

*Environmental Monitoring for
the DOE Coolside and LIMB
Demonstration Extension Projects*

*Final Report for the Period
of February, March and April 1991*

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ENVIRONMENTAL MONITORING FOR THE
DOE COOLSIDE AND LIMB
DEMONSTRATION EXTENSION PROJECTS

Quarrelly
FINAL REPORT FOR THE PERIOD OF
FEBRUARY, MARCH AND APRIL 1991

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February 1992

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1.0 INTRODUCTION

The purpose of this document is to present environmental monitoring data collected during the U.S. Department of Energy Limestone Injection Multistage Burner (DOE LIMB) Demonstration Project Extension at the Ohio Edison Edgewater Generating Station in Lorain, Ohio. These data were collected by implementing the Environmental Monitoring Plan (EMP) for the DOE LIMB Demonstration Project Extension, dated August 1988. This document is the sixth EMP status report to be published and presents the data generated during February, March and April 1991. These reports review a three to four month period and have been published since the project's start in October 1989.

The DOE project is an extension of the U. S. Environmental Protection Agency's (EPA) original LIMB Demonstration. The program is operated under DOE's Clean Coal Technology Program of "emerging clean coal technologies" under the categories of "in boiler control of oxides of sulfur and nitrogen" as well as "post-combustion clean-up." The objective of the LIMB program is to demonstrate the sulfur dioxide (SO₂) and nitrogen oxide (NO_x) emission reduction capabilities of the LIMB system. The LIMB system is a retrofit technology to be used for existing coal-fired boilers equipped with electrostatic precipitators (ESPs).

As required in the Cooperative Agreement between DOE and Babcock and Wilcox (B&W), an Environmental Information Volume (EIV), an Environmental Monitoring Plan Outline (EMPO), and an Environmental Monitoring Plan (EMP) were prepared prior to the onset of the DOE project. The EIV was dated May 20, 1987, the EMPO was dated December 23, 1987, and the final EMP was dated August 11, 1988.

This report is organized as follows: Section 1.0 is the Introduction; Section 2.0 presents a Summary of the project for the stated reporting period; Section 3.0 discusses the LIMB Process and the Project Status; Section 4.0 presents Source Monitoring

Information; Section 5.0 presents Ambient Monitoring Information; Section 6.0 presents the Health and Safety related information; Section 7.0 discusses the Compliance Monitoring Status; Section 8.0 discusses Quality Assurance/Quality Control Results; and Section 9.0 presents Monitoring Problems and Recommendations for Change. Support material related to air emissions, solid waste disposal, and wastewater discharges is presented in the appendices.

2.0 SUMMARY

This section presents a summary of EMP related items which occurred during the February, March and April 1991 reporting period.

The LIMB system was operated this reporting period to evaluate the flue gas desulfurization efficiency of two calcium-based sorbents (dolomitic lime and lignosulfonated lime) during the combustion of two different sulfur content coals (nominal 1.6 and nominal 3.8 percent sulfur by weight). Three sorbent/coal combinations were evaluated during the following three injection periods:

- dolomitic lime/nominal 3.8 percent sulfur coal, 02/04 - 02/20/91;
- lignosulfonated lime/nominal 1.6 percent sulfur coal, 02/21 - 04/05/91; and
- lignosulfonated lime/nominal 3.8 percent sulfur coal, 04/06 - 04/30/91.

The monitoring data and air quality modeling data presented in this report are based on emission data that are specific to the coal/sorbent combination utilized during a specific injection period and the combination of combustion and air pollution control equipment used at the Lorain facility. To determine LIMB operating efficiencies and environmental impacts, monitoring and modeling data collected during the sorbent/coal injection periods were compared to Baseline data. Baseline was the period from February 17 to April 22, 1990, where nominal 1.6 percent sulfur coal or "compliance coal" was fired and no LIMB Extension equipment was in operation.

The Baseline data were collected after circular burners had been replaced with low NO_x burners. An indication of NO_x emissions prior to the demonstration can be found in the paper entitled "Operation of the LIMB/Humidifier Demonstration at Edgewater," presented at the First Combined FGD and Dry SO₂ Control Symposium, October 25, 1988, St. Louis, MO. Some of the data presented in that paper were collected when the circular burners were still in operation.

Since the goal of the LIMB Demonstration Program is to test a wide range of operating conditions, the SO₂ and NO_x emissions averages should not be taken as representative of long-term, optimized operations. Ranges of SO₂ and NO_x data were compiled during periods of formal testing and may include both injection and non-injection periods within a given day.

During this reporting period, the average SO₂ mass emission rate was highest during the dolomitic lime/nominal 3.8 percent sulfur coal and the lignosulfonated lime/nominal 3.8 percent sulfur coal injection periods, and was lowest during the lignosulfonated lime/nominal 1.6 percent sulfur coal injection period. The SO₂ mass emission rate during this reporting period varied from 880 to 3,400 lb/hr. The average SO₂ mass emission rate for each injection period, which ranged from 1,500 to 2,200 lb/hr, was higher than average SO₂ mass emission rate during the Baseline period of 932 lb/hr. The median SO₂ removal efficiency was the highest during the lignosulfonated lime/nominal 3.8 percent sulfur coal injection period and the lowest during the dolomitic lime/nominal 3.8 percent sulfur coal and the lignosulfonated lime/nominal 1.6 percent sulfur coal injection periods. SO₂ removal efficiencies for the reporting period varied widely, from 9 to 73 percent.

The average NO_x mass emission rates for the three sorbent/coal combinations ranged from 160 to 430 lb/hr this reporting period. The average NO_x mass emission rates for each injection period, which ranged from 250 to 260 lb/hr, were greater than the Baseline average NO_x emissions of 181 lb/hr. However, with the installation of B&W XCL low-NO_x burners, NO_x emissions during this reporting period have decreased when compared with emissions which occurred prior to the EPA LIMB Demonstration. Modeled ambient air impacts from SO₂ and NO_x emissions during these three injection periods will be presented in the next report. Additional information on gaseous emission monitoring is presented in Section 4.1.

The wastewater discharge at Outfall 601 was monitored during this reporting period. All discharge parameters were within National Pollution Discharge Elimination System (NPDES) permit requirements. The change in concentration from the Baseline to each sorbent/coal combination for the NPDES discharge parameters is summarized in Table 2-1. No total phosphorus (P) analyses were performed during this reporting period. Additional information on wastewater monitoring is presented in Section 4.2.

Fly ash samples were composited from October 8 to November 27, 1990 and from November 28 to December 20, 1990. The ash compositing periods correspond to the time periods when the primary sorbent coal combinations were dolomitic lime/nominal 3.8 percent sulfur coal and dolomitic lime/nominal 1.6 percent sulfur coal. The resulting samples were submitted for corrosivity and permeability tests, and were leached using the Toxicity Characteristic Leaching Procedure (TCLP) and a deionized water (DI) leaching procedure--American Society for Testing and Materials (ASTM) D3987. These two leaching procedures will allow for the analyses of the targeted parameters listed in the EMP. The results from each of these analyses is summarized in Table 2-2. The corrosivity of the dolomitic lime/nominal 3.8 percent sulfur coal and the dolomitic lime/nominal 1.6 percent sulfur coal samples were below the detection limit. In addition, the fly ash samples from both periods were less permeable than the Baseline period fly ash. The metal concentrations of the TCLP and DI leachates from both fly ash samples were below the TCLP maximum contaminant levels (MCL's) and below the detection limit for silver (Ag), cadmium (Cd), and mercury (Hg). Additional information on solid waste monitoring is presented in Section 4.3.

Air quality modeling was performed in this reporting period using the four sorbent/coal combinations of the previous reporting period. These combinations were dolomitic lime/nominal 1.6 percent sulfur coal, dolomitic lime/nominal 3.0 percent sulfur coal, dolomitic lime/nominal 3.8 percent sulfur coal, and limestone/nominal 1.6 percent sulfur coal. The modeling demonstrated that SO₂ and NO_x concentrations increased over Baseline period concentrations. With the exception of three injection periods, changes in

**TABLE 2-1. COMPARISON OF NPDES OUTFALL 601 MONITORING DATA
DURING BASELINE AND EXTENSION PERIODS OF OPERATION**

Parameter (Units)	SORBENT/COAL COMBINATION		
	Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal	Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal	Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal
Average As ($\mu\text{g/L}$)	-	-	NA
Average Ca (mg/L)	+	+	+
Average TSS (mg/L)	-	-	-
Average O&G (mg/L)	+	-	NA
Max pH (s.u.)	+	+	+
Min pH (s.u.)	+	+	+

+ = increase from Baseline concentration.

- = decrease from Baseline concentration.

NA = no analysis during test period.

