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**PILOT-SCALE INCINERATION TESTING OF
FLUFF WASTE AND CONTAMINATED SOIL
FROM THE M. W. MANUFACTURING
SUPERFUND SITE**

Volume I — Technical Results

by

**J. W. Lee, W. W. Vestal, S. Venkatesh, C. G. Goldman,
and L. R. Waterland
Acurex Environmental Corporation
Incineration Research Facility
Jefferson, Arkansas 72079**

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**Project Officer: R. C. Thurnau
Technical Project Manager: M. K. Richards
Sustainable Technology Division
National Risk Management Research Laboratory
Cincinnati, Ohio 45268**

**NATIONAL RISK MANAGEMENT RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268**

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FOREWORD

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E. Timothy Oppelt, Director
National Risk Management Research Laboratory

ABSTRACT

At the request of EPA Region III and U.S. Army Corps of Engineers, a series of six tests was conducted at the U.S. EPA Incineration Research Facility (IRF) to evaluate the incinerability of the fluff waste and contaminated soil from the M. W. Manufacturing Corporation Superfund site in Danville, Pennsylvania. Both materials are contaminated with volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), chlorinated dioxins and furans, and several trace metals, including antimony, arsenic, barium, cadmium, chromium, copper, lead, nickel, silver, and zinc. Copper and lead, in particular, are at very high concentrations in both the fluff and waste and contaminated soil. The fluff was incinerated at two kiln exit gas temperatures: nominally 871° and 760°C (1,600° and 1,400°F). The soil was incinerated only at the higher kiln exit gas temperature of 871°C (1,600°F). Each test was run in duplicate (i.e., two tests were performed for each incinerator feed/kiln temperature combination). The afterburner exit gas temperature for all tests was nominally at 1,090°C (2,000°F). The primary air pollution control system consisted of a venturi/packed column scrubber system followed by a flue gas reheater and baghouse.

Test results showed that greater than 99.99 percent DRE of the VOC and SVOC contaminants was uniformly achieved. HCl emissions were well below 1.8 kg/hr and system HCl control efficiencies well above 99 percent. Particulate emissions at the baghouse exit were well below 34 mg/dscm (0.015 gr/dscf) corrected to 7 percent O₂, a guideline level announced in the draft waste combustion strategy in May 1993. Baghouse exit flue gas total chlorinated dioxin/furan levels were well below 30 ng/dscm corrected to 7 percent O₂, another draft combustion strategy guideline. Incineration effectively decontaminated both the fluff waste and soil of their VOC and SVOC contaminants. However, the kiln ash discharge from the incineration of contaminated site soil at a kiln gas temperature of 871°C (1,600°F) contained total chlorinated dioxin/furan concentrations of 2.4 to 3.6 µg/kg. Levels in the kiln ash from fluff incineration at the same temperature were 65 to 89 µg/kg, and significantly increased, at 830 to 2,700 µg/kg, for incineration at a kiln gas temperature of 760°C (1,400°F). In addition, the flue gas particulate collected as baghouse ash for all tests was a cadmium- and lead-contaminated toxicity characteristic (TC) hazardous waste.

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SECTION 1

INTRODUCTION

One of the primary missions of the Environmental Protection Agency's (EPA's) Incineration Research Facility (IRF) is to support Regional Offices in evaluations of the potential of incineration as a treatment option for wastes and other contaminated materials at Superfund sites. One priority site is the M. W. Manufacturing site in Danville, Pennsylvania. EPA Region III and the U.S. Army Corps of Engineers (USACE) requested that a pilot-scale test program be conducted at the IRF to support evaluations of the suitability of incineration as a treatment technology for wastes and contaminated soil at the site.

The M. W. Manufacturing site began operation in 1966. M. W. Manufacturing Corporation reclaimed copper from scrap wire using both mechanical and chemical processes. Reclamation activities began in 1969 and continued until 1972 when M. W. Manufacturing filed for bankruptcy. The chemical recovery processes used by M. W. Manufacturing led to site contamination with volatile organic solvents. Warehouse 81, Inc., acquired the site in 1976 and began mechanical recovery operations from the existing waste piles onsite. The mechanical recovery operations generated large volumes of waste material, termed fluff.

The fluff waste produced by the mechanical stripping process consists of fibrous insulation material mixed with plastic. Phthalate esters, copper, and lead are the major contaminants in this material. The chemical recovery process used by M. W. Manufacturing was a two-step process. The first step involved the use of a hot oil bath to melt the plastic insulation

away from the metal in the scrap wire. Residual oils were removed from the separated copper in the second step through the use of chlorinated solvents, including trichloroethene and tetrachloroethene. Thus, these solvents are waste and soil contaminants at the site.

The June 1990 record of decision (ROD) document for the site identified five wastes and contaminated materials for remedial treatment:

- Fluff waste piles
- Organic- and trace-metals-contaminated surface soils
- Organic- and trace-metals-contaminated subsurface soils
- Lagoon water
- Contents of drums and tanks

Onsite incineration was identified as the selected treatment for the fluff and the soil, with possible stabilization of the incineration ash prior to landfill disposal. Other, non-incineration remedies were selected for treating the lagoon water and the drum/tank contents.

Region III requested the pilot-scale test program at the IRF to support the further progress of the remediation of the site, and specifically to supply data on optimum incineration conditions for both fluff waste and contaminated soil to the remediation design effort. The specific objectives of the IRF test program were defined as follows:

- Verify that the fluff waste and the contaminated soil at the site can be incinerated in compliance with the hazardous waste incinerator performance standards and permit requirements of:
 - 99.99 percent principal organic hazardous constituent (POHC) destruction and removal efficiency (DRE)
 - HCl emissions less than 1 percent of the air pollution control system (APCS) inlet flowrate or 1.8 kg/hr (4 lb/hr), whichever is greater

- CO emissions of less than 100 ppm at 7 percent O₂, 1-hour rolling average and the performance guidance announced in 1993 of:
 - Particulate emissions of less than 34 mg/dscm (0.015 gr/dscf) corrected to 7 percent O₂
 - Total tetra- through octa- polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran (PCDD/PCDF) emissions of less than 30 ng/dscm corrected to 7 percent O₂
- Measure the effectiveness of incineration treatment in decontaminating fluff and soil of their organic contaminants and evaluate whether incineration temperature affects the effectiveness of fluff decontamination
 - Measure the distribution of the contaminant metals in the fluff and the contaminated soil among the incineration system discharge streams
 - Determine whether the bottom ash residue and the APCS discharges from the incineration of fluff and contaminated soil will be toxicity characteristic (TC) hazardous wastes
 - Determine whether the bottom ash residue from the incineration of contaminated soil meets the cleanup levels for soil given in the ROD

To address these objectives, a series of seven tests was performed in the rotary kiln incineration system (RKS) at the IRF. Results of this test program are discussed in this report. Section 2 of the report describes the IRF's RKS in which the tests were performed. Section 2 also discusses the composition of the fluff waste and contaminated soil incinerated in the tests, and the test incinerator system operating conditions. The sampling and analysis procedures employed during the tests are discussed in Section 3. Section 4 presents the test results. Section 5 summarizes the test program conclusions. Section 6 discusses the quality assurance

(QA) aspects of the test program. The Appendices provide a complete data set from which information of interest can be extracted for further study.

SECTION 2

FACILITY DESCRIPTION, WASTE CHARACTERISTICS, AND TEST CONDITIONS

A description of the RKS is presented in Section 2.1. Section 2.2 describes the composition and characteristics of the fluff waste and contaminated soil as reported in earlier site remedial investigation reports. The test matrix and incinerator operating conditions are discussed in Section 2.3.

2.1 ROTARY KILN INCINERATION SYSTEM DESCRIPTION

A process schematic of the RKS is shown in Figure 2-1 and the system design characteristics are listed in Table 2-1. The RKS consists of a rotary kiln primary combustion chamber, a transition section, a fired afterburner chamber, and an afterburner extension for flue gas flow conditioning to allow isokinetic sampling of afterburner exit flue gas. After exiting the afterburner extension, flue gas flows through a quench section followed by a primary air pollution control system (APCS). The initial element of the primary APCS for these tests consisted of a venturi/packed column scrubber system which removes most of the coarse particulate and acid gas such as HCl in the flue gas. Downstream of the scrubber system, a 100-kW electric resistance heater reheats the flue gas to about 120°C (250°F) which is about 22°C (40°F) above the saturation temperature. A fabric-filter baghouse downstream of the reheater removes most of the remaining flue gas particulate. Reheating the flue gas prevents moisture condensation in the baghouse, which if allowed to occur, would adversely affect baghouse operation. The flue

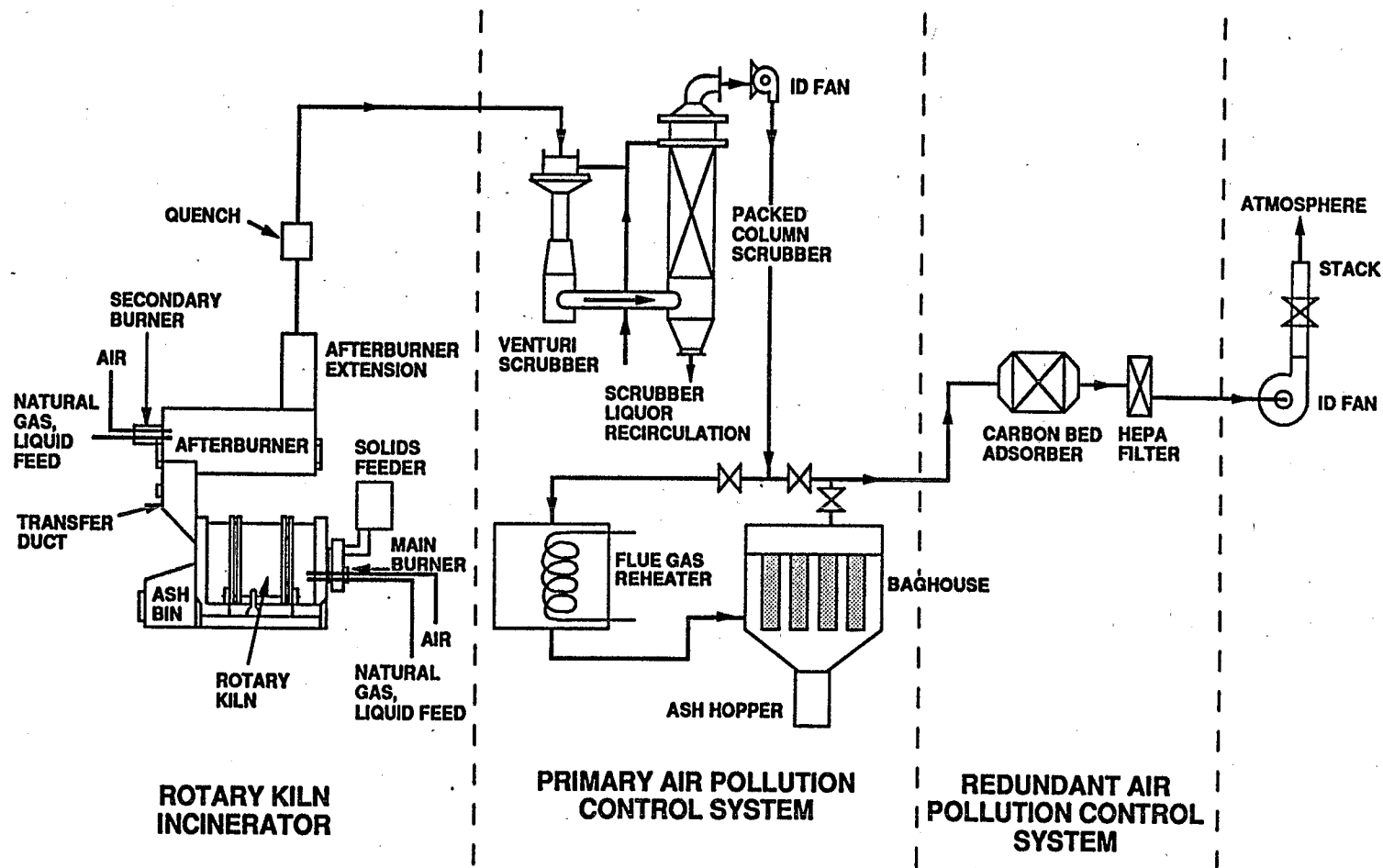


Figure 2-1. Schematic of the rotary kiln incineration system.

TABLE 2-1. DESIGN CHARACTERISTICS OF THE IRF ROTARY KILN INCINERATION SYSTEM

Characteristics of the Kiln Main Chamber	
Length	2.26 m (7 ft-5 in)
Diameter, outside	1.37 m (4 ft-6 in)
Diameter, inside	Nominal 1.04 m (3 ft-4.75 in)
Chamber volume	1.90 m ³ (67.2 ft ³)
Construction	0.95 cm (0.375 in) thick cold-rolled steel
Refractory	18.7 cm (7.375 in) thick high alumina castable refractory, variable depth to produce a frustroconical effect for moving solids
Rotation	Clockwise or counterclockwise, 0.2 to 1.5 rpm
Solids retention time	1 hr (at 0.2 rpm)
Burner	North American burner rated at 590 kW (2.0 MMBtu/hr) with liquid feed capability
Primary fuel	Natural gas
Feed system:	
Liquids	Positive displacement pump via water-cooled lance
Sludges	Moyno pump via front face, water-cooled lance
Solids	Metered screw feeders or fiberpack ram feeder
Temperature (max)	1,010°C (1,850°F)
Characteristics of the Afterburner Chamber	
Length	3.05 m (10 ft)
Diameter, outside	1.22 m (4 ft)
Diameter, inside	0.91 m (3 ft)
Chamber volume	1.80 m ³ (63.6 ft ³)
Construction	0.63 cm (0.25 in) thick cold-rolled steel
Refractory	15.2 cm (6 in) thick high alumina castable refractory
Gas residence time	0.8 to 1.5 s depending on temperature and excess air
Burner	North American Burner rated at 590 kW (2.0 MMBtu/hr) with liquid feed capability
Primary fuel	Natural gas
Temperature (max)	1,200°C (2,200°F)
Characteristics of the Afterburner Extension	
Length, with transition sections	4.43 m (14 ft-6.5 in)
Diameter, outside	0.915 m (3 ft)
Diameter, inside	0.61 m (2 ft)
Chamber volume	1.19 m ³ (41.9 ft ³)
Construction	0.63 cm (0.25 in) thick cold-rolled steel
Refractory	15.2 cm (6 in) thick high alumina castable refractory
Temperature (max)	1,200°C (2,200°F)
Characteristics of the Venturi/Packed-column Scrubber APCS	
System capacity, inlet gas flow	107 m ³ /min (3,773 acfm) at 1,200°C (2,200°F) and 101 kPa (14.7 psia)
Pressure Drop	
Venturi scrubber	7.5 kPa (30 in WC)
Packed column	1.0 kPa (4 in WC)
Liquid flow	
Venturi scrubber	77.2 L/min (20.4 gpm) at 60 kPa (10 psig)
Packed column	116 L/min (30.6 gpm) at 69 kPa (10 psig)
pH control	Feedback control by NaOH solution addition

(continued)

TABLE 2-1. (continued)

Characteristics of the Baghouse Collector	
System capacity, inlet gas flow	70 m ³ /min (2,500 acfm) at 120°C (250°F)
Operating temperature	200°C (400°F)
Operating pressure	±12.4 kPa (±50 in WC)
Diameter	1.8 m (6 ft)
Overall height	4.2 m (13 ft, 8.375 in)
Filter elements (bags)	
Material	16 oz. Nomex
Length	1.8 m (6 ft)
Number	69
Total filter area	45 m ² (488 ft ²)
Material of construction	
Collector internals	304 SS
Airlock	316 SS
Venturi nozzles	Aluminum
Insulation	Heat loss less than 8.8 kW (30,000 Btu/hr) at 200°C (400°F)

gas reheat/baghouse system was installed just prior to the initiation of these tests, in large part to satisfy a Region III request.

Downstream of the baghouse, a backup secondary APCS, comprised of an activated-carbon adsorber and a high-efficiency particulate air (HEPA) filter, is in place for further control of organic compound and particulate emissions. The main components of the RKS and its APCS are discussed in more detail in the following subsections.

2.1.1 Incinerator Characteristics

The rotary kiln combustion chamber has an inside diameter of 1.04-m (40.75-in) and is 2.26-m (7-ft 5-in) long. The chamber is lined with refractory formed into a frustroconical shape to an average thickness of 18.7 cm (7.375 in). The refractory is encased in a 0.95-cm (0.375-in) thick steel shell. Total volume of the kiln chamber, including the transition section, is 1.90 m³ (67.2 ft³). Four steel rollers support the kiln barrel. A variable-speed DC-motor coupled to a reducing gear transmission turns the kiln. Rotation speeds can be varied from 0.2 to 1.5 rpm. For these tests the kiln rotational speed was 0.2 rpm.

The afterburner chamber has a 0.91-m (3-ft) inside diameter, and is 3.05 m (10 ft) long. The afterburner wall is constructed of a 15.2-cm (6-in) thick layer of refractory encased in a 0.63 cm (0.25 in) thick carbon steel shell. The volume of the afterburner chamber is 1.80 m³ (63.6 ft³).

2.1.2 Air Pollution Control System

For this test program, the RKS primary APCS consisted of the venturi scrubber/packed-column scrubber combination, followed by a flue gas reheater and a baghouse. The flue gas exiting the afterburner passes through the refractory-lined transfer section and enters the quench section, where the flue gas temperature is reduced to approximately 82°C (180°F) by direct injection of aqueous caustic scrubber liquor. The cooled flue gas then enters the venturi scrubber, which is fitted with an automatically adjustable-area throat. The scrubber is designed to operate at 6.2 kPa (25 in WC) differential pressure, with a maximum liquor flowrate of 77.2 L/min (20.4 gpm). The scrubber liquor, again an aqueous caustic solution, enters at the top of the scrubber and contacts the flue gas to remove entrained particles and, to some degree, acid gases.

Downstream of the venturi scrubber, the flue gas enters the packed-column scrubber, where additional acid gas and particulate cleanup occurs. The scrubber column is packed with 5.1 cm (2 in) diameter polypropylene ballast saddles to a depth of 2.1 m (82 in). It is designed to operate at 1.0 kPa (4 in WC) differential pressure, with a maximum liquor flowrate of 116 L/min (30.6 gpm).

The quench, venturi scrubber, and packed-column scrubbers receive their scrubber liquor from the same recirculation system. This liquor is a dilute aqueous NaOH solution, the pH of which is monitored continuously by a pH sensor. An integral pH controller automatically meters the amount of NaOH needed to maintain the setpoint pH for proper acid gas removal.

Following the quench, venturi scrubber, and packed-column scrubber systems, the flue gas is reheated to about 120°C (250°F) by a 100-kW electric duct heater, and then passed through the baghouse. The baghouse removes most of the remaining flue gas particulate. Reheating the flue gas ensures that no moisture condenses in the baghouse, which can adversely affect its operation.

In a typical commercial incinerator system, the flue gas would be vented to the atmosphere downstream of the baghouse. However, at the IRF, a backup APCS is in place to further clean up the flue gas. The flue gas exiting the baghouse is passed through a bed of activated carbon to allow the vapor-phase organic compounds to be adsorbed. A set of HEPA filters designed to remove any remaining suspended particulate from the flue gas is located downstream of the carbon bed. An induced-draft (ID) fan draws and vents the treated flue gas to the atmosphere.

2.2 TEST WASTE CHARACTERISTICS

Data on the contaminant concentrations in the fluff waste and the surface and subsurface soil at the site, taken from the site record of decision (ROD) document, are summarized in Table 2-2. Only contaminants present at an average concentration of 1 mg/kg or greater in one or more contaminated site matrix are listed in the table. The data in Table 2-2 show that the major site contaminants are the two phthalate esters, bis(2-ethylhexyl)phthalate (BEHP) and di-n-octylphthalate (DNOP). Thus, these compounds would be considered the POHCs in the site wastes. In addition, Region III was interested in establishing that tetrachloroethene is effectively destroyed by incineration, so tetrachloroethene was also defined to be a POHC. Site wastes are also highly contaminated with copper and lead, with lesser, though still significant, amounts of antimony, barium, chromium, nickel, and zinc.

TABLE 2-2. M. W. MANUFACTURING SITE WASTE CONTAMINANTS FROM THE ROD

Contaminant	Concentration, mg/kg						
	Fluff		Surface soil		Maximum average subsurface soil		
	Range over 17 samples	Average	Range over 21 samples	Average	Range	Average	Depth
Volatile Organic Constituents							
2-Butanone	2.8-6.4	1.6	— ^a	—	up to 3.9	0.78	16-18
Tetrachloroethene	0.72-18.0	4.4	0.023-67	10	0.001-1,600 ^b	56	4-6
Trichloroethene	—	—	0.002-21	1.0	0.002-2.6	2.7	12-14
1,1,2-Trichloroethane	—	—	0.003-2.8	0.28	up to 5.4	1.1	16-18
1,2-Dichloroethene	—	—	0.002-10	0.49	0.004-0.58	0.04	8-10
Methylene chloride	up to 7.7	0.45	up to 0.83	0.04	—	—	—
Semivolatile Organic Constituents							
Bis(2-ethylhexyl)phthalate	72,000-230,000	149,000	3.9-3,000	836	0.30-30,000 ^b	1,480	12-14
Di-n-octyl phthalate	1,800-13,000	4,400	0.2-140	37	0.038-150	7,850	0-2
Di-n-butyl phthalate	—	—	0.48	0.02	0.036-130	3.9	0-2
PCB-1254	0.90-18.1	9.4	0.061-3.7	0.21	0.077-1.0	0.043	0-2
Trace Metals							
Antimony	80-143	65	62-118	16	—	—	—
Barium	20-232	93	22-107	74	47-218	107	0-2
Cadmium	0.65-4.4	2.4	1.2-12	2.0	1-13	1	0-2
Chromium	24-59	40	7.1-59	27	14-70	20	0-14
Copper	5,910-130,000	50,000	742-171,000	21,600	24-38,900	1,850	12-14
Lead	1,600-3,600	2,400	32-9,770	1,450	7-741	160	16-18
Nickel	4.1-15	4.6	8.5-40	22	42-50	46	6-8
Silver	1.6-5.7	1.8	8.6	0.4	—	—	—
Zinc	135-2,580	620	55-787	240	56-319	144	6-8

^a— = Not reported.

^bMaximum value in range represents an estimated value above minimum detection limit but below lowest calibration standard ("J" flag).

Samples of the fluff waste and surface and subsurface soil were sent to the IRF for characterization analyses. Results of the analyses are summarized in Table 2-3. As shown, the soil characterization samples had contaminant concentrations in the range reported in the ROD. Contaminant concentrations in the fluff waste characterization samples were also in the range reported in the ROD for most contaminants. However, the fluff characterization sample

TABLE 2-3. M. W. MANUFACTURING SITE CHARACTERIZATION SAMPLE ANALYSIS RESULTS

Parameter	Sample		
	Fluff	Surface soil	Subsurface soil
Characterization			
Moisture, %	7.7	18	9.8
Ash, %			
at 550°C	41	77	89
at 900°C	14	76	90
Heating value, MJ/kg (Btu/lb)	6.50 (2,800)	0.07 (30)	Will not burn
Volatile Organic Constituents, mg/kg			
Tetrachloroethene	146	69	18
1,1,2-Trichloroethane	4.8	1.5	ND ^a
Semivolatile Organic Constituents, mg/kg			
BEHP	124,000	47.6	4.62
DNOP	17,800	1.95	ND
Trace Metals, mg/kg			
Antimony	230	51	<5
Barium	64	60	78
Cadmium	3.5	<0.2	0.93
Chromium	57	30	21
Copper	31,000	8,300	160
Lead	2,700	1,800	180
Nickel	6.1	15	31
Silver	4.0	<0.4	<0.4
Zinc	890	76	62

^aND = Not detected.

contained substantially more tetrachloroethene, 1,1,2-trichloroethane, DNOP, and antimony that did fluff samples reported in the ROD.

Characterization samples received were also analyzed for hazardous waste characteristics, including the preparation and analysis of toxicity characteristic leaching procedure (TCLP) leachates of the samples. Results are summarized in Table 2-4.

The two semivolatile POHCs in site materials, BEHP and DNOP, are poor candidates for testing the incineration process with regard to destroying other site waste organic contaminants because they are ranked as relatively easy to thermally destroy compounds in the

**TABLE 2-4. M. W. MANUFACTURING SITE CHARACTERIZATION SAMPLE
HAZARDOUS WASTE CHARACTERISTICS ANALYSIS RESULTS**

Characteristic	Fluff waste	Surface soil	Subsurface soil	Regulatory level
Reactivity -S, mg/kg	<0.01	<0.01	<0.01	Contains
Reactivity -CN, mg/kg	<0.01	<0.01	<0.01	and reacts
Corrosivity, pH	6.85	8.15	6.37	<2, >12
Ignitability, °F	>200	>200	>200	<140
TCLP leachate, mg/L				
Arsenic	<0.10	<0.10	<0.10	5.0
Barium	0.10	0.37	0.21	100
Cadmium	0.11	0.07	0.07	1.0
Chromium	0.11	0.09	0.09	5.0
Copper	199	158	1.88	— ^a
Lead	3.1	3.2	0.20	5.0
Mercury	<0.002	<0.002	<0.002	0.2
Nickel	<0.01	<0.01	<0.01	—
Selenium	<0.10	<0.10	<0.10	1.0
Silver	<0.01	<0.01	<0.01	5.0
Zinc	5.1	0.49	0.12	—
Pesticides and other organics	ND ^b	ND	ND	

^a— = No regulatory level.

^bND = Not detected at detection limits ranging from 0.004 to 0.01 mg/L.

thermal stability based incinerability ranking (Reference 1). This ranking groups the 333 compounds ranked into seven stability classes from most stable, or most difficult to destroy (Class 1), to least stable or easiest to destroy (Class 7). Both BEHP and DNOP are ranked in Class 6, or relatively easy to destroy. To present a challenge to the incineration process and develop data that suggest incineration is capable of achieving sufficient DREs for other site organic contaminants, the test waste materials were spiked with naphthalene, a Class 1 (most difficult to destroy) POHC, at 2 percent by weight. This spiking level allows DREs of over 99.999 percent to be quantitated at easily achieved flue gas sampling and analysis method quantitation limits. In addition, it was decided to spike the volatile POHC, tetrachloroethene, into test materials at a level of 3,100 mg/kg by weight. Tetrachloroethene is a Class 2 POHC. Spiking was needed because site material concentrations of tetrachloroethene were too low to allow establishing 99.99 percent DRE at achievable flue gas concentration quantitation limits.

Prior to initiating the test program, all test feed material was packaged into 1.5-gal (5.7-L) polyethylene (PE)-bag-lined cubical cardboard containers for feeding to the RKS. For fluff packaging, the contents of three 55-gal (208-L) shipment drums (of the 14 fluff-containing drums received at the IRF for testing) were emptied into a 250-gal (946-L) mixing trough. The trough contents were manually mixed with hoes until visually homogeneous. Trough contents were then used to fill feed containers. Each container was filled with about 1.8 kg (4 lb) of mixed fluff. A mixed trough would fill 150 containers, with a small quantity left over. This small quantity would be combined with the next three drums added to the trough for mixing.

This mixing process resulted in four full-trough fluff batches, with each batch used to package 150 feed containers. All containers from a given batch were placed on a pallet for short-term storage, resulting in four 150-container pallets. A fifth partial-trough batch was used to package a final 120 feed containers, which were placed on a fifth pallet. The containers on

the fifth pallet were used during scoping tests to verify the ability to feed the test material and maintain target RKS operating conditions. For each of the actual four fluff incineration tests performed, essentially equal numbers of containers were randomly selected from each of the first four pallets to constitute the feed materials for the test.

Naphthalene was added to each fluff-filled box as a preweighed count of solid naphthalene crystals (36.4 g) contained in a 60-mL high-density polyethylene (HDPE) bottle with a polypropylene screw cap closure, weighing about 14.4 g. The tetrachloroethene (5.6 g) was added in a 4-mL HDPE bottle, with a polypropylene screw cap closure, weighing 2.6 g. Feed boxes were spiked the day before a given test by imbedding the HDPE bottles in the feed box contents. The box polyethylene liner was then closed with a plastic tie, and the box itself closed and sealed with paper packaging tape.

Contaminated soil for testing was similarly mixed, except that all five of the drums of soil received at the IRF for testing (whether surface, subsurface, or mixed not specified) were mixed in one trough mixing exercise. Mixed soil was packaged into the 120 feed containers, each containing about 4.5-kg (10-lb) of soil. The naphthalene spike added the day before a soil test was 90.9 g in a 125-mL HDPE bottle, and the tetrachloroethene spike was 14.5 g in an 8-mL bottle.

2.3 TEST CONDITIONS

The test program completed consisted of seven tests. Of these seven, two sets of duplicate tests feeding fluff waste alone and one set of duplicate tests feeding contaminated soil alone were performed. The two sets of fluff feed tests were conducted at different kiln combustion gas temperatures. Soil and fluff were separately tested because the eventual site remediation may treat each material separately for logistical reasons. In addition, Region III

requested data to determine whether the ash from incinerated soil alone would meet the cleanup levels given in the ROD. The target test operating conditions were as given in Table 2-5.

A seventh test, denoted Test 0 in Table 2-5, was performed as a blank burn. Only feed packaging materials — the cardboard box, PE bag liner, HDPE spike bottles (no POHC spike) with closures, plastic tie, and paper tape — were fed to the RKS for the blank burn.

For all tests, the target afterburner exit gas temperature was 1,090°C (2,000°F). The venturi/packed-column scrubber and baghouse APCS units were operated at their normal design settings. Kiln rotation rate was set to give a 30-minute kiln solids residence time.

The target average test material feedrate was 54.5 kg/hr (120 lb/hr) for all tests except the blank burn. Test materials were fed to the RKS via the fiberboard container ram feed system. A total of 30 containers per hour (one container every 2 minutes) was fed to achieve the target feedrate for the fluff tests; 12 containers per hour (one container every 5 minutes) were fed to achieve the target feedrate for the soil tests. The blank burn was conducted feeding 30 containers (no waste or soil) per hour.

