

Category II QUALITY ASSURANCE PROJECT PLAN

PROJECT TITLE: The U.S. Environmental
Protection Agency
Mobile Incinerator System
Pesticide Preproduction Burn
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PERFORMING ORGANIZATION: Enviroresponse, Inc. - EERU, GSA
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08837
DURATION: February 1988 to September 1988
TYPE OF PROJECT: Work Order under EPA Contract
68-03-3255
SUPPORTING ORGANIZATION: U.S. Environmental Protection
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QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM
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Lab Workplan No: Support: Office of
Pesticides

Task Start Date: May 1998 - Sept 1998
 (for measurement, data gathering, and/or data generation activities)

Project Category: II

QA ID No: SP - 236 - E

Date QAPP Received: _____

Task Title: USEPA Mobile Incinerator System Pesticide Preproduction Burn
(2,4,5-T)

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DISTRIBUTION OF TEST BURN QA PROJECT PLAN

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1.0 PROJECT DESCRIPTION

1.1 OVERVIEW

This project covers pesticide preproduction (delisting) test operations of the U.S. Environmental Protection Agency's Office of Research and Development's Mobile Incinerator System by the Environmental Emergency Response Unit (EERU) at the Denney farm, a site in Barry County in the State of Missouri. The system operation and planned tests are described below. 12

1.2 FACILITY DESCRIPTION AND OPERATION

An objective of the incinerator development and testing project is to demonstrate a mobile thermal oxidation and incineration system that is capable of destroying/detoxifying hazardous and toxic organic materials and cleansing the contaminated soil and debris frequently associated with spills or uncontrolled hazardous waste disposal sites. 11

The Mobile Incineration System design includes three heavy duty semi-trailers equipped with the necessary components for response to remote locations that are accessible by over-the-road equipment. A fourth trailer includes the analytical instrumentation for monitoring the combustion and stack gases generated by the incineration process. A Block Flow Diagram of the system is shown in Figure 1.1. The four trailers are equipped as follows:

Trailer No. 1 is equipped with a control system console, solids feeder, waste feed nozzle and refractory lined rotary kiln. the kiln operates in an excess air mode at temperatures up to 982°C (1800°F) and is capable of providing long residence time for those solid materials that do not burn or volatilize readily, and which might tend to release toxic substances into the gas stream. The kiln has two combustion burners, one firing with ambient combustion air and the second firing with pure oxygen. Solid materials are ram fed into the kiln after being shredded; sludges and viscous fluids are pumped into the kiln. Residual ash and inert solids are removed from the discharge end of the kiln while vaporized and partially burned toxic vapors and gases are carried in exhaust gases through the self-aligning expansion joint ducting into the high temperature cyclone. The high temperature cyclone removes 93% of the dust greater than 20 microns in size. This dust deposits in a drum collector under the cyclone. The gas passes into the secondary combustion chamber on trailer No. 2, where complete detoxification/destruction is achieved.

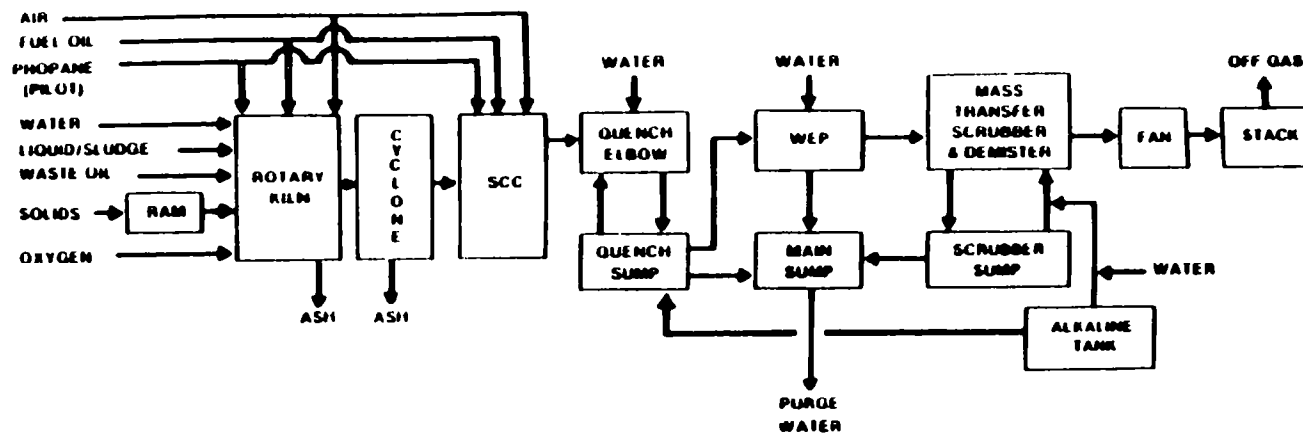


FIGURE 1.1 BLOCK FLOW DIAGRAM OF MOBILE INCINERATION SYSTEM

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Trailer No. 2 is equipped with a refractory-lined Secondary Combustion Chamber (SCC) designed to operate at 1200°C (2200°F) and to maintain a minimum two-second gas stream dwell time with more than 4% oxygen in conformance with the Federal Toxic Substances Control Act (TSCA) regulations covering PCB incineration. The gases leaving the secondary combustion chamber are cooled by quenching in a wetted throat venturi quench elbow. Acid gas removal starts in the quench elbow since the process water contains caustic to neutralize acids. Excess spray drains into a ground-level sump while the cooled flue gases are ducted to the air pollution control equipment. The gas passes into the wet electrostatic precipitator.

Wet Electrostatic Precipitator removes fly ash and sub-micron particulates such as phosphorous pentoxide. It uses a water-wetted, electrostatically charged, fiber reinforced grid as the collection medium. The gas passes into the mass transfer scrubber on trailer No. 3.

Trailer No. 3 is equipped with air pollution control equipment designed to complete the neutralization and removal of acid gases. The gases then pass through a mass transfer (packed bed) scrubber for SO₂, HCl, and other acid gas removal. A demister is provided before the induced draft diesel-engine-driven fan. The fan provides the motive force for gas movement and maintains the negative pressure necessary to avoid toxic fume escape anywhere in the process. The cleaned exhaust leaving the induced draft fan is passed through a sound attenuator and flow straightener before being discharged from the stack.

Trailer No. 4 is equipped with a continuous monitoring system that analyzes the flue and stack gases for combustion components (carbon monoxide (CO), carbon dioxide (CO₂) and oxygen (O₂) and emission components (oxides of nitrogen (NO_x), and total hydrocarbons (THC)). Sulfur dioxide (SO₂) is not monitored because the wastes and fuel oil have low sulfur contents.

Monitoring and recording of flue and stack gas components are accomplished through the use of: (1) a thermal conductivity detector GC for analysis of CO₂ and O₂; (2) a methanizer to flame ionization detector GC for analysis of CO; (3) a flame ionization detector GC for analysis of THC; and (4) a chemiluminescent detector for analysis of NO_x. The analyzer section also has valves and switching to introduce calibration gas (from cylinders) through the sampling/conditioning units

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to the appropriate analyzer. The analyzers are controlled by microprocessors to provide automatic sampling and tabulation of results.

Waste water from the incineration system will be treated by sedimentation and high-pressure filtering. A "bleed" will be collected and held for analysis in two 15,000-gallon tanks. The waste water will be delisted as a hazardous waste by petition to MDNR and EPA Region VII and disposed of under an NPDES permit.

The ash from the rotary kiln drops from the ash discharge chute directly into steel, 55-gallon drums located under the chute. Once a drum is full, the ash discharge chute is closed to allow replacement of drums. Full drums are covered and moved aside on a roller conveyor and allowed to cool to ambient temperature. An empty drum is then placed under the ash discharge chute and the discharge chute is returned to automatic operation.

1.3 TEST PROGRAM DESCRIPTION

The incinerator system has undergone trial burns during which chlorinated liquids and solids were incinerated. Waste feed will be introduced into the mobile incinerator in two matrix forms. A solid matrix form will consist of vermiculite contaminated with 2,4-D, 2,4-DB, 2,4,5-T, and 2,4,5-TP pesticides (herbicides). The feed rate to the incinerator will be approximately 650 lb/hr (600-700 lb/hr). A liquid matrix will consist of 2,4-D, 2,4-DB, 2,4,5-T, and 2,4,5-TP dissolved in heavy naphtha and diesel oil. This liquid will be fed to the incinerator at a rate of 150 lbs/hour. The purpose of this project is to:

Run a series of delisting tests which would exclude from the identification as a hazardous waste by CFR 261.3 the three main byproducts of the MIS generated during the incineration of the pesticide wastes. Separator sludge will not be analyzed since it will be recycled through the process. Thus no separator sludge will be generated. This exclusion will be based upon the analytical results of the wastewater (purge water), and kiln ash/cyclone ash composite.

1.3.1 Test Plan

Waste feeds consist of liquid and solid herbicides whose registrations have been suspended by the EPA due to contamination with trace quantities of dioxin. The solid

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waste is composed of over 99.5% Weedone^R granular weed killer and the liquid waste is composed of over 93% Envert DTR. Remaining materials are other herbicides of similar composition. Compositions of these materials are presented in Table 1-2.

Delisting Tests -- The primary analytical requirements shall be to determine the concentrations of contaminants of the three main byproducts streams during a test burn using methods and procedures that will satisfy the Missouri Department of Natural Resources (MDNR), EPA Region VII and the EPA Office of Solid Waste. The purpose of the test burn is to provide data to verify that the incinerator byproducts meet or exceed the analytical criteria and should therefore be delisted. QA/QC for these tests will meet Category II requirements.

A summary of the analyses and methods required are given in Table 1-1.

Four samples of each matrix will be analyzed. In addition, a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) of each sample matrix is required. The spiking compounds for each method are detailed in Table 1-3. The MS/MSD analyses will be performed on the first set of samples received.

Four replicate eight hour tests are to be completed at the feedrates given above.

Contingency Tests -- Since the cost of maintaining the incinerator in a holding mode is high and the delisting process takes three to four weeks to complete. A second series of four replicate delisting tests will be executed at 75% of the feed rates given above: i.e., 490 lb/hr of solids (450-525 lb/hr) and 115 lb/hr of liquids. The residue streams from these tests will only be analyzed if the residue streams from the first test series fail to meet the delisting criteria.

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TABLE 1-1 SAMPLING AND ANALYSIS SUMMARY FOR ASH, SLUDGE,
AND PURGE WATER DELISTING

This table summarizes the sampling and analytical requirements for delisting of the solid and liquid streams as a hazardous waste. The criteria for delisting presented in this table follow the final rules published in pages 7903-7915 of Vol. 53 No. 48 (March 11, 1988) of the Federal Register.

Sample Matrices: A composite sample of kiln and cyclone ash and a separate sample of purge water.

Sampling Frequencies and Methods:

Sample	Sampling Frequency Per Test	Sampling Method
1. Kiln Ash and cyclone ash	Sample each drum during test run and composite.	S007
2. Purge Water (after carbon filters)	Sample at 2 hour intervals and composite.	S004
3. Purge Water (before carbon filters)	Sample at 2 hour intervals and composite.	S004

Since the purge water before the filters has failed to meet the delisting criteria during previous analyses, the second purge water sample will be analyzed only if the purge water after the carbon filter passes delisting. However, volatile analyses must be preformed before holding times are exceeded.

The following analytical protocol will be followed for each sample:

A. Per 40 CFR 261 Subpart C

	Ash and Solids	Scrubber Waste Liquids
Ignitability	N.A. ¹	N.A. ²
Corrosivity	pH = 2.0-12.5	pH = 2.0-12.5
Reactivity	Not reactive ³	Not reactive ³
EP Toxicity	As per 40 CFR, 261.24, Table 1, and APP. II ³	ICP scan-heavy metals, as per 40 CFR, 261.24, Table 1 ³

B. Analyses to be performed for the following criteria⁵:

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Table 1-1 (cont.)

Toxic Constituent ⁷	Concentration		SW-846 Method		
	Solids	Purge Water			
Dioxins/Dibenzofurans ⁶	5 ppt ⁴	0.002 ppt ⁴	8290*	1	2
Acetone	---	35.3 ppm	8240		
Aldrin	15 ppb	18 ppt	8080	1	2
Benzene	9.7 ppm	44 ppb	8240		
Benzo(a)pyrene	0.43 ppm	27 ppt	8310		
Benzo(b)fluoranthene	1.8 ppm	0.18 ppb	8270/8310 ¹⁰	2	5
Biphenyl	---	15.5 ppm	8270		
Bis-(2-ethylhexyl)phthalate	---	6.1 ppm	8270		
Chlordane	0.37 ppm	0.24 ppb	8080		
Chlorobenzene	---	8.8 ppm	8240		
Trichloromethane (Chloroform)	5.4 ppm	52 ppb	8240		
Chrysene	170 ppm	1.8 ppb	8270/8310 ¹⁰	2	5
2,4-Dichlorophenoxy- acetic acid (2,4-D)	---	3.5 ppm	8150		
Dibenzo(a,h)anthracene	83 ppb	6.0 ppt	8310		5
Dichloromethane (Methylene chloride)	2.4 ppm	42 ppb	8240		
1,3-Dichlorobenzene	---	34 ppm	8270		
1,4-Dichlorobenzene	---	0.66 ppm	8270		
1,2-Dichlorobenzene	---	26.5 ppm	8270		
1,2-Dichloroethane	4.1 ppm	44 ppb	8240		
2,4-Dichlorophenol	480 ppm	0.88 ppm	8270		
Dichlorvos	260 ppm	0.78 ppm	8140		
Diethyl Phthalate	---	4418 ppm	8270		
Disulfaton	23 ppm	16 ppb	8140		
Endosulfan I	310 ppm	20 ppb	8080		
Ethyl benzene	---	35 ppm	8240		
Fluoranthene	---	1.77 ppm	8270		
Fluorene	120 ppm	18 ppb	8270		
Indeno(1,2,3-c,d)pyrene	330 ppm	1.8 ppb	8270/8310 ¹⁰	2	5
Isophorone	---	61.9 ppm	8270		
Chloromethane (Methyl chloride)	---	35.3 ppm	8240		
Methyl parathion	210 ppm	99 ppb	8140		
Naphthalene	---	79.5 ppm	8270		
Nitrosodiphenylamine	130 ppm	63 ppb	8270		
Pentachlorophenol	---	8.8 ppm	8270		
Phenanthrene	150 ppm	18 ppb	8270		
Phenol	---	8.8 ppm	8270		
Polychlorinated biphenyls	0.31 ppm	72 ppt	8080		
Pyrene	---	35 ppm	8270		
Tetrachloroethylene	59 ppm	59 ppb	8240		2
2,3,4,6-Tetrachlorophenol	---	8.8 ppm	8270		
Toluene	---	88.4 ppm	8240		

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Table 1-1 (cont.)

Toxic Constituent	Concentration		SW-846 Method	
	Solids	Purge Water		
2,4,5-TP (Silvex)	110 ppm	88 ppb	8150	
1,2,4-Trichlorobenzene	---	6.2 ppm	8270	1, 2
2,4,5-Trichlorophenol	---	35 ppm	8270	
2,4,6-Trichlorophenol	3.9 ppm	18 ppb	8270	1, 1
2,4,5-Trichlorophenoxy- acetic acid	---	0.88 ppm	8150	
Xylenes (Total)	---	619 ppm	8240	
Metals & Cyanides (EP Toxicity) ³				1, 1
Arsenic ⁸	1.6 ppm	0.44 ppm	6010	
Chromium	1.6 ppm	0.44 ppm	6010	
Lead	1.6 ppm	0.44 ppm	6010	1, 1
Silver	1.6 ppm	0.44 ppm	6010	
Barium	32 ppm	8.8 ppm	6010	
Cadmium	0.3 ppm	0.09 ppm	6010	
Selenium ⁸	0.3 ppm	0.09 ppm	6010	1, 1
Mercury	0.07 ppm	0.02 ppm	7470	
Nickel	16 ppm	4.4 ppm	6010	
Cyanides	6.5 ppm	1.8 ppm	9010	1, 1

* High Resolution GC/High Resolution MS

Footnotes:

- 1 The ash would not be ignitable after having passed through a kiln and having reached approximately 750°C at the time of discharge.
- 2 The scrubber waste liquid (purge water), which is water, is also not considered to be ignitable.
- 3 Analysis will be for the following metals plus total cyanides in the EP toxicity leachate: Arsenic, Barium, Cadmium, Chromium, Lead, Nickel, Selenium, Silver, and Mercury. Nickel is not a EP Toxicity Test requirement.
- 4 Subjected to the constraints given below and calculated as per the following formulae.

A conservative estimate of the level of concern is a weighted sum of the concentration of tetra-, penta-, and hexachlorinated dioxins and -dibenzofuran isomers. The weighting factors are given below, and represent current best estimates of the relative toxicities of the

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Table 1-1 (cont.)

"2,3,7,8-substituted" congeners. In the absence of isomer-specific data, it is prudent to consider all detected isomers in a homologue to be "2,3,7,8-substituted" isomers.

CDD/CDF Isomers and Their Relative Toxicity

POLYCHLORINATED DIBENZO-P-DIOXINS		POLYCHLORINATED DIBENZOFURANS	
Isomer	Relative Toxicity	Isomer	Relative Toxicity
Mono thru Tri CDDs	0		
2,3,7,8-TCDD	1	2,3,7,8-TDCFs	0.1
other TCDD	0.01	other TDCFs	0.001
2,3,7,8-Penta CDDs	0.5	2,3,7,8-Penta CDFs	0.1
other Penta CDDs	0.005	other Penta CDFs	0.001
2,3,7,8-Hexa CDDs	0.04	2,3,7,8-Hexa CDFs	0.01
other Hexa CDDs	0.0004	other Hexa CDFs	0.0001

For the residues to pass the delisting analyses, the equivalent concentration of 2, 3, 7, 8-TCDD as calculated above must be lower than 5 ppt in the waste solids or 0.002 ppt in the purge water or the analyses must achieve non detects at or below the practical quantitation limits (PQLs) given below. The laboratory will achieve these PQLs since failure to achieve them results in failure to pass delisting.

PCDD/PCDF Homologues	Solids	Purge Water
Tetra and Penta = CDD/F	≤ 15	≤ 0.12
Hexa - CDD	≤ 37	≤ 0.30

- 5 All sampling and analysis methods will be defined by SW-846 Test Methods for Evaluating Solid Waste, third Edition 1987 and ASTM methods, where applicable.

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Table 1-1 (cont.)

- 6 PCDD/PCDF analysis includes the quantification of the following dioxin and furan compounds (typical for all sample matrices):
1. 2,3,7,8 tetra, penta and hexa chlorodibenzo-p-dioxins
 2. 2,3,7,8 tetra, penta and hexa chlorodibenzofurans
 3. Total tetra, penta, hexa, hepta, and octa chlorodibenzo-p-dioxins (each total separately)
 4. Total tetra, penta, hexa, hepta, and octa chlorodibenzofurans (each total separately)
- 7 For organic constituents other than dioxins/furans, the constituent must be below the delisting limit or be not detect.
- 8 If arsenic and/or selenium is detected or if the detection limit is above the delisting criteria. The sample will be rerun using Method 7740 for selenium and Method 7060 for arsenic.
- 9 Since the feed materials were commercial household products which were applied to people's lawns, reactivity tests of the residues are not a concern and are not required.
- 10 Method 8310 will be used for purge water to attain greater sensitivity for detecting this analyte. Method 8270 is adequate for the kiln ash/cyclone ash matrix.
- 2
2
1
2
5

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TABLE 1-2 HERBICIDE COMPOSITIONS

<u>Product</u>	<u>Herbicide Concentration (wt)%</u>				<u>Maximum</u> <u>2,3,7,8 TCDD</u>	<u>Main Inert</u> <u>Component (wt) %</u>
	2,4-D	2,4-DB	2,4,5-T	2,4,5-TP	(ppb)	
Weedone Granular Weed Killer	3.22	--	--	1.61	1.52	93% vermiculite
Envert DT	--	18.14	17.71	--	7.83	59% Fuel Oil
Emulsavert 100	11.95	--	11.95	--	30.9	57% Fuel Oil
Emulsamine 2,4,5-T	--	--	34.38	--	9780.	61% Fuel Oil
Emulsaert 248	6.14	--	12.28	--	20.2	67% Fuel Oil
Dinoxol	--	32.45	31.68	--	45 2	36% Fuel Oil
Weedone BK64	--	42.26	20.23	--	20.9	30% Fuel Oil

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TABLE 1-3 QA/QC FOR DELISTING TESTS-SPIKING REQUIREMENTS

1 matrix spike and 1 matrix spike duplicate per sample matrix with the following spiking compounds plus required method blank, surrogate spikes, laboratory control samples, and duplicate analyses:

<u>SW-846 METHODS</u>	<u>SPIKING COMPOUNDS*</u>	
8290	All dioxin/furan homologs	
8150	All delisting compounds using method 8150	
8240	All delisting compounds using method 8240	
8270	The following compounds: 2,4-Dichlorophenol Naphthalene Fluorene 2,4,6-Trichlorophenol Pentachlorophenol Phenanthrene Pyrene 1,2,4-Trichlorobenzene Benzo(a)pyrene Benzo(b)fluoranthene	-5 2
8310	All delisting compounds using Method 8310 in the matrix of interest	1 2
8080	Appropriate Aroclors and Chlordanes, Aldrin, Endosulfan I	
8140	All delisting compounds using method 8140	
6010	Spike all metals at the regulatory level given in Table 1-1.	
7470	Spike mercury at the regulatory level given in Table 1-1.	