**TABLE 2-2. COMPARISON OF ASH LEACHATE COMPOSITION DATA DURING
BASELINE AND EXTENSION PERIODS OF OPERATION**

Parameter	SORBENT/COAL COMBINATION			
	Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal		Dolomitic Lime/Nominal 1.6 Percent Sulfur Coal	
	DI Leachate (mg/L)	TCLP Leachate (mg/L)	DI Leachate (mg/L)	TCLP Leachate (mg/L)
Total Phenolics	-	NA	-	NA
pH	+	NA	+	NA
Calcium	+	NA	+	NA
Chloride	+	NA	+	NA
Fluoride	+	NA	-	NA
Potassium	+	NA	+	NA
Sodium	-	NA	-	NA
Nitrate	-	NA	+	NA
Sulfate	-	NA	-	NA
Total Hardness	NA	NA	NA	NA
Total Alkalinity as CaCO ₃	+	NA	+	NA
Acidity	NA	NA	NA	NA
Bicarbonate	NC	NA	NC	NA
Carbonate	+	NA	+	NA
Chemical Oxygen Demand (COD)	+	NA	+	NA
Total Organic Carbon (TOC)	-	NA	-	NA
Total Dissolved Solids (TDS)	+	NA	+	NA
Iron	NA	NA	NA	NA
Copper	NA	NA	NA	NA
Magnesium	NA	NA	NA	NA
Silver	a	b	a	b
Arsenic	+	+	-	-
Barium	+	+	+	+
Cadmium	-	b	-	b
Chromium	+	+	+	+
Mercury	NC	NC	NC	NC
Lead	+	b	+	a
Selenium	+	+	+	-

+ = Increase from Baseline concentration.

- = Decrease from Baseline concentration.

NC = No change from Baseline concentration.

NA = No analysis during test period.

a = Both Baseline and test period concentrations are below detection limits. No comparison between Baseline and test periods concentrations is possible, for test period detection limit is higher than the Baseline detection limit.

b = Test period concentration is below detection limit. No comparison between Baseline and test periods is possible, for detection limit of the test period is greater than the Baseline concentration.

model predicted concentrations were below the ambient air significance levels, as defined in the Prevention of Significant Deterioration (PSD) air regulations (40 CFR 51.165(b)(2)). During the screening or initial modeling, the 3-hour ($25 \mu\text{g}/\text{m}^3$), 24-hour ($5 \mu\text{g}/\text{m}^3$) and annual ($1.0 \mu\text{g}/\text{m}^3$) significance levels for SO_2 were exceeded during the dolomitic lime/nominal 1.6 percent sulfur coal (11/1 - 11/12/90), dolomitic lime/nominal 3.0 percent sulfur coal, dolomitic lime/nominal 3.8 percent sulfur coal injection periods. Additional modeling, using the Industrial Source Complex Short Term (ISCST) dispersion model, was conducted to determine compliance with the National Ambient Air Quality Standard (NAAQS) for SO_2 during the November and December 1990 and January 1991 reporting period. The modeling results demonstrate that the SO_2 NAAQS were not exceeded during any of the sorbent/coal injection periods. Additional information on dispersion modeling is presented in Section 5.1. Air dispersion modeling will be conducted in the next reporting period, using emission data from the dolomitic lime and lignosulfonated lime sorbent injection periods in this reporting period.

The employee health and safety monitoring specified in the EMP was completed in the November and December 1990 and January 1991 reporting period. No additional employee health and safety monitoring was conducted during this reporting period.

The facility's compliance monitoring status was reviewed for this period. No air or NPDES permit values, as monitored by Ohio Edison and Radian, were exceeded during this reporting period. Additional information on compliance monitoring is presented in Sections 7.0 and 9.0.

3.0 PROJECT STATUS

The section presents information on the background of the LIMB project as well as the current project status.

3.1 Overview

The DOE LIMB Demonstration Project Extension is a continuation of a LIMB technology demonstration sponsored by the EPA. The purpose of the LIMB system is to reduce SO₂ and NO_x emissions from existing utility power generation plants using cost effective retrofit technologies. Specific goals of the EPA program were to demonstrate 50 to 60 percent reduction of SO₂ emissions based on incoming coals containing a nominal 3 percent sulfur. NO_x emissions were expected to be less than 0.5 lb/million Btu heat input. LIMB has the potential to reduce SO₂ emissions at a much lower cost than flue gas desulfurization (FGD) systems, or switching to low-sulfur coals imported from other regions.

3.2 Edgewater Facility Description

The LIMB Technology Demonstration is taking place at the Ohio Edison Edgewater Steam Electric Generating Plant located on Lake Erie in Lorain, Ohio. The Edgewater facility has a total net demonstrated power capability of 214 MW and consists of three pulverized coal-fired boilers serving two turbines and two oil-fired combustion turbine generators. The LIMB system was installed in 1986 on Edgewater Unit No. 4, which has a nameplate capacity of 105 MW. The boiler associated with Unit No. 4 turbine generator is Boiler No. 13. This unit is a B&W, front wall-fired boiler capable of burning 42.5 tons per hour (tph) of coal. Particulate emissions from Unit No. 4 are controlled with a Lodge-Cottrell ESP, which was retrofitted to the system in 1982.

Prior to the LIMB Demonstration, the Edgewater facility burned eastern bituminous coal (nominal 1.6 percent sulfur). Total station coal consumption is approximately 70 tph with all units in operation. The coal is delivered by truck. The facility utilizes 110 million gallons per day (MGD) of once-through cooling water taken from Lake Erie, and discharges 1.1 MGD of wastewater to the lake from the fly ash settling ponds. During LIMB Extension activities, fly ash from Unit No. 4 is trucked to a municipal landfill located in the Dover Township. Figure 3-1 presents a simplified schematic of the Edgewater facility layout.

Additional information on the Edgewater facility can be found in the EIV and previously prepared reports for this project.

3.3 The LIMB Process

The LIMB process utilizes low- NO_x burners to control the formation of NO_x emissions. To accomplish this reduction, Unit No. 4's original circular register burners were replaced with B&W XCL low- NO_x burners. The burner replacement was completed in 1986 during the EPA Demonstration, and these burners remain in use.

Sorbent is injected into the combustion gas stream to provide sites for SO_2 sorption with downstream particulate collection by the ESP. Two injection systems are currently in place at the Edgewater facility. The first system injects sorbent directly into the boiler. EPA LIMB Demonstration tests were completed using this configuration, with a flue gas humidifying chamber installed in a bypass duct downstream of the boiler. The objective was to increase particulate removal efficiency of the ESP. By decreasing the flue gas temperature, the residence time of the flue gas in the ESP was increased, thereby allowing more time for particulate removal. Also, the resistivity of the fly ash was decreased, which allowed for greater particle-ESP plate attraction and hence, removal. The humidifying chamber was constructed in a bypass duct so that it could be isolated during system upsets and not reduce the generation capability of the unit.