For all tests, the scrubber system was operated at its design settings, listed in Table 2-6, and at as close to total recirculation (zero to minimum blowdown) as possible. Given the

TABLE 2-5. TEST MATRIX

Test	Feed	Target kiln exit gas temperature, °C (°F)
0	Packaging container material	870 (1,600)
1	Fluff	870 (1,600)
2	Duplicate of Test 1	
3	Soil	870 (1,600)
4	Duplicate of Test 3	
5	Fluff	760 (1,400)
6	Duplicate of Test 5	

TABLE 2-6. APCS OPERATING CONDITIONS

Venturi liquor flowrate	76 L/min (20 gpm)
Venturi pressure drop	6.2 kPa (25 in WC)
Packed tower liquor flowrate	115 L/min (30 gpm)
Scrubber liquor temperature	49°C (120°F)
Scrubber blowdown rate	0 L/min (0 gpm) or minimum operable

relatively short (nominally 4 to 5 hours) duration of a test, no operational problems due to solids buildup in the scrubber liquor occurred even at total recirculation. For the fluff waste and blank burn tests, kiln ash was continuously deposited in initially clean 20-gal (76-L) drums placed in the RKS ash pit. For the soil tests, kiln ash was continuously removed from the kiln ash hopper via an ash auger transfer system and deposited into 55-gal (208-L) drums.

The actual kiln and afterburner operating conditions achieved for each test are summarized in Tables 2-7 and 2-8, respectively. Table 2-9 provides a similar summary of the APCS operating conditions for each test. Continuous emission monitor (CEM) data are summarized in Table 2-10. The ranges and averages of the temperature, CEM, and scrubber pH data presented in Tables 2-7 through 2-10 were developed for the periods of the flue gas sampling, using the data automatically recorded by a personal computer-based data acquisition system. The values given for the remaining parameters were derived from the control room logbook data.

Transcribed data from the control room logs of the operating parameters, recorded at 15-minute intervals, are given in Appendix A. Appendix B contains graphic presentations of the flue gas temperature and continuous emission monitor data for the kiln and afterburner. Appendix B also contains graphic presentations of the scrubber exit and stack flue gas continuous emissions monitor data. These data plots were based on incinerator system conditions recorded

TABLE 2-7. KILN OPERATING CONDITIONS

Parameter		Test 0 (10/27/93)	Test 1 (11/9/93)	Test 2 (11/16/93)	Test 5 (11/18/93)	Test 6 (11/23/93)	Test 3 (12/1/93)	Test 4 (12/2/93)
Average natural gas feedrate,	scm/hr	33	22	23	13	14	30	31
	(scfh)	(1,156)	(778)	(820)	(475)	(507)	(1,065)	(1,085)
	kW	339	228	240	139	149	312	318
	(kBtu/hr)	(1,156)	(778)	(820)	(475)	(507)	(1,065)	(1,085)
Average combustion air flowrate,	scm/hr	226	214	205	191	174	200	198
	(scfh)	(7,970)	(7,560)	(7,250)	(6,760)	(6,140)	(7,070)	(6,990)
Average total air flowrate (includes inleakage),	scm/hr	679	1,054	1,067	1,000	1,008	675	673
	(scfh)	(23,980)	(37,200)	(37,670)	(35,300)	(35,610)	(23,840)	(23,750)
Average draft,	Pa	7	7	7	5	7	5	5
	(in WC)	0.03	0.03	0.03	0.02	0.03	0.02	0.02
Exit gas temperature,	Range, °C	842-898	827-921	814-918	716-804	724-807	853-912	862-894
	(°F)	(1,548-1,648)	(1,520-1,689)	(1,498-1,685)	(1,321-1,479)	(1,336-1,484)	(1,568-1,674)	(1,583-1,642)
	Average, °C	871	883	876	762	767	876	874
	(°F)	(1,599)	(1,622)	(1,608)	(1,403)	(1,412)	(1,609)	(1,606)
Exit gas O ₂	Range, %	8.4-13.4	7.7-14.0	8.2-14.6	8.7-14.2	9.0-14.0	8.0-13.5	7.7-13.1
	Average, %	11.6	10.9	11.0	11.8	11.8	11.1	10.9
Average waste feedrate,	kg/hr	5	60	59	61	61	59	59
	(lb/hr)	(12)	(132)	(130)	(134)	(134)	(130)	(130)
Average waste heat input,	kW	10	224	218	227	224	30	31
	(kBtu/hr)	(34)	(765)	(743)	(774)	(765)	(104)	(104)
Total heat input,	kW	349	452	458	366	373	342	348
	(kBtu/hr)	(1,190)	(1,543)	(1,563)	(1,249)	(1,272)	(1,169)	(1,189)
Calculated combustion gas residence time,	seconds	2.5	1.6	1.6	1.6	1.9	2.5	2.5

TABLE 2-8. AFTERBURNER OPERATING CONDITIONS

Parameter		Test 0 (10/27/93)	Test 1 (11/9/93)	Test 2 (11/16/93)	Test 5 (11/18/93)	Test 6 (11/23/93)	Test 3 (12/1/93)	Test 4 (12/2/93)
Average natural gas feedrate,	scm/hr	29	27	26	24	25	27	33
	(scfh)	(1,025)	(948)	(903)	(861)	(876)	(943)	(1,166)
	kW	300	278	265	252	257	276	342
	(kBtu/hr)	(1,025)	(948)	(903)	(861)	(876)	(943)	(1,166)
Average combustion air flowrate,	scm/hr	194	165	156	129	118	157	161
	(scfh)	(6,840)	(5,830)	(5,520)	(4,550)	(4,180)	(5,540)	(5,700)
Exit gas temperature,	Range, °C	1,090-1,107	1,089-1,152	1,087-1,105	1,091-1,105	1,091-1,102	1,091-1,115	1,090-1,106
	(°F)	(1,994-2,025)	(1,992-2,106)	(1,989-2,021)	(1,996-2,021)	(1,995-2,016)	(1,196-2,039)	(1,994-2,023)
	Average, °C	1,098	1,103	1,097	1,097	1,097	1,098	1,097
	(°F)	(2,008)	(2,017)	(2,007)	(2,007)	(2,007)	(2,008)	(2,007)
Exit gas O ₂	Range, %	8.8-12.0	4.2-11.7	5.7-10.8	5.1-10.8	5.1-9.1	8.7-14.2	9.0-14.0
	Average, %	10.7	7.6	8.2	8.0	7.9	11.8	11.8

TABLE 2-9. AIR POLLUTION CONTROL SYSTEM OPERATING CONDITIONS

Parameter		Test 0 (10/27/93)	Test 1 (11/9/93)	Test 2 (11/16/93)	Test 5 (11/18/93)	Test 6 (11/23/93)	Test 3 (12/1/93)	Test 4 (12/2/93)
Average quench chamber liquid flowrate,	L/min (gpm)	76 (20)	76 (20)	75 (20)	76 (20)	76 (20)	72 (19)	76 (20)
Average venturi scrubber liquid flowrate	L/min (gpm)	76 (20)	79 (21)	76 (20)	79 (21)	79 (21)	83 (22)	76 (20)
Average packed-column scrubber liquid flowrate,	L/min (gpm)	114 (30)	110 (29)	114 (30)	114 (30)	114 (30)	110 (29)	114 (30)
Scrubber liquor, pH	Range	6.9-7.1	5.9-8.2	6.0-8.3	6.8-8.2	6.4-8.2	6.7-7.5	6.6-7.5
	Average	7.0	6.9	7.1	7.3	7.2	7.2	7.2
Average scrubber makeup flowrate,	L/min (gpm)	1,098 (290)	57 (15)	0 (0)	193 (51)	0 (0)	11 (3)	0 (0)
Average scrubber liquor temperature,	°C (°F)	67 (152)	69 (156)	69 (156)	67 (152)	68 (155)	69 (156)	69 (156)
Average scrubber inlet gas temperature,	°C (°F)	76 (169)	78 (173)	78 (173)	77 (171)	77 (171)	76 (169)	76 (169)
Average scrubber exit gas temperature,	°C (°F)	59 (138)	64 (147)	64 (147)	60 (140)	62 (143)	61 (141)	61 (141)

TABLE 2-10. CONTINUOUS EMISSION MONITOR DATA

Parameter		Test 0 (10/27/93)	Test 1 (11/9/93)	Test 2 (11/16/93)	Test 5 (11/18/93)	Test 6 (11/23/93)	Test 3 (12/1/93)	Test 4 (12/2/93)
Kiln Exit								
O ₂	Range, %	8.4-13.4	7.7-14.0	8.2-14.6	8.7-14.2	9.0-14.0	8.0-13.5	7.7-13.1
	Average, %	11.6	10.9	11.0	11.8	11.8	11.1	10.9
	Target, %	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Afterburner Exit								
O ₂	Range, %	8.8-12.0	4.2-11.7	5.7-10.8	8.7-14.2	9.0-14.0	5.1-10.8	5.1-9.1
	Average, %	10.7	7.6	8.2	11.8	11.8	8.0	7.9
	Target, %	8.0	8.0	8.0	8.0	8.0	8.0	8.0
CO ₂	Range, %	5.3-8.1	6.7-11.6	5.4-11.0	5.4-11.2	5.6-10.3	6.3-10.8	7.6-11.9
	Average, %	6.2	8.9	8.6	8.1	8.3	8.3	8.6
NO _x	Range, ppm	45-82	46-70	41-96	27-66	38-62	37-70	45-75
	Average, ppm	66	59	64	39	47	56	63
Baghouse Exit								
O ₂	Range, %	12.0-15.1	9.8-13.7	10.3-14.0	9.2-14.2	9.4-12.7	9.7-14.1	9.6-12.4
	Average, %	12.9	11.6	11.4	11.4	10.9	11.5	11.4
CO	Range, ppm	<1-10	<1-10	<1-9	<1-10	<1-11	<1-7	<1-6
	Average, ppm	2	2	2	3	2	2	2
CO ₂	Range, %	2.5-5.2	4.4-7.4	4.1-7.0	3.4-8.2	4.0-7.6	3.3-7.1	3.8-7.6
	Average, %	4.3	5.9	5.9	6.2	6.3	5.7	5.8
TUHC	Range, ppm	<1-4	<1-10	<1-11	<1-22	5-36	<1-4	<1-4
	Average, ppm	1	1	1	3	9	2	2
NO _x	Range, ppm	28-62	29-42	38-57	30-51	45-61	28-80	45-73
	Average, ppm	52	38	42	43	53	64	60
Stack								
O ₂	Range, %	13.2-15.7	11.4-14.3	11.8-14.6	11.3-15.1	11.2-13.5	11.6-15.3	11.5-13.9
	Average, %	13.8	12.8	12.6	12.7	12.4	12.8	12.9
CO	Range, ppm	9-14	6-8	5-7	6-7	6-8	6-9	7-8
	Average, ppm	12	7	6	6	7	7	7

at about 35-second intervals on the RKS data acquisition system. In addition, durations of flue gas sampling periods, major events, cumulative amounts of waste fed into the incinerator, and cumulative amounts of ash removed from the incinerator are included in some plots. These data provide the basis for assembling a complete picture of the actual incinerator operating conditions.

SECTION 3

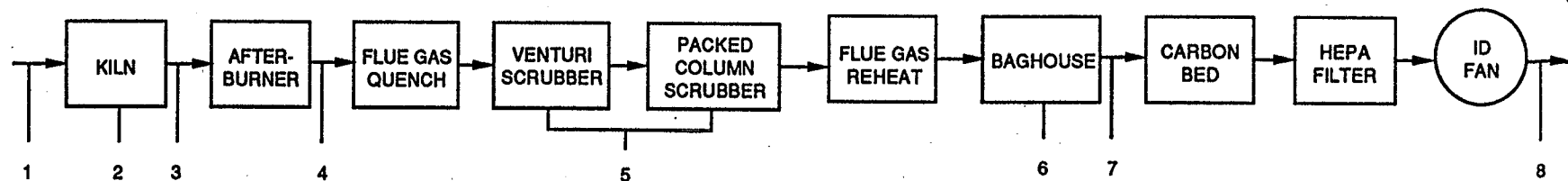
SAMPLING AND ANALYSIS PROCEDURES

The scope of the sampling efforts performed in the test program is illustrated in Figure 3-1. The sampling effort performed is discussed in Section 3.1, followed by a discussion of the sample analysis procedures in Section 3.2.

3.1 SAMPLING PROCEDURES

For all tests, the sampling matrix included:

- Obtaining a composite sample of the test feed material
- Obtaining a composite sample of the kiln ash discharge
- Obtaining a composite sample of the pre-test and post-test scrubber system liquor
- Obtaining a composite sample of the baghouse ash
- Continuously measuring O₂ concentrations in the kiln exit flue gas; O₂, CO₂, and NO_x in the afterburner exit flue gas; O₂, CO, CO₂, NO_x, and total unburned hydrocarbon (TUHC) concentrations in the baghouse exit flue gas; and O₂ and CO concentrations in the stack gas
- Sampling the flue gas at the baghouse exit for trace metals using the EPA multiple metals train (Reference 2)
- Sampling the flue gas at the baghouse exit for the waste and spiked semivolatile POHCs and other semivolatile target compound list (TCL) constituents using an EPA Method 0010 train (Reference 3)



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3-2

Sampling point	Test feed material	Kiln ash	Scrubber liquor	Baghouse ash	Continuous monitors					Flue gas				
					O ₂	CO	CO ₂	NO _x	Heated TUHC	EPA multiple metals train, test trace metals	Method 0010, SVOCs	Method 0030, VOCs	Method 23, PCDD/PCDF	Method 5, particulate and HCl
1. Feed	X													
2. Kiln ash discharge		X												
3. Kiln exit flue gas					X									
4. Afterburner exit flue gas					X		X	X						
5. Scrubber liquor			X											
6. Baghouse hopper				X										
7. Baghouse exit flue gas					X	X	X	X	X	X	X	X	X	X
8. Stack gas					X	X								X

Figure 3-1. Test sampling locations.

- Sampling the flue gas at the baghouse exit for the waste and spiked volatile organic contaminants and other volatile TCL constituents using EPA Method 0030 (Reference 3), the volatile organic sampling train (VOST)
- Sampling the flue gas at the baghouse exit for PCDDs/PCDFs using EPA Method 23 (Reference 2)
- Sampling the baghouse exit and the stack for particulate and HCl using EPA Method 5; the stack sample was needed to comply with the IRF's permit requirements

Composite feed material samples were collected from each mixing trough after feed material was mixed in the trough, as discussed in Section 2.2, but before packaging into the cardboard containers. Samples were collected from six different locations in the trough and combined to form the composite sample representing the trough's contents. Four fluff feed samples resulted, each representing one full trough mixture. Two soil feed samples resulted, the first taken before the packaging of the first soil feed container, the second after the filling of 60 soil feed containers.

The feed sample for Test 0, the blank burn test, consisted of proportionate samples of the cardboard box material, the polyethylene liner bag, the bag plastic tie, the HDPE bottle with screw cap, and the paper packaging tape used to prepare feed boxes.

On a given test day, the incinerator was brought to nominally steady operation at test conditions while firing auxiliary fuel (natural gas) alone. Test material feed was then initiated. Flue gas sampling was started about 1 hour after test material feed initiation. At the conclusion of each test day, the incinerator was operated on natural gas for 2 hours after waste feed cessation while ash material in the kiln continued to discharge until the kiln was empty.

During the fluff waste tests, kiln ash was continuously deposited in an initially clean 20-gal (76-L) drum placed in the RKS ash pit. The amount of collected ash was insufficient to allow representative thief sampling, therefore grab samples consisting of a large fraction of the collected ash were taken.

During the soil feed tests, kiln ash was continuously removed from the kiln ash pit via a transfer auger and deposited into a 55-gal (208-L) drum. After all test ash was deposited in this drum, representative kiln ash samples were taken by thief sampling in at least three locations across the collection drum cross section. The three ash samples were combined to form one composite sample.

No kiln ash resulted from the blank burn test.

Each test was run with the scrubber liquor loop operating at as close to total recycle (no blowdown) as possible. At the end of each test day, a scrubber liquor sample was collected from a tap in the recirculation loop. The scrubber liquor was then drained to a collection tank. In addition, a sample was taken from the scrubber liquor loop from the same tap just before initiating test material feed on a test day. The baghouse ash sample consisted of the entire amount of baghouse ash collected in the baghouse ash hopper for each test.

The Method 5 trains for particulate and HCl collection had dilute caustic-filled impingers (0.1 N NaOH). Admittedly, both HCl and Cl₂ in the flue gas are collected caustic impingers. However, this conservative estimate of HCl concentrations (HCl plus Cl₂) satisfied test program objectives. A nominal 1.4 m³ (50 ft³) sample was collected at the two locations sampled over about a 1-hour time period. The Method 0010, Method 23, and multiple metals trains sampled nominally 2.8 m³ (100 ft³) of flue gas over a 3-hour period. Because mercury was not a trace metal of interest in this program, the permanganate impingers for mercury collection were not used in the multiple metals train, and sample recovery steps specified for eventual

mercury analysis were not performed. Four Method 0030 trap pairs each sampled 20 L of flue gas. Four trap pairs were taken as insurance against trap breakage.

The CEMs available at the IRF and the locations that they monitored during all tests are summarized in Table 3-1. This monitoring arrangement was employed in all tests. Figure 3-2 illustrates the generalized flue gas conditioning and flow distribution system at the IRF. Four independent systems, such as the one illustrated in Figure 3-2, were in place so that the appropriately conditioned sample gas from four separate locations was routed to the respective monitors in Table 3-1. The CEM setup described in Table 3-1, with appropriate gas conditioning per Figure 3-2, was employed throughout this test program. CEM data was recorded continuously on strip charts and also by an automatic data acquisition system.

Test program samples were analyzed for matrix-specific combinations of SVOCs, VOCs, PCDDs/PCDFs, contaminant trace metals, and chloride. Sample analysis procedures are outlined in Table 3-2. The number of test program samples analyzed is summarized in Table 3-3. The numbers of method blank, split sample, and matrix spike/matrix spike duplicate QA analyses are also given in Table 3-3. The large numbers of fluff and soil feed split sample analyses for SVOC and trace metals, and kiln ash split sample analyses for trace metals, were at the request of Region III. The Region III Remediation Project Manager (RPM) requested that the precision of the feed and ash trace metal and feed SVOC concentration measurements be well characterized.

Table 3-4 summarizes the sample aliquoting schedule for dividing samples taken for each test among the various analytical procedures. Each sample was divided among the various analytical procedures according to Table 3-4. Aliquots analyzed as noted in Table 3-4 corresponded to respective method-recommended sample sizes.

TABLE 3-1. CONTINUOUS EMISSION MONITORS USED AND LOCATIONS MONITORED

Location	Monitor				Range
	Constituent	Manufacturer	Model	Principle	
Kiln exit	O ₂	Beckman	755	Paramagnetic	0-10 percent 0-25 percent 0-100 percent
Afterburner exit	O ₂	Rosemount	755	Paramagnetic	0-10 percent 0-25 percent 0-100 percent
	CO ₂	Horiba	PIR 2000	NDIR	0-20 percent 0-80 percent
	NO _x	Thermo Electron	10 AR	Chemiluminescent	0-75 ppm to 0-10,000 ppm in multiples of 2
Baghouse exit	O ₂	Beckman	755	Paramagnetic	0-10 percent 0-25 percent 0-100 percent
	CO	Horiba	VIA 500	NDIR	0-50 ppm 0-500 ppm
	CO ₂	Horiba	PIR 2000	NDIR	0-20 percent 0-80 percent
	TUHC	Beckman	402	FID	0-10 ppm 0-100 ppm 0-1,000 ppm
	NO _x	Thermo Electron	10 AR	Chemiluminescent	0-75 ppm to 0-10,000 ppm in multiples of 2
Stack	O ₂	Teledyne	326A	Fuel cell	0-5 percent 0-10 percent 0-25 percent
	CO	Horiba	VIA 500	NDIR	0-50 ppm 0-500 ppm

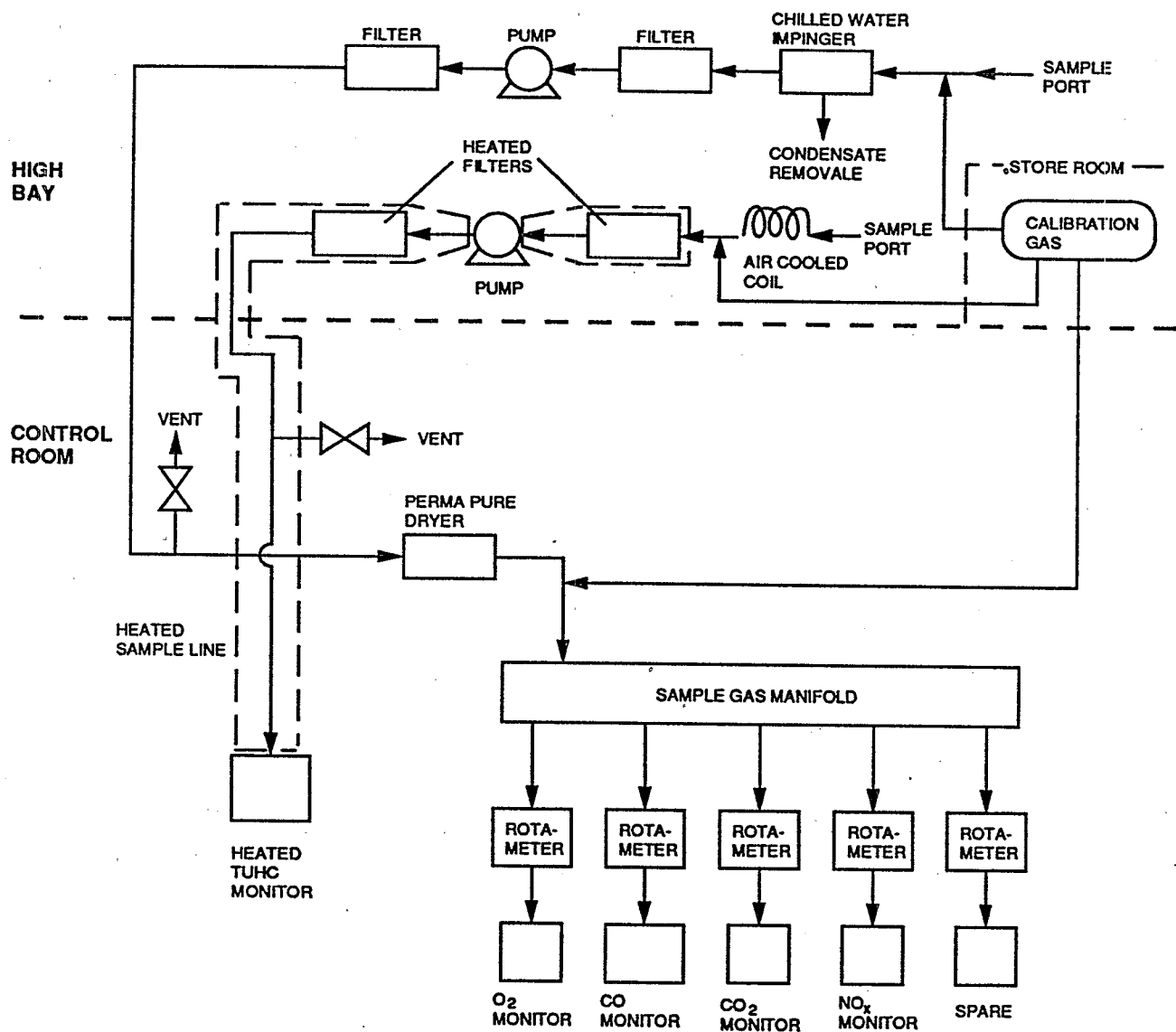


Figure 3-2. Generalized CEM gas flow schematic.

TABLE 3-2. ANALYSIS PROCEDURES

Sample	Parameter	Analysis method	Frequency
Test feed material	Proximate analysis (moisture, volatile matter, fixed carbon, ash)	ASTM D-5142	1 composite for each test material
	Elemental analysis C, H, O, N, S	ASTM D-3176	1 composite for each test material
	Cl	ASTM E-442	1 composite for each test material
	Heating value	ASTM D-3286	1 composite for each test material
	Test semivolatile POHCs	Soxhlet extraction by Method 3540A, GC/MS analysis by Method 8270A ^a	1/test
	Test volatile organic contaminants	Purge and trap GC/FID of methanol extract by Method 8015A ^a	1/test
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1 composite for each test material
	Trace metals ^b	Digestion by the multiple metals filter method ^c , ICAP analysis by Method 6010A ^a	1/fluff test, 1 composite soil
	TCLP extraction	Method 1311 ^a	1/fluff test, 1 composite soil
Test feed TCLP leachate	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/fluff test, 1 composite soil
Feed packaging material	Proximate analysis (moisture, volatile matter, fixed carbon, ash)	ASTM D-5142	1 composite
	Elemental analysis C, H, O, N, S	ASTM D-3176	1 composite
	Cl	ASTM E-442	1 composite
	Heating value	ASTM D-3286	1 composite
	Test semivolatile POHCs	Soxhlet extraction by Method 3540A, GC/MS analysis by Method 8270A ^a	1 composite
	Test volatile organic contaminants	Purge and trap GC/FID of methanol extract by Method 8015A ^a	1 composite
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1 composite
	Trace metals ^b	Digestion by the multiple metals filter method ^c , ICAP analysis by Method 6010A ^a	1 composite
Kiln ash	Test semivolatile POHCs	Soxhlet extraction by Method 3540A, GC/MS analysis by Method 8270A ^a	1/test
	Test volatile organic contaminants	Purge and trap GC/FID of methanol extract by Method 8015A ^a	1/test
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1/test
	Trace metals ^b	Digestion by the multiple metals filter method ^c , ICAP analysis by Method 6010A ^a	1/test
	TCLP extraction	Method 1311 ^a	1/test
Kiln ash TCLP leachate	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/test

^aReference 3, SW-846.^bAs, Sb, Ba, Cd, Cr, Cu, Pb, Ni, Ag, and Zn.^cReference 2, 40 CFR 266, App. IX.

(continued)

TABLE 3-2. (continued)

Sample	Parameter	Analysis method	Frequency
Pre-test scrubber liquor	Test semivolatile POHCs	Extraction by Method 3520A, GC/MS analysis by Method 8270A ^a	1/test
	Test volatile organic contaminants	Purge and trap by Method 5030A, GC/FID by Method 8015A ^a	1/test
	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/test
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1 sample before the first test
Post-test scrubber liquor	Test semivolatile POHCs	Extraction by Method 3520A, GC/MS analysis by Method 8270A ^a	1/test
	Test volatile organic contaminants	Purge and trap by Method 5030A, GC/FID by Method 8015A ^a	1/test
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1/test
	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/test
	TCLP extraction	Method 1311 ^a	1/test
Scrubber liquor TCLP leachate	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/test
Baghouse ash	Test semivolatile POHCs	Soxhlet extraction by Method 3540A, GC/MS analysis by Method 8270A ^a	1/test
	Test volatile organic contaminants	Purge and trap GC/FID of methanol extract by Method 8015A ^a	1/test
	PCDDs/PCDFs	GC/MS by Method 8290 ^a	1/test
	Trace metals ^b	Digestion by the multiple metals filter method ^c , ICAP analysis by Method 6010A ^a	1/test
	TCLP extraction	Method 1311 ^a	1/test
Baghouse ash TCLP leachate	Trace metals ^b	Digestion by Method 3010A, ICAP analysis by Method 6010A ^a	1/test
Baghouse exit flue gas	Semivolatile TCL constituents	Soxhlet extraction of Method 0010A samples by Method 3540A, GC/MS analysis by Method 8270A ^a	1/test
	Volatile TCL constituents	Purge and trap of Method 0030 samples by Method 5040, GC/MS analysis by Method 8240A	3 trap pairs/test
	PCDDs/PCDFs	GC/MS of Method 23 samples by Method 23 ^c	1/test
	Trace metals ^b	Digestion of multiple metals train samples by multiple metals procedure ^c , ICAP analysis by Method 6010A ^a	1/test
	Particulate	Method 5 ^d	1/test
	HCl	ICAP analysis of combined impinger solution by Method 9057 ^c	1/test
Stack gas	Particulate	Method 5 ^d	1/test
	HCl	IC analysis of combined impinger solution by Method 9057 ^c	1/test

^aReference 3, SW-846.^bAs, Sb, Ba, Cd, Cr, Cu, Pb, Ni, Ag, and Zn.^cReference 2, 40 CFR 266, App. IX.^dReference 4, 40 CFR 60, App. A.

TABLE 3-3. TEST PROGRAM SAMPLE ANALYSIS SUMMARY

Sample matrix	Number of analyses			
	SVOCs	VOCs	PCDDs/PCDFs	Trace metals Chloride
Fluff Waste Feed				
Test sample	4	4	1	4
Split sample	16	1		16
Matrix spike	1	1		1
Spike duplicate	1	1		1
Soil Feed				
Test sample	2	2	1	1
Split sample	8	1		4
Matrix spike	1	1		1
Spike duplicate	1	1		1
Packaging Container Material				
Cardboard + packaging tape + HDPE bottle + bottle cap + polyethylene liner + plastic tie	1	1	1	1
Kiln Ash				
Test sample ^a	6	6	6	6
Split sample	1	1	1	22
Matrix spike	1	1		1
Spike duplicate	1	1		1
Pre-test Scrubber Liquor				
Test sample	7	7	1	7
Post-test Scrubber Liquor				
Test sample	7	7	7	7
Split sample	1	1	1	1
Matrix spike	1	1		1
Spike duplicate	1	1		1
Baghouse Ash				
Test sample	7	7	7	7
Split sample	1	1	1	1
Matrix spike	1	1		1
Spike duplicate	1	1		1

^aNo kiln ash resulted from the blank burn test.

(continued)

TABLE 3-3. (continued)

Sample matrix	Number of analyses				
	SVOCs	VOCs	PCDDs/PCDFs	Trace metals	Chloride
TCLP Leachate					
Fluff feed				4	
Soil feed				1	
Kiln ash				6	
Scrubber liquor				7	
Baghouse ash				7	
Method blank				2	
Split sample				2	
Matrix spike				2	
Spike duplicate				2	
Method 0010 Train					
Test sample	7				
Method blank	1				
Matrix spike	1				
Spike duplicate	1				
Method 0030					
Test sample trap pair ^b		21			
Field blank		7			
Trip blank		1			
Matrix spike		9			
Method 23 Train					
Test sample			7		
Method blank			1		
Multiple Metals Train					
Front half					
Test sample				7	
Method blank				1	
Matrix spike				1	
Spike duplicate				1	
Back half					
Test sample				7	
Method blank				1	
Matrix spike				1	
Spike duplicate				1	
Method 5 Train Impingers					
Test sample					14
Matrix spike					1
Spike duplicate					1
Total	81	87	35	140	16

^bFour trap pairs sampled per test, three trap pairs analyzed; fourth trap pair for breakage contingency.

