the contract laboratory arbitrarily decided whether a given sample was analyzed as low concentration or medium concentration. On this basis, the following samples (HU-R1-02, HU-R2-02, and HU-R2-03) required reanalysis. The reanalysis was performed by taking the sample extract from a previous analysis and concentrating the extract from approximately 0.5 g/1 mL to 0.5 g/0.5 mL. Although this procedure should double the concentration and lower the detection limit by half, the reported detection limits were lowered by a factor of four. There was no evidence that sample injection size had been increased. To attain the reported detection limits, the sample injection size would have to double. One other point of concern is that the detection limit is based on the peak height versus background noise level for any given component. It is

therefore highly improbable that each component would have the same detection limit. However, the factor of four improvement in detection level was reported for all compounds.

Based on all of the above, the reported data cannot be validated. However, because of the lower volatility of semivolatile organic compounds, the length of storage time and storage conditions would not be as critical as for the volatile organic compounds. Therefore, the values obtained can probably be used as a guide for evaluating the Huber process.

#### PESTICIDE/PCB ANALYSIS

The pesticide analysis includes the chlorinated insecticides and herbicides as well as the polychlorinated biphenyls. These components were determined using the CLP protocol, which is based on EPA Method 608. One method covers both pesticides and PCBs.

The same storage conditions and time restrictions for sample extraction and analysis exist for the pesticide/PCB samples as for the semivolatile organic material. Like the other organics, a calibration curve is not evident.

Based on the above, the reported pesticide/PCB values cannot be validated. However, like the semivolatile organic components and for the same reason, the reported data can possibly be used as a guide to evaluate the Huber process.

## DIOXIN AND DIBENZOFURAN RESULTS

Two types of analyses were performed for the specified PCDDs and PCDFs: total isomer class content for tetra, penta, and hexachlorinated PCDDs and PCDFs and 2,3,7,8-TCDD isomer-specific. The review methodology was to evaluate all data in terms of applicable ion ratios, retention times, and signal-to-noise ratios to determine if the analytical results were correctly interpreted.

The isomer-specific 2,3,7,8-TCDD data were examined and evaluated, using the same criteria applied during the soil sampling and analysis program conducted previously for the USAF at the NCBC site. These criteria are detailed in the EPA document for reviewing 2,3,7,8-TCDD analytical results (Reference 1), and the criteria are listed in the annex to this appendix. The results of the evaluation will be discussed in two parts, the isomer class analyses and the isomer specific analysis. The isomer class analyses will be discussed first.

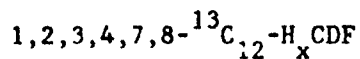
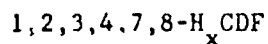
In its original proposal to EG&G Idaho, California Analytical Laboratories proposed to use EPA Method 8280 (as modified by EMSL-Las Vegas) to perform the isomer class analyses. CAL stipulated that extraction methodologies would probably require modification because of the types of sample matrices involved, and this was understood by EG&G Idaho. However, it was not felt that the modification in extraction methodology would significantly alter the remainder of the analytical technique. Upon receipt of the data from CAL and the letter describing the data (January 21, 1986), CAL stated that "the dioxin and furan analyses were performed according to methods acceptable to EPA." No mention of EPA Method 8280 was made. Based upon prior knowledge of 8280 and a review of CAL's isomer class data, it was apparent that a method other than 8280 was performed. The comment must be made that Method 8280 has been under revision for several years and that the final validated 8280 method was not completed and released by EMSL-Las Vegas until March 1986. EG&G Idaho is obtaining a copy of Method 8280 from EMSL-Las Vegas for further review.

Upon review of this document, the use of multipoint calibration curves were confirmed, which CAL did not perform. Other differences in procedure were noted between the EPA Method 8280 method and the method CAL used. However, many of these differences are due to the multiple versions of 8280 in existence. The use of multipoint curves, however, has been mandatory in all versions.

The main concern is the use of single concentrations of analytical standards to determine response factors used in quantification calculations. Method 8280 stipulates multilevel calibration standards be used to determine response factors. This is an important consideration when a wide range of concentration values are anticipated, as was the case of the Huber samples.

As opposed to Method 8280, CAL ran a single point standard on a daily basis to determine response factors for the various analytical parameters. The standard was a mixture of polychlorinated dioxins and furans, which contained the following compounds:

2,3,7,8-TCDD  
2,3,7,8-<sup>13</sup>C<sub>12</sub>-TCDD  
2,3,7,8-<sup>37</sup>Cl<sub>4</sub>-TCDD  
  
1,2,3,7,8-P<sub>5</sub>-CDD  
1,2,3,7,8-<sup>13</sup>C<sub>12</sub>-P<sub>5</sub>CDD  
1,2,3,4,7,8-H<sub>x</sub>CDD  
1,2,3,4,7,8-<sup>13</sup>C<sub>12</sub>-H<sub>x</sub>CDD  
  
2,3,7,8-TCDF  
2,3,7,8-<sup>13</sup>C<sub>12</sub>-TCDF  
  
1,2,3,7,8-P<sub>5</sub>CDF  
1,2,3,7,8-<sup>13</sup>C<sub>12</sub>-P<sub>5</sub>CDF  
2,3,4,7,8-<sup>13</sup>C<sub>12</sub>-P<sub>5</sub>CDF



CAL furnished the raw chromatograms of the standard analyses as well as their calculation sheets. These data were reviewed and calculations checked to verify numerical accuracy. Standards were either obtained commercially or manufactured by CAL. Sources of all standards were documented by CAL. The analyses were conducted using high-resolution gas chromatography/low-resolution mass spectrometry. The isomer class content analyses were conducted using a DB-5 fused silica capillary GC column 60 meters long. The temperature program used was to ramp the GC column temperature from 190°C to 305°C at a rate of 10°C/min.

The following ions were monitored to determine the presence of PCDDs and PCDFs as well as to provide information for quantification:

<u>Compound</u>	<u>Nominal Mass 1</u>	<u>Nominal Mass 2</u>	<u>Theoretical Isotope Ratio Mass 1/Mass 2</u>
TCDD	320	322	0.77
TCDD- $^{13}C_{12}$	332	334	0.77
P <sub>5</sub> CDD	354	356	0.617
P <sub>5</sub> CDD- $^{13}C_{12}$	366	368	0.617
H <sub>x</sub> CDD	390	392	1.235
H <sub>x</sub> CDD- $^{13}C_{12}$	402	404	1.235
TCDF	304	306	0.77
TCDF- $^{13}C_{12}$	316	318	0.77
P <sub>5</sub> CDF	338	340	0.617
P <sub>5</sub> CDF- $^{13}C_{12}$	350	352	0.617
H <sub>x</sub> CDF	372	374	0.514
H <sub>x</sub> CDF- $^{13}C_{12}$	386	390	2.858

The normally accepted practice is that the experimental isotope ratios should be within +15 percent of the theoretical value in order to be considered a positive indicator of the presence of a PCDD or PCDF. Upon reviewing CAL's data and documentation, it was hard to determine if this practice was followed for the isomer class data. CAL's supporting documentation focused primarily on 2,3,7,8-TCDD analysis, with little devoted to specific QA/QC criteria for the PCDD/PCDF analyses. Several Huber samples were determined to have internal standards with isotope ratios slightly outside the 15 percent boundary. As the number of analyses increases, it becomes more probable that some of the criteria will be marginal or slightly outside acceptable limits. Concern was exhibited only where exceptionally wide deviations from the 15 percent criteria were determined.

The following Huber samples were analyzed for total tetra through hexachlorinated PCDD and PCDF content:

HU-NCBC-R1-01	HU-NCBC-R1-02	HU-NCBC-R2-01
HU-NCBC-R2-02	HU-NCBC-R2-03	HU-NCBC-R1-09
HU-NCBC-R2-09	HU-NCBC-R2-09A	

In addition, a method blank and two native spikes were prepared. A duplicate sample using a Huber sample was not analyzed. The native spikes used samples HU-NCBC-R2-09A and HU-NCBC-R1-01.

The following samples were determined not to contain PCDDs or PCDFs at or above the reported detection limits: HU-NCBC-R1-02, HU-NCBC-R2-02, HU-NCBC-R1-09, HU-NCBC-R2-09A, and the method blank. Sample HU-NCBC-R1-02, displayed a 1,2,3,4,7,8- $^{13}\text{C}_{12}$ - $\text{H}_x\text{CDF}$  ratio of 3.389, which is slightly outside the 15 percent range. A more serious concern for this sample is that the M/2 404 ion for the  $\text{H}_x\text{CDF}-^{13}\text{C}_{12}$  standard was not found due to interferences. This necessitated the calculation of the detection limit vs the TCDD- $^{13}\text{C}_{12}$  internal standard. This sample had a total detection limit of 1.304 ppb, with fairly high detection limits for  $\text{H}_x\text{CDF}$ ,  $\text{P}_5\text{CDD}$ , and  $\text{H}_x\text{CDD}$ . Sample HU-NCBC-R2-02 displayed a  $\text{H}_x\text{CDD}$  internal standard

ratio in excess of 15 percent. Samples HU-NCBC-R2-02 and HU-NCBC-R2-03 also displayed internal standard ratios outside 15 percent. None of the ratios was exceptionally far off.

The remainder of the Huber samples were determined to contain chlorinated dioxins or furans. When originally analyzed, sample HU-NCBC-R1-01 was determined to contain 118 ppb of tetra dioxin, with 115 ppb identified as 2,3,7,8-TCDD. This value did not agree with data gathered from isomer-specific data at other labs. CAL reran this sample under isomer-specific conditions and measured a 2,3,7,8-TCDD value of 193 ppb. CAL then scaled the total tetrachlorodibenzo-p-dioxin based on the 2,3,7,8-TCDD value in the following manner:

$$118/115 = 1.026$$

$$1.026 \times 193 = 198.035 \text{ ppb}$$

CAL attributed the differences in values to saturation of the mass spectrometer. However, no evidence of saturation is apparent from the CAL data. The discrepancy in values is probably because the initial analysis was conducted in the regime of nonlinear instrument response. The response of the instrument has reached a plateau and will not significantly change even with increasing concentration. For this type of situation, a multiconcentration calibration curve is essential. When CAL reanalyzed the sample using isomer-specific conditions, a smaller sample size was extracted and analyzed, thus introducing a smaller amount of material into the mass spectrometer. The same type of situation is present for HU-NCBC-R2-01.

Sample HU-NCBC-R2-03 was supplied with two data reports, one of which was a reexamination of the data using an enhanced software routine. Using the enhanced software, TCDF, P<sub>5</sub>CDF, and TCDD were found. A maximum concentration of 0.49 ppb of 2,3,7,8-TCDD was found. Upon isomer-specific analyses, it was determined that the 320/322 ratio was unacceptable (1.01), resulting in a maximum possible concentration of 0.78 ppb for 2,3,7,8-TCDD. Based on these conflicting results, a maximum possible concentration of 0.78 ppb may be present. For sample HU-NCBC-R2-03, the



calculations for H<sub>x</sub>CDD could not be reproduced. The areas and heights used on the calculation sheets do not match those on the actual chromatograms. This is most likely the result of manually determining heights and areas and not noting them on the chromatogram.

Sample HU-NCBC-R2-02 was also supplied with two sets of data, one of which used the enhanced software processing. In the first set of data, results of "not detected" were reported for all isomer classes. Using the enhanced software, a value of 22 ppt (part per trillion) of TCDF was determined. Based upon review of data, the positive identification of a TCDF is marginal because of signal-to-noise factors. The value of 28 ppt should be considered a maximum possible concentration.

As mentioned previously, two native spikes were prepared and analyzed. Of the two, the spike using the HU-NCBC-R2-09A sample was of better quality because the original sample contained no detectable amounts of dioxin and furans. Recovery of the native spikes ranged from 83 to 112 percent. All recoveries were well within the acceptable range. The spike sample using HU-NCBC-R1-01 provided good recoveries, with the exception of the TCDF and TCDD, because of the high levels of material already present in the sample.

In addition to the samples run by CAL for isomer class content, Battelle Columbus Laboratories analyzed sample HU-NCBC-R2-02 for tetra through hexa-chlorinated dioxins and furans using an in-house methodology (refer to Reference 2 for exact details). The results of this analysis show findings of "not detected" for all isomer classes, with detection limits ranging from 0.01 to 0.04 ppb. Battelle's data were examined and determined to meet the QA/QC criteria described in the report.

Examination of CAL's isomer class data was a review and not a validation. When this work was performed, a single accepted, validated method for the isomer class determination was not available. Because of this, a uniform set of evaluation criteria has not been adopted. The review was aimed at understanding the data and analytical results and any inconsistencies noted. The data provided by CAL indicates trends in the

levels of PCDDs and PCDFs present and can be used on a semiquantitative basis to follow the destruction efficiency of the Huber process.

Because of the apparent switch by CAL from Method 8280 to what appears to be an in-house method, and the confused method of reporting, it was time-consuming and difficult to examine the data.

CAL also conducted 2,3,7,8-TCDD isomer specific analyses on specified samples and on those samples found to contain 2,3,7,8-TCDD when analyzed for isomer class content. CAL proposed to perform these analyses according to the U.S. EPA CLP method. According to the final report letter (January 21, 1986), this methodology was used with modifications made for extraction of various sample matrices. As stated previously, the data supplied by CAL were reviewed according to the same criteria used during the previous soil sampling and analysis program. Upon the completion of the review, it was apparent that the CLP procedure was not followed. Inconsistencies included the following:

1. No initial calibration curve established.
2. Incomplete concentration range of standards (100-ppb and 200-ppb standards omitted).
3. Partial scan was not provided until asked for.
4. Incomplete data reporting, including lack of initial calibration, lack of continuing calibration, lack of chronological list of all analyses performed.
5. Nonadherence to protocol concerning performance check standards.
6. Nonadherence to reporting format specified.

Because of these inconsistencies, the isomer-specific data for Huber can be technically considered invalid. The data can be used to project

trends in the Huber system's ability to decontaminate soil but would not be accepted by the Sample Management Office of the CLP.

In addition to the Huber samples, CAL analyzed a series of air filter samples using toluene Soxhlet extraction, followed by CAL's in-house 2,3,7,8-TCDD method. As before, these samples were to have been analyzed by the CLP method using suitable extraction modifications. The same deficiencies noted for the Huber 2,3,7,8-TCDD samples apply to the air filter samples.

Upon review of the data, samples 1138 and 1140 (which were reported to be 0.14 ppb and 0.11 ppb or  $0.03 \text{ pg/m}^3$  and  $0.07 \text{ pg/m}^3$ , respectively) should be changed to not detected, based on signal-to-noise criteria and have detection limits of less than  $0.08 \text{ pg/m}^3$  and less than  $0.18 \text{ pg/m}^3$ , respectively.

## CONCLUSIONS

The CAL-supplied analytical data have numerous shortcomings and omissions that prevent strict validation of any of the results. However, the review/evaluation of the data has shown that the results can be used as indicative. Therefore, the results can be used to identify trends and to evaluate the probable effectiveness of the Huber process technology. The use of the results to provide strict quantitative information about the process is not justified without the additional corroborative information that would be provided by further testing of the Huber process.

The sample analyzed by Battelle was a sample of treated soil. As noted previously, it was found to be free of all isomer classes of both tetra through hexachlorinated dioxins and dibenzofurans down to detection levels that ranged from 0.01 to 0.04 ppb. The Battelle results were supported by adequate QA/QC and met all QA/QC criteria, as described in its report. Thus, these results are the most valid indication of the actual PCDD and PCDF levels in the treated soils. Since Battelle analyzed only a very limited number of samples, however, general conclusions based on its results must take that fact into account.

## REFERENCES

1. Review of Contractor Data from the IFB WA84-A002 Chemical Analytical Services for 2,3,7,8-Tetrachlorodibenzo-p-dioxin, Environmental Protection Agency, November 20, 1984.
2. Determination of Polychlorinated Dibenzo-p-dioxins and Poly-chlorinated Dibenzofurans in Soil Samples, Battelle Columbus Laboratories, Columbus, Ohio, May 21, 1980.

## ANNEX

All 2,3,7,8-TCDD isomer-specific analytical data were reviewed and evaluated according to the requirements detailed in the EPA document for reviewing 2,3,7,8-TCDD analytical results (Reference 1 of Appendix O). This document was adapted to form the working document used for detailed data review/evaluation. The criteria used to review the analytical data are as follows:

1. To ensure isomer specificity for chromatographic separation, the 2,3,7,8-TCDD must be separated from interfering isomers with no more than a 50 percent valley relative to the 2,3,7,8-TCDD peak.
2. The  $m/z$  320/322 and 332/334 ratios must be within the range of 0.67 to 0.87.
3. Ions 320, 322, and 257, which are each monitored separately but concurrently, must all be present; and the signal for all three must maximize simultaneously. The signal-to-noise ratio must be 2.5 to 1 or better for all three ions.
4. The signal-to-noise ratio must be 10 to 1 or better for the 332 and 334 ions, which are the ions due to the internal standard.
5. The retention time of the native 2,3,7,8-TCDD must equal (within 3 seconds) the retention time for the isotopically labeled 2,3,7,8-TCDD.
6. Positive results must be confirmed by obtaining partial scan spectra from mass 150 to mass 350 for selected samples.
7. The surrogate standard results must be within  $\pm 40$  percent of the true value.

APPENDIX R

CALIFORNIA ANALYTICAL LABORATORIES PROTOCOL STANDARDS AND  
FOR ANALYSIS OF DIOXINS AND FURANS

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Exhibit D - Analytical Methods

2,3,7,8-tetrachlorodibenzo-p-dioxin in Soil and  
Sediment by High Resolution Gas Chromatography/  
Low Resolution Mass Spectrometry

## 1. SCOPE AND APPLICATION

- 1.1 This method provides procedures for detection and measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD; CAS Registry Number 1746-01-6; STORET Number 34675) at concentrations of 1  $\mu\text{g}/\text{kg}$  to 200  $\mu\text{g}/\text{kg}$  in 10-g aliquots of wet soil and sediment. The use of 1-g aliquots permits measurement of concentration up to 2000  $\mu\text{g}/\text{kg}$ .
- 1.2 The minimum measurable concentration is estimated to be 0.3  $\mu\text{g}/\text{kg}$ , but is dependent on interfering compounds present in the sample matrix.
- 1.3 This method is designed for use by analysts who are experienced in the use of a gas chromatograph/mass spectrometer.
- 1.4 CAUTION: Because 2,3,7,8-TCDD is extremely toxic, safety procedures described in Section 5 of this method should be followed to prevent exposure of laboratory personnel to materials containing this compound.

## 2. SUMMARY OF METHOD

After 50 ng of  $^{13}\text{C}$ -labeled 2,3,7,8-TCDD and 10 ng of  $^{37}\text{Cl}$ -labeled 2,3,7,8-TCDD are added to a 10 gram aliquot of soil or sediment sample, the wet soil or sediment is mixed with 20 grams of anhydrous sodium sulfate and is extracted with a mixture of hexane and methanol, while the sample aliquot and solvent are agitated continually in a glass jar. Column chromatographic procedures are used to help eliminate sample components that may interfere with detection and measurement of 2,3,7,8-TCDD. The extract is concentrated to 50  $\mu\text{L}$ , and a 2  $\mu\text{L}$  aliquot is injected into a fused silica capillary column in a gas chromatograph (GC) interface to a mass spectrometer (MS) that has at least unit resolution at  $m/z$  334.

Identification of 2,3,7,8-TCDD is based on detection of three characteristic ions, measurement of the appropriate relative abundances of two characteristic ions in the molecular ion cluster, and determination of the retention time of the sample analyte relative to the internal standard,  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, contained in the sample extract. The 2,3,7,8-TCDD concentration is determined by measuring the MS response to the sample component relative to the MS response to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD (the internal standard). The labeled internal



standard method presumes that internal standard losses during method procedures are equal to unlabeled TCDD losses. Therefore, the calculated sample 2,3,7,8,-TCDD concentration is corrected for losses during sample preparation.

The  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD is a surrogate compound that is added to each sample and is analyzed exactly the same as unlabeled TCDD. The accuracy of surrogate compound measurement is used to indicate the accuracy of measurement of unlabeled 2,3,7,8-TCDD in the same sample.

### 3. DEFINITIONS

- 3.1 Concentration calibration solution -- a solution containing known amounts of the analyte (unlabeled 2,3,7,8-TCDD), the surrogate compound ( $^{37}\text{Cl}_4$ -2,3,7,8-TCDD), and the internal standard ( $^{13}\text{C}_{12}$ -2,3,7,8-TCDD); it is used to determine instrument response of the analyte and the surrogate compound relative to the internal standard.
- 3.2 Field blank -- a portion of soil/sediment uncontaminated with 2,3,7,8-TCDD.
- 3.3 Rinsate -- a portion of solvent used to rinse sampling equipment and analyzed to demonstrate that samples are not contaminated during sampling.
- 3.4 Internal standard --  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, which is added to every sample and is present at the same concentration in every blank, quality control sample, and concentration calibration solution. It is added to the soil/sediment sample before extraction and is used to measure the concentrations of analyte and surrogate compound.
- 3.5 Laboratory reagent blank -- a blank prepared by the laboratory by performing all analytical procedures except addition of a sample aliquot to the extraction vessel.
- 3.6 Performance check mixture -- a mixture of known amounts of selected standard compounds; it is used to demonstrate continued acceptable performance of the GC/MS/DS system.
- 3.7 Performance evaluation sample -- a soil or sediment sample containing a known amount of unlabeled 2,3,7,8-TCDD. It is distributed by EPA to potential contractor laboratories who must analyze it and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). It may also be included as an unspecified QC sample in any sample batch submitted to the lab for analysis.

- 3.8 Response factor -- response of the mass spectrometer to a known amount of an analyte relative to a known amount of an internal standard.
- 3.9 Signal-to-noise ratio -- The ratio of the area of the analyte signal to the area of the random background signal; it is determined by integrating the signal for a characteristic ion in a region of the selected ion current profile where only random noise is observed and relating that area to the area measured for a positive response for the same ion. The same number of spectra must be integrated for both areas. (The ratio of peak heights may be used instead of peak areas.)
- 3.10 Surrogate compound --  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD, which is added to the soil/sediment before analysis. Its concentration is measured in each sample, and the accuracy of that concentration measurement is calculated to indicate the accuracy of the unlabeled 2,3,7,8-TCDD measurement.

#### 4. INTERFERENCES

Any organic compound that is within 10 scans (at the rate of 1 scan/second) of  $m/z$  257, 320, 322, or 328 of the internal standard and produces any of the three ions monitored to detect 2,3,7,8-TCDD, is a potential interference. Most frequently encountered interferences are other sample components that are extracted along with TCDD. Because very low levels of TCDD must be measured, elimination of interference is essential. High purity reagents and solvents must be used and all equipment must be scrupulously cleaned. Laboratory reagent blanks (Exhibit E, Quality Control, Section 4) must be analyzed to demonstrate lack of contamination that would interfere with TCDD measurement. Column chromatographic procedures are used to remove some coextracted sample components; these procedures must be performed carefully to minimize loss of TCDD during attempts to enrich its concentration relative to other sample components.

#### 5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a file of current OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are identified. (1-3) 2,3,7,8-TCDD has been identified as a suspected human or mammalian carcinogen.

- 5.2 Each laboratory must develop a strict safety program for handling 2,3,7,8-TCDD. The following laboratory practices are recommended:
- 5.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.
  - 5.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or through a trap containing oil or high-boiling alcohols.
- 5.3 The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory are presented as guidelines only, and are based on safe handling practices included in USEPA Method 613.<sup>(4)</sup> The precautions for safe handling and use are necessarily general in nature because detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual usage. Assistance in evaluating the health hazards or particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. Although 2,3,7,8-TCDD is extremely toxic to laboratory animals, it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.
- 5.3.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and lab hood adequate for radioactive work.
  - 5.3.2 Training: Workers must be trained in the proper method of removing of contaminated gloves and clothing without contacting the exterior surfaces.
  - 5.3.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift) with any mild soap and plenty of scrubbing action.
  - 5.3.4 Confinement: Isolated work area, posted with signs; segregated glassware and tools; and plastic-backed absorbent paper on benchtops.
  - 5.3.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors should not handle wastes.
  - 5.3.6 Disposal of Wastes: 2,3,7,8-TCDD decomposes above 800°C. Low level waste, such as the absorbent paper and plastic gloves, may be burned in a good incinerator. Water containing gross quantities (milligrams) of 2,3,7,8-TCDD should be packaged securely and disposed through commercial or governmental channels that are capable of handling

high-level or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container; residues may then be handled as above.

- 5.3.7 Glassware, Tools, and Surfaces: Satisfactory cleaning may be accomplished by rinsing with 1,1,1-trichloroethane, then washing with any detergent and water. Dishwater may be disposed to the sewer. (Also see Section 6.5.)
- 5.3.8 Laundry: Clothing known to be contaminated should be disposed with the precautions described under Section 5.3.6. Lab coats or other clothing worn in 2,3,7,8-TCDD work may be laundered. Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing. Disposable garments may be used to avoid a laundry problem, but they must be properly disposed or incinerated.
- 5.3.9 Wipe Tests: A useful method to determine cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, which is extracted and analyzed by gas chromatography (limit of sensitivity of approximately 0.1  $\mu\text{g}$  per wipe). Less than 0.1  $\mu\text{g}$  2,3,7,8-TCDD per wipe indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10  $\mu\text{g}$  on a wipe sample indicates an acute hazard that requires prompt cleaning before further use of the equipment or work space and indicates that unacceptable work practices have been employed in the past.
- 5.3.10 Inhalation: Any procedure that may produce airborne contamination must be performed with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in case of an accident. Finely divided soils contaminated with 2,3,7,8-TCDD are hazardous because of the potential for inhalation. Such samples should be handled in a confined environment, such as a hood or glove box, or laboratory personnel should wear masks fitted with a particulate filter and charcoal sorbent.
- 5.3.11 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

## 6. APPARATUS AND EQUIPMENT

### 6.1 Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS)

- 6.1.1 The GC must be capable of temperature programming and be equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port must be designed for capillary columns. Splitless or on-column injection technique is recommended. With this method, a 2- $\mu$ L injection volume is used consistently. With some GC injection ports, however, 1  $\mu$ L may be the maximum volume that produces adequate precision and chromatographic separation. A 1- $\mu$ L injection volume may be used if adequate sensitivity and precision can be achieved. CAUTION: If 1  $\mu$ L is used for any injection volume, the injection volume for all extracts, blanks, calibration solutions and the performance check sample must be 1  $\mu$ L.
- 6.1.2 Mass spectral data are obtained with electron ionization at a nominal electron energy of 70 eV. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five data points for each of six ions while a sample component elutes from the GC.
- 6.1.3 An interfaced data system (DS) is required to acquire, store, reduce and output mass spectral data. The DS must be equipped with a selected ion monitoring (SIM) program to acquire data for at least six ions that are characteristic of labeled and unlabeled 2,3,7,8-TCDD. (The mass spectrum of unlabeled 2,3,7,8-TCDD is shown in Figure 1 at the end of this Exhibit.) The same integration time must be used for each ion monitored, and the integration time used for sample analyses must be the same as the time used to analyze concentration calibration solutions and the performance check solution. Total data acquisition time per cycle (six ions) must not exceed 1.5 seconds.
- 6.2 GC Column -- Two fused silica capillary columns are recommended; one is a 60-m SP-2330 and the other is a 50-m CP-SIL 88. Any capillary column that separates 2,3,7,8-TCDD from all other TCDDs may be used, but this separation must be demonstrated. Minimum acceptance criteria must be determined per Section 9.2.3.1. At the beginning of each 8-hour period during which sample or concentration calibration solutions will be analyzed, column operating conditions must be demonstrated to achieve the required separation on the column to be used for samples. Operating conditions known to produce acceptable results with the recommended columns are shown in Table 1 at the end of this Exhibit.

### 6.3 Miscellaneous Equipment

- 6.3.1 Nitrogen evaporation apparatus with variable flow rate from approximately 30 mL/min to 150 mL/min.
- 6.3.2 Mechanical shaker -- A magnetic stirrer or a wrist-action or platform-type shaker that produces vigorous agitation. Agitation conditions must be determined and demonstrated.
- 6.3.3 Analytical balance capable of accurately weighing 0.01g.
- 6.3.4 Centrifuge capable of operating at 2000 rpm.
- 6.3.5 Water bath -- equipped with concentric ring cover and temperature controlled within  $\pm 2^{\circ}\text{C}$ .
- 6.3.6 Stainless steel spatulas or spoons.
- 6.3.7 Stainless steel (or glass) pan large enough to hold contents of 1-pint sample containers.
- 6.3.8 Glove box.

### 6.4 Glassware

- 6.4.1 Extraction jars -- amber glass with Teflon-lined screw cap; minimum capacity of approximately 500 mL; must be compatible with mechanical shaker to be used.
  - 6.4.2 Kuderna-Danish apparatus -- 500-mL evaporating flask, 10-mL graduated concentrator tubes with ground-glass stoppers, and 3-ball macro Snyder column.
  - 6.4.3 Culture tubes -- 8-mL glass.
  - 6.4.4 Mini-vials -- 1-mL amber borosilicate glass with conical-shaped reservoir and screw caps lined with Teflon-faced silicone disks.
  - 6.4.5 Funnels -- glass; appropriate size to accommodate filter paper used to filter jar extract (volume of approximately 170 mL).
  - 6.4.6 Chromatography columns -- 1 cm ID x 10 cm long and 1 cm ID by 30 cm long.
- 6.5 NOTE: Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure.

Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water. Drain dry and heat in a muffle furnace at 400°C for 15 to 30 min. Volumetric glassware should not be heated in a muffle furnace, and some thermally stable materials (such as PCBs) may not be removed by heating in a muffle furnace. In these cases, rinsing with high-purity acetone and hexane may be substituted for muffle furnace heating. After glassware is dry and cool, store inverted or capped with aluminum foil in a clean environment.

## 7. REAGENTS AND CONSUMABLE MATERIALS

### 7.1 Column Chromatography Reagents

7.1.1 Alumina, acidic -- Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil covered glass container for 24 hours at 190°C.

7.1.2 Silica gel -- high purity grade, type 60, 70-230 mesh; Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil-covered glass container for 24 hours at 130°C.

7.1.3 Silica gel impregnated with 40% (by weight) sulfuric acid -- Add two parts (by weight) concentrated sulfuric acid to three parts (by weight) silica gel (extracted and activated), mix with a glass rod until free of lumps, and store in a screw-capped glass bottle.

7.1.4 Sulfuric acid, concentrated -- ACS grade, specific gravity 1.84.

7.1.5 Graphitized carbon black (Carbopack C or equivalent), surface area of approximately 12 m<sup>2</sup>/g, 80/100 mesh.

7.1.6 Celite 545<sup>R</sup>, reagent grade, or equivalent.

7.2 Filter paper -- pore size of  $\leq 20$  to 25  $\mu$ ; rinse with hexane before use.

7.3 Glass wool, silanized -- Extract with methylene chloride and hexane before use.

7.4 Sodium sulfate -- Granular, anhydrous; before use, extract with methylene chloride and dry for  $\geq 4$  h in a shallow tray placed in an oven operated at 120°C.

- 7.5 Solvents -- High purity, distilled-in-glass; hexane, methanol, methylene chloride, and toluene.
- 7.6 Concentration Calibration Solutions (reference Table 2) -- Five toluene solutions containing unlabeled 2,3,7,8-TCDD at varying concentrations and  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD (the internal standard, CASRN 80494-19-5) at a constant concentration. Three of these solutions also contain  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD (the surrogate compound, CASRN 85508-50-5) at varying concentrations. Concentration calibration solutions are to be obtained from the Quality Assurance Division, USEPA Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. However, if not available from EMSL-LV, standards may be obtained from commercial sources, and solutions may be prepared in the contractor laboratory. Traceability of standards must be verified against EPA-supplied standard solutions, by laboratory SOP's as required in IFB Pre-Award Bid Confirmations, part 2.f.(4).
- 7.6.1 Each of solutions #1-#5 contains  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD at a concentration of 1 ng/ $\mu\text{L}$  which is equivalent to a 50- $\mu\text{L}$  extract of a 10-g sample to which that compound (the internal standard) was added at a concentration of 5  $\mu\text{g}/\text{kg}$ .
- 7.6.2 Solutions #1-#5 contain unlabeled 2,3,7,8-TCDD at concentrations of 0.2, 1, 5, 20 and 40 ng/ $\mu\text{L}$  respectively; those concentrations are equivalent to 50- $\mu\text{L}$  extracts of 10-g samples containing 1, 5, 25, 100 and 200 ppb, respectively.
- 7.6.3 Solutions #1-#3 contain  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD at concentration of 0.06, 0.12, and 0.2 ng/ $\mu\text{L}$ , respectively; those concentrations are equivalent to extracts containing 30, 60, and 100 ppb, respectively, of the amount of  $^{37}\text{Cl}_4$ -TCDD (the surrogate compound) added to each sample before extraction.
- 7.6.4 Store concentration calibration solutions in 1-mL amber mini-vials at room temperature.
- 7.7 Performance Check Solution -- A mixture containing: unlabeled 2,3,7,8-TCDD; 1,2,3,4-TCDD (CASRN 30746-58-8); 1,4,7,8-TCDD (CASRN 40581-94-0); 1,2,3,7-TCDD (CASRN 67028-18-6); 1,2,3,8-TCDD (CASRN 53555-02-5); 1,2,7,8-TCDD (CASRN 34816-53-0) and 1,2,6,7-TCDD (CASRN 40581-90-6) must be obtained from the Quality Assurance Division, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

To this dry mixture add 500  $\mu\text{L}$  of the sample fortification solution (Section 7.8) containing  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD at a concentration of 0.5 ng/ $\mu\text{L}$  and  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD at a concentration of 0.1 ng/ $\mu\text{L}$ . Store in 1-mL amber mini-vial at room temperature.



7.8 Sample Fortification Solution - a toluene solution containing the internal standard at a concentration of 0.5 ng/ $\mu$ L and the surrogate compound at a concentration of 0.1 ng/ $\mu$ L.

7.9 Field Blank Fortification Solution - a toluene solution containing the internal standard at a concentration of 0.5 ng/ $\mu$ L, the surrogate compound at a concentration of 0.1 ng/ $\mu$ L, and the unlabeled 2,3,7,8-TCDD at a concentration of 0.1 ng/ $\mu$ L.