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Table 1-3 (cont.)

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METHODS

SPIKING COMPOUNDS*

7060

Spike arsenic at the regulatory
level given in Table 1-1.

9010

Spike cyanides at the regulatory
level given in Table 1-1.

* See Table 1-1 for delisting compounds and specified SW-846
(3rd Edition) methods. For spiking levels, see Table 3-2.

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1.4 TEST SCHEDULES

The intended schedule of operation of the unit during these test runs will be as follows:

Delisting Tests: (week of August 22, 1988)

<u>Activity</u>	<u>Day</u>	<u>Comments</u>
Obtain steady state operation	1	Test Set-up
Perform Run #1 & 2	2	
Perform Run #3 & 4	3	
Perform Run #5 & 6	4	Contingency
Perform Run #7 & 8	5	Contingency

A final report will be issued in September based upon the anticipated schedule for the analytical laboratory to complete their work. Preliminary analytical data will be available two weeks after the preproduction burn.

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2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The overall project organization and reporting relationships are shown in Fig. 2.1. Enviresponse, Inc., the EPA Environmental Emergency Response Unit (EERU) operating contractor, has the total responsibility for the test burns including the sampling of all matrices, QA management of subcontractors, the preparation for shipment, and the shipment of the samples to EPA Region VII Laboratory. -3

Enviresponse will also be responsible for issuing the final report to the appropriate regulatory authorities.

The analytical work will be performed by two laboratories: EPA Region VII and a CLP laboratory. They will be given copies of this QAPP and will be responsible for carrying out analyses according to the QAPP. The scope of the analytical work for each laboratory is presented in Table 2-1. The primary laboratory is EPA Region VII Laboratory. Region VII laboratory will perform all analyses except those for pesticides by SW Method 8140 and will be responsible for: 1 3 2 3

Providing all bottles, labels, field documents, and preservatives for the samples. The bottles will be precleaned by the appropriate procedures.

Dividing samples and shipping them to the appropriate laboratory for analysis.

Analysis of samples within their scope.

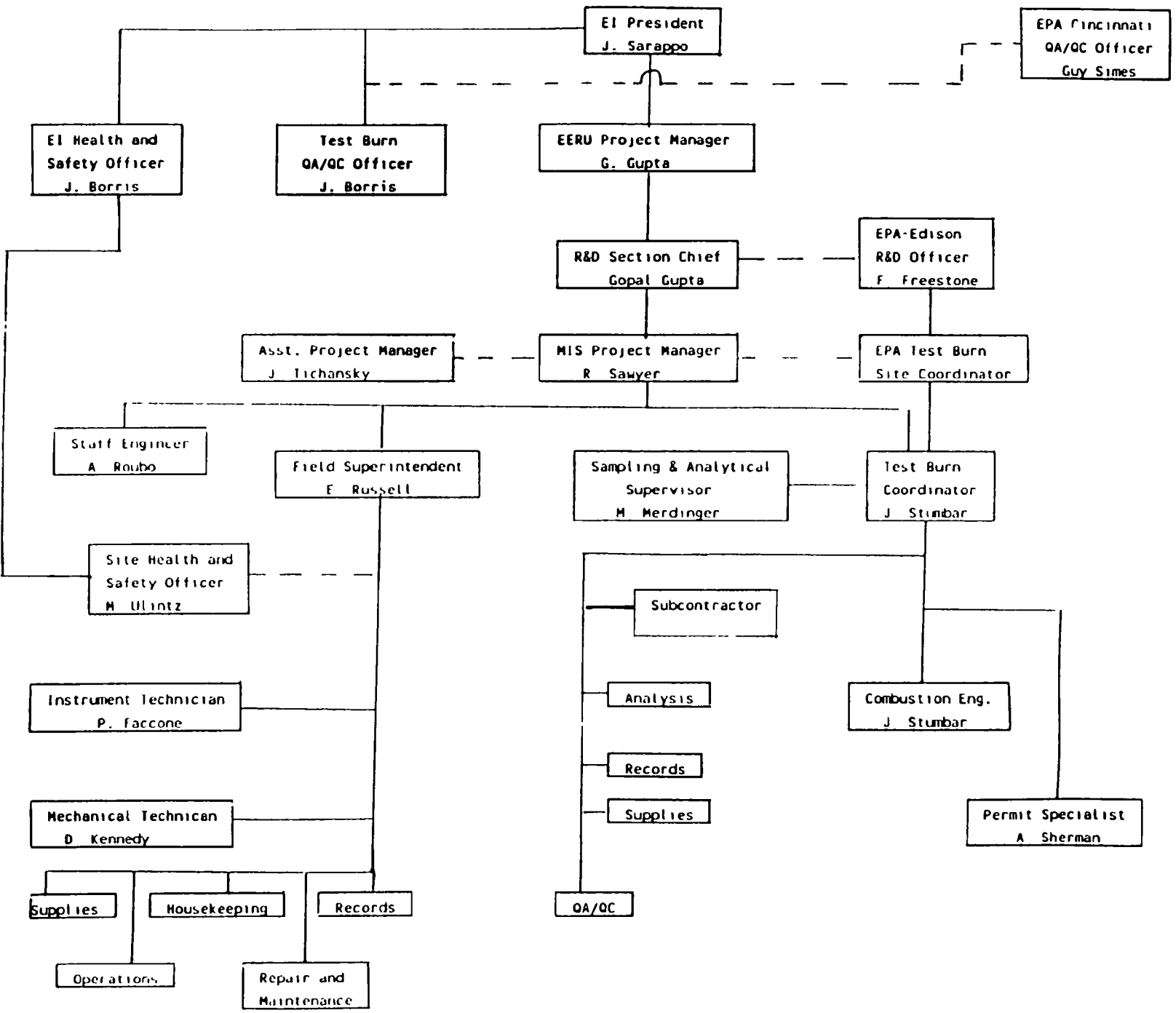
Review, validation and certification of all laboratory data.

The CLP laboratory will be responsible for:

Analysis of all ash and water samples for organo-phosphate pesticides by SW Method 8140 and for polynuclear aromatic hydrocarbons by SW Method 8310. 3 5

Validation of data produced within their laboratory.

Presenting results, including QA/QC results, to Enviresponse and EPA Region VII Laboratories within the required time limits. 1



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FIGURE 2 1 GENERAL PROJECT ORGANIZATION

TABLE 2-1

DIVISION OF LABOR & ANALYTICAL METHODS FOR DELISTING TESTS
DENNEY FARM

MATRIX	PRIORITY	LABORATORY	PARAMETER	ANALYTICAL METHOD QA/QC		Total Samples (REGION VII)	Total Samples (OTHERS)
Kiln Ash/ Cyclone Ash Composite (Solid)	1	REGION VII-ESAT	PCDD/PCDF	8290	MS/MSD	6	
		REGION VII-ESAT	Semivolatiles	8270	MS/MSD	6	
		TBN	PAHs	8310	MS/MSD		6
		REGION VII	Volatiles	8240	MS/MSD	6	
		REGION VII-TAT	Herbicides	8150	MS/MSD	6	
		TBN	Pesticides	8140	MS/MSD		6
		REGION VII	PCBs&Pesticides	8080	MS/MSD	6	
		REGION VII	Heavy Metals	6010	MS/MSD	6	
		REGION VII	Mercury	7470	MS/MSD	6	
		REGION VII	Total Cyanides	9010	MS/MSD	6	
Purge Water filtered (b)	1	Region VII-ESAT	PCDD/PCDF	8290	MS/MSD/BLANK	6	
		Region VII-ESAT	Semivolatiles	8270	MS/MSD/BLANK	6	
		TBN	PAHs	8310	MS/MSD		6
		Region VII	Volatiles	8240	MS/MSD/BLANK	6	
		Region VII-TAT	Herbicides	8150	MS/MSD/BLANK	6	
		TBN	Pesticides	8140	MS/MSD/BLANK		6
		Region VII	PCBs&Pesticides	8080	MS/MSD/BLANK	6	
		Region VII	Heavy Metals	6010	MS/MSD/BLANK	6	
		Region VII	Mercury	7470	MS/MSD/BLANK	6	
		Region VII	Total Cyanides	9010	MS/MSD/BLANK	6	

Continued

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TABLE 2-1 (cont)

DIVISION OF LABOR & ANALYTICAL METHODS FOR DELISTING TESTS
DENNEY FARM

MATRIX	PRIORITY	LABORATORY	PARAMETER	ANALYTICAL METHOD	QA/QC	Total Samples (REGION VII)	Total Samples (OTHERS)
Purge Water unfiltered (b)	2	Region VII-ESAT	PCDD/PCDF	8290	MS/MSD	6	
		Region VII-ESAT	Semivolatiles	8270	MS/MSD	6	
		TBN	PAHs	8310	MS/MSD		6
		Region VII	Volatiles	8240	MS/MSD	6	
		Region VII-TAT	Herbicides	8150	MS/MSD	6	
		TBN	Pesticides	8140	MS/MSD		6
		Region VII	PCBs&Pesticides	8080	MS/MSD	6	
		Region VII	Heavy Metals	6010	MS/MSD	6	
		Region VII	Mercury	7470	MS/MSD	6	
		Region VII	Total Cyanides	9010	MS/MSD	6	
Purge Water unfiltered (Prior to Test)	2	Region VII-ESAT	PCDD/PCDF	8290	(a)	1	
		Region VII-ESAT	Semivolatiles	8270	(a)	1	
		TBN	PAHs	8310	(a)		1
		Region VII	Volatiles	8240	(a)	1	
		Region VII-TAT	Herbicides	8150	(a)	1	
		TBN	Pesticides	8140	(a)		1
		Region VII	PCBs&Pesticides	8080	(a)	1	
		Region VII	Heavy Metals	6010	(a)	1	
		Region VII	Mercury	7470	(a)	1	
		Region VII	Total Cyanides	9010	(a)	1	
Total						152	38

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TABLE 2-1 (cont)

DIVISION OF LABOR & ANALYTICAL METHODS FOR DELISTING TESTS
DENNEY FARM

- NOTES: Number 1 priority to be completed within 14 days of receiving sample.
Number 2 priorities to be completed within 21 days of receiving sample.
- (a) Purge water prior to run will be analyzed only in case of a major hit for the analyte(s) associated with the hit.
 - (b) If filtered purge water samples fail to pass the delisting then the samples for the contingency runs will be analyzed.
If the MS/MSD for the first samples are within the control limits, then No MS/MSD is required for the contingency samples.
 - (c) For volatiles and other analyses, holding times listed in Table 4-4 shall not be exceeded regardless of priority.

3
4

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Presenting method deliverables given in Appendix A to EPA
Region VII Laboratories. |

The quality assurance officer, Jack Borris, has the primary responsibility for reviewing and approving the QA Project Plan and for overseeing the project to assure that the QA objectives are met.

Jack Borris is responsible for:

Approving the project QA Plan

Reviewing and approving QA plans submitted by the
analytical laboratories |4

Advising project manager and EI Corporate QA Officer of
any deficiencies or nonconformance to plan

The MIS project manager, R. Sawyer, has overall responsibility for the mobile incinerator operations including QA/QC. For this purpose he reports to the EERU R&D project director and group manager, G. Gupta, and who has the overall responsibility for the EERU project to the EPA. Some of the MIS project manager's (R. Sawyer) responsibilities are: |2

Managing the overall operation of the mobile incinerator. Included are such activities as obtaining required RCRA, TSCA, and state permits; readiness preparations of equipment, personnel, and waste feeds; operating site preparations; operating and safety procedure; and maintaining communication with USEPA/HWERL Branch on activities.

Deciding when test run sampling will be conducted.

Calling to the attention of the quality assurance officer and others, as appropriate, any problems arising during the testing that affect this QA Project Plan so that the problems can be resolved in a timely manner. Responsible for properly documenting any modifications to this plan.

Issuing a final test burn report to the EPA.

The test coordinator, J. Stumbar, reports to the MIS project manager and has field operations responsibility for conducting the planned tests. Some of the test burn coordinator's responsibilities are:

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TABLE 3-3. METHOD DETECTION LIMITS (MDL) OF THE FLUE GAS COMPONENT MONITORS

Parameter	Method of Measurement	MDL
CO	Continuous non-dispersive infrared analyzer	1 ppm
CO ₂	Gas chromatograph/thermal conductivity detector	0.5%
O ₂	Continuous polarographic detector	0.5%
NO _x	Chemiluminescent detector analyzer	10 ppm

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TABLE 3-4. ANALYTICAL METHODS AND DETECTION LIMITS

Toxic Constituent	DETECTION LIMITS		SW-846 Method	
	Solids	Purge Water		
Acetone	--	100 ug/L	8240	
Aldrin	10 ug/Kg	1.0 ug/L	8080	
Benzene	5 ug/Kg	5 ug/L	8240	
Benzo(a)pyrene	15.4 ug/Kg	0.023 ug/L	8310	
Benzo(b)fluoranthene	330 ug/Kg	0.018 ug/L	8270/8310	15
Biphenyl	-- ug/Kg	10 ug/L	8270	
Bis-2-ethylhexyl phthalate	-- ug/Kg	10 ug/L	8270	
Chlordane	10 ug/Kg	1.0 ug/L	8270	
Chlorobenzene	-- ug/Kg	5 ug/L	8240	
Chloroform	5 ug/Kg	5 ug/L	8240	
Chrysene	330 ug/Kg	0.150 ug/L	8270/8310	15
2,4-D	--	1.0 ug/L	8150	
Dibenz(a,h)anthracene	20.1 ug/Kg	0.030 ug/L	8310	15
Dichloromethane	5 ug/Kg	5 ug/L	8240	
1,3-Dichlorobenzene	--	10 ug/L	8270	
1,4-Dichlorobenzene	--	10 ug/L	8270	
1,2-Dichlorobenzene	--	10 ug/L	8270	
1,2-Dichloroethane	5 ug/Kg	5 ug/L	8240	
2,4-Dichlorophenol	330 ug/Kg	10 ug/L	8270	
Dichlorvos	100 ug/Kg	1.0 ug/L	8140	
Diethyl phthalate	--	10 ug/L	8270	
Disulfaton	100 ug/Kg	1.0 ug/L	8140	
Endosulfan I	10 ug/Kg	1.0 ug/L	8080	
Ethyl benzene	--	5 ug/L	8240	
Fluoranthene	--	10 ug/L	8270	
Fluorene	330 ug/Kg	10 ug/L	8270	
Indeno(1,2,3,cd)pyrene	330 ug/Kg	0.043 ug/L	8270/8310	15
Isophorone	--	10 ug/L	8270	
Methyl chloride	--	100 ug/L	8240	
Methyl parathion	100 ug/Kg	1.0 ug/L	8140	
Napthalene	--	10 ug/L	8270	
Nitrosodiphenylamine	330 ug/Kg	10 ug/L	8270	
Pentachlorophenol	--	10 ug/L	8270	
Phenanthrene	330 ug/Kg	10 ug/L	8270	
Phenol	--	10 ug/L	8270	
Polychlorinated biphenyls	10 ug/Kg	1.0 ug/L	8080	
Pyrene	--	5 ug/L	8270	
Tetrachloroethylene	5 ug/Kg	5 ug/L	8240	
2,3,4,6-Tetrachlorophenol	--	10 ug/L	8270	
Toluene	--	5 ug/L	8240	
2,4,5-TP (silvex)	50 ug/Kg	0.1 ug/L	8150	

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TABLE 3-4. ANALYTICAL METHODS AND DETECTION LIMITS

Toxic Constituent	DETECTION LIMITS		SW-846 Method
	Solids	Purge Water	
1,2,4-Trichlorobenzene	--	10 ug/L	8270
2,4,6-Trichlorophenol	330 ug/Kg	10 ug/L	8270
2,4,5-Trichlorophenol	--	10 ug/L	8270
2,4,5-Trichlorophenoxyacetic acid	50 ug/Kg	0.1 ug/L	8150
Xylenes (total)	--	50 ug/L	8240
Corrosivity	--	--	7.2
EP Toxicity	--	--	1310
Arsenic ¹	2 mg/Kg	53 ug/L	6010
Barium	40 mg/Kg	200 ug/L	6010
Cadmium	1 mg/Kg	5 ug/L	6010
Chromium	2 mg/Kg	10 ug/L	6010
Lead	1 mg/Kg	42 ug/L	6010
Nickel	8 mg/Kg	40 ug/L	6010
Selenium ¹	1 mg/Kg	75 ug/L	6010
Silver	2 mg/Kg	10 ug/L	6010
Mercury	0.1 mg/Kg	0.2 ug/L	7470
TDS	--	1 mg/L	160.1
TSS	--	1 mg/L	160.2
TOC	--	0.5 mg/L	9060
pH	--	0.1 pH units	9040
Total Cyanides (in EP toxicity leachate)	0.5 mg/Kg	10 ug/L	9010

¹ If arsenic and/or selenium is detected or if the detection limit is above the delisting criteria. The sample will be rerun using SW Method 7740 for selenium and Method 7060 for arsenic.

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TABLE 3-5. PCDD/PCDF PRACTICAL DETECTION LIMITS*

Compound	Ash (ppt)	Water (ppt)
2,3,7,8-TCDF	15.0	0.12
Tetra Furans	15.0	0.12
Penta Furans	15.0	0.12
Hexa Furans	37.0	0.30
Hepta Furans	37.0	0.30
Octa Furans	50.0	0.50
2,3,7,8-TCDD	15.0	0.12
Tetra Dioxin	15.0	0.12
Penta Dioxin	15.0	0.12
Hexa Dioxin	37.0	0.30
Hepta Dioxin	37.0	0.30
Octa Dioxin	50.0	0.50

* Per final rule given in the Federal Register/Vol. 53.
No. 48, March 11, 1988 pages 7903-7915

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4.0 SAMPLING PROCEDURES

This section details the planned sampling procedures for this test program. Currently, the program consists of a preproduction burn for delisting purposes consisting of four replicate eight hour tests conducted on consecutive days. Sampling requirements for these tests are listed in Table 4-1. Enviroresponse will have sufficient equipment and sample containers to conduct ten tests.

During each test burn run, the pesticide contaminated wastes will be burned in the incinerator for a period of 8 to 10 hours. All sampling for the run will be performed during this time period except as follows:

- o Kiln ash and cyclone ash, which require cooling, will be sampled approximately 12 hours after the test.

4.1 COMBUSTION PARAMETERS

The sampling and data collection procedures for waste flow rate, fuel flow rate, water flow rate, temperature, CO, CO₂, NO_x, O₂, and air flow rate are described below.

The waste solids feed rate to the rotary kiln is controlled by a weigh cell and a mechanical timer/sequencer that cycles a hydraulic feed ram. The desired feed rate is obtained by adjustment of the weigh cell controller to give the desired weight per cycle and the cycle time of the ram feeder to give the desired frequency. The weigh cell controller will send a weight signal to a printer to record the weight of each charge and a totalized weight.

The waste oil feed will be measured with an EXAC Model 2100 flow meter. This flow meter has a digital display for the instantaneous flow rate, lb/hr, and the totalized flow. The 2100 model has a maximum readable rate of 720 lb/hr with an accuracy of $\pm 0.15\%$. This unit will be field-calibrated with fuel oil before the test. The waste oil feed rate will be displayed on the instrument. The incinerator operator will

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4-1. SAMPLE REQUIREMENTS FOR PREPRODUCTION BURN ANALYSES

BYPRODUCT STREAMS	ANALYSIS	SAMPLE
1. Kiln Ash/Cyclone Ash composite	Dioxins/Furans Metals/Mercury/Cyanide Herbicides/BNAs PAHs Organophosphate Pesticides Pesticides/PCBs Volatile Organics	1-liter bottle 1-liter bottle 1-liter bottle 1-liter bottle 1-liter bottle 1-liter bottle 2-40 mL VOA vials 1 ₃ 1 ₅ 1 ₃ -3
2. Purge Water (after filters)	EP Toxicity Metals/Mercury Total Metals Cyanide Volatile Organics BNAs/Herbicides PAHs Organophosphate Pesticides Pesticides/PCBs Dioxins/Furans	1-liter cubitainer 1-liter cubitainer 1-liter cubitainer 2-40 mL VOA vials 1-gallon bottle 1-gallon bottle 1-gallon bottle 1-gallon bottle 2-gallon bottle 1 ₃ 1 ₅ 1 ₃
3. Purge Water (before filters)	EP Toxicity Metals/Mercury Total Metals Cyanide Volatile Organics BNAs/Herbicides PAHs Organophosphate Pesticides Pesticides/PCBs Dioxins/Furans	1-liter cubitainer 1-liter cubitainer 1-liter cubitainer 2-40 mL VOA vials 1-gallon bottle 1-gallon bottle 1-gallon bottle 1-gallon bottle 2-gallon bottle 1 ₃ 1 ₅ 1 ₃
4. Purge Water before test run (before filters)	EP Toxicity Metals/Mercury Total Metals Cyanide Volatile Organics BNAs/Herbicides PAHs Organophosphate Pesticides Pesticides/PCBs Dioxins/Furans	1-liter cubitainer 1-liter cubitainer 1-liter cubitainer 2-40 mL VOA vials 1-gallon bottle 1-gallon bottle 1-gallon bottle 1-gallon bottle 2-gallon bottle 1 ₃ 1 ₅ 1 ₃

NOTE: Amount of sample to be sufficient to supply two laboratories.