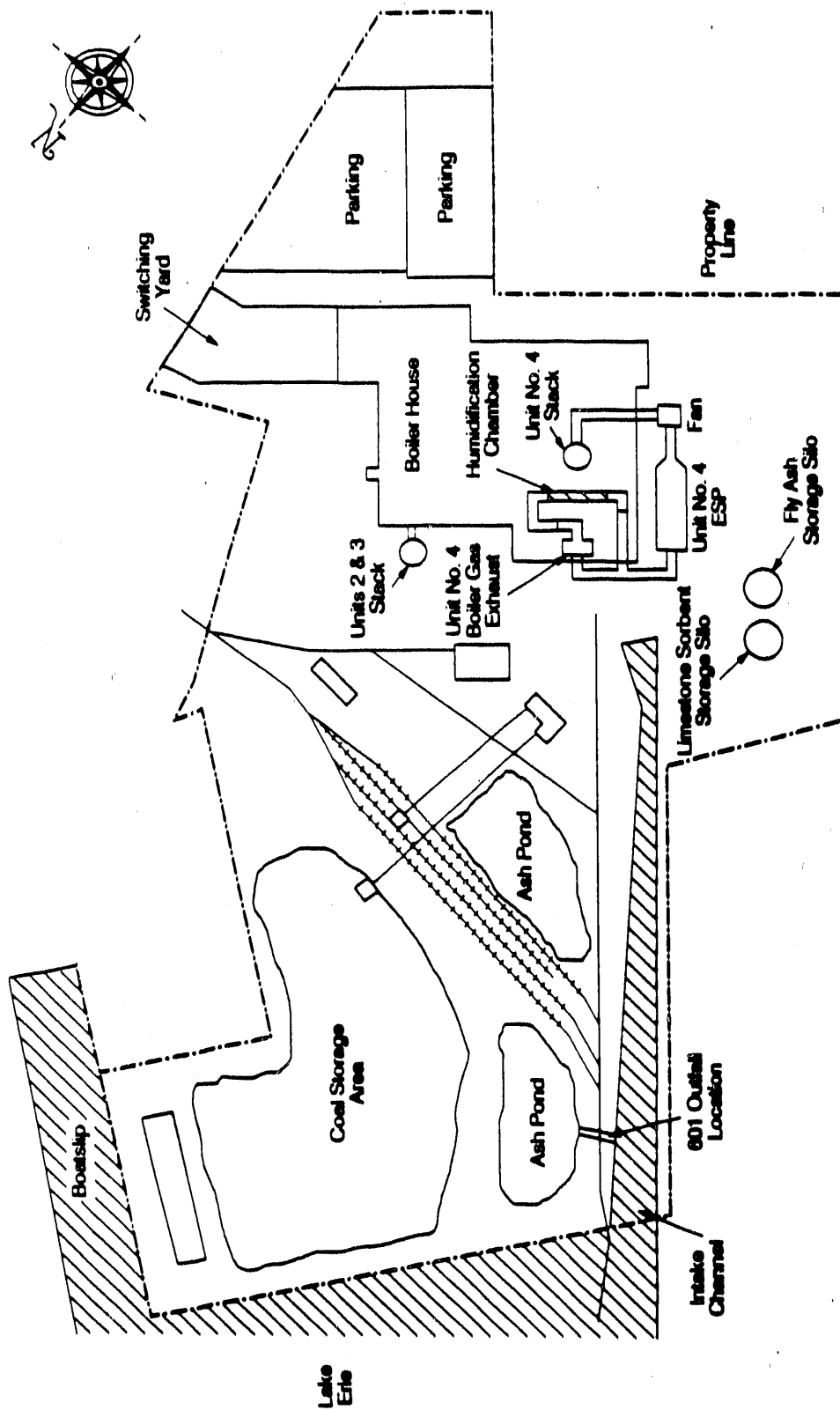


Figure 3-1. Edgewater Plant Site Layout

The DOE LIMB Demonstration Project Extension adds a sorbent injection point located upstream of the flue gas humidification to the original boiler. The Demonstration system, known as the "Coolside" process, involved sorbent injection upstream of the humidification chamber and was used in the initial stages of the LIMB Extension project. In addition to solid sorbent injection, a sodium hydroxide solution was added to the humidifying water to enhance SO₂ removal. Figure 3-2 presents a generalized schematic of the current sorbent injection configurations present at the Edgewater Facility.

In order to accurately document and analyze SO₂ and NO_x reduction efficiencies, as well as boiler operational efficiencies, a variety of parameters are monitored. Boiler operation measurements such as fluid temperatures, pressures, and flow rates are continuously monitored, as are stack gas concentrations of SO₂, NO_x, oxygen (O₂), carbon dioxide (CO₂), carbon monoxide (CO), and opacity. The boiler parameters are monitored by B&W using a computer-based data acquisition system (DAS) known as the Boiler Performance Diagnostic System 140. Up to 1000 data points are scanned and recorded on magnetic media every 60 seconds. System 140 also performs several hundred data calculations using the input measurements. All flue gas component concentrations -- SO₂, NO_x, CO, O₂, and CO₂ -- are monitored at the ESP outlet. Radian also monitors and records component concentrations with a separate personal computer (PC)-based DAS. Data from both DAS's are used in determining stack gas emission rates.

3.4 Project Design and Background

The EPA LIMB Demonstration was initiated in September 1984. B&W, as the prime contractor, has subcontracted with Radian Corporation to perform environmental monitoring throughout the Demonstration. The following testing phases were conducted during the EPA Demonstration:

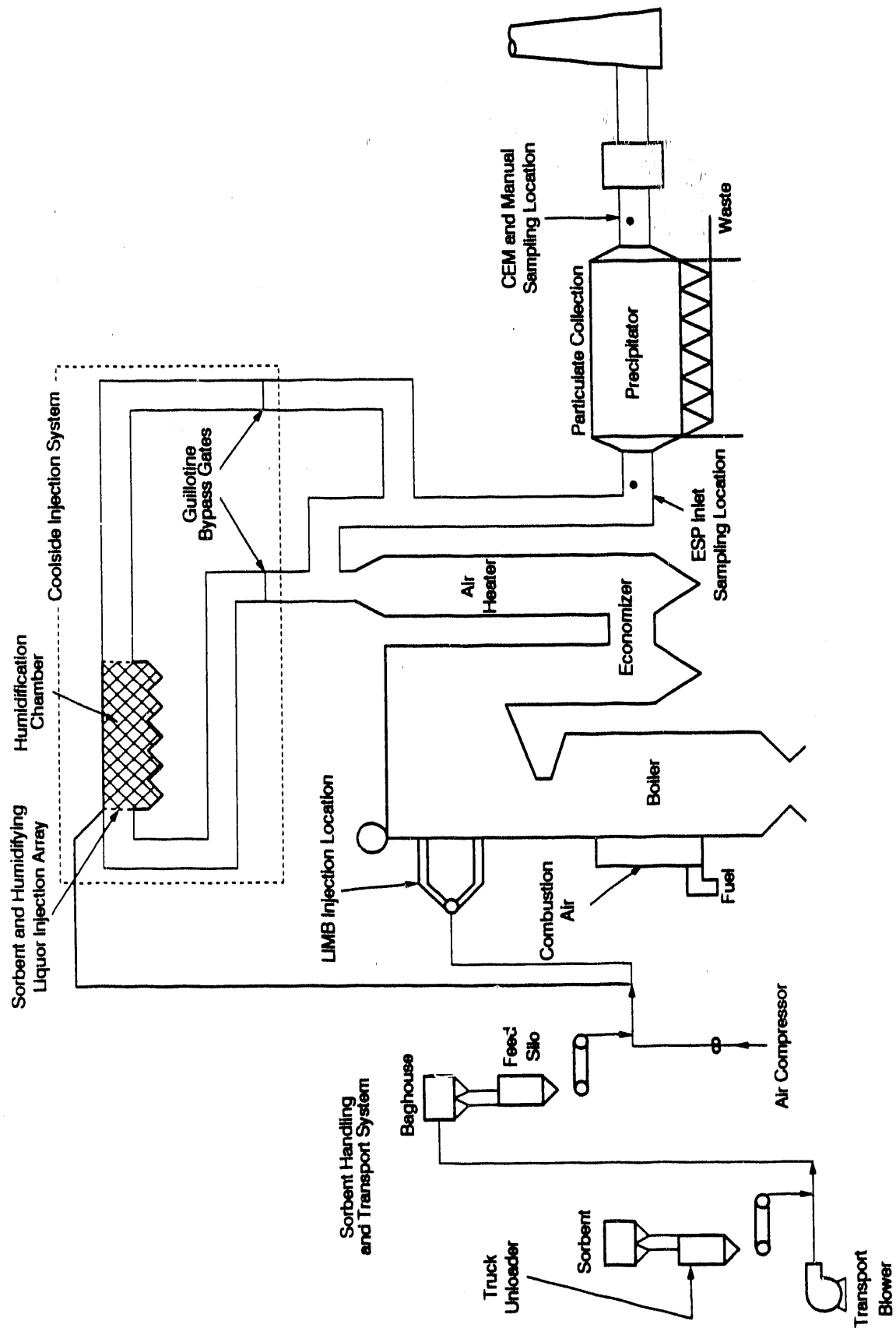


Figure 3-2. LIMB Injection and Coolside Injection Locations and Flue Gas Sampling Locations for the DOE LIMB Extension Project

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Baseline Tests - Conducted prior to any modifications to Unit No. 4. The term "Baseline" in this report also refers to a period when sorbent was not injected into Boiler No. 13 or downstream ductwork.

Low NO_x Burner Tests - Conducted after installation of the low-NO_x burners.

LIMB Optimization and Demonstration - Conducted after installation of the boiler sorbent injection system. Final testing of this phase was completed with the bypass humidification chamber in place.

Preparation for the DOE sponsored LIMB Demonstration with the Coolside testing configuration started in July 1989. A shakedown period was conducted for several months to determine optimum operating conditions. The DOE Coolside tests started in October 1989 and were completed on February 16, 1990. During the period from February 17 to April 22, 1990, a non-LIMB operation or Baseline period was in progress. Following the Baseline period, the DOE LIMB Demonstration Project Extension commenced. The Extension involves sorbent injection into the boiler in conjunction with the humidification chamber operation to maintain ESP performance. When load conditions permit, tests are run close to saturation.

3.5 Project Status

Coolside process tests ended on February 16, 1990. During the weeks following, the system was reconfigured to accommodate boiler injection. The Baseline period occurred from February 17 to April 22, 1990. An equipment/operational shakedown period then followed, during which lignosulfonated lime was used while nominal 3.0 percent sulfur coal was burned. This condition was chosen in order to establish that the system would perform as it had prior to the Coolside test period.