## 8. SAMPLE PRESERVATION AND HANDLING

8.1 Chain-of-custody procedures -- see Exhibit G.

### 8.2 Sample Preservation

8.2.1 When received, each sample will be contained in a 1-pint glass jar surrounded by vermiculite in a sealed metal paint can. Until a portion is to be removed for analysis, store the sealed paint cans in a locked limited-access area where ambient temperature is maintained between 0°C and 35°C. After a portion is removed for analysis, return the unused portion of sample to its original containers and store as stated above. Do not freeze samples; they may contain sufficient water to break the sample jar if frozen.

8.2.2 To avoid photodecomposition, protect samples from light.

### 8.3 Sample Handling

8.3.1 CAUTION: Finely divided soils contaminated with 2,3,7,8-TCDD are hazardous because of the potential for inhalation or ingestion of particles containing 2,3,7,8-TCDD. Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

#### 8.3.2 Pre-extraction sample treatment

8.3.2.1 Homogenization -- Although sampling personnel will attempt to collect homogeneous samples, the contractor shall examine each sample and judge if it needs further mixing. NOTE: Contractor personnel have the responsibility to take a representative sample aliquot; this responsibility entails efforts to make the sample as homogeneous as possible. Stirring is recommended when possible.

8.3.2.2 Centrifugation -- If a sample contains an obvious aqueous/liquid phase, centrifuge it to separate liquid and solid phases. Place the entire sample in a suitable centrifuge bottle and centrifuge for 30

minutes at 2000 rpm. Remove bottle from centrifuge. With a disposable pipet, remove liquid phase and discard. CAUTION: This liquid may contain TCDD and should be disposed as a liquid waste. Mix solid layer with stainless steel spatula and remove a portion to be weighed and analyzed. Return the remaining solid portion to original sample bottle and store.

## 9. CALIBRATION

9.1 Two types of calibration procedures are required. One type, routine calibration, is required at the beginning and end of each 8-hour period during which TCDD analyses are performed. The other type, initial calibration, is required before any samples are analyzed for TCDD, and is required intermittently throughout sample analyses as dictated by results of routine calibration procedures described below. No samples are to be analyzed until acceptable calibration is demonstrated and documented.

### 9.2 Routine Calibration

9.2.1 Calibrate and tune the MS with standards and procedures prescribed by the manufacturers. CAUTION: Some manufacturers may specify baseline resolution at masses higher than necessary for this method; that procedure could significantly reduce sensitivity for TCDD analysis.

9.2.2 Inject 2  $\mu$ L (CAUTION: See Sect. 6.1.1) of the performance check solution (Sect. 7.7) and acquire selected-ion-monitoring mass spectral data for  $m/z$  320, 322, 323, 328, 332, and 334 within a total cycle time of  $\leq 1.5$  seconds. Acquire at least five data points for each GC peak and use the same data acquisition time for each of the six ions being monitored. NOTE: The same data acquisition parameters previously used to analyze concentration calibration solutions during initial calibration must be used for the performance check solution.

9.2.3 Determine and document acceptable system performance with the following criteria:

9.2.3.1 GC column performance -- If SP-2330 column is used, the valley between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers must be resolved with a valley  $\leq 25\%$ . Valley (X) =  $x/y \times 100$ , when y is peak height of 2,3,7,8-TCDD; x is measured as shown in Figures 2 and 3 at the end of this Exhibit. The peak representing 2,3,7,8-TCDD shall be labeled and identified as such on the chromatograms.

- 9.2.3.2 Ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be  $\geq 0.67$  and  $\leq 0.87$ .
- 9.2.3.3 MS resolution -- Ratio of integrated ion current for m/z 323 relative to m/z 322 for unlabeled 2,3,7,8-TCDD should be  $\geq 0.07$  and  $\leq 0.20$ .
- 9.2.3.4 Ratio of integrated ion current for m/z 332 to m/z 334 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD must be  $\geq 0.67$  and  $\leq 0.87$ .
- 9.2.3.5 Response factor (Sect. 9.3.10) for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD must be within  $\pm 10\%$  of the mean value established by triplicate analyses of the concentration calibration solutions (Section 9.3).
- 9.2.4 Inject 2  $\mu\text{L}$  of the concentration calibration solution #1, which contains 0.2 ng/ $\mu\text{L}$  of unlabeled 2,3,7,8-TCDD. Using the same GC/MS/DS conditions as used in Section 9.2.2 except the ions being monitored, acquire data for m/z 257, 320, 322, 328, 332, and 334. Determine and document acceptable performance for:
- 9.2.4.1 MS sensitivity -- signal-to-noise (S/N) ratio (Section 3.8) of  $> 2.5$  for m/z 257 and  $> 10$  for m/z 322 for unlabeled 2,3,7,8-TCDD. The ratio of integrated ion current for m/z 257 to m/z 322 must be  $\geq 0.20$  and  $\leq 0.45$ .
- 9.2.4.2 Measured response factor for unlabeled 2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD is within  $\pm 10\%$  of the mean values established (Section 9.3) by triplicate analyses of the concentration calibration solutions.
- 9.2.5 Remedial actions shall be taken by Contractor if criteria are not met. Possible remedies are:
- 9.2.5.1 Check and adjust GC and/or MS operating conditions.
- 9.2.5.2 Replace GC column (performance of initial calibration procedures then required).
- 9.2.5.3 Tune MS for greater or lesser resolution.
- 9.2.5.4 Calibrate MS mass scale.
- 9.2.5.5 Prepare and analyze new performance check solution.

9.2.3.6 Prepare new concentration calibration curve(s) (Section 9.3.11).

9.3 Initial Calibration

- 9.3.1 In addition to routine calibration procedures described in Section 9.2, before any samples are analyzed, determine response factors for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and for unlabeled 2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD.
- 9.3.2 Concentration calibration solutions -- The five solutions described in Section 7.6 are required.
- 9.3.3 Calibrate and tune the MS with standards and procedures prescribed by the instrument manufacturer.
- 9.3.4 If a column other than the recommended (Section 6.2) SP-2330 or CP-SIL 88 fused silica capillary column is used, determine the GC conditions necessary to separate 2,3,7,8-TCDD from other TCDDs known to have similar relative retention times.
- 9.3.5 Inject a 2- $\mu\text{L}$  aliquot of the performance check solution (CAUTION: See Section 6.1.1) and acquire selected-ion-monitoring (SIM) mass spectral data using the MS operating conditions specified in Section 9.2.2. Determine GC operating conditions necessary to achieve separation described in Section 9.2.3.1.
- 9.3.6 Using specified MS data acquisition procedures and the GC conditions determined in Section 9.3.5, analyze a 2- $\mu\text{L}$  aliquot of the performance check solution.
- 9.3.7 Determine and document acceptable calibration using the criteria specified in Section 9.2.3.2 - 9.2.3.5.
- 9.3.8 Using the same GC conditions that produced acceptable results with the performance check solution, analyze a 2- $\mu\text{L}$  aliquot of each of the five concentration calibration solutions with the following MS operating parameters.
- 9.3.8.1 Acquire selected-ion-monitoring data for m/z 257, 320, 322, 328, 332 and 334.
- 9.3.8.2 Total cycle time for data acquisition must be  $\leq$  1.5 seconds.
- 9.3.8.3 Acquire at least five data points for each ion during elution of the GC peak.

- 9.3.8.4 Use the same data acquisition time for each of the six ions being monitored.
- 9.3.9 Repeat Section 9.3.8 two times to produce triplicate data sets for each solution.
- 9.3.10 Calculate the response factor for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and for unlabeled 2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD:

$$\text{RF} = \frac{A_x \cdot Q_{1s}}{A_{1s} \cdot Q_x}$$

- where  $A_x$  = integrated ion abundance (corrected as specified in Section 12.1.1.3) of m/z 320 for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD or the sum of integrated ion abundances of m/z 320 and m/z 322 for unlabeled 2,3,7,8-TCDD,
- $A_{1s}$  = the sum of integrated abundances of m/z 332 and m/z 334 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD,
- $Q_{1s}$  = quantity of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, and
- $Q_x$  = quantity of unlabeled 2,3,7,8-TCDD or  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD injected.

RF is a unitless number; units used to express quantities must be equivalent.

- 9.3.11 For both  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, calculate the mean RF and its relative standard deviation (RSD) from triplicate analyses of each of the five concentration calibration solutions. Variation of the RF calculated for each compound at each concentration level must not exceed 10% RSD. If the five mean RFs for each compound do not differ by more than  $\pm 10\%$ , the RF can be considered to be independent of analyte quantity for the calibration concentration range, and the mean of the five mean RFs shall be used for concentration calculations.

## 10. QUALITY CONTROL

See Exhibit E for QA/QC Requirements.

## 11. PROCEDURES

### 11.1 Sample Extraction

11.1.1 CAUTION: See Section 5 for safety guidelines and recommendations.

11.1.2 Jar extraction. NOTE: Extremely wet samples may require centrifuging to remove water before addition of sodium sulfate see (Section 8.3.2.2).

11.1.2.1 Accurately weigh to three significant figures a 10 gram (+ 0.5 gram) portion of the wet soil or sediment sample, and transfer it to the extraction jar.

11.1.2.2 Add 100  $\mu$ L of the sample fortification solution (Section 7.8) to the soil or sediment in the extraction jar. Add small portions of the solutions at several sites on the surface of the soil or sediment.

11.1.2.3 Add 20 g of purified anhydrous sodium sulfate, and mix thoroughly using a stainless steel spoon or spatula.

11.1.2.4 Allow the mixture of soil and sodium sulfate to set for two hours at ambient temperature; mix again, break all visible lumps, and allow to set for at least four more hours.

11.1.2.5 Mix again and add 20 mL of methanol; mix again and add 150 mL of hexane.

11.1.2.6 Place the extraction jar containing the soil, sodium sulfate and solvents in the shaker and shake for at least 3 hours.

11.1.2.7 Remove the jar from the shaker and allow solids to settle. Decant the solvent through a glass funnel containing hexane-rinsed filter paper. Rinse the jar, solid sample residue, and filter residue with four 5-mL portions of hexane.

11.1.2.8 Concentrate the extract volume to approximately 2 to 3 mL with a Kuderna-Danish apparatus or a rotary evaporator. NOTE: Glassware used for more than one sample must be carefully cleaned between samples to prevent cross contamination (See Section 6.5).

11.1.2.9 Transfer the concentrated extract to an 8-ml glass culture tube. Rinse the evaporator flask with three 5-ml portions of hexane; transfer each rinse to the culture tube. Between additions of hexane rinse, reduce the extract volume in the culture tube enough to allow addition of another 5-ml volume of rinse. To reduce the volume, place the culture tube in a water bath adjusted to operate at 50°C and position the tube so that the surfaces of the extract and the water are at about the same level. Evaporate the solvent with a stream of nitrogen (flow rate of approximately 150 mL/min) with the tip of the nitrogen delivery tube 2 cm above the solution.

11.1.2.10 After the final rinse has been added, reduce the extract volume to approximately 1 mL.

## 11.2 Column Chromatography

### 11.2.1 Column Preparation

11.2.1.1 Column 1: Place 1.0 g of silica gel into a 1 cm x 20 cm column and tap the column gently to settle the silica gel. Add 2 g sodium hydroxide-impregnated silica gel, 1 g silica gel, 4.0 g of sulfuric acid-impregnated silica gel, and 2 g silica gel. Tap column gently after each addition.

11.2.1.2 Column 2: Place 6.0 g of alumina into a 1 cm x 30 cm column and tap the column gently to settle the alumina. Add a 1-cm layer of purified sodium sulfate to the top of the alumina.

11.2.1.3 Add hexane to each column until the packing is free of channels and air bubbles. A small positive pressure (5 psi) of clean nitrogen can be used if needed.

11.2.2 Quantitatively transfer the hexane sample extract from the culture tube to the top of the sulfuric acid-impregnated silica gel in Column 1. Rinse the culture tube with two 0.5 mL portions of hexane; transfer rinses to Column 1.

11.2.3 With 90 mL of hexane, elute the extract from Column 1 directly into Column 2 containing alumina and sodium sulfate.

11.2.4 Add 20 mL of hexane to Column 2 and elute until the hexane level is just below the top of the sodium sulfate; discard the eluted hexane.

11.2.5 Add 20 mL of 20% methylene chloride/80% hexane (volume/volume) to Column 2 and collect the eluate.

11.2.6 Reduce the volume of eluate with a gentle stream of filtered dry nitrogen. When the volume is about 1 to 2 mL, transfer aliquots to a 1-mL amber mini-vial with conical reservoir. Concentrate and add additional aliquots with further concentration until entire eluate is transferred. Rinse eluate container with two 0.5-mL portions of hexane; transfer rinses to the mini-vial, with further concentration as necessary. CAUTION: Do not evaporate sample extract to dryness.

11.2.7 With the final sample extract volume at approximately 1 mL, store the extract until time for GC/MS analysis.

### 11.3 GC/MS Analysis

11.3.1 Remove the sample extract or blank from storage and allow it to warm to ambient laboratory temperature if necessary.

With a stream of dry, filtered nitrogen, reduce the extract/blank volume to near dryness. Immediately before GC/MS analysis, adjust the extract or blank volume to 50  $\mu$ L with toluene.

11.3.2 Inject a 2- $\mu$ L aliquot of the extract into the GC, operated under conditions previously used (Sect. 9) to produce acceptable results with the performance check solution.

11.3.3 Acquire mass spectral data for the following selected characteristic ions: m/z 257, 320, and 322 for unlabeled 2,3,7,8-TCDD; m/z 328 for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD; and m/z 332 and 334 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD. Use the same data acquisition time and MS operating conditions previously used (Sect. 9.3.8) to determine response factors.

11.4 Identification Criteria. NOTE: Refer to Exhibit E, Section 7, for application of identification criteria.

11.4.1 Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD. Retention times are required for all chromatograms, but scan numbers are optional. These parameters should be printed next to the appropriate peak.

11.4.2 The integrated ion currents detected for m/z 257, 320, and 322 must maximize simultaneously. If there are peaks that will affect the maximization or quantitation of peaks of interest, attempts should be made to narrow the scan window to eliminate the interfering peaks. This should be reported on a separate chromatogram.



11.4.3 The integrated ion current for each analyte and surrogate compound ion (m/z 257, 320, 322 and 328) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background and must not have saturated the detector.

11.4.4 Relative abundance of m/z 257 to m/z 322 should be  $\geq 20\%$  and  $\leq 45\%$ .

11.4.5 Abundance of integrated ion counts detected for m/z 320 must be  $\geq 67\%$  and  $\leq 87\%$  of integrated ion counts detected for m/z 322.

11.5 Column Chromatography Procedure for Difficult Samples -- Use the following procedure for extracts previously subjected to the column chromatography procedures in Section 11.2, but found by GC/MS analysis to contain interfering components.

11.5.1 Mix 3.6 grams of Carbowack C (or equivalent) with 16.4 grams of Celite 545<sup>R</sup> (or equivalent) in a 40-mL vial and activate by heating in an oven at 130°C for 6 hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbowack/Celite<sup>R</sup> to ensure TCDD recovery of  $\geq 50\%$ . Subject the low level concentration calibration solution to this procedure and measure the quantity of labeled and unlabeled 2,3,7,8-TCDD.

11.5.2 Insert a small plug of glass wool into a disposable pipet approximately 15 cm long by 7 mm O.D. Apply suction with a vacuum aspirator attached to the pointed end of the pipet, and add the Carbowack/Celite<sup>R</sup> mixture until a 2 cm column is obtained.

11.5.3 Pre-elute the column with:

11.5.3.1 2 mL of toluene

11.5.3.2 1 mL of a mixture of 75% (by volume) methylene chloride, 20% methanol and 5% benzene

11.5.3.3 1 mL of 50% (by volume) cyclohexane and 50% methylene chloride

11.5.3.4 2 mL of hexane

11.5.4 While the column is still wet with hexane, add the sample extract. Elute the column with the following sequence of solvents and discard eluents.

11.5.4.1 2 mL of hexane

11.5.4.2 1 mL of 50% (by volume) cyclohexane and 50% methylene chloride

11.5.4.3 1 mL of 75% (by volume) methylene chloride, 20% methanol and 5% benzene

11.5.5 Elute with 2 mL of toluene and collect the eluent, which contains the TCDD.

11.5.6 Store the sample extract until just before GC/MS analysis.

## 12. CALCULATIONS

### 12.1 Concentration

12.1.1 Concentration when a linear response factor was obtained:

12.1.1.1 Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_x = \frac{A_x \cdot Q_{is}}{A_{is} \cdot RF \cdot W}$$

where  $C_x$  = 2,3,7,8-TCDD concentration in micrograms per kilogram

$A_x$  = the sum of integrated ion abundance detected for  $m/z$  320 and 322

$A_{is}$  = the sum of integrated ion abundances detected for  $m/z$  332 and 334 (characteristic ions of  $^{13}C_{12}$ -2,3,7,8-TCDD, the internal standard)

$Q_{is}$  = quantity (in nanograms) of  $^{13}C_{12}$ -2,3,7,8-TCDD added to the sample before extraction

RF = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to  $^{13}C_{12}$ -2,3,7,8-TCDD

W = weight (in grams) of wet soil or sediment sample.

- 12.1.1.2 If the calculated concentration of unlabeled 2,3,7,8-TCDD exceeds 200  $\mu\text{g}/\text{kg}$ , which is the maximum concentration of the concentration calibration solutions, the linear range may have been exceeded, and a smaller aliquot of that sample must be analyzed. Accurately weigh to three significant figures a 1-g aliquot of the wet soil/sediment. Add 100  $\mu\text{L}$  of the sample fortification solution (Section 7.8), just as for the larger sample aliquot. Extract and analyze.
- 12.1.1.3 Calculate the concentration of the surrogate compound,  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD, using the formula:

$$C_s = \frac{A_s \cdot Q_{1s}}{A_{1s} \cdot RF \cdot W}$$

$C_s$  = concentration (in micrograms per kilogram) of the surrogate compound

$A_s$  = total integrated ion abundance of m/z 328 after correction for the contribution by unlabeled 2,3,7,8-TCDD (correction -- subtract 0.9% of the total integrated ion abundance detected for m/z 322 in the same sample extract)

$A_{1s}$  = the sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, the internal standard)

$Q_{1s}$  = quantity (in nanograms) of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD added to the sample before extraction

RF = calculated mean response factor for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD

W = weight (in grams) of wet soil or sediment sample.

- 12.2 Accuracy -- Calculate the accuracy (A) of the measurement of surrogate,  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD, using the formula:

$$\text{Surrogate Percent Accuracy} = \frac{\text{amount measured (nanograms)}}{10 \text{ ng}} \times 100$$

12.3 Estimated Detection Limit -- For samples in which no unlabeled 2,3,7,8-TCDD was detected, calculate the estimated minimum detectable concentration, which is the concentration required to produce a signal with area (or peak height) of 2.5 times the background signal area (or peak height). The background area is determined by integrating ion abundances for either m/z 320 or 322 in the appropriate region of the SICP, multiplying that area by 2.5, and relating the product area to an estimated concentration that would produce that product area.

Use the formula:

$$C_E = \frac{2.5 \cdot A_x \cdot Q_{IIS}}{A_{IIS} \cdot RF \cdot W}$$

where  $C_E$  = estimated concentration of unlabeled 2,3,7,8-TCDD required to produce  $A_x$

$A_x$  = peak height or integrated ion abundance for either m/z 320 or 322 in the same group of  $\geq 5$  spectra used to measure  $A_{IIS}$

$A_{IIS}$  = peak height or integrated ion abundance for the appropriate ion characteristic of the internal standard, m/z 332 when m/z 320 is used to determine  $A_x$ , and m/z 334 when m/z 322 is used to determine  $A_x$

$Q_{IIS}$ , RF, and W retain the definitions previously stated in Section 12.1.1.

The use of the area (or peak height) for m/z 320 to calculate  $C_E$  is preferred to m/z 322, but m/z 322 can be used when interference is observed for m/z 320 but not for m/z 322.

NOTE: This calculation is not applicable to all samples in which 2,3,7,8-TCDD was not identified (see Section 12.4).

12.4 Estimated Maximum Possible Concentration -- For samples where interference is observed for both m/z 320 and 322 or when an unacceptable ratio prevented identification of unlabeled 2,3,7,8-TCDD as a sample component, the procedure in Section 12.1 can be used to estimate the maximum concentration that could be represented by detected signals.

12.5 The relative percent difference (RPD) is calculated as follows: (See Section 5.1.1, Exhibit E.)

$$RPD = \frac{|S_1 - S_2|}{\text{Mean Concentration}} = \frac{|S_1 - S_2|}{\frac{S_1 + S_2}{2}}$$

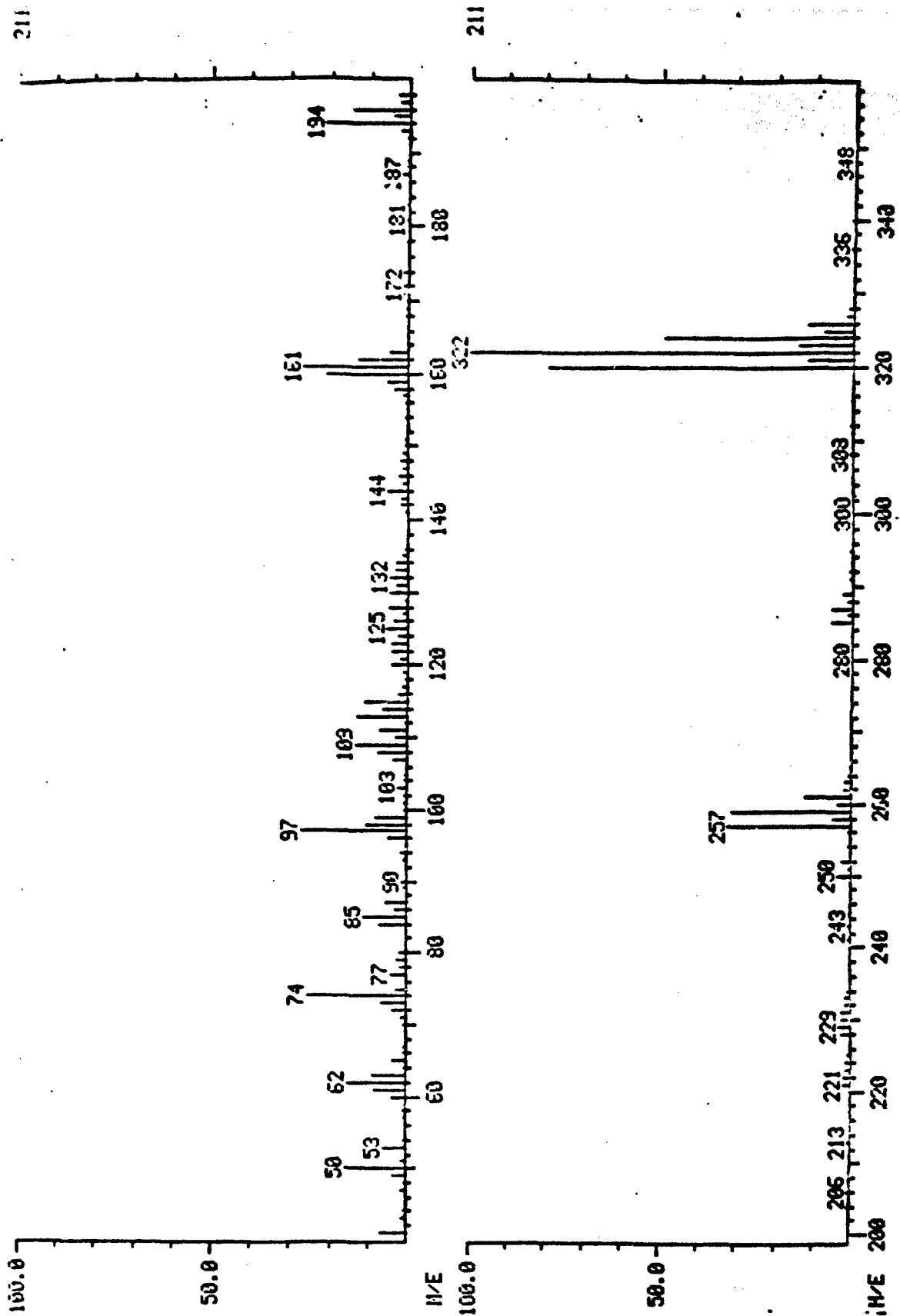
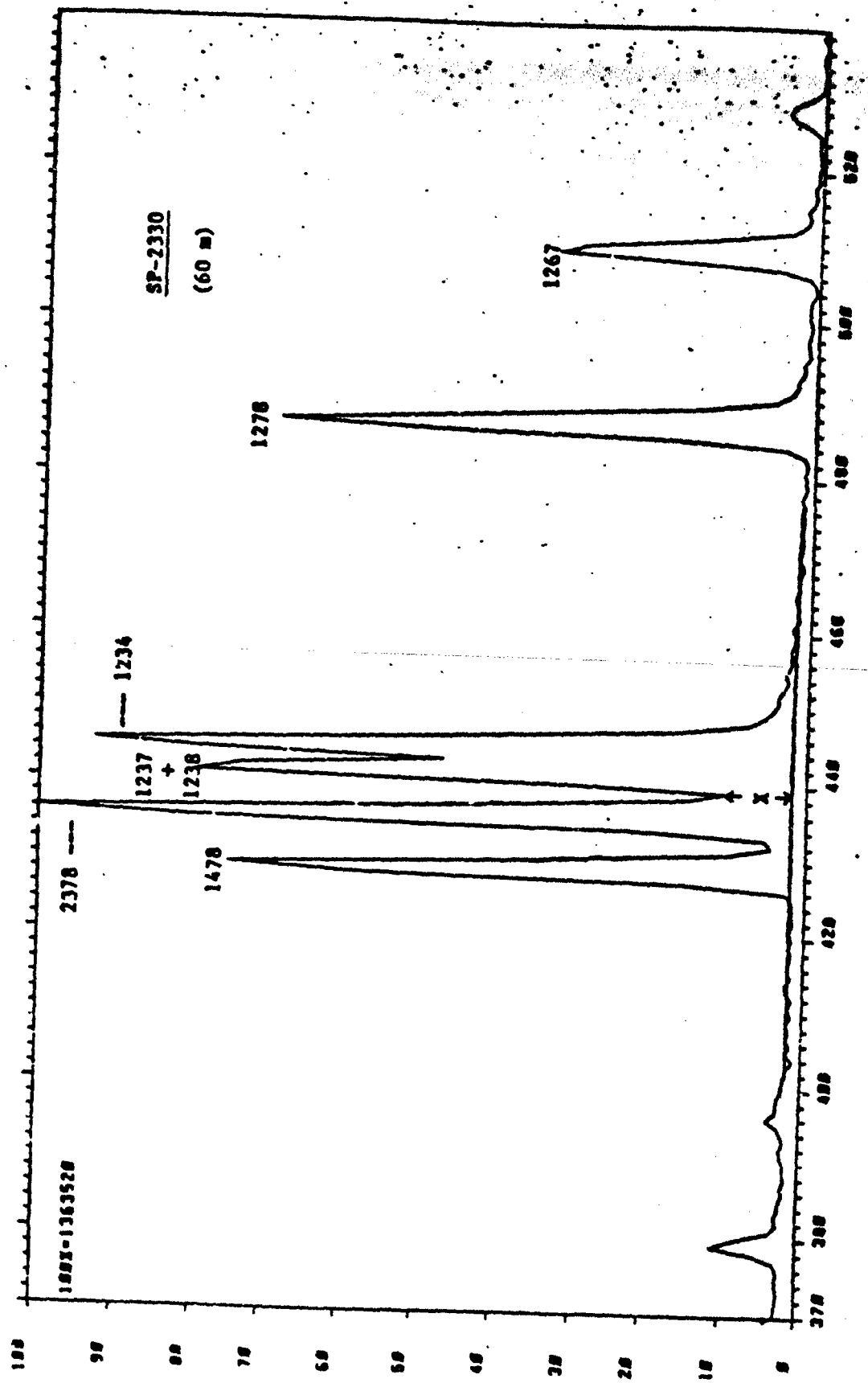


Figure 1. Complete mass spectrum of unlabeled 2,3,7,8-TCDD acquired with recommended GC conditions (Table 1).

05-58 CROSS SCAN REPORT, RUN: GCMJXC001

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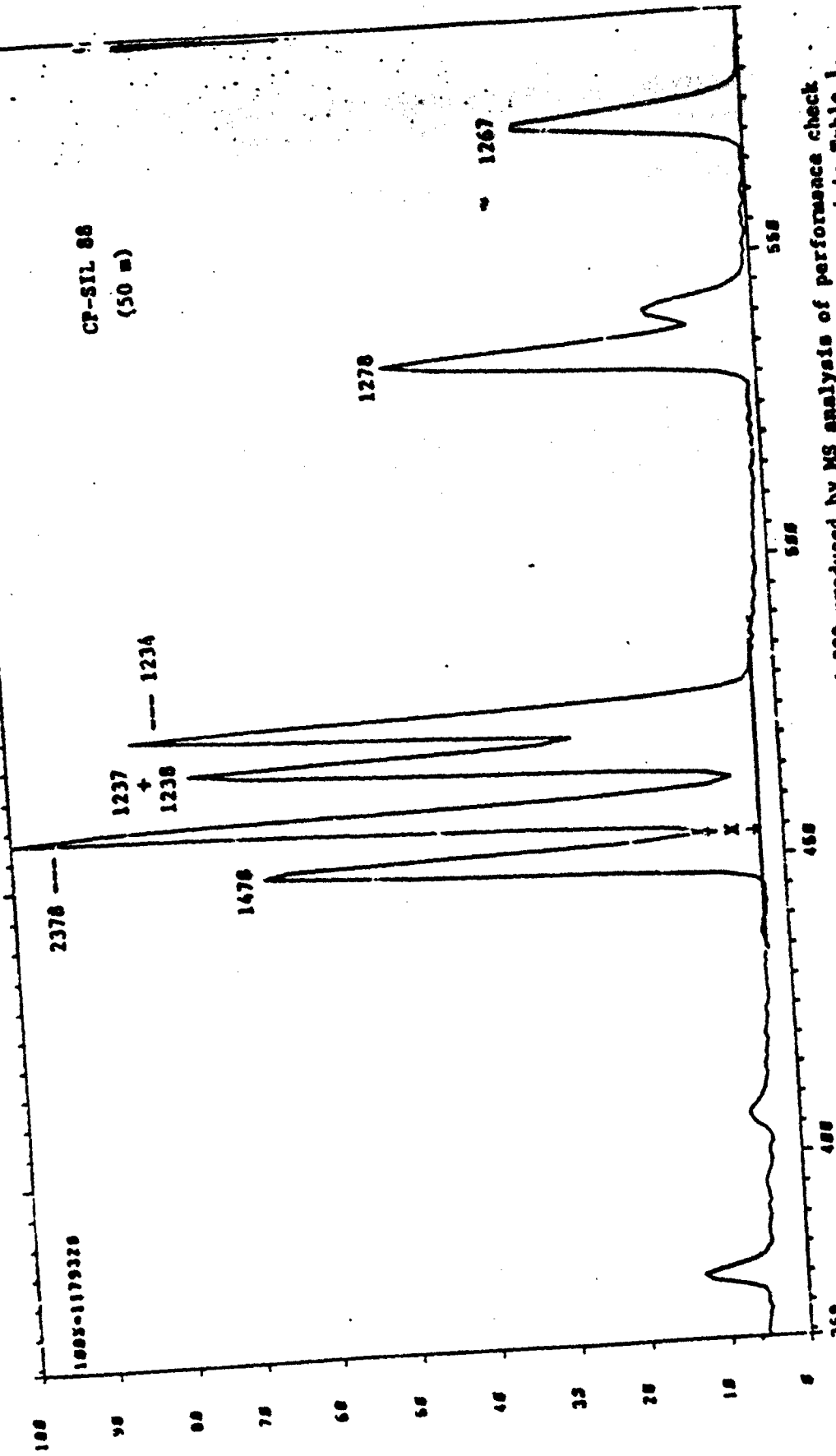


Figure 3. Selected ion current profile for m/z 320 and 322 produced by MS analysis of performance check solution using a 50-m CP-SIL 88 fused silica capillary column and conditions listed in Table 1.

TABLE 1. RECOMMENDED GC OPERATING CONDITIONS

Column coating	SP-2330	CP-SIL 88
Film thickness	0.2 $\mu$ m	0.22 $\mu$ m
Column dimensions	60 m x 0.24 mm	50 m x 0.22 mm
Helium* linear velocity	28-29 cm/sec at 260°C	28-29 cm/sec at 240°C
Initial temperature	70°C	45°C
Initial time	4 min	3 min
Temperature program	Rapid increase to 200°C 200°C to 260°C at 4°C/min	Rapid increase to 190°C 190°C to 240°C at 5°C/min
2,3,7,8-TCDD retention time	24 min	26 min

\* Hydrogen is an acceptable carrier gas.



TABLE 2. COMPOSITION OF CONCENTRATION CALIBRATION SOLUTIONS

<u>Solution #</u>	<u>Concentration of 2,3,7,8-TCDD</u>		
	<u>Isotopically Labeled</u>		<u>Unlabeled</u>
	<u><math>^{13}\text{C}_{12}</math></u>	<u><math>^{37}\text{Cl}_4</math></u>	
1	1 ng/ $\mu\text{L}$	0.06 ng/ $\mu\text{L}$	0.2 ng/ $\mu\text{L}$
2	1 ng/ $\mu\text{L}$	0.12 ng/ $\mu\text{L}$	1 ng/ $\mu\text{L}$
3	1 ng/ $\mu\text{L}$	0.2 ng/ $\mu\text{L}$	5 ng/ $\mu\text{L}$
4	1 ng/ $\mu\text{L}$	0	20 ng/ $\mu\text{L}$
5	1 ng/ $\mu\text{L}$	0	40 ng/ $\mu\text{L}$

**Exhibit E - QA/QC Requirements****SUMMARY OF QC ANALYSES**

1. Initial and periodic calibration and instrument performance checks.
2. Laboratory reagent blank analyses (Sect. 4.1); minimum of one blank shall be analyzed with each sample batch; an additional blank analyzed when new reagents are used.
3. Analysis of a batch of samples with accompanying QC analyses:
  - 3.1 Sample Batch --  $\leq$  24 samples, including field blank and rinseate sample(s).
  - 3.2 Additional QC Analyses Per Batch:
 

Laboratory reagent blank	1
Duplicate sample analysis	1
Confirmatory partial scan analysis	1
TOTAL	<u>3</u>
4. "Blind" QC samples may be submitted to contractor as an ordinary soil or sediment sample included among the batch of samples. Blind samples include:
  - 4.1 Uncontaminated soil,
  - 4.2 Split samples,
  - 4.3 Unlabeled duplicates, and
  - 4.4 Performance evaluation samples.