TABLE 3-4. SAMPLE ANALYSIS ALIQUOT SCHEDULE FOR EACH TEST

Sample	Total quantity of each sample collected	Analyte/ procedure	Aliquot size	Number of aliquots needed
Fluff waste feed	1 kg	SVOCs	10 g	5 replicates each test + 1 MS + 1 MSD
		VOCs	4 g	1 each test + 1 duplicate + 1 MS + 1 MSD
		PCDDs/PCDFs	10 g	1 total
		Trace metals	1 g	5 replicates each test + 1 MS + 1 MSD
		TCLP extraction	100 g	1 each test
Soil feed	1 kg	SVOCs	10 g	5 replicates each test + 1 MS + 1 MSD
		VOCs	4 g	1 each test + 1 duplicate + 1 MS + 1 MSD
		Trace metals	1 g	5 replicates total + 1 MS + 1 MSD
		TCLP extraction	100 g	1 total
Packaging container material	50 g	SVOCs	10 g	1
		VOCs	4 g	1
		PCDDs/PCDFs	10 g	1
		Trace metals	1 g	1
Kiln ash	1 kg	SVOCs	10 g	1 each test + 1 split + 1 MS + 1 MSD
		VOCs	4 g	1 each test + 1 split + 1 MS + 1 MSD
		PCDDs/PCDFs	10 g	1 each test + 1 split
		Trace metals	1 g	5 replicates each test + 1 MS + 1 MSD
		TCLP extraction	100 g	1 each test
Pre-test scrubber liquor	4 L	SVOCs	1 L	1 each test
		VOCs	80 mL (two 40-mL aliquots)	1 each test
		PCDDs/PCDFs	1 L	1 total
		Trace metals	100 mL	1 each test
Post-test scrubber liquor	8 L	SVOCs	1 L	1 each test + 1 split + 1 MS + 1 MSD
		VOCs	80 mL (two 40-mL aliquots)	1 each test + 1 split + 1 MS + 1 MSD
		PCDDs/PCDFs	1 L	1 each test + 1 split
		Trace metals	100 mL	1 each test + 1 split + 1 MS + 1 MSD
		TCLP extraction	2 L	1 each test

(continued)

TABLE 3-4. (continued)

Sample	Total quantity of each sample collected	Analyte/ procedure	Aliquot size	Number of aliquots needed
Baghouse ash	As collected	SVOCs	10 g	1 each test + 1 split + 1 MS + 1 MSD
		VOCs	4 g	1 each test + 1 split + 1 MS + 1 MSD
		PCDDs/PCDFs	10 g	1 each test + 1 split
		Trace metals	1 g	1 each test + 1 split + 1 MS + 1 MSD
		TCLP extraction	100 g	1 each test
TCLP leachate	2 L	Trace metals	100 mL	25 test samples + 2 method blank + 2 split + 2 MS + 2 MSD
Method 0010 train	As collected	SVOCs	Total	1 each test + 1 method blank + 1 MS + 1 MSD
Method 0030 train	As collected	VOCs	Total	4 ^a each test + 7 field blanks + 1 trip blank + 9 MS
Method 23 train	As collected	PCDDs/PCDFs	Total	1 each test + 1 method blank
Multiple metals train				
Front half	As collected	Trace metals	Total	1 each test + 1 method blank + 1 MS + 1 MSD
Back half	As collected	Trace metals	Total	1 each test + 1 method blank + 1 MS + 1 MSD
Method 5 train impinger	As collected	Cl	100 mL	2 each test + 1 MS + 1 MSD

^aThree of four analyzed; fourth represents breakage contingency.

Table 3-5 summarizes the containers used for sample aliquot storage until analysis, preservation methods used, and analysis hold times required. Only new containers were used for sample storage. They were purchased, precleaned to meet EPA standards, from a laboratory supply vendor and are certified by the vendor as appropriate for use in storing samples for the respective analyte class. No containers or preservation is shown in Table 3-5 for Method 0010 train samples. The procedure at the IRF is to transfer samples recovered from Method 0010 trains directly into the extraction apparatus immediately after a test and begin overnight extractions the day of each test.

Filters and other sampling train components for Method 0010, Method 23, the multiple metals method train, and the Method 5 trains were cleaned according to the procedures

TABLE 3-5. SAMPLE CONTAINERS, PRESERVATION METHODS, AND HOLD TIMES

Sample	Analyte	Sample container ^a	Sample preservation method	Analysis hold time
Solid samples (fluff waste, soil, packaging container material, kiln ash, baghouse ash)	SVOCs	G, T	Cool to 4°C	Extraction: 14 days Analysis ^b : 40 days
	VOCs	G, T, zero headspace	Cool to 4°C	14 days
	PCDDs/PCDFs	G, T	Cool to 4°C	Extraction: 30 days Analysis ^b : 45 days
	Trace metals	G or P	None	6 months
Aqueous liquid samples (scrubber liquor, TCLP leachates)	SVOCs	G, T	Cool to 4°C	Extraction: 7 days Analysis ^b : 40 days
	VOCs	G, T VOA vial	Cool to 4°C	14 days
	PCDDs/PCDFs	G, T	Cool to 4°C	Extraction: 30 days Analysis ^b : 45 days
	Trace metals	G or P	HNO ₃ to pH <2	6 months
Method 0010 train	SVOCs	None	None	40 days
Method 0030 traps	VOCs	Sealed glass traps	Cool to 4°C	42 days
Method 23 train filter	PCDDs/PCDFs			Extraction: 30 days Analysis ^b : 45 days
Filter		Glass petri dish	Cool to 4°C	
Rinses and impinger solutions		G, T	Cool to 4°C	
Multiple metals train	Trace metals			6 months
Filter		Glass petri dish	None	
Rinses and impinger solutions		G or P	None	
Method 5 train impinger solution	Chloride	G or P	None	28 days

^aG = glass, P = polyethylene, T = Teflon-lined cap.

^bAfter extraction.

documented in the respective methods. Sorbent resin for use in Method 0010 and Method 0030 trains was cleaned prior to use according to the procedures in the respective methods. Method 23 sorbent resin cartridges were cleaned and charged with clean sorbent, spiked with method surrogates, by the analytical laboratory that performed the PCDD/PCDF analyses.

A single container was used to store each sample collected for each analysis. Aliquots were taken from this container as needed. MS and MSD samples were prepared from aliquots from this container as well. After preparation, however, MS and MSD samples were stored in separate containers until analyzed.

Unused sample collected was stored in appropriate containers with appropriate preservation until the expiration of method hold times. After method hold time expired, unused samples were archived.

3.2 ANALYSIS METHODS

Table 3-2 summarizes the analytes determined in each test program sample, and the analysis procedures used. As indicated in the table, the fluff feed samples for each fluff test, a composite soil feed sample, the packaging container material, and the kiln ash, pre-test and post-test scrubber liquor, and baghouse ash samples for each test analyzed for the site contaminant trace metals. Trace metal analyses were by ICAP spectroscopy in accordance with Method 6010A. Solid samples (feed material, packaging container material, kiln ash, and baghouse ash) were digested using the HNO_3/HF procedure specified for use with filter particulate in the EPA multiple metals method (Reference 2). Liquid samples (pre-test and post-test scrubber liquor) were digested using a minor variation of Method 3010A. This minor variation consists of using concentrated HNO_3 instead of 1:1 HCl in the last step of Section 7.2 of Method 3010A. The 10 site contaminant metals measured in the test samples were antimony,

arsenic, barium, cadmium, chromium, copper, lead, nickel, silver, and zinc. Omission of the HCl in the last Method 3010A step allows analysis for silver.

Each fluff test's fluff feed, a composite soil feed sample, and each test's kiln ash, baghouse ash, and post-test scrubber liquor samples were subjected to TCLP extraction by Method 1311. The TCLP leachates were analyzed for the 10 site contaminant trace metals by Method 6010A. The minor variation of Method 3010A, noted above, was used for leachate digestion.

In addition, each fluff test's fluff feed, each soil test's soil feed, the packaging container material, and the kiln ash, baghouse ash, and pre-test and post-test scrubber liquor samples for each test were analyzed for the test semivolatile POHCs (BEHP, DNOP, and naphthalene). Semivolatile POHC analyses were by gas chromatography/mass spectrometry (GC/MS) in accordance with Method 8270A. Solid samples (feed, packaging container material, baghouse ash, and kiln ash) were Soxhlet-extracted by Method 3540A. Pre-test and post-test scrubber liquor samples were extracted via continuous liquid-liquid extraction by Method 3520A.

One composite fluff feed sample, a composite soil feed sample, the packaging container material, the pre-test scrubber liquor sample taken before the first test program test, and the kiln ash, post-test scrubber liquor, and baghouse ash samples for each test were also analyzed for PCDDs/PCDFs by the high-resolution GC/MS method, Method 8290. The single composite fluff feed sample was prepared by combining aliquots of the four test feed samples collected.

Each test's fluff feed or soil feed, the packaging container material, and each test's kiln ash, baghouse ash, and pre-test and post-test scrubber liquor samples were also analyzed for the test volatile organic contaminants by GC/FID in accordance with Method 8015A. Sample introduction was by Method 5030A.

The Method 0010 samples for each test were analyzed for the semivolatile organic TCL constituents listed in Table 3-6. The target analytes were the semivolatile test POHCs, BEHP, DNOP, and naphthalene. However, the other TCL constituents were also quantitated. Sample preparation was performed according to Method 0010, with final GC/MS analysis by Method 8270A. The Method 23 samples for each test were analyzed for PCDDs/PCDFs by the method.

TABLE 3-6. SEMIVOLATILE ORGANIC TCL CONSTITUENTS

Acenaphthene	2,6-Dinitrotoluene
Acenaphthylene	Di-n-octyl phthalate
Anthracene	Fluoranthene
Benz(a)anthracene	Fluorene
Benzo(b)fluoranthene	Hexachlorobenzene
Benzo(k)fluoranthene	Hexachlorobutadiene
Benzo(a)pyrene	Hexachloroethane
Benzo(ghi)perylene	Indeno(1,2,3-cd)pyrene
Benzyl butyl phthalate	Isophorone
Bis(2-chloroethyl) ether	Naphthalene
Bis(2-chloroethoxy) methane	Nitrobenzene
Bis(2-chloroisopropyl) ether	N-Nitrosodi-n-propylamine
Bis(2-ethylhexyl) phthalate	Phenanthrene
4-Bromophenyl phenyl ether	Pyrene
2-Chloronaphthalene	1,2,4-Trichlorobenzene
4-Chlorophenyl phenyl ether	4-Chloro-3-methylphenol
Chrysene	2-Chlorophenol
Dibenzo(a,h)anthracene	2,4-Dichlorophenol
Di-n-butyl phthalate	2,4-Dimethylphenol
1,2-Dichlorobenzene	2,4-Dinitrophenol
1,3-Dichlorobenzene	2-Methyl-4,6-dinitrophenol
1,4-Dichlorobenzene	2-Nitrophenol
3,3'-Dichlorobenzidine	4-Nitrophenol
Diethyl phthalate	Pentachlorophenol
Dimethyl phthalate	Phenol
2,4-Dinitrotoluene	2,4,6-Trichlorophenol

As noted in Section 3.1, four Method 0030 trap pair samples were collected for each test. Three trap pair samples for each test were analyzed for the test volatile organic TCL constituents listed in Table 3-7 by purge and trap GC/MS via Methods 5040 and 8240A. The fourth trap pair was collected for breakage contingency, so that the probability that three trap pair analyses could be done for each test was increased, given the inevitability of trap breakage. The target analytes were the volatile site material organic contaminants, tetrachloroethene, trichloroethene, and 1,1,2-trichloroethane. However, the other TCL constituents were also quantitated.

Multiple metals train samples were analyzed for the 10 site contaminant trace metals. Sample preparation was performed according to the method, with final ICAP analysis by Method 6010A. Flue gas HCl levels were determined by analyzing the combined Method 5 train impinger solutions for chloride via IC according to Method 9057.

One composite fluff feed sample, the composite soil feed sample, and the packaging container material sample were also subjected to proximate, elemental, and heating value analyses by the ASTM procedures noted in Table 3-2.

TABLE 3-7. VOLATILE ORGANIC TCL CONSTITUENTS

Acetone	trans-1,2-Dichloroethene
Benzene	1,2-Dichloropropane
Bromodichloromethane	cis-1,3-Dichloropropene
Carbon disulfide	trans-1,3-Dichloropropene
Carbon tetrachloride	Methylene chloride
Chlorobenzene	Tetrachloroethene
Chlorodibromomethane	Toluene
Chloroform	1,1,1-Trichloroethane
1,1-Dichloroethane	1,1,2-Trichloroethane
1,2-Dichloroethane	Trichloroethene
1,1-Dichloroethene	Trichlorofluoromethane

Proximate and elemental analyses were performed by Galbraith Laboratories in Knoxville, Tennessee. SVOC (Method 8270A), VOC (Method 8015A), HCl (Method 9057), and TCLP (Method 1311) procedures were performed in the IRF analytical laboratories. Trace metal analyses (Method 6010A) were performed by the American Interplex Laboratories in Little Rock, Arkansas. Method 0030 sample and PCDD/PCDF (Methods 8290 and 23) analyses were performed by Triangle Laboratories in Research Triangle Park, North Carolina.

SECTION 4

TEST RESULTS

The results of the test program are discussed in this section. Test results are grouped by analyte class. Thus, Section 4.1 presents the fluff waste and soil feed proximate and elemental analysis results. Section 4.2 discusses the SVOC measurements. Section 4.3 discusses the VOC measurements. Section 4.4 discusses the PCDD/PCDF measurements. Each of the sections, 4.2, 4.3, and 4.4, includes discussion on the effectiveness of incineration in removing the contaminants of interest in the fluff waste and contaminated soil. Where appropriate, contaminant destruction removal efficiencies (DREs) are presented and discussed. The concentrations of contaminants of interest in the RKS discharge streams, kiln ash, baghouse ash, and scrubber liquor, are also discussed. Section 4.5 discusses the trace metals measurements. Section 4.6 discusses the results of the flue gas particulate and HCl measurements, as well as the flue gas particulate size distribution results. For the reader who is interested in studying the analytical results in more detail, the analytical laboratory reports are given in Appendix C of this report.

4.1 PROXIMATE AND ULTIMATE ANALYSIS RESULTS

The proximate and elemental analysis results for the fluff waste, soil, and packaging container material samples analyzed are presented in Table 4-1. As shown in the table, the fluff waste was distinctly organic in nature as evidenced by the high level of volatile matter at 39.1 percent and its higher heating value of 12.4 MJ/kg (5,330 Btu/lb). The fluff was also quite moist, with 46.8 percent moisture. The high moisture content was consistent with the fact that

**TABLE 4-1. PROXIMATE AND ELEMENTAL ANALYSIS RESULTS FOR
COMPOSITE FLUFF AND SOIL FEED SAMPLES**

	Fluff waste	Contaminated soil	Packaging container material
Proximate Analysis (as received)			
Moisture, %	46.8	23.0	4.8
Ash, %	5.2	64.4	1.4
Fixed carbon, %	8.9	2.6	13.3
Volatile matter, %	39.1	10.0	80.5
Higher heating value, MJ/kg (Btu/lb)	12.4 (5,330)	0.58 (250)	— ^a
Elemental Analysis, % (dry basis)			
C	44.2	7.3	46.4
H	6.2	1.1	7.0
O	7.4	5.8	45.0
N	<0.5	<0.5	<0.5
S	0.2	<0.04	0.2
Cl	32.2	2.3	<0.5
Ash	9.8	83.6	1.4

^a— = Not measured.

the fluff had been accumulated and stored in large piles outdoors, thereby being exposed to precipitation. The fluff waste contained 32.2 percent chlorine (dry basis), a level suggesting the presence of chlorinated plastics (e.g., polyvinyl chloride) and possibly other chloro-organic solvents, both of which were present or used at the site.

The character of the contaminated soil was distinctly different from the fluff, although a small amount of fluff-like material was found to have commingled into the soil. The heating value of the soil was low, as expected at 0.58 MJ/kg (250 Btu/lb). Its moisture content, at

23 percent, and ash content, at 64.4 percent are typical of soils. The soil chlorine content, at 2.3 percent (dry), could have been due to the presence of a small amount of fluff or other chlorine containing organic contaminants.

Table 4-2 summarizes the cumulative weights of the fluff waste and contaminated soil fed for each test and the total amount of corresponding kiln ash collected. As indicated in the table, for Tests 1 and 2 (average kiln exit gas temperature at 875° to 883°C [1,608° and 1,622°F], respectively), the collected ash weights were equal to about 5 percent of the amount of fluff fed. The collected ash amounts accounted for 98 to 101 percent of the theoretical ash amount that could be expected. For Tests 5 and 6, during which the average kiln exit temperatures were at 762 and 767°C (1,403° and 1,412°F), respectively, the collected ash amounts were equal to 5.7 and 6.7 percent of the amount of fluff fed. These amounts of collected ash were greater than the theoretical ash quantity in the fluff fed. The amounts of ash collected were 128 and 109 percent of the theoretical ash amount. The greater-than-100-percent ash collected for these

TABLE 4-2. WEIGHTS OF TEST MATERIAL FED AND KILN ASH COLLECTED

Test	Date	Total fed		Kiln ash collected			
				Weight		Fraction of feed %	Fraction of expected ash %
		kg	(lb)	kg	(lb)		
Fluff Waste Tests							
1	11/9/93	303	(666)	15	(34)	5.1	98
2	11/16/93	304	(669)	16	(35)	5.2	101
5	11/18/93	307	(675)	20	(45)	6.7	128
6	11/23/93	305	(671)	17	(38)	5.7	109
Soil Feed Tests							
3	12/1/93	288	(634)	150	(329)	52	81
4	12/2/93	290	(638)	145	(320)	50	78

tests would be consistent with incomplete fluff oxidation for these lower kiln exit gas temperature tests.

For the soil tests (Tests 3 and 4), the kiln ash weights were about 50 percent of the fed soil weights, which corresponds to about 80 percent of the theoretical ash amounts. Evidently, about 20 percent of the theoretical ash in the soil tests was entrained in the kiln exit combustion gas and carried out of the kiln.

4.2 SVOC ANALYSIS RESULTS

Table 4-3 summarizes the measured concentrations of the target SVOC analytes in test program samples collected. For the entries in the table noted as less than values, the value represents the MDL of the analysis procedure. Laboratory analysis reports are given in Appendix C-3. For each test performed, Table 4-3 also indicates the average kiln exit gas temperature measured over the flue gas sampling period corresponding to each test.

The data in Table 4-3 show that the BEHP concentrations in the actual fluff waste fed for each fluff waste test, at 48,300 to 53,300 mg/kg, were about half the level measured in the pretest characterization sample as reported in Table 2-3, as well as being below the lowest concentration reported in the ROD. Similarly, the DNOP levels in actual test fluff waste, at 1,850 to 2,870 mg/kg, were also substantially lower than the 17,800 mg/kg level measured in the pretest characterization sample, and were at the low end of the range of concentrations reported in the ROD. Nevertheless, contamination levels of these two constituents in the test fluff waste were still significant. The 20,200 mg/kg naphthalene concentration noted in incinerator feed samples in the table represents the quantity of naphthalene spike added to feed containers. No fluff waste or soil sample contained naphthalene at an MDL of 25 mg/kg before spiking.

The data in Table 4-3 show that the contaminated soil tested contained 9,440 to 9,810 mg/kg of BEHP and 550 to 580 mg/kg DNOP. These levels are substantially greater than

TABLE 4-3. SEMIVOLATILE ORGANIC CONTAMINANT ANALYSIS RESULTS

Sample	Concentration		
	BEHP	DNOP	Naphthalene
Test 0 (10/27/93), kiln temperature: 871°C (1,599°F)			
Packaging container material, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	6.6	4.1	<0.3
Baghouse exit flue gas, µg/dscm	8.4	<0.9	<0.8
Fluff Waste Tests			
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)			
Fluff feed, mg/kg	48,800	1,850	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	14.3	9.9	<0.3
Baghouse exit flue gas, µg/dscm	7.0	<1.2	<0.9
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)			
Fluff feed, mg/kg	53,300	2,610	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	4.5	2.2	<0.3
Baghouse exit flue gas, µg/dscm	9.9	<1.3	<1.1
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)			
Fluff feed, mg/kg	48,300	2,870	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.2
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	21.1	13.4	<0.3
Baghouse exit flue gas, µg/dscm	9.8	<1.1	<0.9
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)			
Fluff feed, mg/kg	49,000	2,810	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	18.7	12.1	<0.3
Baghouse exit flue gas, µg/dscm	6.2	<1.2	<1.0

^aSpiked concentration.

(continued)

TABLE 4-3. (continued)

Sample	Concentration		
	BEHP	DNOP	Naphthalene
Soil Feed Tests			
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)			
Soil feed, mg/kg	9,810	580	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	23.5	17.0	<0.3
Baghouse exit flue gas, µg/dscm	7.8	<1.2	<1.0
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)			
Soil feed, mg/kg	9,440	547	20,200 ^a
Kiln ash, mg/kg	<1.3	<0.4	<0.3
Scrubber liquor, mg/L	<0.013	<0.004	<0.003
Baghouse ash, mg/kg	14.2	9.7	<0.3
Baghouse exit flue gas, µg/dscm	7.0	<1.2	<1.0

^aSpiked concentration.

those measured in the pretest soil characterization samples analyzed, although they fall within the concentration ranges reported in the ROD for these contaminants. Again, the naphthalene concentrations in test soil feed samples correspond to spiked amounts.

The data in Table 4-3 also show that the native and spiked SVOC contaminants were essentially completely removed from the fluff waste by incineration at both kiln temperatures tested, as evidenced by their absence in the kiln ash discharge for all fluff waste tests at method detection limits (MDLs) of 0.3 to 1.3 mg/kg. Similarly these contaminants were removed from the contaminated soil for both soil tests at the single kiln temperature tested for this matrix. No kiln ash concentration data are given for the blank burn test, Test 0, in Table 4-3 because no kiln ash was discharged for this test.

None of the three SVOC contaminants was found in the post-test scrubber liquor for any test at MDLs of 0.003 to 0.013 mg/L.

Naphthalene was absent from the baghouse ash for all tests at an MDL of 0.3 mg/kg. However, low levels of both BEHP (6.6 to 23.5 mg/kg) and DNOP (2.2 to 17.0 mg/kg) were found in the baghouse ash for all tests, including the blank burn. No explanation as to why these site contaminants are found at these levels in the baghouse ash is offered, other than the fact that phthalates are commonly encountered laboratory contaminants. Neither naphthalene nor DNOP was present in the baghouse exit flue gas for any test, at MDLs of about 1 $\mu\text{g}/\text{dscm}$. BEHP was found in the baghouse exit flue gas for all tests, including the blank burn, at levels ranging from 6.2 to 9.8 $\mu\text{g}/\text{dscm}$.

Feed SVOC contaminant concentration, feedrate, baghouse exit flue gas SVOC contaminant concentration, and flue gas flowrate data can be combined to calculate SVOC contaminants DREs for each of the tests. Calculated DREs are summarized in Table 4-4. As shown in the table, the measured levels of BEHP in the baghouse exit flue gas corresponded to BEHP DREs ranging from 99.99932 to 99.99962 percent for the fluff waste tests and 99.9974 to 99.9980 percent for the soil feed tests. Kiln temperature had no apparent affect on BEHP DRE from fluff waste. Neither the spiked naphthalene nor the native DNOP contaminants were detected in the baghouse exit flue gas for any test. The DREs corresponding to baghouse exit flue gas MDLs, and noted with the ">" sign in Table 4-4, were 99.99982 to 99.99987 percent for naphthalene for all tests, 99.9982 to 99.9987 percent for DNOP in the fluff waste tests, and 99.9933 to 99.9940 percent for DNOP in the soil feed tests. All DREs demonstrated were greater than the 99.99 percent level required by the current hazardous waste incinerator performance standard.

TABLE 4-4. SVOC POHC DREs

Parameter	BEHP	DNOP	Naphthalene
Fluff Waste Tests			
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)			
Feed concentration, mg/kg	48,800	1,850	20,200
Feedrate, kg/hr	2.93	0.11	1.21
Baghouse exit flue gas:			
Concentration, µg/dscm	7.0	<1.2	<0.9
Emission rate, mg/hr	11.9	<2.0	<1.5
DRE, %	99.99959	>99.9982	>99.99987
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)			
Feed concentration, mg/kg	53,300	2,610	20,200
Feedrate, kg/hr	3.15	0.15	1.19
Baghouse exit flue gas:			
Concentration, µg/dscm	9.9	<1.3	<1.1
Emission rate, mg/hr	19.4	<2.5	<2.2
DRE, %	99.99939	>99.9984	>99.99982
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)			
Feed concentration, mg/kg	48,300	2,870	20,200
Feedrate, kg/hr	2.94	0.18	1.23
Baghouse exit flue gas:			
Concentration, µg/dscm	9.8	<1.1	<0.9
Emission rate, mg/hr	20.1	<2.3	<1.8
DRE, %	99.99932	>99.9987	>99.99985
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)			
Feed concentration, mg/kg	49,000	2,810	20,200
Feedrate, kg/hr	2.98	0.17	1.23
Baghouse exit flue gas:			
Concentration, µg/dscm	6.2	<1.2	<1.0
Emission rate, mg/hr	11.3	<2.2	<1.8
DRE, %	99.99962	>99.9987	>99.99985
Soil Feed Tests			
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)			
Feed concentration, mg/kg	9,810	580	20,200
Feedrate, kg/hr	0.58	0.034	1.19
Baghouse exit flue gas:			
Concentration, µg/dscm	7.8	<1.2	<1.0
Emission rate, mg/hr	15.0	<2.3	<1.0
DRE, %	99.9974	>99.9933	>99.99984
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)			
Feed concentration, mg/kg	9,440	547	20,200
Feedrate, kg/hr	0.56	0.032	1.19
Baghouse exit flue gas:			
Concentration, µg/dscm	7.0	<1.2	<1.0
Emission rate, mg/hr	11.3	<1.9	<1.6
DRE, %	99.9980	>99.9940	>99.99986

As noted in Section 3.2, the flue gas Method 0010 train samples were analyzed for the full list of SVOC TCL compounds given in Table 3-6. None were found at compound-specific MDLs ranging from 0.4 to 8 $\mu\text{g}/\text{dscm}$, with the exception of dimethylphthalate, which was found in the flue gas for all tests (including the blank test, Test 0) at concentrations ranging from 1.1 to 2.1 $\mu\text{g}/\text{dscm}$.

Also recall from the discussion in Section 3 that the five replicates of each fluff waste and soil feed samples collected were to be analyzed for the SVOC POHCs in the test program. This was done for the soil feed samples collected. However, only BEHP was quantitated in all fluff waste replicate samples. In order to accurately quantitate the DNOP levels measured in fluff waste samples, the analysis of undiluted sample extracts was required. However, the concentration of BEHP in undiluted extracts was so high that the instrument cleanup times required after an undiluted extract analysis were quite lengthy. For this reason, it was decided to quantitate DNOP and naphthalene in a composite undiluted extract, formed by combining aliquots of each replicate fluff waste SVOC extract, to give a test composite analysis result for these two analytes. Each replicate sample extract was then diluted and analyzed for BEHP. The DNOP concentrations for fluff waste samples given in Table 4-3 represent these test-specific composite extract analysis results. Naphthalene was not detected in any fluff waste composite extract sample at an MDL of 130 mg/kg.

Table 4-5 summarizes the BEHP replicate sample analysis results for the fluff waste samples. The average concentration noted for each test is the fluff waste concentration reported in Table 4-3. The data in the table show that the percent relative standard deviation (% RSD) of the BEHP concentration measured in the five replicate fluff waste samples analyzed for each test ranged from 5.8 to 24.6. All are well within the precision data quality objective (DQO) for this measurement of 50% RSD. The four test average fluff waste BEHP concentration, from

TABLE 4-5. BEHP CONCENTRATIONS IN REPLICATE FLUFF WASTE SAMPLES

Test	BEHP concentration, mg/kg					Average concentration, mg/kg	% RSD
	Sample	Replicate 1	Replicate 2	Replicate 3	Replicate 4		
1 (11/9/93)	27,800	52,800	57,200	50,700	55,600	48,800	24.6
2 (11/16/93)	55,400	51,200	49,000	56,100	54,900	53,300	5.8
5 (11/18/93)	51,300	53,300	43,200	47,300	46,200	48,300	8.4
6 (11/23/93)	54,000	43,300	51,900	54,000	42,000	49,000	12.1
4 test total (20 samples)						49,900	13.8

the analysis of 20 separate fluff samples, was 49,900 mg/kg, with the 20-sample % RSD at 13.8 percent.

Table 4-6 summarizes the results of the replicate soil feed SVOC analyses. Again, the average concentrations of BEHP and DNOP from the five samples analyzed for each test are those given in Table 4-3. As shown in Table 4-6, naphthalene was not detected in any soil feed sample at an MDL of 25 mg/kg. The data in Table 4-6 show that the % RSDs for the individual test replicate soil feed analyses were 32.8 and 27.9 percent for BEHP and 48.7 and 30.2 percent for DNOP. The two test average SVOC contaminant concentrations representing 10 separate

TABLE 4-6. SVOC CONTAMINANT CONCENTRATIONS IN REPLICATE SOIL SAMPLES

Soil sample	Concentration, mg/kg		
	BEHP	DNOP	Naphthalene
Test 3			
Sample	10,700	508	<25
Replicate 1	14,900	614	<25
Replicate 2	6,450	296	<25
Replicate 3	8,530	440	<25
Replicate 4	8,470	1,040	<25
Average	9,810	580	<25
% RSD	32.8	48.7	—
Test 4			
Sample	11,000	728	<25
Replicate 1	8,270	598	<25
Replicate 2	9,450	417	<25
Replicate 3	5,780	335	<25
Replicate 4	12,700	655	<25
Average	9,440	547	<25
% RSD	27.9	30.2	—
2 test total (10 samples)			
Average	9,630	563	<25
% RSD	28.9	38.8	—

analyses were 9,630 mg/kg for BEHP with 28.9 % RSD, and 563 mg/kg for DNOP with 38.8% RSD. All % RSD variances achieved were within the precision DQO for this measurement of 50% RSD.