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also manually record the rate on the log sheet every 15 minutes. The totalized flow for each test will be recorded every 15 minutes to check that the instantaneous flow rates are consistent, and the feed tank levels will be also measured, using a sight level gage, to verify the quantity fed.

The flue gas composition (O_2 , CO, CO_2 and NO_x) will be continuously monitored at two locations. The first location is in the quench elbow at the SCC outlet immediately upstream of the water spray. This location will have two gas sample probes. One will supply the sample to the non-dispersive infrared/polarographic primary monitoring system and the other will supply the sample to the chromatograph backup monitoring system. The second sampling location is at the stack. One advantage of monitoring at these two points is that the amount of air in-leakage from the air pollution control equipment can be calculated.

The gaseous samples are withdrawn from the center of the ducts through a 1/2-in. Inconel probe. The extracted sample passes through a ceramic inertial filter, located inside the probe, to remove particulate material (greater than 100 micron) from the gas sample. There is another filter at the Perma Pure dryer. Filtration is required for instrument protection. Next, the filtered gas is partially cooled in an air-air heat exchanger to lower the temperature to approximately $38^{\circ}C$ ($100^{\circ}F$). Entrained liquids are collected in a liquid trap at the bottom of the exchanger. The gas sample then passes through a vaporizer ($120^{\circ}C$) to ensure that any entrained liquids from the process are vaporized before entering the gas drying unit. Gas drying will be accomplished in a Perma Pure dryer (hygroscopic, ion-exchange membrane) that removes water vapor from the gas sample without using a condensation process since condensation often removes key gaseous components from the sample. The cooled and dried gas sample is then transported to the gas chromatographic/chemiluminescent or the nondispersive infrared/polarographic analyzers through Teflon tubing. The sampling and sample transport functions are all controlled by a microprocessor. The sampling function of the gas monitoring system continuously operates to provide fresh, up-to-date gas samples to the analyzers for stack and flue gas analysis.

The temperatures at the rotary kiln and SCC outlets will be monitored with thermocouples, ANSI, type S (Pt/Pt-Rh), in direct contact with the flue gases. The thermocouples are shielded from the burner flames to eliminate temperature

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measurement error associated with flame radiation. The millivolt thermocouple signal is converted to a (4-20 mA) signal and is recorded on a strip chart recorder which is in the control panel on the kiln trailer.

The fuel flow rates will be read from rotameters and the air flow rates will be read from indicators. These data will be recorded every 30 minutes in the log sheets. These instruments will be calibrated before the test burn program according to manufacturers' recommendations. Wastewater collected during a test is measured by a flow meter and also will be measured at the end of each test by the change in liquid level in the wastewater tank.

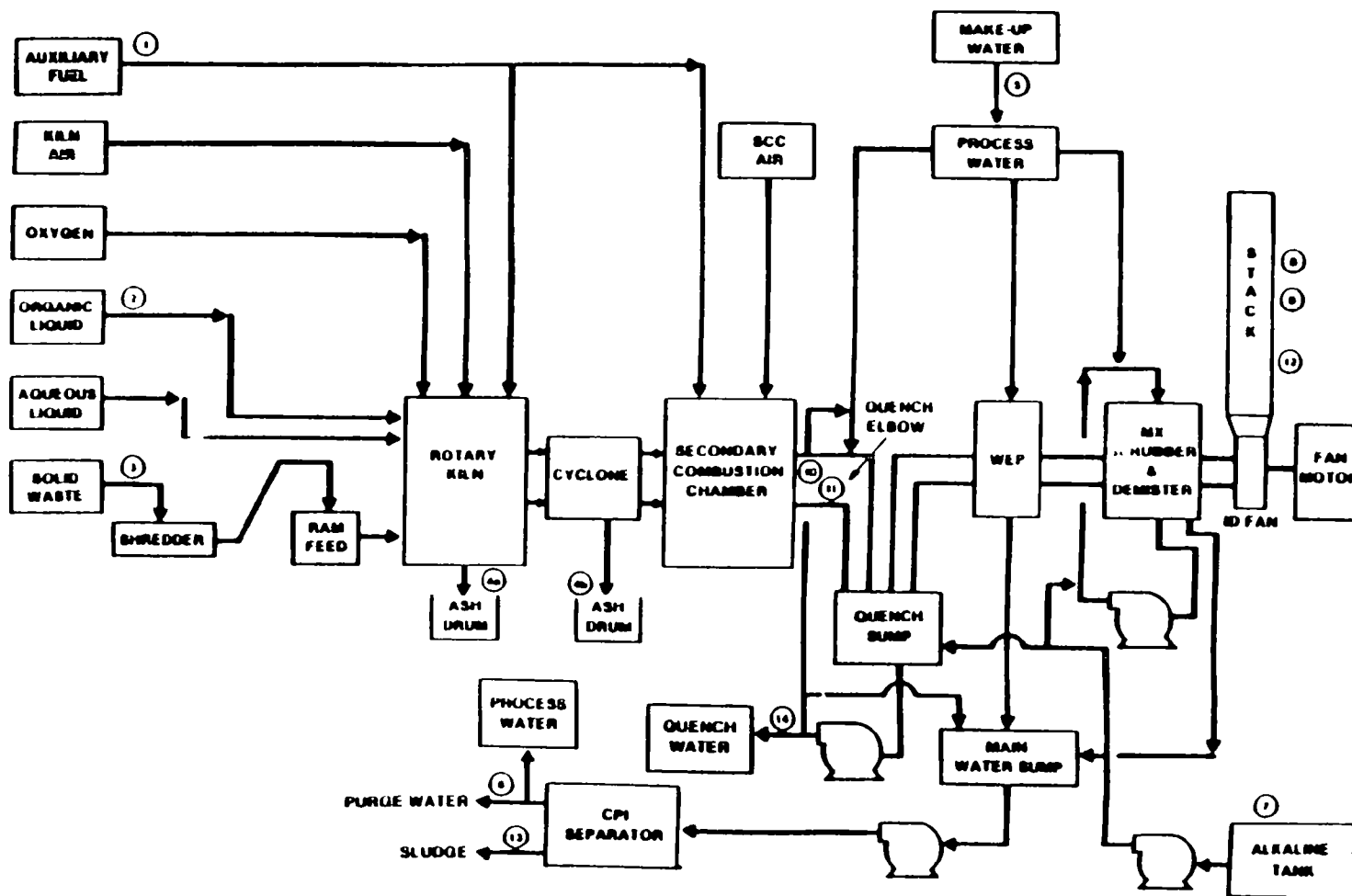
4.2 SAMPLING PERIODS

Each eight hour sampling period will begin when the incineration system is at steady-state. The project manager will determine when sampling may begin, based on a review of data from the continuous monitoring equipment. During sampling, the test coordinator will periodically verify that steady-state conditions exist. Steady-state operation is defined as consistent operation during a 30 minute period with less than 5% drift during 30 minutes for the SCC temperature and less than 10% drift for waste feed, and flue gas O₂ concentrations. These parameters will be checked at least every 30 minutes. If a transient condition develops during sampling, the test coordinator will decide whether to temporarily interrupt or to terminate sampling.

4.3 SAMPLING LOCATIONS AND PROCEDURES

The locations where solid, liquid, and gaseous samples are collected from the Incineration System are shown in Fig. 4-1. Materials supplied to the incineration process including liquid and solid waste feeds, are sampled at locations 2 and 3. Samples of process solids (ash) and liquid (purge water) effluents are collected at locations 4 and 6. The composition of the combustion gases is continuously monitored at the exit duct from the SCC (locations 11 and 12) and at the stack (location 13).

The sampling equipment and the procedures for collecting samples at each location are summarized in Table 4-2. Sampling frequency and reference methods are included in the table. Additional details regarding each sampling location are discussed below.



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FIGURE 4-1. Sampling locations for Test Burn

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TABLE 4-2. SAMPLE COLLECTION LOCATIONS, EQUIPMENT, AND METHODS

Description/Location	Access	Equipment	General Procedures/Frequency	Reference Methods ^a
1. Kiln and Cyclone Ash Container (4a,4b)	Open top	Spoon; glass bottle	Composite grab samples collected from drums for each run	S007, SW846
2. Purge water line downstream of carbon filters (6)	Tap	Glass bottle	Sample continuously during run (2 hour intervals) and composite	S004, SW846
3. Purge water line upstream of carbon filters (6)	Tap	Glass bottle	Sample continuously during run (2 hour intervals) and composite	S004, SW846
4. SCC exit duct (10)	4-in. port	Sample extraction/conditioning system	Continuously filters, cools dries, and transports combustion gases to instruments for O ₂ , NO _x , CO ₂ and CO analyses	
5. SCC exit duct (10)	4-in. port	Sample extraction/conditioning system	Continuously filters, cools dries, and transports combustion gases to instruments for O ₂ and CO analyses	
6. Stack (12)	4-in. port	Sample extraction/conditioning system	Continuously filters, cools dries, and transports combustion gases to instruments for O ₂ , NO _x , CO ₂ and CO analyses	

^a Prefix S refers to Sampling and Analysis Methods for Hazardous Waste Combustion, EPA-600/8-84-002. SW refers to Test Methods for Evaluating Solid Waste, SW846, Third Ed., 1987.

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Kiln and Cyclone Ash (4) -- Bulk ash will be collected from the kiln and cyclone in open-top containers, covered, and allowed to cool. A scoop will be used to collect a sample from each container. The samples collected during each replicate test run will be composited in a stainless steel mixing apparatus. |

Purge Water (6) -- Samples will be taken from taps in the discharge line downstream and upstream of the carbon filters. Before the first run, a sample upstream of the carbon filters will be collected to provide a blank sample. Samples will then be taken at 1 hour intervals after test start, and ending with test completion. The samples for volatile organic analysis will be composited as described in the procedure given in Appendix B. The other samples will be composited as per ASTM Method D-3370. |
-2, -3

4.4 Flue Gas Sampling

Continuous emission monitors will be utilized for measuring the concentration of the following gases: oxygen, carbon dioxide, carbon monoxide, oxides of nitrogen, and total hydrocarbons during all tests. A side view of the exhaust stack is shown in Figure 4-2. The stack is 2 foot by 2 foot square and the top of the stack is approximately 40 ft above ground level. -2

The continuous emission monitoring system (CEM) probe is located in a port hole located 2 feet above the stack silencer. Other stack sampling is not required because the program is solely for purposes of delisting byproducts streams as hazardous wastes.

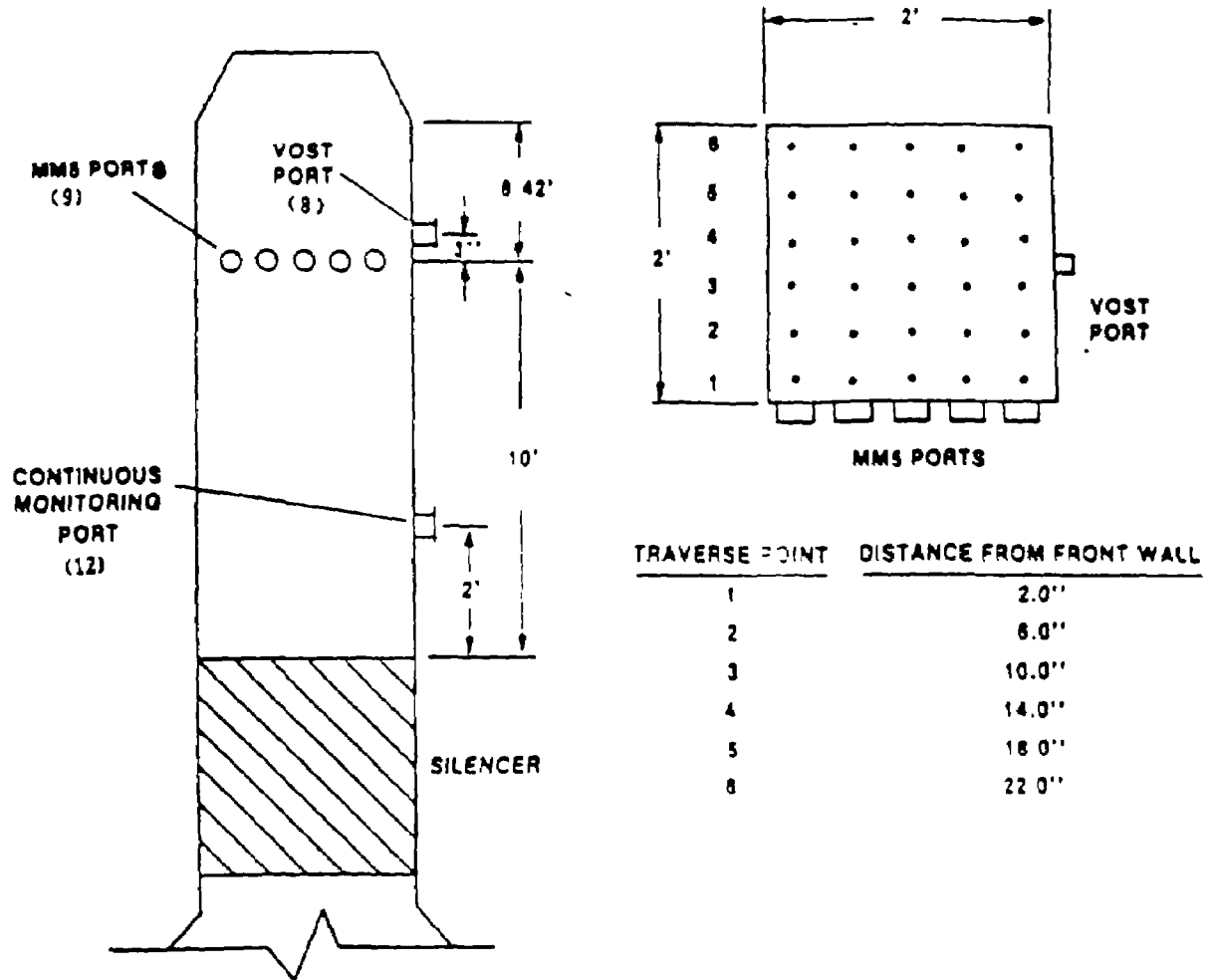
The CEM system will monitor the stack during the entire test period.

4.5 SAMPLING PROGRAM OPERATIONS

All field activities will be conducted per the guidance of Section 3.0 in the (OSWER) Guidance Directive 9355.0-14 for implementing field activities. This will include Sections 3.2 (Control of Fieldwork Generated Contaminated Material), 3.3 (Organization of the Field Team), 3.4 (Decontamination), and 3.5 (General Health and Safety Considerations).

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FIGURE 4-2.
 Stack gas sampling ports and sampling probe traverse points.



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4.6 SAMPLE CONTAINERS AND PRESERVATION REQUIREMENTS

Table 4.3 summarizes the container requirements for the samples. Table 4-4 summarizes preservation requirements and presents holding times for the samples. Precleaned sample bottles will be obtained for I-Chem Research (415/782-3905) in California (presently the official bottle repository for the Superfund Program). 4

4.7 SAMPLE CONTROL

The purpose of sample control is to maintain the quality of samples during collection, transportation and storage for analysis. Sample control in the field will be per OSWER directive 9355.0-14 Sections 4.0 and 6.2. Information detailed in these sections include:

- o Records (OSWER Section 4.5)
- o Procedures for sample identification tags, sample traffic reports, chain-of-custody records, receipt-for-samples forms, custody seals and field notebooks (Section 4.6)
- o Packaging, labeling and shipping (Section 6.2)

4.7.1 Chain of Custody

Chain of custody procedures will be followed to ensure a documented, traceable link between any given measurement results and the sample and parameter which it represents. These procedures are intended to provide a legally acceptable record of sample preparation, storage, and analysis.

To track sample custody transfers before ultimate disposition, sample custody will be documented using the form shown in Figure 4.3. A chain of custody seal is shown in Figure 4.4. In addition, a master logbook will be used as a centralized mechanism for documenting project activities.

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TABLE 4-3. PREPRODUCTION BURN SAMPLE FREQUENCY AND CONTAINER REQUIREMENTS

Process Stream	Sampling Frequency	Total Containers For Compositing	Total Sample Containers Shipped	Total Sample Containers Required	Sample Container Description	
Kiln ash/Cyclone ash composite	One grab per drum and composite	0	6	6	1000 mL wide-mouth glass bottles w/teflon-lined screw cap	13 15
		0	2	2	40 mL glass vials w/ teflon-lined screw cap	
		1	0	1	12" x 24" shallow stainless steel pan	13 -3
Scrubber Purge	One grab every 2	8	2	10	40 mL glass vials w/teflon-lined screw cap	
Water (after carbon filter)	hours during run and composite	1	0	1	1000 mL glass Erlenmeyer flask	
		0	1	1	125 mL amber glass bottle w/teflon-lined screw cap	
		8	0	8	1000 mL amber glass bottles w/teflon-lined screw cap	13
		0	3	3	1000 mL cubitainer w/teflon-lined screw cap	
		0	5	5	1 gallon amber glass bottle w/teflon-lined screw cap	14 15
		1	0	1	32 oz wide mouth jar	13

(continued)

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TABLE 4-3. PREPRODUCTION BURN SAMPLE FREQUENCY AND CONTAINER REQUIREMENTS (cont.)

14

Process Stream	Sampling Frequency	Total Containers For Compositing	Total Sample Containers Shipped	Total Sample Containers Required	Sample Container Description	
Scrubber Purge Water (before carbon filter)	One grab every 2 hours during run and composite	8	2	10	40 mL glass vials w/teflon-lined screw cap	
		1	0	1	1000 mL glass Erlenmeyer flask	
		0	1	1	125 mL amber glass bottle w/teflon-lined screw cap	
		8	0	8	1000 mL amber glass bottles w/teflon-lined screw cap	13
		0	3	3	1000 mL cubitainer w/teflon-lined screw cap	14
		0	5	5	1 gallon amber glass bottle w/teflon-lined screw cap	15
Scrubber Purge Water (Collected prior to run)	One grab taken prior to any waste feed going to incinerator	1	0	1	32 oz wide mouth jar	13
		0	2	2	40 mL glass vials w/teflon-lined screw cap	
		0	1	1	125 mL amber glass bottle w/teflon-lined screw cap	13
		0	3	3	1000 mL cubitainer w/teflon-lined screw cap	14
		8	0	8	1000 mL amber glass bottles w/teflon-lined screw cap	
		0	5	5	1 gallon amber glass bottle w/teflon-lined screw cap	15
		1	0	1	32 oz wide mouth jar	13

NOTE: Basis is for 1 run. Equipment for ten runs is required.

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TABLE 4-4. PREPRODUCTION BURN PRESERVATION
REQUIREMENTS AND HOLDING TIMES

Analyses	Preservation	Holding Times
Dioxins/Furans ¹	Cool to <4°C	7 days until extraction
Metals	HNO ₃ to pH <2	6 mos.
Mercury	HNO ₃ to pH <2	13 days
Cyanide	Cool <4°C NaOH to pH 12 0.6 g ascorbic acid	14 days
BNAs ¹	Cool <4°C	7 days until extraction
PAHs ¹	Cool <4°C	7 days until extraction
Organochlorine Pesticides ¹	Cool <4°C	7 days until extraction
Pesticides ¹	Cool <4°C	7 days until extraction
PCBs	Cool <4°C	7 days until extraction
Vol. Org.	Cool <4°C 4 drops conc. HCl	14 days
TOC	Cool <4°C HCl to pH <2	28 days
TDS, TSS	Cool <4°C	48 hours



Note:

1. Analysis within 30 days after extraction.

FIGURE 4.3

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FIGURE 4.4

CUSTODY SEAL			CUSTODY SEAL
_____ Date			_____ Date
_____ Signature			_____ Signature

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4.7.2 Logbooks

Field notebooks and a master sample log will be used to record sampling activities and information. Field notebooks will be bound field survey books. Notebooks will be surrendered to the field sampling task leader upon completion of the assignment. The cover of each logbook will contain:

- o The name of the person to whom the book is assigned
- o The book number;
- o The project name;
- o Entry start date; and
- o Entry completion date.

Entries will include general sampling information to reconstruct the site activities without reliance on memory. The beginning of each entry will include the date, sampling site, start time, weather conditions, field personnel present, and level of personal protection. Other possible entries would be names and purpose of any visitors to the vicinity during sampling, unusual conditions which might impact the interpretation of the subsequent sampling data, or problems with the sampling equipment. All entries will be in ink with no erasures. Incorrect entries will be crossed out with a single strike and initialed.

A master sample log will be maintained on site for all samples taken. A full description of the sample, its origin, and condition will be included in the master log entry. An example of the master log heading is shown in Figure 4.5.

4.7.3 Sample Labels

Each sample will be assigned a unique identification number. Examples of a sample label and a sample Identification Tag are shown in Figures 4.6 and 4.7.