The LIMB Extension system was started on April 23, 1990. System performance was monitored in the April to July 1990 reporting period for the following sorbent/coal combinations: (1) lignosulfonated lime/nominal 3.0 percent sulfur coal;

(2) limestone/nominal 3.0 percent sulfur coal; and (3) limestone/nominal 1.6 percent sulfur coal. The system was then shut down on July 21, 1990 for a scheduled maintenance outage.

The LIMB Extension system was again started on August 14, 1990. When low sulfur coal was burned, a number of short sorbent/coal injection test periods (each less than 8 hours per day) followed, a format which has continued to date. When high sulfur coal was burned, the injection system was run full-time and data were collected continuously. The coal and sorbent combinations tested during the August to October 1990 reporting period were: (1) dolomitic lime/nominal 1.6 percent sulfur coal; and (2) dolomitic lime/nominal 3.0 percent sulfur coal. The coal and sorbent combinations tested during the November and December 1990, and January 1991 reporting period were: (1) dolomitic lime/nominal 1.6 sulfur coal; (2) dolomitic lime 3.0 percent sulfur coal; (3) dolomitic lime/nominal 3.8 percent sulfur coal; and (4) limestone/nominal 1.6 percent sulfur coal.

The LIMB system was operated this reporting period to evaluate the flue gas desulfurization efficiency of two calcium-based sorbents (dolomitic lime and lignosulfonated lime) during the combustion of two different sulfur content coals (nominal 1.6 and 3.8 percent sulfur by weight). The following three sorbent/coal combinations were evaluated during three injection periods:

- dolomitic lime/nominal 3.8 percent sulfur coal, February 4 - 20, 1991;
- lignosulfonated lime/nominal 1.6 percent sulfur coal, February 21 - April 5, 1991; and
- lignosulfonated lime/nominal 3.8 percent sulfur coal, April 6 - 30, 1991.

4.0 SOURCE MONITORING

The Edgewater facility has several environmental discharge streams that are affected by the DOE LIMB program. This section divides the discharge source monitoring reporting into three areas. Unit No. 4 gaseous emissions are covered in Section 4.1, wastewater discharges are covered in Section 4.2, and solid waste discharges are covered in Section 4.3. Monitoring of pollution control limits and equipment is discussed in Section 4.4.

4.1 Gaseous Emissions Monitoring

There are two stacks at the Edgewater facility. Exhaust gases from Unit No. 4 are emitted through a stack located on the roof of the Unit No. 4 boiler house. Unit No. 3 flue gases are emitted through a brick chimney located adjacent to the northern side of the boiler house. As a part of the DOE LIMB Extension test matrix, Unit No. 4 flue gas concentrations of NO_x , SO_2 , CO , CO_2 , and O_2 as well as opacity measurements are continuously monitored. No manual stack gas tests were conducted for total particulate matter (PM), total particulate matter below 10 microns (PM_{10}) and particle size distribution tests.

A summary of average air emissions data are presented in Table 4-1, by test period. Average values in the table for the three coal/sorbent combinations and the overall reporting period average are arithmetic means of nonzero daily values recorded or calculated on days when Unit No. 4 and the sorbent injection equipment were operating at least some period of time. The goal of the demonstration program is to test a wide range of operating conditions, therefore, these averages should not be taken as representative of long-term, optimized operations. For this reason, ranges of SO_2 data have been shown and may include both injection and noninjection periods within a given day. A detailed analysis that breaks down emission monitoring data into shorter averaging periods is outside the scope of the EMP reporting requirements.

TABLE 4-1. SUMMARY OF AVERAGE AIR EMISSIONS DATA^a

	Average Coal Firing Rate (klb/hr)	Average Higher Heating Value (Btu/lb)	Average Opacity (%)	Average, Maximum and Minimum SO ₂ Emission		Median, Maximum & Minimum SO ₂ Removal Efficiency (%) ^c	Average, Maximum & Minimum NO _x Emissions	
				(lb/MMBtu)	(lb/hr) ^b		(lb/MMBtu)	(lb/hr) ^b
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal: 02/04 through 02/20/91.								
Average	49	12,355	3.6	3.6	2,200	23	0.42	250
Maximum				4.4	3,400	34	0.47	430
Minimum				2.1	1,000	21	0.36	170
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal: 02/21 through 04/05/91.								
Average	53	12,431	2.4	2.2	1,500	23	0.37	250
Maximum				3.3	2,200	73	0.44	330
Minimum				1.5	900	9	0.30	160
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal: 04/06 through 04/30/91.								
Average	52	12,269	1.5	3.4	2,200	39	0.41	260
Maximum				4.1	3,100	56	0.43	360
Minimum				2.9	1,500	33	0.38	180
Overall Reporting Period Average 02/04 through 04/30/91.								
Average	55	12,388	2.3	2.8	1,900	23	0.40	280
Maximum				4.4	3,400	73	0.48	430
Minimum				1.5	880	9	0.30	160
Baseline Period: 02/17 through 04/22/90. ^d								
Average	53	11,680	1.3	1.4	932	NA ^e	0.28	181
Maximum								
Minimum								

^a All emissions are calculated for each day, as shown in Appendix A. The values represent the average of those daily calculated values.

Average lb/hr values for each reporting period can be verified using the formula in footnote "b".

^b Values calculated as lbs/hr = [(lbs/MMBtu)(klb/hr)(Btu/lb)(1000 lb/klb)/(10⁶ Btu/MMBtu)].

^c Values presented here are not a direct indication of system performance. Calculations incorporate recorded data taken only during days when there was at least some LIMB operation. Zero values for off-line days were not used in calculating averages.

^d The data for baseline period results are presented in the report for the period of February, March and April 1990.

^e NA = Not applicable.

The summary of average air emissions data for the Baseline period are also arithmetic means of daily values recorded or calculated on days when Unit No. 4 was operating. However, the quality of these arithmetic means cannot be verified. As a result, the Baseline emission averages presented in Table 4-1 may be lower than actual emissions. An indication of NO_x emissions prior to the demonstration can be found in the technical paper "Operation of the LIMB/Humidifier Demonstration at Edgewater," presented at the First Combined FGD and Dry SO₂ Control Symposium, October 25, 1988, St. Louis, MO. This paper indicates that with B&W XCL burners, NO_x emissions range from 0.39 lb NO_x/MMBtu at a main steam flow of 425,000 lb/hr, to 0.48 lb NO_x/MMBtu at a main steam flow of 775,000 lb/hr.

Air emissions data for O₂ and CO₂ are not included in the summary table, since they are not considered pollutants. Data for CO is only used as a measurement of combustion efficiency and is therefore also not included in the summary tables. Monitoring data for O₂, CO₂, and CO are only evaluated and reported if modeled ambient air concentrations of SO₂ or NO_x are higher than the NAAQS. The results from total hydrocarbon (THC) testing, which were below 1 ppmv for the U.S. EPA portion of the Base LIMB Testing project (Baseline Report, 1988), demonstrated that no further THC monitoring was required.

The testing which occurred during this reporting period was largely performed during short time periods (2 to 6 hours per day). This method of testing was employed throughout most of this reporting period. However, when high sulfur coal was burned, testing took place continuously.

The SO₂ mass emission rate during this reporting period varied from 880 to 3,400 lb/hr. The average SO₂ mass emission rate was highest during the dolomitic lime/nominal 3.8 percent sulfur coal and lignosulfonated lime/nominal 3.8 percent sulfur coal injection periods, and was the lowest during the lignosulfonated lime/nominal 1.6 percent sulfur coal injection period. The average SO₂ mass emission rate for each

injection period, which ranged from 1,500 to 2,200 lb/hr, was higher than average SO₂ emissions during the Baseline period of 932 lb/hr. The median SO₂ removal efficiency was highest during the lignosulfonated lime/nominal 3.8 percent sulfur coal injection period and lowest during the dolomitic lime/nominal 3.8 percent sulfur coal and lignosulfonated lime/nominal 1.6 percent sulfur coal injection periods. SO₂ removal efficiencies for the reporting period varied widely, from 9 to 73 percent.

The average NO_x mass emission rates for the three sorbent/coal combinations ranged from 160 to 430 lb/hr this reporting period. The average NO_x mass emission rates for each injection period, which ranged from 250 to 260 lb/hr, were greater than the Baseline average NO_x emissions of 181 lb/hr. With the installation of B&W XCL low-NO_x burners, NO_x emissions during this reporting period have decreased when compared with emissions which occurred prior to the EPA LIMB Demonstration. Ambient air impacts from SO₂ and NO_x emissions during these three injection periods will be evaluated in the next report.