4.3 VOC ANALYSIS RESULTS

Table 4-7 summarizes the measured concentrations of the target VOC analytes in test program samples collected. As for the SVOC analysis data reported in Table 4-3, entries in Table 4-7 noted as less than values were not detected at the MDLs noted with the "<" sign. Complete analytical laboratory reports on the VOC analyses are given in Appendices C-1 and C-2.

As shown in Table 4-7, no fluff waste sample contained 1,1,2-trichloroethane at an MDL of 1 mg/kg. This contaminant was absent from one test soil sample, but found at 28 mg/kg in the other test soil. Trichloroethene was not found in three of four fluff feeds at an MDL of 1 mg/kg. It was present in the fourth fluff feed at 2.4 mg/kg, and in the soil test feeds at 2.7 to 3.9 mg/kg. Tetrachloroethene was not detected in two tests' fluff feed (before spiking) at an MDL of 4 mg/kg, though it was present at 4.9 and 17 mg/kg in the other two tests' fluff feed. These levels are substantially lower than the 146 mg/kg found in the pretest fluff feed characterization sample indicated in Table 2-3, although they are comparable to levels reported in the ROD, indicated in Table 2-2. The contaminated soil tested contained 50 to 93 mg/kg of native (before spiking) tetrachloroethene, in the range of the levels measured in pretest soil characterization samples, as well as within the range of concentrations reported in the ROD. The addition of the tetrachloroethene spike to all test feed samples raised spiked fluff feed concentrations to 3,100 mg/kg and spiked soil feed concentrations to the 3,200 to 3,300 mg/kg range, as indicated in Table 4-7.

TABLE 4-7. VOLATILE ORGANIC CONTAMINANT ANALYSIS RESULTS

Sample	Concentration		
	Tetrachloro-ethene	1,1,2-Trichloro-ethane	Trichloro-ethene
Test 0 (10/27/93), kiln temperature: 870°C (1,599°F)			
Packaging container material, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.66	<0.09	0.15
Fluff Waste Tests			
Test 1 (11/9/94), kiln temperature: 883°C (1,622°F)			
Fluff feed, mg/kg, native	4.9	<1	2.4
Fluff feed, mg/kg, spiked	3,100		
Kiln ash, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.27	<0.14	0.16
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)			
Fluff feed, mg/kg, native	<4	<1	<1
Fluff feed, mg/kg, spiked	3,100		
Kiln ash, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.71	<0.09	0.14
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)			
Fluff feed, mg/kg, native	<4	<1	<1
Fluff feed, mg/kg, spiked	3,100		
Kiln ash, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.68	<0.09	0.23
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)			
Fluff feed, mg/kg, native	17	<1	<1
Fluff feed, mg/kg, spiked	3,100		
Kiln ash, mg/kg	5.6	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.61	0.23	0.09

(continued)

TABLE 4-7. (continued)

Sample	Concentration		
	Tetrachloro- ethene	1,1,2-Trichloro- ethane	Trichloro- ethene
Soil Feed Tests			
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)			
Soil feed, mg/kg, native	50	<1	2.7
Soil feed, mg/kg, spiked	3,200		
Kiln ash, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	1.57	1.27	0.73
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)			
Soil feed, mg/kg, native	93	2.8	3.9
Soil feed, mg/kg, spiked	3,300		
Kiln ash, mg/kg	<4	<1	<1
Scrubber liquor, mg/L	<0.015	<0.004	<0.004
Baghouse ash, mg/kg	<4	<1	<1
Baghouse exit flue gas, µg/dscm	0.14	<0.05	0.17

As was the case for the SVOC contaminants, incineration treatment of the fluff waste at both temperatures tested and of the contaminated soil at the one temperature tested was essentially completely effective in decontaminating the feed materials of their native and spiked VOC contaminants. The kiln ash discharge for all tests contained no detectable VOC contaminants at MDLs ranging from 1 to 4 mg/kg with the single exception of a 5.6 mg/kg concentration of tetrachloroethene in the kiln ash from one low temperature fluff waste test. This detected level is just above the MDL of 4 mg/kg. In addition, neither the post-test scrubber liquor nor the baghouse ash from any test contained detectable VOC contaminants at MDLs of 0.004 to 0.15 mg/L in scrubber liquor and 1 to 4 mg/kg in baghouse ash. Pretest scrubber liquor samples similarly contained no detectable VOC contaminants at the same MDLs.

The baghouse exit flue gas for all tests, including the blank burn test, contained low levels of both trichloroethene, at 0.09 to 0.73 µg/dscm, and tetrachloroethene, at 0.14 to

1.57 $\mu\text{g}/\text{dscm}$. No 1,1,2-trichloroethane was found in the baghouse exit flue gas at MDLs of 0.05 to 0.14 $\mu\text{g}/\text{dscm}$ for the blank burn test, either fluff test at the higher incinerator temperature, one of the two fluff tests at the lower incineration temperature, and one of the two soil feed tests. This contaminant was found in the baghouse exit flue gas from the two tests detected at 0.23 to 1.27 $\mu\text{g}/\text{dscm}$. The baghouse exit flue gas concentration data noted represent averages for the three Method 0030 trap pairs analyzed for each test.

Feed contaminant concentration, feedrate, baghouse exit flue gas contaminant concentration, and flue gas flowrate data can be combined to calculate spike tetrachloroethene DREs for each of the tests. Calculated DREs are summarized in Table 4-8. As shown in the table, the measured baghouse exit flue gas tetrachloroethene concentrations corresponded to tetrachloroethene DREs of 99.9984 to 99.99988 percent over all tests. Comparable tetrachloroethene DREs were measured for both fluff and soil, and for fluff treated at both incineration temperatures. All measured DREs were greater than the 99.99 percent level required by the current hazardous waste incinerator performance standard.

As noted in Section 3, the Method 0030 train samples taken at the baghouse exit were also analyzed for the extended list of VOCs given in Table 3-7. The results of these analyses are summarized in Table 4-9 for those VOCs not given in Table 4-7. Concentrations in Table 4-9 noted as less than values were either not detected at the MDL of the procedure used to measure flue gas VOC concentration, or had a measured concentration not significantly different from the method blank concentration. In these latter cases, the method blank concentration is noted with the "<" sign.

The data in Table 4-9 show that acetone, bromodichloromethane, carbon tetrachloride, and chloroform were found in the baghouse exit flue gas for all tests, including the blank test (Test 0). Flue gas acetone concentrations were the highest for the blank test and the two fluff

TABLE 4-8. TETRACHLOROETHENE DREs

Parameter	Tetrachloroethene
Fluff Waste Tests	
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)	
Feed concentration, mg/kg	3,100
Feedrate, kg/hr	0.19
Baghouse exit flue gas:	
Concentration, µg/dscm	0.27
Emission rate, mg/hr	0.46
DRE, %	99.99975
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)	
Feed concentration, mg/kg	3,100
Feedrate, kg/hr	0.18
Baghouse exit flue gas:	
Concentration, µg/dscm	0.71
Emission rate, mg/hr	1.4
DRE, %	99.99924
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)	
Feed concentration, mg/kg	3,100
Feedrate, kg/hr	0.19
Baghouse exit flue gas:	
Concentration, µg/dscm	0.68
Emission rate, mg/hr	1.4
DRE, %	99.99926
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)	
Feed concentration, mg/kg	3,100
Feedrate, kg/hr	0.19
Baghouse exit flue gas:	
Concentration, µg/dscm	0.61
Emission rate, mg/hr	1.1
DRE, %	99.99941
Soil Feed Tests	
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)	
Feed concentration, mg/kg	3,200
Feedrate, kg/hr	0.19
Baghouse exit flue gas:	
Concentration, µg/dscm	1.57
Emission rate, mg/hr	3.0
DRE, %	99.9984
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)	
Feed concentration, mg/kg	3,300
Feedrate, kg/hr	0.19
Baghouse exit flue gas:	
Concentration, µg/dscm	0.14
Emission rate, mg/hr	0.23
DRE, %	99.99988

TABLE 4-9. FLUE GAS VOC CONCENTRATIONS

Compound	Baghouse exit flue gas concentration, $\mu\text{g}/\text{dscm}$						
	Blank test	Fluff waste tests				Soil tests	
	Test 0 10/27/93	Test 1 11/9/93	Test 2 11/6/93	Test 5 11/18/93	Test 6 11/23/93	Test 3 12/1/93	Test 4 12/2/93
Acetone	130	420	460	23	2.5	1.4	1.2
Benzene	63	32	300	8.3	<6.4	14	<5.2
Bromodichloromethane	0.48	0.17	0.15	1.2	1.9	4.4	1.6
Carbon disulfide	<0.5	<18	<73	<17	<64	<23	<22
Carbon tetrachloride	0.25	0.83	0.44	0.19	0.41	1.1	0.36
Chlorobenzene	<0.19	<0.05	1.1	<0.04	<0.05	0.60	<0.05
Chloroform	1.1	3.6	2.4	2.3	3.8	12	4.2
Dibromochloromethane	<0.09	1.3	1.3	0.94	1.2	1.9	0.74
1,1-Dichloroethene	<0.09	<0.09	<0.09	<0.09	<0.09	<0.09	<0.10
c-1,3-Dichloropropene	<0.05	<0.05	<0.05	<0.04	0.21	<0.05	<0.05
Methylene chloride	<2.5	18	97	15	34	100	72
Toluene	39	11	210	<2.0	<2.1	9.2	<2.1
1,1,1-Trichloroethane	<0.09	<0.09	0.39	0.16	0.39	6.1	<0.10
Trichlorofluoromethane	<1.0	<2.3	<2.4	<2.6	<0.8	<1.5	<0.39

waste tests at the higher incineration temperature tested; levels for the fluff waste tests at the lower incineration temperature and for the soils tests were substantially lower. No explanation for this observation is offered. Flue gas carbon tetrachloride concentrations were comparable from test to test, including the blank test, and ranged from a few to several tenths of a $\mu\text{g}/\text{dscm}$. Flue gas chloroform concentrations were also comparable from test to test, but were higher, in the 1 to 12 $\mu\text{g}/\text{dscm}$ range. Methylene chloride was measured in the baghouse exit flue gas for all tests except the blank test; concentrations ranged from 18 to 100 $\mu\text{g}/\text{dscm}$.

The presence of bromodichloromethane, seen in the flue gas for all tests, and dibromochloromethane, seen in the flue gas for all tests except the blank test, arises from the trihalomethane (THM) compounds present in the recirculating scrubber liquor. The plant water used for scrubber makeup is local well water subjected to a water treatment process that includes disinfection. The disinfection process leaves low levels of THM compounds in the water. The observation that dibromochloromethane was not detected in the Test 0 flue gas, but was at concentrations substantially above the MDL for the fluff waste and soil tests suggests that HCl or organochlorine compounds, such as methylene chloride, need to be present in the flue gas being scrubbed in order to form dibromochloromethane.

4.4 PCDD/PCDF ANALYSIS RESULTS

As noted in Section 3, incinerator feed, kiln ash, post-test scrubber liquor, and baghouse ash samples for all tests were analyzed for PCDDs and PCDFs. In addition, the baghouse exit flue gas was sampled, and collected samples correspondingly analyzed. Analyses were performed for the total concentration of each homologue grouping of total tetra-, penta-, hexa-, hepta-, and octa-chlorinated dibenzo-p-dioxins (TCDD, PeCDD, HxCDD, HpCDD, and OCDD) and dibenzofurans (TCDF, PeCDF, HxCDF, HpCDF, and OCDF) as well as the concentration of

each congener chlorinated in the 2, 3, 7, and 8 positions within each group. Analysis results are given, by sample matrix, in Tables 4-10 through 4-14.

Two summary measures of dioxin/furan concentrations are commonly cited. The first measure, total PCDD/PCDF, represents the sum of the homologue group total concentrations analyzed. The second measure, 2,3,7,8-TCDD toxicity equivalent (TEQ), is a weighted sum of each 2,3,7,8-chlorinated congener's concentration. In calculating TEQs, the measured concentration of each specific 2,3,7,8-chlorinated congener is weighted by a toxicity equivalent factor (TEF). The TEF is a measure of the congener's toxicity relative to 2,3,7,8-TCDD, which has a TEF of 1. The TEFs used to calculate the TEQs here are those specified by EPA (Reference 2) and given in Table 4-15.

Table 4-16 summarizes the chlorinated dioxin and furan concentrations in test program samples in terms of the two summary measures. In many cases, concentrations in Table 4-16 are reported as ranges. This arises out of the fact that analyzed concentrations for both homologue group totals and specific congeners are often reported as being less than an MDL, as indicated by the less than values in Tables 4-10 through 4-14. Thus, in cases where a concentration is listed as a range in Table 4-16, the maximum value in the range corresponds to the assumption that constituents not detected were present at the MDL, and the minimum value in the range corresponds to the assumption that they were not present, i.e., at zero concentration.

The data in Table 4-16 show that the fluff feed contained 56 $\mu\text{g}/\text{kg}$ of total PCDD/PCDF or 0.73 $\mu\text{g}/\text{kg}$ on a TEQ basis (1 $\mu\text{g}/\text{kg}$, often reported as parts per billion, or ppb, equals 1,000 ng/kg, the unit used for solid samples in Table 4-16; 1 ng/kg is often reported as parts per trillion, or ppt). Levels in the kiln ash discharge from the higher temperature incineration tests were somewhat higher at 65 to 89 $\mu\text{g}/\text{kg}$ total, or 1.2 to 2.0 $\mu\text{g}/\text{kg}$ TEQ. Levels in the kiln ash discharge from the lower temperature incineration tests were substantially higher, at 830 to

TABLE 4-10. PCDDs AND PCDFs IN TEST FEED SAMPLES

Analyte	Test feed concentration, ng/kg		
	Composite fluff	Composite soil	Packaging container material
Total TCDD	140	170	1.6
2,3,7,8-TCDD	7.1 ^a	2.8	0.20 ^a
Total PeCDD	180	310	0.41
1,2,3,7,8-PeCDD	31	18	0.15 ^a
Total HxCDD	1,360	680	3.2
1,2,3,4,7,8-HxCDD	45	28	<0.5
1,2,3,6,7,8-HxCDD	110	54	0.37
1,2,3,7,8,9-HxCDD	150	77	0.30 ^a
Total HpCDD	3,160	790	20.2
1,2,3,4,6,7,8-HpCDD	1,590	430	10.0
OCDD	10,700	2,290	126
Total TCDF	2,660	1,360	6.3
2,3,7,8-TCDF	110	34	0.50 ^a
Total PeCDF	2,740	1,220	3.0
1,2,3,7,8-PeCDF	250	51	0.44
2,3,4,7,8-PeCDF	220	100	0.37
Total HxCDF	4,660	1,260	1.9
1,2,3,4,7,8-HxCDF	1,580	420	0.79
1,2,3,6,7,8-HxCDF	470	100	0.27
2,3,4,6,7,8-HxCDF	530	150	0.74 ^a
1,2,3,7,8,9-HxCDF	180	7.2 ^a	<0.4
Total HpCDF	9,360	1,000	2.9
1,2,3,4,6,7,8-HpCDF	4,180	550	2.3
1,2,3,4,7,8,9-HpCDF	2,240	120	<0.7
OCDF	22,100	920	15.7

^aEstimated maximum possible concentration; see Appendix C-6.

TABLE 4-11. PCDDs AND PCDFs IN KILN ASH SAMPLES

Kiln exit gas temperature, °C (°F)	Kiln ash concentration, ng/kg					
	Fluff waste tests				Soil tests	
	Test 1 (11/9/93)	Test 2 (11/16/93)	Test 5 (11/18/93)	Test 6 (11/23/93)	Test 3 (12/1/93)	Test 4 (12/2/93)
	883 (1,622)	876 (1,608)	762 (1,403)	767 (1,412)	876 (1,609)	874 (1,606)
Analyte						
Total TCDD	320	38	3,190	450	3.4	6.6
2,3,7,8-TCDD	12	1.0	230	23 ^a	0.09 ^a	0.18
Total PeCDD	310	290	10,500	7,120	14	19
1,2,3,7,8-PeCDD	31	19	990	400	1.2 ^a	2.2 ^a
Total HxCDD	910	2,050	40,400	53,900	47	96
1,2,3,4,7,8-HxCDD	51	120	1,850	2,080	31	6.5
1,2,3,6,7,8-HxCDD	71	160	3,100	3,650	3.6	7.4
1,2,3,7,8,9-HxCDD	210	570	8,300	9,770	14	36
Total HpCDD	3,830	8,790	117,000	107,000	110	160
1,2,3,4,6,7,8-HpCDD	1,810	4,480	56,100	107,000	57	88
OCDD	11,600	14,100	109,000	739,000	230	180
Total TCDF	3,330	2,820	64,300	72,200	150	310
2,3,7,8-TCDF	82	28	760	500	3.2	3.6
Total PeCDF	5,730	9,550	110,000	179,000	300	600
1,2,3,7,8-PeCDF	160	120	3,450	2,370	7.2	11
2,3,4,7,8-PeCDF	270	470	9,520	15,500	24	44
Total HxCDF	10,800	15,200	101,000	448,000	430	830
1,2,3,4,7,8-HxCDF	2,770	4,540 ^a	54,800 ^a	325,000 ^a	120	230
1,2,3,6,7,8-HxCDF	610	1,220	18,800	25,800	29	54
2,3,4,6,7,8-HxCDF	2,310	4,110	46,900	287,000	80	140
1,2,3,7,8,9-HxCDF	25	83	5,840	1,210	1.7	4.6
Total HpCDF	15,800	21,200	164,000	360,000	610	910
1,2,3,4,6,7,8-HpCDF	6,180	6,210	51,600	106,000	320	120
1,2,3,4,7,8,9-HpCDF	1,360	3,810	233,000	14,800	47	95
OCDF	12,200	15,400	114,000	728,000	510	500

^aEstimated maximum possible concentration; see Appendix C-6.

TABLE 4-12. PCDDs AND PCDFs IN SCRUBBER LIQUOR SAMPLES

Kiln exit gas temperature, °C (°F)	Post-test scrubber concentration, pg/L						
	Blank test	Fluff waste tests				Soil tests	
	Test 0	Test 1	Test 2	Test 5	Test 6	Test 3	Test 4
	(10/27/93)	(11/9/93)	(11/16/93)	(11/18/93)	(11/23/93)	(12/1/93)	(12/2/93)
870	883	876	762	767	876	874	
(1,599)	(1,622)	(1,608)	(1,403)	(1,412)	(1,609)	(1,606)	
Analyte							
Total TCDD	<6.1	<2.8	<5.6	13	<5.5	<3.4	<3.8
2,3,7,8-TCDD	<6.1	<2.8	<5.6	3.4	<5.5	<3.4	<3.8
Total PeCDD	<9.7	<4.3	<8.5	7.9	<10	<6.7	<9.1
1,2,3,7,8-PeCDD	<9.7	<4.3	<8.5	7.9	<10	<6.7	<9.1
Total HxCDD	<9.6	3.5 ^a	<6.9	9.1 ^a	8.2 ^a	34	<6.1
1,2,3,4,7,8-HxCDD	<11	<4.4	<7.8	<4.6	<6.1	<5.6	<6.7
1,2,3,6,7,8-HxCDD	<8.8	<3.4	<6.2	<3.6	<4.8	6.6	<5.6
1,2,3,7,8,9-HxCDD	<9.4	<3.8	<6.8	9.0 ^a	<5.3	8.2 ^a	<6.0
Total HpCDD	<15	12	18	6.6	40	100	22
1,2,3,4,6,7,8-HpCDD	<15	12	17 ^a	11 ^a	22	100	17 ^a
OCDD	<30	51	116	49	94 ^a	440	140
Total TCDF	45	4.3	8.6 ^a	8.1	5.0	98	<2.8
2,3,7,8-TCDF	28	4.3	4.9 ^a	8.1	4.9	18	<2.8
Total PeCDF	9.9	5.9 ^a	5.7	19	13 ^a	180	<4.1
1,2,3,7,8-PeCDF	<6.4	<3.1	<6.2	11	<6.7	24	4.0
2,3,4,7,8-PeCDF	9.9	<3.0	<5.9	8.5	6.4 ^a	22	<4.1
Total HxCDF	13	28	36	34	11	220	7.2
1,2,3,4,7,8-HxCDF	4.1 ^a	7.8	12 ^a	8.9	13 ^a	80	3.2
1,2,3,6,7,8-HxCDF	4.6 ^a	3.3 ^a	6.7	7.8	5.4 ^a	30	<2.5
2,3,4,6,7,8-HxCDF	6.3	12	18	<3.0	17 ^a	60	7.3
1,2,3,7,8,9-HxCDF	<6.4	3.0	<5.7	<3.3	<4.2	<3.4	<3.8
Total HpCDF	<8.2	82	110	16	130	540	29
1,2,3,4,6,7,8-HpCDF	<6.6	32	68	19 ^a	53	250	23
1,2,3,4,7,8,9-HpCDF	<11	17	36 ^a	13	30	86	<7.4
OCDF	<26	190	430	130	220	720	56

^aEstimated maximum possible concentration; see Appendix C-6.

TABLE 4-13. PCDDs AND PDCFs IN BAGHOUSE ASH SAMPLES

Kiln exit gas temperature, °C (°F)	Baghouse ash concentration, ng/kg						
	Blank test	Fluff waste tests				Soil tests	
	Test 0	Test 1	Test 2	Test 5	Test 6	Test 3	Test 4
	(10/27/93) 870 (1,559)	(11/9/93) 883 (1,622)	(11/16/93) 876 (1,608)	(11/18/93) 762 (1,403)	(11/23/93) 767 (1,412)	(12/1/93) 876 (1,609)	(12/2/93) 874 (1,606)
Analyte							
Total TCDD	0.17	0.90	<0.2	4.6	0.6 ^a	0.48	0.3 ^a
2,3,7,8-TCDD	0.08 ^a	<0.3	<0.2	0.36	<0.6	0.23	<0.1
Total PeCDD	0.19	1.5	0.62	5.3	3.9	4.6	0.85 ^a
1,2,3,7,8-PeCDD	<0.1	0.47	<0.2	1.1	<1.0	0.58	<0.2
Total HxCDD	1.3	6.4	6.2	6.0	19	21	3.3
1,2,3,4,7,8-HxCDD	<0.2	<0.4	<0.2	0.53	<0.9	1.2	0.3 ^a
1,2,3,6,7,8-HxCDD	<0.1	0.69 ^a	0.85	0.76 ^a	1.8 ^a	1.9 ^a	0.68
1,2,3,7,8,9-HxCDD	0.36	1.5	1.7	1.6	5.0	1.6 ^a	1.4 ^a
Total HpCDD	10	39	40	20	72	90	27
1,2,3,4,6,7,8-HpCDD	5.3	21	21	11	37	44	14
OCDD	38	180	180	51	200	250	67
Total TCDF	1.1	15	12.	39	15	46	8.5
2,3,7,8-TCDF	0.70	1.1	0.69	1.9	3.0	2.8	1.2
Total PeCDF	2.5	27	26	50	86	130	26
1,2,3,7,8-PeCDF	0.28	1.5	1.1	3.4	5.1	6.9	1.5 ^a
2,3,4,7,8-PeCDF	0.55	3.2	2.3	4.7	11	11	3.5
Total HxCDF	4.0	40	65	46	150	320	61
1,2,3,4,7,8-HxCDF	1.0	11	18	10	39	80	15
1,2,3,6,7,8-HxCDF	0.41	3.9 ^a	5.2	4.7	11	24	5.5
2,3,4,6,7,8-HxCDF	1.0	9.1	17	8.9	37	67	15
1,2,3,7,8,9-HxCDF	<0.2	<0.3	<0.2	1.9 ^a	2.9 ^a	1.4	0.38 ^a
Total HpCDF	3.4	83	120	59	250	740	110
1,2,3,4,6,7,8-HpCDF	2.4	40	79	29	110	41	54
1,2,3,4,7,8,9-HpCDF	0.56	8.1	15 ^a	7.0	34	52	11
OCDF	3.6	120	380	56	230	990	85

^aEstimated maximum possible concentration; see Appendix C-6.

TABLE 4-14. PCDDs AND PCDFs IN BAGHOUSE EXIT FLUE GAS

Kiln exit gas temperature, °C (°F)	Baghouse exit flue gas concentration, ng/dscm						
	Blank test	Fluff waste tests				Soil tests	
	Test 0 (10/27/93) 870 (1,599)	Test 1 (11/9/93) 883 (1,622)	Test 2 (11/16/93) 876 (1,608)	Test 5 (11/18/93) 762 (1,403)	Test 6 (11/23/93) 767 (1,412)	Test 3 (12/1/93) 876 (1,609)	Test 4 (12/2/93) 874 (1,606)
Analyte							
Total TCDD	<0.002	0.006 ^a	0.002	<0.003	0.003	<0.002	0.001
2,3,7,8-TCDD	<0.002	<0.002	<0.002	<0.003	<0.003	<0.002	<0.001
Total PeCDD	<0.002	0.006 ^a	0.006	<0.006	<0.006	<0.003	<0.001
1,2,3,7,8-PeCDD	<0.002	<0.002	<0.003	<0.006	<0.006	<0.003	<0.001
Total HxCDD	<0.005	0.021	0.006	<0.003	0.003	0.013	0.003
1,2,3,4,7,8-HxCDD	<0.005	<0.002	<0.002	<0.006	<0.006	<0.003	<0.001
1,2,3,6,7,8-HxCDD	<0.002	0.002	0.002	<0.003	<0.003	<0.002	0.002
1,2,3,7,8,9-HxCDD	<0.005	0.003	0.002	<0.003	<0.003	<0.002	0.001 ^a
Total HpCDD	0.005	0.024	0.021	0.009 ^a	0.009 ^a	0.013	0.019
1,2,3,4,6,7,8-HpCDD	0.005	0.012	0.009	0.006 ^a	0.006 ^a	0.013	0.009
OCDD	0.017	0.021	0.027	0.024	0.016	0.066	0.044
Total TCDF	0.017	0.175	0.246	0.065	0.169	0.078	0.078
2,3,7,8-TCDF	0.002	0.015	0.012	0.006	0.019	0.009	0.006
Total PeCDF	0.012 ^a	0.275	0.249	0.065	0.228	0.097	0.062
1,2,3,7,8-PeCDF	<0.002	0.021	0.024	0.009	0.019	0.009	0.006
2,3,4,7,8-PeCDF	<0.002	0.024	0.021	0.009	0.022	0.013	0.009
Total HxCDF	0.022	0.206	0.224	0.059	0.169	0.085	0.056
1,2,3,4,7,8-HxCDF	0.010	0.057	0.058	0.018	0.047	0.031	0.019
1,2,3,6,7,8-HxCDF	0.005	0.021	0.024	0.006	0.016	0.009	0.006 ^a
2,3,4,6,7,8-HxCDF	0.002 ^a	0.024	0.024	0.012	0.022	0.016 ^a	0.012
1,2,3,7,8,9-HxCDF	<0.002	<0.002	<0.002	<0.003	<0.003	<0.002	0.001
Total HpCDF	0.024 ^a	0.067	0.061	0.042	0.056	0.066	0.044
1,2,3,4,6,7,8-HpCDF	0.012 ^a	0.051	0.048	0.027	0.047	0.035	0.025
1,2,3,4,7,8,9-HpCDF	<0.005	0.006 ^a	0.009	<0.006	<0.006	0.006	0.006
OCDF	0.017	0.039	0.058	0.027	0.031	0.035	0.025

^aEstimated maximum possible concentration; see Appendix C-6.

**TABLE 4-15. 2,3,7,8-TCDD TOXICITY EQUIVALENT FACTORS
(Reference 2)**

Compound	Toxicity equivalent factor
2,3,7,8-TCDD	1
Other TCDDs	0
2,3,7,8-PeCDD	0.5
Other PeCDDs	0
2,3,7,8-HxCDD	0.1
Other HxCDDs	0
2,3,7,8-HpCDD	0.01
Other HpCDDs	0
OCDD	0.001
2,3,7,8-TCDF	0.1
Other TCDFs	0
1,2,3,7,8-PeCDF	0.05
2,3,4,7,8-PeCDF	0.5
Other PeCDFs	0
2,3,7,8-HxCDFs	0.1
Other HxCDFs	0
2,3,7,8-HpCDFs	0.01
Other HpCDFs	0
OCDF	0.001

TABLE 4-16. TOTAL DIOXINS AND TEQs IN TEST PROGRAM SAMPLES

Sample	Total PCDD/PCDF	TEQ
Test 0 (10/27/93), kiln temperature: 870°C (1,599°F)		
Packaging container material, mg/kg	180	1.2-1.3
Scrubber liquor, pg/L	68-170	9.7-25
Baghouse ash, ng/kg	64	0.94-1.0
Baghouse exit flue gas ng/dscm at 7% O ₂	0.21	0.005-0.017
Fluff Waste Tests		
Fluff feed, ng/kg	56,000	730
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)		
Kiln ash, ng/kg	65,000	1,200
Scrubber liquor, pg/L	370-380	4.6-12
Baghouse ash, ng/kg	520	6.8-7.0
Baghouse exit flue gas, ng/dscm at 7% O ₂	1.3	0.048-0.052
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)		
Kiln ash, ng/kg	89,000	2,000
Scrubber liquor, pg/L	730-750	7.0-23
Baghouse ash, ng/kg	740	8.9-9.2
Baghouse exit flue gas, ng/dscm at 7% O ₂	1.3	0.044-0.049
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)		
Kiln ash, ng/kg	830,000	29,000
Scrubber liquor, pg/L	290	17-18
Baghouse ash, ng/kg	340	81
Baghouse exit flue gas, ng/dscm at 7% O ₂	0.44	0.016-0.027
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)		
Kiln ash, ng/kg	2,700,000	110,000
Scrubber liquor, pg/L	520-540	6.7-23
Baghouse ash, ng/kg	1,000	22-23
Baghouse exit flue gas, ng/dscm at 7% O ₂	0.96	0.038-0.049

(continued)

TABLE 4-16. (continued)

Sample	Total PCDD/PCDF	TEQ
Soil Feed Tests		
Soil feed, ng/kg	10,000	210
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)		
Kiln ash, ng/kg	2,400	55
Scrubber liquor, pg/L	2,300-2,400	46-54
Baghouse ash, ng/kg	2,600	39
Baghouse exit flue gas, ng/dscm at 7% O ₂	0.68	0.025-0.032
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)		
Kiln ash, ng/kg	3,600	98
Scrubber liquor, pg/L	260-280	1.3-15
Baghouse ash, ng/kg	390	8.2-8.4
Baghouse exit flue gas, ng/dscm at 7% O ₂	0.48	0.018-0.020

2,700 µg/kg total, or 29 to 110 µg/kg TEQ. These data indicate that, not only was incineration treatment ineffective in destroying contaminant dioxins and furans in the fluff waste, in fact conditions experienced by the noncombustible fraction of the fluff waste during incineration likely led to PCDD/PCDF formation at the lower temperature.