**QUALITY CONTROL**

1. Performance Evaluation Samples -- Included among samples in some batches will be samples containing known amounts of unlabeled 2,3,7,8-TCDD that may or may not be marked as other than ordinary samples.
2. Performance Check Solution
  - 2.1 At the beginning of each 8-hour period during which samples are to be analyzed, an aliquot of the performance check solution and an aliquot of concentration calibration solution #1 shall be analyzed to demonstrate adequate GC and MS resolution and sensitivity, response factor reproducibility, and mass range calibration.

These procedures are described in Section 9 of Exhibit D. If any required criteria are not met, remedial action must be taken before any samples are analyzed.

- 2.2 To validate sample data, the performance check solution must be analyzed also at the end of each 8-hour period during which samples are analyzed.
  - 2.2.1 If the contractor laboratory operates only during one 8-hour period (shift) each day, the performance check solution must be analyzed twice (at the beginning and end of the 8-hour period) to validate data acquired during the interim period.
  - 2.2.2 If the contractor laboratory operates during consecutive 8-hour periods (shifts), analysis of the performance check solution at the beginning of each 8-hour period and at the end of the final 8-hour period is sufficient.
- 2.3 Results of at least two analyses of the performance check solution must be reported with sample data collected during an 8-h period.
- 2.4 Deviations from criteria specified for the performance check solution (Section 9.2.3, Exhibit D) invalidate all sample data collected between analyses of the performance check solution, and samples shall be rerun (see Exhibit C).
3. The performance check mixture, concentration calibration solutions, and the sample and field blank fortification solutions are to be obtained from EMSL-LV. However, if not available from EMSL-LV, standards can be obtained from other sources, and solutions can be prepared in the contractor laboratory. Concentrations of all solutions containing unlabeled 2,3,7,8-TCDD and not obtained from EMSL-LV must be verified by comparison to the unlabeled 2,3,7,8-TCDD standard solution (concentration of 7.87  $\mu\text{g/mL}$ ) that is available from EMSL-LV.
4. Blanks
  - 4.1 Laboratory reagent blank -- Perform all steps in the analytical procedure (Section 11, Exhibit D) using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis, but omit an aliquot of soil or sediment.
    - 4.1.1 Except in the case noted in Section 4.1.3, a laboratory reagent blank must contain the same amount of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD that is added to samples before extraction.
    - 4.1.2 Analyze a laboratory reagent blank before any samples are extracted and analyzed.

- 4.1.3 Analyze two laboratory reagent blanks before a new batch of solvents or reagents is used for sample extraction or for column chromatographic procedures. Do not add any  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD or  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD to one blank, to demonstrate that reagents contain no impurities producing an ion current above the level of background noise for  $m/z$  328, 332 and 334.
- 4.1.4 Analyze a laboratory reagent blank along with each batch of samples.
- 4.1.5 Acceptable laboratory reagent blanks contain no ion current above the level of background signal-to-noise for any of the selected characteristic ions ( $m/z$  257, 320, 322) for unlabeled 2,3,7,8-TCDD. If the reagent blank which was extracted along with a batch of samples is contaminated, the entire batch of samples must be rerun (see Exhibit C).
- 4.1.5.1 If the above criterion is not met, check solvents, reagents, apparatus, and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed.
- 4.1.5.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.
- 4.2 Field Blanks -- Each batch of samples contains a sample of uncontaminated soil/sediment that is to be fortified with unlabeled 2,3,7,8-TCDD at a concentration of  $1 \mu\text{g}/\text{kg}$  before analysis. In addition to that field blank, a batch of samples may include a rinsate, that is a portion of solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that samples have not been contaminated by sampling equipment.
- 4.2.1 Unfortified field blank -- Analyze with procedures used for environmental samples (Section 11, Exhibit D). This blank may or may not be labeled as such (i.e., it may be a "blind" QC sample).
- 4.2.2 Fortified (Spiked) Field Blank
- 4.2.2.1 Weigh a 10-g aliquot of the specified field blank sample and add  $100 \mu\text{L}$  of the solution containing  $0.1 \text{ ng}/\mu\text{L}$  of unlabeled 2,3,7,8-TCDD,  $0.5 \text{ ng}/\mu\text{L}$  of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, and  $0.1 \text{ ng}/\mu\text{L}$  of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD. (Analysis before fortification is not required because this field blank is known not to contain a detectable concentration of unlabeled 2,3,7,8-TCDD.)

4.2.2.2 Extract with the jar procedure (Section 11.1.2, Exhibit D) and analyze a 2- $\mu$ L aliquot.

4.2.2.3 Calculate concentration (Section 12.1, Exhibit D) of both  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD and unlabeled 2,3,7,8-TCDD, and accuracy (Section 12.2, Exhibit D) of each measured concentration.

4.2.2.3.1 If accuracy of measured concentration of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD is  $> +40\%$ , discard the results and repeat the fortified field blank extraction and analysis with a second aliquot of the specified field blank sample (see Exhibit C).

#### 4.2.3 Rinsate Sample

4.2.3.1 To a 100- $\mu$ L aliquot of equipment rinse solvent (rinsate sample), add 100  $\mu$ L of the solution containing 0.5 ng/ $\mu$ L of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD and 0.1 ng/ $\mu$ L solution of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD.

4.2.3.2 Using a Kuderna-Danish apparatus or a rotary evaporator, concentrate the volume to approximately 5 mL.

4.2.3.3 Transfer the total 5- $\mu$ L concentrate in 1- $\mu$ L portions to a 1 mL-amber mini-vial, reducing volume as necessary with a gentle stream of dry nitrogen.

4.2.3.4 Rinse container with two 0.5 mL portions of hexane and transfer rinses to the 1- $\mu$ L amber mini-vial.

4.2.3.5 Just before analysis, reduce volume to near dryness; make to final volume of 50  $\mu$ L with isoctane. (Column chromatography is not required.)

4.2.3.6 Analyze an aliquot with the same procedures used to analyze samples (Section 11, Exhibit D).

#### 5. Duplicate Analyses

5.1 Laboratory duplicates -- In each batch of samples, locate the sample specified for duplicate analyses and analyze a second 10-g sample aliquot.

5.1.1 Results of laboratory duplicates must agree within 50% relative difference (difference expressed as percentage of the mean). If relative difference is  $> 50\%$ , Contractor shall immediately contact the Sample Management Office for resolution of the problem. Report all results.

**5.1.2 Recommended actions to help locate problem:**

**5.1.2.1** Analyze an aliquot of the performance check sample to verify satisfactory instrument performance (Section 9, Exhibit D.)

**5.1.2.2** If possible, determine that no error was made while weighing sample aliquots.

**5.1.2.3** Review analytical procedures with performing laboratory personnel.

**6. Accuracy of Measured Concentration of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD** -- For each sample and blank, calculate the percent accuracy (Section 12.2, Exhibit D) of the measured concentration of  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD. If percent accuracy is  $> +40\%$  for a sample, analyze a second aliquot of that sample and report both results (see Exhibit C). NOTE: Low or high accuracy for a blank does not require discarding sample data but indicates a potential problem with future sample data.

**7. Identification Criteria**

**7.1** If any of the four initial identification criteria (Sections 11.4.1 -11.4.4, Exhibit D) are not met, the sample is reported not to contain unlabeled 2,3,7,8-TCDD at the calculated detection limit (Section 12.3, Exhibit D).

**7.2** When the four initial identification criteria are met, but the fifth criteria, the isotopic abundance ratio for m/z 320 and 322 (Section 11.4.5, Exhibit D) is not met, that sample is presumed to contain interfering contaminants. Contractor shall use the second column chromatography procedure (Section 11.5, Exhibit D) to remove interferences from the extract, and shall reanalyze the sample. (See Exhibit C.)

**8. Blind QC Samples** -- Included among soil and sediment samples may be QC samples that are not specified as such to the performing laboratory. Types that may be included are:

**8.1 Uncontaminated soil.**

**8.1.1** If a false positive is reported for this sample, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C).

**8.2 Split samples** -- composited sample aliquots sent to more than one laboratory.

**8.3 Unlabeled field duplicates** -- two aliquots of a composited sample.

**8.4 Performance evaluation sample** -- soil/sediment sample containing a known amount of unlabeled 2,3,7,8-TCDD.

8.4.4 If the performance evaluation sample result falls outside the acceptance windows established by EPA, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C). NOTE: EPA acceptance windows are based on historical data results.

#### 9. Confirmatory Partial Scan Analysis

9.1 From each sample batch, select the sample extract containing the highest concentration of unlabeled 2,3,7,8-TCDD and analyze an aliquot by GC/MS under the same GC conditions used previously but with the MS tuned and calibrated to acquire data for the mass range m/z 150 to m/z 350. (If no sample in a batch contains unlabeled 2,3,7,8-TCDD, no confirmatory analysis is required for that batch.) Required calibration criteria for decafluorotriphenylphosphine introduced through the GC column shall be:

<u>m/z</u>	<u>Relative Intensity</u>
51	30 - 60 percent of base peak
68	< 2 percent of m/z = 69
70	< 2 percent of m/z = 69
127	40 - 60 percent of base peak
197	< 1 percent of base peak
198	100 percent (base peak)
199	5 - 9 percent of base peak
275	10 - 30 percent of base peak
365	> 1 percent of base peak
441	less than m/z = 443
442	> 40 percent of base peak
443	17 - 23 percent of m/z = 442

9.2 MS data acquisition requirements shall be:

9.2.1 Cycle time  $\leq$  1.5 seconds.

9.2.2 Acquisition of  $\geq$  5 spectra during elution of 2,3,7,8-TCDD from the GC.

9.3 Subtract an appropriate background spectrum, and plot a spectrum of 2,3,7,8-TCDD after background subtraction. (The person responsible for MS data interpretation is responsible for demonstrating that the background spectrum selected for subtraction was an appropriate spectrum.) Provide a hard copy of the background spectrum, the TCDD spectrum before subtraction, and the TCDD spectrum after subtraction. The quality of the plotted spectrum will be affected by other sample components that have approximately the same GC retention time and will be highly variable. Desired spectral features are:

Base peak = m/z 322  
Ratio of m/z 320 to 322 = 0.77  
Ratio of m/z 320 to 324 = 1.58  
Ratio of m/z 257 to 322 = 0.32  
Ratio of m/z 257 to 259 = 1.03  
Ratio of m/z 194 to 196 = 1.54  
m/z 160 and 161 =  $\geq$  1.0% of m/z 322

Because  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, the internal standard, is present in every sample and has essentially the same retention time as unlabeled 2,3,7,8-TCDD, the spectrum after background subtraction will represent a mixture. When  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD is present at a higher concentration than unlabeled 2,3,7,8-TCDD, the resultant spectrum (Figure E-1) must be normalized to m/z 322 to demonstrate desired spectral features.

10. Records - At each contractor laboratory, records must be maintained on site for six months after contract completion to document the quality of all data generated during contract performance. Before any records are disposed, written concurrence of the Contracting Officer must be obtained.
11. Magnetic tapes containing all raw GC/MS data (including performance check solution, blanks, and concentration calibration solutions) must be delivered to EMSL-LV when sufficient data to fill or nearly fill a tape have been collected or when all samples are completed, depending on which event occurs first.
12. Unused portions of samples and sample extracts must be preserved for six months after sample receipt; appropriate samples may be selected by EPA personnel for further analyses.
13. Reuse of glassware is to be minimized to avoid the risk of using contaminated glassware.

#### LABORATORY EVALUATION PROCEDURES

On a quarterly basis, the EPA Project Officer and/or designated representatives shall conduct an evaluation of the laboratory to ascertain that the laboratory is meeting contract requirements. This evaluation will consist of: 1) laboratory analysis of a performance evaluation sample, and 2) laboratory site visit by EPA officials and/or representatives. The evaluation procedures will be similar, but may not be identical to the evaluation performed as part of the pre-award bidder evaluation. (See IFB Pre-Award Bid Confirmations section.)



QA/QC Requirements for Dioxin and Furan  
Total Isomer Analysis

~~Our~~<sup>The</sup> approach to QA/QC for the tetra-hexa furans and dioxins parallels closely that for 2,3,7,8-TCDD. Items 3 through 9 of the outline below point out additional items not specifically mentioned in 2,3,7,8-TCDD methods.

1. Materials Examined for Contamination
  - A. Prior to start of project
  - B. Along with each set of analyses
2. Traceable Standards
  - A. 2,3,7,8-TCDD traceable to EPA reference
  - B. Other dioxins and furans traceable to EPA
3. Internal Standards for EACH chlorination level
  - A. TCDD internal standards traceable to EPA
  - B. Others synthesized at CAL Lab and reference to Rappe's materials
4. Column Performance
  - A. 2,3,7,8-TCDD resolution as per EPA requirements
  - B. Resolution of 1,2,3,4,7,8 and 1,2,3,6,7,8-HxCDF
5. Documentation of Mass Spectrometer Resolution (Low or High)
  - A. Hardcopy of profile data
6. Cleanup column chromatography Performance
  - A. 1,3,6,8-TCDD to HxCDD recovery within 30% of Internal Standards
7. Duplicate Analyses
  - A. Duplicates within 30% for most congeners.
8. Spikes of Representative Tetra-Hexa Chlorodioxins and Furans
  - A. Recovery should be within 30%
9. Detection Limit Calculated for each Chlorination Level in each Sample

Please note that duplicates and matrix spikes are regarded as billable samples.

APPENDIX D: SOURCES OF STANDARDS AND INTERNAL STANDARDS

## CAL LABS OWNED DIBENZODIOXINS

<u>Isomer</u>	<u>Source</u>
2,7-DCDD	RFR Corp.
1,2,4-TrCDD	RFR Corp.
1,2,3,4-TCDD	Christoffer Rappe
1,3,6,8-TCDD	Christoffer Rappe
2,3,7,8-TCDD	Radian Corp, USEPA
1,2,3,7,8-PnCDD	KOR Isotopes
1,2,3,4,7,8-HxCDD	KOR Isotopes
1,2,3,4,6,7,8-HpCDD	KOR Isotopes
OCDD	Ultra Scientific

## CAL LABS OWNED INTERNAL STANDARDS

<u>Isomer</u>	<u>Source</u>
13C-2,3,7,8-TCDF	Cambridge Isotope Lab (CIL)
13C-1,2,3,7,8-PnCDF	CAL LAB Synthesized, CIL
13C-2,3,4,7,8-PnCDF	CAL LAB Synthesized, CIL
13C-1,2,3,4,7,8-HxCDF	CAL LAB Synthesized
13C-1,2,3,4,6,7,8-HpCDF	CAL LAB Synthesized
13C-1,2,3,4,7,8,9-HpCDF	CAL LAB Synthesized
13C-OCDF	CAL LAB Synthesized
13C-2,3,7,8-TCDD	KOR Istopes, USEPA
13C-1,2,3,7,8-PnCDD	CAL LAB Synthesized, CIL
13C-1,2,3,4,7,8-HxCDD	CAL LAB Synthesized, CIL
13C-1,2,3,4,6,7,8-HpCDD	CAL LAB Synthesized
13C-OCDD	KOR Istopes & CAL LAB Synthes
Surrogate:	
37Cl-2,3,7,8-TCDD	KOR Isotopes, USEPA

CAL LABS OWNED DIBENZOFURANS

<u>Isomer</u>	<u>Source</u>
2,8-DCDF	RFR Corp.
1,2,3,9-TCDF	Christoffer Rappe
1,2,4,7-TCDF	Christoffer Rappe
1,2,4,8-TCDF	Christoffer Rappe
1,2,6,7-TCDF	Christoffer Rappe
1,2,7,8-TCDF	Christoffer Rappe
1,2,7,9-TCDF	Christoffer Rappe
1,3,4,6-TCDF	Christoffer Rappe
1,3,6,7-TCDF	Christoffer Rappe
1,3,6,8-TCDF	Christoffer Rappe
1,3,7,9-TCDF	Christoffer Rappe
1,4,6,7-TCDF	Christoffer Rappe
1,4,6,9-TCDF	Christoffer Rappe
2,3,4,7-TCDF	Christoffer Rappe
2,3,4,8-TCDF	Christoffer Rappe
2,3,6,7-TCDF	Christoffer Rappe
2,3,6,8-TCDF	Christoffer Rappe
2,3,7,8-TCDF	Christoffer Rappe & Radian Corp.
2,4,6,7-TCDF	Christoffer Rappe
2,4,6,8-TCDF	Christoffer Rappe
1,2,3,4,8-PnCDF	Christoffer Rappe
1,2,3,7,8-PnCDF	Cambridge Isotope Lab
1,2,4,6,8-PnCDF	Christoffer Rappe
1,2,4,7,8-PnCDF	Christoffer Rappe
2,3,4,6,8-PnCDF	Christoffer Rappe
2,3,4,7,8-PnCDF	Christoffer Rappe
1,2,3,4,6,8-HxCDF	Christoffer Rappe
1,2,3,4,7,8-HxCDF	Cambridge Isotope Lab
1,2,3,4,7,9-HxCDF	Christoffer Rappe
1,2,4,6,7,8-HxCDF	Christoffer Rappe
1,2,4,6,8,9-HxCDF	Christoffer Rappe
2,3,4,6,7,8-HxCDF	Christoffer Rappe
1,2,3,4,6,7,8-HpCDF	Christoffer Rappe & Cambridge Isotopes
1,2,3,4,6,8,9-HpCDF	Christoffer Rappe
1,2,3,4,7,8,9-HpCDF	Christoffer Rappe
OCDF	Christoffer Rappe & Ultra Scientific

APPENDIX S

CALIFORNIA ANALYTICAL LABORATORIES DATA SHEETS FOR  
DIOXIN/FURAN ANALYSES, ORGANIC COMPOUND ANALYSES,  
AND INORGANIC ANALYSES

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CCC

TCDD DATA REPORT  
California Analytical Laboratories  
2544 Industrial Blvd.  
W. Sacramento, CA 95691

Lab: California Analytical Laboratories  
Case No. 21932  
Batch/Shipment No.

Report Date: 9-25-85  
Column: SP-2331

Cal Labs ID	Sample Number	C U	Aliquot (sample)	ng/sample		Det. Inst	Time	320/ 322		332/ 334		320 322		320	322	325	328*	332	334	Ca
				TCDD Meas	ng/sample			Surry Mess	Surry Acc't	ng/sample	Surry Mess	Surry Acc't								
21932MB	METHOD BLANK	Y	1.00	MD	0.14	8	09/25/85	16:59:00	0.72	0.72	9.42	94	1117640	1556910	555546	938761	1096630	1526890		
21932BNS	METH.BLNK NS	Y	1.00	13.6	0.14	8	09/25/85	17:20:00	0.72	0.75	9.81	98	1117640	1556910	555546	2036677	2336860	3127880	1348 Recover	
21932-1	1131	Y	1.00	MD	0.59	8	09/25/85	17:38:00	0.71	0.71	9.09	91	21096	16597	12632	203381	265439	343673		
21932-2	1132	Y	1.00	MD	0.27	8	09/25/85	17:58:00	1.27	0.77	9.69	97	21096	16597	12632	2315250	2730770	3559650	ntie Unrecq	
21932-3	1133	Y	1.00	1.5	0.27	8	09/25/85	18:15:00	0.85	0.75	9.82	98	133020	157416	62752	1952223	2239600	2994490		
21932-4	1134	Y	1.00	MD	0.17	8	09/25/85	18:59:00	1.16	0.75	9.58	96	5477	4724	5756	850411	999907	1336710		
21937-1	1135	Y	1.00	MD	0.14	8	09/25/85	19:28:00	0.65	0.73	9.76	98	11472	17556	1997447	2302480	3060840	ntie Unrecq		
21937-2	1136	Y	1.00	MD	0.28	8	09/25/85	20:03:00	0.74	0.75	9.52	95	6080	8208	1603652	1873420	2559090	ntie Unrecq		
21937-3	1137	Y	1.00	MD	0.22	8	09/25/85	20:21:00	0.75	0.75	9.70	97	116104	141300	53943	1022516	1173730	1586130		
21937-4	1138	Y	1.00	0.14	0.13	8	09/25/85	20:46:00	0.75	0.74	9.75	97	6960	11936	4581	1444240	1684630	2235380		
21937-5	1139	Y	1.00	MD	0.13	8	09/25/85	21:13:00	0.75	0.78	9.54	95	116104	141300	53943	1863680	2247320	2896000		
21937-6	1140	Y	1.00	0.11	0.11	8	09/25/85	21:36:00	0.82	0.77	10.04	100	116104	141300	53943	1891322	2279920	2959730		
21937-7	1141	Y	1.00	1.1	0.12	8	09/25/85	22:12:00	0.82	0.77	10.04	100	116104	141300	53943	2419758	2749520	3591470		
21937-8	1143	Y	1.00	MD	0.12	8	09/25/85	22:55:00	0.77	0.77	9.72	97	116104	141300	53943	1231550	1448880	1886030		

MB = Method Blank  
 P = Partial Scan/Confirmatory Analysis  
 NS = Native TCDD Spike  
 D = Duplicate/Fortified Field Blank  
 RI = Re-injection  
 FB = Field Blank  
 MD = Not Detected  
 DL = Detection Limit  
 RA = Re-extraction

\*Corrected for contribution by native TCDD; 0.9% of m/z 322 subtracted

Comments:

Ratio Unacceptable - The sample shows ion current for m/e 320 and m/e 322, but their ratio is unacceptable (outside the 0.67 - 0.87 window) for positive 2,3,7,8-TCDD identification.

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21591

CLIENT ID: HU-NCBC-R1-01

Date Analyzed: 8/30/85

Column: DB-

CAL ID: 21591-1

Weight: 10.06 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	16.3 3.7	- -
penta (12378) (23478)	20.7 0.21 0.11	- - -
hexa (123478)	1.0 ND	- 0.084
DIOXINS		
tetra (total) (2378+1234)	196 A 193	- -
penta (12378)	4.6 1.5	- -
hexa (123478)	2.1 ND	- 0.21

‡ Accuracy 37Cl-TCDD = 94‡

A = Data taken from 2,3,7,8-TCDD specific analysis

ND = Not Detected

PREPARED BY:

[Signature]

APPROVED BY:

[Signature]

DATE:

1/21/86

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21413

HU-MC BC

CLIENT ID: R1-02

Date Analyzed: 10/16/85

Column: DB-

CAL ID: 21413-2RXRX\$

Weight: 10.15 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	ND ND	0.036 0.036
penta	ND	0.063
hexa	ND	0.15
DIOXINS		
tetra (total) (2378+1234)	ND ND	0.057 0.057
penta	ND	0.29
hexa	ND	0.24

‡ Accuracy 37Cl-TCDD = 94‡

ND = Not Detected

PREPARED BY: CJH

APPROVED BY: [Signature]

DATE: 11/2/86

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21591

R2-02

CLIENT ID: HV-NCBC-02

Date Analyzed: 10/16/85 Column: DB-

CAL ID: 21591-3RX\$

Weight: 10.00 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total) (2378*)	0.028 0.028	- -
penta	ND	0.019
hexa	ND	0.020
DIOXINS		
tetra (total) (2378+1234)	ND ND	0.036 0.036
penta	ND	0.18
hexa	ND	0.070

% Accuracy 37Cl-TCDD = 89%

ND = Not Detected

PREPARED BY: RJA

APPROVED BY: JMO

DATE: 1/21/86



California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO. 21591

CLIENT ID: HV-NCBC-R2-03

Date Analyzed: 10/16/85 Column: DB-

CAL ID: 21591-04RX\$

Weight: 10.06 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
tetra (total)	0.54	-
penta (12378)	0.29	-
(23478)	ND	0.033
	ND	0.033
hexa	ND	0.074

DIOXINS

tetra (total)	0.87	-
(2378+1234)	0.49	-
penta	ND	0.47
hexa	ND	0.26

Accuracy 37Cl-TCDD = 101%

ND = Not Detected

PREPARED BY: Paul

APPROVED BY: MMS

DATE: 1/2/86

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO: 21413

CLIENT ID: HU-NCBC-R1-09

DATE ANALYZED: 9/17/85

CAL ID: 21413-3

WEIGHT: 10.05 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
Total TCDF	ND	0.011
2,3,7,8-TCDF	ND	0.011
Total PCDF	ND	0.040
1,2,3,7,8-PCDF	ND	0.040
2,3,4,7,8-PCDF	ND	0.040
Total HCDF	ND	0.021
1,2,3,4,7,8-HCDF	ND	0.021
<b>DIOXINS</b>		
Total TCDD	ND	0.037
2,3,7,8-TCDD	ND	0.037
Total PCDD	ND	0.18
1,2,3,7,8-PCDD	ND	0.18
Total HCDD	ND	0.046
1,2,3,4,7,8-HCDD	ND	0.046

ND = Not Detected  
RX = Re-extraction

RI = Reinjection

PREPARED BY:                     

APPROVED BY:                     

DATE:

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO: 21591

CLIENT ID: HU-NCBC-R2-09A (19:00)

DATE ANALYZED: 9/17/85

CAL ID: 21591-5

WEIGHT: 10.13 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
Total TCDF	ND	0.0034
2,3,7,8-TCDF	ND	0.0034
Total PCDF	ND	0.013
1,2,3,7,8-PCDF	ND	0.013
2,3,4,7,8-PCDF	ND	0.013
Total HCDF	ND	0.025
1,2,3,4,7,8-HCDF	ND	0.025
DIOXINS		
Total TCDD	ND	0.013
2,3,7,8-TCDD	ND	0.013
Total PCDD	ND	0.15
1,2,3,7,8-PCDD	ND	0.15
Total HCDD	ND	0.043
1,2,3,4,7,8-HCDD	ND	0.043

ND = Not Detected  
RX = Re-extraction

RI = Reinjection

PREPARED BY: JW

APPROVED BY: PIV

DATE: 10/22/85

California Analytical Laboratories, Inc.

POLYCHLORINATED DIOXIN/FURAN ANALYSIS

TICKET NO: 21591

CLIENT ID: HU-NCBC-R2-09A (19:40) *unclassified*

DATE ANALYZED: 9/17/85

CAL ID: 21591-6

WEIGHT: 10.14 g

FURANS	AMOUNT FOUND (ng/g)	DETECTION LIMIT (ng/g)
Total TCDF	ND	0.0075
2,3,7,8-TCDF	ND	0.0075
Total PCDF	ND	0.021
1,2,3,7,8-PCDF	ND	0.021
2,3,4,7,8-PCDF	ND	0.021
Total HCDF	ND	0.033
1,2,3,4,7,8-HCDF	ND	0.033
DIOXINS		
Total TCDD	ND	0.018
2,3,7,8-TCDD	ND	0.018
Total PCDD	ND	0.27
1,2,3,7,8-PCDD	ND	0.27
Total HCDD	ND	0.058
1,2,3,4,7,8-HCDD	ND	0.058

ND = Not Detected  
RX = Re-extraction

RI = Reinjection

PREPARED BY: *[Signature]*

APPROVED BY: *[Signature]*

DATE: 10/22/85

Organics Analysis Data Sheet  
(Page 1)

Exhibit 3

Laboratory Name: California Analytical Laboratories, Inc.

Case No: 21591

Lab Sample ID No: 21591-1

QC Report No: NR

Sample Matrix: SOIL

Contract No: NR

Data Release Authorized By: PRS

Date Sample Received: 8/29/85

Volatile Compounds

Concentration: Medium

Date Extracted/Prepared: 9/16/85

Date Analyzed: 9/16/85

Conc/Dil Factor: 100 pH: NR

Percent Moisture: NR

Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-4	Chloromethane	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	800 U
67-64-1	Acetone	800 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethane	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-5	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-83-3	2-Butanone	800 U
71-55-6	1,1,1-Trichloroethane	200 U
56-23-6	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
75-37-6	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichloropropane	200 U
78-01-6	Trichloroethane	200 U
124-48-1	Dibromochloromethane	200 U
78-00-6	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-6	cis-1,3-Dichloropropane	200 U
110-75-8	2-Chloroethylvinylether	1000 U
75-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	800 U
881-78-6	2-Hexanone	800 U
127-18-4	Tetrachloroethane	200 U
78-34-6	1,1,2,2-Tetrachloroethane	200 U
108-86-3	Toluene	200 U
108-86-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	800 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

Value If the result is a value greater than or equal to the detection limit, report the value.

U Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample

J Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g. 10U) if limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3J

C This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides  $\geq 10\text{ng}/\mu\text{l}$  in the final extract should be confirmed by GC/MS

B This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.

Other Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

NA Not Analyzed.  
S See cover letter.  
NR Not Required.  
S Spiked Compound.

1006

# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85, 9/3/85  
 Date Analyzed: 9/12/85  
 Conc:DiL Factor: 0.57G/ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	1000 U
111-44-4	bis(2-Chloroethyl)Ether	4000 U
95-57-8	2-Chlorophenol	1000 U
541-73-1	1,3-Dichlorobenzene	4000 U
106-46-7	1,4-Dichlorobenzene	4000 U
100-51-6	Benzyl Alcohol	4000 U
95-50-1	1,2-Dichlorobenzene	4000 U
95-48-7	2-Methylphenol	4000 U
39638-32-9	bis(2-chloroisopropyl)Ether	8000 U
106-44-5	4-Methylphenol	4000 U
621-64-7	N-Nitroso-Di-n-Propylamine	4000 U
67-72-1	Hexachlorocyclohexane	4000 U
98-95-3	Nitrobenzene	1000 U
78-59-1	Isophorone	4000 U
78-75-5	2-Nitrophenol	1000 U
105-67-9	2,4-Dimethylphenol	4000 U
65-85-0	Benzole Acid	2000 U
111-91-1	bis(2-Chloroethoxy)Methane	4000 U
120-83-2	2,4-Dichlorophenol	1000 U
120-82-1	1,2,4-Trichlorobenzene	4000 U
91-20-3	Naphthalene	4000 U
106-47-8	4-Chloroaniline	4000 U
87-68-3	Hexachlorocyclopentadiene	4000 U
59-50-7	4-Chloro-2-Methylphenol	4000 U
91-57-6	2-Methylnaphthalene	4000 U
124-14	Hexachlorocyclopentadiene	4000 U
89-06-2	2,4,6-Trichlorophenol	2000 U
95-95-4	2,4,5-Trichlorophenol	4000 U
91-58-7	2-Chloronaphthalene	4000 U
88-74-4	2-Nitroanisole	2000 U
131-11-1	Dimethylphthalate	4000 U
208-96-8	Acenaphthylene	4000 U
99-09-2	3-Nitroaniline	2000 U

CAS Number		ug/Kg
83-32-9	Acenaphthene	4000 U
51-28-5	2,4-Dinitrophenol	2000 U
100-02-7	4-Nitrophenol	2000 U
132-64-9	Dibenzofuran	4000 U
121-14-2	2,4-Dinitrotoluene	8000 U
606-20-2	2,6-Dinitrotoluene	8000 U
84-65-2	Diethylphthalate	4000 U
7005-72-3	4-Chlorophenyl-phenylether	4000 U
86-73-7	Fluorene	4000 U
100-01-6	4-Nitroaniline	2000 U
534-52-1	4,6-Dinitro-2-Methylphenol	8000 U
86-30-6	N-Nitrosodiphenylamine(1)	4000 U
101-65-3	4-Bromophenyl-phenylether	4000 U
118-74-1	Hexachlorobenzene	4000 U
87-86-6	Pentachlorophenol	4000 U
85-01-3	Phenanthrene	4000 U
120-12-7	Anthracene	4000 U
84-74-2	Di-n-Butylphthalate	4000 U
206-44-0	Fluoranthene	4000 U
129-00-0	Pyrene	4000 U
85-64-7	Butylbenzylphthalate	4000 U
91-94-1	3,3'-Dichlorobenzidine	8000 U
56-53-3	Benzo(a)Anthracene	4000 U
117-91-7	bis(2-Ethylhexyl)Phthalate	4000 U
218-01-9	Chrysene	8000 U
117-84-0	Di-n-Octyl Phthalate	4000 U
205-99-2	Benzo(b)Fluoranthene	8000 U
207-06-9	Benzo(k)Fluoranthene	8000 U
50-32-8	Benzo(a)Pyrene	8000 U
193-33-5	Indeno(1,2,3-cd)Pyrene	8000 U
53-70-3	Dibenzo(a,h)Anthracene	8000 U
191-24-2	Benzo(g,h,i)Perylene	8000 U

(1) - Cannot be separated from diphenylamine

1007

## Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85, 9/3/85  
 Date Analyzed: 9/19/85  
 Conc/Dil Factor: 0.11G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	88 U
319-86-7	Beta-BHC	88 U
319-86-8	Delta-BHC	88 U
58-89-9	Gamma-BHC (Lindane)	88 U
76-44-8	Heptachlor	88 U
308-00-3	Aldrin	88 U
1024-87-3	Heptachlor Epoxide	88 U
988-88-8	Endosulfan I	100 U
80-57-1	Dieldrin	100 U
73-88-8	4,4'-DDE	100 U
73-30-8	Endrin	100 U
33213-66-8	Endosulfan II	100 U
73-84-8	4,4'-DDD	200 U
1031-87-8	Endosulfan Sulfate	200 U
89-29-3	4,4'-DDT	200 U
73-43-6	Methoxychlor	1000 U
83494-70-8	Endrin Ketone	NA
87-74-8	Chlordane	1000 U
8001-36-2	Tempone	10000 U
12874-11-2	Aroclor-1016	NA
11104-29-2	Aroclor-1221	NA
11141-16-8	Aroclor-1232	NA
83489-21-8	Aroclor-1242	1000 U
12873-29-6	Aroclor-1248	1000 U
11087-69-1	Aroclor-1254	1000 U
11096-82-8	Aroclor-1260	1000 U

$V_i$  = Volume of extract injected (ul)  
 $V_s$  = Volume of water extracted (ml)  
 $W_s$  = Weight of sample extracted (g)  
 $V_t$  = Volume of total extract (ul)

1008

$V_s = NR$       or       $W_s = 0.11$        $V_t = 5000$        $V_i = 5$

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Organics Analysis Data Sheet  
(Page 1)

Laboratory Name: California Analytical Laboratories, Inc.  
 Lab Sample ID No: 21591-2  
 Sample Matrix: SOIL  
 Data Release Authorized By: PLS

Case No: 21591  
 GC Report No: NR  
 Contract No: NR  
 Date Sample Received: 3/29/85

**Volatile Compounds**

Concentration: ug/Kg  
 Date Extracted/Prepared: 3/16/85  
 Date Analyzed: 3/16/85  
 Conc/Dil Factor: 100 pH: NR  
 Percent Moisture: NR  
 Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-0	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	500 U
67-64-1	Acetone	500 U
75-15-0	Carbon Disulfide	200 U
5-35-4	1,1-Dichloroethane	200 U
78-34-3	1,1-Dichloroethane	200 U
156-80-6	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-92-3	2-Butanone	500 U
71-55-8	1,1,1-Trichloroethane	200 U
56-23-5	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
78-87-8	1,2-Dichloropropene	200 U
10061-02-6	Trans-1,3-Dichloropropene	200 U
79-01-6	Trichloroethene	200 U
124-48-1	Dibromochloromethane	200 U
79-00-6	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-6	cis-1,3-Dichloropropene	200 U
110-78-8	2-Chloroethylvinylether	1000 U
78-25-2	Eromofom	200 U
102-10-1	4-Methyl-2-Pentanone	500 U
981-78-6	2-Hexanone	500 U
127-18-4	Tetrachloroethene	200 U
79-34-6	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-90-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	500 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

**Data Reporting Qualifiers**

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value.
- U** Indicates compound was analyzed for but not detected. Report the maximum detection limit for the sample with the U (e.g. 100U) based on the necessary concentration/dilution factors. (This is not necessarily the instrument detection limit). The footnote should read: U Compound was analyzed for but not detected. The number is the maximum detection limit for the sample.
- J** Indicates an estimate value. This flag is used only when estimating a concentration. It is used only for unidentified compounds where a 10% response is assumed or when the mass spectral data is used for the identification of a compound that meets the 10% criterion. If the result is less than the specified detection limit but greater than zero (e.g. 10U) and the detection limit is 100U and a concentration of sufficient data is reported, report as U.
- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides  $\geq 10\text{ng}/\mu\text{l}$  in the final extract should be confirmed by GC/MS.
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed
- R** See cover letter
- NR** Not Required
- S** Spiked Compound.