The average opacity during each dolomitic lime injection period ranged from 1.5 to 3.6 percent, as compared to average opacity during the Baseline period of 1.3 percent. The opacity remained well below the State of Ohio permit limit of 20 percent. The average opacity during all injection periods was slightly higher than the average opacity during the Baseline period. The average higher heating value (HHV) of the coal increased this reporting period over the Baseline period.

Daily emission rate data are presented in Appendix A. No manual flue gas testing was conducted for PM and particle size distribution during this reporting period. This type of testing has been previously performed and reported in LIMB Demonstration Extension: Emission Test Report Calcium Chloride Injection Study and the Draft LIMB Demonstration Extension Quality Assurance Project Plan. Testing for PM and PM₁₀ requires that the boiler and air pollution control equipment be operating at steady conditions for a minimum of 4 and 24 hours respectively. The injection of sorbent in

4-8 hour periods precluded the PM or PM₁₀ tests from being performed on a regular basis.

4.2 Wastewater Monitoring

The wastewater discharge points at the Edgewater facility are shown in Figure 4-1. The wastewater Outfalls are listed below:

1. **Outfall 001** - consists of condenser cooling water and discharges to Lake Erie.
2. **Outfall 002** - consists of intermittent storm water runoff from the fuel tank spill containment basin area, and also discharges to Lake Erie.
3. **Outfall 601** - discharges secondary ash pond effluent. Outfall 601 consists of all major plant wastewater streams and storm water runoff, including runoff from the truck loading area.
4. **Outfall 606** - consists of intermittent boiler blowdown discharge and drains to the Outfall 001 tunnel.

Only Outfall 601 contains any additional effluent or pollutant loadings as a result of the Coolside or LIMB Demonstration Project Extension testing. Compliance monitoring was conducted as required by the NPDES permit. Monthly discharge reports are submitted by Ohio Edison to the Ohio EPA for Outfalls 601, 606, 001, and 002 for the following parameters: pH, total suspended solids (TSS) (referred to in the permit as nonfilterable residue), flow, oil and grease (O&G), P, and As. TSS, pH, and flow were measured twice a week; O&G, and As were measured once a month. Total P was not analyzed during this reporting period. Outfall 601 was monitored daily for pH and temperature. Daily wastewater samples were also composited during the three sorbent/coal combinations for Ca analyses. Appendix B provides NPDES analytical data for the months of February, March and April 1991.

Temperature and pH data are shown in Appendix C for the period of February, March and April 1991. The Outfall 601 Ca analyses are shown in Appendix D.

Wastewater discharges at Outfall 601 were monitored during this reporting period. All discharge parameters were within NPDES permit requirements. Table 4-2 shows a comparison of the Baseline values versus the average, maximum, and minimum values for each parameter during the month indicated. Table 4-3 provides Outfall 601 wastewater quality data categorized by sorbent/coal combination. Table 4-4 presents the supplemental pH and Ca concentration data for Outfall 601 with the data categorized by sorbent/coal combination. It is important to note that Radian and Ohio Edison perform wastewater pH monitoring at different locations and times. As specified in the EMP, Radian takes daily pH measurements at the 601 outfall into Lake Erie. Ohio Edison monitors the pH as the wastewater flows from the primary settling pond to the secondary settling pond. Because of the differences in time and location of data collection, pH measurements are likely to differ. The pH may change as operations change at the facility. For example, the pH may fluctuate in the settling ponds when ash trucks are loaded or sorbent is unloaded. Therefore, the values in Tables 4-3 and 4-4, hence Appendix B and C, are not necessarily the same. Generally, the pH data in the two tables are within 0.5 pH units, a variation that is expected in neutral wastewater.

In addition, the monitoring data summarized in each report is from a variety of sources. Depending on the medium being sampled and the type of monitoring being performed, the dates of sampling may not match the sorbent/coal injection period. The dates of air emission and wastewater sampling recorded in Tables 4-1 and 4-3 match the injection period. The sampling dates recorded in Table 4-4, however, differ from other sampling episodes, in that wastewater samples are being composited for calcium analyses on a weekly basis (as per the EMP). Therefore, if a sorbent/coal injection period does not start on a Monday, then the injection period will not match the sampling period.

TABLE 4-2. NPDES OUTFALL 601 MONITORING DATA -
FEBRUARY, MARCH AND APRIL 1991

Reporting Period	Parameters					
	pH (s.u.)	TSS (mg/L)	Flow (MGD)	O&G (mg/L)	P (mg/L)	As (μg/L)
Sampling Frequencies	2/week	2/week	2/week	1/month	1/month	1/month
Permit Requirements						
Daily Limit	6-9	100	--	20	--	--
Monthly Limit	6-9	30	--	15	--	--
<u>February 1991^a</u>						
Average	---	7	1.1	1 ^c	NA ^c	25 ^c
Maximum	8.5	16	1.3	1 ^c	NA ^c	25 ^c
Minimum	7.1	3	0.7	1 ^c	NA ^c	25 ^c
<u>March 1991^a</u>						
Average	---	10	1.4	d	NA ^c	38
Maximum	8.2	24	2.6	d	NA ^c	43
Minimum	7.4	4	0.8	d	NA ^c	33
<u>April 1991^a</u>						
Average	---	13	1.1	d	NA ^c	NA ^c
Maximum	8.1	41	1.6	d	NA ^c	NA ^c
Minimum	7.0	2	0.7	d	NA ^c	NA ^c
<u>Baseline^b</u> (2/17-4/22/90)						
Average	---	14	1.9	1	0.15	70
Maximum	7.70	26	2.1	1	0.25	90
Minimum	7.40	7	0.2	d	0.05	48

- ^a Analytical data shown in Appendix B.
^b Analytical data from February, March and April 1990 Reporting Period.
^c Single data point for the month.
^d Below detection limits.
^e Not analyzed during test period.

**TABLE 4-3. SUMMARY OF NPDES OUTFALL 601 MONITORING DATA
DURING BASELINE AND EXTENSION PERIODS OF OPERATIONS**

Reporting Period Average, Maximum, and Minimum	Parameters					
	pH (s.u.)	TSS (mg/L)	Flow (MGD)	O&G (mg/L)	P (mg/L)	As (µg/L)
Sampling Frequencies	2/week	2/week	2/week	1/month	1/month	1/month
Permit Requirements						
Daily Limit	6-9	100	--	20	--	--
Monthly Limit	6-9	30	--	15	--	--
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal: 02/04 through 02/20/91 ^a						
Average	---	5.5	1.1	1 ^c	NA ^b	25 ^c
Maximum	7.6	8	1.3	1 ^c	NA ^b	25 ^c
Minimum	7.4	3	0.7	1 ^c	NA ^b	25 ^c
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal: 02/21 through 04/05/91 ^a						
Average	--	12	1.4	d	NA ^b	38
Maximum	8.5	41	2.6	d	NA ^b	43
Minimum	7.1	4	0.8	d	NA ^b	33
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal: 04/06 through 04/30/91 ^a						
Average	--	10	1.0	NA ^b	NA ^b	NA ^b
Maximum	8.1	24	1.5	NA ^b	NA ^b	NA ^b
Minimum	7.0	2	0.7	NA ^b	NA ^b	NA ^b
Overall Reporting Period Average: 02/01 through 04/30/91 ^a						
Average	--	10	1.2	1	NA ^b	34
Maximum	8.5	41	2.6	1	NA ^b	43
Minimum	7.0	2	0.7	d	NA ^b	25
Baseline Period: 02/17 through 04/22/90 ^c						
Average	--	14	1.9	1.0	0.15	70
Maximum	7.70	26	2.1	1	0.25	90
Minimum	7.40	7	0.2	d	0.05	48

^a Analytical data shown in Appendix B.

^b Not analyzed during test period.

^c Single data point for the period.

^d Below detection limits.

^e Analytical data from February, March and April 1990 Reporting Period.