That PCDD/PCDF formation in the kiln ash discharge occurred, at least for the fluff waste tests at the lower incineration temperatures, is further substantiated by the data in Table 4-17. The total weight of fluff waste or soil fed and the total weight of kiln ash collected for each test are combined with respective PCDD/PCDF concentrations from Table 4-16 to ultimately give the ratios of the amounts of dioxins and furans discharged in the kiln ash to the amounts introduced in the incinerator feed for each test. The data show that, for the fluff waste tests at the target 870°C (1,600°F) kiln exit gas temperature, the amount of total PCDD/PCDF discharged was 5.9 to 8.3 percent of the amount introduced to the incinerator in the fluff feed.

TABLE 4-17. RATIO OF DISCHARGED DIOXINS AND FURANS TO FED AMOUNTS

Test	Waste feed			Kiln ash discharge			Ratio of PCDD/PCDF discharged to amount fed, %	
	Total weight fed, kg	PCDD/PCDF fed, μg		Total weight discharged, kg	PCDD/PCDF discharged, μg		Total PCDD/PCDF	TEQ
		Total PCDD/PCDF	TEQ		Total PCDD/PCDF	TEQ		
Fluff Waste Tests								
Test 1 (11/9/93) Kiln temperature: 883°C (1,622°F)	303	17,000	220	15.4	1,000	18	5.9	8.4
Test 2 (11/16/93) Kiln temperature: 876°C (1,608°F)	304	17,000	220	15.9	1,420	32	8.3	14.3
Test 5 (11/18/93) Kiln temperature: 762°C (1,403°F)	307	17,200	220	20.4	16,900	590	98.5	264
Test 6 (11/23/93) Kiln temperature: 767°C (1,412°F)	305	17,100	220	17.3	46,700	1,900	273	855
Soil Feed Tests								
Test 3 (12/1/93) Kiln temperature: 876°C (1,609°F)	288	2,900	60	101	240	5.6	8.5	9.2
Test 4 (12/2/93) Kiln temperature: 874°C (1,606°F)	290	2,900	61	145	520	14.2	18.0	23.4

This would correspond to an effectiveness of dioxin/furan decontamination by incineration at this higher tested temperature of 91.7 to 94.1 percent. On a TEQ basis, the ratio of discharged-to-fed dioxins/furans was 8.4 to 14.3 percent, corresponding to incineration decontamination effectiveness or a TEQ basis of 85.7 to 91.6 percent at the higher incineration temperature.

However, for the fluff waste tests at the 760°C (1,400°F) target kiln exit gas temperature, the amount of total PCDD/PCDF discharged was 98.5 to 273 percent of the amount fed. In other words, the quantity of total PCDD/PCDF discharged for the lower kiln temperature fluff tests was roughly the same to 2.7 times the amount fed to the incinerator. On a TEQ basis the ratios of discharged to feed dioxins/furans were even larger, at 264 to 855 percent. Clearly, at the lower incineration temperatures, dioxins/furans were being produced in the noncombustible fraction of the fluff waste feed ultimately discharged as kiln ash.

This should not be surprising, however. It has become recognized over the past few years that dioxins and furans arising out of combustion processes result from the formation of these compounds from precursor organic constituents and a chlorine source, such as HCl, at relatively low temperatures (Reference 5). The presence of metal-containing solids, such as particulate, appears to catalyze the process. Copper has specifically been shown to catalyze reactions leading to dioxin/furan formation. The rate of dioxin/furan formation is highest at temperatures near 300°C (570°F), and this rate decreases as the temperature at which precursors, a chlorine source, and metal-bearing solids are held is either increased or decreased.

Evidently, the right combination of conditions were in place in the kiln solids bed before, or shortly after, discharge from the kiln into the ash collection pit of the RKS during the incineration of the fluff waste at the 760°C (1,400°F) target kiln temperature. Dioxin/furan precursors were likely present in the near-bed combustion gas, and chlorine, likely in the form of HCl from the chloroorganic components of the fluff, was likely in abundance. The fluff waste

tested contained 17 percent by weight chlorine. As discussed below, a major contaminant metal in the fluff waste was copper, so this likely dioxin-formation catalyst was present. Apparently kiln solids bed temperatures were sufficiently close to the peak reaction temperature of 300°C (570°F) to be within a "dioxin formation" window. The data clearly show that dioxin formation occurred at the lower incineration temperature tested.

All other dioxin formation conditions would have been in effect for the fluff incineration tests at the 870°C (1,600°F) target kiln exit gas temperature. However, at this higher incineration temperature, kiln solids bed temperatures were apparently above the window associated with more rapid dioxin formation. Similar results were seen in the soil feed tests, also performed at the higher, 870°C (1,600°F), target kiln exit gas temperature. Ratios of discharged to feed PCDD/PCDFs noted in Table 4-17 for the soil tests are comparable to those experienced for the fluff waste tests at the higher incineration temperature.

Returning to the data in Table 4-16, the scrubber liquor for the fluff waste tests contained total PCDD/PCDF concentrations in the 290 to 750 pg/L (1 pg/L is often reported as parts per quadrillion, or ppq). Scrubber liquor concentrations were in the 4.6 to 23 pg/L ranges on a TEQ basis. No apparent difference in the scrubber liquor concentrations with incineration temperature was seen. The scrubber liquor concentration measured during the blank burn test was comparable to those for the fluff waste tests on a TEQ basis, though total PCDD/PCDF concentrations were slightly lower. Scrubber liquor dioxin/furan concentrations for one of the two soil feed tests were also comparable to those measured for the fluff waste tests, although levels measured for the other soil feed test were substantially higher.

Baghouse ash total PCDD/PCDF concentrations ranged from 340 to 1,000 ng/kg (ppt) for the fluff waste tests, with no apparent change associated with changing incineration temperature. On a TEQ basis, the measured range was 6.8 to 23 ng/kg. Baghouse ash dioxin

levels were lower for the blank burn test on both bases. As for the scrubber liquor, baghouse ash dioxin levels for one of the two soil feed tests were comparable to those measured for the fluff waste tests; they were higher for the other soil feed test.

Baghouse exit flue gas total PCDD/PCDF levels were 0.021 ng/dscm corrected to 7 percent O₂ for the blank burn test. Measured levels were increased, at 1.3 ng/dscm at 7 percent O₂, for the fluff waste tests at the 870°C (1,600°F) target kiln exit gas temperature. Levels for the fluff waste test at the 760°C (1,400°F) target kiln exit gas temperature, at 0.44 to 0.96 ng/dscm at 7 percent O₂, were slightly lower than for the higher temperature tests. Levels for the soil feed tests were comparable, at 0.48 to 0.68 ng/dscm. All measured levels were significantly lower than the EPA guidance announced in 1993 of 30 ng/dscm at 7 percent O₂.

On a TEQ basis, baghouse exit flue gas dioxin/furan levels were 0.005 to 0.017 ng/dscm at 7 percent O₂ for the blank burn test, increased, at 0.044 to 0.052 ng/dscm at 7 percent O₂ for the fluff waste tests at the 870°C (1,600°F) target kiln exit gas temperature. Compared to these latter levels, comparable to slightly decreased emissions, at 0.016 to 0.049 ng/dscm at 7 percent O₂, were measured for the fluff waste tests at the 760°C (1,400°F) target kiln exit gas temperature. Levels measured for the soil feed tests were also comparable, at 0.018 to 0.032 ng/dscm at 7 percent O₂. The European suggested dioxin emission limit for waste incinerators is 0.1 ng/Nm³ TEQ corrected to 11 percent O₂. Thus, while the temperature correction for scm is slightly different than for Nm³, and the O₂ correction for the European standard, at 11 percent O₂, differs from the 7 percent O₂ used in the Table 4-16 data, all emission levels reported in Table 4-16 are lower than the suggested European standard.

4.5 TRACE METAL AND TCLP ANALYSIS RESULTS

Trace metal concentrations measured in test program samples are summarized in Table 4-18. The data in the table clearly show that the major metal contaminants in both the

TABLE 4-18. TRACE METAL ANALYSIS RESULTS

Sample	Concentration									
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn
Test 0 (10/27/93), kiln temperature: 870°C (1,599°F)										
Packaging container material, mg/kg	<20	<20	6.7	<0.5	1.0	<2	<10	<4	<0.7	8.9
Scrubber liquor, mg/L	0.7	<0.05	8.5	0.05	0.7	0.6	18	0.30	<0.007	6.6
Baghouse ash, mg/kg	120	<20	18	6.6	510	58	410	150	<0.7	1,800
Fluff Waste Tests										
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)										
Fluff feed, mg/kg	120	<20	74	1.4	34	8,800	2,400	3.1	0.9	100
Kiln ash, mg/kg	1,100	<20	270	<0.5	390	186,000	3,000	250	1.7	190
Scrubber liquor, mg/L	4.7	0.12	2.4	0.20	1.9	210	789	1.2	0.07	19
Baghouse ash, mg/kg	830	24	38	33	520	31,000	30,000	120	<0.7	4,100
Baghouse exit flue gas, µg/dscm	<21	<19	50	<50	<9	85	1,600	6	<2	26
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)										
Fluff feed, mg/kg	180	<20	70	1.5	38	8,400	2,400	3.4	1.3	180
Kiln ash, mg/kg	940	30	220	<0.5	480	142,000	3,800	840	4.4	260
Scrubber liquor, mg/L	3.7	0.08	1.8	0.14	1.4	130	490	1.0	0.08	11
Baghouse ash, mg/kg	400	<20	22	18	320	14,000	19,000	130	<0.7	2,100
Baghouse exit flue gas, µg/dscm	<11	<22	6	<1	<4	44	130	<5	<2	18
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)										
Fluff feed, mg/kg	90	<20	47	1.3	27	8,500	1,100	<3	1.2	140
Kiln ash, mg/kg	950	<20	240	0.6	460	115,000	5,700	150	3.0	240
Scrubber liquor, mg/L	4.7	0.20	1.5	0.20	1.4	170	560	0.8	0.06	12
Baghouse ash, mg/kg	1,300	<20	35	54	470	56,000	28,000	150	<0.7	4,400
Baghouse exit flue gas, µg/dscm	<11	<16	6	<1	<4	38	46	<5	<2	13

(continued)

TABLE 4-18. (continued)

Sample	Concentration									
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn
Fluff Waste Tests (concluded)										
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)										
Fluff feed, mg/kg	100	<20	81	1.0	24	8,400	900	4.6	1.3	120
Kiln ash, mg/kg	950	46	250	<0.5	530	176,000	5,700	340	3.0	250
Scrubber liquor, mg/L	4.9	0.10	1.4	0.20	1.4	180	610	0.9	0.08	12
Baghouse ash, mg/kg	1,900	<20	19	70	510	77,000	38,000	160	1.0	5,000
Baghouse exit flue gas, µg/dscm	<10	<14	5	<1	<4	26	38	<4	<2	44
Soil Feed Tests										
Average soil feed, mg/kg	66	<20	72	1.3	85	14,000	3,100	29	<2	190
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)										
Kiln ash, mg/kg	190	<20	120	<0.5	73	53,000	4,100	62	1.5	320
Scrubber liquor, mg/L	1.5	<0.05	1.3	0.10	0.8	110	120	0.3	0.02	3
Baghouse ash, mg/kg	1,400	<70	29	57	540	64,000	48,000	190	<0.7	4,200
Baghouse exit flue gas, µg/dscm	<10	<15	10	<1	<3	51	190	<9	<2	20
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)										
Kiln ash, mg/kg	190	<20	91	<0.5	56	35,000	4,100	51	1.9	290
Scrubber liquor, mg/L	1.3	<0.05	1.3	0.20	1.0	160	180	0.4	0.02	4
Baghouse ash, mg/kg	950	<20	19	43	520	52,000	41,000	200	<0.7	2,900
Baghouse exit flue gas, µg/dscm	<13	<29	6	<2	<3	620	2,030	<6	<2	42

fluff waste and the contaminated soil were copper and lead. Both of these metals were also present in the kiln ash discharge for all tests. The presence of high concentrations of these metals, especially copper, in the kiln ash discharge substantiates that presumed catalysts for the relatively low temperature reactions in the dioxin formation pathway would be present in the kiln ash so that dioxin formation in this matrix, especially as noticed for the lower incineration temperature fluff waste tests, can be understood.

Fluff and soil feed, kiln ash, scrubber liquor, and baghouse ash samples from the test program were subjected to the TCLP, and resulting TCLP leachates were analyzed for the test program trace metals. Leachate analysis data are summarized in Table 4-19. For the six TCLP metals with regulatory levels defined, the regulatory level is also given in the table. The data in the table show that the fluff waste from two of the four tests would be a lead-contaminated TC hazardous waste. Further, the lead concentrations in the leachates of the fluff for the other two tests were very close to the regulatory level for lead. Despite this, no resulting kiln ash discharge from the incineration of fluff waste would be a TC hazardous waste due to its leachable lead, or any other metal analyzed, concentration. Similarly, the scrubber liquor from all fluff waste tests was not TC hazardous. However, the baghouse ash for all fluff waste tests would be a lead-contaminated TC hazardous waste, and for three of the four tests a cadmium-contaminated TC hazardous waste.

Although the contaminated soil tested was not a TC hazardous waste, conclusions regarding the TC status of the residual discharges from its incineration were the same as for the fluff waste. Namely, neither the kiln ash discharge nor the scrubber liquor resulting from its incineration under the conditions tested would possess the TC, and the baghouse ash for both tests performed would be considered both cadmium- and lead-contaminated TC hazardous waste.

TABLE 4-19. TCLP LEACHATE ANALYSIS RESULTS

Sample leached	Leachate concentration, mg/L									
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn
Regulatory Level	— ^a	5	100	1	5	—	5	—	5	—
Test 0 (10/27/93), kiln temperature: 870°C (1,599°F)										
Scrubber liquor	<0.03	<0.05	0.17	<0.004	0.052	0.05	<0.04	<0.01	<0.007	0.12
Baghouse ash	3.3	0.34	0.25	0.30	4.5	1.4	0.07	1.7	<0.007	84
Fluff Waste Tests										
Test 1 (11/9/93), kiln temperature: 883°C (1,622°F)										
Fluff feed	0.23	<0.05	0.25	0.03	<0.007	120	3.6	0.02	<0.007	3.2
Kiln ash	<0.03	<0.05	1.2	<0.004	0.32	0.14	0.42	<0.01	<0.007	0.03
Scrubber liquor	0.55	<0.05	0.93	0.03	0.06	0.91	0.52	0.08	0.08	0.16
Baghouse ash	0.85	0.20	0.23	1.4	0.29	720	3,800	2.0	<0.007	150
Test 2 (11/16/93), kiln temperature: 876°C (1,608°F)										
Fluff feed	0.26	<0.05	0.26	0.04	<0.007	130	5.4	0.02	<0.007	4.1
Kiln ash	0.05	<0.05	1.3	<0.004	0.41	0.05	0.09	0.01	<0.007	0.07
Scrubber liquor	0.42	<0.05	0.76	0.03	0.04	0.09	0.40	0.06	0.09	0.13
Baghouse ash	0.43	0.08	0.05	0.9	0.20	340	1,900	28	0.02	100
Test 5 (11/18/93), kiln temperature: 762°C (1,403°F)										
Fluff feed	0.26	<0.05	0.19	0.03	<0.007	130	3.7	0.02	<0.007	3.7
Kiln ash	0.24	<0.2	2.2	<0.005	0.05	<0.02	<0.1	<0.01	<0.007	0.02
Scrubber liquor	0.60	<0.05	0.60	0.03	0.04	1.3	2.2	0.05	0.06	0.10
Baghouse ash	<0.2	<0.2	0.4	1.8	0.13	1,400	5,200	2.3	0.02	180
Test 6 (11/23/93), kiln temperature: 767°C (1,412°F)										
Fluff feed	0.26	<0.05	0.55	0.03	<0.007	130	5.8	0.02	<0.007	4.6
Kiln ash	0.28	<0.2	1.2	<0.005	0.01	0.072	0.50	<0.01	<0.007	0.02
Scrubber liquor	0.71	<0.05	0.6	0.02	0.05	1.2	0.6	0.06	0.08	0.10
Baghouse ash	<0.2	<0.2	0.6	1.8	0.2	1,400	4,400	2.8	0.02	200

^a — = No regulatory level; not a TCLP metal.

(continued)

TABLE 4-19. (continued)

Sample leached	Leachate concentration, mg/L									
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn
Regulatory Level	— ^a	5	100	1	5	—	5	—	5	—
Soil Feed Tests										
Average soil feed	0.09	<0.05	0.91	0.02	<0.007	21	0.67	0.02	<0.007	1.1
Test 3 (12/1/93), kiln temperature: 876°C (1,609°F)										
Kiln ash	<0.2	<0.2	0.26	<0.005	0.05	0.25	<0.1	<0.01	<0.007	1.3
Scrubber liquor	0.09	<0.05	0.16	<0.004	0.04	0.02	0.08	<0.01	<0.007	0.01
Baghouse ash	0.90	0.2	0.50	2.2	0.3	760	6,600	2.7	0.03	120
Test 4 (12/2/93), kiln temperature: 874°C (1,606°F)										
Kiln ash	<0.2	<0.2	0.70	<0.005	0.03	0.2	0.2	<0.01	<0.007	0.04
Scrubber liquor	0.08	<0.05	0.09	<0.004	0.10	0.09	0.39	0.24	<0.007	0.03
Baghouse ash	0.80	0.24	0.2	1.7	0.3	640	5,700	3.2	0.03	100

^a — = No regulatory level; not a TCLP metal.

The scrubber liquor TCLP leachate metals content data given in Table 4-19 deserve some discussion, especially when compared to the scrubber liquor metals content data given in Table 4-18. The scrubber liquor metals data in Table 4-18 correspond to complete scrubber liquor samples, including the suspended solids in the liquor. For liquid samples containing suspended solids, the TCLP specifies filtering the sample. If the resulting filtered solids content of the sample is less than 0.5 percent (the case for all scrubber liquor samples collected in this test program), then the filtrate is defined to be the sample TCLP leachate.

Comparing the scrubber liquor data in Table 4-19 to the data in Table 4-18 shows that the metals concentrations in the scrubber liquor TCLP leachate are almost always less than, and in many cases much less than, the corresponding concentrations in the complete scrubber liquor. This suggests that most of the scrubber liquor's metals content was accounted for in the suspended solids fraction of the liquor.

Special attention to the lead concentrations is warranted. Lead concentrations in bulk scrubber liquor samples were in the 120 to 789 mg/L range for the fluff waste and soil feed tests. These levels far exceed the regulatory level for lead of 5 mg/L. However, lead concentrations in scrubber liquor TCLP leachates (scrubber liquor filtrate after removal of the suspended solids) ranged from 0.08 to 2.2 mg/L over the fluff waste and soil feed tests, all below lead's regulatory level.

4.6 PARTICULATE AND HCl EMISSIONS

The baghouse exit flue gas particulate and HCl emission data developed in the test program are summarized in Table 4-20. The data show that baghouse exit particulate concentrations were less than 10 mg/dscm corrected to 7 percent O₂ for all but one test for which they were 14 mg/dscm at 7 percent O₂. All measured levels were well below the current

TABLE 4-20. PARTICULATE AND HCl EMISSIONS

Test	Cl feedrate, kg/hr	Baghouse exit		
		Particulate concentration, mg/dscm at 7% O ₂	HCl emission rate, g/hr	Apparent system HCl collection efficiency, %
Test 0 (10/27/93)	0.28	7	<0.2	>99.93
Fluff Waste Tests				
Test 1 (11/9/93)	9.48	7	1.7	99.98
Test 2 (11/16/93)	9.48	4	2.0	99.98
Test 5 (11/18/93)	9.48	6	2.0	99.98
Test 6 (11/23/93)	9.48	14	2.3	99.98
Soil Feed Tests				
Test 3 (12/1/93)	1.1	5	2.6	99.76
Test 4 (12/2/93)	1.1	9	0.7	99.94

hazardous waste incinerator performance standard of 180 mg/dscm at 7 percent O₂, and even substantially below the EPA's announced 1993 guidance of 34 mg/dscm at 7 percent O₂.

Baghouse exit flue gas HCl emission rates were at most 2.6 g/hr. Apparent system collection HCl efficiencies were greater than 99.9 percent for all except one soil feed test for which the apparent system HCl collection efficiency was 99.76 percent.

Particle size distribution measurements were also performed for each test using an Andersen cascade impactor train in the afterburner extension. The cascade impactor particle size distribution data analysis worksheets are presented in Appendix D-6. Figure 4-1 shows the size distribution data in the form of log-probability plot of cumulative mass percent less than particle diameter (y-axis, probability scale) versus particle diameter (x-axis, log scale). A straight line on such a plot corresponds to a log-normal size distribution.

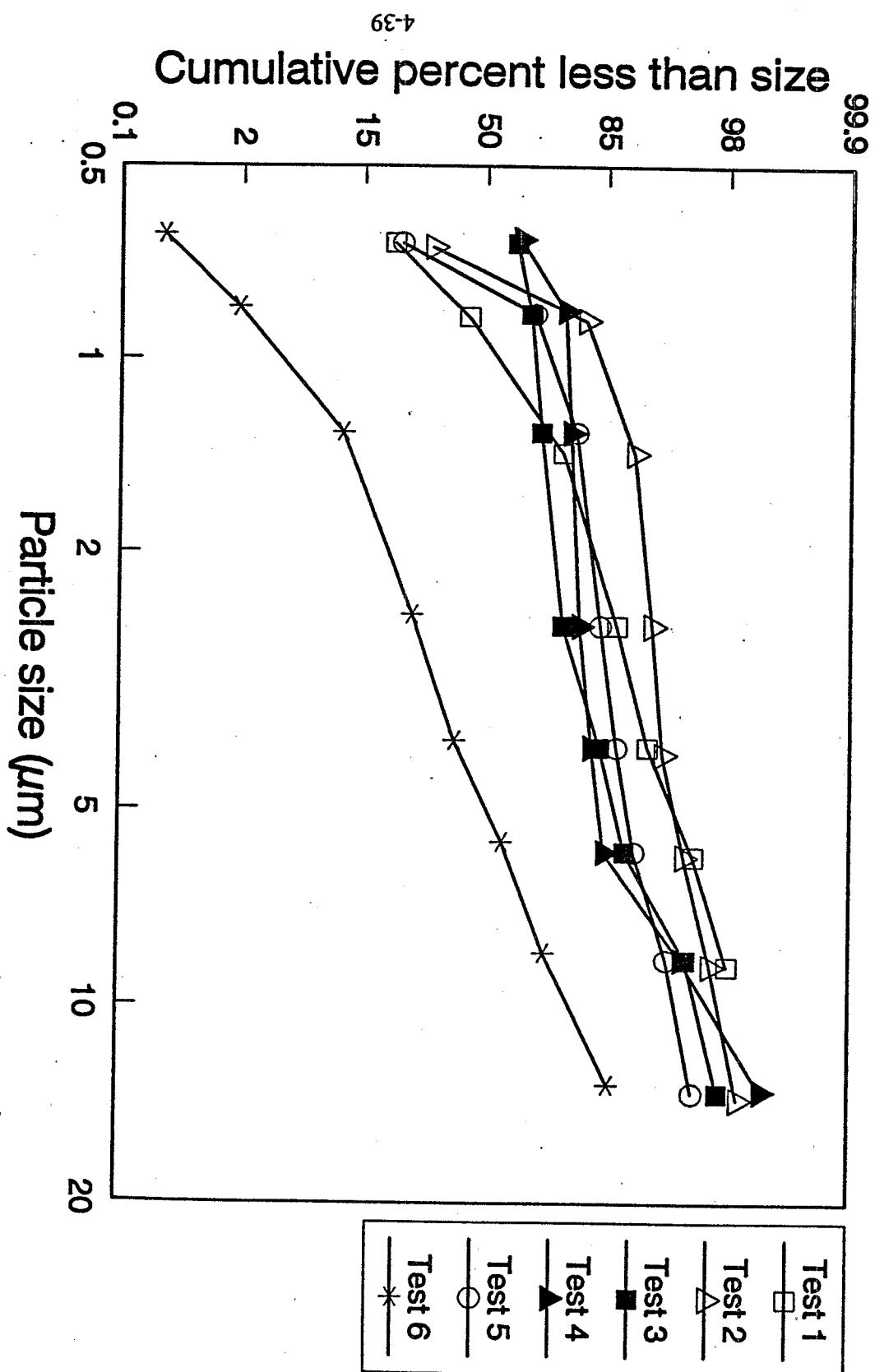


Figure 4-1. Afterburner exit particle size distributions.

The distribution data shown in Figure 4-1 indicate that the size distributions of the afterburner exit particulate were comparable for 5 of the 6 tests, with the particulate for these 5 tests being relatively fine, with 50 percent less than 1 μm . The distribution for Test 6 was distinctly shifted to much larger particle size, with over 90 percent greater than 1 μm . No explanation for this shift is offered here.

SECTION 5

CONCLUSIONS

Results of the test program conducted to evaluate the incineration treatment of fluff waste and contaminated soil from the M. W. Manufacturing Superfund site confirm that incineration represents an effective treatment option, but several cautions regarding its use need emphasis. Indeed, incineration of the fluff waste offers several benefits including substantial waste volume reduction, and effective, near complete, decontamination and destruction of both the VOC and SVOC contaminants in the waste. While the volume reduction benefit is less significant in the incineration treatment of the contaminated soil, the benefit of effective and near complete decontamination and destruction of organic POHC contaminants remains.

Both site materials can be incinerated in compliance with the current hazardous waste incinerator performance standards in a rotary kiln incineration system of the type in place at the IRF with an APCS consisting of a wet scrubber for acid gas control and a baghouse for final particulate control. Specifically:

- Greater than 99.99 percent POHC DREs were uniformly measured
- HCl emissions were well below 1.8 kg/hr and system HCl control efficiencies well above 99 percent

In addition, compliance with the more recent incinerator emissions guidance announced in 1993 was demonstrated. Specifically:

- Particulate emissions measured were well below 34 mg/dscm corrected to 7 percent O₂
- Total PCDD/PCDF emissions measured were well below 30 ng/dscm corrected to 7 percent O₂

In fact, measured dioxin/furan emissions on a TEQ basis were well below the suggested European emission limit of 0.1 ng/Nm³ dry at 11 percent O₂.

However, the kiln ash discharge from the incineration of both site materials remains dioxin-contaminated. The kiln ash discharge from the incineration of contaminated site soil at a kiln temperature of nominally 870°C (1,600°F) contained total PCDD/PCDF concentrations of 2.4 to 3.6 µg/kg. Levels in the kiln ash discharge from the incineration of fluff waste at a nominal kiln temperature of 870°C (1,600°F) were higher, at 65 to 89 µg/kg. Levels in the kiln ash discharge from the incineration of fluff waste at a nominal kiln temperature of 760°C (1,400°F) were substantially higher, at 830 to 2,700 µg/kg.

Thus, with respect to fluff waste, incineration offers substantial volume reduction, however the resulting treated waste discharge (kiln ash) may still need to be managed as a dioxin-contaminated material. Dioxin contamination levels were decreased at higher incineration temperatures, but they remained significant nonetheless. Perhaps even higher incineration temperatures, with or without the use of an ash water quench system, would give a kiln ash discharge relatively free of dioxin contamination. However, further tests are needed to investigate this possibility.

In addition, the flue gas particulate collected as baghouse ash in essentially all tests was a cadmium- and lead-contaminated TC hazardous waste. Thus, this discharge would need to be appropriately managed as a hazardous waste.

All quality assurance (QA) objectives for the test program were met, with the exception of:

- The MDL objectives for:
 - Tetrachloroethene in solid residues
 - Trichloroethene in aqueous liquids
 - 2-nitrophenol in flue gas
 - Zinc in aqueous liquids
- The accuracy and precision objectives for antimony, barium, and silver as assessed by analyte recovery from MS samples and the RPD of MS/MSD analyses
- The accuracy objective for the other seven test trace metals as assessed by analyte recovery from MS samples

The MDLs achieved for the above four analyte/sample matrix combinations were not significantly greater than the respective objectives, so no measurable effects on test program conclusions resulted.

Failure to meet the precision and accuracy objectives for the antimony, barium, and silver analyses leads to the conclusion that test program results for these three metals are compromised. The reported data for these three metals should be treated with caution, and test program conclusions regarding these three metals must be viewed as tentative.

The accuracy objective of 70 to 130 percent recovery from MS samples was not met for the other seven test trace metals. However, an objective of 60 to 140 percent recovery would have been met. This suggests that test program results and conclusions regarding these seven analytes are valid and defensible, but are just not as certain as had been planned.

SECTION 6

QUALITY ASSURANCE

This test program was carried out as outlined in the Quality Assurance Project Plan (QAPP) for Pilot-Scale Incineration Testing of Fluff Waste and Contaminated Soil from the M. W. Manufacturing Superfund Site. Thus, all QA aspects of the program were completed as specified in the QAPP. Except as noted, all tests were performed in accordance with the procedures documented in the QAPP.

All samples analyzed to obtain data reported in this report were taken at the IRF by members of the IRF operating staff. All samples were collected and/or recovered in accordance with the methods appropriate to their eventual analysis. After appropriate preservation, the samples were relinquished to the custody of the onsite Sample Custodian. The Sample Custodian subsequently directed the splitting of samples and the transport of these to the appropriate laboratories for analysis. The sample chain-of-custody procedures described in the QAPP for these tests were followed. No compromise in sample integrity occurred, with one exception as noted below.