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# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: LOW  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/12/85  
 Conc/Dil Factor: 30G/10ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	3400
111-44-4	bis(2-Chloroethyl)Ether	1000 U
95-57-8	2-Chlorophenol	1000 U
341-73-1	1,3-Dichlorobenzene	1000 U
106-46-7	1,4-Dichlorobenzene	1000 U
100-51-6	Benzyl Alcohol	1000 U
95-50-1	1,2-Dichlorobenzene	1000 U
95-48-7	2-Methylphenol	1000 U
39638-32-8	bis(2-chloroisopropyl)Ether	2000 U
106-44-5	4-Methylphenol	1000 U
621-64-7	N-Nitroso-Di-n-Propylamine	1000 U
67-72-1	Hexachloroethane	1000 U
98-95-3	Nitrobenzene	1000 U
78-59-1	Isophorene	1000 U
88-75-5	2-Nitrophenol	2000 U
105-67-9	2,4-Dimethylphenol	1000 U
65-85-0	Benzoic Acid	3000 U
111-91-1	bis(2-Chloroethoxy)Methane	2000 U
120-83-2	2,4-Dichlorophenol	10000
120-82-1	1,2,4-Trichlorobenzene	1000 U
91-20-3	Naphthalene	1000 U
106-47-8	4-Chloroaniline	1000 U
67-68-3	Hexachlorobutadiene	1000 U
59-50-7	4-Chloro-3-Methylphenol	1000 U
91-57-6	2-Methylnaphthalene	1000 U
77-47-4	Hexachlorocyclopentadiene	1000 U
88-06-2	2,4,6-Trichlorophenol	3000 S
95-85-4	2,4,5-Trichlorophenol	S
91-58-7	2-Chloronaphthalene	1000 U
88-74-4	2-Nitroaniline	3000 U
131-11-3	Dimethyl Phthalate	1000 U
208-96-8	Acenaphthylene	1000 U
89-09-2	3-Nitroaniline	3000 U

CAS Number		ug/Kg
83-32-8	Acenaphthene	1000 U
81-28-6	2,4-Dinitrophenol	3000 U
100-02-7	4-Nitrophenol	3000 U
132-64-8	Diisoxafuran	1000 U
121-14-2	2,4-Dinitrotoluene	2000 U
206-20-2	2,6-Dinitrotoluene	2000 U
84-86-2	Diethylphthalate	1000 U
7005-72-3	4-Chlorophenyl-phenylether	1000 U
86-73-7	Fluorene	1000 U
100-01-6	4-Nitroaniline	3000 U
834-62-1	4,6-Di-nro-2-Methylphenol	2000 U
86-30-6	N-Nitrosodiphenylamine(1)	1000 U
101-65-3	4-Bromophenyl-phenylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
67-86-6	Pentachlorophenol	1000 U
88-01-8	Phenanthrene	1000 U
120-12-7	Anthracene	1000 U
84-74-2	Di-n-Butylphthalate	1000 U
206-44-0	Fluoranthene	1000 U
129-00-0	Pyrene	1000 U
85-66-7	Butylbenzylphthalate	1000 U
91-84-1	3,3'-Dichlorobenzidine	2000 U
86-66-3	Benzo(a)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthalate	1000 U
218-01-8	Chrysene	2000 U
117-84-0	Di-n-Octyl Phthalate	1000 U
206-99-2	Benzo(b)Fluoranthene	2000 U
207-08-9	Benzo(k)Fluoranthene	2000 U
50-32-6	Benzo(a)Pyrene	2000 U
193-39-6	Indeno(1,2,3-cd)Pyrene	2000 U
53-70-3	Dibenz(a,h)Anthracene	2000 U
191-24-2	Benzo(g,h,i)Perylene	2000 U

1040

(1) - Cannot be separated from diphenylamine

CLF: 10/11/85

Form I

Prepared by: JB

7A

# Organics Analysis Data Sheet (Page 3)

## Pesticide/PCBs

Concentration: LOW GPC Cleanup: NO  
 Date Extracted/Prepared: 8/27/85 Separatory Funnel Extraction: YES  
 Date Analyzed: 9/19/85 Continuous Liquid - Liquid Extraction: NO  
 Conc/Dil Factor: 1.57G/50ML

CAS Number		ug/Kg
319-84-6	Alpha-BHC	30 U
319-85-7	Beta-BHC	30 U
319-86-8	Delta-BHC	30 U
58-89-9	Gamma-BHC (Lindene)	30 U
75-44-8	Heptachlor	30 U
309-00-2	Aldrin	30 U
1024-87-3	Heptachlor Epoxide	30 U
959-98-8	Endosulfan I	70 U
60-57-1	Dieldrin	70 U
72-55-6	4,4'-DDE	70 U
72-20-8	Endrin	70 U
33213-65-9	Endosulfan II	70 U
72-54-8	4,4'-DDD	130 U
1031-07-8	Endosulfan Sulfate	130 U
50-29-3	4,4'-DDT	130 U
72-43-5	Methoxychlor	670 U
53464-70	Endrin Ketone	NA
57-74-9	Chlordane	670 U
61-35-2	Toxaphene	6700 U
12674-11-2	Aroclor-1016	NA
11104-28-2	Aroclor-1221	NA
11141-16-5	Aroclor-1232	NA
53469-21-9	Aroclor-1242	670 U
12672-29-6	Aroclor-1248	670 U
11097-69-1	Aroclor-1254	670 U
11096-82-5	Aroclor-1260	670 U

$V_i$  = Volume of extract injected (ul)

$V_s$  = Volume of water extracted (ml)

$W_s$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

$V_s = NR$

or  $W_s = 1.57$

$V_t = 50000$

$V_i = 5$

1041

# Organics Analysis Data Sheet (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.

Case No: 21413

Lab Sample ID No: 21413-2

QC Report No: NR

Sample Matrix: SOIL

Contract No: NR

Data Release Authorized By: PLS

Date Sample Received: 8/21/85

## Volatile Compounds

Concentration: Medium

Date Extracted/Prepared: 11/14/85

Date Analyzed: 11/14/85

Conc/Dil Factor: 100 pH: NR

Percent Moisture: NR

Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
78-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	800 U
67-64-1	Acetone	800 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethane	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-6	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-93-3	2-Butanone	800 U
71-55-6	1,1,1-Trichloroethane	200 U
56-23-5	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
78-87-6	1,2-Dichloropropene	200 U
10061-02-6	Trans-1,3-Dichloropropene	200 U
78-01-6	Trichloroethane	200 U
124-48-1	Dibromochloromethane	200 U
78-00-5	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-5	cis-1,3-Dichloropropene	200 U
110-75-8	2-Chloroethylvinylether	1000 U
78-29-2	Bromobenzene	200 U
108-10-1	4-Methyl-2-Pentanone	800 U
891-78-6	2-Hexanone	800 U
127-18-4	Tetrachloroethane	200 U
75-34-5	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-80-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	800 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

### Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- |  |  |
|--|--|
| <p><b>Value</b> If the result is a value greater than or equal to the detection limit, report the value.</p> <p><b>U</b> Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution factors. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample</p> <p><b>J</b> Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g. 10U). If limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3J</p> | <p><b>C</b> This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides <math>\geq</math> 10ng/ml in the final extract should be confirmed by GC/MS</p> <p><b>B</b> This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.</p> <p><b>Other</b> Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.</p> <p><b>NA</b> Not Analyzed.</p> <p><b>#</b> See cover letter.</p> <p><b>NR</b> Not Required.</p> <p><b>S</b> Spiked Compound.</p> |
|--|--|

253

CLF: 11/14/85

Form I

Prepared by: gbs

10/85

501

4 2-86  
submitted

# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: MEDIUM  
 Date Extracted/Prepared: 9/3/85, 8/27/85  
 Date Analyzed: 4/11/88  
 Conc/Dil Factor: 0.5g/0.5ml

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
106-95-2	Phenol	1000 U
111-44-4	bis(2-Chloroethyl)Ether	1000 U
95-57-8	2-Chlorophenol	1000 U
541-73-1	1,3-Dichlorobenzene	1000 U
106-46-7	1,4-Dichlorobenzene	1000 U
100-61-6	Benzyl Alcohol	1000 U
96-80-1	1,2-Dichlorobenzene	1000 U
95-48-7	2-Methylphenol	1000 U
39638-32-9	bis(2-chloroisopropyl)Ether	2000 U
106-44-5	4-Methylphenol	1000 U
621-64-7	N-Nitroso-Di-n-Propylamine	1000 U
67-72-1	Hexachloroethane	1000 U
98-08-3	Nitrobenzene	1000 U
78-59-1	Isochlorane	1000 U
84-73-6	2-Nitrophenol	2000 U
105-67-9	2,4-Dimethylphenol	1000 U
55-85-0	Benzoic Acid	5000 U
111-91-1	bis(2-Chloroethoxy)Methane	2000 U
120-83-2	2,4-Dichlorophenol	1000 U
120-82-1	1,2,4-Trichlorobenzene	1000 U
91-20-3	Naphthalene	1000 U
106-47-8	4-Chloroaniline	1000 U
77-48-1	Hexachlorobutadiene	1000 U
55-70-7	4-Chloro-3-Methylphenol	1000 U
91-57-6	2-Methylnaphthalene	1000 U
77-47-4	Hexachlorocyclopentadiene	1000 U
68-06-2	2,4,6-Trichlorophenol	1000 U
95-95-4	2,4,5-Trichlorophenol	5000 U
91-68-7	2-Chloronaphthalene	1000 U
84-74-4	2-Nitroaniline	5000 U
131-11-3	Dimethyl Phthalate	1000 U
208-96-8	Acenaphthylene	1000 U
98-08-2	3-Nitroaniline	5000 U

CAS Number		ug/Kg
83-37-6	Acenaphthene	1000 U
81-28-6	2,4-Dinitrophenol	5000 U
100-02-7	4-Nitrophenol	5000 U
132-64-9	O-Benzofuran	1000 U
121-14-2	2,4-Dinitrotoluene	2000 U
606-20-2	2,6-Dinitrotoluene	2000 U
84-66-2	Diethylphthalate	1000 U
7006-72-3	4-Chlorophenyl-phenylether	1000 U
86-73-7	Fluorene	1000 U
100-01-6	4-Nitroaniline	3000 U
534-52-1	4,6-Dinitro-2-Methylphenol	2000 U
86-30-6	N-Nitrosodiphenylamine(1)	1000 U
101-88-3	4-Bromophenyl-phenylether	1000 U
116-74-1	Hexachlorobenzene	1000 U
87-38-6	Pentachlorophenol	1000 U
85-01-8	Phenanthrene	1000 U
120-12-7	Anthracene	1000 U
84-74-2	Di-n-Butylphthalate	1200
206-44-0	Fluoranthene	1000 U
129-00-0	Pyrene	1000 U
88-68-7	Butylbenzylphthalate	1000 U
91-94-1	3,3'-Dichlorobenzidine	2000 U
56-55-3	Benzo(a)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthalate	480 J
218-01-8	Chrysene	1000 U
117-84-0	Di-n-Octyl Phthalate	1000 U
205-99-2	Benzo(b)Fluoranthene	1000 U
207-08-3	Benzo(k)Fluoranthene	1000 U
50-32-6	Benzo(a)Pyrene	1000 U
193-39-1	Indeno(1,2,3-cd)Pyrene	1000 U
53-70-3	Dibenz(a,h)Anthracene	1000 U
191-24-2	Benzo(g,h,i)Perylene	1000 U

254 cannot be separated from diphenylamine  
 Form I Prepared by: *[Signature]*

## Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85, 9/13/85  
 Date Analyzed: 9/18/85  
 Conc/Dil Factor: 0.10G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	80 U
319-84-7	Beta-BHC	80 U
319-84-8	Delta-BHC	80 U
58-89-8	Gamma-BHC (Lin Janu)	80 U
76-44-8	Heptachlor	80 U
309-00-2	Aldrin	80 U
1024-67-3	Heptachlor Epoxide	80 U
969-89-8	Endosulfan I	100 U
89-87-1	Dieldrin	100 U
72-85-8	4,4'-DDE	100 U
72-20-8	Endrin	100 U
33213-88-9	Endosulfan II	100 U
72-84-8	4,4'-DDD	200 U
1021-67-8	Endosulfan Sulfate	200 U
89-29-3	4,4'-DDT	200 U
72-43-5	Methoxychlor	1000 U
83494-70-8	Endrin Ketone	NA
87-74-8	Chlordane	1000 U
8001-35-2	Toxaphene	10000 U
12674-11-2	Aroclor-1016	NA
11104-28-2	Aroclor-1221	NA
11141-18-5	Aroclor-1232	NA
83469-21-8	Aroclor-1242	1000 U
12672-29-6	Aroclor-1248	1000 U
11087-89-1	Aroclor-1254	1000 U
11086-82-5	Aroclor-1260	1000 U

- $V_i$  = Volume of extract injected (ul)
- $V_s$  = Volume of water extracted (ml)
- $W_s$  = Weight of sample extracted (g)
- $V_t$  = Volume of total extract (ul)

503

$V_s = \text{NR}$       or       $W_s = 0.10$        $V_t = 5000$        $V_i = 5$

Organics Analysis Data Sheet  
(Page 1)

Laboratory Name: California Analytical Laboratories, Inc.  
 Lab Sample ID No: 21591-3  
 Sample Matrix: SOIL  
 Data Release Authorized By: PLS

Case No: 21591  
 QC Report No: NR  
 Contract No: NR  
 Date Sample Received: 6/29/85

Volatile Compounds

Concentration: Medium  
 Date Extracted/Prepared: 9/16/85  
 Date Analyzed: 9/16/85  
 Conc/Dil Factor: 100 pH: NR  
 Percent Moisture: NR  
 Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-8	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	500 U
67-64-1	Acetone	500 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethene	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-5	Trans-1,2-Dichloroethene	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-23-3	2-Butanone	500 U
71-55-6	1,1,1-Trichloroethane	200 U
56-23-8	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
78-87-5	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichloropropane	200 U
79-01-6	Trichloroethene	200 U
124-48-1	Dibromochloromethane	200 U
79-00-5	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-6	cis-1,3-Dichloropropane	200 U
110-75-5	2-Chloroethylvinylether	1000 U
75-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	500 U
591-78-6	2-Hexanone	500 U
127-18-4	Tetrachloroethene	200 U
78-34-5	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-90-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	500 U
100-42-5	Styrene	200 U
	Total Xylenes	200 U

Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value.
- U** Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- E** Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10U). If limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3U.
- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS.
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed.
- S** See cover letter.
- NR** Not Required.
- S** Spiked Compound.

libe

256

4-22-86  
submittal

# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85 & 9/3/85  
 Date Analyzed: 4/11/86  
 Conc/DIL Factor: 0.51g/0.5ml

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
106-95-2	Phenol	1000 U
111-44-4	bis(2-Chloroethoxy)Ether	1000 U
95-57-8	2-Chlorophenol	1000 U
841-73-1	1,3-Dichlorobenzene	1000 U
106-46-7	1,4-Dichlorobenzene	1000 U
100-91-8	Benzyl Alcohol	1000 U
98-90-1	1,2-Dichlorobenzene	1000 U
95-48-7	2-Methylphenol	1000 U
39638-32-9	bis(2-chloroisopropyl)Ether	2000 U
106-44-8	4-Methylphenol	1000 U
621-44-7	N-Nitroso-Di-n-Propylamine	1000 U
67-72-1	Hexachloroethane	1000 U
98-95-3	Nitrobenzene	1000 U
78-96-1	Isophenone	1000 U
88-75-5	2-Nitrophenol	2000 U
106-67-9	2,4-Dimethylphenol	1000 U
65-85-3	Benzole Acid	8000 U
111-91-1	bis(2-Chloroethoxy)Methane	2000 U
120-83-2	2,4-Dichlorophenol	1000 U
120-83-1	1,2,4-Trichlorobenzene	1000 U
91-20-3	Naphthalene	1000 U
106-47-6	4-Chloroaniline	1000 U
87-68-3	Hexachlorobutadiene	1000 U
59-50-7	4-Chloro-3-Methylphenol	1000 U
91-57-5	2-Methylnaphthalene	1000 U
77-47-4	Hexachlorocyclopentadiene	1000 U
88-06-2	2,4,6-Trichlorophenol	1000 U
86-86-4	2,4,5-Trichlorophenol	3000 U
91-58-7	3-Chloronaphthalene	1000 U
88-74-4	2-Nitroaniline	8000 U
131-11-3	Dimethyl Phthalate	1000 U
208-98-8	Acenaphthylene	1000 U
88-08-2	3-Nitroaniline	8000 U

CAS Number		ug/Kg
83-32-8	Acenaphthene	1000 U
91-28-1	2,4-Dinitrophenol	8000 U
106-62-7	4-Nitrophenol	8000 U
132-64-6	Dibenzofuran	1000 U
121-14-2	2,4-Dinitrobenzene	2000 U
608-99-9	2,6-Dinitrobenzene	2000 U
84-92-2	Diethylphthalate	1000 U
7098-72-9	4-Chlorophenyl-phenylether	1000 U
88-73-7	Fluorene	1000 U
108-91-6	4-Nitroaniline	8000 U
84-82-1	4,6-Dinitro-3-Methylphenol	2000 U
88-30-6	N-Nitrosodiphenylamine(1)	800 J
101-85-3	4-Bromophenyl-phenylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
87-88-5	Pentachlorophenol	1000 U
85-91-8	Phenanthrene	1000 U
128-12-7	Anthracene	1000 U
84-74-2	Di-n-Butylphthalate	700 J
208-44-0	Fluoranthene	1000 U
129-00-9	Pyrene	1000 U
86-88-7	Butylbenzylphthalate	1000 U
91-84-1	3,5-Dichlorobenzidine	2000 U
86-86-3	Benz(a)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthalate	1000 U
218-01-9	Chrysene	1000 U
117-84-0	Di-n-Octyl Phthalate	1000 U
208-98-2	Benz(b)Fluoranthene	1000 U
207-06-8	Benz(a)Fluoranthene	1200 U
80-32-3	Benz(e)Pyrene	1000 U
193-39-6	Indeno(1,2,3-cd)Pyrene	1000 U
83-70-2	Dibenz(a,h)Anthracene	1000 U
191-24-2	Benz(g,h,i)Perylene	1000 U

(1) - Cannot be separated from diphenylamine

CLF: 10/11/85

257

Prepared by: *RJY*

7/86

## Organics Analysis Data Sheet (Page 3)

### Pesticide/PCPs

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85, 9/3/85  
 Date Analyzed: 9/19/85  
 Conc/Dil Factor: 0.11G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	50 U
319-85-7	Beta-BHC	50 U
319-86-8	Delta-BHC	50 U
58-89-9	Gamma-BHC (Lindane)	50 U
75-44-8	Heptachlor	50 U
309-00-2	Aldrin	50 U
1024-87-3	Heptachlor Epoxide	50 U
859-98-8	Endosulfan I	100 U
80-87-1	Dieldrin	100 U
72-55-9	4,4'-DDE	100 U
72-20-8	Endrin	100 U
33213-65-9	Endosulfan II	100 U
72-54-8	4,4'-DDD	200 U
1031-07-8	Endosulfan Sulfate	200 U
50-29-3	4,4'-DDT	200 U
72-43-3	Methoxychlor	1000 U
53494-72-5	Endrin Ketone	NA
57-74-9	Chlordane	1000 U
8001-35-2	Toxaphene	10000 U
12674-11-2	Aroclor-10100	NA
11104-28-2	Aroclor-1221	NA
11141-100-5	Aroclor-1232	NA
53469-21-9	Aroclor-1242	1000 U
12672-29-6	Aroclor-1248	1000 U
11097-69-1	Aroclor-1254	1000 U
11096-82-5	Aroclor-1260	1000 U

$V_i$  = Volume of extract injected (ul)

$V_s$  = Volume of water extracted (ml)

$W_s$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

1070

$V_s = NR$

or  $W_s = 0.11$

$V_t = 5000$

$V_i = 5$



# Organics Analysis Data Sheet (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.  
 Lab Sample ID No: 21591-4  
 Sample Matrix: SOIL  
 Data Release Authorized By: FVA

Case No: 21591  
 QC Report No: NR  
 Contract No: NR  
 Date Sample Received: 9/29/85

## Volatile Compounds

Concentration: Medium  
 Date Extracted/Prepared: 9/16/85  
 Date Analyzed: 9/16/85  
 Conc/Dil Factor: 100 pH: NR  
 Percent Moisture: NR  
 Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-8	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-08-2	Methylene Chloride	800 U
67-64-1	Acetone	800 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethane	200 U
75-34-3	1,1-Dichloroethane	200 U
156-80-5	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-83-3	2-Butanone	800 U
71-55-6	1,1,1-Trichloroethane	200 U
56-23-5	Carbon Tetrachloride	200 U
108-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
78-87-6	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichloropropene	200 U
78-01-6	Trichloroethane	200 U
124-48-1	Dibromochloromethane	200 U
78-00-5	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	150 J
10061-01-5	cis-1,3-Dichloropropene	200 U
110-78-6	2-Chloroethylvinylether	1000 U
73-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	800 U
981-78-6	2-Hexanone	800 U
127-18-4	Tetrachloroethane	200 U
78-34-6	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-88-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	800 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

### Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value.
- U** Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution factors. (This is not necessarily the instrument detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- J** Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g. 10U) if limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3J
- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides  $\geq 10$ ng/ul in the final extract should be confirmed by GC/MS
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed.
- R** See cover letter.
- NR** Not Required.
- S** Spiked Compound.

CLF: 11/14/85

259  
Form I

Prepared by: FVA

10/85

4-22-80  
S. L. Con. Hall

# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: MEDIUM  
 Date Extracted/Prepared: 2/27/85 & 9/3/85  
 Date Analyzed: 4/11/86  
 Conc/Dil Factor: 0.5g/0.5ml

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	1000 U
111-44-4	bis(2-Chloroethyl)Ether	1000 U
20-67-8	2-Chlorophenol	1000 U
141-73-1	1,3-Dichlorobenzene	1000 U
108-48-7	1,4-Dichlorobenzene	1000 U
100-51-6	Benzyl Alcohol	1000 U
98-60-1	1,2-Dichlorobenzene	1000 U
65-48-7	2-Methylphenol	1000 U
3638-32-8	bis(2-chloroisopropyl)Ether	2000 U
108-44-8	4-Methylphenol	1000 U
621-64-7	N-Nitroso-Di-n-Propylamine	1000 U
67-72-1	Hexachlorocyclohexane	1000 U
92-95-3	Nitrobenzene	1000 U
72-59-1	Isophorone	1000 U
8-75-5	2-Nitrophenol	2000 U
103-57-9	2,4-Dimethylphenol	1000 U
5-25-0	Benzoic Acid	5000 U
111-91-1	bis(2-Chloroethoxy)Methane	2000 U
130-33-2	2,4-Dichlorophenol	1000 U
100-82-1	1,2,4-Trichlorobenzene	1000 U
91-20-3	Naphthalene	18000
102-47-8	4-Chloroaniline	1000 U
27-58-3	Hexachlorobutadiene	1000 U
85-62-7	4-Chloro-3-Methylphenol	1000 U
101-75-2	2-Methylnaphthalene	1000 U
27-37-4	Hexachlorocyclopentadiene	1000 U
88-06-2	2,4,6-Trichlorophenol	1000 U
95-95-4	2,4,5-Trichlorophenol	5000 U
21-33-7	2-Chloronaphthalene	1000 U
86-74-1	2-Nitroaniline	5000 U
131-11-3	Dimethyl Phthalate	1000 U
209-96-8	Acenaphthylene	36000
98-09-2	3-Nitroaniline	5000 U

CAS Number		ug/Kg
53-32-8	Acenaphthene	1800
51-28-8	2,4-Dinitrophenol	5000 U
100-02-7	4-Nitrophenol	5000 U
132-44-9	Dibenzofuran	720 J
121-14-2	2,4-Dinitrotoluene	1000 U
608-30-2	2,6-Dinitrotoluene	2000 U
84-86-2	Diethylphthalate	1000 U
7006-72-3	4-Chlorophenyl-phenylether	1000 U
86-73-7	Fluorene	1300
100-01-6	4-Nitroaniline	5000 U
534-62-1	4,5-Dinitro-2-Methylphenol	2000 U
86-30-6	N-Nitrosodiphenylamine (1)	140 J
101-65-3	4-Bromophenyl-phenylether	1000 U
118-74-1	Hexachlorobenzene	1000 U
87-88-6	Pentachlorophenol	1000 U
85-01-6	Phenanthrene	13000
120-12-7	Anthracene	2000
84-74-2	Di-n-Butylphthalate	1200
208-44-9	Fluoranthene	33000
128-00-9	Pyrene	47000
85-68-7	Butylbenzylphthalate	1000 U
91-84-1	3,3'-Dichlorobenzidine	2000 U
88-55-3	Benzo(a)Anthracene	1000 U
117-81-7	bis(2-Ethylhexyl)Phthalate	800 J
218-01-9	Chrysene	1000 U
117-84-0	Di-n-Octyl Phthalate	1000 U
206-89-2	Benzo(b)Fluoranthene	1700
207-08-9	Benzo(k)Fluoranthene	1700
50-32-8	Benzo(a)Pyrene	1000 U
193-39-6	Indeno(1,2,3-cd)Pyrene	1000 U
51-75-3	Dibenz(a,h)Anthracene	1000 U
191-24-2	Benzo(g,h,i)Perylene	1000 U

260

Cannot be separated from diphenylamine

CLF: 10/11/85

Form I

Prepared by: *[Signature]*

7/85

**Organics Analysis Data Sheet  
(Page 3)**

**Pesticide/PCBs**

Concentration: MEDIUM  
 Date Extracted/Prepared: 8/27/85, 9/3/85  
 Date Analyzed: 9/19/85  
 Conc/Dil Factor: 0.11G/5ML

GPC Cleanup: N<sup>o</sup>  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	80 U
319-86-7	Beta-BHC	80 U
319-86-8	Delta-BHC	80 U
58-89-9	Gamma-BHC (Lindane)	80 U
75-44-8	Heptachlor	80 U
308-00-2	Aldrin	80 U
1024-67-3	Heptachlor Epoxide	80 U
968-98-8	Endosulfan I	100 U
80-67-1	Dieldrin	100 U
72-55-5	4,4'-DDE	100 U
72-27-8	Endrin	100 U
33213-65-8	Endosulfan II	100 U
72-84-8	4,4'-DDD	200 U
1031-67-8	Endosulfan Sulfate	200 U
80-29-3	4,4'-DDT	200 U
72-43-5	Methoxychlor	1000 U
83404-70-8	Fluorfen Ketone	NA
57-74-9	Chlordane	1000 U
8001-35-2	Tepphone	10000 U
12874-11-2	Aroclor-10100	NA
11104-28-2	Aroclor-1221	NA
11141-100-6	Aroclor-1232	NA
83489-21-9	Aroclor-1242	1000 U
12672-29-6	Aroclor-1248	1000 U
11097-68-1	Aroclor-1254	1000 U
11096-82-5	Aroclor-1260	1000 U

$V_i$  = Volume of extract injected (ul)

$V_s$  = Volume of water extracted (ml)

$W_s$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

$V_s = NR$

or  $W_s = 0.11$

$V_t = 5000$

$V_i = 5$

1097

Organics Analysis Data Sheet  
(Page 1)

Laboratory Name: California Analytical Laboratories, Inc.  
 Lab Sample ID No: 21413-3  
 Sample Matrix: SOIL  
 Data Release Authorized By: PAS

Case No: 21413  
 QC Report No: NR  
 Contract No: NR  
 Date Sample Received: 8/21/85

Volatile Compounds

Concentration: Medium  
 Date Extracted/Prepared: 9/16/85  
 Date Analyzed: 9/16/85  
 Conc/Dil Factor: 100 pH: NR  
 Percent Moisture: NR  
 Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-09-2	Methylene Chloride	500 U
67-64-1	Acetone	500 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethene	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-5	Trans-1,2-Dichloroethene	200 U
75-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
75-93-3	2-Butanone	500 U
75-75-6	1,1,1-Trichloroethene	250
75-23-5	Carbon Tetrachloride	200 U
75-45-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
78-87-6	1,2-Dichloropropane	200 U
10061-02-8	Trans-1,3-Dichloropropene	200 U
78-01-6	Trichloroethene	200 U
124-48-1	Dibromochloromethane	200 U
78-00-5	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-8	cis-1,3-Dichloropropene	200 U
110-75-8	2-Chloroethylvinylether	1000 U
75-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	500 U
591-78-6	2-Hexanone	500 U
127-18-4	Tetrachloroethene	200 U
78-34-8	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-90-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	500 U
100-42-5	Styrene	200 U
	Total Xylenes	200 U

Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value.
- U** Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: "U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample."
- J** Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10U). If limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3J.
- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides  $\geq$  10ng/l in the final extract should be confirmed by GC/MS.
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed
- #** See cover letter
- NR** Not Required
- S** Spiked Compound

262

11/14/85

Form 1

Prepared by: [Signature]

10/85

570

## Organics Analysis Data Sheet (Page 2)

### Semivolatile Compounds

Concentration: LOW  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/10/85  
 Conc/Dil Factor: 29G/ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	200 U
111-44-4	bis(2-Chloroethyl)Ether	200 U
95-57-8	2-Chlorophenol	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlorobenzene	200 U
100-81-6	Benzyl Alcohol	200 U
95-80-1	1,2-Dichlorobenzene	200 U
95-48-7	2-Methylphenol	200 U
39638-32-8	bis(2-chloroisopropyl)Ether	400 U
108-44-6	4-Methylphenol	200 U
621-64-7	N-Nitroso-Di-n-Propylamine	200 U
67-72-1	Hexachloroethane	200 U
98-95-3	Nitrobenzene	200 U
78-59-1	Isophorone	200 U
88-75-5	2-Nitrophenol	400 U
105-67-8	2,4-Dimethylphenol	200 U
65-85-0	Benzoic Acid	1000 U
111-91-1	bis(2-Chloroethoxy)Methane	400 U
120-83-2	2,4-Dichlorophenol	200 U
120-82-1	1,2,4-Trichlorobenzene	200 U
91-20-3	Naphthalene	200 U
106-47-8	4-Chloroaniline	200 U
87-88-3	Hexachlorobutadiene	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
91-57-6	2-Methylnaphthalene	200 U
77-47-4	Hexachlorocyclopentadiene	200 U
88-06-2	2,4,6-Trichlorophenol	200 U
98-95-4	2,4,5-Trichlorophenol	5
91-88-7	2-Chloronaphthalene	200 U
88-74-4	2-Nitroaniline	1000 U
131-11-3	Dimethyl Phthalate	200 U
208-98-8	Acenaphthylene	200 U
99-09-2	3-Nitroaniline	1000 U

CAS Number		ug/Kg
83-32-8	Acenaphthene	200 U
51-28-6	2,4-Dinitrophenol	100 U
100-02-7	4-Nitrophenol	1000 U
132-64-9	Dibenzofuran	200 U
121-14-2	2,4-Dinitrotoluene	400 U
806-30-2	2,6-Dinitrotoluene	400 U
84-86-2	Dichlorophthalate	200 U
7005-72-3	4-Chlorophenyl-phenylether	200 U
86-73-7	Fluorene	200 U
100-01-6	4-Nitroaniline	1000 U
834-83-1	1,4-Dinitro-2-Methylphenol	400 U
88-30-6	N-Nitrosodiphenylamine(1)	200 U
101-85-3	4-Bromophenyl-phenylether	200 U
118-74-1	Hexachlorobenzene	200 U
87-85-6	Pentachlorophenol	200 U
85-01-8	Phenanthrene	200 U
120-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthalate	200 U
208-44-0	Fluoranthene	200 U
129-00-0	Pyrene	200 U
86-68-7	Butylbenzylphthalate	200 U
91-84-1	3,3'-Dichlorobenzidine	400 U
56-55-3	Benzo(a)Anthracene	200 U
117-81-7	bis(2-Ethylhexyl)Phthalate	200 U
218-01-8	Chrysene	400 U
117-84-0	Di-n-Octyl Phthalate	200 U
208-88-2	Benzo(b)Fluoranthene	400 U
207-08-9	Benzo(k)Fluoranthene	400 U
80-32-8	Benzo(a)Pyrene	400 U
193-39-5	Indeno(1,2,3-cd)Pyrene	400 U
53-70-3	Dibenz(a,h)Anthracene	400 U
191-24-2	Benzo(g,h,i)Perylene	400 U

(1) - Cannot be separated from diphenylamine

653

CLF: 10/11/85

Form I

Prepared by: *[Signature]*

7/85

Organics Analysis Data Sheet  
(Page 3)

Pesticide/PCBs

Concentration: LOW  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/18/85  
 Conc/Dil Factor: 1.54G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	3.0 U
319-85-7	Beta-BHC	3.0 U
319-86-8	Delta-BHC	3.0 U
58-89-9	Gamma-BHC (Lindane)	3.0 U
75-44-8	Heptachlor	3.0 U
309-00-2	Aldrin	3.0 U
1024-87-3	Heptachlor Epoxide	3.0 U
558-98-8	Endosulfan I	7.0 U
60-57-1	Dieldrin	7.0 U
72-55-9	4,4'-DDE	7.0 U
72-20-8	Endrin	7.0 U
33213-65-9	Endosulfan II	7.0 U
72-54-8	4,4'-DDD	13 U
1031-07-8	Endosulfan Sulfate	13 U
50-29-3	4,4'-DDT	13 U
72-43-5	Methoxychlor	67 U
53484-70-5	Endrin Ketone	NA
57-74-9	Chlordane	67 U
8001-35-2	Toxaphene	670 U
12674-11-2	Aroclor-1016	NA
11104-28-2	Aroclor-1221	NA
11141-16-5	Aroclor-1232	NA
53469-21-9	Aroclor-1242	67 U
12672-29-6	Aroclor-1248	67 U
11097-69-1	Aroclor-1254	67 U
11096-82-5	Aroclor-1260	67 U

$V_i$  = Volume of extract injected (ul)

$V_s$  = Volume of water extracted (ml)

$W_s$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

$V_s = NR$

or  $W_s = 1.54$

$V_t = 5000$

$V_i = 5$

654

CLF 11/14/85

264  
 Furn I Prepared by: *[Signature]*

7/85

# Organics Analysis Data Sheet (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.

Case No: 21581

Lab Sample ID No: 21581-5

QC Report No: NR

Sample Matrix: SOIL

Contract No: NR

Data Release Authorized By: PJS

Date Sample Received: 8/28/85

## Volatile Compounds

Concentration: Medium

Date Extracted/Prepared: 9/16/85

Date Analyzed: 9/16/85

Conc/Dil Factor: 100 pH: NR

Percent Moisture: NR

Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-08-2	Methylene Chloride	800 U
67-64-1	Acetone	800 U
75-13-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethane	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-4	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
78-93-3	2-Butanone	800 U
71-58-6	1,1,1-Trichloroethane	200 U
86-23-5	Carbon Tetrachloride	200 U
108-06-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/l
78-87-6	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichloropropene	200 U
78-01-6	Trichloroethane	200 U
124-46-1	Dibromochloromethane	200 U
78-09-6	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-6	cis-1,3-Dichloropropene	200 U
119-78-6	3-Chloroethylvinylether	1000 U
75-28-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	800 U
891-78-6	2-Hexanone	800 U
127-18-4	Tetrachloroethane	200 U
78-34-5	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-90-7	Chlorobenzene	200 U
108-41-4	Ethylbenzene	800 U
100-42-5	Styrene	200 U
	Total Xylenes	200 U

### Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value.
- U** Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- J** Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10U). If limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3U.

- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides  $\geq 10\text{ng/l}$  in the final extract should be confirmed by GC/MS.
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed.
- S** See cover letter.
- NR** Not Required.
- S** Spiked Compound.

1133

265

CLF: 11/14/85

Form I

Prepared by: PJS

10/85

Organics Analysis Data Sheet  
(Page 2)

Semivolatile Compounds

Concentration: Low  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/12/85  
 Conc/DIL Factor: 29G/ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-95-2	Phenol	200 U
111-44-4	bis(2-Chloroethyl)Ether	200 U
95-57-8	2-Chlorophenol	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlorobenzene	200 U
100-51-8	Benzyl Alcohol	200 U
95-50-1	1,2-Dichlorobenzene	200 U
35-48-7	2-Methylphenol	200 U
39638-32-0	bis(2-chloroisopropyl)Ether	400 U
106-44-5	4-Methylphenol	200 U
621-64-7	N-Nitroso-Di-n-Propylamine	200 U
67-72-1	Hexachloroethane	200 U
98-95-3	Nitrobenzene	200 U
78-59-1	Isophorone	200 U
88-75-5	2-Nitrophenol	400 U
105-67-2	2,4-Dimethylphenol	200 U
35-43-0	Benzoic Acid	1000 U
111-91-1	bis(2-Chloroethoxy)Methane	400 U
120-83-2	2,4-Dichlorophenol	200 U
120-82-1	1,2,4-Trichlorobenzene	200 U
91-20-3	Naphthalene	200 U
105-47-8	4-Chloroaniline	200 U
87-68-3	Hexachlorobutadiene	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
127-51-5	2-Methylnaphthalene	200 U
127-47-4	Hexachlorocyclopentadiene	200 U
38-06-2	2,4,5-Trichlorophenol	200 U
85-95-4	2,4,5-Trichlorophenol	200 U
91-58-7	2-Chloronaphthalene	200 U
62-74-4	2-Nitroaniline	1000 U
111-11-3	Dimethyl Phthalate	200 U
208-96-8	Acenaphthylene	200 U
39-09-2	3-Nitroaniline	1000 U

CAS Number		ug/Kg
83-32-9	Acenaphthene	200 U
51-28-5	2,4-Dinitrophenol	1000 U
100-02-7	4-Nitrophenol	1000 U
132-64-9	Dibenzofuran	200 U
121-14-2	2,4-Dinitrotoluene	400 U
606-20-2	2,6-Dinitrotoluene	400 U
84-66-2	Diethylphthalate	200 U
7005-72-3	4-Chlorophenyl-phenylether	200 U
86-73-7	Fluorene	200 U
100-01-6	4-Nitroaniline	1000 U
534-52-1	4,6-Dinitro-2-Methylphenol	400 U
86-30-6	N-Nitrosodiphenylamine(1)	200 U
101-55-3	4-Bromophenyl-phenylether	200 U
118-74-1	Hexachlorobenzene	200 U
87-86-5	Pentachlorophenol	200 U
85-01-8	Phenanthrene	200 U
120-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthalate	200 U
206-44-0	Fluorenone	200 U
129-00-0	Pyrene	200 U
85-68-7	Butylbenzylphthalate	200 U
91-94-1	3,3'-Dichlorobenzidine	400 U
56-55-3	Benzo(a)Anthracene	200 U
117-81-7	bis(2-Ethylhexyl)Phthalate	200 U
218-01-9	Chrysene	400 U
117-84-0	Di-n-Octyl Phthalate	200 U
205-99-2	Benzo(b)Fluoranthene	400 U
207-08-9	Benzo(k)Fluoranthene	400 U
50-32-8	Benzo(a)Pyrene	400 U
193-39-5	Indeno(1,2,3-cd)Pyrene	400 U
53-70-3	Dibenz(a,h)Anthracene	400 U
191-24-2	Benzo(g,h,i)Perylene	1000 U

(1) - Cannot be separated from diphenylamine



## Organics Analysis Data Sheet (Page 3)

### Pesticide/PCBs

Concentration: LOW  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/19/85  
 Conc/Dil Factor: 1.5G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	3.0 U
319-86-7	Beta-BHC	3.0 U
319-88-8	Delta-BHC	3.0 U
58-89-9	Gamma-BHC (Lindane)	3.0 U
75-44-8	Heptachlor	3.0 U
308-00-2	Aldrin	3.0 U
1024-67-3	Heptachlor Epoxide	3.0 U
888-88-8	Endosulfan I	7.0 U
88-87-1	Dieldrin	7.0 U
72-86-8	4,4'-DDE	7.0 U
72-30-8	Endrin	7.0 U
33213-88-8	Endosulfan II	7.0 U
72-84-8	4,4'-DDD	13.0 U
1031-07-8	Endosulfan Sulfate	13.0 U
80-29-3	4,4'-DDT	13.0 U
72-43-8	Methoxychlor	67.0 U
83494-70-8	Endrin Ketone	NA
87-74-8	Chlordane	67.0 U
8001-36-2	Toxaphene	670 U
12874-11-2	Aroclor-1018	NA
11104-28-2	Aroclor-1221	NA
11141-16-6	Aroclor-1232	NA
83469-21-8	Aroclor-1242	67.0 U
12872-28-6	Aroclor-1248	67.0 U
11087-68-1	Aroclor-1254	67.0 U
11096-82-6	Aroclor-1260	67.0 U

$V_i$  = Volume of extract injected (ul)

$V_s$  = Volume of water extracted (ml)

$W_s$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

$V_s = NR$

or  $W_s = 1.5$

$V_t = 5000$

$V_i = 5$     1135

# Organics Analysis Data Sheet (Page 1)

Laboratory Name: California Analytical Laboratories, Inc.  
 Lab Sample ID No: 21591-6  
 Sample Matrix: SOIL  
 Data Release Authorized By: PLS

Case No: 21591  
 QC Report No: NR  
 Contract No: NR  
 Date Sample Received: 8/29/85

## Volatile Compounds

Concentration: Medium  
 Date Extracted/Prepared: 9/16/85  
 Date Analyzed: 9/16/85  
 Conc/Dil Factor: 100 pH: NR  
 Percent Moisture: NR  
 Percent Moisture (Decanted): NR

CAS Number		ug/Kg
74-87-3	Chloromethane	200 U
74-83-9	Bromomethane	200 U
75-01-4	Vinyl Chloride	200 U
75-00-3	Chloroethane	200 U
75-08-2	Methylene Chloride	500 U
67-64-1	Acetone	500 U
75-15-0	Carbon Disulfide	200 U
75-35-4	1,1-Dichloroethane	200 U
75-34-3	1,1-Dichloroethane	200 U
156-60-5	Trans-1,2-Dichloroethane	200 U
67-66-3	Chloroform	200 U
107-06-2	1,2-Dichloroethane	200 U
75-93-3	2-Butanone	500 U
71-55-6	1,1,1-Trichloroethane	200 U
75-21-5	Carbon Tetrachloride	200 U
100-05-4	Vinyl Acetate	1000 U
75-27-4	Bromodichloromethane	200 U

CAS Number		ug/Kg
75-87-6	1,2-Dichloropropane	200 U
10061-02-6	Trans-1,3-Dichloropropene	200 U
79-01-6	Trichloroethane	200 U
124-48-1	Dibromochloromethane	200 U
79-06-8	1,1,2-Trichloroethane	200 U
71-43-2	Benzene	200 U
10061-01-5	cis-1,3-Dichloropropene	200 U
110-75-8	2-Chloroethylvinylether	1000 U
75-25-2	Bromoform	200 U
108-10-1	4-Methyl-2-Pentanone	500 U
591-78-6	2-Hexanone	500 U
127-18-4	Tetrachloroethane	200 U
75-34-5	1,1,2,2-Tetrachloroethane	200 U
108-88-3	Toluene	200 U
108-90-7	Chlorobenzene	200 U
100-41-4	Ethylbenzene	500 U
100-42-6	Styrene	200 U
	Total Xylenes	200 U

### Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

- Value** If the result is a value greater than or equal to the detection limit, report the value
- U** Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g. 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U - Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- J** Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g. 10U). If limit of detection is 10ug/l and a concentration of 3ug/l is calculated, report as 3U.
- C** This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides >= 10ng/ul in the final extract should be confirmed by GC/MS.
- B** This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other** Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.
- NA** Not Analyzed.
- #** See cover letter.
- NR** Not Required.
- S** Spiked Compound.

1167

SEP 11/14/85

Form I

Prepared by: AS

10/85

# Organics Analysis Data Sheet (Page 2)

## Semivolatile Compounds

Concentration: Low  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/12/85  
 Conc/Dil Factor: 29G/ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
108-85-2	Phenol	200 U
111-44-4	bis(2-Chloroethyl)Ether	200 U
85-57-8	2-Chlorophenol	200 U
541-73-1	1,3-Dichlorobenzene	200 U
106-46-7	1,4-Dichlorobenzene	200 U
100-51-6	Benzyl Alcohol	200 U
95-50-1	1,2-Dichlorobenzene	200 U
95-48-7	2-Methylphenol	200 U
39838-32-9	bis(2-chloroisopropyl)Ether	400 U
108-44-8	4-Methylphenol	200 U
621-64-7	N-Nitroso-Di-n-Propylamine	200 U
67-72-1	Hexachlorocyclohexane	200 U
88-85-3	Nitrobenzene	200 U
78-59-1	Isophorone	200 U
88-75-5	2-Nitrophenol	400 U
105-67-9	2,4-Dimethylphenol	200 U
65-85-0	Benzole Acid	1000 U
111-91-1	bis(2-Chloroethoxy)Methane	400 U
120-83-2	2,4-Dichlorophenol	200 U
120-82-1	1,2,4-Trichlorobenzene	200 U
91-20-3	Naphthalene	200 U
106-47-6	4-Chloroaniline	200 U
87-68-3	Hexachlorobutadiene	200 U
59-50-7	4-Chloro-3-Methylphenol	200 U
91-57-6	2-Methylnaphthalene	200 U
77-47-4	Hexachlorocyclopentadiene	200 U
88-08-2	2,4,6-Trichlorophenol	200 U s
95-95-4	2,4,5-Trichlorophenol	s
91-58-7	2-Chloronaphthalene	200 U
88-74-4	2-Nitroaniline	1000 U
131-11-3	Dimethyl Phthalate	200 U
208-96-8	Acenaphthylene	200 U
99-09-2	3-Nitroaniline	1000 U

CAS Number		ug/Kg
83-32-9	Acenaphthene	200 U
81-29-8	2,4-Dinitrophenol	1000 U
109-02-7	4-Nitrophenol	1000 U
132-84-9	Dibenzofuran	200 U
121-14-2	2,4-Dinitrotoluene	400 U
606-20-2	2,6-Dinitrotoluene	400 U
84-86-2	Diethylphthalate	200 U
7005-72-3	4-Chlorophenyl-phenylether	200 U
86-73-7	Fluorene	200 U
100-91-6	4-Nitroaniline	1000 U
834-82-1	4,6-Dinitro-2-Methylphenol	400 U
86-30-6	N-Nitrosodiphenylamine(1)	200 U
101-68-3	4-Bromophenyl-phenylether	200 U
118-74-1	Hexachlorobenzene	200 U
87-88-6	Pentachlorophenol	200 U
85-01-8	Phenanthrene	200 U
129-12-7	Anthracene	200 U
84-74-2	Di-n-Butylphthalate	200 U
206-44-0	Fluoranthene	200 U
129-00-0	Pyrene	200 U
86-62-7	Butylbenzylphthalate	200 U
91-84-1	3,3'-Dichlorobenzidine	400 U
86-66-3	Benzo(a)Anthracene	200 U
117-81-7	bis(2-Ethylhexyl)Phthalate	200 U
218-01-9	Chrysene	400 U
117-84-0	Di-n-Octyl Phthalate	200 U
206-99-2	Benzo(b)Fluoranthene	400 U
207-08-9	Benzo(k)Fluoranthene	400 U
50-32-8	Benzo(a)Pyrene	400 U
193-39-5	Indeno(1,2,3-cd)Pyrene	400 U
53-70-3	Dibenz(a,h)Anthracene	400 U
191-24-2	Benzo(g,h,i)Perylene	400 U

(1) - Cannot be separated from diphenylamine

1168

# Organics Analysis Data Sheet (Page 3)

## Pesticide/PCBs

Concentration: LOW  
 Date Extracted/Prepared: 8/27/85  
 Date Analyzed: 9/19/85  
 Conc/Dil Factor: 1.5G/5ML

GPC Cleanup: NO  
 Separatory Funnel Extraction: YES  
 Continuous Liquid - Liquid Extraction: NO

CAS Number		ug/Kg
319-84-6	Alpha-BHC	3.0 U
319-85-7	Beta-BHC	3.0 U
319-86-8	Delta-BHC	3.0 U
58-89-9	Gamma-BHC (Lindane)	3.0 U
76-44-8	Heptachlor	3.0 U
309-00-2	Aldrin	3.0 U
1024-57-3	Heptachlor Epoxide	3.0 U
959-98-8	Endosulfan I	7.0 U
60-57-1	Dieldrin	7.0 U
72-56-9	4,4'-DOE	7.0 U
72-20-8	Endrin	7.0 U
33213-65-9	Endosulfan II	7.0 U
72-84-8	4,4'-DDD	13.0 U
1031-07-8	Endosulfan Sulfate	13.0 U
50-29-3	4,4'-DDT	13.0 U
72-43-5	Methoxychlor	67.0 U
53494-77-5	Endrin Ketone	NA
57-74-9	Chlordane	67.0 U
6001-35-2	Toxaphene	67.0 U
12674-11-2	Aroclor-1016	NA
11104-28-2	Aroclor-1221	NA
11141-16-5	Aroclor-1232	NA
53469-21-9	Aroclor-1242	67.0 U
12672-29-6	Aroclor-1248	67.0 U
11097-69-1	Aroclor-1254	67.0 U
11096-82-5	Aroclor-1260	67.0 U

$V_i$  = Volume of extract injected (ul)

$V_S$  = Volume of water extracted (ml)

$W_S$  = Weight of sample extracted (g)

$V_t$  = Volume of total extract (ul)

$V_S = NR$

or  $W_S = 1.5$

$V_t = 5000$

$V_i = 5$       1169

HU-NCBC-R1-01

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21591  
SOW NO.: 784 QC RPT. # 21591  
LAB SAMPLE NO.: 21591-1 DATE: 3-19-86

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: Soil UNITS: MG/KG DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<3U R
3. ARSENIC.....P	7.2
5. BERYLLIUM...P	<0.3U
6. CADMIUM.....P	<0.3U
8. CHROMIUM....P	2.3
10. COPPER.....P	3.3
12. LEAD.....P	29 R
15. MERCURY....CV	20.1U
16. NICKEL.....P	4.2
18. SELENIUM...P	<0.3U R
19. SILVER.....P	<0.3U R
21. THALLIUM....F	<0.3U R
24. ZINC.....P	108
25. CYANIDE.....C	<0.2U

COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

FOOTNOTES:

- NR - not required by contract at this time
- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- S - Indicates value determined by Method of Standard Addition.
- R - Indicates spike sample recovery is not within control limits.
- \* - Indicates duplicate analysis is not within control limits.
- + - Indicates the correlation coefficient for method of standard addition is less than 0.995.

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB.      CASE NO: 21591  
SOW NO.: 784      QC RPT.# 21591  
LAB SAMPLE NO.: 21591-2      DATE: 3-14-86

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: Soil      UNITS: MG/KG  
DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<3U R
3. ARSENIC.....P	5.5
5. BERYLLIUM...P	<0.3U
6. CADMIUM.....P	<0.3U
8. CHROMIUM....P	6.1
10. COPPER.....P	10.2
12. LEAD.....P	15 R
15. MERCURY....CV	<0.1U
16. NICKEL.....P	1.10
18. SELENIUM...P	<0.3U R
19. SILVER.....P	<0.5U R
21. THALLIUM....F	<0.5U R
24. ZINC.....P	7.0
25. CYANIDE.....C	<0.5U

COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

FOOTNOTES:

- NR - not required by contract at this time
- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- I - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- S - Indicates value determined by Method of Standard Addition.
- R - Indicates spike sample recovery is not within control limits.
- D - Indicates duplicate analysis is not within control limits.
- - Indicates the correlation coefficient for method of standard addition is less than 0.995.

FORM V

SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

HU-NCBC-R2-01

DATE: 3-19-86  
 MATRIX: SOIL

UNITS: ppb

COMPOUNDS METALS:	ELEMENTS..METHOD	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	% R
	2. ANTIMONY....P	75 TO 125	53.3	24.4	200	14
	3. ARSENIC....P	75 TO 125	408	104	300	100
	5. BERYLLIUM...P	75 TO 125	92.5	<20	100	03
	6. CADMIUM....P	75 TO 125	84.6	<20	100	91
	8. CHROMIUM....P	75 TO 125	451	122	400	82
	10. COPPER.....P	75 TO 125	358	123	250	44
	12. LEAD.....P	75 TO 125	448	300	100	128
	15. MERCURY....CV	75 TO 125	NIR	NIR	-	-
	16. NICKEL.....P	75 TO 125	154	31.7	150	82
	18. SELENIUM....P	75 TO 125	7.1	<20	50	14
	19. SILVER.....P	75 TO 125	101	<20	100	61
	21. THALLIUM....F	75 TO 125	22.8	<20	50	26
	24. ZINC.....P	75 TO 125	5760	1340	5000	87
	25. CYANIDE.....C	75 TO 125	NIR	NIR	-	-

COMMENTS:

DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

HL-NEBC-R2-01

DATE: 3-19-86  
MATRIX: SOIL

UNITS: ppb

COMPOUNDS METALS: CONTROL SAMPLE(S) LIMIT DUPLICATES (D) RPD

ELEMENTS..METHOD	CONTROL SAMPLE(S) LIMIT	DUPLICATES (D)	RPD
2. ANTIMONY....P	244	28.5	16
3. ARSENIC.....P	124	102	10.0
5. BERYLLIUM...P	<20	<20	0
6. CADMIUM.....P	<20	<20	0
8. CHROMIUM....P	<20	<20	0
10. COPPER.....P	122	124	1.16
12. LEAD.....P	123	122	1.19
15. MERCURY....CV	281	283	5.9
16. NICKEL.....P	NIR	NIR	-
18. SELENIUM...P	31.7	35	0.6
19. SILVER.....P	<20	<20	0
21. THALLIUM...F	<20	<20	0
24. ZINC.....P	<20	<20	2
25. CYANIDE.....C	1340	1340	1
	NIR	NIR	-

COMMENTS:



HU-NCBC-R1-02

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21413  
SOW NO.: 784 QC RPT. # 21413  
LAB SAMPLE NO.: 21413-2 DATE: 2-19-76

## ELEMENTS IDENTIFIED AND MEASURED

MATRIX: SOIL UNITS: MG/KG  
DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<u>&lt;3.0</u> R
3. ARSENIC....P	<u>4.7</u>
5. BERYLLIUM....P	<u>10.427</u>
6. CADMIUM....P	<u>&lt;0.30</u>
8. CHROMIUM....P	<u>4.2</u>
10. COPPER.....P	<u>3.0</u>
12. LEAD.....P	<u>4.2</u>
15. MERCURY....CV	<u>&lt;0.10</u>
16. NICKEL.....P	<u>3.4</u> R
18. SELENIUM....P	<u>&lt;0.30</u> R
19. SILVER.....P	<u>50.617</u> R
21. THALLIUM....F	<u>&lt;0.50</u> R
24. ZINC.....P	<u>81</u>
25. CYANIDE.....C	<u>&lt;0.50</u>

## COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

## FOOTNOTES:

- NR - not required by contract at this time  
Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).  
U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).  
E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.  
S - Indicates value determined by Method of Standard Addition.  
R - Indicates spike sample recovery is not within control limits.  
\* - Indicates duplicate analysis is not within control limits.  
+ - Indicates the correlation coefficient for method of standard addition is less than 0.995.

1990

SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

HU-NCBC-R1-02

DATE: 3-19-86  
 MATRIX: SOIL

UNITS: ppb

COMPOUNDS METALS:	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	% R
ELEMENTS..METHOD					
2. ANTIMONY....P	75 TO 125	391	29	200	5.1
3. ARSENIC.....P	75 TO 125	51.4	173	350	110
5. BERYLLIUM...P	75 TO 125	22.6	<20	150	29
6. CADMIUM.....P	75 TO 125	40.7	<20	100	91
8. CHROMIUM....P	75 TO 125	511	184	400	82
10. COPPER.....P	75 TO 125	282	59.0	250	89
12. LEAD.....P	75 TO 125	1028	183	500	89
15. MERCURY....CV	75 TO 125	NIR	NIR	-	-
16. NICKEL.....P	75 TO 125	269	78.2	250	74
18. SELENIUM...P	75 TO 125	17.6	<20	50	35
19. SILVER.....P	75 TO 125	105.8	<20	100	106
21. THALLIUM...F	75 TO 125	30.7	<20	50	101
24. ZINC.....P	75 TO 125	5270	1020	5000	95
25. CYANIDE.....C	75 TO 125	NIR	NIR	-	-

COMMENTS:

1993

FM VI

DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

HU-NCBC-R1-02

DATE: 3-19-86  
 MATRIX: SOIL

UNITS: ppb

COMPOUNDS METALS:	ELEMENTS..METHOD	CONTROL SAMPLE(S) LIMIT	DUPLICATES (D)	RPD
	2. ANTIMONY....P	29	21.1	32
	3. ARSENIC.....P	173	175	1.7
	5. BERYLLIUM...P	<20	<20	0
	6. CADMIUM.....P	<20	<20	0
	8. CHROMIUM....P	188	172	6.7
	10. COPPER.....P	54.0	66.7	12
	12. LEAD.....P	173	177	3.3
	15. MERCURY....CV	NIR	NIR	-
	16. NICKEL.....P	72.2	53.5	34
	18. SELENIUM....P	<20	<20	0
	19. SILVER.....P	<20	<20	0
	21. THALLIUM....P	<20	<20	0
	24. ZINC.....P	1620	1450	4.0
	25. CYANIDE.....C	NIR	NIR	-

COMMENTS: Antimony and nickel were not flagged with an "\*" for high RPD because the values were only at the detection limits.

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21591  
SOW NO.: 784 QC RPT. # 21591  
LAB SAMPLE NO.: 21591-3 DATE: 3-14-82

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: SOIL UNITS: MG/KG  
DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<30 R
3. ARSENIC.....P	102
5. BERYLLIUM...P	0.247
6. CADMIUM.....P	<0.30
8. CHROMIUM....P	1.2
10. COPPER.....P	5.2
12. LEAD.....P	7.6
15. MERCURY....CV	<0.10
16. NICKEL.....P	2.0
18. SELENIUM...P	<0.2 U R
19. SILVER.....P	10.5 U R
21. THALLIUM...F	<0.5 U R
24. ZINC.....P	102
25. CYANIDE....C	<0.5 U

COMMENTS:

Interelement and background corrections applied? Yes.

Corrections consist of Zeeman effect background correction for Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections applied before generation of raw data.

NOTES:

- not required by contract at this time
- If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]).
- Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- Indicates value determined by Method of Standard Addition.
- Indicates spike sample recovery is not within control limits.
- Indicates duplicate analysis is not within control limits.
- Indicates the correlation coefficient for method of standard addition is less than 0.995.

FORM V

SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

HU-NBC-R2-02

DATE: 3-19-86  
 MATRIX: SCIL

UNITS: ppb

COMPOUNDS METALS:	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	
ELEMENTS...METHOD					
2. ANTIMONY....P	75 TO 125	57.7	460	200	26
3. ARSENIC....P	75 TO 125	243	127	360	10
5. BERYLLIUM...P	75 TO 125	74.8	230	100	91
6. CADMIUM....P	75 TO 125	23.4	220	100	6
8. CHROMIUM....P	75 TO 125	468	163	400	71
10. COPPER.....P	75 TO 125	320	116	250	8
12. LEAD.....P	75 TO 125	270	151	500	8
15. MERCURY....CV	75 TO 125	NIR	NIR	-	-
16. NICKEL.....P	75 TO 125	247	59.1	250	70
18. SELENIUM....P	75 TO 125	10	230	50	10
19. SILVER.....P	75 TO 125	12.5	230	100	7
21. THALLIUM....F	75 TO 125	210.3	230	50	2
24. ZINC.....P	75 TO 125	320	1240	5000	8
25. CYANIDE.....C	75 TO 125	NIR	NIR	-	-

COMMENTS:

DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

DATE: 3-19-86  
 MATRIX: SOIL

HU-N:BC-R2-02

UNITS: ppb

COMPOUNDS METALS: CONTROL SAMPLE(S) LIMIT DUPLICATES (D) RPD

ELEMENTS..METHOD	CONTROL SAMPLE(S) LIMIT	DUPLICATES (D)	RPD
2. ANTIMONY....P	32.9	<20	0
3. ARSENIC....P	127	21	4.1
5. BERYLLIUM...P	<20	<20	0
6. CADMIUM....P	<20	<20	0
8. CHROMIUM....P	16.3	16.2	0.99
10. COPPER.....P	116	129	1.12
12. LEAD.....P	51	151	0
15. MERCURY....CV	NIR	NIR	-
16. NICKEL.....P	241	57.4	2.1
18. SELENIUM...P	<20	<20	0
19. SILVER.....P	<20	<20	0
21. THALLIUM...F	<20	<20	0
24. ZINC.....P	<20	<20	0
25. CYANIDE.....C	12.0	12.0	3.5
	NIR	NIR	-

COMMENTS:

HU-NCBC-R2-03

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21591  
SOW NO.: 784 QC RPT. # 21591  
LAB SAMPLE NO.: 21591-4 DATE: 3-14-86

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: Soil (DUST) UNITS: MG/KG DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	< 3.0 R
3. ARSENIC.....P	1.3
5. BERYLLIUM...P	< 1.0 R
6. CADMIUM.....P	3.8
8. CHROMIUM....P	21
10. COPPER.....P	29
12. LEAD.....P	212
15. MERCURY....CV	0.4
16. NICKEL.....P	4.9
18. SELENIUM....P	< 1.0 R
19. SILVER.....P	2.9 R
21. THALLIUM...P	< 0.5 U R
24. ZINC.....P	16.0
25. CYANIDE....C	< 0.5 U

COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

FOOTNOTES:

- NR - not required by contract at this time
- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- S - Indicates value determined by Method of Standard Addition.
- R - Indicates spike sample recovery is not within control limits.
- \* - Indicates duplicate analysis is not within control limits.
- + - Indicates the correlation coefficient for method of standard addition is less than 0.995.

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21413  
SOW NO.: 784 QC RPT. # 21413  
LAB SAMPLE NO.: 21413-3 DATE: 3-14-76

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: CHARCOAL UNITS: MG/KG DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<3U R
3. ARSENIC.....P	<0.5U
5. BERYLLIUM...P	<0.2U
6. CADMIUM.....P	<0.2U
8. CHROMIUM....P	2.4
10. COPPER.....P	<1.2U
12. LEAD.....P	1.5
15. MERCURY....CV	<0.1U
16. NICKEL.....P	2.2
18. SELENIUM...P	<0.3U R
19. SILVER.....P	<0.5U R
21. THALLIUM...F	<0.5U R
24. ZINC.....P	14 *
25. CYANIDE.....C	<0.5U

COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections applied before generation of raw data.

FOOTNOTES:

- 1 - not required by contract at this time
- 2 - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- 3 - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- 4 - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- 5 - Indicates value determined by Method of Standard Addition.
- 6 - Indicates spike sample recovery is not within control limits.
- 7 - Indicates duplicate analysis is not within control limits.
- 8 - Indicates the correlation coefficient for method of standard addition is less than 0.995.



FORM V

SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

HU-NCBC-R1-09

DATE: 3-19-86  
 MATRIX: UNIDENTIFIED

UNITS: ppb

COMPOUNDS METALS:	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	% R
ELEMENTS...METHOD					
2. ANTIMONY....P	75 TO 125	60	<20	200	30
3. ARSENIC.....P	75 TO 125	40.3	<20	50	81
5. BERYLLIUM...P	75 TO 125	48.4	<20	150	68
6. CADMIUM.....P	75 TO 125	91.8	<20	150	93
8. CHROMIUM....P	75 TO 125	218	37.1	200	95
10. COPPER.....P	75 TO 125	43.2	<20	100	93
12. LEAD.....P	75 TO 125	1070	165	1000	91
15. MERCURY....CV	75 TO 125	NIR	NIR	-	-
16. NICKEL. ....P	75 TO 125	125	43.4	150	44
18. SELENIUM....P	75 TO 125	15.2	<20	50	30
19. SILVER.....P	75 TO 125	55.4	<20	100	35
21. THALLIUM....F	75 TO 125	24.8	<20	50	50
24. ZINC.....P	75 TO 125	746	379	500	83
25. CYANIDE.....C	75 TO 125	N.R	NIR	-	-

COMMENTS:

1988

DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

HU-NCBC-R1-09

DATE: 3-10-86  
 MATRIX: HFRSOP

UNITS: ppb

COMPOUNDS  
 METALS:

CONTROL SAMPLE(S) LIMIT  
 DUPLICATES(D)

RPD

ELEMENTS..METHOD	CONTROL SAMPLE(S) LIMIT	DUPLICATES(D)	RPD
ANTIMONY....P	<20	<20	0
ARSENIC....P	<20	<20	0
BERYLLIUM...P	<20	<20	0
5. CADMIUM....P	<20	<20	0
7. CHROMIUM....P	<20	<20	0
10. COPPER.....P	57.1	66.4	3.6
12. LEAD.....P	<20	<20	0
15. MERCURY....CV	100	100	3.6
16. NICKEL.....P	NIR	NIR	-
18. SELENIUM....P	43.4	45	3.6
19. SILVER.....P	<20	<20	0
21. THALLIUM....F	<20	<20	0
22. TIN.....P	<20	<20	0
23. CYANIDE....C	279	267	35 *
	NIR	NIR	

NONMETALS:

MODIFIED PRIORITY POLLUTANT LIST  
 (EG & G Subcontract No. C85-130761-KAM-177-85)  
 INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21591  
 SOW NO.: 78: QC RPT.# 21591  
 LAB SAMPLE NO.: 21591-5 DATE: 5-14-86

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: CHARCOAL UNITS: MG/KG DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<30 R
3. ARSENIC.....P	1.01
5. BERYLLIUM...P	<0.30 U
6. CADMIUM.....P	<0.30 U
8. CHROMIUM....P	4.9
10. COPPER.....P	1.2
12. LEAD.....P	3.3
15. MERCURY....CV	<0.10 U
16. NICKEL.....P	2.9
18. SELENIUM...P	<0.30 R
19. SILVER.....P	<0.30 R
21. THALLIUM...F	<0.30 R
24. ZINC.....P	16
25. CYANIDE.....C	<0.50 U

COMMENTS:

ICP Interelement and background corrections applied? Yes.