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FIGURE 4.5

Example of Master Logbook Format

[illegible]

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FIGURE 4.6

EPA REGION VII ** VOLATILES	
ABC LANDFILL	WCC/HOUSER
EPA#: K09ZA001	SMO#: _____
11/ /86	TIME: _____
DEPTH: _____	LOCATION: _____
COLOR: LIME	

EPA REGION VII ** VOLATILES	
ABC LANDFILL	WCC/HOUSER
EPA#: K09ZA001	SMO#: _____
11/ /86	TIME: _____
DEPTH: _____	LOCATION: _____
COLOR: LIME	

EPA REGION VII ** VOLATILES	
ABC LANDFILL	WCC/HOUSER
EPA#: K09ZA001	SMO#: _____
11/ /86	TIME: _____
DEPTH: _____	LOCATION: _____
COLOR: LIME	

EPA REGION VII ** METALS-TOTAL	
ABC LANDFILL	WCC/HOUSER
EPA#: K09ZA001	SMO#: _____
11/ /86	TIME: _____
DEPTH: _____	LOCATION: _____
COLOR: WHITE	


EPA REGION VII ** METALS-FILTERED	
ABC LANDFILL	WCC/HOUSER
EPA#: K09ZA001	SMO#: _____
11/ /86	TIME: _____
DEPTH: _____	LOCATION: _____
COLOR: GRAY	

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FIGURE 4.7

Designation		Date	Camp	Preservative Yes <input type="checkbox"/> No <input type="checkbox"/>
Time	Station	Station	Station	ANALYSES
Station	Station	Station	Station	BOD Amens
				Solids (total) (non fat)
Station	Station	Station	Station	COD, TOC, Nutrients
				Phenolics
Station	Station	Station	Station	Mercury
				Metals
Station	Station	Station	Station	Cyanide
				Oil and Grease
Station	Station	Station	Station	Organics GC/MS
				Priority Pollutants
Station	Station	Station	Station	Volatile Organics
				Pesticides
Station	Station	Station	Station	Mutagenicity
				Bacteriology
Station	Station	Station	Station	Remarks
Tag No.		Lab Sample No.		
3 60966				

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY



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5.0 ANALYTICAL PROCEDURES AND CALIBRATIONS

This section delineates the analytical protocols which will be used to analyze samples at the laboratories.

As discussed in Sections 1.0 and 4.0, the following process streams will be sampled:

- o Kiln ash, -2
- o Cyclone ash, -3
- o Purge water downstream of carbon filter, -2
- o Purge water upstream of carbon filter, and
- o Stack gases.
 - CO₂, O₂, NO_x, THC

These samples will be analyzed for the parameters previously discussed in Section 4.0 using appropriate laboratory analytical techniques described in this section and outlined in Table 3-4.

5.1 INORGANIC ANALYSIS

5.1.1 Total Dissolved & Total Suspended Solids

Purge water upstream of the carbon filter will be analyzed for Total Dissolved Solids by Method 160.1 and Total Suspended Solids according to Method 160.2 of "Methods for Chemical Analyses of Water and Wastes", EPA 600/4-79-020. Measurements are to be performed with an analytical balance which has been calibrated using standardized weights traceable to the National Bureau of Standards and capable of weighing to the nearest milligram (mg).

5.1.2 pH

The pH of the purge water upstream and downstream of the carbon filters will be determined according to EPA Method 9040 (SW-846, 3rd Edition). The meter is calibrated to a pH of 7.0 and 10.0 using two buffer solutions.

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5.1.3 Total Organic Carbon

Total organic carbon content will be determined for the purge water upstream of the carbon filters following the procedures outlined in Method 9060 (SW-846, 3rd Edition). The analyses will be performed on a Dohrman DC80 TOC Analyzer the instrument will be calibrated daily with standard solutions of potassium hydrogen phthalate over the expected concentration range, 0, 0.25 and 0.50% TOC.

5.1.4a Metals - (Total)

Samples of purge water (downstream and upstream of the carbon filters) from the incineration system will be analyzed for total metals prior to the analysis for EP toxicity. If the target analytes meet the delisting criteria then the EP toxicity test is unnecessary for the target analytes. The samples for the total metals analysis will be acidified in the field with 5 mL/L of reagent grade HNO_3 . The samples will be extracted in the laboratory using SW Method 3005 and the extract will be analyzed by ICP according to Method 6010 of U.S. EPA SW-846. The calibration for ICP and the separate analysis by SW Method 7470 required for mercury are discussed in Section 5.1.4b.

5.1.4b Metals - (EP Toxicity & Nickel)

Samples of purge water (downstream and upstream of the carbon filters) from the incineration system and kiln ash/cyclone ash composite will be submitted for metals determination in accordance with delisting requirements (arsenic, barium, cadmium, chromium, lead, mercury, nickel, selenium, and silver). Extraction procedures will follow those outlined in 40 CFR Chapter 1, Part 261, Appendix II for EP toxicity test procedures. After extraction is complete, the extracts will be acid digested per procedures discussed below and analyzed. The analyses will be performed by ICP according to Method 6010 of U.S. EPA SW-846 with the exception of Mercury. Mercury will be analyzed by Method 7470 (cold vapor). If selenium and/or arsenic is detected or if matrix interferences raise the method detection limits above the delisting criteria for these compounds, the sample will be rerun using atomic adsorption Method 7740 for selenium and Method 7060 for arsenic. Detection limits are provided in Table 3-4.

Digestion For ICP - The EP Toxicity extract will be digested according to the procedure given in Method 3010. This consists of an acid digestion with concentrated nitric acid (HNO_3) under gentle reflux conditions until the digestate is

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light in color and does not change in appearance. This is followed by addition of 1:1 hydrochloric acid (HCl) and dilution to 100 mL with dionized water (ASTM Type II).

Digestion For Methods 7060 and 7740 - The EP Toxicity extract will be digested according to the procedure given in section 7.1 of the method description. This consists of an acid digestion with 30% hydrogen peroxide solution (H_2O_2) and concentrated nitric acid (HNO_3). This is followed by addition of 1% nickel nitrate solution and dilution with Type II water.

Dissolution Procedure For Method 7470 (Hg) - The EP Toxicity extract will be digested according to the procedure given in section 7.1 of the method description. This consists of addition of sulfuric acid (H_2SO_4) and concentrated HNO_3 followed by addition of 5% potassium permanganate solution and 5% potassium persulfate solution. Sodium chloride-hydroxylamine sulfate solution is then added to reduce excess permanganate. Finally, stannous sulfate solution is added and the sample bottle is immediately attached to the aeration apparatus for Hg analysis.

Calibration For ICP - Prepare mixed calibration standard solutions combining appropriate volumes of stock solutions. Five different sets of mixed solutions are required as specified in Method 6010. Each set should be prepared at three different concentration levels. The concentrations should span the linear range of the instrument and bracket the target concentrations for delisting (Table 1-1). Establish the calibration curve by running the blank first and then working toward the highest standard.

Calibration For Methods 7060 (As), 7470 (Hg) and 7740 (Se) - The calibration will be made according to section 8.2 of the respective method description using appropriate standard solutions as described above. A calibration curve will be constructed by analyzing a blank and three standard concentrations. A new calibration curve will be constructed after each hour of continuous sample requirements.

5.1.5 Total Cyanides

Total cyanides will be analyzed in liquid matrices using Method 9010 of SW-846 which is a colorimetric technique. Per the Federal Register requirement, cyanides in the EP toxicity leachate from the solid sample will also be measured by Method 9010.

ENVIRESPONSE, INC.

ENVIRONMENTAL EMERGENCY RESPONSE UNIT

GSA RARITAN DEPOT, WOODBRIDGE AVENUE BUILDING 209 BAY F EDISON, N J 08837
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DATE: July 21, 1988
TO: Joyce Perdek
Project Officer, HWERL
FROM: James P. Stumbar *JPS*
SUBJECT: QA ID No. SP-236-E
Lab Work Plan No. Support Office of Pesticides
Project Category II

Title: "USEPA Mobile Incinerator System Pesticide
Preproduction Burn (2,4,5-T)

Prepared by: Enviresponse, Inc.

CC: Guy Simes

Revision 5 of QAPP SP-236-E is attached. This contains minor changes to the previously approved Revision 4. These changes are described below:

1. SW-846 Method 8310 has been specified for those polynuclear aromatic hydrocarbons (PAHs) for which the delisting criteria require lower detection limits than achievable with Method 8270.

Affected pages: Section 1 pages 7, 10, and 12
Section 2 pages 1, 3, and 4
Section 3 pages 4, 6, and 9
Section 4 pages 10 and 11
Section 5 pages 25 and 26

2. Minor revisions and corrections have been made to the holding times and preservation requirements in Table 4-4 to reflect SW-846 requirements. A 30 day maximum holding time has been specified to expedite results for semivolatiles, PAHs, etc.

Affected page: Section 4 page 12

3. Method 8080 write-up has been changed to reflect external standardization used by the analytical laboratory.

Affected page: Section 5 page 24

Category II QUALITY ASSURANCE PROJECT PLAN

PROJECT TITLE: The U.S. Environmental
Protection Agency
Mobile Incinerator System
Pesticide Preproduction Burn
EPA PROJECT OFFICER: James J. Yezzi, Jr.
EERU PROGRAM DIRECTOR: Gopal Gupta
PERFORMING ORGANIZATION: Enviresponse, Inc. - EERU, GSA
Raritan
Depot, Edison, New Jersey
08837
DURATION: February 1988 to September 1988
TYPE OF PROJECT: Work Order under EPA Contract
68-03-3255
SUPPORTING ORGANIZATION: U.S. Environmental Protection
Agency
Hazardous Waste Engineering
Research
Office of Research and
Laboratory Development Releases
Control Branch

13

APPROVALS:

EI CORPORATION

NAME: Gopal Gupta
TITLE: Project Manager
SIGNATURE: Gopal Gupta

DATE 7/21/88

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DATE 5/21/88

NAME: James P. Stumbar
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DATE 7/21/88

NAME: Jack Borris
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TITLE: Project Officer
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Date _____

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TITLE: EPA Task Monitor
SIGNATURE: _____

Date _____

NAME: Guy F. Simes
TITLE: QA Officer HWERL
SIGNATURE: _____

Date _____

13

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QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM
for
HWERL Contracts/IAGs/Cooperative Agreements/In-house Projects

Lab Workplan No: Support: Office of
Pesticides

Task Start Date: May 1988 - Sept 1988
(for measurement, data gathering, and/or data generation activities)

Project Category: II

QA ID No: SP - 236 -E

Date QAPP Received: _____

Task Title: USEPA Mobile Incinerator System Pesticide Preproduction Burn
(2,4,5-T)

Technical Project Officer: Joyce Perdek

Contractor: Enviresponse

APPROVALS:

<u>Robert Sawyer</u> Contractor Project/Task Manager	<u>R Sawyer</u> Signature	<u>7/21/88</u> Date
<u>John Borris</u> Contractor QA Manager	<u>J. B. Borris</u> Signature	<u>7/21/88</u> Date
<u>TBN</u> Affiliate Task Manager*	_____ Signature	_____ Date
_____ Other (as appropriate)	_____ Signature	_____ Date
<u>Joyce Perdek</u> HWERL Technical Project Officer	_____ Signature	_____ Date
<u>John S. Farlow</u> HWERL Branch or Staff Chief**	_____ Signature	_____ Date
<u>Guy Simes</u> HWERL Quality Assurance Officer	_____ Signature	_____ Date

* Approval signature is required for any ancillary sampling, analytical, or data gathering support provided by a subcontractor or HWERL principal investigator.

** Approval signature from the HWERL Branch or Staff Chief is required for Category I, II, and III extramural projects and for all in-house projects.

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5.2 VOLATILE ORGANICS

5.2.1 Analysis for Volatile Organics

The samples of kiln ash/cyclone ash composites and purge water downstream and upstream of the carbon filters for volatiles analysis will be conducted using Method 8240 of EPA SW-846. These samples will be analyzed for acetone, 1,2-dichloroethane, chloroform, tetrachloroethylene, methyl chloride, methylene chloride, xylenes, benzene, ethyl benzene, toluene, and chlorobenzene. Detection limits are presented in Table 3-4. A portion of the ash samples will be dispersed in methanol to dissolve the volatile organic constituents. An aliquot of the methanol solution will be combined with organic free water in the purging chamber. Methanol is freely miscible with water in all proportions. 13

Approximately 1 gram of the sample will be transferred into a preweighed 50 mL glass centrifuge tube or 20 mL glass vial, with Teflon-lined caps, containing 15 mL methanol. The capped centrifuge tube and methanol will be reweighed on an analytical balance to determine the sample weight. Care will be taken not to touch the sample transfer implement to the methanol. The sample will be dispersed in the methanol as expeditiously as possible to prevent loss of volatiles from the sample. After the sample weight has been determined, additional methanol will be added to the 20 mL mark of the centrifuge tube or glass vial. The sample container will be securely recapped and then vigorously agitated for 1 minute. The mixture will be agitated manually or with the aid of a vortex mixer. If the sample does not disperse during the process, the mixture will be sonicated in an ultrasonic bath for 30 minutes. The mixture will be allowed to stand until a clear supernatant is obtained. Centrifugation may be necessary to facilitate phase separation. The supernatant solution will then be analyzed or stored for future analytical needs in a 10 mL screw cap vial with Teflon cap line at 4°C.

Analysis of the methanol extract will proceed by taking an appropriate aliquot of the methanol solution using a microsyringe. An aliquot of the methanol solution extract will be dispersed directly into 5 mL reagent water in the purging device. The sample will then be purged according to Method 5030 of SW-846 for GC/MS analysis.

Aqueous samples will be analyzed by the same method as the methanol extracts. Table 5-1 provides the GC/MS operating

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TABLE 5-1. GC/MS INSTRUMENT OPERATING CONDITIONS FOR VOLATILES ANALYSIS

GC/MS Conditions

<u>Instrument</u>	Hewlett-Packard 5985 or Finnigan OWA
-------------------	--------------------------------------

GC Conditions

Column	1% SP-1000 on Carbopack B, 6 ft x 2 mm ID column
Temperature	45 ⁰ C held for 3 min, then 8 ⁰ /min to 220 ⁰ C and held
Injector temperature	200 - 225 ⁰ C
Carrier flow	Helium at 30 cm/sec or hydrogen 50 cm/sec

MS Conditions

Emission	300 ua
Ionization	70 eV
Scan time	5 scans/peak but not to exceed 7 scans/peak
Mass interval	35-260 amu

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conditions for these analyses. Surrogate compounds and internal standards shown in Table 5-2 will be injected prior to GC/MS analysis.

5.2.2 Calibration Standards for Volatile Organics

Stock standard solutions will be prepared from pure standard materials or purchased certified solutions. The stock standards will be prepared in methanol by serially diluting EPA EMSL (Las Vegas, Nevada) standard solutions. Because of the toxicity of some of the organohalides, primary dilutions of these materials will be prepared in a hood.

Fresh standards will be prepared at least weekly for volatile compounds with boiling points at $<30^{\circ}\text{C}$. All other standards will be replaced monthly, or sooner if comparison with check standards indicates a problem. Table 5-3 lists the compounds in the calibration mixture along with internal standards and surrogate compounds.

Secondary dilution standards in methanol will be prepared from stock standard solutions which contain the compounds of interest, at concentrations such that the desorbed calibration standards will bracket the working range of the analytical system.

Calibration standards will be prepared at a minimum of five concentration levels. The calibration standards will be prepared in reagent water.

5.2.3 Tuning for Volatile Analysis

The GC/MS will be tuned at the beginning of each day. The instrument tuning will be verified using a direct injection of bromfluorobenzene (BFB), using the criteria presented in Table 5-4. | 3

If the performance criteria listed above are not met, the analyst will retune the instrument and repeat the performance check. The performance criteria will be met before any standards, blanks, or samples are analyzed.

After allowing the column to cool, a Laboratory Control Sample, consisting of an EPA EMSL Quality Assurance (QA) spike, will be analyzed. If the measured value departs from | 2

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TABLE 5-2. SURROGATE COMPOUNDS AND INTERNAL
STANDARDS USED TO ANALYZE VOLATILES

=====

Surrogate Compounds

d₈-toluene

Bromofluorobenzene

d₄-1,2-dichloroethane

Internal Standards

Bromochloromethane

1,4-difluorobenzene

d₅-chlorobenzene

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TABLE 5-3. VOLATILE ORGANIC CALIBRATION MIXTURE

Compound		Typical relative retention times
<u>Region A</u> - 1	Chlorobromomethane (internal standard)	1.0
2	d ₄ -1,2-dichloroethane (surrogate)	1.2
3	Methylene chloride	0.73
4	Chloroform	1.17
5	1,2-dichloroethane	1.23
<u>Region B</u> - 1	1,4-difluorobenzene (internal standard)	1.0
2	d ₈ -toluene (surrogate)	1.17
3	Trichloroethene	0.87
4	Benzene	0.89
<u>Region C</u> - 1	d ₅ -chlorobenzene (internal standard)	1.0
2	Bromofluorobenzene (surrogate)	1.23
3	Tetrachloroethylene	0.91
4	Toluene	0.96
5	Clorobenzene	1.01

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TABLE 5-4. BFB ION ABUNDANCE CRITERIA

=====	
Mass	Ion abundance criteria
<hr/>	
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	less than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176
<hr/>	

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the true value by more than two standard deviations as listed on the EMSL specification sheet, corrective action will be taken before further analyses will be performed.

2

5.2.4 Calibration for Volatile Analysis

After all system criteria have been met, the GC/MS will be initially calibrated to determine response by generating a five-point calibration curve. System calibration will be accomplished by the analysis of reagent water spiked with the compounds of interest. Spiked solutions will be prepared for at least five concentration levels and responses of the compounds at these levels will be recorded to form a calibration curve. One of the concentration levels will be at a concentration near, but above, the method detection limit found in real samples. The remaining concentration levels should correspond to the concentration levels found in real samples or not exceed the working range of the GC/MS system.

2

On every day that samples are analyzed, verification of the five-point calibration curve is necessary. A standard set of solutions with concentrations of the compounds of interest between the low and high points of the calibration curve will be analyzed. If the response varies by more than ± 25 percent, the calibration check procedure will be repeated. A second failure indicates the calibration curve is invalid and the instrument must be recalibrated. Calibration will be performed a minimum of once per calendar week.

The GC/MS data system will be programmed to operate in the Total Ion Current Profile (TCIP) mode collecting the mass spectra for each of the compounds of interest. The peak for the major ion of each compound will be measured versus concentration at five levels of calibration.

The information regarding response ratio versus area ratio for each standard is stored in the computer and used to calculate sample concentration. The response factor (RF) for each compound is calculated using the following equation:

$$RF = \frac{(A_x \cdot C_{is})}{(A_{is})(C_x)}$$

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where: A_x = Area of the prime characteristic ion for the compound to be measured.

A_{is} = Area of the primary characteristic ion of the internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound to be measured.

5.3 SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270

5.3.1 Analysis for Semivolatile Organic Compounds

Samples of the purge water (downstream and upstream of the carbon filters), and kiln ash/cyclone ash composite will be sent to the laboratory for analysis of delisting requirement organic extractable compounds by EPA Method 8270 of SW-846. For Method 8270 the compounds of interest include: phenol, chlorinated phenols, di- and tri- chlorobenzenes, polynuclear aromatic hydrocarbons and phthalates. Detection limits are presented in Table 3-4.

Aqueous samples will be spiked with the surrogate compounds shown in Table 5-5, then serially extracted with methylene chloride at a pH greater than 11 and again at pH less than 2, using a separatory funnel. The methylene chloride extract is dried and subsequently concentrated to a volume of 1 to 2 mL. The concentrated extract is then analyzed by gas chromatography/mass spectrometry using the operating conditions shown in Table 5-6.

Qualitative identification is performed using the retention time data and spectral matches to standards. Quantitative analysis will be performed using an internal standard method of quantification.

The potential for contamination introduced as a result of sample handling and analysis procedures will be assessed by analyzing daily method blanks. These consist of deionized water taken from the Laboratory System to detect contamination introduced by extraction and sample handling.

Preparation of ash samples for semivolatile organic analysis will be conducted in accordance with Method 3540 of EPA

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TABLE 5-5. SURROGATE COMPOUNDS ADDED TO SAMPLE
FOR SEMIVOLATILE ORGANIC ANALYSIS

=====
d ₅ -Nitrobenzene
2-fluorobiphenyl
d ₁₄ -terphenyl
d ₆ -phenol
2-fluorophenol
2,4,6-tribromophenol
=====

TABLE 5-6. GC/MS OPERATING CONDITIONS FOR METHOD 8270
SEMIVOLATILE ORGANIC ANALYSES

=====	
<u>Instrument</u>	Hewlett-Packard 5985 or 5988A, Quadrupole Mass Spectrometer
<u>GC Conditions</u>	
Column	DB5 30M fused silica capillary or equivalent
Temperature program	40 ⁰ C held for 4 min, then 10 ⁰ /min to 270 ⁰ C and held
Injector type	Grob type
Injector temperature	250 ⁰ - 300 ⁰ C
Injection volume	1-2 uL, splitless
Column flow	Helium at 30 cm/sec or hydrogen at 50 cm/sec
<u>MS Conditions</u>	
Electron energy	70 eV
Scan rate	1.0 sec/scan
Mass range	35 - 500 amu
Source temperature	According to manufacturer specifications

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SW-846. The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble, and extracted first with methylene chloride in a Soxhlet extractor. The methylene chloride extract is dried, concentrated and analyzed by gas chromatography/mass spectrometry in accordance with the GC/MS procedures of Method 8270 in SW-846. The operating conditions are shown in Table 5-6.