Numerous QA procedures were followed to assess the data quality of laboratory analytical measurements performed in this test program. These included blank sample analyses, duplicate analyses, and matrix spike (MS) and matrix spike duplicate (MSD) sample analyses. Method blank samples were analyzed for all sample matrices for which logical matrix blanks

could be prepared. Results of QA procedures performed for the critical laboratory measurements are discussed, by analyte group, in the following subsections.

6.1 VOC ANALYSES

A total of 50 solid and aqueous samples was analyzed for VOCs by GC/FID using Method 8015A. Included in this group were 5 MS/MSD sample sets and 5 duplicate analyses of test samples. Table 6-1 lists sample collection date, analysis date, and analysis hold time for these samples. The data in Table 6-1 show that 49 of the 50 samples were analyzed within the method hold time limit of 14 days. Table 6-2 provides an analogous listing for flue gas samples analyzed for volatile organic constituents by GC/MS using Method 5040. The data in Table 6-2 show that 35 out of 36 samples were analyzed, and that all of those analyzed were analyzed within the method hold time limit of 42 days. One sample, the Test 3 field blank, was lost in shipment (the compromise in sample integrity mentioned above). Still, analytical completeness was 97 percent (35 of 36).

Table 6-3 summarizes the VOC analysis quality assurance objectives (QAOs) for precision, accuracy, and completeness. Table 6-4 shows the method detection limit (MDL) objectives and the achieved values for the VOC analyses. As can be seen in Table 6-4 the MDL objective was met for all primary target analytes except tetrachloroethene in solid residues and trichloroethene in aqueous liquids. In addition, all secondary target analyte MDLs in flue gas samples were met.

VOC analysis precision and accuracy were assessed for GC/FID analyses by preparing one MS/MSD sample set for each of the fluff waste, contaminated soil, scrubber liquor, kiln ash, and baghouse ash sample matrices and measuring spike recovery. One pair of VOST traps was spiked with the primary target analytes for each test and analyzed by GC/MS. In addition, one

TABLE 6-1. SAMPLE HOLD TIMES FOR THE VOC ANALYSES OF SOLID AND LIQUID SAMPLES BY GC/FID

Sample	Collection date	Analysis date	Analysis hold time, days
Test Feed			
Test 0	12/21/93	01/10/94	20
Composite fluff	10/14/93	10/25/93	11
Composite fluff duplicate	10/14/93	10/25/93	11
Composite fluff MS	10/14/93	10/27/93	13
Composite fluff MSD	10/14/93	10/27/93	13
Test 1	10/14/93	10/25/93	11
Test 2	10/14/93	10/26/93	12
Test 5	10/18/93	10/26/93	8
Test 6	10/18/93	10/29/93	11
Test 3	10/20/93	10/28/93	8
Test 3 duplicate	10/20/93	10/28/93	8
Test 3 MS	10/20/93	10/29/93	9
Test 3 MSD	10/20/93	10/29/93	9
Test 4	10/20/93	10/29/93	9
Scrubber Liquor			
Test 0 pretest	10/27/93	11/03/93	7
Test 0 post-test	10/27/93	11/03/93	7
Test 0 post-test duplicate	10/27/93	11/04/93	8
Test 0 post-test MS	10/27/93	11/03/93	7
Test 0 post-test MSD	10/27/93	11/03/93	7
Test 1 pretest	11/09/93	11/11/93	2
Test 1 post-test	11/09/93	11/11/93	2
Test 2 pretest	11/16/93	11/23/93	7
Test 2 post-test	11/16/93	11/23/93	7
Test 3 pretest	12/01/93	12/07/93	6
Test 3 post-test	12/01/93	12/07/83	6
Test 4 pretest	12/02/93	12/07/93	5
Test 4 post-test	12/02/93	12/07/93	5
Test 5 pretest	11/18/93	11/29/93	11
Test 5 post-test	11/18/93	11/29/93	11
Test 6 pretest	11/23/93	12/06/93	13
Test 6 post-test	11/23/93	12/06/93	13
Method Requirement			14

(continued)

TABLE 6-1. (continued)

Sample	Collection date	Analysis date	Analysis hold time, days
Kiln Ash			
Test 1	11/09/93	11/11/93	2
Test 2	11/16/93	11/23/93	7
Test 3	12/01/93	12/08/93	7
Test 3 duplicate	12/01/93	12/08/93	7
Test 3 MS	12/01/93	12/08/93	7
Test 3 MSD	12/01/93	12/08/93	7
Test 4	12/02/93	12/10/93	8
Test 5	11/18/93	11/29/93	11
Test 6	11/23/93	12/06/93	13
Baghouse Ash			
Test 0	10/28/93	11/04/93	7
Test 1	11/09/93	11/11/93	2
Test 2	11/16/93	11/23/93	7
Test 3	12/01/93	12/09/93	8
Test 3 duplicate	12/01/93	12/09/93	8
Test 3 MS	12/01/93	12/09/93	8
Test 3 MSD	12/01/93	12/09/93	8
Test 4	12/02/93	12/10/93	8
Test 5	11/18/93	11/29/93	11
Test 6	11/23/93	12/06/93	13
Method Requirement			14

**TABLE 6-2. SAMPLE HOLD TIMES FOR THE VOC ANALYSES OF METHOD 0030
SAMPLES BY GC/MS**

Sample	Collection date	Analysis date	Analysis hold time, days
Test 0, Set 1	10/27/93	11/09/93	12
Test 0, Set 2	10/27/93	11/09/93	12
Test 0, Set 3	10/27/93	11/09/93	12
Test 0, Field blank	10/27/93	11/09/93	12
Test 0, MS	10/27/93	11/09/93	12
Test 1, Set 1	11/09/93	11/22/93	13
Test 1, Set 2	11/09/93	11/22/93	13
Test 1, Set 3	11/09/93	11/22/93	13
Test 1, Field blank	11/09/93	11/22/93	13
Test 1, MS	11/09/93	11/22/93	13
Test 2, Set 1	11/16/93	12/03/93	17
Test 2, Set 2	11/16/93	12/03/93	17
Test 2, Set 3	11/16/93	12/03/93	17
Test 2, Field blank	11/16/93	12/03/93	17
Test 2 MS	11/16/93	12/03/93	17
Test 3, Set 1	12/01/93	12/16/93	15
Test 3, Set 2	12/01/93	12/16/93	15
Test 3, Set 3	12/01/93	12/16/93	15
Test 3, Field blank	12/01/93	lost	
Test 3, MS	12/01/93	12/16/93	15
Test 4, Set 1	12/02/93	12/17/93	15
Test 4, Set 2	12/02/93	12/17/93	15
Test 4, Set 3	12/02/93	12/16/93	14
Test 4, Field blank	12/02/93	12/16/93	14
Test 4, MS	12/02/93	12/16/93	14
Test 5, Set 1	11/18/93	12/08/93	20
Test 5, Set 2	11/18/93	12/08/93	20
Test 5, Set 3	11/18/93	12/08/93	20
Test 5, Field blank	11/18/93	12/03/93	15
Test 5, MS	11/18/93	12/03/93	15
Test 6, Set 1	11/23/93	12/16/93	23
Test 6, Set 2	11/23/93	12/16/93	23
Test 6, Set 3	11/23/93	12/16/93	23
Test 6, Field blank	11/23/93	12/16/93	23
Test 6, MS	11/23/93	12/16/93	23
Trip blank	12/07/93	12/16/93	9
Method Requirement			42

TABLE 6-3. VOC MEASUREMENT QAOS

Measurement parameter	Measurement/ analytical method	Reference	Conditions	Precision, % RSD or RPD	Accuracy, %	Completeness, %
Volatile organic contaminants in feed, and residue samples	Purge and trap GC/FID	SW-846 Method 8015A	Methanol extract of solid samples	50	52 to 157 ^a	70
Volatile organic contaminants in flue gas sampling trains	Method 0030 sampling, GC/MS analysis	SW-846 Methods 5040 and 8240A	—	70	52 to 157 ^a	70

^aCompound-specific criteria taken from Table 6, Method 8240A.

TABLE 6-4. VOC MEASUREMENT MDLs: OBJECTIVES AND ACHIEVED LEVELS

Compound	MDL objective		
	Solid residues, mg/kg objective / achieved	Aqueous liquids, µg/L objective / achieved	Flue gas, µg/dscm objective / achieved
Primary Target Analytes			
Tetrachloroethene	2 / 3.9	20 / 4.1	2 / 0.05
1,1,2-Trichloroethane	1 / 1	10 / 3.9	1 / 0.14
Trichloroethene	1 / 1	10 / 15.4	1 / 0.05
Secondary Analytes			
Acetone	NA ^a	NA	2 / 0.05
Benzene	NA	NA	2 / 0.23
Bromodichloromethane	NA	NA	2 / 0.05
Carbon disulfide	NA	NA	2 / 0.05
Carbon tetrachloride	NA	NA	2 / 0.05
Chlorobenzene	NA	NA	2 / 0.05
Chlorodibromomethane	NA	NA	2 / 0.05
Chloroform	NA	NA	2 / 0.05
1,1-Dichloroethane	NA	NA	2 / 0.05
1,2-Dichloroethane	NA	NA	2 / 0.05
1,1-Dichloroethene	NA	NA	2 / 0.05
trans,1,2-Dichloroethene	NA	NA	2 / 0.05
1,2-Dichloropropane	NA	NA	2 / 0.05
cis-1,3-Dichloropropene	NA	NA	2 / 0.05
trans-1,3-Dichloropropene	NA	NA	2 / 0.05
Methylene chloride	NA	NA	2 / 0.09
Toluene	NA	NA	2 / 0.23
1,1,1-Trichloroethane	NA	NA	2 / 0.05
Trichlorofluoromethane	NA	NA	2 / 0.05

^aNA = Not applicable; analyte not measured in this matrix.

MS/MSD VOST sample set (two pairs of VOST traps) was spiked with the full set of VOC analytes listed in Table 3-7.

Table 6-5 summarizes VOC recoveries achieved from solid and liquid MS samples analyzed by GC/FID. The data in Table 6-5 show 29 out of 30 measurements, or 97 percent, were within the compound-specific recovery range. As the completeness QAO was 70 percent

TABLE 6-5. VOC RECOVERIES FROM MS SAMPLES ANALYZED BY GC/FID

Sample	Spike recovery, %			Precision QAO, RPD
	Tetrachloroethene	1,1,2-Trichloroethane	Trichloroethene	
Test Feed				
Composite fluff MS	91.1	90.6	90.7	50
Composite fluff MSD	95.4	94.0	94.8	
RPD, %	4.6	3.7	4.4	
Test 3 soil MS	125	102	102	50
Test 3 soil MSD	155	98.8	99.6	
RPD, %	21.4	3.2	2.4	
Post-test Scrubber Liquor				
Test 0 MS	102	101	104	50
Test 0 MSD	101	100	102	
RPD, %	1.0	1.0	1.9	
Kiln Ash				
Test 3 MS	88.8	87.1	90.7	50
Test 3 MSD	87.0	87.1	91.1	
RPD, %	2.0	0	0.4	
Baghouse Ash				
Test 3 MS	94.7	85.4	100	50
Test 3 MSD	97.8	99.8	102	
RPD, %	3.2	15.6	2.0	
Accuracy QAO	64-148	52-150	71-157	

for this measurement, the accuracy QAO, as measured by spike recovery from MS/MSD samples, was met.

The data in Table 6-5 also show that all 15 duplicate measurements were within the precision QAO of 50 percent RPD. Thus, the VOC measurement precision QAO for the GC/FID analyses, as measured by MS/MSD sample analyses, was met.

Table 6-6 summarizes the VOC spike recoveries from the VOST trap MS/MSD sample analyses by GC/MS. The data in Table 6-6 show that 53 out of 61 measurements, or 87 percent, were within the compound-specific recovery ranges. The completeness QAO for this

TABLE 6-6. VOC RECOVERIES FROM MS SAMPLES ANALYZED BY GC/MS

Primary target analytes	Spike recovery, %							
	Test 0	Test 1	Test 2	Test 3	Test 4	Test 6	Test 6	QAO*
Trichloroethene	97	99	102	50	62	66	73	71-157
1,1,2-trichloroethane	51	68	74	48	63	52	64	52-150
Tetrachloroethene	85	94	91	58	74	51	86	64-148

Secondary analytes	Spike recovery, %			
	MS	MSD	QAO*	RPD, %
Trichlorofluoromethane	82.6	69.4	17-181	17.4
1,1-Dichloroethene	92.2	88.8	59-155	3.8
Methylene chloride	97.6	108.2	D-221	10.3
trans-1,2-Dichloroethene	88.2	85.4	54-156	3.2
1,1-Dichloroethane	75.0	82.6	59-155	9.6
Chloroform	71.8	83.2	51-138	14.7
1,2-Dichloroethane	87.8	80.8	49-155	8.3
1,1,1-Trichloroethane	66.8	71.0	52-162	6.1
Carbon tetrachloride	72.6	76.2	70-140	4.8
Benzene	97.4	76.2	37-151	24.4
Trichloroethene	76.2	70.2	71-157	8.2
1,2-Dichloropropane	80.4	74.2	D-210	8.0
Bromodichloromethane	84.6	76.2	35-155	10.4
cis-1,3-Dichloropropene	78.2	61.2	D-227	24.4
trans-1,3-Dichloropropene	81.2	37.6	17-183	73.4
1,1,2-Trichloroethane	72.6	64.6	52-150	11.7
Dibromobloromethane	78.6	68.8	53-149	13.3
Toluene	87.6	127	47-150	37.0
Tetrachloroethene	71.0	64.8	64-148	9.1
Chlorobenzene	77.2	68.0	37-160	12.7
Precision QAO, RPD				70

*Compound-specific criteria taken from Table 6, Method 8240A; D denotes detected.

measurement was 70 percent; therefore, the accuracy QAO, as measured by spike recovery, was met. The data in Table 6-6 also show that 19 out of 20 RPD measurements, or 95 percent, were within the precision QAO of 70 percent RPD. Therefore, the VOC measurement precision QAO for the GC/MS analyses, as measured by MS/MSD sample analyses, was met.

One sample from each of the fluff feed, soil feed, kiln ash, baghouse ash, and post-test scrubber liquor matrices was analyzed in duplicate as a further check on measurement precision. Neither of the kiln ash, baghouse ash, or scrubber liquor duplicate samples contained any detectable volatile organic target analytes, so no precision information was obtained from these analyses. Table 6-7 summarizes the duplicate sample analysis results for the two test feed matrices. All precision measurements shown met the precision QAO of 50 percent RPD.

Table 6-8 summarizes the method surrogate recoveries achieved in the GC/FID analyses of test samples. Table 6-9 presents an analogous summary for the GC/MS analyses. The data in Table 6-8 show that 95 out of 100, or 95 percent, of the surrogate recoveries were within the surrogate-specific accuracy QAO range. The data in Table 6-9 show that 105 out of 140, or 75 percent, of the surrogate recoveries were within the surrogate-specific accuracy QAO range. Because the completeness QAO was 70 percent for this measurement, the accuracy QAO, as measured by surrogate recovery, was met for both the GC/FID and the GC/MS analyses.

TABLE 6-7. DUPLICATE SAMPLE VOC ANALYSIS RESULTS

Sample	Concentration		RPD, %
	Analysis	Duplicate analysis	
Test Feed, mg/kg			
Composite fluff			
Tetrachloroethene	4.8	<4	18
1,1,2-Trichloroethane	<1	<1	0
Trichloroethene	1.2	<1	18
Test 3 soil			
Tetrachloroethene	50	50	0
1,1,2-Trichloroethane	<1	<1	0
Trichloroethene	2.7	2.7	0
Precision QAO, RPD			50

TABLE 6-8. VOC SURROGATE RECOVERIES IN THE GC/FID ANALYSIS OF TEST SAMPLES

Sample	Surrogate recovery, %		Sample	Surrogate recovery, %	
	1,1,1-Trichloro-ethane	4-Bromo-fluorobenzene		1,1,1-Trichloro-ethane	4-Bromo-fluorobenzene
Test Feed			Kiln Ash		
Test 0	97.5	113	Test 1	87.7	98.4
Composite fluff	93.6	97.2	Test 2	94.4	99.6
Composite fluff duplicate	67.2	115	Test 3	70.2	83.4
Composite fluff MS	81.9	125	Test 3 duplicate	79.3	88.9
Composite fluff MSD	82.1	120	Test 3 MS	76.4	86.7
Test 1	86.4	82.1	Test 3 MSD	73.0	83.4
Test 2	134	141	Test 4	80.2	92.9
Test 5	118	123	Test 5	92.5	73.0
Test 6	140	150	Test 6	90.0	93.6
Test 3	70.3	85.2			
Test 3 duplicate	72.9	85.7			
Test 3 MS	77.7	87.0	Baghouse Ash		
Test 3 MSD	73.5	90.7	Test 0	93.7	105
Test 4	71.0	94.5	Test 1	93.8	104
Scrubber Liquor			Test 2	86.7	106
Test 0 pretest	91.6	105	Test 3	78.1	103
Test 0 post-test	84.6	96.7	Test 3 duplicate	81.2	105
Test 0 post-test duplicate	89.3	101	Test 3 MS	76.4	96.9
Test 0 post-test MS	89.9	103	Test 3 MSD	78.7	102
Test 0 post-test MSD	91.6	106	Test 4	90.4	105
Test 1 pretest	94.1	104	Test 5	92.1	107
Test 1 post-test	92.5	106	Test 6	90.4	107
Test 2 pretest	90.4	103			
Test 2 post-test	92.4	109			
Test 3 pretest	89.1	102			
Test 3 post-test	87.4	100			
Test 4 pretest	83.3	93.0			
Test 4 post-test	87.9	100			
Test 5 pretest	87.7	101			
Test 5 post-test	86.3	97.9			
Test 6 pretest	88.5	103			
Test 6 post-test	88.4	100			
Recovery QAO	52-162	74-121	Recovery QAO	52-162	74-121

TABLE 6-9. VOC SURROGATE RECOVERIES IN THE GC/MS ANALYSIS OF METHOD 0030 SAMPLES

Sample	Surrogate recovery, %			
	1,2-Dichloroethane-d ₄	Toluene-d ₈	Benzene-d ₆	4-Bromofluorobenzene
Test 0				
Set 1	98	90	84	92
Set 2	98	88	89	61
Set 3	99	89	64	61
Field blank	96	98	65	62
MS	101	97	57	45
Test 1				
Set 1	128	84	96	90
Set 2	128	88	96	65
Set 3	115	93	98	85
Field blank	111	104	100	84
MS	119	98	99	76
Test 2				
Set 1	20	80	96	152
Set 2	104	121	107	94
Set 3	100	105	130	93
Field blank	108	98	116	80
MS	104	106	119	109
Test 3				
Set 1	175	90	118	168
Set 2	256	90	127	204
Set 3	134	98	108	86
Field blank		Sample Lost		
MS	126	102	107	75
Test 4				
Set 1	128	97	109	72
Set 2	127	97	109	83
Set 3	131	98	108	56
Field blank	131	101	108	94
MS	128	103	108	82
Test 5				
Set 1	118	88	91	49
Set 2	126	85	94	48
Set 3	108	97	88	53
Field blank	98	97	110	76
MS	111	105	132	68
Test 6				
Set 1	128	98	110	101
Set 2	122	100	106	57
Set 3	123	100	105	62
Field blank	131	100	108	64
MS	121	105	113	94
Trip blank	135	103	105	46
Recovery QAO	70-121	81-117	74-121	74-121

6.2 SVOC ANALYSES

A total of 101 samples was analyzed for SVOCs. Included in this group were 17 method blanks, 1 field blank, 7 MS/MSD sample sets, 3 sets of duplicate test samples, and 6 sets of replicate feed samples. Table 6-10 lists the sample collection date, extraction date, analysis date, and analysis hold time for these samples. As shown in Table 6-10, 89 of 101 samples, or 88 percent were extracted within the specified method extraction hold time. The data also show that all sample extracts were analyzed within the analysis hold time specified by the method.

Table 6-11 summarizes the SVOC measurement QAOs for precision, accuracy, and completeness. Table 6-12 lists the MDL objectives and the levels achieved. As can be seen in Table 6-12, the MDL objective was achieved for all of the analytes except 2-nitrophenol in flue gas, a secondary analyte. SVOC measurement precision and accuracy were assessed by preparing two MS/MSD sample sets for the test feed (one set for each feed type) and kiln ash matrices, and one MS/MSD sample set for the baghouse ash, scrubber liquor, and Method 0010 train matrices. Table 6-13 summarizes the spike recovery data obtained for solid and liquid samples. Table 6-14 presents an analogous summary for Method 0010 train samples.

The data in Table 6-13 show that all achieved spike recoveries were within the compound-specific recovery objective ranges. Thus, the measurement accuracy QAO, as measured by spike recovery from solid and liquid samples, was met. Table 6-13 also shows that all 12 RPD measurements for MS/MSD analyses were within the precision QAO of 50 percent RPD. Thus, the SVOC measurement precision QAO, as measured by solid and liquid MS/MSD sample analyses, was also met.

The data in Table 6-14 show that all 112 spike recovery measurements from Method 0010 train samples were within the compound-specific recovery objective ranges. Thus, the measurement accuracy QAO, as measured by spike recovery, was met. Table 6-14 also shows

TABLE 6-10. SAMPLE HOLD TIMES FOR THE SVOC ANALYSES BY GC/MS

Sample	Collection date	Extraction date	Extraction hold time, days	Analysis date	Analysis hold time, days
Test 1 Feed					
Extract 1	10/14/93	10/26/93	12	11/19/93	24
Extract 2	10/14/93	10/26/93	12	11/19/93	24
Extract 3	10/14/93	10/26/93	12	11/19/93	24
Extract 4	10/14/93	10/26/93	12	11/19/93	24
Extract 5	10/14/93	10/26/93	12	11/19/93	24
MS	10/15/93	11/05/93	21	12/10/93	35
MSD	10/15/93	11/05/93	21	12/10/93	35
Test 2 Feed					
Extract 1	10/15/93	10/27/93	12	11/21/93	25
Extract 2	10/15/93	10/27/93	12	11/21/93	25
Extract 3	10/15/93	10/27/93	12	11/21/93	25
Extract 4	10/15/93	10/27/93	12	11/21/93	26
Extract 5	10/15/93	10/27/93	12	11/21/93	26
Test 3 Feed					
Extract 1	10/20/93	11/04/93	15	12/06/93	32
Extract 2	10/20/93	11/04/93	15	12/06/93	32
Extract 3	10/20/93	11/04/93	15	12/06/93	32
Extract 4	10/20/93	11/04/93	15	12/07/93	33
Extract 5	10/20/93	11/04/93	15	12/07/93	33
MS	10/20/93	11/05/93	16	12/10/93	35
MSD	10/20/93	11/05/93	16	12/10/93	35
Test 4 Feed					
Extract 1	10/20/93	11/03/93	14	12/07/93	34
Extract 2	10/20/93	11/03/93	14	12/07/93	34
Extract 3	10/20/93	11/03/93	14	12/07/93	34
Extract 4	10/20/93	11/03/93	14	12/07/93	34
Extract 5	10/20/93	11/03/93	14	12/07/93	34
Test 5 Feed					
Extract 1	10/18/93	10/28/93	10	11/21/93	24
Extract 2	10/18/93	10/28/93	10	11/21/93	24
Extract 3	10/18/93	10/28/93	10	11/21/93	24
Extract 4	10/18/93	10/28/93	10	11/22/93	25
Extract 5	10/18/93	10/28/93	10	11/22/93	25
Method Requirement			14		40

(continued)

TABLE 6-10. (continued)

Sample	Collection date	Extraction date	Extraction hold time, days	Analysis date	Analysis hold time, days
Test 6 Feed					
Extract 1	10/18/93	10/29/93	11	11/21/93	23
Extract 2	10/18/93	10/29/93	11	11/22/93	24
Extract 3	10/18/93	10/29/93	11	11/22/93	24
Extract 4	10/18/93	10/29/93	11	11/22/93	24
Extract 5	10/18/93	10/29/93	11	11/23/93	25
Feed Packaging Container Material (Test 0)					
Sample 1	12/21/93	01/05/94	15	01/13/94	8
Sample 2	12/31/93	01/05/94	5	01/13/94	8
Feed Method Blanks					
Blank 1	10/21/93	10/22/93	1	11/21/93	30
Blank 2	11/02/93	11/03/93	1	12/09/93	36
Blank 3	11/03/93	11/04/93	1	12/10/93	36
Kiln Ash					
Test 1	11/09/93	11/16/93	7	12/17/93	31
Test 2	11/16/93	11/18/93	2	12/21/93	33
Test 3	12/01/93	12/03/93	2	01/07/94	35
Test 3 duplicate	12/01/93	12/08/93	7	01/10/94	33
Test 3 MS	12/01/93	12/07/93	6	01/07/94	31
Test 3 MSD	12/01/93	12/07/93	6	01/07/94	31
Test 4	12/02/93	12/08/93	6	01/10/94	33
Test 5	11/18/93	11/24/93	6	12/23/93	29
Test 6	11/23/93	12/01/93	8	01/06/94	36
Test 6 MS	11/23/93	12/09/93	15	01/10/94	32
Test 6 MSD	11/23/93	12/09/93	15	01/10/94	32
Baghouse Ash					
Test 0	10/28/93	11/09/93	12	12/16/93	37
Test 1	11/10/93	11/16/93	6	12/17/93	31
Test 2	11/16/93	11/18/93	2	12/21/93	33
Test 3	12/01/93	12/03/93	2	01/07/94	35
Test 3 duplicate	12/01/93	12/08/93	7	01/10/94	33
Test 4	12/02/93	12/08/93	6	01/10/94	33
Test 5	11/18/93	11/24/93	6	12/23/93	29
Test 6	11/23/93	12/01/93	8	01/06/94	36
Test 6 MS	11/23/93	12/07/93	14	01/07/94	31
Test 6 MSD	11/23/93	12/07/93	14	01/07/94	31
Method Requirement			14		40

(continued)

TABLE 6-10. (continued)

Sample	Collection date	Extraction date	Extraction hold time, days	Analysis date	Analysis hold time, days
Ash Method Blanks					
Blank 1	11/08/93	11/09/93	1	12/10/93	31
Blank 2	11/15/93	11/16/93	1	12/21/93	35
Blank 3	11/17/93	11/18/93	1	12/21/93	33
Blank 4	11/23/93	11/24/93	1	12/23/93	29
Blank 5	11/30/93	12/01/93	1	01/06/94	36
Blank 6	12/02/93	12/03/93	1	01/07/94	34
Blank 7	12/07/93	12/08/93	1	01/10/94	32
Blank 8	12/08/93	12/09/93	1	01/10/94	31
Scrubber Liquor					
Test 0 pretest	10/27/93	11/02/93	6	12/09/93	37
Test 0 post-test	10/27/93	11/02/93	6	12/09/93	37
Test 1 pretest	11/09/93	11/16/93	7	12/17/93	31
Test 1 post-test	11/09/93	11/16/93	7	12/17/93	31
Test 2 pretest	11/16/93	11/22/93	6	12/21/93	29
Test 2 post-test	11/16/93	11/22/93	6	12/21/93	29
Test 3 pretest	12/01/93	12/09/93	8	01/11/94	33
Test 3 post-test	12/01/93	12/09/93	8	01/11/94	33
Test 3 post-test duplicate	12/01/93	12/09/93	8	01/11/94	33
Test 4 pretest	12/02/93	12/09/93	7	01/11/94	33
Test 4 post-test	12/02/93	12/09/93	7	01/11/94	33
Test 4 MS	12/02/93	12/10/93	8	01/11/94	32
Test 4 MSD	12/02/93	12/10/93	8	01/11/94	32
Test 5 pretest	11/18/93	11/22/93	4	12/23/93	31
Test 5 post-test	11/18/93	11/22/93	4	12/23/93	31
Test 6 pretest	11/23/93	11/30/93	7	12/23/93	23
Test 6 post-test	11/23/93	11/30/93	7	12/23/93	23
Scrubber Liquor Method Blanks					
Blank 1	11/01/93	11/02/93	1	12/09/93	37
Blank 2	11/15/93	11/16/93	1	12/21/93	35
Blank 3	11/22/93	11/22/93	0	12/23/93	31
Blank 4	11/29/93	11/30/93	1	12/23/93	23
Blank 5	12/09/93	12/10/93	1	01/11/94	31
Method Requirement			14		40

(continued)

TABLE 6-10. (continued)

Sample	Collection date	Extraction date	Extraction hold time, days	Analysis date	Analysis hold time, days
Method 0010 Train					
Test 0	10/27/93	10/28/93	1	11/24/93	27
Test 1	11/09/93	11/10/93	1	11/24/93	14
Test 2	11/16/93	11/17/93	1	12/20/93	33
Test 3	12/01/93	12/02/93	1	12/20/93	35
Test 4	12/02/93	12/03/93	1	01/07/94	35
Test 5	11/18/93	11/19/93	1	12/21/93	32
Test 6	11/23/93	11/24/93	1	12/23/93	29
Method blank	11/02/93	11/03/93	1	12/10/93	37
Field blank	12/01/93	12/02/93	1	01/07/94	35
Blank spike	12/08/93	12/09/93	1	01/10/94	31
Blank spike duplicate	12/08/93	12/09/93	1	01/10/94	31
Method Requirement			14		40

TABLE 6-11. SVOC MEASUREMENT QAOS

Measurement parameter	Measurement/ analytical method	Reference	Conditions	Precision, % RSD or RPD	Accuracy, %	Completeness, %
Semivolatile organic contaminants in feed, residue, and flue gas samples	Extraction, concentration, GC/MS	SW-846 Methods 0010, 3520A, 3540A, and 8270A	Methylene chloride extraction	50	D-262*	70

*Compound-specific criteria taken from Table 6, Method 8270A.