AA corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 875 instruments. Corrections are applied before generation of raw data.

FOOTNOTES:

- NR - not required by contract at this time
- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]). Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <10U).
- E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- S - Indicates value determined by Method of Standard Addition.
- R - Indicates spike sample recovery is not within control limits.
- \* - Indicates duplicate analysis is not within control limits.
- + - Indicates the correlation coefficient for method of standard addition is less than 0.995.

SPIKE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID

HU-NCBC-R2-09

DATE: 3-19-86  
 MATRIX: ILWAP/CAL

UNITS: ppb

COMPOUNDS (METALS):	CONTROL LIMIT % R	SPIKED SAMPLE RESULT (SSR)	SAMPLE RESULT (SR)	SPIKED ADDED (SA)	% R
ELEMENTS..METHOD					
1. ANTIMONY.....P	75 TO 125	22.2	22.7	200	OR
2. ARSENIC.....P	75 TO 125	45.4	20.8	20	82
3. BERYLLIUM...P	75 TO 125	47.4	<20	100	97
4. CADMIUM.....P	75 TO 125	40.2	<20	100	97
5. CHROMIUM....P	75 TO 125	42.6	49.0	400	97
11. COPPER.....P	75 TO 125	110.6	24.2	100	82
12. LEAD.....P	75 TO 125	100X	106	500	82
13. MERCURY....CV	75 TO 125	NIR	NIR	—	88
14. NICKEL.....P	75 TO 125	220	52.1	250	—
16. SELENIUM....P	75 TO 125	16.2	<20	50	90
17. SILVER.....P	75 TO 125	51.5	<20	100	34R
18. THALLIUM....F	75 TO 125	27.4	<20	50	52R
19. ZINC.....P	75 TO 125	222	313	500	57.8
20. CYANIDE.....C	75 TO 125	NIR	NIR	—	102

ELEMENTS:

2013

DUPLICATE SAMPLE RECOVERY

LAB NAME: CALIF. ANAL. LABS.

EG & G ID NO.:

HU-NCBC-R2-09

DATE: 3-19-86  
 MATRIX: UFORLADAL

UNITS: ppb

COMPOUNDS METALS: CONTROL SAMPLE(S) LIMIT DUPLICATES (D) RPD

ELEMENTS..METHOD	CONTROL SAMPLE(S) LIMIT	DUPLICATES (D)	RPD
2. ANTIMONY....P	22.7	<20	0
3. ARSENIC.....F	20.8	22.6	2.3
5. BERYLLIUM...P	<20	<20	0
6. CADMIUM.....P	<20	<20	0
8. CHROMIUM....P	47.0	101	4.5
10. COPPER.....P	24.2	25.2	4.0
12. LEAD.....P	116.6	109	1.8
15. MERCURY....CV	NIR	NIR	-
16. NICKEL.....P	58.1	70.5	1.9
18. SELENIUM....P	<20	<20	0
19. SILVER.....P	<20	<20	0
21. THALLIUM....F	<20	<20	0
24. ZINC.....P	313	329	5.0
25. CYANIDE....C	NIR	NIR	-

COMMENTS:

MODIFIED PRIORITY POLLUTANT LIST  
(EG & G Subcontract No. C85-130761-KAM-177-85)  
INORGANIC ANALYSIS SHEET

LAB NAME: CALIF. ANAL. LAB. CASE NO: 21591  
SOW NO.: 784 QC RPT. # 21591  
LAB SAMPLE NO.: 21591-6 DATE: 2-10-76

ELEMENTS IDENTIFIED AND MEASURED

MATRIX: CLAY UNITS: MG/KG  
DRY WEIGHT

ELEMENTS..METHOD	
2. ANTIMONY....P	<30 R
3. ARSENIC.....P	7.5
5. BERYLLIUM...P	<1.30
6. CADMIUM.....P	<1.30
8. CHROMIUM....P	3
10. COPPER.....P	5
12. LEAD.....P	10
15. MERCURY....CV	<100
16. NICKEL.....P	11.7
18. SELENIUM...P	<0.30 R
19. SILVER.....P	<0.50 R
21. THALLIUM...F	<0.50 R
24. ZINC.....P	14
25. CYANIDE.....C	<0.50

COMMENTS:

ICP Interelement and background corrections applied? Yes.

All corrections consist of Zeeman effect background correction on Perkin-Elmer 3030 instruments and correction of background absorption by D2 lamp on Varian 375 instruments. Corrections applied before generation of raw data.

NOTES:

- Not required by contract at this time
- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (ie. [10]).
- Indicate the analytical method used with P (for ICP/Flame AA), F (for furnace), or CV (for cold vapor).
- Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., <100).
- Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
- Indicates value determined by Method of Standard Addition.
- Indicates spike sample recovery is not within control limits.
- Indicates duplicate analysis is not within control limits.
- Indicates the correlation coefficient for method of standard addition is less than 0.995.

2009

2,4-D and 2,4,5-T by EPA Method 8150

CAL I.D.	MG/KG (ppm) 2,4-D	MG/KG (ppm) 2,4,5-T
21349-MB	<0.01	<0.01
-1	870	1400
-2	920	1300
-4	1100	1600
-5	990	1200
-6	1200	2400
-7	0.18	0.50
-7MS (0.05)	0.26 (160%)	0.73 (460%)
-7MSD (0.05)	0.27 (180%)	0.70 (400%)
-8	<0.01	0.014
-9	<0.01	0.01
21591-1MB	<0.01	<0.01
-1	380	770
-2	280	610
-3	<0.01	<0.01
-4	<0.01	<0.01
-5	<0.01	0.011
-6	<0.01	<0.01
21484-2MB	<0.01	0.06
-2	<0.01	<0.01
-9	0.17	0.54
-10	<0.01	<0.01
-11	<0.01	<0.01
-13	<20	<2.0
21413-1MB	<0.01	<0.01
-1	<1.0	<0.2
-2	<0.01	<0.01
-3	<0.01	<0.01
-5	0.05	0.17
-7	<0.01	<0.01
-8	<0.01	<0.01
-11	0.02	0.06

Phenoxy Acids  
Reanalyses

21349-MB	<0.005	<0.005
-MBS or MS 50 ppb	0.051	0.067
7RX	0.039	0.220
-7MS (50 ppb)	0.077 (76%)	0.260 (80%)
-7MSD (50 ppb)	0.0102 (130%)	0.420 (400%)

California Analytical Laboratories, Inc.

1786

## CREOSOTE

Creosote (the 'coal tar' variety) is a mixture of polynuclear aromatic hydrocarbons used as biocide/biostat in the preservation of wood. Most of the principal components of the mixture are priority pollutants. We have examined both commercial creosote and the chromatogram in the reference cited in K. McKay's letter of December 23, 1985 and compared these data to the raw mass chromatograms raw Quan Lists for the samples IT-NCBC-R1-01 and IT-NCBC-R2-01. There are a variety of polynuclear aromatic hydrocarbons present in the samples. Nevertheless, it is our conclusion that it is impossible to say that the creosote profile is present in the data. We base this conclusion on the low amounts of 2-methyl naphthalene and fluorene in one or both of the samples. Further, our own creosote data shows that phenanthrene dominates anthracene by a factor of greater than 6 to 1 in creosote. This does not occur in the cited samples. The data does not rule out the presence of creosote, but it is clear that the bulk of polynuclear aromatic hydrocarbons came from other hydrocarbon sources and further that the analysis for only "creosote" is not possible.



Summary of TCDD & TSP in Filter Samples

CAL Lab I.D.	Sample Number	Method	Blank	Y	C Aliquot U (sample)	ng/sample TCDD Mess	ng/sample Detection Limit	TSP (ug/m <sup>3</sup> )	Vol (m <sup>3</sup> )
21932-MB	1131	MBNS	Blank	Y	1.00	ND	0.14	--	--
-1	1132		Blank	Y	1.00	13.6	0.59	18.6	4845.99
-2	1133		Blank	Y	1.00	ND	0.27	44.9	4635.54
-3	1134		Blank	Y	1.00	1.5	--	36.4	4845.99
-4	1135		Blank	Y	1.00	ND	0.17	17.4	2877.06
-1	1136		Blank	Y	1.00	ND	0.14	26.9	4547.10
-2	1137		Blank	Y	1.00	ND	0.28	31.8	4537.07
-3	1138		Blank	Y	1.00	0.14	0.22	25.2	4547.10
-4	1139		Blank	Y	1.00	ND	--	24.4	4721.23
-5	1140		Blank	Y	1.00	0.11	0.13	41.9	1761.05
-6	1141		Blank	Y	1.00	1.1	--	38.5	1596.16
-7	1143		Blank	Y	1.00	ND	--	55.7	1761.05
-8			Blank	Y	1.00	ND	0.12	35.8	1828.50

Lab: California Analytical Laboratories  
 Case No. 21349  
 Batch/Shipment No.

LOW DATA PLANT  
 California Analytical Labs, Suite  
 2544 Industrial Blvd.,  
 W. Sacramento, CA 95591

Report Date: 10-22-85  
 Column: SP-2331

Cal Labs ID	Sample Number	Aliquot C Met Wt. U (grams)	PPB TCOD Meas	Inst ID	Date	Time	320/322	332/334 Meas	Surrq % Acc't	320	322	332	334	Cor
21349-1	11NCBC-R1-01	Y 10.00	ND	8	10/07/85	21:59:00	0.011	0.77	1.03	103	977032	1046270	1362800	
21349-2	11NCBC-R1-01	Y 0.51	260	8	10/07/85	22:31:00	0.79	0.77	20.37	104	727070	774825	1004100	
21349-3	11NCBC-R2-01	Y 0.60	272	8	10/07/85	22:53:00	0.79	0.79	16.29	98	799955	917779	1163060	
21349-4	11NCBC-R1-03	Y 50.00 ml	43.3	8	10/17/85	15:55:00	0.81	0.71	0.17	84	53560800	582468	823854	
21349-5	11NCBC-R3-01	Y 0.61	236	8	10/07/85	21:59:00	0.79	0.78	16.39	100	3592830	609775	778939	
21349-6	11NCBC-R4-01	Y 0.52	266	8	10/07/85	21:59:00	0.79	0.75	19.23	100	3709570	640468	855600	
21349-7	11NCBC-R5-01	Y 0.58	233	8	10/07/85	23:56:00	0.78	0.77	17.74	103	4821660	866648	1132170	
21349-8	11NCBC-R1-09	Y 10.19	ND	8	10/08/85	23:50:00	0.73	1.10	112		167708	163716	223665	
21349-9	11NCBC-R1-09A	Y 10.19	ND	8	10/08/85	23:50:00	0.73	1.10	112		167708	163716	223665	
21413-1	11NCBC-R1-04	Y 50.00 ml	0.36	8	10/17/85	15:35:00	0.79	0.77	0.17	86	794464	1004730	390389	
21413-2	HUNCBC-R1-02	ND	ND	8	10/17/85	15:35:00	0.79	0.77	0.17	86	794464	1004730	390389	
21413-3	HUNCBC-R1-09	ND	ND	8	10/17/85	15:35:00	0.79	0.77	0.17	86	794464	1004730	390389	
21413-4	11NCBC-R2-02	Y 10.07	ND	8	10/08/85	11:27:00	0.76	1.08	100		56944	58080	76604	
21413-5	11NCBC-R2-03	Y 50.00 ml	148	8	10/18/85	11:23:00	0.78	0.82	0.15	74	27890300	35784000	14976200	
21413-6	11NCBC-R2-09	Y 10.01	ND	8	10/17/85	16:55:00	0.80	0.94	94		430407	556721	692407	
21413-7	11NCBC-R2-09A	ND	ND	8	10/17/85	16:55:00	0.80	0.94	94		430407	556721	692407	
21413-8	11NCBC-R3-02	Y 10.08	ND	8	10/08/85	12:00:00	0.80	0.94	94		127582	155404	193962	
21413-9	11NCBC-R3-02NS	Y 10.14	0.97	8	10/08/85	12:20:00	0.84	0.80	0.96	98	82455	97716	41608	
21484-10	11NC-R1-5-06	Y 0.53	515	8	10/08/85	12:36:00	0.78	0.82	18.67	99	9101720	11679500	5278470	
21484-11	11NC-R1-5-10	ND	ND	8	10/08/85	00:00:00	1.00	1.00	100		732871	861074	1051050	
21484-12	11NCBC-R3-03	Y 50.00	250	8	10/18/85	11:46:00	0.78	0.87	0.18	91	60332100	77398900	31778300	
21484-13	11NCBC-R4-02	Y 10.07	ND	8	10/08/85	13:19:00	0.80	0.99	91		102590	131960	152101	
21484-14	11NCBC-R5-02	Y 10.13	0.51	8	10/08/85	13:37:00	0.74	0.70	0.92	94	290802	366952	460250	
21484-15	11NCBC-R5-09	ND	ND	8	10/08/85	13:37:00	0.74	0.70	0.92	94	187165	213156	303039	
21484-16	11NCBC-R5-09A	ND	ND	8	10/08/85	13:37:00	0.74	0.70	0.92	94	187165	213156	303039	
21484-17	11NCBC-R3-04	Y 50.00 ml	227	8	10/18/85	10:46:00	0.78	0.88	0.20	102	39956600	51048500	21022100	
21591-18	HUNCBC-R1-01	Y 0.51	193	8	10/08/85	13:55:00	0.80	0.79	18.75	96	3497650	4369880	888910	
21591-19	HUNCBC-R2-01	Y 0.54	111	8	10/08/85	14:20:00	0.79	0.81	18.74	101	1421090	1806100	804151	
21591-20	HUNCBC-R2-02	ND	ND	8	10/08/85	14:20:00	0.79	0.81	18.74	101	1421090	1806100	804151	
21591-21	HUNCBC-R2-03	Y 10.00	ND	8	10/22/85	12:17:00	1.01	0.72	1.01	101	171056	169312	155680	
21591-22	HUNCBC-R2-09A (19:00)	ND	ND	8	10/22/85	12:17:00	1.01	0.72	1.01	101	171056	169312	155680	
21591-23	HUNCBC-R2-09A (19:40)	ND	ND	8	10/22/85	12:17:00	1.01	0.72	1.01	101	171056	169312	155680	

FB = Field Blank  
 P = Partial Scan/Confirmatory Analysis  
 NS = Native TCOD Spike  
 D = Duplicate/Fortified Field Blank  
 RI = Re-injection  
 MD = Not Detected  
 DL = Detection Limit  
 RX = Re-extraction

APPENDIX T

CLEOSOTE CHROMATOGRAM SUPPLIED BY SUPELCO, INC.

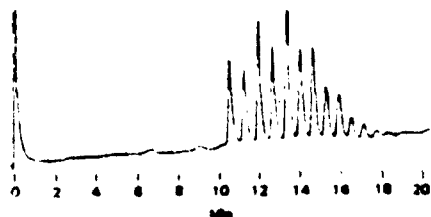
# Stable Phases for Separating High Boiling Mixtures

erations of very high boiling mixtures (glycerides, cholesteryl esters, etc.) require a stationary phase with good stability at temperatures above 300°C.

For packed columns, Dexsil® 300, a low polarity carborane silicone, can be used at up to 450°C with negligible bleed. Short (12" to 18") columns of 1% Dexsil are recommended for many types of samples (Figures A - D). For complex hydrocarbon mixtures, 3% Dexsil is suggested (Figure E). For more details, request Bulletins 743 and 768.

SPB-1 capillary columns (bonded SE-30 phase) offer a stable baseline and thermal stability to 320°C. We recommend you use a 0.75mm ID column and on-column injection (flash vaporization) when analyzing high boiling mixtures. This will prevent the discrimination that occurs with splitter systems. Under these conditions, branched hydrocarbons (short peaks) and n-alkanes (tall peaks) in a wax sample are well separated (Figure F). You can easily install and use a 0.75mm ID column in a packed column GC (see "Wide Bore Capillary Columns" in the index or request Bulletin 814 for more details).

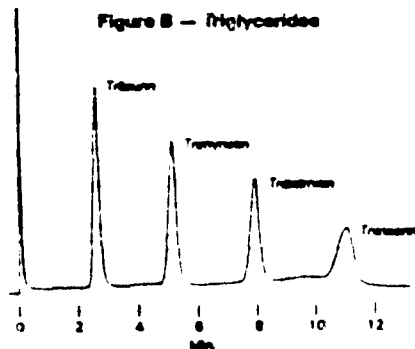
Figure A - Pentaerythritol Esters



1% Dexsil 300 on 100/120 Supelcoport, 18" x 1/8" SS. Col. Temp. 125° to 300°C at 8°C/min. Inlet Temp. 325°C. Det. Temp. 350°C. Flow Rate 20ml/min. N<sub>2</sub>. Sample 1 µl chloroform containing 10 µg esters.

Packing: Cat. No. 1-1972. \$81/20g

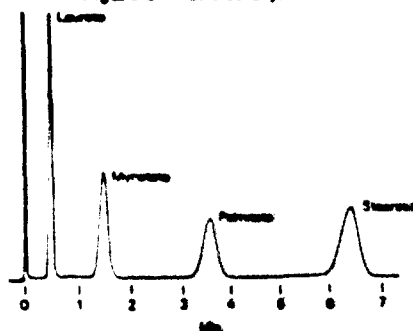
Figure B - Triglycerides



1% Dexsil 300 on 100/120 Supelcoport, 18" x 1/8" SS. Col. Temp. 275° to 350°C at 8°C/min. Inlet Temp. 325°C. Det. Temp. 350°C. Flow Rate 20ml/min. N<sub>2</sub>. Sample 1 µl chloroform containing 1 µg each triglyceride.

Packing: Cat. No. 1-1972. \$81/20g

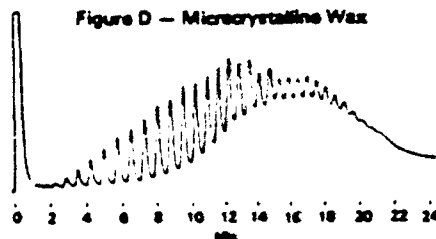
Figure C - Cholesteryl Esters



1% Dexsil 300 on 100/120 Supelcoport, 18" x 2mm ID glass. Col. Temp. 300° to 350°C at 6°C/min. Inlet Temp. 325°C. Det. Temp. 350°C. Flow Rate 40ml/min. N<sub>2</sub>. Sample 1 µl chloroform containing 1 µg each ester.

Packing: Cat. No. 1-1972. \$81/20g

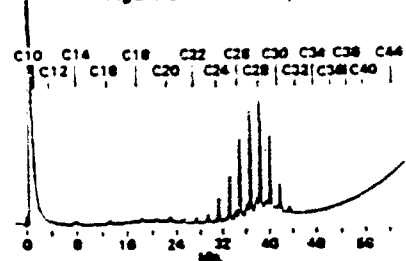
Figure D - Microcrystalline Wax



1% Dexsil 300 on 100/120 Supelcoport, 18" x 1/8" SS. Col. Temp. 175° to 350°C at 8°C/min. Inlet Temp. 325°C. Det. Temp. 350°C. Flow Rate 20ml/min. N<sub>2</sub>. Det. FID Sens. 16 x 10<sup>11</sup> AFS. Sample 1 µl chloroform containing 30 µg wax.

Packing: Cat. No. 1-1972. \$81/20g

Figure E - Wax Sample



3% Dexsil 300 on 100/120 Supelcoport, 6" x 1/8" SS. Col. Temp. 100° to 360°C at 4°C/min. Flow Rate 20ml/min. N<sub>2</sub>. Sample 1 µl chloroform containing 30 µg wax.

Packing: Cat. No. 1-1973. \$107/20g

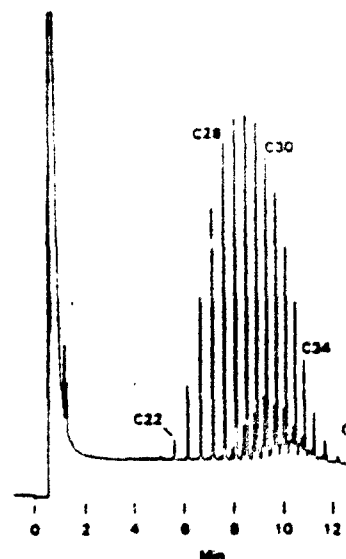
Additional Packing for High Boiling Aromatics

1-2132 10% SP-2250 on 100/120 Supelcoport, 20g \$62

For packed columns, see "Columns" in the index.

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Figure F - Wax Sample Injected on 0.75mm ID Column in a Packed Col



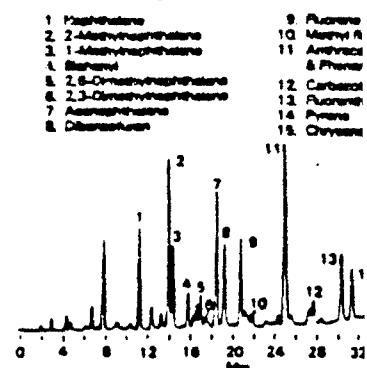
SPB-1 wide bore capillary column, 30m x 0.10mm I.D. Col. Temp. hold 2 min at 130°C, 320°C at 18°C/min, and hold 15 min. Temp. 350°C. Flow Rate, 15cc/min. He. Inlet. Det. FID. Sens.: 4 x 10<sup>11</sup> AFS. Sample commercial wax in undecane, 130 µg/µl.

Column: Cat. No. 2-3755.

## High Boiling Aromatics

We recommend 10% SP-2100 silicone for separating high boiling aromatics (Figure G). A 10% S methyl phenyl silicone is also useful for such separations. For more details request Bulletin 743.

Figure G - Cresols



10% SP-2100 on 100/120 Supelcoport, 1 SS. Col. Temp. 100° to 300°C at 6°C/min. 20ml/min. N<sub>2</sub>. Det. FID. Sample, 0.1 µl.

Packing: Cat. No. 1-1989. \$

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APPENDIX U

BATTELLE COLUMBUS LABORATORIES ANALYTICAL METHODOLOGY AND RESULTS FOR  
DIOXIN/FURANS IN NCBC SAMPLES

## APPENDIX U

### BATTELLE COLUMBUS LABORATORIES ANALYTICAL METHODOLOGY AND RESULTS FOR DIOXIN/FURANS IN NCBC SAMPLES

#### Introduction

This appen'ix<sup>a</sup> describes the analytical procedures used to determine the levels of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in three soil samples and three QA/QC samples submitted by EG&G Idaho, Inc. One of these soil samples, HU-NCBC-R2-02, was treated soil from the Huber NCBC test. The other two soil samples were from the IT Corporation testing NCBC, which was concurrent with the Huber testing. These data are included because of the QA/QC program interactions. The specific levels of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) and 2,3,7,8-tetrachlorodibenzofurans (2,3,7,8-TCDF) as well as the congener class concentrations of the tetrachloro- through octachloro- PCDD/PCDF classes were determined.

#### Extraction

Ten grams of each of the three samples, as well as the duplicate and native spike samples, were weighed and transferred to Soxhlet extractors. Sample number IT-NCBC-R1-01 was used as the duplicate, and HU-NCBC-R2-02 was used as the native spike. These five samples and the method blank were spiked with 25.0 ng each of three isotopically labelled internal standards: 2,3,7,8-tetrachlorodibenzo-p-dioxin-<sup>13</sup>C<sub>12</sub>  
(2,3,7,8-TCDD-<sup>13</sup>C<sub>12</sub>) 2,3,7,8-tetrachlorodibenzofuran-<sup>13</sup>C<sub>12</sub>

a. Information is referenced from Battelle Columbus Laboratories "Final Report on Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Soil Samples", prepared for EG&G Idaho, Inc., dated May 21, 1986.

(2,3,7,8-TCDF-<sup>13</sup>C<sub>12</sub>), and octachlorodibenzo-p-dioxin-<sup>13</sup>C<sub>12</sub> (OCDD-<sup>13</sup>C<sub>12</sub>). The samples were then Soxhlet-extracted for 18 hours using benzene.

#### Extract Cleanup

The benzene extracts were concentrated to approximately 5 ml using three stage Snyder columns, diluted with 5 ml of hexane, and transferred to multilayered columns containing activated silica gel, 44 percent concentrated sulfuric acid on silica gel, and 33 percent 1M sodium hydroxide on silica gel. The columns were rinsed with 70 ml of hexane and the entire eluates were collected. The purpose of these columns was to remove acidic and basic compounds from the extracts as well as oxidizable materials.

The benzene/hexane eluates were concentrated using a gentle stream of nitrogen gas and solvent-exchanged into hexane. The hexane solutions were chromatographed through columns containing approximately 1 gm of activated basic alumina using hexane/methylene chloride (97:3, v/v), and hexane/methylene chloride 1:1, v/v) as elution solvents. The 1:1 hexane/methylene chloride eluates were collected, concentrated to near dryness, and dissolved in 20 µl of n-decane containing 10 ng of 1,2,3,4-TCDD-<sup>13</sup>C<sub>12</sub>, which was used as an absolute recovery standard. The solutions were stored at 0°C and protected from light until analyzed.

#### Analysis

The extracts were analyzed and quantified for PCDD/PCDF using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The HRGC/HRMS consisted of a Carlo Erba Model 4160 gas chromatograph interfaced directly into the ion source of a VG Model 7070 mass spectrometer. The chromatographic column was a 6CM DB-5 fused silica column using helium carrier gas at a flow velocity of 25 cm/sec. The mass spectrometer was operated in the electron impact (EI) ionization mode at a mass resolution of 9,000-12,000 (M/ΔM, 10 percent valley definition).

The operating parameters of the HRGC/HRMS are summarized in Table U-1. All HRGC/HRMS data were acquired by multiple mass-detection (MID) using a VG Model 2035 Data System. The exact masses monitored are shown in Table U-2.

#### Quality Assurance

The operation of the HRGC/HRMS was evaluated each day by analyzing standard mixtures of PCDD/PCDF isomers. These consisted of native and isotopically labelled isomer mixtures used to determine response factors, mixtures of selected PCDD/PCDF isomers to evaluate the stability of the chromatographic elution windows, the TCDD isomer mixtures to evaluate isomer resolution. The mass accuracy of the MID unit was evaluated at least every 4 hours by focusing selected ion masses from perfluorokerosene (PFK) and correcting the slope to account for minor variations. Mass focus stability was assured by the use of a reference PKF "lock mass" to correct for any mass focus drift.

A method blank and a native spiked sample were processed during the extraction and cleanup of the samples. The results of these analyses are summarized in Table U-3. The raw mass spectral data, areas and heights, are presented in Table U-4. The method blank was free from PCDD/PCDF contamination except for trace levels of HpCDD and OCDD. Background levels of higher chlorinated dioxins are periodically observed in low level PCDD/PCDF analyses. The average native spike recoveries were within approximately 6.5 percent of the spiked value.

#### Quantification

The PCDF/PCDD isomers were quantified by comparing the sum of the two ion masses monitored for each class to the sum of the two ion masses monitored for the corresponding internal standard. The 2,3,7,8-TCDF- $^{13}\text{C}_{12}$ \* was used to quantify the tetrachlorodibenzodioxins

\* was used to quantify the tetrachlorodibenzofuran isomers, and 2,3,7,8-TCDD- $^{13}\text{C}_{12}$



... and heptachloro- dioxin and furan isomers. The OCDD-<sup>13</sup>C<sub>12</sub> was used for the heptachloro- and octachlorodioxins and furans. Experimental relative response factors were obtained by analyzing a test mixture that contained representatives of the tetrachloro- through octachloro- PCDD/PCDF classes. These response factors were included in all calculations used to quantify the data. The response factors were calculated by comparing the sum of the areas of the two ion masses monitored for each congener class to the corresponding internal standard ion masses. The experimental response factors were:

Data File			
Analyte	591018	591109	Average
TCDF	1.0620	0.9982	1.0301
TCDD	1.0270	1.1209	1.0740
PCDF	0.9463	1.0413	0.9938
PCDD	0.3942	0.4622	0.4282
HxCDF	0.9669	1.0277	0.9973
HxCDD	0.4425	0.4893	0.4659
HpCDF	2.0288	2.4330	2.2309
HpCDD	3.054	3.0823	3.0682
OCDF	0.9205	1.0532	0.9869
OCDD	1.0568	1.0776	1.0672

Internal Standard RRF Relative to  
1,2,3,4-TCDD<sup>13</sup>C<sub>12</sub> for Recovery Calculations

TCDF- <sup>13</sup> C <sub>12</sub>	0.9695
TCDD- <sup>13</sup> C <sub>12</sub>	1.1-49
OCDD- <sup>12</sup> C <sub>12</sub>	0.2491

The formula used for quantifying the PCDD/PCDF isomers was:

$$\text{Level (ppb)} = \frac{\text{Areas of Quantification Masses} \times \text{Amount of Internal Standard (ng)}}{\text{Areas of Internal Std. Masses} \times \text{Resp. Factor} \times \text{Wt. Sample (g)}}$$

The criteria used to identify PCDD and PCDF isomers were:

- (1) Simultaneous responses at both masses
- (2) Chlorine isotope ratios within  $\pm 15$  percent of the theoretical values
- (3) Chromatographic retention times within windows determined by analyses of standards
- (4) Signal-to-noise ratio equal to or greater than 2.5 to 1.

The 2,3,7,8-TCDF/TCDD isomers included the additional criterion that they elute within  $\pm 2$  seconds of their isotopically labeled analogs. A limit of detection was calculated for samples in which a particular chlorination class was not detected. The formula used was:

$$\text{Limit of Detection (ppb)} = \frac{\text{Hts. of Quant. Masses} \times \text{Amt. Int. Std. (ng)} \times 2.5}{\text{Hts. of Int. Std. Masses} \times \text{Resp. Factor} \times \text{Wt. Sample (g)}}$$

#### Results

The levels of PCDD/PCDF determined in the samples are summarized in Table U-3. Analysis of sample number IT-NCBC-R1-01 in duplicate indicated the presence of 2,3,7,8-TCDD at the levels of 165 ppb and 170 ppb. Since the level of native 2,3,7,8-TCDD in the sample is approximately 70 times higher than the level of the internal standard, it is possible that the response of the ion source of the mass spectrometer was not linear. The sample was reinjected with a 0.1  $\mu\text{l}$  injection size (rather than 2.0  $\mu\text{l}$ ). This analysis indicated the level of 2,3,7,8-TCDD to be 220 ppb; however, it is possible that the actual level is even higher. In an attempt to obtain a more representative value for the level of native

material in this sample, 100 ppb of 2,3,7,8-tetrachlorodibenzo-p-dioxin- $^{37}\text{Cl}_4$  (2,3,7,8-TCDD  $^{37}\text{Cl}_4$  was spiked into the sample extract. The amount of 2,3,7,8-TCDD  $^{37}\text{Cl}_4$  was then determined relative to 2,3,7,8-TCDD  $^{13}\text{C}_{12}$ . The sample was diluted by a factor of ten, and the native 2,3,7,8-TCDD was quantified based on the 2,3,7,8-TCDD- $^{37}\text{Cl}_4$ . As in the 0.1  $\mu\text{l}$  injection of the nondiluted sample, this set of analysis indicated the level of native material to be 220 ppb. The formulas used for this calculation were:

$$\text{Std } R_f \text{ } ^{13}\text{C}_{12}/^{37}\text{Cl}_4 = \frac{\text{Area } ^{13}\text{C}_{12} \text{ Quant Masses in Std} \times \text{Amt. } ^{37}\text{Cl}_4 \text{ (ng)}}{\text{Area } ^{13}\text{C}_{14} \text{ Quant Masses in Std} \times \text{Amt. } ^{13}\text{C}_{12} \text{ (ng)}}$$

and  $1, (\text{Std } R_f \text{ } ^{13}\text{C}_{12}/^{37}\text{Cl}_4$

$$\text{Native Conc} = \frac{\text{Area Native Quant Masses} \times \text{Amt. } ^{37}\text{Cl}_4 \text{ (mg)}}{\text{Area } ^{37}\text{Cl}_4 \text{ Quant Mass} \times \text{Wt. Sample (g)}}$$

Table U-4 lists the area data and the concentrations used in these calculations. The reconstructed ion chromatograms for these three data files (Std, Lower Sensitivity, High Sensitivity) are contained in the section of this report that is labeled "IT-NCBC-R1-01  $^{37}\text{Cl}_4$ -Spike". The only alternative available to obtain a reliable value for the amount of native material in this sample involves the additional cost of repeating in extraction, using a smaller sample size (1 gram versus 10 grams). Because of cost and time constraints, it was decided that it be inappropriate to proceed any further with this sample.

$$\text{Sample } R_f \text{ } ^{13}\text{C}_{12}/^{37}\text{Cl}_4 = \frac{\text{Area } ^{13}\text{C}_{12} \text{ Quant Masses in Std} \times \text{Amt. } ^{37}\text{Cl}_4 \text{ (ng)}}{\text{Area } ^{37}\text{Cl}_4 \text{ Quant Masses} \times \text{Amt. } ^{13}\text{C}_{12} \text{ (ng)}}$$

For those samples in which a particular chlorination class was not detected, a detection in parts-per-billion (ppb) is listed in Table U-3. The height and area data used to calculate the concentrations and detection limits can be found in Table U-5.

The percent recoveries of the internal standards in each sample and the chlorine isotope ratios for isomers or isomer classes detected are reported in Table U-6 and U-7, respectively. The formula used to calculate the percent recoveries was:

$$\text{Percent Recovery} = \frac{(\text{Area of Quant. Masses of Stnd}) \times (\text{Amt. 1,2,3,4,-TCDD-}^{13}\text{C}_{12}) \times 100}{(\text{Area of Quant. Masses of 1,2,3,4-TCDD-}^{13}\text{C}_{12}) \times (\text{Amt. Stnd}) \times R_f}$$

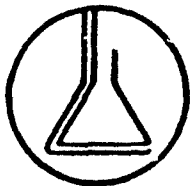
where  $R_f$  is relative to 1,2,3,4-TCDD- $^{13}\text{C}_{12}$ .