5.3.2 Instrument Tuning

At the beginning of each working day, the system will be tuned by injecting bis-decafluorotriphenylphospene (DFTPP) to obtain a spectrum. The GC/MS will be tuned to meet all the key ion criteria shown in Table 5-7.

5.3.3 Calibration Standards

The GC/MS unit will be calibrated to the analytes of concern and the internal standards and surrogates shown in Table 5-8. Calibration standards will be prepared at five concentration levels by adding volumes of stock EMSL standards to a volumetric flask and diluting to volume with acetone or other suitable solvent. These calibration standards will cover the working range of 20 to 200 ppb in the pre-extracted samples. Each new set of standards will be verified by analyzing EPA/EMSL QA samples of base, neutral, and acid extractable compounds.

5.3.4 Instrument Calibration

Internal standards, shown in Table 5-8, will be used to calibrate the instrument. One (1) of each calibration mixture will be analyzed and the area of the primary characteristic ion will be tabulated against the concentration for each compound and internal standard. A response factor, defined in Section 5.2.4 will then be calculated.

A five-point calibration curve will be constructed by plotting the response factor against the standard concentration. The concentration of the standards will be in the range of 20 to 160 ug/mL.

The working calibration curve or response factor will be verified on each working day by measurement of the calibration standards. The working calibration curve will be considered valid if the criteria specified in Section 7.4 of the method description for Calibration Check Compounds (CCC) and System Performance Check Compounds (SPCC) are met and 90% of the semivolatiles have an actual response within ± 25 percent of

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3

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TABLE 5-7. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA*

=====	
Mass	Ion abundance criteria
<hr/>	
51	30 - 60% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	40 - 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	greater than 1% of mass 198
441	present but less than mass 443
442	greater than 40% of mass 198
443	17 23% of mass 442
<hr/>	

* For the following instrumental parameters:
electron energy: 70 volts (nominal); mass range
35-450 amu; scan time: at least 5 scans/peak but
not greater than 7 seconds per scan.

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TABLE 5-8. SURROGATES AND INTERNAL STANDARDS USED TO
CALIBRATE THE GC/MS FOR EXTRACTABLE ANALYSES

=====	
Parameter	Relative retention time
<hr/>	
<u>Region 1</u>	
d ₄ -1,4-dichlorobenzene (IS)*	1.0
d ₅ -phenol	0.94
2-fluorophenol	0.69
<u>Region 2</u>	
d ₈ -naphthalene (IS)	1.0
d ₅ -nitrobenzene	0.84
<u>Region 3</u>	
d ₁₀ -acenaphthene (IS)	1.0
2-fluorobiphenyl	0.90
<u>Region 4</u>	
d ₁₀ -phenanthrene (IS)	1.0
2,4,6-tribromophenol	0.91
<u>Region 5</u>	
d ₁₂ -chrysene	1.0
d ₁₄ -terphenyl	0.90
<u>Region 6</u>	
d ₁₂ -perylene	1.0
<hr/>	

* IS - Internal Standard.

the predicted response. If this criterion is not met, the test will be repeated using a fresh calibration standard. Alternatively, a new calibration curve will be prepared.

3

5.3.5 Calibration Procedures

1. The instrument will be calibrated using five calibration (working) standards when it has failed to pass verification.
2. A laboratory control sample will then be analyzed. If the reported values are within control limits of the expected values, analysis will proceed.
3. All instrument operating conditions and quality control results will be entered in the instrument logbook.

3

5.4 PCDD/PCDF BY METHOD 8290

Samples submitted for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans analysis will be processed according to Method 8290, U.S. EPA EMSL (Las Vegas), a method for the determination of tetra-, penta-, hepta, and octa-chlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) in chemical wastes including still bottoms, fuel oils, sludges, fly ash, reactor residues, soil and water.

-2

Method 8290 uses a matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques.

Because of the extreme toxicity of these compounds, the analyst must take necessary precautions to prevent exposure to himself, or to others, of materials known or believed to contain PCDDs or PCDFs.

5.4.1 Sample Preparation and Extraction

Fly ash samples are extracted by placing a sample (e.g. 10g) and an equivalent amount of anhydrous sodium sulfate in a Soxhlet extraction apparatus charged with 100 mL of toluene (benzene) and extracted for 16 hours using a three cycle per hour schedule. The toluene extract is cooled and filtered through a glass fiber filter paper into a 500 mL round bottom flask. The filter is then rinsed with 5 mL of toluene. The combined toluene solution is then concentrated to near dryness and transferred to a separatory funnel with hexane.

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The solvent is partitioned with a basic solution and an acidic solution and placed on an alumina column and eluted with methylene chloride in hexane. The sample is then cleaned up on a carbon column.

Aqueous samples are extracted with methylene chloride for a 25-hour period then dried through anhydrous sodium sulfate. Samples are concentrated on a water bath and the solvent exchanged to hexane. The solvent is then partitioned and cleaned as described above.

5.4.2 GC/MS Analysis

Response factors are calculated for standard relative to 7 internal standards. The recovery standards ($^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD) are added to the samples prior to injection. The TCDD standard is used to determine the percent recoveries of the tetra- and pentachlorinated PCDD/PCDF congeners while the HxCDD standard is used for the determination of hexa, hepta and octa-chlorinated PCDD/PCDF congeners percent recoveries. The concentration of the recovery standard in the sample extract will be the same as that in the calibration standards used to measure the response factors. The GC operating conditions and temperature programs are listed in Table 5-9. The high resolution calibration solution concentrations are listed in Table 5-10.

5.4.3 Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 5.4.4) does not meet the required criteria. All seven high-resolution concentration calibration solutions listed in Table 5-10 will be used for the initial calibration. The instrument will be tuned with PFK molecular leak to meet the minimum resolving power of 10,000. The instrument is then injected with 2 μL of the GC column performance check solution to acquire the SIM mass spectral data. The total cycle time must be less than or equal to 1 second. The laboratory will not perform any further analysis until it is demonstrated and documented that the criterion listed in the reference method are met.

Using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, a 2- μL portion of each of the seven concentration calibration solutions is then analyzed.

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TABLE 5-9. RECOMMENDED GC OPERATING CONDITIONS
FOR METHOD 8290

Column coating	DB-5
Film thickness	0.25 μ m
Column dimension	60 m x 0.32 mm
Injector temperature	270°C
Splitless value time	45 s
Interface temperature	Function of the final temperature

TEMPERATURE PROGRAM

Stage	Init. Temp. (° C)	Init. Hold. Time (min)	Temp. Ramp (° C/min)	Fin. Temp. (° C)	Fin. Hold. Time
1	200	2	5	220	16
2			5	235	7
3			5	330	5
Total time: 60 min					

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TABLE 5-10. HIGH RESOLUTION CALIBRATION SOLUTIONS CONCENTRATIONS

Compound	HRCC	Concentration (pg/uL)						
		7	6	5	4	3	2	1
<u>Unlabeled Analytes</u>								
2,3,7,8-TCDD		200	100	50	25	10	5	2.5
2,3,7,8-TCDF		200	100	50	25	10	5	2.5
1,2,3,7,8-PeCDD		200	100	50	25	10	5	2.5
1,3,4,7,8-PeCDF		200	100	50	25	10	5	2.5
2,3,4,7,8-PeCDF		200	100	50	25	10	5	2.5
1,2,3,4,7,8-HxCDD		500	250	125	62.5	25	12.5	6.25
1,2,3,6,7,8-HxCDD		500	250	125	62.5	25	12.5	6.25
1,2,3,7,8,9-HxCDD		500	250	125	62.5	25	12.5	6.25
1,2,3,4,7,8-HxCDF		500	250	125	62.5	25	12.5	6.25
1,2,3,6,7,8-HxCDF		500	250	125	62.5	25	12.5	6.25
1,2,3,7,8,9-HxCDF		500	250	125	62.5	25	12.5	6.25
2,3,4,6,7,8-HxCDF		500	250	125	62.5	25	12.5	6.25
1,2,3,4,6,7,8-HpCDD		500	250	125	62.5	25	12.5	6.25
1,2,3,4,6,7,8-HpCDF		500	250	125	62.5	25	12.5	6.25
1,2,3,4,7,8,9-HpCDF		500	250	125	62.5	25	12.5	6.25
OCDD		1,000	500	250	125	50	25	12.5
OCDF		1,000	500	250	125	50	25	12.5
<u>Internal Standards</u>								
¹³ C ₁₂ -2,3,7,8-TCDD		50	50	50	50	50	50	50
¹³ C ₁₂ -2,3,7,8-TCDF		50	50	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDD		50	50	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8-PeCDF		50	50	50	50	50	50	50
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD		125	125	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF		125	125	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD		125	125	125	125	125	125	125
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF		125	125	125	125	125	125	125
¹³ C ₁₂ -OCDD		250	250	250	250	250	250	250
<u>Recovery Standards</u>								
¹³ C ₁₂ -1,2,3,4-TCDD (a)		50	50	50	50	50	50	50
¹³ C ₁₂ -1,2,3,7,8,9-HxCDD (b)		125	125	125	125	125	125	125

(a) Used for recovery determinations of TCDD, TCDF, PeCDD and PeCDF internal standards

(b) Used for recovery determinations of HxCDD, HxCDF, HpCDD, HpCDF, and OCDD internal standards.

- o The criteria listed below for acceptable calibration must be met before the analysis is performed.
- o The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from each of the 26 determinations (17 for the unlabeled standards and 9 for the labeled reference compounds) must be less than 20 percent.
- o The S/N for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 2.5 .
- o The isotopic ratios must be within the specified control limits.

5.4.4 Routine Calibration (Continuing Calibration Check)

Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks. A routine calibration is also required at the end of a 12-hour shift.

The routine calibration is completed by injecting 2 μL of the concentration solution HRCC-3 containing 10 $\text{pg}/\mu\text{L}$ of tetra- and pentachlorinated congeners, 25 $\text{pg}/\mu\text{L}$ of hexa- and heptachlorinated congeners, 50 $\text{pg}/\mu\text{L}$ of octachlorinated congeners, and the respective internal and recovery standards (Table 5-10).

The measured RRFs [RRF(n) for the unlabeled standards] obtained during the routine calibration runs must be within 20 percent of the mean values established during the initial calibration.

The following criteria will be met before further analysis is performed. If these criteria are not met, corrective action will be taken.

The measured RRFs [RRF(m) for the labeled standards] obtained during the routine calibration runs must be within 20 percent of the mean values established during the initial calibration.

The ion-abundance ratios must be within the allowed control limits.

If either of the first two above criteria is not satisfied, the entire initial calibration process must be repeated. If the ion-abundance ratio is not satisfied corrective action will be taken.

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5.5 CHLORINATED HERBICIDE ANALYSIS BY METHOD 8150

Chlorinated Herbicide analyses on the kiln ash/cyclone ash composite and the purge waters will be performed according to Method 8150 of U.S. EPA SW-846, a GC/ECD Method for determining the concentration of 2,4-D, 2,4-DB, 2,4,5-T, and 2,4,5-TP. | 3

5.5.1 Summary of Method

Method 8150 provides extraction, esterification, and gas chromatographic conditions for the analysis of chlorinated acid herbicides. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using the diazomethane as the derivatizing agent. After excess reagent is removed, the esters are determined by gas chromatography employing an electron capture detector, microcoulometric detector, or electrolytic conductivity detector (Goerlitz and Lamar, 1967). The results are reported as the acid equivalents.

Based upon the specific herbicides to be quantified, a Type 1a GC column will be used, as defined in the method. The GS operating conditions for the herbicide analyses are listed in Table 5-11.

5.5.2 External Calibration Standards

Calibration standards at a minimum of five concentration levels for each analyte of interest will be prepared through dilution of the stock standards with diethyl ether. One of the concentration levels will be at a concentration near, but above, the method detection range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem. | 2

The results will be used to prepare a calibration curve for each analyte or if appropriate, the ratio of response to amount injected or calibration factor (CF) will be calculated for each analyte at each standard calculation. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20% over the working range, linearity through the range will be assumed and the average calibration factor can be used in place of the calibration curve. | 2

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TABLE 5-11. GC OPERATING CONDITIONS FOR
CHLORINATED HERBICIDE ANALYSES

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GC Conditions

Column	1.8m x 4mm ID glass, packed with 1.5% SP-2250/1.95% SP-2410 on Supelcoport (100/200 mesh)
Temperature	185°C isothermal
Column flow	5% methane/95% argon at 70 ml/min

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$$CF = \frac{\text{Total Area of Peak or Peaks (multiple response)}}{\text{Mass injected (ng)}}$$

5.5.3 Calibration Verification

The working calibration curves or calibration factors will be verified on each working day by injection of one or more calibration standards. If the response for any analyte varies by more than $\pm 15\%$ from the predicted response, a new calibration curve will be prepared for that analyte.

$$\text{Percent Difference} = \frac{R_1 - R_2 \times 100}{R_1}$$

where:

R_1 = Calibration factor from first analysis.

R_2 = Calibration factor from succeeding analyses.

5.5.4 Surrogate Standards

The analyst will monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with dicamba herbicide. Deuterated analogs of analytes will not be used as surrogates for gas chromatographic analysis due to coelution problems.

5.6 ORGANOCHLORIDE PESTICIDES AND PCBs BY METHOD 8080

Samples of the purge water (downstream and upstream of the carbon filters), kiln ash/cyclone ash composites, and the separator sludge will be sent to the laboratory for analysis of the delisting requirement pesticides and PCBs by Method 8080 of SW-846. For Method 8080 the compounds of interest are Aldrin, Chlorodane, Endosulfan I, and total PCBs which are calculated as the sum of all PCB Arochlor mixtures contained in the method.

Method 8080 provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides and PCBs. Prior to the use of this method, solid matrices will be extracted using either Method 3540 or 3550. The purge water will be extracted at a neutral or as is pH with methylene chloride using either Method 3510 or 3520. The extraction solvent is then exchanged to hexane and the extract is concentrated prior to injection into a gas chromatograph. The extract will be analyzed by direct

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injection. A 2- to 5-uL sample is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD) or a halogen specific detector (HSD).

The analysis will be performed with a gas chromatograph suitable for on-column injections and all required accessories. The columns and detectors to be used are as follows:

Column 1: Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8-m x 4-mm I.D. glass column or equivalent.

Column 2: Supelcoport (100/120 mesh) coated with 3% OV-1 in a 1.8-m x 4-mm I.D. glass column or equivalent.

Detectors: Electron capture (ECD) or halogen specific (HSD) (i.e., electrolytic conductivity detector).

5.6.1 External Calibration

15

Calibration standards at a minimum of five concentration levels for each parameter of interest are prepared through dilution of the stock standards with isooctane. One of the concentration levels will be at a concentration near, but above, the method detection limit. The remaining concentration levels will correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions will be replaced after six months, or sooner, if comparison with check standards indicates a problem.

-5

5.6.2 Surrogate standards

The analyst will monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with pesticide surrogates. Because GC/ECD data are much more subject to interference than GC/MS, a secondary surrogate is to be used when sample interference is apparent. Dibutylchlorodate (DBC) is also subject to acid and base degradation. Therefore, two surrogate standards are added to each sample; however, only one need be calculated for recovery. DBC is the primary surrogate and should be used whenever possible. However, if DBC recovery is low or compounds interfere with DBC, then the 2,4,5,6-tetrachloro-meta-xylene should be evaluated for acceptance. Corrective action will be taken when both

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surrogates are out of limits for a sample as defined in Method 8080. Method 3500 indicates the proper procedure for preparing these surrogates.

5.7 POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs) BY METHOD 8310

Samples of the purge waters, and kiln ash/cyclone ash composites will be sent to the laboratory for analysis of the specified delisting requirement PAHs by Method 8310 of SW-846. Method 8310 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of PAHs. For Method 8310 the compounds of interest are listed in Table 1-1.

Prior to analysis, solid matrices will be extracted using either Method 3540 or 3550. The purge water will be extracted at a neutral pH with methylene chloride using either Method 3510 or 3520. The extraction solvent is then exchanged to acetonitrile with a Kuderna-Danish (K-D) procedure and the extract is concentrated prior to injection into a gas chromatograph.

The extract will be analyzed by direct injection. A 2- to 5-uL aliquot of the extract is injected into a gas chromatograph, using the solvent flush technique, and compounds in the GC effluent are detected by a fluorescence detector.

The analysis will be performed with a gas chromatograph suitable for on-column injections and all required accessories. The columns and detectors to be used are as follows:

Fluorescence Detector: For excitation at 280-nm and emission greater than 389-nm cutoff (Corning 3-75 or equivalent). Fluormeters will have dispersive optics for excitation and will utilize either filter or dispersive optics at the emission detector.

5.7.1 External Calibration

Calibration standards at a minimum of five concentration levels for each analyte of interest are prepared through dilution of the stock standards with acetonitrile. One of the concentration levels will be at a concentration near, but above, the method detection limit. The remaining concentration levels will correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions will be replaced after six months, or sooner, if comparison with check

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standards indicates a problem. Calibration factors are to be generated and verified as discussed in Sections 5.5.2 and 5.5.3.

5.7.2 Surrogate standards

The analyst will monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with decafluorobiphenyl and anthracene or other PAH not expected to be present in the sample. These surrogates encompass the range of temperature program to target analytes. Deuterated analogs of analytes will not be used due to coelution problems.

5.8 ORGANOPHOSPHOROUS PESTICIDES BY METHOD 8140

Samples of the purge waters, and kiln ash/cyclone ash composites will be sent to the laboratory for analysis of the delisting requirement by Method 8140 of SW-846. For Method 8140 the compounds of interest are Dichlorvos, Disulfaton, and Methyl parathion.

Method 8140 provides gas chromatographic conditions for the detection of ppb levels of organophosphorous pesticides. Prior to analysis, appropriate sample extraction techniques will be used for the solid matrices. The purge water will be analyzed by direct injection. A 2- to 5-uL aliquot of the extract is injected into a gas chromatograph, and compounds in the GC effluent are detected with a flame photometric or thermionic detector.

The analysis will be performed with a gas chromatograph suitable for on-column injections and all required accessories. The columns and detectors to be used are as follows:

Column: 1.8-m x 2-mm I.D. glass, packed with 5% SP-2401 on Supelcoport, 100/120 mesh (or equivalent)

Detector: Phosphorous-specific, Nitrogen/Phosphorous (N/P) operated in the phosphorous sensitive mode or Flame Photometric (FPD)

5.8.1 External Calibration

Calibration standards at a minimum of five concentration levels for each analyte of interest are prepared through

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dilution of the stock standards with acetonitrile. One of the concentration levels will be at a concentration near, but above, the method detection limit. The remaining concentration levels will correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions will be replaced after six months, or sooner, if comparison with check standards indicates a problem. Calibration factors are to be generated and verified as discussed in Sections 5.5.2 and 5.5.3.

5.7.2 Surrogate standards

The analyst will monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with tetrachlorvinphos, dimeton O, and ethoprop.

5.9 CALIBRATION OF CRITICAL COMBUSTION PARAMETER MEASUREMENT SYSTEMS

The critical combustion parameter measurements are the kiln and SCC exit temperatures, the waste solid and liquid feed rates and the combustion gas monitoring systems. The calibration procedures and frequency are detailed below.

5.9.1 Temperature Monitoring System

The thermocouple converter and recorder are calibrated by inputting a millivolt signal which corresponds to a given temperature signal according to ANSI Standard Thermocouple Tables. The output signal is then adjusted to a proper temperature reading. The temperature monitoring system cannot be calibrated during the operation of the mobile incinerator, so calibration of the instrument will be done before the start of the testing program. If the calibrations show a significant drift (greater than 2%) in the accuracy of the temperature monitor, the deviation will be recorded and the instrument recalibrated.

5.9.2 Contaminated Waste Organic Liquid Flow Rate

The EXAC Model #2100 unit will be field-calibrated with fuel oil. Calibration will be done by gravimetric changes in a separate feed container with time. The liquid waste feed rate will be manually recorded by the kiln operator every 15 minutes. The total flow will be recorded to confirm that the instantaneous flow rates are consistent. The periodic

recording of flow rates will be used to evaluate the steady-state operation of the incinerator before and during each test.

5.9.3 Waste Solids Feed Rate

The weigh cell will be calibrated according to the manufacturer's specifications, and the cycle time will be checked with a watch prior to the start of the testing program.

5.9.4 CO, CO₂, NO_x, THC and O₂ Stack and SCC Gas Monitors

The gas chromatographs, chemiluminescent detector analyzer, nondispersive infrared analyzer, and the polarographic detector analyzer will be calibrated by selectively opening valves on certified gas standards that direct calibrated gas mixtures into the sampling probe assemblies on the incinerator. The calibration gas passes through the same sample conditioning and transfer systems to the analyzers as the actual incinerator samples. This technique not only calibrates the analyzers but corrects for losses that can occur during sample conditioning. The calibration of the analyzers generates a four-point calibration curve for each gas component using a zero gas (pure nitrogen) and three standard gases. Approximate concentrations of standard gases are listed in Table 5-12. The four-point calibration will be performed prior to the start of the testing program for CO, CO₂, O₂, and NO_x. Zero and span calibration gases will be analyzed daily for CO, CO₂, O₂, and NO_x or will be analyzed during each run if the tests are run consecutively. If the zero-span gas analysis data show an analyzer response drift greater than 5%, a new four-point calibration will be performed. The gas mixtures used for calibration will be certified gas standards traceable to the National Bureau of Standards where possible. The concentrations of the calibration standard gases used will be close to and will span the expected values in the gas stream.