TABLE 4-4. OUTFALL 601 pH AND Ca CONTENT

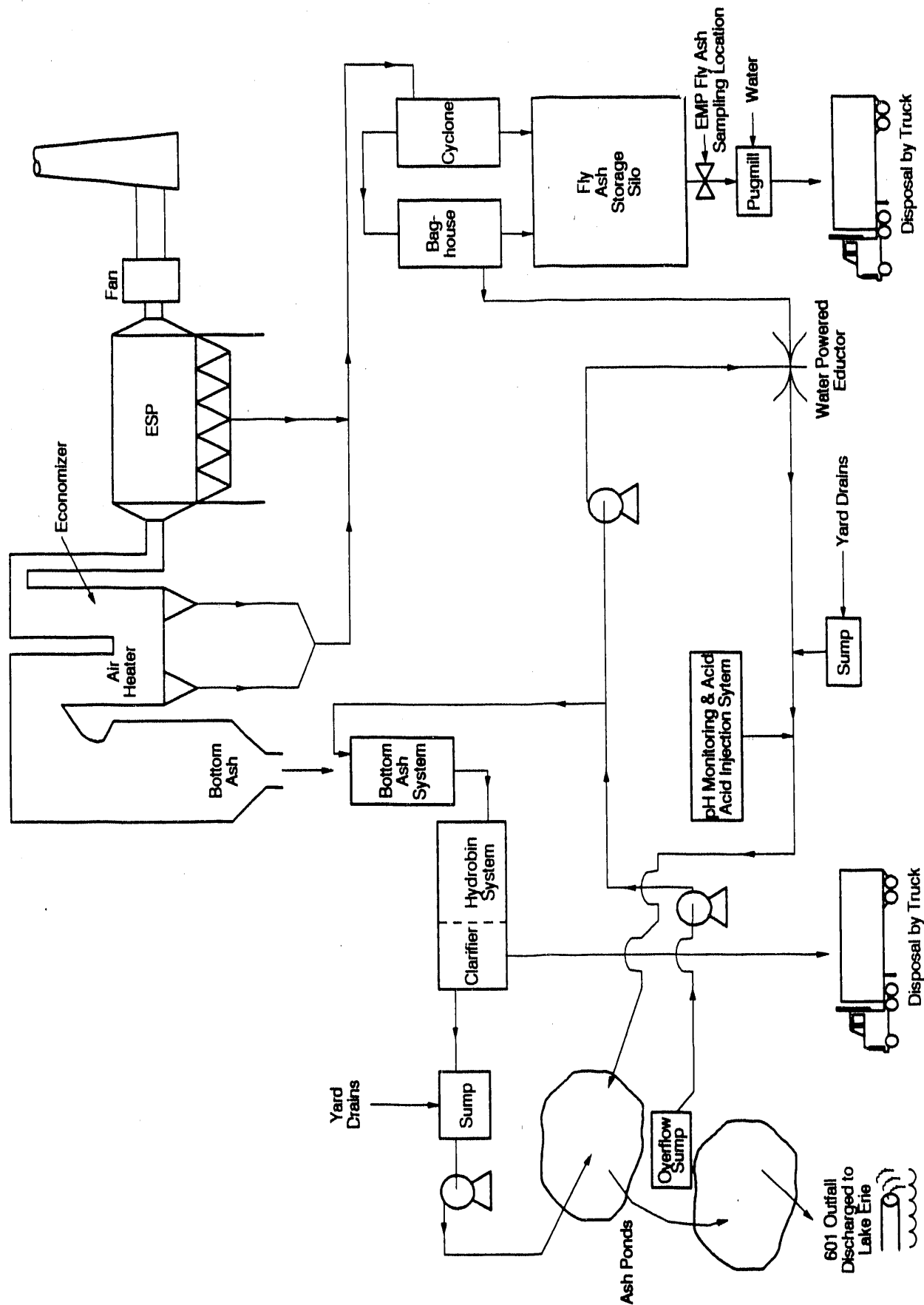
Sorbent/Coal Combination	Date of Sampling	Maximum pH (s.u.)	Minimum pH (s.u.)	Average Ca (mg/L)
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal ^{a,b}	02/04 - 02/20/91	8.31	7.20	59
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal ^{a,b}	02/21 - 04/05/91	8.37	7.07	58
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal ^{a,b}	04/06 - 04/30/91	7.87 --	7.09	50
Baseline: ^c	02/17 - 04/22/90	6.85	6.32	45

^a Daily pH data shown in Appendix C.^b Calcium analysis shown in Appendix D.^c Analytical data from February, March and April 1990 Reporting Period.

The maximum and minimum pH measurements recorded during the three sorbent/coal combinations were greater than the maximum and minimum pH measurements recorded during the Baseline period. The wastewater Ca concentration values ranged from 50 to 59 mg/L during the reporting period. The average, maximum, and minimum concentration of As during the dolomitic lime/nominal 3.8 percent sulfur coal and lignosulfonated lime/nominal 1.6 percent sulfur coal injection period were below Baseline values. The TSS average and minimum concentrations during this reporting period were at or below Baseline levels, however, the maximum concentration was higher than Baseline. The average and maximum O&G concentrations reported this period increased when compared to the Baseline. However, the increase in the O&G concentration was well below permit requirements and these O&G values were measured very near the stated detection limit of 1 mg/l. No P analyses were performed during this reporting period.

4.3 Solid Waste Discharges

The two solid waste streams generated from coal combustion at the Edgewater facility are boiler bottom ash and fly ash. A generalized schematic of the ash handling system is presented in Figure 4-2. Bottom ash generated during the project is not expected to present a major environmental impact. The bottom ash will not be analyzed, since LIMB Extension activities will not impact bottom ash generation and the quantity of bottom ash generated is a small percentage of the total ash produced. Only fly ash will be sampled and analyzed during the LIMB Extension periods of operation. The fly ash was sampled and analyzed during the dolomitic lime/nominal 3.8 percent sulfur coal and the dolomitic lime/nominal 1.6 percent sulfur coal injection periods. The dolomitic lime/3.8 percent sulfur coal ash sample was composited from October 8 to November 27, 1991. The dolomitic lime/1.6 percent sulfur coal ash sample was composited from November 28 to December 20, 1990.



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Figure 4-2. Edgewater Ash Handling System

During August and September 1990, lime injection was limited to daily test periods. Because these injection periods were so short, it was not possible to collect representative ash samples from the electrostatic precipitator (ESP) during these injection and non-injection periods. In October 1990, the testing program continued with short duration injection periods. At that time, Radian decided to attempt collection of representative ash samples from the ESP and from the ash truck loading chute. Fly ash, which was removed from the flue gas by the ESP and air heater/economizer, was sampled daily at the bottom of the ash storage silo.

The analyses on the composite ash sample produced for each sorbent/coal injection period will be compared to the results from the Baseline ash sample. The Baseline ash sample was composited from samples collected from February 17 through April 22, 1990.

As a part of the EMP, the fly ash generated during the LIMB Extension activities is subjected to the TCLP and the DI leaching procedure (ASTM 3987), with analysis of the DI leachate for 28 targeted parameters. Utility waste, such as fly ash, is an exempt category under the Resource Conservation and Recovery Act (RCRA), therefore leaching tests are not mandated to characterize a waste prior to disposal. However, the EMP specifies performing the TCLP and ASTM 3987 DI leaching procedures to provide an initial indication of leachate strength when the ash is disposed of in a sanitary landfill or monofill.

The TCLP and DI leaching procedures generate leachates from solid wastes, which are then analyzed for metals and inorganic elements and compounds. The fly ash samples generated during the LIMB Extension activities were leached using both procedures. The leachate from each DI procedure is analyzed for 23 targeted parameters and the TCLP for 8 parameters. Prior to March 1990, RCRA stipulated that the Extraction Procedure (EP) Toxicity Test be used to assess the toxicity characteristics of a solid waste. EPA promulgated a new toxicity characteristics ruling in March 1990

requiring that the TCLP replace the EP toxicity test. To ensure regulatory compliance during the Baseline period, ash composited during this period was subjected to the EP Toxicity Test and the TCLP as well as the DI leaching procedure. After the Baseline period, the EP toxicity test was no longer used and the ashes generated during the Extension periods of operation were leached using the TCLP and the DI procedures only. All extraction procedures and reference methods are provided in Appendix E.

The leachates from the TCLP and the DI leaching procedure were analyzed for the eight RCRA-regulated metals [silver (Ag), As, barium (Ba), cadmium (Cd), chromium (Cr), mercury (Hg), lead (Pb), and selenium (Se)]. The TCLP and the EP Toxicity Tests are acid extractions ($\text{pH} < 5.0$) and are not suitable for the non-metal analyses. Therefore, a neutral leaching procedure was selected (ASTM D3987) to produce a leachate to monitor for the following secondary constituents: total phenolics, pH, Ca, chloride (Cl), fluoride (F), potassium (K), sodium (Na), nitrate, sulfate, total hardness, total alkalinity, acidity, bicarbonate, carbonate, chemical oxygen demand (COD), total organic carbon (TOC), total dissolved solids (TDS), iron, and copper.

The corrosivity and permeability results on the composite samples from the two sorbent/coal injection periods are compared to the Baseline in Table 4-5. Tests of the Baseline ash demonstrated a corrosivity of 1.3 mm/yr. Corrosivities of both ash samples were below the detection limits for the test. The permeability of the ash from each of the two sorbent/coal injection periods was lower than that of the Baseline ash sample. This decrease in permeability was expected, since the two sorbents can act as a cement material. This characteristic is advantageous in a landfill because it demonstrates a decreased tendency for leachate to flow through the spent sorbent/ash matrix. The permeability data summary is provided in Appendix G.