#### Single Ion Current Chromatograms

The single ion current chromatograms for the samples, standards, and decane analyses are included in the referenced Battelle report (see footnote at beginning of this appendix). They were assembled in analysis order and are cross-referenced by the table that prefaces each section. The data files are six-digit numbers, with the first two numbers denoting the instrument logbook in which the analysis is recorded. The third and fourth numbers indicate the package in the logbook, and the fifth and sixth numbers mark the line on which the entry was made. All information pertaining to extraction and workup of the samples can be found in Battelle Laboratory Book Number 40196. The GC/MS acquisition parameters can be found in Laboratory Record Book Number 41270.

APPENDIX V

CAL EP TOXICITY TEST RESULTS FOR HUBER TEST BAGHOUSE FILTER MATERIAL



California Analytical Laboratories, Inc.  
2544 Industrial Boulevard • West Sacramento, CA 95691 • (916) 372-1393

January 31, 1986  
Lab No. 21591-A


Kathy MacKay  
EG & G Idaho, Inc.  
1955 Fremont Ave.  
Idaho Falls, Idaho 83415

One soil sample was re-submitted for metals analysis by EP  
Toxicity Test Method (EPA 1310).


CAL I.D.  
21591-4

RESULTS:

Analytical and quality assurance results are presented in the attached data sheets. Spike recoveries for post-digestion spike are low for some elements. This may be attributed to the high initial sample pH (11.9). The maximum allowable volume of 0.5N acetic acid to be added to a sample is 4 milliliters per gram according to Method 1310. In this sample, a final pH of 9.7 was achieved after addition of 8 milliliters acid to a 2 gram sample. This pH is too high for dissolution of some elements.

  
Paul Taylor  
President

ds

  
Donna Gilmore  
Manager Inorganic Services

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This report is for the sole and exclusive use of the client to whom it is addressed.  
Samples not destroyed in testing are retained a maximum of thirty (30) days unless otherwise requested.

EF TOXICITY

DATA SHEET

SAMPLE I.D.: HU-NCBC-R2-03

CAL I.D.: 21591-4

<u>Metals</u>	<u>mg/L Leachate Regulatory Limit</u>	<u>mg/L Found in Leachate</u>
Arsenic	5.0	<1.0
Barium	100	0.75
Cadmium	1.0	<0.1
Chromium	5.0	<0.1
Mercury	0.2	<0.01
Lead	5.0	1.1
Selenium	1.0	<0.5
Silver	5.0	<0.5

PREPARED BY: JRB

APPROVED BY: [Signature]

EP TOXICITY TEST  
 QUALITY ASSURANCE  
 DATA SHEET

POST-DIGESTION

SAMPLE I.D.: HU-NC BC-R2-03

CAL I.D. 21591-4

Metals	mg/L Leachate Regulatory Limit	Sample mg/L	Dup. Sample mg/L	Rel. % Diff.	Post Digestion Spike		% Recovery
					mg/L Spike Result	mg/L Spike Level	
Arsenic	5.0	<1.0	<1.0	0	1.85	2.00	93
Barium	100	0.75	0.68	9.8	2.24	1.50	99
Cadmium	1.0	<0.1	<0.1	0	0.214	0.200	107
Chromium	5.0	<0.1	<0.1	0	0.191	0.250	76
Mercury	0.2	<0.01	<0.01	0	2.10	2.0	105
Lead	5.0	1.1	1.7	43	3.03	2.00	96
Selenium	1.0	<0.5	<0.5	0	0.420	1.00	42
Silver	5.0	<0.5	<0.5	0	0.063	1.00	6.3

PREPARED BY: RB  
 APPROVED BY: DRG

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California Analytical Laboratories, Inc.



APPENDIX W

BATTELLE COLUMBUS LABORATORIES PRIORITY POLLUTANT METALS  
AND CYANIDE ANALYTICAL RESULTS FOR SIX NCBC SOIL SAMPLES

APPENDIX W  
BATTELLE COLUMBUS LABORATORIES PRIORITY POLLUTANT METALS  
AND CYANIDE ANALYTICAL RESULTS FOR SIX NCBC SOIL SAMPLES

Ag, As, Be, Cd, Cr, Cu, Ni, Pb, Se, Ti & Zn Analysis

A 5.0 g sample was weighed from the thoroughly mixed content of the jar containing the original sample. The sample was placed in a 150 mL and 5.0 mL of concentrated  $\text{HNO}_3$  were added to it. The beaker was placed on a hot plate set at approximately  $90^\circ\text{C}$  and left to digest for 3 hours without allowing it to boil. The beaker contents were then evaporated to near dryness prior to the addition of 5.0 mL of concentrated  $\text{HNO}_3$ . The beaker was placed back on the hot plate of the contents were evaporated to near dryness again. This step was repeated three times after which 5 mL of 1:1  $\text{HNO}_3$ :DI  $\text{H}_2\text{O}$  and 7.5 mL DI water were added. The mixture was heated for 15 minutes prior to filtering through white ribbon paper (No. 584) with pulp into a 50 mL volumetric flask. The contents of the volumetric flask were brought to volume using DI water. This solution was analyzed using either Zeeman graphite furnace atomic absorption spectrophotometry (Z-GF-AAS) or inductively coupled argon plasma (ICAP) according to the instructions given in the manufacturer's manual and the wave lengths given in the attached Tables 3 & 4.

Hg Analysis

A 1.0 g sample was weighed from the thoroughly mixed contents of the jar containing the original sample. The sample was then placed in a 125 mL Erlenmeyer flask and to it 10 mL aqua regia solution were added. The flask was placed on a steam bath and heated for approximately 1 hour then diluted to 50 mL with DI water. 2.5 mL concentrated  $\text{H}_2\text{SO}_4$ , 10 mL  $\text{KMnO}_4$ , and 4 mL  $\text{F}_2\text{S}_2\text{O}_8$  were added. This solution was heated in a steam bath for approximately 4 hours\* were added prior to analysis by cold vapor atomic

\* After which 1 drop of hydroxylamine hydrochloride and 5 mL stannous chloride.

absorption spectrophotometry (CV-AAS) at the wavelength given in the attached Table 3.

#### Sb Analysis

A 5.0 g sample was weighed from the thoroughly mixed contents of the jar containing the original sample. The sample was placed in a 150 mL beaker and 10 mL aqua regia were added to it. The mixture was placed on a hot plate set at approximately 90°C and left to digest for three hours. The beaker was removed, cooled and its contents filtered into a 100 mL volumetric flask through a white ribbon (No. 589) paper with pulp. The beaker and the filter were washed with DI water and the washings were added to the volumetric flask DI water was used to bring the volume to 100 mL prior to analysis by Zeem graphite furnace atomic absorption spectrophotometry (Z-GF-AAS) at the wave length given in the attached Table 3.

#### QA/QC

All the samples were logged into the central laboratory record system and given Battelle numbers prior to distribution to the individual analysts. All the laboratory activities and results were recorded in Laboratory Book No.'s 40602 and 39818. Sample 40196 53-2 was used for both duplicate and spike recovery studies. The spiking was accomplished by adding the spike level given in Table 2 to the soil sample immediately after adding the acid to it. The spiked sample was then taken through the sample analytical procedure as for the unspiked sample.

APPENDIX X

DISCUSSION ON SOIL PRETREATMENT FACILITY

## DISCUSSION ON SOIL PRETREATMENT FACILITY

The soil pretreatment facility can prepare a variety of contaminated soils for treatment in the AER. The dry system is capable of crushing, blending, drying, pulverizing, and sizing a variety of composite soils, of sizes up to 10 inches, to produce a ground, free-flowing material that will pass through a 35-mesh screen, thereby assuring the prepared soil will have the desired AER retention time.

Figure X-1 presents the detailed flow diagram for the soils pretreatment facility. Figure X-2 shows the general arrangement plans and key views to aid in understanding the following discussion of the different phases.

### Soils Receiving and Handling

All solid feed material is delivered to the soil pretreatment facility in sealed, portable containers (bins). Each bin is designed to hold approximately 12 cubic yards or 30,000 lb of material.

Plant operators will use a recently loaded bin or will take a loaded bin out of the bin staging area with a roll-off truck or forklift and place them into a 40,000-lb capacity feed surge bin.

As the bin is positioned exactly over the feed chute, a hydraulic mechanism is actuated to gently lower the bin into the feed opening. The feed chute has peripheral gasketing to prevent any dust leakage.

The feed bin has a stationary 10-inch grid grizzly located under the feed chute to divert oversize material to a gravity chute, which will collect occasional trash solids in a tote bin. The grizzly oversize material will be removed and handled separately.

A 42-inch-wide bucket elevator feeder is installed under the feed bin inside an enclosed housing, which is vented to the primary bag filter. This feeder has a variable-speed drive, which can be adjusted to match the feed rate established by the operator in the control room. The bucket elevator discharges onto a 30-inch feeder belt with a self-cleaning, cross-belt, electromagnetic tramp iron magnet mounted over the 30-inch feeder belt to remove magnetic metal objects to a separate chute and tote bin. A metal detector is installed downstream from the tramp iron magnet to sound an alarm and stop the weight belt if nonmagnetic metal appears on the 30-inch belt. A manually operated hydraulic picker mechanism pushes the metal or other objects off the belt to another chute and tote bin, which collects this material for disposal by other means. The 30-inch belt gravity feeds the primary crusher.

From this point in the process, all solid materials are handled either by gravity flow between process equipment or by dense-phase pneumatic solids transport systems, which convey fine dry material to the next process step.

#### Soils Crushing, Drying, and Pulverizing

The feed to the process can be a maximum of 10 inches diameter into the primary crusher, which is a 10-hp impact breaker, having the capability to open circuit crush the incoming feed at the rate of 6,700 lb/hr to a 1-1/2-inch maximum particle size. This crushed product is then fed by gravity into the indirect-heated dryer.

The primary crusher can process feed soil mixtures containing as high as 25-percent moisture, but second-stage comminution in hammer mill and screen separations at 35 mesh are not possible with solids containing more than about 1-percent moisture. Therefore, the material is dried ahead of the hammer mill. The process drying system consists of a disc dryer, to dry 30-percent moisture feed soil to 1-percent moisture at a feed rate of 6700 lb/hr. The dryer solids at 210°F gravity discharge into the hammer

mill feed bin. The indirect-heated dryer is fed by a gravity discharge from the primary crusher located above the dryer in Module 02.

The heat for the dryer system comes from a package gas-fired Dowtherm system with 3.0-million-Btu/hr heat capacity. The unit is equipped with a 1-1/2-hp blower and 20-hp pump. This dryer will have its torus disc flights hard-surfaced to resist the wear from the 1-1/2-inch-solids in the feed soil.

The hammer mill operates in closed circuit with a multideck vibrating screen to produce 6000 lb/hr of -35 mesh product. An approximate 150 percent circulating load of +35 mesh solids recirculates back to the hammer mill to achieve the fineness of grind required for the process specification. This is achieved by gravity feeding the 1-1/2-inch-sized dryer discharge into the hammer mill feed bin where a gravity discharge chute from the screen located in the module above the dryer drops about 8900 lb/hr of +35 mesh screen oversize product into the hammer mill surge bin. The combined flow passes through a rotary valve and into the hammer mill, which can produce pulverized material that is 99 percent -1/4 inch with an average particle size of about 16 mesh with 40 percent by weight of -35 mesh material.

The hammer mill discharge is fed to the dense-phase pneumatic conveyor system, which elevates the coarse ground product to feed the vibrating screen. The system could accumulate a circulating load of lightweight fibrous material resulting from shredding wood or cloth in the mill. This material is intermittently passed through a fiber chopper, which drops the fibrous material at the rate of 100 lb/hr into a very fine powder. This material is gravity-dropped into a surge bin that also collects -35 mesh screen product and dust from the bag filters.

The surge bin discharges into a rotary valve, which feeds the dense-phase pneumatic transport system. This system conveys the comminuted product at an approximate rate of 6,000 lb/hr to its bag filter, which has been modified to provide a 10,000-lb surge capacity in the bottom of the bag filter. The product surge bin feeds a loss-in-weight feed system that

gravimetrically controls the rate of feed to the AER in the demonstration plant. The inlet flank to the AER is located 80 feet above the ground. The AER is fed through a rotary valve capable of discharging against the 1 psig pressure in the AER.

#### Dryer Off-Gas and Plant Ventilation Systems

The water vapor, air, and dust leaving the dryer vent are heated electrically, if necessary, and conducted to a separate bag filter. A backup bag filter is installed in the off-gas line to intercept any solids flow in the event of a fabric bag failure. Vapors entrained with the air and water vapor are swept out of the dryer into the vent gas ductwork with an ID fan and are captured in five carbon adsorption filters.

To prevent fugitive emissions from the facility, a complete plant ventilation system is provided to allow every bin, crusher, screen, and pneumatic conveyor to have an outlet into a bag filter system. Motive force is provided by a 3500-scfm, 25-hp, ID fan.

The pretreatment facility will receive 13.8 kV line power from outside the battery limits to feed a 500 kVA transformer. This transformer supplies 400-V, 3-phase\* motor starter modules, a 480-V, 5-k kVA transformer for one-phase lighting power, a 50-a, 30-pole lighting receptacle panel, a 20-a, 12-pole instrument panel, and a 60-a welding receptacle. All electrical switchgear is housed in a single module.

#### Descriptor of Modules

The pretreatment facility equipment has been arranged so that it can be fitted into 11 modules, each 12 feet wide by 11 feet 2 inches high by 39 feet long, with a maximum weight of 72,000 lb (i.e., module steel plus empty equipment). These dimensions and weight were chosen to allow for unrestricted transport on lowboys by road throughout the U.S. The modules are identified as follows:

\*Power to a motor control center containing fifty 445-V, 3-phase.



- Module 01 Feed surge bin, 42-inch bucket elevator, 30-inch belt feeder, grizzly, hopper, and 5-ton electric hoist.
- Module 02 30-inch belt feeder, tramp iron magnet, picker, and primary crusher.
- Module 03 Dryer, Dowtherm unit, and hammer mill feed bin.
- Module 04 Hammer mill, flexible screw conveyor, and rotary valve.
- Module 05 Vibrating screen, fiber chopper, knifegate, collection bin, rotary valve, and automatic sampler.
- Module 06 Plant ventilation primary bag filter, dust leak detector, electric air heaters, and screen air conveyor.
- Module 07 Dryer primary bag filter, dry secondary bag filter, dust leak detector, air heaters, blower, and activated carbon adsorbers.
- Module 08 Plant ventilation secondary bag filter, electric air heater, and blower.
- Module 09A 500-kVA transformer, 5-kVA transformer and motor control center circuit breakers, motor starters, and lighting and instrument panels.
- Module 09B Stairways.
- Module 21 Product air conveyor, product surge bin, and product feeder.

The first four modules in the process flow are stacked to achieve gravity transfer of material between equipment. Screw conveyors, or chain conveyors have not been used, as these would present added cleanup

difficulties. Belt feeders are used at the feed end for process feed control and to provide for tramp metal removal and pickup. Any dribble from these two feeders will be collected in tote bins for disposal. Elevation of material is by two air conveyor systems, one for the recycle load between the secondary crusher and screen and one for the product feed to the reactors.

A monorail/hoist installed in the top module will be used for raising and lowering bins containing grizzly oversize, tramp iron, etc., through a hoistway

Heating and cooling of the electrical control center during winter and summer will be a 2-ton unit heat pump. The temperatures in the module will be maintained between 40°F minimum and 90°F maximum. Siding will be provide for the modules to shield against wind during maintenance and to provide security

APPENDIX Y

DETAILED COST ESTIMATE FOR AER TREATMENT AND CLEANUP

APPENDIX V, EXHIBIT 1. DETAILED COST ESTIMATE FOR AER TREATMENT OF 20,000 TONS OF SOIL (reference case)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>CATEGORY 1: COMMON REMEDIAL COSTS</b>					
<b>MOBILIZATION</b>					
General mobilization and demobilization	1	LS	95,000	95	5% of Category 1 subtotal cost
Trial burn	1	LS	150,000	150	--
Community relations	1	LS	40,000	40	--
<b>CONSTRUCTION</b>					
Ancillary buildings and equipment:					
a) Water treatment facility	1	LS	300,000	300	GAC facility, 25 gpm (50 gpm peak) for decon, runoff, quench
b) Office trailer	12	M0	380	5	50 ft x 10 ft trailer
c) Employee trailer - breakroom	12	M0	380	5	50 ft x 10 ft trailer
d) Forklift for material preparation	12	M0	1,581	19	--
Utility upgrade:					
a) Electrical services construction	1	LS	68,000	68	\$58,000 for main facility, \$10,000 for excavation site area
b) Outdoor lights	5	EA	1,400	7	--
c) Water supply allowance	1	LS	20,000	20	--
d) Sewer connection	1	LS	30,000	30	--
e) Natural gas line	1	LS	30,000	30	Fuel to heat soil dryer
f) Telephone service allowance	1	LS	10,000	10	--
Excavation site buildings:					
a) Decontamination trailer rental (2)	28	M0	1,000	28	--
b) Vehicle decontamination station	1	LS	30,000	30	--

APPENDIX Y, EXHIBIT 1. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>EXCAVATION: LOAD AND HAUL</b>					
<b>Equipment:</b>					
a) Backhoe rental	12	MO	7,100	85	--
b) Tractor (with hoe ram) rental	12	MO	3,000	36	--
c) Rolloff truck rental (2)	24	M <sup>1</sup>	1,984	48	--
d) Rolloff boxes for operations	3	EA	5,000	15	3 for daily operation
e) Rolloff boxes for untreated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net vol, total storage for 7 days of operation
f) Rolloff boxes for treated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net vol, total storage for 7 days holding
<b>Labor:</b>					
a) Backhoe operator	1,760	HR	26	46	8 hr/day for 220 days
b) Tractor operator	1,760	HR	26	46	8 hr/day for 220 days
c) Rolloff truck drivers (2)	3,520	HR	26	92	8 hr/day for 220 days for each driver
d) Excavation foreman	1,760	HR	27	48	8 hr/day for 220 days
<b>HEALTH AND SAFETY</b>					
Health and safety personnel (2)	3,520	HR	25	88	8 hr/day for 220 days each worker
Per diem and general expense for H&S personnel	220	DY	100	22	25% of H&S personnel cost
Physicals (2/worker)	16	EA	600	10	--
Training	8	EA	1,100	9	--
Protective equipment	1,320	MD	63	83	Purchase and disposal of protective equipment
--Level C (6 sets)	440	MD	26	12	Purchase and disposal of protective equipment for truck drivers
--Level D (2 sets)					
Dust control	12	MO	1,980	24	--

APPENDIX Y, EXHIBIT 1. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>SITE RESTORATION</b>					
Spreading treated soil:					
a) Dump truck	12	MO	1,984	24	--
b) Truck driver	1,760	HR	26	46	8 hr/day for 220 days
Regrade site	84,700	SY	0.81	69	
Topsoil placement	2,820	CY	13.50	38	15% of 18,800 cy
Erosion matting	84,700	SY	0.80	68	--
Seeding and revegetation	84,700	SY	1.40	119	--
Salvage of rolloff boxes	59	EA	-2,500	-148	50% of cost, 3 operations, 28 untreated soil, and 28 treated soil
<b>COMMON REMEDIAL COST SUBTOTAL</b>				<b>1,997</b>	
Contingencies (25%)	--	--	--	499	Factor applied to subtotal
General administration (13%)	--	--	--	324	Factor applied to subtotal and contingencies
Contractor fee (8%)	--	--	--	226	Factor applies to subtotal and contingencies and GA
				<u>3,046</u>	
<b>CATEGORY 1: TOTAL COMMON REMEDIAL COST</b>					
<b>CATEGORY 2: COMMON OPERATING AND MONITORING COSTS</b>					
<b>GENERAL</b>					
On-scene coordinator	12	MO	5,500	66	\$25/hr x 8 hr/d x 27.5 d/mo
Per diem and general expense	330	DY	50	17	25% of labor cost
<b>MONITORING AND ANALYSIS PROGRAM</b>					
On-site lab facility	1	LS	250,000	250	Includes enclosure, high resolution GC/MS, ancillary equipment
<b>Analytical Lab Operation:</b>					
a) Chemists (4)	48	MO	8,333	400	--
b) Per diem and general expense	330	DY	303	100	25% of labor cost
c) Lab expendables and miscellaneous	12	MO	800	10	--
<b>Ambient air monitoring:</b>					
a) Residential stations	4	EA	30,000	120	High-volume air samplers
b) Background database	1	LS	20,000	20	--
c) Air sampling analysis	12	MO	4,000	48	--

APPENDIX Y, EXHIBIT 1. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>Sampling collection:</b>					
a) Sampling technicians (2)	24	MO	4,888	117	--
b) Per diem and general expense	330	DY	88	29	25% of labor cost
c) Expendables and miscellaneous	12	MO	800	10	--
<b>FACILITY OPERATIONS</b>					
<b>Water treatment operation:</b>					
a) General	12	MO	550	7	\$20/day x 27.5 d/mo
b) Carbon replacement	17,000	lbs	2.00	34	1 lb carbon/1000 gal for 17,000,000 gal; decon, quench, runoff
c) Facility operator	2,640	HR	25.5	67	8 hr/d for 330 days
d) Discharge to POTW	17,000	1000 gal	1.20	20	
e) Maintenance (misc)	1	LS	30,000	30	10% of facility capital cost
Water user charge	4,752	1000 gal	0.80	4	10 gpm x 60 min/hr x 24 hr/d
Electricity	12	MO	3,168	38	x 330 d; decon and ancil facility
				<u>1,387</u>	100 kW x 24 h/d x 27.5 d/mo x \$0.048/kWh
<b>COMMON OPERATING AND MONITORING COST SUBTOTAL</b>					
Contingencies (20%)	--	--	--	277	Factor applied to subtotal
General Administration (13%)	--	--	--	216	Factor applied to subtotal and contingencies
Contractor fee (8%)	--	--	--	150	Factor applied to subtotal + contingencies and GA
				<u>2,030</u>	
<b>CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST</b>					
<b>CATEGORY 3: AER FACILITY SITE SETUP/REMOVAL COSTS</b>					
Transportation trailers to and from site	27	TRL	6,000	162	Transport from Borger, TX to Gulfport, MS and back
Erect AER and pretmt building	972	TON	125	122	27 modules @ 72,000 lb or 36 tons each
Startup/shakedown	1	LS	15,000	15	--

APPENDIX V, EXHIBIT 1. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
Remove AER and pretmt building	972	TON	100	97 396	27 modules @ 72,000 lb or 36 tons each
<u>FACILITY SITE SETUP/REMOVAL COST SUBTOTAL</u>					
Contingencies (25%)	--	--	--	99	Factor applied to subtotal
General administration (13%)	--	--	--	64	Factor applied to subtotal and contingencies
Contractor fee (8%)	--	--	--	45 604	Factor applied to subtotal and contingencies + GA
<u>CATEGORY 3: TOTAL AER SITE SETUP/REMOVAL COSTS</u>					
<u>CATEGORY 4: AER FACILITY OPERATING AND MAINTENANCE COSTS</u>					
EQUIPMENT USE CHARGE	14,500	MO	111,000	1,610	Pretreatment and AER equipment includes transportation time
<u>OPERATION OF AER FACILITY</u>					
Utilities:					
a) AER power requirements	20,000	TON	52.80	1,056	1100 kWh/ton * \$.048/kWh
b) Ancillary power requirements	12	MO	6,336.00	76	200 kWh * 24 h/d * 27.5 d/mo * \$.048/kWh
c) Nitrogen	950,000	100 ft <sup>3</sup>	0.40	380	200 scfm use rate during operation or ~4750 ft <sup>3</sup> /ton
d) Water user charge	5,227	1000 ft <sup>3</sup>	0.80	4	11 gpm for ash quenching * 60 min/h * 24 h/d * 330 d/y
e) Natural gas	12 23,760	1000 ft <sup>3</sup>	5.00	119	3000 cf/h use rate during operation or ~1200 ft <sup>3</sup> /ton
Maintenance of AER:					
a) Electrode replacement	20,000	TON	1.25	25	Replace @ 20,000 tons/electrode
b) Core replacement	20,000	TON	25.00	500	Replace @ 2,000 tons/core
c) Misc. materials	20,000	TON	6.00	120	3% of equipment capital cost
d) Baghouse and carbon replacement	20,000	TON	2.00	40	--



APPENDIX Y, EXHIBIT 1. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>Labor at AER facility</b>					
a) Operations manager (1)	12	MO	8,532.48	102	Salary @ \$48,000/y x burden
b) Secretary (1)	12	MO	2,844.16	34	Salary @ \$16,000/y x burden
c) Maintenance personnel (2)	24	MO	4,614.29	111	Salary @ \$25,958/y x burden
d) Reactor operator (4)	48	MO	4,477.60	215	Salary @ \$25,189/y x burden
e) Yard operator (4)	48	MO	4,204.02	202	Salary @ \$23,650/y x burden
f) Pretreatment area operator (4)	48	MO	4,340.90	208	Salary @ \$24,420/y x burden
g) Relief operator (2)	24	MO	4,340.90	104	Salary @ \$24,420/y x burden
h) Engineers/safety personnel (3)	36	MO	6,754.88	243	Salary @ \$38,000/y x burden
i) Per diem and general expense	330	DY	923.48	305	25% of total labor cost for 21 employees
<b>Health and safety program</b>					
a) Physicals (2 per worker)	42	EA	600.00	25	--
b) Protective equip. - Level C (20)	6,600	MD	63.25	417	Purchase and disposal of protective equipment
c) Protective equip. - Level D (1)	330	MD	26.45	9	Purchase and disposal of protective equipment
				<u>5,905</u>	
<b>AER FACILITY OPERATING AND MAINTENANCE COST SUBTOTAL</b>					
Contingencies (20%)	--	--	--	1,181	Factor applied to subtotal
General administration (13%)	--	--	--	921	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	<u>641</u>	Factor applied to subtotal + contingencies and GA
				<u>8,648</u>	
<b>CATEGORY 4: TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS</b>					
<b>TOTAL ESTIMATED COST</b>				<b>14,333</b>	

APPENDIX Y, EXHIBIT 2. DETAILED COST ESTIMATE FOR 11,200 TON INTERIM SOIL STORAGE FACILITY

Item	Quantity	Cost Units	Unit Price (\$1000)	Comments
<b>COMMON REMEDIAL COSTS</b>				
<b>GENERAL</b>				
Mobilization and demobilization	1	LS	5%	--
Construction admin. trailer	2	MO	270.00	--
Utilities connection	1	LS	5,000.00	--
Water supply	1	LS	5,000.00	--
<b>SITE PREPARATION</b>				
Clear, strip and grub	3,000	SY	1.50	Storage for bulk waste pile of 11,200 tons of soil
Grading and gravel surfacing	3,000	SY	5.50	--
Ditching for site runoff	800	LF	0.56	--
Access roads	350	SY	5.70	20 ft wide with distance of 150 ft to AER facility
<b>CONTAINMENT BASE</b>				
Geotextile	2,844	SY	1.85	--
45 Mil. Hypalon liner	2,244	SY	6.75	--
Geotextile	2,844	SY	1.85	--
12-inch leak detection gravel	948	CY	10.95	--
4-inch perforated PVC piping	622	LF	14.00	--
8-inch PVC Piping	333	LF	20.00	--
8-inch reinforced concrete	632	CY	150.00	--
Impervious layer	2,844	SY	11.70	--
Geotextile	2,844	SY	1.85	--
12-inch drainage gravel	948	CY	10.95	--
4-inch perforated PVC piping	622	LF	14.00	--
8-inch PVC piping	333	LF	20.00	--
Geotextile	2,844	SY	1.85	--

APPENDIX Y, EXHIBIT 2. (continued)

Item	Quantity	Cost Units	Unit Price	(\$1000)	Comments
<b>CONTAINMENT SIDEWALLS</b>					
Impervious layer	400	SY	11.70	5	
10-inch reinforced concrete wall	100	CY	400.00	40	5 ft high concrete wall around perimeter of wastepile
Vertical drain	400	SY	6.00	2	--
4-inch perforated PVC pipe	640	LF	14.00	9	Around perimeter of wall
Waterproofing panels	400	SY	6.00	2	--
<b>CONTAINMENT ENCLOSURE</b>					
Pre-engineered steel building	25,600	SF	18.00	461	Steel frame building, assumed eave height 30 ft
Lighting and electrical	25,600	SF	0.50	13	--
HVAC	1	LS	40,000.00	40	--
Double air-lock doors	1	LS	60,000.00	60	--
<b>BUILDING CLEANING AND RESTORATION</b>					
12-inch drainage gravel disposal	948	CY	350.00	332	Decontaminate and reuse building instead of demolition
Impervious layer disposal	2844	SY	36.00	102	Dispose of gravel at pretreatment and AER facility
Decontaminate building	25,600	SF	6.00	154	--
<b>OPERATION AND MAINTENANCE</b>					
Utilities	6	MO	1,000.00	6	--
Soil rehandling	11,200	CY	10.00	112	--

APPENDIX V, EXHIBIT 2. (concluded)

<u>Item</u>	<u>Quantity</u>	<u>Cost Units</u>	<u>Unit Price</u>	<u>(\$1000)</u>	<u>Comments</u>
<u>CONSTRUCTION SUBTOTAL</u>				1,672.5	
Contingencies (25%)	--	--	--	481.1	Factor applied to subtotal
General administration (13%)	--	--	--	271.8	Factor applied to subtotal and contingencies
Contractor fee (8%)	--	--	--	194.0	Factor applied to subtotal and contingencies and GA
<u>TOTAL INTERIM STORAGE FACILITY COST</u>				<u>2,619.3</u>	

APPENDIX Y, EXHIBIT 3. DETAILED COST ESTIMATE FOR IDLE AER OPERATIONS FOR 6 MONTHS

Item	Quantity	Cost Units	Unit Price (\$1000)	Comments
<u>AER FACILITY OPERATING AND MAINTENANCE COSTS</u>				
GENERAL				
Second startup & shakedown	1	LS	15,000.00	--
AER equipment and facility decon	1	LS	10,000.00	--
Equipment use charge	6	MO	111,000.00	666
STANDBY O&M COSTS				
Labor at AER facility				
Operations manager (1)	6	MO	6,532.48	51
Maintenance personnel (2)	12	MO	4,614.29	55
Reactor operator (1)	6	MO	4,477.60	27
Per diem and general expense	165	OY	201.52	33
Rental equipment remaining onsite				
Office trailer	6	MO	379.50	2
Employee trailer - breakroom	6	MO	379.50	2
Excavation admin trailer	6	MO	270.00	2
Decor. trailer rental (2)	12	MO	1,000.00	12
Utilities	6	MO	1,000.00	6
<u>OPERATING AND MAINTENANCE COST SUBTOTAL</u>				881
Contingencies (20%)				176
General administration (13%)				137
Contractor fee (8%)				96
<u>TOTAL COST DURING 6-MONTH IDLE OPERATIONS</u>				1,290

APPENDIX Y, EXHIBIT 4. DETAILED COST ESTIMATE FOR AER TREATMENT OF 40,000 TONS OF SOIL

Item	Quantity	Cost Units	Unit Price (\$1000)	Comments
<b>CATEGORY 1: COMMON REMEDIAL COSTS</b>				
<b>MOBILIZATION</b>				
General mobilization and demobilization	1	LS	130,000	5% of Category 1 subtotal cost
Trial burn	1	LS	150,000	--
Community relations	1	LS	40,000	--
<b>CONSTRUCTION</b>				
Ancillary buildings and equipment:				
a) Water treatment facility	1	LS	300,000	GAC Facility, 25 gpm (50 gpm peak) for decon, runoff, quench
b) Office trailer	23	MO	380	50 ft x 10 ft trailer
c) Employee trailer - breakroom	23	MO	380	50 ft x 10 ft trailer
d) Forklift for material preparation	23	MO	1,561	--
Utility upgrade:				
a) Electrical services construction	1	LS	68,000	\$58,000 for main facility, \$10,000 for excavation site area
b) Outdoor lights	5	EA	1,300	--
c) Water supply allowance	1	LS	20,000	--
d) Sewer connection	1	LS	30,000	--
e) Natural gas line	1	LS	30,000	Fuel to heat soil dryer
f) Telephone service allowance	1	LS	10,000	--
Excavation site buildings:				
a) Decontamination trailer Rental (2)	46	MO	1,000	--
b) Vehicle decontamination station	1	LS	30,000	--

APPENDIX V, EXHIBIT 4. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>EXCAVATION LOAD AND HAUL</b>					
Equipment:					
a) Backhoe rental	23	MO	7,100	163	--
b) Tractor (with hoe ram) rental	23	MO	3,000	69	--
c) Rolloff truck rental (2)	46	MO	1,984	91	--
d) Rolloff boxes for operations	3	EA	5,000	15	3 for daily operation
e) Rolloff boxes for untreated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net volume, total storage for 7 days of operation
f) Rolloff boxes for treated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net volume, total storage for 7 days of holding
Labor:					
a) Backhoe operator	3,360	HR	26	87	8 hr/day for 420 days
b) Tractor operator	3,360	HR	26	87	8 hr/day for 420 days
c) Rolloff truck driver (2)	6,720	HR	26	174	8 hr/day for 420 days for each driver
d) Excavation foreman	3,360	HR	27	91	8 hr/day for 420 days
<b>HEALTH AND SAFETY</b>					
Health and safety personnel (2)	6,720	HR	25	168	8 hr/day for 420 days each worker
Per diem and general expenses of H&S personnel	420	DY	100	42	25% of H&S personnel cost
Physicals (2/worker)	16	EA	600	10	--
Training	8	EA	1,100	9	--
Protective equipment Level C	2,520	MD	63	159	Purchase and disposal of protective equipment
Level D	840	MD	26	22	Purchase and disposal of protective equipment for truck drivers
Dust control	23	MO	1,980	46	--