Both the primary and backup monitoring systems must be operational and calibrated before a test is started. Failure of one system during a test, however, will not be a cause for interrupting a sampling run because both systems have equal status in activating alarms, safety interlocks, or waste feed shutdown and will provide the required gas composition data.

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TABLE 5-12 Approximate Concentrations of
Calibration Gases

<u>Constituent</u>	<u>Zero</u>	<u>Low</u>	<u>Mid</u>	<u>Span</u>
Carbon monoxide (vppm)	0	25	90	500
Total hydrocarbons (vppm)	0	10	50	100
Oxygen (vol %)	0	4.5	8.0	18.0
Carbon dioxide (vol %)	0	5.0	13.0	19.5

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Preparing, obtaining approval of, and distributing the QA Project Plan for the mobile incinerator operations.

Managing the incinerator test operations and sampling.

Monitoring to see that field QC and sampling procedures are carried out.

Deciding when test run sampling must be interrupted or terminated.

Reviewing operating logs, stack sampling report, and analytical reports for completeness and transferring all test data and reports to the project manager.

Making weekly telephone calls to the three analytical laboratories to monitor performance and progress.

Assisting in the interim test burn report preparation.

The site supervisors have the responsibility for the field operation of the mobile incinerator according to directives during their shift. Some of the site supervisor's responsibilities are:

Assuring that equipment and instrument calibrations are performed and recorded.

Assuring that the process conditions (temperatures, pressures, flowrates, etc.) established for the test are set and maintained.

Ensuring that the required operating data are recorded, collected, and delivered to the test coordinator.

Monitoring and coordinating with the sample coordinator the taking of all test samples.

The test burn sample coordinator, M. Merdinger, has the overall responsibilities for field activities pertaining to sample collection, handling, packaging, and shipping. The analytical laboratory will supply the required supplies such as the resin columns for the stack sampling, sample containers, and field sample preservation reagents. Some of the test burn sample manager's responsibilities are:

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Ensuring that appropriate sampling equipment, sample containers, and field blanks are supplied by the subcontractor at the test site.

Reviewing the assignment and recording of sample numbers.

Directing sample collection activities.

Overseeing sample preservation in the field.

Documenting sampling activities in field log book.

Ensuring chain of custody of samples in the field.

Overseeing the preparation samples for shipping and shipping them to Region VII Laboratory.

The remaining members of the test burn field test team are identified in Table 2-2 along with their lines of responsibility.

The analytical laboratory directors and the laboratories lines of responsibility are shown on Table 2-3. The laboratory analysis coordinator, has the overall responsibility for the test burn samples and sample analyses. Some of the analysis coordinator's responsibilities include:

Training and qualifying laboratory personnel in the analytical procedures specified in the test burn plan, prior to receiving the test samples.

Directing the distribution of the samples for analyses.

Monitoring and verifying that the specified analytical and QC procedures are being followed.

Insuring that analytical personnel have the required information to perform the analyses in accordance with this Quality Assurance Project Plan.

Reviewing the analytical and QC data as soon as each major group is analyzed and notifying the project manager if data quality appears to warrant repeat analyses of some or all samples.

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If for any reason, any personnel assigned to this project are lost to it before completion, they will be replaced with others of equal or better qualifications.

Byproduct streams will be sampled by Enviresponse. Region VII Laboratory will be the prime subcontractor for analytical activities. Reporting requirements of the subcontractors to the main contractor, Enviresponse, Inc., are included in Section 6.0. | 4

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Table 2-2. Personnel List and Line of Responsibility for MIS
Operation and Sampling During Test Burns

Position	Person
R&D Group Manager/EPA Liaison	G. Gupta
Project Manager	R. Sawyer
Assistant Project Manager	J. Tichansky
Test Coordinator	J. Stumbar
Site Supervisors	E. Russell H. Gragg 14 J. Irwin 13 S. Jones 13
Incinerator/APC System Operators	T. Miller 13 R. L. Turner 13 J. Collins 13 R. B. Turner 13
Waste Feed Handlers	a
Ash Handlers	a
Electrical/Utility	D. Weigel 14 C. Webb 14 J. Wheeler 14 P. Wilson 14
Monitoring System Operator	TBN 14 G. Tieman 13 K. Schaedel 13 G. Thompson 13
Sample Coordinator	M. Merdinger 1
Field Team Leader	M. Merdinger/ 1 J. Stumbar 3

a Local personnel trained and experienced in handling same or similar hazardous wastes, and who are under a medical surveillance program.

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Table 2-3 Personnel List and Line of Responsibility for Test Burn Sample Analyses

Position	Person	
MIS Project Manager	R. Sawyer	
Prime Analytical Laboratory Region VII		
Analytical Laboratory Director	Dr. Harold Brown	14
Analysis Coordinator	William Bunn	
Volatiles	Diane Easley	
Herbicides	Audra Gier	
Dioxins/Furans/Semivolatiles	Dr. Tenkasi S. Viswanathan	
Metals	Dr. Harold Brown	-3
Subcontracting Laboratory - TBN		3
Analytical Laboratory Director	TBN	
Analysis Coordinator	TBN	
Organo phosphate pesticides	TBN	1

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3.0 QUALITY ASSURANCE OBJECTIVES

3.1 PRECISION, ACCURACY AND COMPLETENESS

The collection of data to characterize the waste feed, kiln and cyclone ash, purge water and stack gas requires that sampling and analysis procedures be conducted with properly operated and calibrated equipment by trained personnel. Precision and accuracy goals for this program are presented in Tables 3-1 and 3-2. Table 3-1 presents precision, accuracy and completeness goals for field measurements which consist of process measurements and flue gas composition measurements. Table 3-2 lists the goals for analysis of process stream samples.

Precision is defined as the degree of mutual agreement among measurements made under prescribed conditions. Accuracy is the degree of agreement of a measurement with an accepted reference or true value. Completeness is defined as the percent of samples judged to be valid compared to the total number of samples collected. Every attempt will be made to have all data generated to be valid data. However, realistically, some samples may be lost in laboratory accidents and some results may be deemed questionable based on internal QC procedures. Enviresponse anticipates that some 10 percent of the recovery values will be outside the QC limits owing to matrix interferences. In the event of gross matrix interferences, revised QA objectives will be submitted for approval. The objective will be to have 90 percent of the data valid.

3.2 REPRESENTATIVENESS AND COMPARABILITY

It is recognized that the usefulness of the data is also contingent upon meeting the criteria for representativeness and comparability. Wherever possible, reference methods and standard sampling procedures will be used. The QA objective is that all measurements be representative of the media and operation being evaluated. The detailed requirements for the continuous monitoring, EPA Methods (3A, 6C, 7E, 10, and 25A), will be followed to ensure representative sampling of flue gases. The frequent grab sampling of ash and the composite sampling of process water during each test run will provide representative samples of these media.

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TABLE 3-1. QUALITY ASSURANCE OBJECTIVES FOR
COMBUSTION AND OPERATING PARAMETERS

Parameter	Method of Measurement	Precision (%) ^a	Accuracy (%) ^b	Completeness (%) ^c
Rotary kiln gas temperature	Thermocouple	5	5	90
SCC temperature	Thermocouple	5	5	90
Waste oil flow rate	Electronic flow meter	5	5	90
Waste soil flow rate	Weigh cell	10	10	90
CO	Continuous non-dispersive infrared analyzer ^d	15 ^e	15 ^e	90
CO ₂	Gas chromatograph/thermal conductivity detector	5	5	90
O ₂	Continuous polarographic detector analyzer ^f	5	5	90
NO _x	Chemiluminescent detector analyzer	10 ^e	10 ^e	90

^aExpressed in terms of the relative standard deviation as defined in Section 9.0.

^bExpressed as the percentage difference from the true (standard) value.

^cExpressed as the amount of valid data obtained compared to the total amount expected.

^dBackup is a methanizer coupled to a gas chromatograph/flame ionization detector.

^eBased on results of the previous trial burn, the level of CO and NO_x in the combustion gases was very low. Precision and accuracy will need to be with $\pm 15\%$ or 5 ppm for CO and $\pm 10\%$ or 10 ppm for NO_x, whichever is greater.

^fBackup is a gas chromatograph/thermal conductivity detector.

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TABLE 3-2. ANALYTICAL QA OBJECTIVES FOR PRECISION AND ACCURACY(a)

Parameter	Matrix	Precision (RPD)	Accuracy (% Recovery)	Spiking Level ug/L or ug/kg
Volatiles(b)				
Acetone	Aqueous Solid(d)	≤ 25(c) NA	50-150(c) NA	500 0
Benzene	Aqueous Solid	≤ 20 ≤ 21	76-127 66-142	50 50
Chlorobenzene	Aqueous Solid	≤ 20 NA	75-130 NA	50 0
Chloromethane (methyl chloride)	Aqueous Solid(d)	≤ 37 NA	5-204 NA	500 0
1,2-Dichloroethane	Aqueous Solid	≤ 20 ≤ 22	61-145 59-172	50 50
Ethylbenzene	Aqueous Solid(d)	≤ 20 NA	59-141 NA	50 0
Dichloromethane (methylene chloride)	Aqueous Solid	≤ 20 ≤ 25	24-140 14-150	50 50
Trichloromethane (chloroform)	Aqueous Solid	≤ 20 ≤ 25	27-133 17-143	50 50
Toluene	Aqueous Solid(d)	≤ 20 NA	76-125 NA	50 0
Tetrachloroethylene	Aqueous Solid	≤ 20 ≤ 20	74-127 64-137	60 60
Semi-volatiles(b)				
2,4-Dichlorophenol	Aqueous Solid	≤ 40 ≤ 50	27-123 25-102	50 1600
Naphthalene	Aqueous Solid(d)	≤ 25 NA	36-120 NA	50 0
Fluorene	Aqueous Solid	≤ 20 ≤ 25	72-110 62-120	50 1600

(cont.)

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TABLE 3-2. ANALYTICAL QA OBJECTIVES FOR PRECISION AND ACCURACY^(a) (cont)

Parameter	Matrix	Precision (RPD)	Accuracy (% Recovery)	Spiking Level ug/L or ug/kg
2,4,6 Trichlorophenol	Aqueous	≤ 30	52-129	50
	Solid	≤ 35	42-139	1600
Pentachlorophenol	Aqueous	≤ 50	9-103	50
	Solid(d)	NA	NA	0
Phenanthrene	Aqueous	≤ 20	65-109	60
	Solid	≤ 25	55-119	1600
Pyrene	Aqueous	≤ 31	26-127	50
	Solid(d)	NA	NA	0
1,4-Dichlorobenzene	Aqueous	≤ 28	36-97	50
	Solid(d)	NA	NA	0
1,2,4-Trichlorobenzene	Aqueous	≤ 28	39-98	50
	Solid(d)	NA	NA	0
Benzo(b)fluoranthene	Aqueous	≤ 26	42-140	50
	Solid	≤ 31	32-150	1600
PAHs (b)				
Benzo(a)pyrene	Aqueous	≤ 38	5-128	10
	Solid	≤ 43	5-138	400
Benzo(b)fluoranthene	Aqueous	≤ 20	6-150	10
	Solid	≤ NA	NA	NA
Chrysene	Aqueous	≤ 30	5-159	10
	Solid	≤ NA	NA	NA
Dibenzo(a,h)anthracene	Aqueous	≤ 25	5-110	10
	Solid	≤ 30	5-120	100
Indeno(1,2,3-c,d)pyrene	Aqueous	≤ 29	5-116	10
	Solid	≤ NA	NA	NA

(cont.)

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2 -5
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TABLE 3-2. (cont.)

Parameter	Matrix	Precision (RPD)	Accuracy (% Recovery)	Spiking Level ug/L or ug/kg
Herbicides(b)	Aqueous	≤ 25	50-130	100
	Solid	≤ 25	50-130	250
PCDD/PCDFs(b,f)	Aqueous	≤ 25	40-120	--
	Solid	≤ 25	40-120	--
PCBs (as Arochlors) and organochlorine pesticides	Aqueous	≤ 20	70-130	5
	Solid	≤ 25	50-150	50
Organophosphorous pesticides	Aqueous	≤ 20	40-140	20
	Solid	≤ 25	40-140	500
Metals(e)	Aqueous	≤ 15	85-115	
	Solid	≤ 20	80-120	
pH	Aqueous	≤ 10	$\pm 10\%$	
Total Suspended/ Dissolved Solids	Aqueous No matrix spike or matrix spike duplicate required			
Cyanides (c)	Aqueous	≤ 20	80-120	
	non-aqueous	≤ 30	70-130	

NOTE: All precision and accuracy objectives are based upon historical laboratory results.

a - Accuracy will be measured as percent recovery of a matrix spike compounds. Precision will be estimated as the relative percent difference of a matrix spike duplicate.

(cont.)

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TABLE 3-2. (cont.)

b - Precision and accuracy targets were determined as follows:

When applicable values were taken from tables for water and soil MS/MSD recovery in Chapter 1 of the 3rd Ed. SW-846.

Minimum precision of 20% was assumed to be adequate since most compounds will be nondetect and proof of the ability to recover compounds present in the matrices is the most important QC objective.

For constituents not listed in the above tables, the data, presented in Tables 6 and 7 of the SW Methods 8240 & 8270 write-ups, and in Table 3 and 4 of the SW Method 8310 write-up were used to estimate target values for water.

Target values for soil were obtained from water target values by adding 5% to the precision value and increasing the range of recoveries by 20%.

c - Targets are estimated.

d - NA means that the constituent will not be spiked in the solid matrix because, analysis is not required for delisting purposes.

e - Accuracy will be determined as percent recovery of matrix spikes when appropriate or the percent recovery of a QC sample if spiking is inappropriate. Precision will be determined as relative percent difference of duplicate spikes, or duplicate sample if spiking is inappropriate.

f - From PCDD/PCDF spiking requirements see Table 5-10.

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The combustion parameters for which representativeness could be a problem are the rotary kiln and SCC gas temperatures and the flue gas component concentrations. The thermocouples for temperature measurement will be shielded from flame radiation and will have combustion gases circulated around them. The sampling points for flue gas compounds will be located in the center of the ducts. At test conditions the gas flow at the sample points will be turbulent, ensuring complete mixing.

3.3 METHOD DETECTION LIMITS

The objectives for the method detection limits (MDL) for the flue gas component monitors are listed in Table 3-3. These detection limits have been selected based upon instrument vendor information, system analysis range and prior operating experience. Since the MDL for these instruments are significantly below any system alarm/shutdown condition or typical operating conditions, the verification of these MDL's will not be included as part of this testing program.

The method detection limits for the various analytical methods are listed in Table 3-4. The method detection limits for dioxin analyses will be calculated using the procedure given in Method 8290. For other analytes, method detection limits will be determined by the procedure given in Section 1.3 of the third edition of SW-846. The method detection limits for PCDD and PCDF analysis are listed in Table 3-5.

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6.0 DATA REDUCTION, VALIDATION AND REPORTING

Extensive QC measures will be used to ensure the generation of reliable data from sampling and analysis activities. Proper collection and organization of accurate information followed by clear and concise reporting of the data is a primary goal in all projects.

6.1 FIELD DATA REDUCTION

Appendix C of this QA Plan presents the standardized forms that will be used to record field data. The data collected will be reviewed in the field by at least two field crew members. Errors or discrepancies will be noted in the field log book. Flue gas data will be treated as follows:

- o Continuous Monitoring Data--the monitoring data will be reduced and presented in terms of 30 minute averages for the parameters listed below:

- Oxides of nitrogen--ppm, lb/hr
- Carbon monoxide--ppm, lb/hr
- Carbon dioxide--percent
- Oxygen--percent
- Total Hydrocarbons--ppm, lb/hr

6.2 LABORATORY ANALYSIS DATA REDUCTION

Analytical results will be reduced to concentration units specified by Enviresponse, Inc. or the analytical procedure, using the equations given in the analytical procedures. If units are not specified, data from the analysis of water samples will be converted to units of mg/L using the following equation:

$$X_w = X_v \times W \times DF / V_s$$

where X_w = reported value, mg/L

X_v = reported sample value, mg/kg

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V_s = sample volume, L
DF = dilution factor
W = sample weight, kg

Data from the analysis of gas samples will be reported as $\mu\text{g}/\text{m}^3$. This will be calculated by dividing the total weight of the substance detected by the volume of gas sampled.

The concentration of compounds identified in a sample will be calculated using either the internal standard or external standard method. The internal standards are similar in analytical behavior to compounds of interest. The calculated concentrations is based upon the chromatographic peak areas and response factors obtained from the calibration standards according to the following equation:

$$C_x = \frac{(A_x \ C_{is} \ DF)}{(A_{is} \ RF \ V_s)} \quad \text{for aqueous samples}$$

$$C_x = \frac{(A_x \ C_{is} \ DF)}{(A_{is} \ RF \ W)} \quad \text{for solid samples}$$

where: C_x = concentration of analyte x in $\mu\text{g-C/L}$ (aqueous or $\mu\text{g} - \text{c/kg}$ (solid))
 A_x = peak area for analyte x
RF = response factor for the appropriate quantitative standard in area counts $\mu\text{g-C/L}$. (RF is defined in Section 5).
DF = dilution factor.

The concentration of compounds identified by the external standard method will be determined by calculating the amount of standard purged or injected from the peak response using the calibration curve or the calibration factor given in Section 5.0 according to the following equations:

$$C_x = \frac{(A_x \ C_s \ V_t \ DF)}{(A_s \ V_i \ V_s)} \quad \text{for aqueous samples}$$

$$C_x = \frac{(A_x \ C_s \ V_t \ DF)}{(A_s \ V_i \ W)} \quad \text{for solid samples}$$

where: A_x , C_x , V_s , W , and DF are defined above.

C_s = Amount of standard injected or purged, ng.

V_t = Volume of total extract, uL.

V_i = Volume of extract injected, uL.

2

The data generated will include the following information:

- o Compound; and
- o Concentration.

6.3 DATA VALIDATION

6.3.1 Combustion Parameters

Backup measurements will help validate the primary data. The total contaminated solids fed during each test will be measured by counting the number of bags of pesticide fed and multiplying by the weight of each bag. This backup measurement for contaminated solids quantity used must agree within $\pm 10\%$ of the primary measurement during the test to be valid.

The change in the contaminated organic liquid waste feed tank level during the test will be measured with a dip stick as a check on the primary mass flow readings. The backup measurement for these feeds must agree within $\pm 10\%$ to meet the test objective. Failure to meet this objective will be discussed in the final report.

-4

6.3.2 Analytical Parameters

Data validation is the process of filtering data and accepting or rejecting it on the basis of sound criteria. Laboratory supervisory and QC personnel will use validation methods and criteria appropriate to the type of data and the purpose of the measurement. Records of all data will be maintained, even

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that judged to be an "outlying" or spurious value. The persons validating the data will have sufficient knowledge of the technical work to identify questionable values.

Field sampling data will be validated by the Field Team Leader and/or the Field QC Coordinator based on their judgment of the representativeness of the sample, maintenance and cleanliness of sampling equipment and the adherence to an approved, written sample collection procedure.

Region VII Laboratory (ENSV) will evaluate all analytical data. Analytical subcontractor, the CLP laboratory, will provide data deliverables required of EPA Contract Laboratory Program laboratories. Appendix A presents requirements for general deliverables, and additional method deliverables for: (1) PCDD/PCDF, (2) GC/MS, (3) GC, and (4) ICP/Furnace methods.

Analytical data will be validated by the laboratory QC or supervisory personnel using criteria outlined in the QA Project Plan. The laboratory uses results from field and laboratory method blanks, replicate samples and internal QC samples to validate analytical results. Analytical results on field blanks and replicate field samples are valuable for validation of sample collection also.

The following criteria will be used to evaluate the field sampling data:

- o Use of approved test procedures
- o Proper operation of the process being tested
- o Use of properly operating and calibrated equipment
- o Leak checks conducted before and after tests
- o Use of reagents that have conformed to QC specified criteria
- o Proper chain-of-custody maintained

The criteria listed below will be used to evaluate the analytical data:

- o Use of approved analytical procedures
- o Use of properly operating and calibrated instrumentation

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- o Acceptable results from analyses of QC samples (i.e., the reported values should fall within the 95 percent confidence interval for these samples)
- o Precision and accuracy achieved should be comparable to that achieved in previous analytical programs and consistent with objectives stated in Section 3.0 of this QAPP

6.4 IDENTIFICATION AND TREATMENT OF OUTLIERS

Any data point which deviates markedly from others in its set of measurements will be investigated; however, the suspected outlier will be recorded and retained in the data set while it is investigated. One or both of the following tests will be used to identify outliers.

Dixon's test for extreme observations is an easily computed procedure for determining whether a single very large or very small value is consistent with the remaining data. The one-tailed t-test for difference may also be used in this case. It should be noted that these tests are designed for testing a single value. If more than one outlier is suspected in the same data set, other statistical sources will be consulted and the most appropriate test of hypothesis will be used and documented.

Since an outlier may result from unique circumstances at the time of sample analysis or data collection, those persons involved in the analysis and data reduction will be consulted. This may provide an experimental reason for the outlier to determine its effect on the conclusions. In many cases, two data sets will be reported, one including, and one excluding the outlier.