The analytical results for metals and secondary analytes from the two sorbent/coal injection periods are compared to the Baseline concentrations in Table 4-6. The concentrations of metals for the two injection periods, both TCLP and DI leachates,

TABLE 4-5. ESP ASH CORROSIVITY AND PERMEABILITY RESULTS

Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal ^a	
Parameter	Results
Corrosivity ^b	c
Permeability	1.3×10^{-5} cm/sec
Dolomitic Lime/Nominal 1.6 Percent Sulfur Coal ^a	
Parameter	Results
Corrosivity ^b	c
Permeability	3.7×10^{-6} cm/sec
Baseline ^d	
Parameter	Results
Corrosivity ^b	1.3 mm/yr
Permeability	5.3×10^{-4} cm/sec

^a Analytical data shown in Appendix G.

^b Conducted on DI leachate sample.

^c Below detection limits.

^d Analytical data from May, June and July 1990 Report.

**TABLE 4-6. ANALYSES PERFORMED ON FLY ASH LEACHATES FROM THE
BASELINE AND EXTENSION PERIODS OF OPERATION**

	BASELINE ASH ^a		DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL ^b		DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL ^b	
Parameter	DI Leachate (mg/L)	DI Detection Limit (mg/L)	DI Leachate (mg/L)	DI Detection Limit (mg/L)	DI Leachate (mg/L)	DI Leachate Detection Limit (mg/L)
Total Phenolics	0.023 ^c	0.0050	ND ^d	0.0050	0.012 ^c	0.0050
pH	4.0	--	12	--	12	--
Calcium	460	1.0	1500	10	1700	4.0
Chloride	3.2 ^c	1.0	98	1.0	460	50
Fluoride	3.3	0.10	3.8	0.20	2.3	0.20
Potassium	ND ^d	3.0	23	3.0	12 ^c	3.0
Sodium	43	1.0	9.6	1.0	4.9 ^c	1.0
Nitrate	0.2	0.020	ND ^d	0.020	0.26	0.020
Sulfate	1700	100	1000	100	1300	100
Total Hardness	1268	66	NA ^e	--	NA ^e	NA ^e
Total Alkalinity as CaCO ₃	ND ^d	1.0	2300	1.0	2300	1.0
Acidity	250	1.0	NA ^e	--	NA ^e	NA ^e
Bicarbonate	ND ^d	1.0	ND ^d	--	ND ^d	NA ^e
Carbonate	ND ^d	1.0	110	--	120 ^e	--
Chemical Oxygen Demand (COD)	ND ^d	5.0	8.3 ^c	5.0	11 ^c	5.0
Total Organic Carbon (TOC)	2.2 ^c	1.0	ND ^d	1.0	ND ^d	1.0
Total Dissolved Solids (TDS)	2700	9.0	4000	9.0	5000	9.0
Iron	3.8	0.040	NA ^e	--	NA ^e	--
Copper	1.1	0.020	NA ^e	--	NA ^e	--
Magnesium	29	1.0	NA ^e	--	NA ^e	--
Silver	ND ^d	0.010	ND ^d	0.10	ND ^d	0.40
Arsenic	0.0042 ^c	0.0040	0.011 ^c	0.0040	0.0040 ^c	0.0040
Barium	0.062	0.010	0.60	0.010	0.48	0.040
Cadmium	0.069	0.0050	ND ^d	0.050	ND ^d	0.020
Chromium	0.27	0.010	0.030	0.010	0.29	0.040
Mercury	ND ^d	0.0002	ND ^d	0.0002	ND ^d	0.0002
Lead	0.0097 ^c	0.0030	0.027	0.0030	0.012 ^c	0.0030
Selenium	0.059	0.0050	0.13	0.025	0.071	0.0050

^a Analytical data from May, June and July 1990 Report.

^b Analytical data shown in Appendix F.

^c Estimated results less than 5 times the practical quantitation limit.

^d Below detection limits.

^e Not analyzed.

TABLE 4-6. (CONTINUED)

Parameter	BASELINE ASH ^a		DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL ^b		DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL ^b	
	TCLP (mg/L)	TCLP Detection Limit (mg/L)	TCLP Leachate (mg/L)	TCLP Detection Limit (mg/L)	TCLP Leachate (mg/L)	TCLP Detection Limit (mg/L)
Total Phenolics	NA ^c	--	NA ^c	--	NA ^c	--
pH	NA ^c	--	NA ^c	--	NA ^c	--
Calcium	NA ^c	--	NA ^c	--	NA ^c	--
Chloride	NA ^c	--	NA ^c	--	NA ^c	--
Fluoride	NA ^c	--	NA ^c	--	NA ^c	--
Potassium	NA ^c	--	NA ^c	--	NA ^c	--
Sodium	NA ^c	--	NA ^c	--	NA ^c	--
Nitrate	NA ^c	--	NA ^c	--	NA ^c	--
Sulfate	NA ^c	--	NA ^c	--	NA ^c	--
Total Hardness	NA ^c	--	NA ^c	--	NA ^c	--
Total Alkalinity as CaCO ₃	NA ^c	--	NA ^c	--	NA ^c	--
Acidity	NA ^c	--	NA ^c	--	NA ^c	--
Bicarbonate	NA ^c	--	NA ^c	--	NA ^c	--
Carbonate	NA ^c	--	NA ^c	--	NA ^c	--
Chemical Oxygen Demand (COD)	NA ^c	--	NA ^c	--	NA ^c	--
Total Organic Carbon (TOC)	NA ^c	--	NA ^c	--	NA ^c	--
Total Dissolved Solids (TDS)	NA ^c	--	NA ^c	--	NA ^c	--
Iron	NA ^c	--	NA ^c	--	NA ^c	--
Copper	NA ^c	--	NA ^c	--	NA ^c	--
Magnesium	NA ^c	--	NA ^c	--	NA ^c	--
Silver	0.013 ^c	0.010	ND ^d	0.10	ND ^d	0.040
Arsenic	1.5	0.30	3.7 ^c	3.0	ND ^d	1.2
Barium	0.053	0.010	0.47	0.010	1.1	0.040
Cadmium	0.013 ^c	0.0050	ND ^d	0.050	ND ^d	0.020
Chromium	0.016 ^c	0.010	0.19	0.010	0.018 ^c	0.010
Mercury	ND ^d	0.0002	ND ^d	0.0002	ND ^d	0.0002
Lead	ND ^d	0.050	ND ^d	0.50	ND ^d	0.20
Selenium	0.058	0.0050	0.21	0.025	0.024 ^c	0.0050

^a Analytical data from May, June and July 1990 Report.^b Analytical data shown in Appendix F.^c Estimated results less than 5 times the practical quantitation limit.^d Below detection limits.^e Not analyzed.

were below all TCLP MCL's, and the concentrations were below the detection limit for Ag, Cd, and Hg.

An increase was observed in the pH, Ca, Cl, K, total alkalinity, and TDS concentrations for both injection periods over the Baseline. This increase was expected, due to the presence of spent sorbent material in the fly ash. A decrease was observed in the Na, sulfate, and TOC concentrations for both injection periods over the Baseline. The Fl concentration of the dolomitic lime/nominal 3.8 percent sulfur coal injection period increased over the Baseline; whereas, the Fl concentration of the dolomitic lime/nominal 1.6 percent sulfur coal injection period decreased over the Baseline. The nitrate concentration of the dolomitic lime/nominal 3.8 percent sulfur coal injection period decreased over the Baseline; whereas, the nitrate concentration of the dolomitic lime/nominal 1.6 percent sulfur coal injection period increased over the Baseline. The analytical data summary is provided in Appendix F.

4.4 Pollution Control Limit Monitoring

The pollution control systems for gaseous and aqueous discharges from Unit No. 4 were continuously monitored throughout the months of February, March and April 1991. Stack gas emissions from Unit No. 4 were controlled with the LIMB system and the existing ESP.

The LIMB operating log for the months indicated is presented in Table 4-7. Some of the reasons for various outages and system upsets are included in the table. In February 1991, the system operated for a total of 384 hours of formal testing. During March 1991, no formal testing was conducted for 13 days. The system operated for 122 hours of formal testing during the month, with the system off-line due to low power demands. In April 1991, the system operated for a total of 213 hours of formal testing. Occasional shutdowns were reported for sorbent injection line repairs, plugged sorbent injection hoses, and to "zero" monitoring equipment.