TABLE 6-12. SVOC MEASUREMENT MDLs: OBJECTIVES AND ACHIEVED LEVELS

Compound	MDL objective					
	Solid residues, mg/kg		Aqueous liquids, µg/L		Flue gas, µg/dscm	
	Objective	Achieved	Objective	Achieved	Objective	Achieved
Primary Target Analytes						
Naphthalene	2	0.3	20	3.1	10	0.9
Bis(2-ethylhexyl)phthalate	2	1.3	20	13	10	3.9
Di-n-octylphthalate	2	0.4	20	3.8	10	1.2
Compound	Flue gas, µg/dscm		Compound	Flue gas, µg/dscm		
	Objective	Achieved		Objective	Achieved	
Secondary Analytes^a			Secondary Analytes^a			
Phenol	10	4.5	2,4-Dinitrophenol	10	3.3	
Bis(2-chloroethyl)ether	10	1.3	4-Nitrophenol	10	2.5	
2-Chlorophenol	10	7.1	Dibenzofuran	10	2.1	
1,3-Dichlorobenzene	10	1.5	2,4-Dinitrotoluene	10	3.9	
1,4-Dichlorobenzene	10	1.8	Diethylphthalate	10	1.7	
1,2-Dichlorobenzene	10	1.3	Fluorene	10	1.3	
Bis(2-chloroisopropyl)ether	10	1.0	4-Chlorophenylphenylether	10	1.5	
N-nitroso-di-n-propylamine	10	1.6	4,6-Dinitro-2-methylphenol	10	2.8	
Hexachloroethane	10	1.3	N-nitrosodiphenylamine	10	4.3	
Nitrobenzene	10	2.3	4-Bromophenylphenylether	10	0.7	
Isophorone	10	1.4	Hexachlorobenzene	10	0.6	
2-Nitrophenol	10	13.9	Pentachlorophenol	10	1.7	
2,4-Dimethylphenol	10	1.5	Phenanthrene	10	0.7	
Bis(2-chloroethoxy)methane	10	4.9	Anthracene	10	0.8	
2,4-Dichlorophenol	10	7.3	Di-n-butylphthalate	10	5.3	
1,2,4-Trichlorobenzene	10	1.8	Fluoranthene	10	0.4	
Hexachlorobutadiene	10	0.9	Pyrene	10	1.3	
4-Chloro-3-methylphenol	10	5.8	Butylbenzylphthalate	10	6.7	
2-Methylnaphthalene	10	1.0	Benzo(a)anthracene	10	0.5	
2,4,6-Trichlorophenol	10	7.9	Chrysene	10	0.7	
2,4,5-Trichlorophenol	10	7.3	Benzo(b)fluoranthene	10	0.7	
2-Chloronaphthalene	10	0.7	Benzo(k)fluoranthene	10	0.8	
Dimethylphthalate	10	0.4	Benzo(a)pyrene	10	0.7	
Acenaphthylene	10	2.3	Indeno(1,2,3-cd)pyrene	10	1.2	
2,6-Dinitrotoluene	10	1.3	Dibenz(a,h)anthracene	10	1.3	
Acenaphthene	10	1.8	Benzo(ghi)perylene	10	0.9	

^aMeasurement not performed on solid and liquid matrices.

**TABLE 6-13. SVOC RECOVERIES FROM SOLID AND LIQUID MS SAMPLES
ANALYZED BY GC/MS**

Sample	Spike recovery, %			Precision QAO, RPD
	Naphthalene	BEHP	DNOP	
Test 1 Feed ^a				
MS	88.0	NS ^b	NS	50
MSD	83.5	NS	NS	
RPD, %	5.2	—	—	
Test 3 Feed ^a				
MS	86.9	NS	NS	50
MSD	88.7	NS	NS	
RPD, %	2.1	—	—	
Test 3 Kiln Ash				
MS	88.1	81.8	81.3	50
MSD	93.9	90.3	87.2	
RPD, %	6.4	9.9	7.0	
Test 6 Kiln Ash				
MS	78.8	71.0	79.3	50
MSD	90.3	83.5	91.0	
RPD, %	13.6	16.2	13.7	
Test 6 Baghouse Ash ^c				
MS	91.6	IS ^d	IS	50
MSD	85.3	IS	IS	
RPD, %	7.1	—	—	
Test 4 Scrubber Liquor				
MS	85.7	74.0	80.7	50
MSD	86.2	59.9	72.3	
RPD, %	0.6	21.1	11.0	
Accuracy QAO	21-133	8-158	4-146	

^aThe amount of phthalates in the fluff and soil feed exceeded 1 percent therefore spiking was not required.

^bNS = Not spiked.

^cThe phthalate spiking levels were insignificant compared to the native amount found in the baghouse ash.

^dIS = Insufficient spike.

**TABLE 6-14. SVOC RECOVERIES FROM THE METHOD 0010 MS SAMPLES
ANALYZED BY GC/MS**

Compound	Spike recovery, %			
	MS	MSD	QAO	RPD
Phenol	75.6	82.3	5-112	8.5
Bis(2-chloroethyl)ether	84.6	88.2	12-158	4.2
2-Chlorophenol	83.5	88.2	23-134	5.5
1,3-Dichlorobenzene	84.4	85.3	D-172	1.1
1,4-Dichlorobenzene	87.0	87.8	20-124	0.9
1,2-Dichlorobenzene	83.7	90.4	32-129	7.7
Bis(2-chloroisopropyl)ether	70.2	74.9	36-166	6.5
N-Nitroso-di-n-propylamine	89.2	96.6	D-230	8.0
Hexachloroethane	84.5	82.3	40-113	2.6
Nitrobenzene	89.3	88.9	35-180	0.5
Isophorone	86.8	88.9	21-196	2.4
2-Nitrophenol	88.5	90.0	29-182	1.7
2,4-Dimethylphenol	37.2	52.8	32-119	34.7
Bis(2-chloroethoxy)methane	84.3	87.5	33-184	3.7
2,4-Dichlorophenol	87.3	93.5	39-135	6.9
1,2,4-Trichlorobenzene	95.2	92.1	44-142	3.3
Naphthalene	90.2	87.8	21-133	2.7
Hexachlorobutadiene	96.3	95.1	24-116	1.3
4-Chloro-3-methylphenol	93.2	102	22-147	9.0
2-Methylnaphthalene	88.4	91.6	21-133	3.6
2,4,6-Trichlorophenol	87.2	103	37-144	16.6
2,4,5-Trichlorophenol	95.8	101	37-144	5.3
2-Chloronaphthalene	85.2	93.6	60-118	9.4
Dimethylphthalate	85.7	96.3	D-112	11.7
Acenaphthylene	80.7	88.6	33-145	9.3
2,6-Dinitrotoluene	89.7	101	50-158	11.9
Acenaphthene	89.5	95.1	47-145	6.0
2,4-Dinitrophenol	64.1	87.2	D-191	30.5
4-Nitrophenol	80.9	91.5	D-132	12.3
Dibenzofuran	83.5	94.0	27-133	1.8
2,4-Dinitrotoluene	89.3	103	39-139	14.3
Diethylphthalate	84.5	94.8	D-114	11.5
Fluorene	82.1	91.7	59-121	11.1
4-Chlorophenylphenylether	88.2	98.3	25-158	10.8
4,6-Dinitro-2-methylphenol	69.4	87.5	D-181	23.1
Precision QAO, RPD				50

TABLE 6-14. (continued)

Compound	Spike recovery, %			
	MS	MSD	QAO	RPD
N-Nitrosodiphenylamine	86.0	93.8	D-230	8.7
4-Bromophenylphenylether	97.0	106	53-127	8.9
Hexachlorobenzene	103	109	D-152	5.7
Pentachlorophenol	104	114	14-176	9.2
Phenanthrene	91.2	97.5	54-120	6.7
Anthracene	89.4	96.4	27-133	7.5
Di-n-butylphthalate	89.7	95.4	1-118	6.2
Fluoranthene	92.2	96.7	26-137	4.8
Pyrene	82.2	90.6	52-115	9.7
Butylbenzylphthalate	76.9	82.6	D-152	7.2
Benzo(a)anthracene	86.3	94.9	33-143	9.5
3,3-Dichlorobenzidine	79.6	94.6	D-262	17.2
Chrysene	87.0	96.2	17-168	10.0
Bis(2-ethylhexyl)phthalate	86.8	93.0	8-158	6.9
Di-n-octylphthalate	82.6	91.7	4-146	10.4
Benzo(b)fluoranthene	85.5	94.5	24-159	10.0
Benzo(k)fluoranthene	80.4	87.6	11-162	8.6
Benzo(a)pyrene	79.4	84.8	17-163	6.8
Indeno(1,2,3-cd)pyrene	49.6	83.8	D-171	51.3
Dibenzo(a,h)anthracene	82.2	80.9	D-227	1.6
Benzo(g,h,i)perylene	83.4	82.7	D-219	0.8
Precision QAO, RPD				50

that 55 of 56 RPD determinations from MS/MSD sample analyses, or 98 percent, were within the precision QAO of 50 percent RPD. As the completeness QAO for this measurement was 70 percent, the precision QAO for the Method 0010 train SVOC analyses, as measured by the RPD of MS/MSD sample analyses, was met.

All samples extracted for SVOC analyses were spiked with method surrogates prior to extraction, and surrogate recoveries were measured. Table 6-15 summarizes the surrogate recoveries achieved for solid and liquid samples. Table 6-16 provides an analogous summary for flue gas samples. The data in Table 6-15 show that 154 out of 159 surrogate recovery

TABLE 6-15. SVOC SURROGATE RECOVERIES IN THE GC/MS ANALYSIS OF SOLID AND LIQUID SAMPLES

Sample	Surrogate recovery, %		
	Nitrobenzene-d ₅	2-Fluorobiphenyl	4-Terphenyl-d ₁₄
Feed Packaging Container Material			
Sample 1	79	71	100
Sample 2	73	67	116
Kiln Ash			
Test 1	72	77	82
Test 2	77	83	94
Test 3	85	80	84
Test 3 duplicate	74	76	79
Test 3 MS	83	79	77
Test 3 MSD	90	84	86
Test 4	89	92	92
Test 5	74	75	76
Test 6	81	83	86
Test 6 MS	72	77	75
Test 6 MSD	82	79	84
Baghouse Ash			
Test 0	85	85	95
Test 1	81	82	88
Test 2	80	85	91
Test 3	127	112	120
Test 3 duplicate	79	78	81
Test 4	81	80	81
Test 5	82	87	94
Test 6	89	84	86
Test 6 MS	87	79	93
Test 6 MSD	81	73	77
Ash Method Blanks			
Blank 1	78	82	86
Blank 2	80	85	95
Blank 3	88	91	98
Blank 4	76	78	95
Blank 5	72	73	84
Blank 6	90	85	89
Blank 7	78	83	82
Blank 8	74	67	78
Solid Sample Recovery QAO*	23-120	30-115	18-137

*Compound-specific criteria taken from Table 8 of Method 8270A.

(continued)

TABLE 6-15. (continued)

Sample	Surrogate recovery, %		
	Nitrobenzene-d ₅	2-Fluorobiphenyl	4-Terphenyl-d ₁₄
Scrubber Liquor			
Test 0 pretest	76	63	82
Test 0 post-test	74	69	84
Test 1 pretest	20	19	25
Test 1 post-test	79	89	108
Test 2 pretest	75	74	97
Test 2 post-test	67	69	89
Test 3 pretest	65	71	84
Test 3 post-test	68	68	83
Test 3 post-test duplicate	84	79	86
Test 4 pretest	80	81	82
Test 4 post-test	67	71	83
Test 4 post-test MS	82	83	83
Test 4 post-test MSD	82	82	82
Test 5 pretest	72	72	89
Test 5 post-test	55	66	92
Test 6 pretest	84	73	95
Test 6 post-test	74	63	86
Scrubber Liquor Method Blanks			
Blank 1	79	83	85
Blank 2	77	76	96
Blank 3	79	76	93
Blank 4	84	81	91
Blank 5	82	81	79
Liquid Sample Recovery QAO^a	35-114	43-116	33-141

^aCompound-specific criteria taken from Table 8 of Method 8270A.

TABLE 6-16. SVOC SURROGATE RECOVERIES IN THE GC/MS ANALYSIS OF METHOD 0010 FLUE GAS SAMPLES

Sample	Surrogate recovery, %							
	2-Fluoro-phenol	Phenol-d ₆	Nitro-benzene-d ₅	2-Fluoro-biphenyl	2,4,6-Tribromo-phenol	4-Terphenyl-d ₁₄	Octafluoro-biphenyl	9-Phenyl-anthracene
Test 0	57	43	76	76	80	78	87	86
Test 1	36	25	72	64	61	69	73	84
Test 2	40	35	81	82	78	91	55	106
Test 3	74	75	90	89	85	93	103	115
Test 4	74	75	90	79	78	83	98	110
Test 5	66	57	86	87	83	101	95	102
Test 6	70	64	76	74	84	83	102	96
Method blank	71	73	69	71	92	78	NS ^a	NS
Field blank	69	70	86	82	84	83	NS	NS
Blank spike	84	89	120	106	119	108	NS	NS
Blank spike duplicate	88	95	121	115	125	118	NS	NS
Recovery QAO	21-100	10-94	35-114	43-116	10-123	33-141	30-115 ^b	18-137 ^c

^aNS = Not spiked.

^bRecovery QAO the same as for 2-fluorobiphenyl.

^cRecovery QAO the same as for 4-terphenyl-d₁₄.

measurements, or 97 percent, were within the surrogate-specific recovery objective range. Because the completeness QAO was 70 percent for this measurement, the accuracy QAO, as measured by surrogate recovery, was met for solid and liquid samples.

Table 6-16 summarizes surrogate recoveries achieved for flue gas samples. The data in Table 6-16 show that 76 out of 80 surrogate recovery measurements, or 95 percent, were within the surrogate-specific recovery objective range. Again, because the completeness QAO for this measurement was 70 percent, the accuracy QAO, as measured by surrogate recovery from the flue gas samples, was met.

One kiln ash, one baghouse ash, and one post-test scrubber liquor sample were analyzed in duplicate as a further measure of analysis precision. The target SVOC analytes were not detected in either duplicate kiln ash or baghouse ash sample, so no precision information was obtained via this procedure. Both BEHP and DNOP were found in both duplicate baghouse ash samples. The RPDs from the duplicate analyses were 37 percent for BEHP and 47 percent for DNOP. Both measurements met the precision QAO of 50 percent RPD.

As discussed in Section 4, five replicates of each fluff waste sample were analyzed for BEHP. The percent RSDs of the five analyses ranged from 6 to 25 percent, all within the precision QAO of 50 percent RSD. In addition, five replicates of each soil feed sample were analyzed for BEHP, DNOP, and naphthalene. Naphthalene was not detected in any soil feed replicate analysis at an MDL of 25 mg/kg. The percent RSDs of the replicate sample analyses were 33 and 28 percent, for BEHP, and 49 and 30 percent, for DNOP. All met the precision QAO for the measurement of 50 percent RSD.

6.3 TRACE METAL ANALYSES

A total of 139 samples was analyzed by ICAP using Method 6010A. Included in this number were 3 method blanks, 46 sample duplicates or replicates, and 9 MS/MSD sample sets.

Table 6-17 summarizes the sample collection and analysis dates for these samples. As shown in Table 6-17, all trace metal analyses were completed within the method-required hold time of 180 days.

Table 6-18 summarizes trace metal measurement QAOs for precision, accuracy, and completeness. Table 6-19 shows the MDL objectives and the achieved values for the trace metal analyses. As can be seen in Table 6-19, the MDL objective was met for all metals with the exception of zinc. The zinc MDL was achieved for solid residues but not aqueous liquid or flue gas matrices.

Three method blanks were analyzed for trace metals. Included in this group were one TCLP extraction fluid blank and the front half and back half of a multiple metals train field blank. Analysis results are shown in Table 6-20.

Measurement precision was assessed by performing duplicate or replicate sample analyses. Table 6-21 summarizes the results of these analyses. The data in Table 6-21 show that 103 out of 143 precision calculations performed, or 72 percent, were within the precision QAO of 25 percent RSD or RPD. As the completeness QAO was 70 percent for this measurement, the precision QAO, as measured by duplicate or replicate sample analyses, was met.

Trace metal measurement accuracy was assessed by preparing and analyzing MS/MSD samples. The MS/MSD sample analysis results are given in Table 6-22. The data in Table 6-22 show that only 79 out of 168 measurements, or 47 percent, were within the accuracy QAO range of 70 to 130 percent recovery. Antimony, barium, and silver recoveries were particularly poor. The reason for poor antimony recoveries likely lay with the digestion method used. The method employed, heated HNO_3 /HF for solid samples and heated HNO_3 for aqueous liquid samples, may have caused evaporative loss of the relatively volatile antimony. The poor silver recoveries may have been caused by the presence of chlorides in test program samples. The data in

TABLE 6-17. SAMPLE HOLD TIMES FOR TRACE METAL ANALYSES BY ICAP

Sample	Collection/ preparation date ^a	Analysis date	Analysis hold time, days
Test Feed			
Test 0	01/04/94	01/18/94	14
Test 1 ^b	10/14/93	12/09/93	56
Test 2 ^b	10/15/93	12/09/93	55
Test 5 ^b	10/18/93	12/09/93	52
Test 6 ^b	10/18/93	12/09/93	52
Composite soil ^b (Tests 3 and 4)	10/20/93	12/09/93	50
Kiln Ash			
Test 1 ^b	11/09/93	12/09/93	30
Test 2 ^b	11/16/93	12/14/93	28
Test 3 ^c	12/01/93	01/03/94	33
Test 4 ^c	12/02/93	01/03/94	32
Test 5 ^b	11/18/93	12/14/93	26
Test 6 ^b	11/23/93	12/14/93	21
Baghouse Ash			
Test 0	10/28/93	12/09/93	42
Test 1	11/10/93	12/09/93	29
Test 2 ^d	11/16/93	12/17/93	31
Test 3	12/01/93	12/23/93	22
Test 4	12/02/93	12/23/93	21
Test 5	11/18/93	12/17/93	29
Test 6	11/23/93	12/17/93	24
Scrubber Liquor			
Test 0 pretest	10/27/93	12/09/93	43
Test 0 post-test	10/27/93	12/09/93	43
Test 1 pretest	11/09/93	12/09/93	30
Test 1 post-test ^d	11/09/93	12/09/93	30
Test 2 pretest	11/16/93	12/14/93	28
Test 2 post-test	11/16/93	12/14/93	28
Test 3 pretest	12/01/93	12/23/93	22
Test 3 post-test	12/01/93	12/23/93	22
Test 4 pretest	12/02/93	12/23/93	21
Test 4 post-test	12/02/93	12/23/93	21
Test 5 pretest	11/18/93	12/14/93	28
Test 5 post-test	11/18/93	12/14/93	28
Test 6 pretest	11/23/93	12/14/93	21
Test 6 post-test	11/23/93	12/14/93	21
Method Requirement			180

^aPreparation date corresponds to TCLP leachates.

^bFive split samples were prepared and analyzed.

^cFour split samples were prepared and analyzed.

^dTwo split samples were prepared and analyzed.

(continued)

TABLE 6-17. (continued)

Sample	Collection/ preparation date ^a	Analysis date	Analysis hold time, days
Multiple Metals Train (front half)			
Test 0	10/27/93	12/09/93	43
Test 1	11/09/93	12/09/93	30
Test 2	11/16/93	12/17/93	31
Test 3	12/01/93	12/23/93	22
Test 4	12/02/93	12/23/93	21
Test 5	11/18/93	12/17/93	29
Test 6	11/23/93	12/27/93	24
Multiple Metals Train (back half)			
Test 0	10/27/93	12/09/93	43
Test 1	11/09/93	12/09/93	30
Test 2	11/16/93	12/14/93	28
Test 3	12/01/93	12/23/93	22
Test 4	12/02/93	12/23/93	21
Test 5	11/18/93	12/14/93	26
Test 6	11/23/93	12/17/93	24
TCLP Leachates:			
Test Feed			
Test 1	10/14/93	12/14/93	61
Test 2	10/15/93	12/14/93	60
Test 5	10/18/93	12/14/93	57
Test 6	10/18/93	12/14/93	57
Composite soil (Tests 3 and 4)	10/20/93	12/14/93	55
Kiln Ash			
Test 1	11/09/93	12/09/93	30
Test 2	11/16/93	12/23/93	39
Test 3 ^d	12/01/93	01/18/94	48
Test 4	12/02/93	01/18/94	47
Test 5	11/18/93	12/23/93	35
Test 6	11/23/93	12/23/93	30
Method Requirement			180

^aPreparation date corresponds to TCLP leachates.^dTwo split samples were prepared and analyzed.

(continued)

TABLE 6-17. (continued)

Sample	Collection/ preparation date*	Analysis date	Analysis hold time, days
TCLP Leachates (continued):			
Baghouse Ash			
Test 0	10/28/93	12/09/93	42
Test 1	11/10/93	12/09/93	29
Test 2	11/16/93	12/23/93	37
Test 3	12/01/93	01/18/94	48
Test 4 ^d	12/02/93	01/18/94	47
Test 5	11/18/93	12/23/93	35
Test 6	11/23/93	12/23/93	30
Post-test Scrubber Liquor			
Test 0	10/27/93	12/14/93	48
Test 1	11/09/93	12/14/93	35
Test 2	11/16/93	12/23/93	37
Test 3	12/01/93	01/18/94	48
Test 4	12/02/93	01/18/94	47
Test 5	11/18/93	12/23/93	35
Test 6	11/23/93	12/23/93	30
Blanks			
Multiple metals train field blank front half	12/07/93	01/18/94	42
Multiple metals train field blank back half	12/07/93	01/18/94	42
TCLP extraction fluid	12/30/93	01/18/94	19
Spikes:			
Test Feed			
Test 1 MS	10/14/93	01/26/94	104
Test 1 MSD	10/14/93	01/26/94	104
Composite soil MS	10/20/93	01/18/94	98
Composite soil MSD	10/20/93	01/18/94	98
Kiln Ash			
Test 1 MS	11/09/93	01/18/94	70
Test 1 MSD	11/09/93	01/18/94	70
Baghouse Ash			
Test 1 MS	11/10/93	01/18/93	69
Test 1 MSD	11/10/93	01/18/93	69
Method Requirement			180

*Preparation date corresponds to TCLP leachates.

^dTwo split samples were prepared and analyzed.

(continued)

TABLE 6-17. (continued)

Sample	Collection/ preparation date*	Analysis date	Analysis hold time, days
Spikes (continued):			
Scrubber Liquor			
Test 6 MS	11/23/93	01/18/94	56
Test 6 MSD	11/23/93	01/18/94	56
Multiple Metals Train (front half)			
Blank spike MS	01/11/94	01/18/94	7
Blank spike MSD	01/11/94	01/18/94	7
Multiple Metals Train (back half)			
Blank spike MS	01/11/94	01/18/94	7
Blank spike MSD	01/11/94	01/18/94	7
TCLP Leachates:			
Kiln Ash			
Test 3 MS	12/01/93	01/18/94	48
Test 3 MSD	12/01/93	01/18/94	48
Baghouse Ash			
Test 3 MS	12/01/93	01/18/94	48
Test 3 MSD	12/01/93	01/18/94	48
Method Requirement			180

*Preparation date corresponds to TCLP leachates.

TABLE 6-18. TRACE METAL MEASUREMENT QAOS

Measurement parameter	Method	Reference	Conditions	Precision, % RSD or RPD	Accuracy, %	Completeness, %
Trace metals in solid samples	ICAP	BIF methods, SW-846 Method 6010A	Microwave digestion by BIF methods	25	70-130	70
Trace metals in aqueous liquid samples	ICAP	BIF methods, SW-846 Method 6010A	Conventional digestion by BIF methods	25	70-130	70

TABLE 6-19. TRACE METAL MEASUREMENT MDLs: OBJECTIVES AND ACHIEVED LEVELS

Measurement parameter	MDL objective		
	Solid residues, mg/kg objective / achieved	Aqueous liquids, µg/L objective / achieved	Flue gas, µg/dscm objective / achieved
Antimony	10 / 0.01	100 / 30	50 / 8.5
Arsenic	10 / 0.01	100 / 50	50 / 12.1
Barium	1 / 0.01	10 / 3	5 / 3.6
Cadmium	2 / 0.003	20 / 4	10 / 0.8
Chromium	5 / 0.01	50 / 7	30 / 4.4
Copper	5 / 0.002	50 / 23	30 / 4.5
Lead	10 / 0.005	100 / 77	50 / 15.4
Nickel	5 / 0.0049	50 / 10	30 / 3.3
Silver	5 / 0.0004	50 / 7	30 / 1.4
Zinc	1 / 0.26	10 / 25	5 / 12.6

TABLE 6-20. TRACE METAL ANALYSES OF METHOD BLANK SAMPLES

Sample	Concentration									
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn
TCLP extraction fluid, mg/L	<0.03	<0.05	0.56	<0.004	<0.007	0.11	0.87	0.045	<0.007	98
Multiple metals train field blank										
Front half, mg	<0.01	<0.01	0.010	<0.0003	0.010	0.0023	<0.005	0.0049	<0.0004	0.026
Back half, mg/L	<0.03	<0.05	0.0030	<0.004	<0.007	0.023	0.077	<0.01	<0.007	0.025

TABLE 6-21. REPLICATE TRACE METAL SAMPLE ANALYSIS RESULTS

Sample	Concentration										Precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Test Feed:											
Test 1											
Sample	62	48	43	2.0	27	8,800	1,300	5.2	<0.7	95	
Replicate 1	200	<20	87	2.2	32	9,000	2,400	4.5	1.2	120	
Replicate 2	110	<20	120	<0.5	31	13,000	1,600	<3.0	<0.7	100	
Replicate 3	94	<20	75	0.9	40	8,400	2,800	5.1	1.1	100	
Replicate 4	110	<20	47	1.2	40	5,000	3,800	<3.0	<0.7	96	
RSD, %	45	— ^a	42	53	17	32	42	26	28	10	25
Test 2											
Sample	290	<20	76	1.3	37	8,200	3,000	<3.0	1.3	110	
Replicate 1	190	<20	44	1.4	36	7,100	1,400	3.7	<0.7	130	
Replicate 2	130	<20	72	1.5	45	8,900	5,100	<3.0	2.6	310	
Replicate 3	140	<20	70	2.2	34	8,000	1,300	4.3	1.0	240	
Replicate 4	130	<20	90	1.1	38	9,800	1,200	<3.0	<0.7	120	
RSD, %	39	0	24	28	11	12	70	17	63	49	25
Test 5											
Sample	98	<20	52	1.5	33	7,600	1,200	<3	<0.7	150	
Replicate 1	80	<20	50	1.3	23	28,000	1,100	<3	1.2	140	
Replicate 2	91	<20	48	1.4	32	9,800	1,100	<3	<0.7	150	
Replicate 3	120	<20	37	<0.5	21	7,600	980	<3	1.4	110	
Replicate 4	63	<20	48	1.8	27	9,000	1,000	<3	2.1	140	
RSD, %	23	0	12	37	20	71	8	0	48	12	25

^a — = RSD, RPD not calculated.

(continued)

TABLE 6-21. (continued)

Sample	Concentration										Precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Test Feed (continued):											
Test 6											
Sample	66	<20	110	<0.5	21	8,300	980	<3.0	3.6	130	
Replicate 1	86	<20	42	<0.5	23	7,900	800	9.0	<0.7	120	
Replicate 2	75	<20	56	1.5	23	7,700	900	<3.0	<0.7	150	
Replicate 3	92	<20	100	1.1	27	9,500	970	<3.0	<0.7	120	
Replicate 4	200	<20	98	1.3	26	15,000	840	5.1	<0.7	89	
RSD, %	53	0	37	47	10	32	9	57	— ^a	18	25
Composite Soil (Tests 3 and 4)											
Sample	75	<20	66	1.5	70	12,000	4,200	19	1.4	170	
Replicate 1	66	<20	69	1.2	73	14,000	2,300	27	1.8	210	
Replicate 2	87	<20	76	1.4	130	26,000	3,800	46	2.3	210	
Replicate 3	52	<20	80	1.6	83	11,000	2,500	28	1.6	220	
Replicate 4	48	<20	67	0.9	68	7,400	2,900	24	<0.7	130	
RSD, %	25	0	9	21	31	50	26	36	38	20	25
Kiln Ash:											
Test 1											
Sample	1,100	42	280	<0.5	360	130,000	3,000	230	1.9	180	
Replicate 1	1,100	<20	260	<0.5	390	200,000	2,600	280	1.3	180	
Replicate 2	1,200	<20	270	<0.5	400	240,000	3,100	250	1.6	200	
Replicate 3	1,100	<20	230	<0.5	350	210,000	3,100	220	1.7	190	
Replicate 4	1,200	<20	290	<0.5	440	150,000	3,000	410	2.0	190	
RSD, %	5	—	9	0	9	24	7	28	16	4	25

^a— = RSD, RPD not calculated.