In summary, every effort will be made to include the outlying values in the reported data. If the value is rejected, it will be identified as an outlier, reported with its data set and its omission noted.

6.5 DATA REPORTING

6.5.1 Combustion Parameters

The incinerator operators' log sheets and the continuous monitoring system operators' logs will be used for reporting data to the project manager. Calibration check results will be recorded in the "Remarks" sections of the log sheets. The results of calibration checks for all instruments will be reported along with the data in the final report.

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6.5.2 Figure 6-1 shows the sampling, analysis, and reporting responsibilities for the demonstration test.

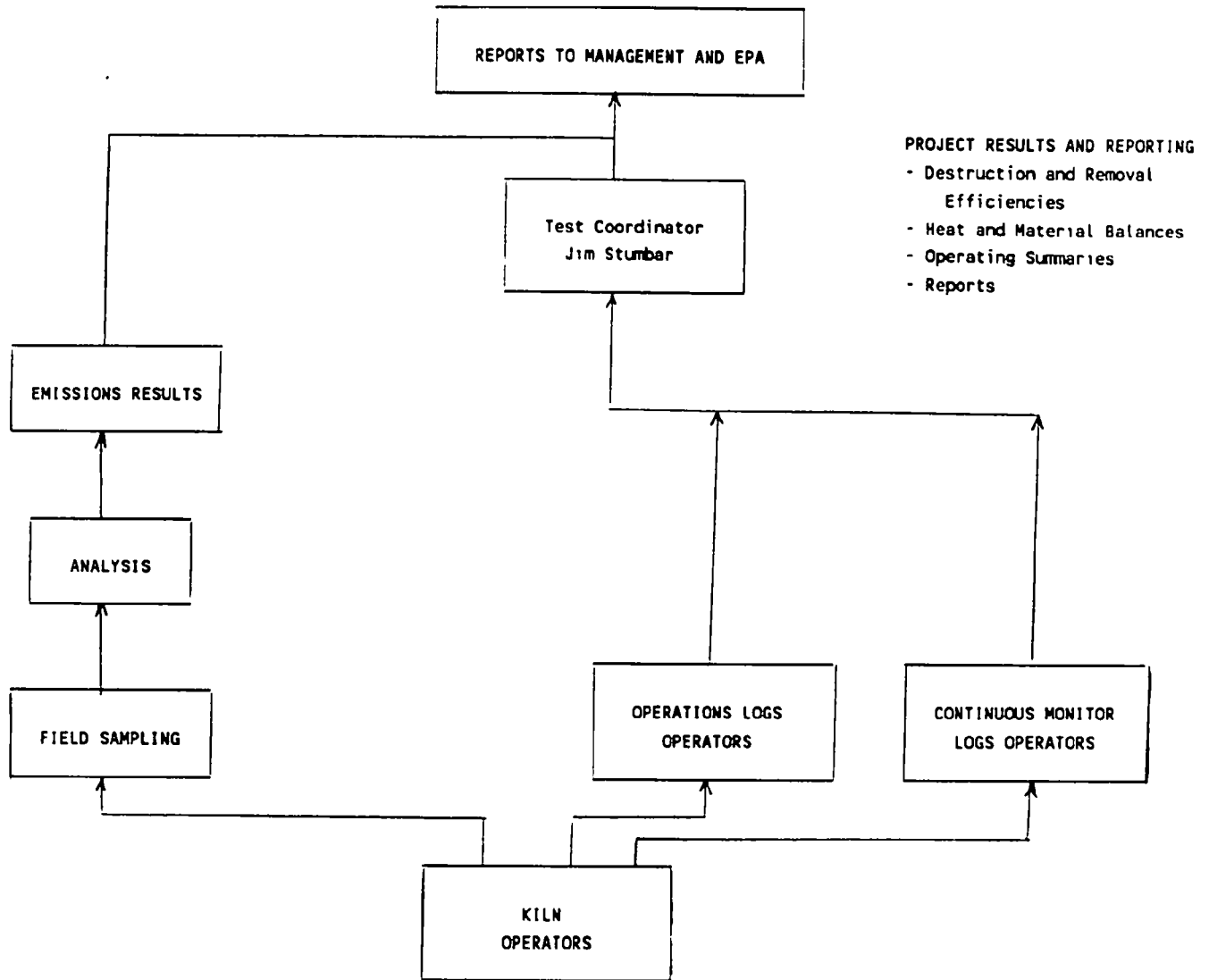
6.5.3 Analytical Data

A flow chart depicting a proposed measurement data reporting scheme is shown in Figure 6-2. Figure 6-3 presents the analytical data validation and reporting scheme routinely used at the laboratory. The Work Assignment Organization Chart identifies the supervisory and QC personnel who will validate the data.

All data will be reported in standard units depending on the measurement and the ultimate use of the data. The bulk of the data will be computer processed and reported as follows:

- o Continuous monitoring data (30 minute averages)
 - Oxides of nitrogen--to nearest 1.0 ppm NO_x
 - Carbon dioxide--to nearest 1.0 percent CO₂
 - Oxygen--to nearest 1.0 percent O₂
 - Carbon monoxide--to nearest 1.0 ppm CO
 - Dry molecular weight--to nearest tenth gram
 - Total Hydrocarbons--to nearest 1.0 ppm

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PROJECT RESULTS AND REPORTING

- Destruction and Removal Efficiencies
- Heat and Material Balances
- Operating Summaries
- Reports

SOURCE SAMPLING AND ANALYSIS

- Stack Gas Sampling
- Waste Feed Composition
- Kiln and Cyclone Ash
- Scrubber Liquid
- Lab Audit Samples

OPERATIONS AND MONITORING

- System Conditions
- Waste Feed Flow
- Combustion Temperature (continuous monitoring)
- Field Audit Samples

FIGURE 6-1 DATA FLOW AND REPORTING SCHEME

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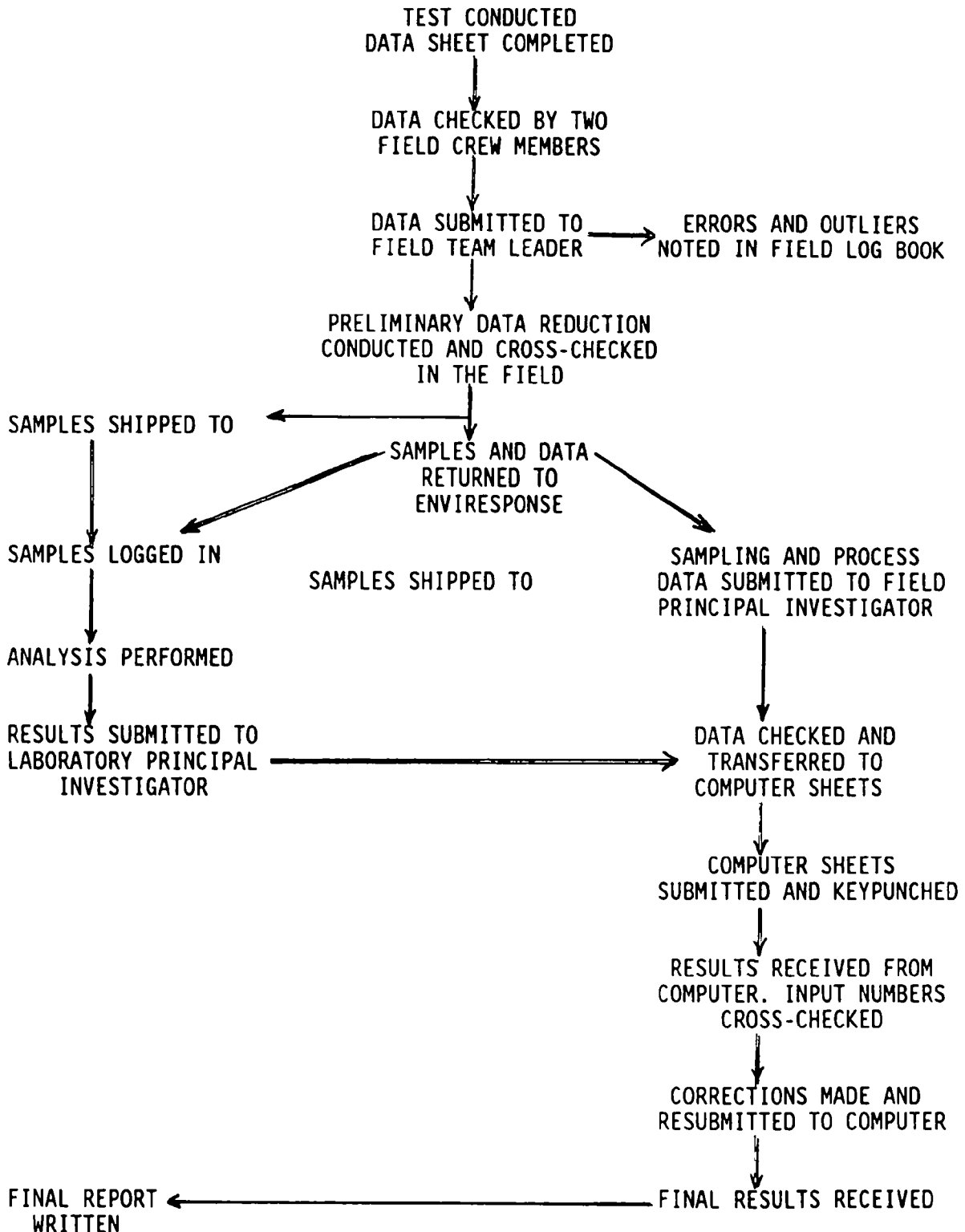


FIGURE 6-2. MEASUREMENT DATA FLOW SCHEME

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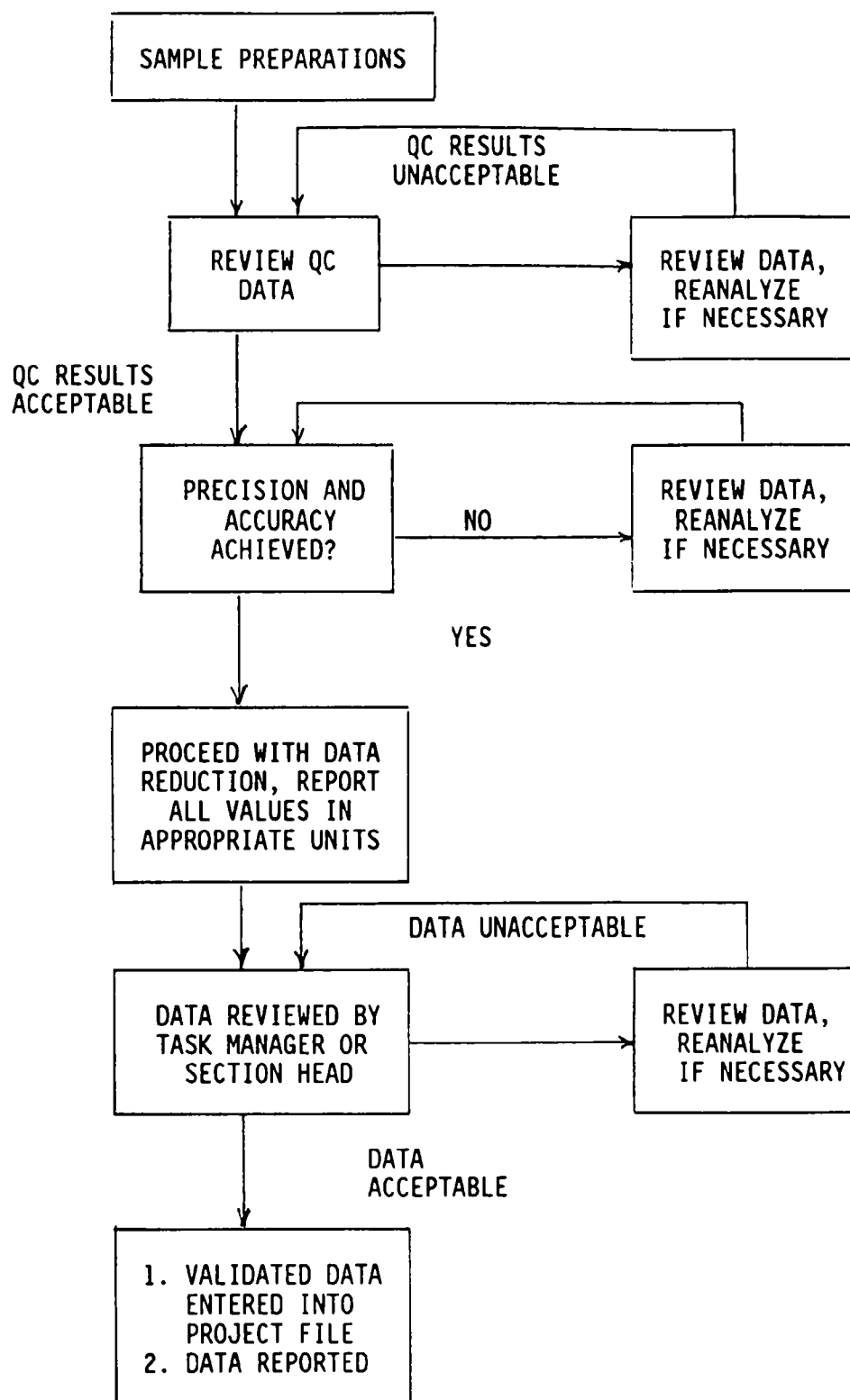


FIGURE 6-3 ANALYTICAL DATA REPORTING SCHEME

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7.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Quality control checks will be performed to ensure the collection of representative samples and the generation of valid analytical results on these samples. These checks will be performed by project participants through the program under the guidance of the QA Director and Field and Laboratory QC Coordinators.

7.1 DATA COLLECTION AND SAMPLING QC PROCEDURES

Subcontractors' QC checks for the process data collection and sampling aspects of this program will include, but not be limited to, the following:

1. Use of standardized checklists and field notebooks to ensure completeness, traceability, and comparability of the process information and samples collected.
2. Field checking of standardized forms by a second person to ensure accuracy and completeness.
3. Strict adherence to the sample chain-of-custody procedures outlined in Section 5.0 of the QAPP.
4. Submission of field biased blanks.

Duplicate samples will not be collected during the project because the scoping burn does not require this level of QC and the preproduction burn essentially gives four duplicate samples because the unit operating conditions will be the same over the entire preproduction burn.

7.1.1 Sampling Equipment QC Checks and Frequency

Calibration of the field sampling equipment will be performed prior to and at the conclusion of the field sampling effort. Copies of the calibration sheets will be submitted to the field team leader to take on site for reference, and to the

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project file. Calibrations will be performed as described in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Method;" Section 3 presents acceptance limits.

Leak checks of the sample trains will be conducted in accordance to the protocol called out for each method. Leak checks will be conducted prior to and at the end of sample collection.

7.1.2 Span Drift Check of Continuous Monitors

The continuous monitoring equipment will be checked at the beginning and end of each test period by inputting a combination span gas through the probe for analysis. The responses obtained from these span checks will be reduced into units of concentration using the appropriate calibration equation. These data will be plotted on a control chart to determine if a significant drift has occurred. These limits were determined by using the 24-hour drift criteria (contained in PST No. 2: 2.5 percent of span). Data falling outside the chart limits will necessitate corrective actions.

7.1.3 Sample Collection QC Checks

Field-biased blanks of reagent and collection media (deionized water, etc.) will be placed in appropriately cleaned and sized sample containers in the field and handled in the same way as actual field samples, to provide a QC check on sample handling.

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Sample collection QC checks and frequency for samples to be analyzed in the laboratory are listed below:

- o Scrubber Water Sampling:
 - One field-biased blank (i.e., DI water) for the first sampling day for volatile organic compounds.
- o Ash Sampling:
 - No duplicates.

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Method blanks will also be provided with the various sample sets. These blanks are portions of analytical reagents, filters, and recovery reagents set aside and not transported to the field. Method blanks will be obtained for each reagent and sampling media lot.

Field duplicate samples will not be included as QC samples for this project because they are inappropriate and unnecessary. Duplicated samples are inappropriate because previous sampling history has shown that most compounds are none detected. For such a case the use of matrix spikes and matrix spike duplicates provides the necessary estimates of accuracy and precision. Duplicate samples are unnecessary because the preproduction runs consist of tests which are essentially under the same process conditions. Hence, the repetition of four tests should provide the "duplicate field samples".

7.2 ANALYTICAL QC PROCEDURES FOR SAMPLES TO BE ANALYZED IN THE LABORATORY

7.2.1 Quality Control Samples and Blanks

The Quality Control program for laboratory analysis makes use of a number of different types of QC samples to document the validity of the generated data. The following types of QC samples are used routinely:

- 7.2.1.1 Method Blanks - Method blanks contain all the reagents used in the preparation and analysis of samples and are processed through the entire analytical scheme to assess spurious contamination arising from reagents, glassware, and other materials used in the analysis.
- 7.2.1.2 Calibration Check Samples - One of the working calibration standards which is periodically used to check that the original calibration is still valid.
- 7.2.1.3 Replicate Samples and Spiked Samples - Analysis of replicate samples is used to enable estimation of the precision of the analytical procedure. Replicate aliquots of project samples are spiked with components of interest and carried through the entire preparative and analytical scheme.

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- 7.2.1.4 Laboratory Control Samples (LCS) - These samples are prepared from EPA EMSL concentrates or NBS standard reference materials. The LCS are used to establish that an instrument or procedure is in control. An LCS is normally carried through the entire sample preparation and analysis procedure also.
- 7.2.1.5 Surrogate Spikes - Samples requiring analysis by GC/MS are routinely surrogate-spiked with a series of deuterated analogues of the components of interest. It is anticipated that these compounds would assess the behavior of actual components in individual program samples during the entire preparative and analysis scheme.
- 7.2.1.6 "Blind" Quality Control Samples - Blind quality control samples are inserted in the sample load in a fashion unrecognizable to the analyst. These samples may be blanks, duplicate or spiked project samples, or prepared or purchased reference material samples.
- 7.2.1.7 Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
A MS/MSD pair will be run for each different matrix analyzed. These pairs will be spiked with the target compounds of concern for that matrix (e.g., 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP and PCDD/PCDF for ash samples).

All values which fall outside the QC limits, given in Table 3-2, and described in the Analytical Method will be noted. Enviresponse anticipates that some of these recovery values will be outside the QC limit owing to matrix interferences. The following guidelines will be used:

1. All recovery data are evaluated to determine if the QC limits are appropriate and if a problem may exist even though the limits are being achieved (e.g., one compound that is consistently barely within the lower limit).
2. All recovery data which are outside the established limits are evaluated. This evaluation will include an independent check of the calculation.
3. Corrective action will be performed if any of the following are observed:

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- o All recovery values in any one analysis are outside the established limits,
- o Over 50 percent of the values for a given sample set are outside limits, or
- o One compound is outside the limits in over 50 percent of the samples.

The type and frequency of use of each of these QC measure is listed in Table 7-1. An analysis batch is defined as a group of 10 or fewer samples carried through the entire preparation and analysis procedure in one batch.

Reagents used in the laboratory are normally of analytical reagent grade or higher purity; each lot of acid or solvent used is checked for acceptability prior to lab use. All reagents are labeled with the date received and ate opened. The quality of the laboratory deionized water is routinely checked. All glassware used in the sampling and analysis procedures will be precleaned according to the method requirements. Standard laboratory practices for laboratory cleanliness, personnel training and other general procedures are used.

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TABLE 7-1. ANALYTICAL QC MEASURES

QA Measure	Minimum frequency
Method Blank	Each sample set or every 10 samples
Laboratory Duplicate sample	Each sample set or every 10 samples per matrix
Spiked sample	Each sample set or every 10 samples per matrix when spiking is possible
Laboratory control sample	Each sample set or every 10 samples
Calibration check sample	Daily
Surrogate spike (GC/MS analysis)	Each sample
Matrix Spike/Matrix Spike Duplicate	Each matrix for the preproduction burn as given in Table 1-1 for the scoping burn

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8.0 QUALITY ASSURANCE PERFORMANCE AND SYSTEM AUDITS

The laboratory's Quality Assurance Program includes both performance and system audits as independent checks on the quality of data obtained from sampling, analysis, and data gathering activities. Every effort is made to have the audit assess the measurement process in normal operation. Either type of audit may show the need for corrective action.

8.1 PERFORMANCE AUDITS

The sampling, analysis, and data handling segments of a project are checked in performance audits. Blind QC samples are used or a different operator/analyst performs these audit operations to ensure the independence of the quantitative results.

EPA Quality Control Concentrates and NBS Standard Reference Materials will be used to assess the analytical work. The Chemistry Division QC Coordinator will direct the inclusion in the sample load of QC samples appropriate to the analyses performed in each batch of 20 or fewer samples so that they are not recognizable to the analyst. In addition, any appropriate interlaboratory study samples which are available during this program will be analyzed to further audit the analytical work.

8.2 SYSTEM AUDITS

A system audit is a qualitative review to ensure that the quality measures outlined in the Project QA Plan are in place. The Director of Quality Assurance selects projects representing different types of measurement activities for audit by the QA staff; this project may be audited under that policy (to ensure the integrity of system audits, it is not known in advance whether such an audit will be performed). If it is, a written audit report will be submitted as noted in Section 11.0.