APPENDIX Y EXHIBIT 4. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>SITE RESTORATION</b>					
Spreading treated soil:					
a) Dump truck	23	MO	1,984	46	--
b) Truck driver	3,360	HR	26	89	8 hr/day for 420 days
Regrade site	84,700	SY	0.81	68	880 ft x 220 ft
Topsoil placement	3,585	CY	13.50	48	6-inch depth x 880 ft x 220 ft
Clean fill	2,055	CY	7.00	14	15% of 37,600 yd <sup>3</sup> less top soil placement
Erosion matting	84,700	SY	0.80	68	--
Seeding and revegetation	84,700	SY	1.40	119	--
Salvage of rolloff boxes	59	EA	- 2,500	- 148	50% of cost, 3 operations, 28 untreated soil, and 28 treated soil
<b>COMMON REMEDIAL COST SUBTOTAL</b>				<b>2,731</b>	
Contingencies (25%)	--	--	--	683	Factor applied to subtotal
General administration (13%)	--	--	--	444	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	309	Factor applied to subtotal + contingencies and GA
<b>CATEGORY 1: TOTAL COMMON REMEDIAL COST</b>				<b>4,167</b>	
<b>CATEGORY 2: COMMON OPERATING AND MONITORING COSTS</b>					
<b>GENERAL</b>					
On-scene coordinator	23	MO	5,500	127	\$25/hr x 8 hr/day x 27.5 day/mo.
Per diem and general expense	640	DY	50	32	25% of labor cost
<b>MONITORING AND ANALYSIS PROGRAM</b>					
Onsite lab facility	1	LS	250,000	250	Includes enclosure, high resolution GC/MS, ancillary equipment
<b>Analytical Lab Operation:</b>					
a) Chemists (4)	92	MO	8,333	767	
b) Per diem and general expense	640	DY	300	192	25% of labor cost
c) Lab expendables and miscellaneous	23	MO	800	18	--



APPENDIX Y, EXHIBIT 4. (Continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>Ambient air monitoring:</b>					
a) Residential stations	4	EA	30,000	120	High volume air samplers
b) Background database	1	LS	20,000	20	--
c) Air sampling analysis	23	MO	4,000	92	--
<b>Sampling collection:</b>					
a) Sampling technicians (2)	46	MO	4,888	225	--
b) Per diem and general expense	640	UY	58	56	25% of labor cost
c) Expensables and miscellaneous	23	MO	800	18	--
<b>FACILITY OPERATIONS</b>					
<b>Water Treatment Operation:</b>					
a) General	23	MO	550	13	\$20/day x 27.5 day/mo.
b) Carbon replacement	31,000	lbs	2.00	62	1 lb carbon/1,000 gal, 31,000,000 gal; decon, quench, runoff
c) Facility operator	5,064	HR	25.5	129	8 hr/day for 633 days
d) Discharge to POTW	31,000	1000 gal	1.20	37	
e) Maintenance (misc)	1	LS	30,000	30	10% of facility capital cost
Water User Charge	9,115	KGAL	0.80	7	10 gpm x 60 min/hr x 24 hr/day x 633 days; decon and ancil facility
Electricity	23	MO	3,168	73	100 kW x 24 h/d x 27.5 d/mo. x \$0.048/kWh
<b>COMMON OPERATING AND MONITORING COST SUBTOTAL</b>				2,268	
Contingencies (20%)	--	--	--	454	Factor applied to subtotal
General administration (13%)	--	--	--	354	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	246	Factor applied to subtotal + contingencies and GA
<b>CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST</b>				<u>3,322</u>	

APPENDIX Y, EXHIBIT 4. (Cont'd)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<u>CATEGORY 3: AER FACILITY SITE SETUP/REMOVAL COSTS</u>					
transportation trailers to and from site	27	TRL	6,000	162	Transport from Borger, TX to Gulfport, MS and back
Erect AER and pretmt building	972	TON	125	122	27 modules @ 72,000 lb or 36 tons each
Startup/shakedown	1	LS	15,000	15	--
Remove AER and pretmt building	972	TON	100	97	27 modules @ 72,000 lb or 36 tons each
<u>FACILITY SITE SETUP/REMOVAL COST SUBTOTAL</u>				396	
Contingencies (25%)	--	--	--	99	Factor applied to subtotal
General administration (13%)	--	--	--	64	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	45	Factor applied to subtotal + contingencies and GA

CATEGORY 3: TOTAL AER SITE SETUP/REMOVAL COSTS 604

CATEGORY 4: AER FACILITY OPERATING AND MAINTENANCE COSTS

<u>EQUIPMENT USE CHARGE</u>	24	MO	111,000.00	2,664	Pretreatment and AER equipment including transportation time
<u>OPERATION OF AER FACILITY</u>					
<u>Utilities:</u>					
AER power requirements	40,000	TON	52.80	2,112	1100 kWh/ton x \$.048/kWh
Ancillary power requirements	23	MO	6,336.00	146	200 kWh x 24h/d x 27.5d/mo x .048/kWh
Nitrogen	1,300,000	100 ft <sup>3</sup>	0.40	760	200 scfm use rate during operation or 4750 ft <sup>3</sup> /ton
Water user charge	10,027	1000	0.80	8	11 gpm for ash quenching x 60 min/h x 24 h/d x 633 days
Natural gas	48,000	1000 ft <sup>3</sup>	5.00	240	3000 cf/h use rate during operation or 1200 ft <sup>3</sup> /ton

APPENDIX Y EXHIBIT 4. (concluded)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
Maintenance of AER:					
Electrode replacement	40,000	TON	1.25	50	Replace @ 20,000 tons/electrode
Core replacement	40,000	TON	25.00	1,000	Replace @ 2,000 tons/core
Miscellaneous materials	40,000	TON	6.00	240	3% of equipment capital cost
Baghouse and carbon replacement	40,000	TON	2.00	80	
Labor at AER facility:					
Operations manager (1)	23	MO	8,532.48	196	Salary @ \$48000/y x burden
Secretary (1)	23	MO	2,844.16	65	Salary @ \$16000/y x burden
Maintenance personnel (2)	46	MO	4,614.29	212	Salary @ \$25958/y x burden
Reactor operator (4)	92	MO	4,477.60	412	Salary @ \$25189/y x burden
Yard operator (4)	92	MO	4,204.02	387	Salary @ \$23650/y x burden
Pretreatment area operator (4)	92	MO	4,340.90	399	Salary @ \$24420/y x burden
Relief operator (2)	46	MO	4,340.90	200	Salary @ \$24420/y x burden
Engineers/safety personnel (3)	69	MO	6,754.88	466	Salary @ \$38000/y x burden
Per diem and general expense	633	DY	924.02	584	25% of total labor cost for 21 employees
Health and safety program:					
Physicals (2 per worker)	42	EA	600.00	25	--
Protective equip. - Level C (20)	12,650	MD	63.25	800	Purchase and disposal of protective equipment
Protective equip. - Level D (1)	633	MD	26.45	17	Purchase and disposal of protective equipment
<b>AER FACILITY OPERATING AND MAINTENANCE SUBTOTAL COST</b>				<b>11,063</b>	
Contingencies (20%)	--	--	--	2,213	Factor applied to subtotal
General administration (13%)	--	--	--	1,726	Factor applied to subtotal and contingencies
Contractor fee (8%)	--	--	--	1,200	Factor applied to subtotal and contingencies and GA
<b>TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS</b>				<b>16,202</b>	
<b>TOTAL ESTIMATED COST</b>				<b>24,295</b>	

APPENDIX Y, EXHIBIT 5. DETAILED COST ESTIMATE FOR AER TREATMENT OF 10,000 TONS OF SOIL

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>CATEGORY 1: COMMON REMEDIAL COSTS</b>					
<b>MOBILIZATION</b>					
General mobilization and demobilization	1	LS	79,000	79	5% of Category 1 subtotal cost
Trial burn	1	LS	150,000	150	--
Community relations	1	LS	40,000	40	--
<b>CONSTRUCTION</b>					
Ancillary buildings and equipment:					
a) Water treatment facility	1	LS	300,000	300	GAC facility, 25 gpm (50 gpm peak) for decon, runoff, quench
b) Office trailer	7	M0	380	3	50 ft x 10 ft trailer
c) Employee trailer - breakroom	7	M0	380	3	50 ft x 10 ft trailer
d) Forklift for material preparation	7	M0	1,581	11	--
Utility Upgrade:					
a) Electrical services construction	1	LS	68,000	68	\$58,000 for main facility, \$10,000 for excavation site area
b) Outdoor lights	5	EA	1,400	7	--
c) Water supply allowance	1	LS	20,000	20	--
d) Sewer connection	1	LS	30,000	30	--
e) Natural gas line	1	LS	30,000	30	Fuel to heat soil dryer
f) Telephone service allowance	1	LS	10,000	10	--
Excavation site buildings:					
a) Decontamination trailer Rental (2)	16	M0	1,000	16	--
b) Vehicle decontamination station	1	LS	30,000	30	--

APPENDIX Y, EXHIBIT 5. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>EXCAVATION LOAD AND HAUL</b>					
<b>Equipment:</b>					
a) Backhoe rental	7	MO	7,100	50	--
b) Tractor (with hoe ram) rental	7	MO	3,000	21	--
c) Rolloff truck rental (2)	14	MO	1,984	28	--
d) Rolloff boxes for operations	3	EA	5,000	15	3 for daily operation
e) Rolloff boxes for untreated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net vol, total storage for 7 days of operation
f) Rolloff boxes for treated soil storage	28	EA	5,000	140	12 yd <sup>3</sup> net vol, total storage for 7 days of holding
<b>Labor:</b>					
a) Backhoe operator	1,040	HR	26	27	8 hr/day for 130 days
b) Tractor operator	1,040	HR	26	27	8 hr/day for 130 days
c) Rolloff truck drivers (2)	2,080	HR	26	54	8 hr/day for 130 days for each driver
d) Excavation foreman	1,040	HR	27	28	8 hr/day for 130 days
<b>HEALTH AND SAFETY</b>					
Health and safety personnel (2)	2,080	HR	25	52	8 hr/day for 130 days each worker
Per diem and general expense for H&S personnel	130	DY	100	13	25% of H&S personnel cost
Physicals (2/worker)	16	EA	600	10	--
Training	8	EA	1,100	9	--
Protective Equipment Level C (6 sets)	780	MD	63	49	Purchase and disposal of protective equipment
Level D (2 sets)	260	MD	26	7	Purchase and disposal of protective equipment for truck drivers
Dust Control	7	MO	1,980	14	--

APPENDIX 1, EXHIBIT 5. (continued)

Item	Quantity	Cost Units	Unit Price	(\$1000)	Comments
<b>SITE RESTORATION</b>					
Spreading treated soil:					
a) Dump truck	7	MO	1,584	14	--
b) Truck driver	1,040	HR	26	27	8 hr/day for 130 days
Regrade site	84,700	SY	0.81	69	880 ft x 220 ft
Topsoil replacement	1,410	CY	13.50	19	15% of 9,400 yd <sup>3</sup>
Erosion matting	84,700	SY	0.80	68	--
Seeding and revegetation	84,700	SY	1.40	119	--
Salvage of rolloff boxes	59	EA	- 2,500	- 148	50% of cost, 3 operations, 28 untreated soil, and 28 treated soil
<b>COMMON REMEDIAL COST SUBTOTAL</b>				<b>1,649</b>	
Contingencies (25%)	--	--	--	412	Factor applied to subtotal +
General administration (13%)	--	--	--	268	contingencies
Contractor fee (8%)	--	--	--	186	Factor applied to subtotal +
<b>CATEGORY 1: TOTAL COMMON REMEDIAL COST</b>				<b>2,515</b>	contingencies and GA
<b>CATEGORY 2: COMMON OPERATING AND MONITORING COSTS</b>					
<b>GENERAL</b>					
On-scene coordinator	7	MO	5,500	39	\$25/hr x 8 hr/day x 27.5 day/mo.
Per diem and general expense	192	DY	50	10	25% of labor cost
<b>MONITORING AND ANALYSIS PROGRAM</b>					
Onsite lab facility	1	LS	250,000	250	Includes enclosure, high resolution GC/MS, ancillary equipment
Analytical Lab Operation:					
a) Chemists (4)	28	MC	8,333	233	
b) Per diem and general expense	192	DY	303	58	25% of labor cost
c) Lab expendables and miscellaneous	7	MO	800	6	--

APPENDIX Y, EXHIBIT 5. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>Ambient air monitoring:</b>					
a) Residential stations	4	EA	30,000	120	High-volume air samplers
b) Background database	1	LS	20,000	20	--
c) Air sampling analysis	7	MO	4,000	28	--
<b>Sampling Collection:</b>					
a) Sampling technicians (2)	14	MO	4,888	68	--
b) Per diem and general expense	192	DAY	88	17	25% of labor cost
c) Expendables and miscellaneous	7	MO	800	6	--
<b>FACILITY OPERATIONS</b>					
<b>Water treatment operation:</b>					
a) General	7	MO	550	4	\$20/day x 27.5 day/mo.
b) Carbon replacement	10,000	lbs	2.00	20	1 lb carbon/1000 gal, 10,000,000 gal; decon, quench, runoff
c) Facility operator	1,544	HR	25.5	39	8 hr/day for 193 days
d) Discharge to POTW	10,000	1000 gal	1.20	12	
e) Maintenance (misc)	1	LS	30,000	30	10% of facility capital cost
Water User Charge	2,779	1000 gal	0.80	2	10 gpm x 60 min/hr x 24 hr/day x 193 days; decon and ancil facility
Electricity	7	MO	3,168	22	100 kW x 24 h/d x 27.5 d/mo. x \$0.048/kWh
<b>COMMON OPERATING AND MONITORING COST SUBTOTAL</b>				<u>984</u>	
Contingencies (20%)	--	--	--	197	Factor applied to subtotal
General administration (13%)	--	--	--	154	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	107	Factor applied to subtotal and contingencies and GA
<b>CATEGORY 2: TOTAL COMMON OPERATING AND MONITORING COST</b>				<u>1,44</u>	

APPENDIX 1, EXHIBIT 5. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<u>CATEGORY 3: AER FACILITY SITE SETUP/REMOVAL COSTS</u>					
Transportation trailers to and from site	27	TRL	6,000	162	Transport from Borger, TX to Gulfport, MS and back
Erect AER and pretmt building	972	TON	125	122	27 modules @ 72,000 lb or 36 tons each
Startup/shakedown	1	LS	15,000	15	--
Remove AER and pretmt building	972	TON	100	97	27 modules @ 72,000 lb or 36 tons each
<u>FACILITY SITE SETUP/REMOVAL COST SUBTOTAL</u>					
				396	
Contingencies (25%)	--	--	--	97	Factor applied to subtotal
General administration (13%)	--	--	--	64	Factor applied to subtotal + contingencies
Contractor fee (8%)	--	--	--	45	Factor applied to subtotal and contingencies and GA
<u>CATEGORY 3: TOTAL AER SITE SETUP/REMOVAL COSTS</u>					
				604	
<u>CATEGORY 4: AER FACILITY OPERATING AND MAINTENANCE COSTS</u>					
<u>EQUIPMENT USE CHARGE</u>					
	8	MO	111,000.00	888	Pretreatment and AER equipment, includes transportation time
<u>OPERATION OF AER FACILITY</u>					
<u>Utilities:</u>					
a) AER power requirements	10,000	TON	52.80	528	1100 kWh/ton x \$.048/kWh
b) Ancillary power requirements	7	MO	6,336.00	44	200 kWh x 24 h/d x 27.5d/mo x \$.048/kWh
c) Nitrogen	475,000	100 ft <sup>3</sup>	0.40	190	200 scfm use rate during operation or 4750 ft <sup>3</sup> /ton
d) Water user charge	3,057	1000 GAL	0.80	2	11 gpm for ash quenching x 60 min/h x 24 h/d x 193 days
e) Natural gas	12,000	1000 ft <sup>3</sup>	5.00	70	3000 cf/h use rate during operation or 1200 ft <sup>3</sup> /ton



APPENDIX Y, EXHIBIT B. (continued)

Item	Quantity	Units	Unit Price	Cost (\$1000)	Comments
<b>Maintenance of AER:</b>					
a) Electrode replacement	10,000	TON	1.25	13	Replace @ 20,000 tons/electrode
b) Core replacement	10,000	TON	25.00	250	Replace @ 2,000 tons/core
c) Miscellaneous materials	10,000	TON	6.00	60	3% of equipment capital cost
d) Baghouse and carbon replacement	10,000	TON	2.00	20	--
<b>Labor at AER facility:</b>					
a) Operations manager (1)	7	MO	8,532.48	60	Salary @ \$48000/y x 202% overhead x 5.6% G&A
b) Secretary (1)	7	MO	2,814.16	20	Salary @ \$16000/y x 202% overhead x 5.6% G&A
c) Maintenance personnel (2)	14	MO	4,614.29	65	Salary @ \$25958/y x 202% overhead x 5.6% G&A
d) Reactor operator (4)	28	MO	4,477.60	125	Salary @ \$25189/y x 202% overhead x 5.6% G&A
e) Yard operator (4)	28	MO	4,204.02	118	Salary @ \$23650/y x 202% overhead x 5.6% G&A
f) Pretreatment area operator (4)	28	MO	4,340.90	122	Salary @ \$24420/y x 202% overhead x 5.6% G&A
g) Relief operator (2)	14	MO	4,340.90	61	Salary @ \$1420/y x 202% overhead x 5.6% G&A
h) Engineers/safety personnel (3)	21	MO	6,754.88	142	Salary @ \$38000/y x 202% overhead x 5.6% G&A
i) Per diem and general expense	193	OY	925.97	178	25% of total labor cost for 21 employees
<b>Health and safety program:</b>					
a) Physicals (2 per worker)	42	EA	600.00	25	--
b) Protective equip. - Level C (20)	3,860	MD	63.25	244	Purchase and disposal of protective equipment
c) Protective equip. - Level D (1)	193	MD	26.45	5	Purchase and disposal of protective equipment

APPENDIX Y, EXHIBIT 5. (concluded)

<u>Item</u>	<u>Quantity</u>	<u>Units</u>	<u>Unit Price</u>	<u>Cost (\$1000)</u>	<u>Comments</u>
<u>AER FACILITY OPERATING AND MAINTENANCE SUBTOTAL COST</u>					
Contingencies (20%)	--	--	--	3,230	
General administration (13%)	--	--	--	646	Factor applied to subtotal
Contractor fee (8%)	--	--	--	504	Factor applied to subtotal + contingencies
				350	Factor applied to subtotal + contingencies and GA
<u>CATEGORY 4: TOTAL AER FACILITY OPERATING AND MAINTENANCE COSTS</u>				<u>4,730</u>	
<u>TOTAL ESTIMATED COST</u>				<u>9,291</u>	

APPENDIX Z

UPDATED COST ESTIMATE FOR THE J. M. HUBER METHOD OF  
PROCESSING TOXIC WASTE AT THE GULFPORT, MISSISSIPPI, SITE

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**INTEROFFICE CORRESPONDENCE**

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Date: March 20, 1987  
To: R. W. Thomas  
From: H. J. Welland *HJW*  
Subject: UPDATED COST ESTIMATE FOR THE J. M. HUBER METHOD OF PROCESSING TOXIC WASTE AT THE GULFPORT, MISSISSIPPI SITE - HJW-13-87

Cost Estimating has updated the cost estimate for the J. M. Huber Corp. Category 1, Common Remedial Costs and Category 2, Common Operating and Maintenance Costs for processing the toxic waste located at the Gulfport, Mississippi site. A copy of the assumptions stated in the original transmittal letter (K. A. Jenjum to R. W. Thomas letter dated June 18, 1986) is included for information. The changes considered in this update are:

1. The size of the excavation area and the associated regrading costs were revised to agree with the latest contaminated volume and depth estimates.
2. Per diem expenses have been added for the Chemists and the On Scene Coordinator because it was felt it would be more efficient if trained personnel were brought to the job site rather than trying to find or train local personnel.
3. The full time decontamination worker has been deleted because only minor, sporadic decontamination will be required during the excavation, which the equipment operator can perform. The major decontamination effort will occur at the conclusion of the excavation effort. The original estimate assumed that the excavation personnel would participate in the final decontamination effort, thus full-time decontamination personnel are not required.
4. The costs for general mobilization have been revised to 95 percent of the Category 1 subtotal costs.
5. A contractor fee of 8 per cent has been added.

The detailed backup sheets have been revised and are attached to this letter.

hjl

Attachments:  
As Stated

cc: Central Files  
Project File #8076-A  
H. J. Welland Letter File

353

*"Providing research and development services to the government"*

Assumptions taken from letter from K. A. Henjum to R. W. Thomas, dated June 18, 1986:

1. Facility/equipment site development can take place as a normal construction operation. Since the facility/equipment site is in a clean area, there is no danger of contamination spread to workers as long as dust-creating activities are not occurring on the excavation site. Unit prices for equipment area and yard floor slabs need not reflect added labor costs due to personal-protective clothing effects on workers, and thus were lowered.
2. Excavation site preparation was eliminated. The excavation site has been stabilized with soil cement and additional fill material. Grasses have grown through the surface of the site. It is assumed that the ground is capable of supporting heavy equipment, thus eliminating the need for on-site roads. Contaminated soil on the surface of the site cannot be removed by clearing and grubbing as is the normal method of removing grasses from an excavation site because the removed soil would constitute a hazardous waste. It is assumed that the soil pretreatment facility can handle the amount of grass excavated from the site. The excavation site is very flat with ditches on site to control water flow on the site. These ditches are monitored for contamination. Further ditching for site run on will be unnecessary.
3. Soil stabilization occurs to a depth of six to twelve inches on site. Contamination in this soil is expected to reach a maximum depth of approximately 1 1/2 feet. This soil will need to be broken up before it can be excavated. A tractor, such as a Case 580 with a hoe ram attachment, is suggested for this work. The hoe ram could break up sufficient soil for one day's excavation, leaving the rest of the site intact. In this way, the soil stabilization can be taken advantage of to minimize the spread of contaminated dust from the excavation site while maintaining a surface for hauling the excavated soil offsite.
4. There is one vehicle decontamination station. This station will be capable of decontaminating the equipment which will be removed from the site at the end of the project.
5. Excavation will be done five days a week in an eight-hour day. It is assumed that loading of soil will occur in a five-hour period. Assume that 220 days will be available to work, allowing for storms, holidays and equipment down time. Excavation equipment is oversized to allow for doubling the daily requirement of the treatment facility to allow the soil supply to catch up after periods of depletion caused by excavation down time.

6. Excavation crew rates were lowered to reflect DOE and Davis-Bacon labor rates for that area. Per diem and general expense allowance of 25 percent of labor costs was included in the labor hourly rates. The rate using local operating engineers and training them within the handling of hazardous waste is a minimum of 14 percent less costly than using a hazardous waste trained operating engineer and paying him per diem.
7. Mapping of the excavation site contamination has been performed to provide a preliminary scope for excavation. For the purpose of this estimate, it was assumed that the depth of the base case excavation area will double for twice the base soil volume or will be halved for one half of the base soil volume. This assumption is based on the expectation that subsurface contamination will not cover a larger area than the known surface contamination.
8. After treatment, the original soil will be returned to the excavation site. It is expected that fifteen percent of the original soil volume will be lost during treatment. Clean fill top soil of appropriate quality will be used to fill the remaining volume of the excavation site so that the site may be recontoured for its final intended use as a parking area.
9. The federal facility which has the waste site will provide several services. Specifically, these are:
  - o All permitting associated with the work
  - o All licensing and ash delisting procedures
  - o A site warehouse
  - o A site parking area
  - o A prepared area for placement of the treatment facility
10. The costs for bonding and insurance is included in the overhead costs.
11. The estimate does not include costs for footings, foundations or slabs to place the process equipment on. It is assumed that the stabilized soil is adequate for all intended loads.
12. One other area of discrepancy is that CH2M Hill maintains that the soil may not become completely delistable and thus a disposal site must be provided for the treated soil and fill material will be needed for the excavation site. This estimate was prepared on the assumption that the soil will be completely delistable and can be returned to the original excavation site or else nothing will be done and the soil will remain undisturbed in its present state.

Eckman, Inc.

DATE: MAR 10, 67

PLANNING

PROJECT: REVISION TO J. M. MURKIN ESTIMATE FOR

LOCATION: CLEANUP OF WILFORD, MASS. SITE

REQUESTER: F. W. THOMAS

Page 1 of 2

Prep. By: H. J. WELLS

Appr'd By: *[Signature]*

TYPE OF EST. : FLANNING  
 SOURCE : (E) Eng. Est.  
 (V) Vendor  
 (F) Pur. Order  
 (H) Handbook Ref.

NO.	DESCRIPTION	EA. P.H.	MAT'L UNIT	MAT'L COST	UNIT LAB. HRS	TOTAL LAB. RATE	LABOR COST	MAT'L COST	OTHER COST	TOTAL COST
	CONSTRUCTION									
	SEALED & DEPOT		1 LS	95000				95,000		95,000.0
	PAVING		1 CC	150000				150,000		150,000.0
	SECURITY RELATIONS		1 LS	40000				40,000		40,000.0
	CONSTRUCTION									
	WATER TREATMENT FACILITY		1 LS	300000				300,000		300,000.0
	OFFICE TRAILER		12 MO	380				4,560		5,000.0
	EMP TRAILER ROOM		12 MO	380				4,560		5,000.0
	FERTILIZER FOR MAT'L PREF		12 MO	1581				18,972		19,000.0
	UTILITY UPGRADE									
	ELECT SERVICE CONSTRUCTION		1 LS	68000				68,000		68,000.0
	OUTDOOR LIGHTS		5 EA	1400				7,000		7,000.0
	WATER SUPPLY ALLOWANCE		1 LS	20000				20,000		20,000.0
	SEWER CONNECTION		1 LS	30000				30,000		30,000.0
	NATURAL GAS LINE		1 LS	30000				30,000		30,000.0
	TELEPHONE ALLOWANCE		1 LS	10000				10,000		10,000.0
	TOTAL							778092		779,000.0

FORM NO. 100

DATE MAR 16, 87

FLAMING

TYPE OF EST.

PROJECT REGION TO B. P. WHEEL ESTIMATE FOR

Page 2 of 6

SOURCE (E) Eng. Est.

LOCATION CLEBURG OF BUREAU, MISS. SITE

Prep. by: H. J. PELLARD

(V) Vendor

REQUESTER E. W. THOMAS

App'd By: *[Signature]*

(F) For. Order

REQUESTER E. W. THOMAS

ACT. NO.	DESCRIPTION	E.V. P.H.	MAT'L UNIT	MAT'L COST	UNIT LAF. HRS	UNIT LAF. RATE	TOTAL LAB. COST	MAT'L COST	OTHER COST	TOTAL COST
	EXCAVATION SITE SLOPE					0.0	0.0	0.0	0.0	0.0
	SCUM TRUCKER RENTAL (2)		28 MD	1000			0	28,000		28,000.0
	VEHICLE SIGN STATION		1 LS	30000			0	30,000		30,000.0
	EXCAVATION AND BACK EQUIPMENT						0	0	0	0.0
	SPACE RENTAL		12 MD	7100			0	85,200		85,200.0
	TRACTOR (W/SHO AND ATTACH)		12 MD	3000			0	36,000		36,000.0
	ROLLOFF TRUCK RENTAL (2)		24 MD	1984			0	47,616		48,000.0
	ROLLOFF BONES FOR OPER		3 EA	5000			0	15,000		15,000.0
	ROLLOFF BONES FOR W/RT STOR		28 EA	5000			0	140,000		140,000.0
	ROLLOFF BONES FOR TRT STOR		28 EA	5000			0	140,000		140,000.0
	LABOR						0	0	0	0.0
	BACKHOE OPER					1760	26	45760	0	46,000.0
	TRACTOR OPER					1760	26	45760	0	46,000.0
	ROLLOFF TRUCK DRIVER (2)					3520	26	91520	0	92,000.0
	EXCAVATION FOREMAN					1760	27	47520	0	48,000.0
						0		0	0	0.0
						0		0	0	0.0
						0		0	0	0.0
	TOTAL					8800		521816		754,000.0



EG&T 10/20/84

Date Mar 11, 87

FLANNING

TYPE OF EST.

PROJECT RELISSION TO J. M. HARPER ESTIMATE FOR

Page 3 of 6

SURBLE (E) Eng. Est.

LOCATION CLEARING OF 20 ACRES, PAVED SITE

Prep. By: K. J. NEULAND

(F) Pur. Order

REQUESTER S. W. THOMAS

APPROVED BY: *[Signature]*

ACT. NO.	DESCRIPTION	QTY	MAT'L UNIT	MAT'L COST	UNIT LAB. HRS	TOTAL LAB. RATE	LABOR COST	MAT'L COST	OVER COST	TOTAL COST
	HEALTH AND SAFETY				0	0	0	0		0.0
	HEALTH & SAFETY - PERSONNEL				350	25	8750	0		88,000.0
	BASE PIER FOR H.A.S. (50)				200	100	20000	0		22,000.0
	PHYSICALS - (CONCRETE)		16 EA	600	0	0	0	0	9600	10,000.0
	TRAINING		8 EA	1100	0	0	0	0	8800	9,000.0
	PROTECTIVE EQUIP - LEVEL 0 - 6 SETS		1320 EA		0	0	0	0	63160	63,160.0
	PROTECTIVE EQUIP - LEVEL 0 - 2 SETS		440 EA		0	0	0	0	11440	11,440.0
	NOISE CONTROL		12 MO	1984	12	1980	23760	0		24,000.0
	SITE RESTORATION				0	0	0	0		0.0
	SPREAD TREATED SOIL				0	0	0	0		0.0
	DUMP TRUCK RENTAL		12 MO	1984	0	0	0	23,808		24,000.0
	TRUCK DRIVER		1 EA		1760	26	45760	0		46,000.0
	REGRADE SITE		184700 SY	0.81	0	0	0	68,607		69,000.0
	TOPSOIL PLACEMENT		2820 CY	13.5	0	0	0	38,070		38,070.0
	EROSION MATTING		184700 SY	0.8	0	0	0	67,760		68,000.0
	SEEDING & REVEGETATION		184700 SY	1.4	0	0	0	118,580		119,000.0
	SALVAGE ROLLOFF BOXES		59 EA	-2500	0	0	0	(147,500)		(147,500.0)
	TOTAL				5510		14905			444,000.0

8888 JONES, INC.

PROJECT: BEAVERHEAD M. W. WASTE ESTIMATE EOB.  
 LOCATION: CLEAR P. OF BULFOFT, MISS. SITE  
 REQUESTER: R. W. FAYAS  
 TYPE OF EST.: PLANNING  
 SOURCE: TET EFG. EST.  
 (V) Vendor  
 (F) Sur. Order  
 (H) Handbook Ref.  
 Date: MAR 14, 87  
 Page 4 of 6  
 Prep. by: H. J. WELAND  
 Appr'd By: *[Signature]*

ACCT. NO.	DESCRIPTION	E, V, F, H	MAT'L UNIT	MAT'L COST	UNIT LAB. HRS	TOTAL LAB. HRS	LAB. RATE	LABOR COST	MAT'L COST	OTHER COST	TOTAL COST
	COMMON REMEDIAL COSTS SUBTOTAL				0	0	0	0	0	0	0.0
	FROM PAGE 1										779,000.0
	FROM PAGE 2										754,000.0
	FROM PAGE 3										464,000.0
	SUBTOTAL										1,997,000.0
	CONTINGENCY @ 25 %										499,000.0
	GENERAL ADMIN @ 13 %										324,000.0
	CONTRACTOR FEE @ 8 %										226,000.0
	CATEGORY 1 TOTAL COSTS										3,046,000.0
											6.0
											0.0
											0.0
	CAT 2 - COMMON OPER & MONIT COSTS										0.0
	GENERAL										0.0
	ON-SCENE COORDINATOR		12 MO		1	12	5500	66000			66,000.0
	FEEDIER & GEN EXPENSE @ 25 %		320 DY		50					16500	16,500.0
	MONITORING PROGRAM										0.0
	ON-SITE LAB FACILITY		1 LS	250000					250,000		250,000.0
	LAB OPERATION										0.0
	CHEMISTS (4)		48 MO		1	48	8333	399984			399,984.0
	PER DIEM & GEN EXPENSE @ 25 %		330 DY	303						99990	99,990.0
	LAB EXPENDABLES AND MISC		12 MO	800					9,600		9,600.0
											0.0
	TOTAL					60		259800			842,000.0

665a (Rev. 1-65)

PROJECT: TO BE DETERMINED ESTIMATE FOR  
 LOCATION: DEPT OF TRANSPORTATION, MISS. STATE  
 REQUESTER: R. A. TRENGE

TITLE OF EST. : FURNISHING  
 SOURCE: (E) Eng. Est.  
 (F) Vendor  
 (H) Pur. Order  
 (M) Handbook Ref.

Rate: Ref. 14, 87  
 Page 5 of 6  
 Prep. By: M. J. WELLS  
 Appr'd by: *[Signature]*

ACT. NO.	DESCRIPTION	E.V. PAR	MAT'L UNIT	MAT'L COST	UNIT	HRS	TOTAL LAB. HR	LAB. RATE	LABOR COST	MAT'L COST	OTHER COST	TOTAL COST
	APPOINT AIR METERING					0	0		0	0		0.0
	RESIDENTIAL STATIONS		4 EA	30000		0	0		0	120,000		120,000.0
	ENGINEERING DATA BASE		1 LS	20000		0	0		0	20,000		20,000.0
	WATER SAMPLING ANALYSIS		12 MO	4000		0	0		0	48000		48,000.0
	SAMPLING COLLECTION					0	0		0	0		0.0
	ENGINEERING TECHNICIANS (2)		24 MO			1	24	4669	117312	0		117,312.0
	PER DIEM & CAR EXPENSE 25%		33 DY	96		0	0		0	2340		29,040.0
	EXPENSABLES & MISC		12 MO	800		0	0		0	1000		10,000.0
	FACILITY OPERATIONS					0	0		0	0		0.0
	WATER TREATMENT OPERATION					0	0		0	0		0.0
	GENERAL		12 MO	550		0	0		0	6600		7,000.0
	CARBON REPLACEMENT		117000 LB	2		0	0		0	34,000		34,000.0
	FACILITY OPERATOR		1 EA			2640	2640	25.5	67320	0		67,320.0
	DISCHARGE TO FOTH		117000 KGAL	1.2		0	0		0	20,400		20,400.0
	MAINTENANCE		1 LS	30000		0	0		0	30,000		30,000.0
	WATER USER CHARGE		4752 KGAL	0.8		0	0		0	3,872		3,801.6
	ELECTRICITY		12 MO	3168		0	0		0	38,016		38,016.0
						0	0		0	0		0.0
						0	0		0	0		0.0
						0	0		0	0		0.0
						0	0		0	0		0.0
	TOTAL						2664		2664	266217.6		545,000.0