(continued)

TABLE 6-21. (continued)

Sample	Concentration										Precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Kiln Ash (continued):											
Test 2											
Sample	880	<20	240	0.6	370	83,000	3,600	320	3.0	220	
Replicate 1	870	<20	170	<0.5	430	190,000	3,300	750	4.8	270	
Replicate 2	1,000	<20	210	<0.5	600	150,000	3,800	1,300	3.9	290	
Replicate 3	930	63	220	0.6	410	99,000	3,600	450	3.5	270	
Replicate 4	1,000	33	240	<0.5	600	190,000	4,500	1,400	7.0	260	
RSD, %	7	60	13	10	23	35	12	58	35	10	25
Test 3											
Sample	180	<20	120	<0.5	67	46,000	4,000	65	2.1	320	
Replicate 1	200	<20	99	<0.5	66	69,000	4,300	59	1.5	350	
Replicate 2	180	<20	150	<0.5	79	32,000	4,400	66	1.7	340	
Replicate 3	190	<20	140	<0.5	70	67,000	3,800	70	1.3	310	
RSD, %	5	0	18	0	8	33	7	7	21	6	25
Test 4											
Sample	190	<20	120	<0.5	41	18,000	4,000	42	1.7	290	
Replicate 1	190	<20	98	<0.5	60	21,000	4,000	60	1.8	300	
Replicate 2	190	<20	72	<0.5	56	67,000	3,700	56	2.3	300	
Replicate 3	200	<20	95	<0.5	50	50,000	4,000	48	2.2	300	
RSD, %	3	0	20	0	16	60	4	16	15	2	25

(continued)

TABLE 6-21. (continued)

Sample	Concentration										Precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Kiln Ash (continued):											
Test 5											
Sample	1,000	<20	270	<0.5	470	110,000	5,500	160	5.0	250	
Replicate 1	980	<20	290	0.8	480	110,000	5,800	140	2.4	250	
Replicate 2	790	<20	140	<0.5	500	85,000	6,000	140	2.6	180	
Replicate 3	1,000	<20	240	<0.5	440	160,000	5,600	160	2.4	260	
Replicate 4	1,000	<20	240	0.6	420	110,000	5,600	140	1.0	260	
RSD, %	10	0	24	22	7	24	4	7	54	14	25
Test 6											
Sample	1,100	<20	270	1.0	550	170,000	5,700	350	2.3	280	
Replicate 1	820	<20	210	<0.5	520	180,000	5,600	330	3.6	260	
Replicate 2	940	<20	270	<0.5	520	170,000	7,100	360	2.3	240	
Replicate 3	890	120	220	<0.5	570	190,000	5,500	640	5.6	230	
Replicate 4	1,000	48	270	<0.5	510	170,000	5,800	310	2.7	250	
RSD %	13	95	12	— ^a	5	5	11	34	48	8	25
Baghouse Ash											
Test 2 sample	400	<20	22	18	320	14,000	19,000	130	<0.7	2,100	
Duplicate	340	20	20	17	310	14,000	44,000	110	1.0	2,100	
RPD, %	16	—	10	6	3	0	79	17	—	0	25
Post-test Scrubber Liquor											
Test 1 sample	0.47	0.12	2.4	0.20	1.9	210	780	1.2	0.072	19	
Duplicate	0.65	<0.05	5.3	0.25	2.9	230	900	1.7	0.094	23	
RPD, %	32	—	75	22	42	9	14	35	25	19	25

^a— = RSD, RPD not calculated.

(continued)

TABLE 6-21. (continued)

Sample	Concentration										Precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
TCLP Leachate											
Test 3 kiln ash sample	<0.2	<0.2	0.26	<0.005	0.046	0.28	<0.1	<0.01	<0.007	1.2	
Duplicate	<0.2	<0.2	0.26	<0.005	0.044	0.22	<0.1	<0.01	<0.007	1.3	
RPD, %	0	0	0	0	4	24	0	0	0	8	25
Test 4 sample	0.80	0.26	0.22	1.7	0.27	640	5,700	3.2	0.027	97	
Duplicate	0.84	0.21	0.22	1.8	0.30	650	5,800	3.3	0.028	100	
RPD, %	5	21	0	6	11	2	2	3	4	3	25

TABLE 6-22. TRACE METAL RECOVERIES FROM MS SAMPLES ANALYZED BY ICAP

Sample	% recovery										Accuracy/precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Test 1 Fluff Feed											
MS	17	65	63	195	92	NS ^a	30	9	9	108	70-130
MSD	0	68	27	81	85	NS	57	8	2	94	70-130
RPD, %	200	5	80	83	8	— ^b	62	12	127	14	25
Test 3 and 4 Soil Feed											
MS	40	66	910	76	100	NS	87	61	5	63	70-130
MSD	103	38	4	61	68	NS	63	63	35	50	70-130
RPD, %	88	54	198	22	38	—	32	3	150	23	25
Test 1 Kiln Ash											
MS	62	56	0	75	64	NS	19	84	13	95	70-130
MSD	16	72	0	90	65	NS	4	80	0	70	70-130
RPD, %	118	25	0	18	2	—	130	5	200	30	25
Test 1 Baghouse Ash											
MS	52	<13	17	60	50	NS	NS	44	<12	53	70-130
MSD	95	66	37	64	44	NS	NS	42	<11	44	70-130
RPD, %	59	>134	74	6	13	—	—	5	0	19	25
Test 6 Scrubber Liquor											
MS	0	79	46	67	82	112	99	75	46	71	70-130
MSD	0	79	54	61	71	112	63	69	89	234	70-130
RPD, %	0	0	16	9	14	0	44	8	64	107	25

^aNS = Not spiked.

^b— = RPD not calculated.

(continued)

TABLE 6-22. (continued)

Sample	% recovery										Accuracy/precision QAO
	Sb	As	Ba	Cd	Cr	Cu	Pb	Ni	Ag	Zn	
Multiple Metals Train Blank Spike											
Back half											
MS	117	85	88	84	91	102	109	80	72	131	70-130
MSD	100	90	85	84	91	102	101	79	46	131	70-130
RPD, %	15	6	3	0	0	0	8	1	44	0	25
Front half											
MS	<10	68	17	97	12	108	85	100	90	95	70-130
MSD	<10	71	0	77	8	85	70	36	<10	47	70-130
RPD, %	0	4	200	23	40	24	19	94	>160	68	25
Test 3 Kiln Ash TCLP Leachate											
MS	<10	80	73	85	75	119	120	69	45	IS ^c	70-130
MSD	<10	85	77	85	73	94	109	66	43	IS	70-130
RPD, %	0	6	5	0	3	23	10	4	5	— ^b	25
Test 3 Baghouse Ash TCLP Leachate											
MS	0	58	42	83	39	105	73	86	178	111	70-130
MSD	0	78	83	78	44	109	62	84	228	115	70-130
RPD, %	0	29	66	6	12	4	16	2	25	4	25

^b— = RPD not calculated.^cIS = Spiked amount not significant compared to native sample amount.

Section 4.1 show that both test feed material matrices contained significant amounts of chlorine. Thus, test program samples used to prepare the MS/MSD samples may have contained chlorine in the form of chlorides. The presence of chlorides will definitely interfere with the sample digestion method employed. The reason for the poor barium recoveries is not clear.

If antimony, barium, and silver are removed from consideration, then 67 out of 114 other metal spike recovery measurements, or 59 percent, fell within the accuracy QAO range of 70 to 130 percent recovery. This still fails the completeness objective of 70 percent. However, had the accuracy QAO range instead been a slightly relaxed 60 to 140 percent recovery, then 89 of 114 other metal (antimony, barium, and silver excluded) recovery measurements, or 78 percent, would have been acceptable, a 70 percent completeness objective at this relaxed accuracy QAO range would have been met.

Trace measurement precision was also measured by calculating the RPD of each pair of MS/MSD measurements. The data in Table 6-22 show that 55 out of 84 RPDs, or 65 percent, met the precision QAO of 25 percent RPD. However, again, the precision measures for antimony, barium, and silver were particularly poor. If these three metals are excluded, then 43 of the remaining 57 RPD determinations, or 75 percent, met the precision QAO of 25 percent RPD. This would have satisfied the completeness objective of 70 percent.

Based on both the precision and accuracy checks employing MS/MSD sample analyses, test program results for antimony, barium, and silver appear to have been compromised, and the reported data should be treated with caution. Test program conclusions regarding the concentrations of these three metals in incinerator discharges, and their distributions among these discharges, must be viewed as tentative at best.

6.4 CHLORIDE ANALYSES

A total of 17 samples was analyzed for chloride ion to support flue gas HCl concentration measurements. Included in this number were 1 method blank and 1 MS/MSD sample set. Table 6-23 lists the sample collection and analysis dates and the analysis hold times for these samples. As the data in Table 6-23 show, all 17 samples were analyzed within the method required hold time limit of 28 days.

Table 6-24 summarizes the flue gas chloride measurement precision, accuracy, and completeness QAOs. The MDL objective for the measurement was 100 $\mu\text{g}/\text{dscm}$. This objective was met with an achieved MDL of 49 $\mu\text{g}/\text{dscm}$.

Table 6-25 shows that the chloride recoveries for the MS/MSD sample set were both within the accuracy QAO range of 75 to 125 percent recovery. Table 6-25 also shows that the RPD of the MS/MSD sample set was within the precision QAO of 30 percent RPD. Thus, both the accuracy and precision QAOs were met as measured by the MS/MSD sample analyses.

Table 6-26 summarizes the chloride analysis results for the duplicate test samples analyzed. As can be seen from the data in Table 6-26, 9 of the 11 RPDs, or 82 percent, met the precision QAO of 30 percent RPD. As the completeness objective for the measurement was 70 percent, the precision QAO, as measured by duplicate sample analyses, was met.

6.5 PCDD/PCDF ANALYSES

A total of 35 samples was analyzed for PCDDs and PCDFs by GC/MS using Method 8290 or Method 23. Included in this number were one Method 23 sorbent resin method blank, one pretest scrubber liquor blank, and three duplicates of test samples. Table 6-27 lists the sample collection, extraction, and analysis dates for these samples, and the corresponding analysis hold times. As shown in Table 6-27, all but five samples were extracted within the specified method limit. Three of the five samples not extracted within the method hold time were sample

TABLE 6-23. SAMPLE HOLD TIMES FOR CHLORIDE ANALYSES BY ION CHROMATOGRAPHY

Sample*	Collection/preparation date	Analysis date	Analysis hold time, days
Stack Exit Flue Gas			
Test 0	10/27/93	11/22/93	26
Test 1	11/09/93	11/29/93	20
Test 1 MS	11/09/93	11/29/93	20
Test 1 MSD	11/09/93	11/29/93	20
Test 2	11/16/93	11/29/93	7
Test 3	12/01/93	12/06/93	5
Test 4	12/02/93	12/06/93	4
Test 5	11/18/93	12/06/93	18
Test 6	11/23/93	11/29/93	6
Method blank	12/07/93	12/10/93	3
Baghouse Exit Flue Gas			
Test 0	10/27/93	11/22/93	26
Test 1	11/09/93	11/29/93	20
Test 2	11/16/93	11/29/93	7
Test 3	12/01/93	12/06/93	5
Test 4	12/02/93	12/06/93	4
Test 5	11/18/93	11/29/93	11
Test 6	11/23/93	11/29/93	6
Method Requirement			28

*All samples were analyzed in duplicate.

TABLE 6-24. FLUE GAS CHLORIDE MEASUREMENT QAOS

Measurement parameter	Method	Reference	Precision, % RSD or RPD	Accuracy, %	Completeness, %
HCl in flue gas	Ion chromatography	40 CFR 60, Appendix A, Method 5; BIF methods; Method 9057	30	75-125	70

TABLE 6-25. CHLORIDE RECOVERIES FROM MS SAMPLES ANALYZED BY ION CHROMATOGRAPHY

Sample	Chloride recovery, %	Precision QAO
Stack Exit Flue Gas		
Test 1 MS	105	
Test 1 MSD	109	
RPD, %	3.7	30
Recovery QAO	75-125	

TABLE 6-26. DUPLICATE SAMPLE CHLORIDE ANALYSIS RESULTS

Sample	Chloride concentration, mg/L		RPD, %
	Analyses	Duplicate analyses	
Stack Exit Flue Gas			
Test 1	0.40	<0.29	>31.9
Test 2	0.88	0.78	12.1
Test 3	0.52	0.64	20.7
Test 4	<0.29	0.60	>69.7
Test 5	<0.29	<0.29	0
Test 6	<0.29	<0.29	0
Baghouse Exit Flue Gas			
Test 1	19.0	18.8	1.1
Test 2	12.3	12.0	2.5
Test 3	28.3	28.4	0.4
Test 4	19.6	20.0	2.0
Test 6	42.0	42.7	1.7
QAO			30

TABLE 6-27. SAMPLE HOLD TIMES FOR THE PCDD/PCDF ANALYSES BY GC/MS

Sample	Collection date	Extraction date	Extraction hold time, days	Analysis date	Analysis hold time, days
Test Feed					
Packaging container material	12/21/93	01/12/94	22	01/20/94	8
Composite fluff feed	10/20/93	11/08/93	13	11/16/93	8
Composite soil feed	10/20/93	12/02/93	43	12/05/93	3
Kiln Ash					
Test 1	11/09/93	11/19/93	10	11/22/93	3
Test 1 duplicate	11/09/93	01/21/93	73	01/24/93	3
Test 2	11/16/93	12/02/93	16	12/05/93	3
Test 3	12/01/93	12/10/93	9	12/17/93	7
Test 4	12/02/93	12/10/93	8	12/17/93	7
Test 5	11/18/93	12/02/93	14	12/05/93	3
Test 6	11/23/93	12/07/93	14	12/17/93	10
Baghouse Ash					
Test 0	10/28/93	11/08/93	11	11/16/93	8
Test 1	11/10/93	12/02/93	22	12/05/93	3
Test 1 duplicate	11/10/93	01/12/94	63	01/20/94	8
Test 2	11/16/93	12/02/93	16	12/05/93	3
Test 3	12/01/93	12/10/93	9	12/18/93	8
Test 4	12/02/93	12/10/93	8	12/18/93	8
Test 5	11/18/93	12/02/93	14	12/05/93	3
Test 6	11/23/93	12/07/93	14	12/17/93	10
Post-test Scrubber Liquor					
Test 0	10/27/93	11/10/93	14	11/15/93	5
Test 1	11/09/93	12/10/93	31	12/14/93	4
Test 1 duplicate	11/09/93	01/14/94	66	01/21/94	7
Test 2	11/16/93	12/03/93	17	12/07/93	4
Test 3	12/01/93	12/16/93	15	12/19/93	3
Test 4	12/02/93	12/16/93	14	12/19/93	3
Test 5	11/18/93	12/10/93	22	12/15/93	5
Test 6	12/23/93	12/06/93	13	12/15/93	9
Pretest Scrubber Liquor					
Test 1	10/09/93	12/03/93	14	12/07/93	4
Baghouse Exit Flue Gas Method 23 Train					
Test 0	10/27/93	11/10/93	14	11/15/93	5
Test 1	11/09/93	11/19/93	10	11/24/93	5
Test 2	11/16/93	12/02/93	16	12/06/93	4
Test 3	12/01/93	12/09/93	8	12/13/93	4
Test 4	12/02/93	12/09/93	7	12/13/93	4
Test 5	11/18/93	12/02/93	14	12/06/93	4
Test 6	11/23/93	12/02/93	9	12/06/93	4
Method blank	12/07/93	12/09/93	2	12/13/93	4
Method Requirement			30		45

duplicates for analysis; one of the five missed extraction hold time by 1 day.

Table 6-28 summarizes the PCDD/PCDF measurement QAOs for precision, accuracy, and completeness. Measurement precision was assessed by analyzing split samples in duplicate. Table 6-29 summarizes the results of these analyses and shows that 57 out of 75 RPD measurements, or 76 percent, were within the precision QAO of 50 percent RPD. As the completeness objective for the measurement was 70 percent, the precision QAO, as measured by duplicate sample analyses, was met.

PCDD/PCDF measurement accuracy was assessed adding the method-specified internal standards and surrogates to all test samples and measuring their recovery. Table 6-30 shows the internal standards recoveries achieved from the test samples. As can be seen in Table 6-30, 290 out of 315 individual internal standard recovery measurements, or 92 percent, were within the compound-specific recovery ranges. Since the completeness objective for this measurement was 70 percent, the accuracy QAO, as measured by internal standards recovery, was met.

Table 6-31 lists the surrogate recoveries achieved from test programs samples. As shown, 149 out of 210 individual surrogate recovery measurements, or 71 percent, were within the method specified recovery ranges. Again, with a completeness objective of 70 percent, the accuracy QAO, as assessed by surrogate recoveries, was met.

Table 6-32 shows the MDL objectives and the achieved values for the PCDD/PCDF measurements. As indicated, all MDL objectives were achieved.

TABLE 6-28. PCDD/PCDF MEASUREMENT QAOs

Measurement parameter	Measurement/analytical method	Reference	Conditions	Precision, %	Accuracy, %	Completeness, %
PCDDs/PCDFs in feed, residual, and flue gas sampling trains	Extraction, concentration, GC/MS	40 CFR 266, Appendix IX, Method 23; SW-846 Method 8290	Matrix-specific extraction	50	25-130	70

TABLE 6-29. DUPLICATE SAMPLE PCDD/PCDF ANALYSIS RESULTS

Compound	Test 1 kiln ash, ng/kg analysis / duplicate analysis	RPD, %	Test 1 baghouse ash, ng/kg analysis / duplicate analysis	RPD, %	Test 1 post-test scrubber liquor, pg/L analysis / duplicate analysis	RPD, %
2,3,7,8-TCDD	12.0 / 1.8	148	0.3 / 1.0	108	2.8 / 2.0	33
1,2,3,7,8-PeCDD	31.1 / 6.5	131	0.47 / 0.30	44	4.3 / 3.8	12
1,2,3,4,7,8-HxCDD	50.8 / 57.5	12	0.40 / 0.46	14	4.4 / 3.8	15
1,2,3,6,7,8-HxCDD	71.1 / 67.7	5	0.69 / 0.74	7	3.4 / 3.0	13
1,2,3,7,8,9-HxCDD	212 / 286	30	1.5 / 1.4	7	3.8 / 3.3	14
1,2,3,4,6,7,8-HpCDD	1,810 / 2,340	26	21.3 / 18.1	16	12.4 / 13.1	5
OCDD	11,590 / 24,580	72	179 / 110	48	51.2 / 77.5	41
2,3,7,8-TCDF	496 / 281	55	3.6 / 3.1	15	4.3 / 4.4	2
1,2,3,7,8-PeCDF	155 / 49.1	104	1.5 / 1.2	22	3.1 / 2.6	18
2,3,4,7,8-PeCDF	273 / 215	24	3.2 / 3.7	14	3.0 / 2.5	18
1,2,3,4,7,8-HxCDF	2,770 / 3,160	13	10.7 / 9.4	13	7.8 / 8.3	6
1,2,3,6,7,8-HxCDF	614 / 676	10	3.9 / 3.3	17	3.3 / 3.0	10
2,3,4,6,7,8-HxCDF	2,130 / 2,560	18	9.1 / 9.7	6	11.8 / 13.1	10
1,2,3,7,8,9-HxCDF	25.2 / 35.8	35	0.3 / 1.1	114	3.0 / 2.8	7
1,2,3,4,6,7,8-HpCDF	6,180 / 15,220	84	39.8 / 25.9	42	31.8 / 40.0	23
1,2,3,4,7,8,9-HpCDF	1,360 / 2,070	41	8.1 / 6.3	25	17.1 / 16.7	2
OCDF	12,160 / 44,810	115	123 / 60.3	68	185 / 219	17
Total TCDD	232 / 2.4	196	0.9 / 0.88	2	2.8 / 2.0	33
Total PeCDD	305 / 76.6	120	1.5 / 0.42	113	4.3 / 3.8	12
Total HxCDD	914 / 905	1	6.4 / 6.8	6	3.5 / 3.3	6
Total HpCDD	3,830 / 4,660	20	38.6 / 32.6	17	12.4 / 18.8	41
Total TCDF	3,330 / 1,430	80	15.4 / 3.1	133	4.3 / 4.4	2
Total PeCDF	5,730 / 4,630	21	26.8 / 10.1	91	5.9 / 8.9	41
Total HxCDF	10,780 / 11,190	4	40.2 / 41.5	3	27.6 / 9.8	95
Total HpCDF	15,810 / 28,870	58	83.3 / 57.3	37	81.8 / 84.1	3
Precision QAO		50		50		50

TABLE 6-30. INTERNAL STANDARD RECOVERIES IN THE PCDD/PCDF ANALYSES

Sample	% recovery								
	¹³ C ₁₂ - 2,3,7,8- TCDF	¹³ C ₁₂ - 2,3,7,8- TCDD	¹³ C ₁₂ - 1,2,3,7,8- PeCDF	¹³ C ₁₂ - 1,2,3,7,8- PeCDD	¹³ C ₁₂ - 1,2,3,6,7,8- HxCDF	¹³ C ₁₂ - 1,2,3,6,7,8- HxCDD	¹³ C ₁₂ - 1,2,3,4,6,7,8- HpCDF	¹³ C ₁₂ - 1,2,3,4,6,7,8- HpCDD	¹³ C ₁₂ -OCDD
Test Feed									
Packaging container material	54.3	53.6	58.2	59.3	59.0	61.1	47.2	44.0	31.1
Composite fluff feed	72.5	79.7	82.2	92.5	76.4	83.3	77.7	94.9	95.2
Composite soil feed	85.5	97.0	94.4	107	111	111	117	142	120
Kiln Ash									
Test 1	70.4	75.8	83.2	114	96.1	109	107	135	138
Test 1 duplicate	64.2	62.3	69.4	80.5	71.0	79.6	82.2	92.2	81.5
Test 2	56.4	55.5	65.9	76.0	74.5	79.0	86.1	98.2	105
Test 3	49.8	48.4	47.7	54.4	67.4	73.0	61.2	67.6	53.1
Test 4	41.0	42.0	48.9	60.7	58.4	70.0	62.2	73.7	62.3
Test 5	90.9	81.2	99.4	90.7	113	95.7	164	130	185
Test 6	74.3	84.9	77.3	77.2	92.8	99.6	392	145	397
Baghouse Ash									
Test 0	60.0	63.0	69.6	88.3	76.2	88.8	70.9	91.7	85.2
Test 1	78.6	76.5	86.6	103	93.8	108	110	115	114
Test 1 duplicate	5.7	6.5	17.5	33.9	43.7	54.8	57.5	68.2	77.2
Test 2	71.8	71.5	80.9	97.4	91.8	101	95.4	108	100
Test 3	53.5	52.0	58.0	68.0	74.4	77.5	69.6	77.2	60.1
Test 4	42.1	45.1	46.6	63.8	64.4	81.6	61.2	76.8	62.6
Test 5	68.9	68.1	75.7	93.0	87.8	96.8	95.2	107	111
Test 6	64.4	63.0	64.4	67.6	57.6	85.0	66.0	85.3	163
Pretest Scrubber Liquor									
Test 1	59.1	57.2	63.3	66.7	79.7	81.9	76.7	83.1	66.9
Recovery QAO	40-130	40-130	40-130	40-130	40-130	40-130	25-130	25-130	25-130

(continued)

TABLE 6-30. (continued)

Sample	% recovery								
	¹³ C ₁₂ - 2,3,7,8- TCDF	¹³ C ₁₂ - 2,3,7,8- TCDD	¹³ C ₁₂ - 1,2,3,7,8- PeCDF	¹³ C ₁₂ - 1,2,3,7,8- PeCDD	¹³ C ₁₂ - 1,2,3,6,7,8- HxCDF	¹³ C ₁₂ - 1,2,3,6,7,8- HxCDD	¹³ C ₁₂ - 1,2,3,4,6,7,8- HpCDF	¹³ C ₁₂ - 1,2,3,4,6,7,8- HpCDD	¹³ C ₁₂ -OCDD
Post-test Scrubber Liquor									
Test 0	52.0	59.6	58.5	55.0	73.6	75.6	62.8	63.2	41.3
Test 1	52.2	48.3	51.9	57.4	58.3	62.6	53.1	57.4	50.4
Test 1 duplicate	36.1	30.1	28.4	28.9	30.9	33.4	29.1	36.7	26.3
Test 2	41.0	39.0	42.4	47.5	53.0	55.7	45.8	50.8	39.7
Test 3	56.0	59.9	65.9	55.5	90.1	79.2	76.1	78.2	66.3
Test 4	47.8	48.2	51.5	37.5	74.5	62.1	53.4	59.9	54.2
Test 5	20.0	19.3	21.7	24.0	30.6	31.5	28.2	30.5	26.2
Test 6	34.3	25.6	25.2	25.3	39.5	39.2	33.2	37.2	30.6
Baghouse Exit Method 23 Train									
Test 0	83.0	94.1	87.2	96.6	108	112	89.1	90.7	71.8
Test 1	56.9	57.2	61.3	75.7	72.4	77.9	69.7	81.3	68.2
Test 2	65.0	59.3	62.4	65.0	85.3	82.4	79.9	83.7	74.5
Test 3	61.8	56.5	60.9	64.7	72.4	74.3	66.0	69.9	58.2
Test 4	63.9	59.6	71.0	87.9	84.4	88.0	83.1	89.0	78.5
Test 5	42.7	38.7	38.9	44.5	57.4	56.7	51.3	56.1	50.8
Test 6	69.4	60.1	60.1	59.6	81.7	83.8	76.8	81.7	70.4
Method blank	69.6	65.3	71.6	77.0	89.3	89.9	88.0	97.3	92.4
Recovery QAO	40-130	40-130	40-130	40-130	40-130	40-130	25-130	25-130	25-130

TABLE 6-31. SURROGATE RECOVERIES IN THE PCDD/PCDF ANALYSES

Sample	% recovery					
	³⁷ Cl ₄ -2,3,7,8-TCDD	¹³ C ₁₂ -2,3,4,7,8-PeCDF	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	¹³ C ₁₂ -1,2,3,7,8,9-HxCDF
Test Feed						
Packaging container material	49.5	57.3	63.9	65.1	47.3	50.5
Composite fluff feed	78.6	81.2	81.7	84.1	89.4	79.1
Composite soil feed	74.6	74.5	92.5	91.7	110	89.3
Kiln Ash						
Test 1	81.3	86.5	92.9	89.5	107	89.0
Test 1 duplicate	60.5	72.3	71.5	81.3	88.7	74.6
Test 2	61.0	77.5	87.7	83.7	107	85.4
Test 3	52.8	54.3	78.9	77.1	67.2	72.3
Test 4	46.5	58.2	69.1	75.3	78.8	69.5
Test 5	73.2	86.3	114	81.6	118	98.7
Test 6	87.7	81.5	144	79.7	122	85.5
Baghouse Ash						
Test 0	66.0	80.2	87.1	90.8	90.4	85.6
Test 1	63.8	79.1	79.5	82.1	93.5	82.6
Test 1 duplicate	6.6	27.3	49.4	57.5	70.4	57.7
Test 2	58.5	75.5	79.6	80.2	95.6	84.4
Test 3	53.9	59.9	75.6	77.7	75.6	70.3
Test 4	48.5	63.7	82.3	85.7	83.8	79.5
Test 5	56.7	70.5	70.9	75.1	90.4	76.4
Test 6	65.7	66.7	69.5	77.4	74.4	65.6
Pretest Scrubber Liquor						
Test 1	63.6	75.4	81.2	85.4	95.0	85.1
Recovery QAO	70-130	70-130	70-130	70-130	70-130	70-130

(continued)

TABLE 6-31. (continued)

Sample	% recovery					
	³⁷ Cl ₄ -2,3,7,8- TCDD	¹³ C ₁₂ -2,3,4,7,8- PeCDF	¹³ C ₁₂ -1,2,3,4,7,8- HxCDF	¹³ C ₁₂ -1,2,3,4,7,8- HxCDD	¹³ C ₁₂ -1,2,3,4,7,8,9- HpCDF	¹³ C ₁₂ -1,2,3,7,8,9- HxCDF
Post-test Scrubber Liquor						
Test 0	71.1	72.6	82.7	86.1	75.0	75.4
Test 1	58.8	68.3	62.6	74.4	80.0	75.9
Test 1 duplicate	56.2	60.7	69.6	88.4	86.6	71.8
Test 2	68.3	71.1	73.0	77.0	83.3	81.5
Test 3	59.7	73.2	80.6	90.8	93.8	91.3
Test 4	58.7	67.1	69.0	73.9	81.0	80.8
Test 5	49.0	55.1	72.3	76.8	73.5	71.1
Test 6	45.7	52.6	62.0	66.4	81.1	68.8
Baghouse Exit Method 23 Train						
Test 0	102	112	97.0	107	97.1	79.6
Test 1	66.4	79.1	82.9	88.2	96.1	74.0
Test 2	122	128	110	121	137	84.4
Test 3	119	130	109	110	139	64.1
Test 4	114	129	108	108	131	71.8
Test 5	124	133	103	107	138	54.6
Test 6	115	124	104	103	129	72.3
Method blank	113	133	106	117	138	75.6
Recovery QAO	70-130	70-130	70-130	70-130	70-130	70-130

TABLE 6-32. PCDD/PCDF MEASUREMENT MDLS: OBJECTIVES AND ACHIEVED LEVELS

Measurement parameter	Solid residues, ng/kg objective / achieved	Aqueous liquids, pg/L objective / achieved	Flue gas, ng/dscm objective / achieved
2,3,7,8-TCDD	20 / 0.6	200 / 1.3	0.2 / 0.0009
1,2,3,7,8-PeCDD	20 / 0.9	200 / 2.2	0.2 / 0.0012
1,2,3,4,7,8-HxCDD	20 / 1.0	200 / 2.3	0.2 / 0.0015
1,2,3,6,7,8-HxCDD	20 / 0.8	200 / 1.8	0.2 / 0.0012
1,2,3,7,8,9-HxCDD	20 / 0.9	200 / 2.0	0.2 / 0.0015
1,2,3,4,6,7,8-HpCDD	20 / 1.0	200 / 2.2	0.2 / 0.0015
OCDD	20 / 1.1	200 / 4.7	0.2 / 0.0061
2,3,7,8-TCDF	20 / 0.6	200 / 1.0	0.2 / 0.0009
1,2,3,7,8-PeCDF	20 / 0.7	200 / 1.5	0.2 / 0.0009
2,3,4,7,8-PeCDF	20 / 0.7	200 / 1.5	0.2 / 0.0009
1,2,3,4,7,8-HxCDF	20 / 0.7	200 / 1.6	0.2 / 0.0012
1,2,3,6,7,8-HxCDF	20 / 0.5	200 / 1.2	0.2 / 0.0009
2,3,4,6,7,8-HxCDF	20 / 0.7	200 / 2.7	0.2 / 0.0025
1,2,3,7,8,9-HxCDF	20 / 0.8	200 / 1.7	0.2 / 0.0012
1,2,3,4,6,7,8-HpCDF	20 / 0.6	200 / 1.4	0.2 / 0.0009
1,2,3,4,7,8,9-HpCDF	20 / 1.0	200 / 2.3	0.2 / 0.0015
OCDF	20 / 0.9	200 / 2.6	0.2 / 0.0018
Total TCDD	30 / 0.6	300 / 1.3	0.5 / 0.0009
Total PeCDD	30 / 0.9	300 / 2.2	0.5 / 0.0012
Total HxCDD	30 / 0.9	300 / 2.1	0.5 / 0.0015
Total HpCDD	30 / 1.0	300 / 2.2	0.5 / 0.0015
Total TCDF	30 / 0.6	300 / 1.0	0.5 / 0.0009
Total PeCDF	30 / 0.7	300 / 1.5	0.5 / 0.0009
Total HxCDF	30 / 0.7	300 / 2.6	0.5 / 0.0021
Total HpCDF	30 / 0.8	300 / 1.8	0.5 / 0.0012

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