8.3 EXTERNAL AUDITS

The laboratory will cooperate fully in any performance of system audits conducted or arranged by EPA or Enviroresponse. The QA Director and Division QC Coordinators are available to aid in scheduling such audits.

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9.0 CALCULATION OF DATA QUALITY INDICATORS

9.1 PRECISION

9.1.1 Reference Method Testing and Analytical Measurements

Precision will be determined through the analysis of replicate or replicate spiked samples and will be expressed as either relative standard deviation (RSD) or relative percent difference (RPD). The following equations will be used when three or more measurements are made:

$$RSD = 100 \left(\frac{s}{\bar{x}} \right)$$

where \bar{x} = arithmetic mean
 s = standard deviation

Standard deviation will be determined as follows:

$$s = \sqrt{\frac{\sum_{i=1}^N x_i^2 - \frac{1}{N} \left(\sum_{i=1}^N x_i \right)^2}{N - 1}}$$

x_i = individual measurement
 N = number of measurements

Precision of duplicate analyses will be calculated as follows:

$$RPD = \frac{\text{Value 1} - \text{Value 2}}{\text{Arithmetic mean of values 1 and 2}} \times 100$$

9.1.2 Continuous Monitoring System

Precision will be estimated from the periodic span check data for each monitor using mid-span Manufacturer's Certified gases

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as noted in section 7.1. The following equations will be used to estimate precision from at least five data points:

Percent difference (d_i)

$$d_i = \frac{(Y_i - X_i)}{X_i} \quad (100) \quad (100)$$

where: Y_i = Monitor indicated concentration from the i -th span check.

X_i = The span check reference concentration for the i -th precision check.

Mean percent difference (\bar{d}_j)

$$\bar{d}_j = \frac{1}{n} \sum_{i=1}^n d_i$$

where: n = number of valid precision checks made during the test period j .

Standard deviation of the percent difference (S_j)

$$S_j = \sqrt{\frac{1}{n-1} \sum_{i=1}^n d_i^2 - \frac{1}{n} \left(\sum_{i=1}^n d_i \right)^2}$$

Upper and lower 95 percent probability limits (UPL and LPL) will be computed as follows:

$$UPL = \bar{d}_j + 1.96 S_j$$

$$LPL = \bar{d}_j - 1.96 S_j$$

9.2 ACCURACY

9.2.1 Reference Method Tests and Analytical Measurements

Accuracy will be estimated from the analysis of matrix spiked samples and/or laboratory control samples, whose true values are known to the analyst, and the analysis of "Blind" QC samples whose values are known to the Chemistry Division QC Coordinator. Accuracy will be expressed as percent recovery or as relative error. The formulas to calculate these values are:

$$\text{Percent Recovery} = 100 \times \frac{(C_m - C_s)}{C_{sp}}$$

where:

C_m = Measured value in spiked sample

C_s = Measured value in sample

C_{sp} = Known value of spike added

$$\text{Relative Error} = 100 \times \left(\frac{\text{Measured Value} - \text{True Value}}{\text{True Value}} \right)$$

9.2.2 Continuous Monitoring System

Relative accuracy will be estimated using blind audit cylinders or comparison with Reference test procedures. The cylinder value will be used as the reference value. The following equations will be used.

The arithmetic differences will be computed as follows:

$$X_i = C_m - C_r$$

where: X_i is the the difference between the current concentration and previous calibration value

C_m is the monitor indicated concentration

C_r is the reference value concentration

Next, the arithmetic mean of the individual differences will be calculated:

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$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n}$$

where: \bar{X} is the mean of the difference

X_i is the individual differences

n is the number of data points

The confidence interval at the 95 percent confidence level will be calculated as follows:

$$CI_{95} = \frac{t_{.975}}{n(n-1)} \sqrt{n \sum_{i=1}^n X_i^2 - \left(\sum_{i=1}^n X_i \right)^2}$$

where: CI_{95} is the 95 percent confidence interval

$t_{.975}$ is a statistical "t factor"

n is the number of data points

X_i are the individual differences

The Relative Accuracy (R.A.) will be calculated from the preceding values:

$$R.A. = \frac{\bar{X} + [CI_{95}]}{C_r}$$

9.3 COMPLETENESS

Completeness will be reported as the percentage of all measurements made whose results are judged to be valid. The procedures to be used for validating data and determination of outliers are contained in Section 6.0 of this QA Plan. The following formula will be used to estimate completeness:

$$C = 100 \left(\frac{V}{T} \right)$$

where: C = percent completeness

V = number of measurements judged valid

T = total number of measurements

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10.0 CORRECTIVE ACTION

The acceptance limits for the sampling and analyses to be conducted in this program will be those stated in the method or defined previously in this QA Project Plan. The corrective actions are likely to be immediate in nature and most often will be implemented by the analyst or Project Manager; the corrective action will usually involve recalculation, reanalysis, or repeating a sample run. The laboratory's ongoing corrective action policy is described here.

10.1 IMMEDIATE CORRECTIVE ACTION

Specific QC procedures and checklists are designed to help analysts detect the need for corrective action. Often the person's experience will be more valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action can be taken at this point, as part of normal operating procedures, the collection of poor quality data can be avoided. Instrument and equipment malfunctions are amenable to this type of action and the laboratory's QC procedures include troubleshooting guides and corrective action suggestions. The actions taken should be noted in field or laboratory notebooks but no other formal documentation is required, unless further corrective action is necessary. These on-the-spot corrective actions are an everyday part of the QA/QC system.

Corrective action during the field sampling portion of a program is most often a result of equipment failure or an operator oversight and may require repeating a run. When equipment is discovered to be defective (i.e., pre- and post-sampling leak check) it is repaired or replaced and a corrective factor is established as per the EPA method. If a correction factor is unacceptable the run is repeated. Operator oversight is best avoided by having field crew members audit each others' work before and after a test. Every effort is made by the field team leader to ensure that all QC procedures are followed. Economically, it is preferred to repeat a run during a particular field trip rather than return at a later date.

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Corrective action for the continuous monitors will involve constructing a new 3-point calibration equation. In order to minimize the time that the analyzer is offline, the span gases will be sampled intermittently (e.g., zero, flue gas, mid span, flue gas, high span).

Corrective action for analytical work would include recalibration of instruments, reanalysis of known QC samples and, if necessary, of actual field samples.

If the problem is not solved in this way, more formalized long-term corrective action may be necessary.

10.2 LONG-TERM CORRECTIVE ACTION

The need for this action may be identified by standard QC procedures, control charts, performance or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category. The laboratory uses a system to ensure that the condition is reported to a person responsible for correcting it who is part of the closed-loop action and follow-up plan.

The essential steps in the closed-loop corrective action system are:

- o Identify and define the problem.
- o Assign responsibility for investigating the problem.
- o Determine a corrective action to eliminate the problem.
- o Assign and accept responsibility for implementing the corrective action.
- o Establish effectiveness of the corrective action and implement it.
- o Verify that the corrective action has eliminated the problem.

Documentation of the problem is important to the system. A Corrective Action Request Form (shown in Figure 10-1) is filled out by the person finding the quality problem. This form identifies the problem, possible causes and the person responsible for action on the problem. The responsible person may be an analyst, field team leader, Division QC coordinator or the QA Director. If no person is identified as responsible for action, the QA Director investigates the situation and determines who is responsible in each case.

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CORRECTIVE ACTION REQUEST FORM NO. _____

Originator _____ Date _____

Person Responsible _____ Contract
for Replying _____ Involved _____

Description of problem and when identified: _____

State cause of problem, if known or suspected: _____

Sequence of Corrective Action: (If no responsible person is identified, notify QA Director immediately. Submit all CA forms to QA Director for initial approval of CA.)

State Date, Person, and Action Planned: _____

CA Initially Approved By: _____ DATE _____

Follow-up Dates _____

Final CA Approval By: _____ DATE _____

Information copies to:

Responsible Person/Division QC Coordinator: _____

QA Director: _____

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The Corrective Action Request Form includes a description of the corrective action planned and the date it was taken, and space for follow-up. The QA Director checks to be sure that initial action has been taken and appears effective and, at an appropriate later date, checks again to see if the problem has been fully solved. The QA Director receives a copy of all Corrective Action Forms and then enters them in the Corrective Action Log. This permanent record aids the QA Director in follow-up and makes any quality problems visible to management; the log may also prove valuable in listing a similar problem and its solution.

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11.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

11.1 INTERNAL REPORTS

The Chemistry Division QC Coordinator and the Air and Process Engineering Division QC Coordinator prepare written monthly reports on QC activities for their Division Manager and the QA Director. These reports detail the results of quality control procedures, problems encountered and any corrective action which may have been required.

All Corrective Action Forms are submitted to the QA Director for initial approval of the corrective action planned and a copy is provided to the Division Manager. All system audit reports are provided to the project manager, Division Manager and the laboratory President.

11.2 REPORTS TO CLIENT

Each data transmittal will contain a summary of QA/QC activities; this summary will include:

- o Estimates of precision, accuracy and completeness of reported data
- o Reports of performance and system audits
- o Quality Control problems found
- o Corrective actions taken

The final report will include a section summarizing QA/QC activities during the program. The Chemistry and Air and Process Engineering Division QC Coordinators and the QA Director will participate in preparing this section.

APPENDIX A
DELIVERABLES REQUIRED FROM ANALYTICAL SUBCONTRACTORS

APPENDIX B

PROCEDURES FOR COMPOSITING PURGE WATER SAMPLES FOR VOLATILE ORGANIC ANALYSES

APPENDIX C
FIELD DATA SHEETS

APPENDIX D
COMMENTS ON REGION VII VARIATION OF METHOD 8290

COMMENTS ON REGION VII VARIATION OF METHOD 8290

From: Robert D. Kloepper, Ph.D.
Chief, Laboratory Branch, ENSV

Date: April 1, 1988

The comments of ESAT chemist, Mr. Saleh Arghestani on the differences between the 8290 and Region VII methods for the analysis of PCDDs and PCDFs is attached. I discussed the status of the RCRA method 8290 with Mr. Werner Beckert (EMSL-LV/EPA) on March 10, 1988. He informed me that this method is still in the single laboratory evaluation stage and that the projected completion date for this activity is September 1988. Apparently, the information that appeared in the March 11 Federal Register on this method is inaccurate. A few changes are also proposed for this method after this evaluation. The method will be subjected to a multi-laboratory evaluation in 1989. Although the Region VII method has not been tested on a multi-laboratory basis, it has been peer-reviewed by a number of scientists with expertise in this area and has been used for the analysis of up to 30 batches of environmental samples in the last 18 months.

APPENDIX A

DELIVERABLES REQUIRED FROM ANALYTICAL SUBCONTRACTORS

GENERAL DELIVERABLES

Please note that some elements are method, technique, or parameter specific and, therefore, additional deliverables may be required. Additional deliverables will be outlined on method/parameter specific attachments to this document.

I. PACKAGE NARRATIVE

1. Method(s) used (citations acceptable if commonly available)
2. Summary of problems and/or decision tree processes utilized to complete the analyses.

II. QUALITY CONTROL

1. Listing of the in-house control limits used as a basis for corrective action for each of the QC elements.
2. Summary of QC results for reagent blanks, field blanks, matrix spikes, duplicates, and laboratory control standards analyzed.
 - a. Matrix Spike: Tabulated results of % recovery
 - b. Duplicates: Tabulated results for \overline{X} SD or \overline{X} RPD
 - c. Lab Control Standard: Tabulated results and true values
 - d. Method Blank: Tabulated result or non-detected value
3. Raw QC data chromatograms, quantitation reports, instrument readout records, ICP/AA printouts, strip chart recordings, ~~or~~ RICs or logbook entries of meter readings. All raw data must be clearly labeled with the sample ID number and the parameter.

III. SAMPLE DATA PACKAGE

1. Holding time information (date/time sampled and analyzed)
2. Sample results summary
3. Raw sample data chromatograms, instrument readout records, RICs, ICP/AA printouts, strip chart recordings, or logbook entries of meter readings. All raw data must be clearly labeled with the sample ID number and the parameter.
4. Quantitation reports
 - a. Amount of sample aliquots
 - b. Dilution factors used
 - c. Calculations used to determine sample results
5. Percent solids determination for soil data

IV. STANDARDS DATA PACKAGE

1. Listing of laboratory detection limits
2. Initial calibration data
3. Continuing calibration check data
4. Raw standards' data chromatograms, quantitation reports, *R*_{ics}, instrument readout records, ICP/AA printouts, strip chart recordings, or meter readings. All standards' raw data must be clearly labeled as to parameter and concentration they represent.

These deliverables are in addition
to the General Deliverables.

ADDITIONAL
PCDD/PCDF Method Deliverables

I. SAMPLE DATA PACKAGE

1. Listing of ions monitored for each compound along with area responses found, if any, per sample.

II. STANDARDS DATA PACKAGE

Initial Calibration:

1. List of retention time (RT) windows established for each homolog group for both dioxins and furans along with ions monitored for each compound.
2. All Selected Ion Monitoring (SIM) reconstructed ion chromatograms and ion responses for each analyte in the initial calibration analyses. All raw data must be clearly labeled as to parameter and concentration they represent.
3. SIM chromatograms for the performance check mixture.
4. % Valley tabulation for resolution between 2,3,7,8- and 1,2,3,4-TCDD.
5. Signal to noise for m/z 320 (2,3,7,8-TCDD) for the low level calibration standard.
6. Tabulated RRF for each compound in the initial calibration along with the average RRF and %RSD calculated based results from the multiple point initial calibration.

Routine Calibration:

1. SIM chromatograms for the performance check mixture.
2. % Valley tabulation for resolution between 2,3,7,8- and 1,2,3,4-TCDD.
3. SIM chromatograms and ion responses for the routine calibration analysis.
4. Tabulated RRF for each compound plus % Difference between initial calibration RRFs and routine calibration RRFs.

SPECIAL NOTE

For 2,3,7,8-substituted PCDD/PCDF analysis (versus total PCDD/PCDF plus 2,3,7,8-TCDD), we also require that each of the 2,3,7,8-PCDD/PCDF analytes be analyzed in the calibration standard and that RT windows for each analyte be submitted.

ADDITIONAL
GC/MS Method Deliverables

I. QUALITY CONTROL

1. Summary of QC results for surrogate compounds.
 - a. Tabulated % recovery for each surrogate used.
2. Summary of internal standard responses.
 - a. Listing of area response for each internal standard compound in the daily calibration solution as well as area responses for the internal standard compounds in each associated sample analysis.
3. Summary of ion abundances for tuning compounds.
 - a. Tabulated ion abundance results for BFB (volatiles) and/or DFTPP (semi-volatiles).
 - b. Raw data mass listing of ion abundances.

II. SAMPLE DATA PACKAGE

1. Mass spectra for all positive hits including reference spectrum.

III. STANDARDS DATA PACKAGE

Initial Calibration:

1. Tabulated RRFs for each compound plus average RRF and %RSD for multiple point initial calibration.

Continuing Calibration:

1. Tabulated RRF for each compound plus % Difference between initial calibration RRF and daily calibration RRF.

ADDITIONAL
GC Method Deliverables

I. QUALITY CONTROL

1. Summary of QC results for surrogate compounds
 - a. Tabulated % recovery for each surrogate used.

II. SAMPLE & STANDARD DATA PACKAGE

1. Summary of instrument operating conditions, temperature programs, and column type or packing.
2. Second or confirmatory column chromatograms and integration printouts for all samples having positive hits for compounds of interest including all standard runs.
3. All chromatograms must be clearly labelled with the sample number and any peaks representing compounds of interest must be labelled with the compound name. A corresponding integration printout must be submitted for each sample which also contains the sample number, retention time of all peaks, and peak area response. Each retention time representing a peak corresponding to a compound of interest must be so labelled on the integration printout.

ADDITIONAL ICP/Furnace Method Deliverables

I. QUALITY CONTROL

1. Tabulated results and raw data for all blanks including initial calibration blank, continuing calibration blank, and preparation blanks.
2. Tabulated results and raw data for ICP Interference Check Sample analysis.

II. SAMPLE DATA PACKAGE

Furnace AA:

1. Duplicate injection results for all furnace work.
2. MSA data results for all furnace parameters.
3. Tabulated correlation coefficients for MSA results.

APPENDIX B

PROCEDURES FOR COMPOSITING PURGE WATER SAMPLES FOR VOLATILE ORGANIC ANALYSES

PROCEDURE FOR THE COLLECTION OF A REPRESENTATIVE VOA SAMPLE DURING A 8-12 HOUR SAMPLING PERIOD FROM A SOURCE OF WATER THAT IS GREATER IN TEMPERATURE THAN AMBIENT

This method is written for use in the collection of a VOA sample from a source that is hot and representative of a time period of several hours.

EQUIPMENT

40ml VOA vials, cleaned and baked out and known to be free of contamination

1000ml Erlenmeyer flask, washed and baked out at 130 degrees centigrade for 1 hour and cooled to room temperature before use.

A refrigerator used for the cooling of the samples.

A source of organic free water to be used for blanks.

PROCEDURE

From a suitable exit port, quickly fill one 40 ml VOA vial completely. Cap with the teflon lined septum facing the water layer. Label with sample number, time and date of collection. Cool. Collect all required portions of the sample using the same sample number and recording the time and date. Cool all portions of the sample.

During the cooling of the sample, a bubble of air will form in the vial. This is normal and should not be of concern.

After the samples have been cooled, remove all portions of the sample from the refrigerator.

Composite in erlenmeyer flask by emptying the contents of all vials that make up the sample. Swirl 2 times and then quickly fill two clean 40 ml VOA vials full with the sample. Label and date, noting the time frame of the composite, eg 02/03/88, 3:00am to 4:00pm.

Pack the VOA vials in a plastic subcontainer with an extractor tube filled with activated charcoal and refrigerate.

Ship to the analytical laboratory with the appropriate paperwork.

CLEANING PROCEDURE FOR REUSABLE GLASSWARE

Rinse with tap water

Wash with hot water and detergent

Rinse with tap water

Rinse with distilled water

Bake in an oven for one hour at 130 degrees centigrade

Cool to room temperature before use

QA/QC Samples

One composite blank should be prepared for each set of 20 samples collected. This would mean filling the appropriate number of vials with organic free water. Refrigerating for the same number of hours as the samples. Compositing the sample, using the same glassware and method. Filling two 40ml VOA vials and shipping them for analysis. A "field blank" would consist of filling 2x40ml VOA vials with organic free water and shipping them to the analytical laboratory. This should be done with the first set of samples collected. If the "composite blank" is found to be contaminated, a "field blank" should be routine.

APPENDIX C
FIELD DATA SHEETS

TITLE _____

OPERATING PARAMETERS

Project No. _____

Book No. _____

From Page N _____

SHEET ____ 0

RUN NUMBER _____

DATE _____

DATA RECORDED BY _____

PARAMETER	UNITS	TIME					
BLOWDOWN TOTALIZER	GAL						
ID FAN SPEED	RPM						
ID FAN INLET PRESS	IN.W.C.						
QUENCH CIRC PH	-						
PROCESS WATER pH	-						
MX SCRUBBER pH	-						
MX RECIRC FLOWRATE	GPM						
MX RECIRC TEMP	°F						
WEP GAS INLET TEMP	°F						
WEP INLET PRESSURE	IN.W.C.						
QUENCH WATER FLOW	GPM						
WEP AC VOLTAGE	VOLTS						
WEP DC VOLTAGE	KV						
WEP AC AMPERAGE	AMPS						
WEP DC AMPERAGE	MAHP						
SCC FUEL BURNER B	GPH						
SCC PRESSURE	IN.W.C.						
ASH GATE TEMP	°F						
ASH TEMP	°F						
SCC FUEL BURNER A	GPH						
SCC COMB AIR TEMP	°F						
SCC COMB AIR FLOW	SCFM						
CHEMF INLET TEMP	°F						
WEP FLOWRATE FTS22	% of SCALE						

To Pa

Witnessed & Understood by me.

Date

Invented by

Date

TITLE _____

OPERATING PARAMETERS

Project No. _____

Book No. _____

From Page No. _____

SHEET _____ OF

RUN NUMBER _____

DATE _____

DATA RECORDED BY _____

PARAMETER	UNITS	TIME							
WASTE OIL TOTALIZER	LB								
WASTE OIL FLOW	LB/HR								
WATER INT. FLOW	GPM								
KILN PRESSURE	IN. W.C.								
KILN AIR TEMP	°F								
KILN COMB. AIR	SCFM								
WEP OUTLET FUE GAS	ACFM								
SCC TEMPERATURE	°F								
KILN O ₂ OUTLET	%								
KILN OXYGEN FLOW	MCFH								
FUEL TO O ₂ BURNER	LB/HR								
KILN TEMP. LINDE	°F								
KILN TEMP. CHART	°F								
WASTE OIL TOTALIZER	LB								
WASTE OIL FLOW	LB/HR								
WATER INT. FLOW	GPM								
KILN PRESSURE	IN. W.C.								
KILN AIR TEMP	°F								
KILN COMB. AIR	SCFM								
WEP OUTLET FUE GAS	ACFM								
SCC TEMPERATURE	°F								
KILN O ₂ OUTLET	%								
KILN OXYGEN FLOW	MCFH								
FUEL TO O ₂ BURNER	LB/HR								
KILN TEMP. LINDE	°F								
KILN TEMP. CHART	°F								

To Page

Witnessed & Understood by me.

Date

Invented by

Date