**TABLE 4-7. LIMB OPERATION LOG FOR JANUARY, FEBRUARY, MARCH,
AND APRIL 1991**

Date	Hours of Line Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Ratio	Humidifier Outlet Temp (F)	Daily Hours out of service
	From	To	From	To				
910121	1000	1300	-	-	3	2	-	21
910122	1200	1400	-	-	2	1.0	-	22
910123	LIMB unit off-line.							24
910124	1000	1300	-	-	3	2.0	-	21
910125	1100	1330	-	-	2.5	1.6	-	21.5
910126	LIMB unit off-line.							24
910127	LIMB unit off-line.							24
910128	1000	1300	-	-	3	2.0	-	21
910129	1030	1500	1030	1500	4.5	1.6	-	19.5
910130	1030	1530	1030	1530	5	1.6	260	19
910131	1200	1730	-	-	5.5	1.2/2.2	-	18.5
910201	930	2100	-	-	13.5	2	-	10.5
910202	LIMB unit off-line.							24
910203	LIMB unit off-line.							24
910204	1030	1800	1430	1800	7.5	2	260	16.5
910205	1300	2400	1300	2400	11	2	260	13
910206	0	2400	0	2400	24	2	260	0
910207	0	2100	0	2100	21	1.2/1.6	260	3
910208	LIMB unit off-line.							24
910209	LIMB unit off-line.							24
910210	LIMB unit off-line.							24
910211	300	2400	1145	2400	21	0.8/2.2	260	3
910212	0	2400	0	2400	24	0.8/1.0	260	0
910213	0	2400	0	2400	24	2	145/260	0
910214	0	2400	0	2400	24	1.2	250	0
910215	0	2400	0	2400	9	1.5	250	15
910216	0	2400	0	2400	24	1.5	250	0
910217	0	2400	0	2400	24	1.0/1.5	250	0
910218	0	2400	0	2400	24	1.75	250	0
910219	0	2400	0	2400	24	1.05	250	0
910220	0	2400	0	2400	24	1.2/1.5	250	0
910221	0	2400	0	2400	24	1.0/2.1	240	0
910222	0	2400	0	2400	24	1.5	250	0
910223	LIMB unit off-line.							24
910224	LIMB unit off-line.							24
910225	1300	2400	1300	2400	11	1.5	275	13
910226	0	1500	0	1600	10	2.2	275	14
910227	1200	1500	1200	1500	3	2	275	21
910228	1100	2400	1100	2400	13	1.8	275	11
910301	0	1330	0	1330	13.5	1.6	275	10.5
910302	LIMB unit off-line.							24
910303	LIMB unit off-line.							24
910304	1030	1200	1030	1200	1.5	1.5	275	22.5
910305	0	2400	0	2400	24	1.0/1.5	275	0
910306	0	1530	0	1530	15.5	1.3	275	8.5
910307	1000	1700	1000	1700	7	1.0/1.8	275	17
910308	930	1400	930	1400	4.5	0.8	275	19.5
910309	LIMB unit off-line.							24
910310	LIMB unit off-line.							24

TABLE 4-7. (CONTINUED)

Date	Hours of Line Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Ratio	Humidifier Outlet Temp (F)	Daily Hours out of service
	From	To	From	To				
910311	1000	2400	1000	2400	14	1.4/2.0	275	10
910312	0	2400	0	2400	24	1.2/1.4	275	0
910313	0	1500	0	1500	15	1.0/1.2	275	9
910314	LIMB unit off-line.							24
910315	LIMB unit off-line.							24
910316	LIMB unit off-line.							24
910317	LIMB unit off-line.							24
910318	LIMB unit off-line.							24
910319	LIMB unit off-line.							24
910320	LIMB unit off-line.							24
910321	LIMB unit off-line.							24
910322	1300	1600	1300	1600	3	1.6	275	21
910323	LIMB unit off-line.							24
910324	LIMB unit off-line.							24
910325	LIMB unit off-line.							24
910326	LIMB unit off-line.							24
910327	LIMB unit off-line.							24
910328	LIMB unit off-line.							24
910329	LIMB unit off-line.							24
910330	LIMB unit off-line.							24
910331	LIMB unit off-line.							24
910401	1300	1700	1300	1700	0	2.2	275	20
910402	1000	1700	1000	1700	7	2.2	264	17
910403	900	1700	900	1700	8	1.45	275	16
910404	1500	2400	1500	2400	9	1.0/1.85	275	15
910405	0	1700	0	1700	17	1.0/2.0	275	7
910406	LIMB unit off-line.							24
910407	LIMB unit off-line.							24
910408	LIMB unit off-line.							24
910409	LIMB unit off-line.							24
910410	LIMB unit off-line.							24
910411	1000	2400	1000	2400	14	1.8	275	10
910412	0	1800	0	1800	18	2	275	6
910413	LIMB unit off-line.							24
910414	LIMB unit off-line.							24
910415	1100	2400	1100	2400	13	1.4	275	11
910416	0	2400	0	2400	24	1.0/1.8	275	0
910417	0	2400	0	2400	24	2	275	0
910418	0	2200	0	2200	22	1.0/1.8	275	2
910419	LIMB unit off-line.							24
910420	LIMB unit off-line.							24
910421	LIMB unit off-line.							24
910422	1100	2400	1100	2400	13	1.2/1.5	275	11
910423	0	2400	0	2400	24	0.8/1.65	275	0
910424	0	1600	0	1600	16	1.75	275/145/275	8
910425	LIMB unit off-line.							24
910426	LIMB unit off-line.							24
910427	LIMB unit off-line.							24
910428	LIMB unit off-line.							24
910429	LIMB unit off-line.							24
910430	LIMB unit off-line.							24

5.0 AMBIENT MONITORING

This section presents the results of ambient air dispersion modeling and ground-water monitoring. Section 5.1 discusses ambient air impacts predicted by using dispersion models with data from the November and December 1990, and January 1991 reporting period. Section 5.2 discusses ground-water monitoring.

5.1 Ambient Air Dispersion Modeling

Air dispersion modeling was conducted using EPA-approved models to assess ground level pollutant concentrations during sorbent injection periods. The air quality results presented in this section are based on emission and stack parameter data that are specific to the coal/sorbent combination utilized during a specific test period and the combination of combustion/control equipment used at this facility. Ambient air dispersion modeling was performed to determine site-specific air quality impacts. However, since the predicted impacts are dependent on site-specific factors, the results of air quality modeling conducted at another facility using this combustion and control technology would be expected to vary on a case-by-case basis. The site-specific parameters that would affect the modeling results are meteorological data, size of property (distance to nearest ambient air receptor), type of combustion and air pollution control equipment employed, operating conditions (including percent sulfur of coal and degree of pulverization), and stack parameter data.

Air dispersion modeling was conducted to assess ground level pollutant concentrations during seven injection periods. Table 5-1 shows the sorbent/coal combinations and dates used for each period. Modeling results show that the change in concentration (i.e., injection period maximum impacts minus Baseline maximum impacts) is less than $1.0 \mu\text{g}/\text{m}^3$ for NO_x for all sorbent/coal injection periods modeled. Therefore, no further modeling of NO_x was required. During the screening or initial modeling, results for SO_2 indicated an increase in impacts over the Baseline values that were

TABLE 5-1. INJECTION PERIODS MODELED FOR THE CURRENT REPORT

SORBENT/COAL COMBINATION	INJECTION PERIOD
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL	(11/01/90 - 11/12/90)
DOLOMITIC LIME/NOMINAL 3.0 PERCENT SULFUR COAL	(11/13/90 - 11/16/90)
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL	(11/17/90 - 11/27/90)
DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL	(11/28/90 - 12/04/90)
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL	(12/05/90 - 12/21/90)
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL	(12/22/90 - 01/06/91)
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL	(01/07/91 - 01/23/91)

greater than the PSD significance criteria for the 3-hour, 24-hour and annual averaging periods. Therefore, additional modeling of SO₂ impacts was required. As shown in this section, additional modeling demonstrated that the SO₂ NAAQS were not exceeded during any of the sorbent/coal injection periods.

The modeling methodology followed for this analysis and all other analyses was outlined in the report covering the period of October 1989 to January 1990. The five-part methodology compares modeled ground level concentrations for the no sorbent injection (Baseline) case and modeled ground level concentrations during each of the coal/sorbent combinations to be evaluated. The ambient air modeling protocol is provided in Appendix H.

5.1.1 Air Quality Source Parameters

To determine the combination of stack parameter and emission rate data that will predict the maximum air quality impacts, two data sets of representative stack parameters and emission rates were evaluated for each sorbent/coal injection period. In general, plume dispersion, and therefore, maximum predicted impacts are dependent on the stack parameters input to the model.

Therefore, prior to the ISCST modeling, two sets of data were evaluated, the maximum SO₂ and NO_x emission rates with the maximum stack exit velocity for each injection period, and the mean SO₂ and NO_x emission rates with the mean stack exit velocity for each sorbent/coal injection period. These two data sets were input to the EPA SCREEN dispersion model to determine the most conservative set of operating conditions for each injection period. Table 5-2 shows the scenario (i.e., maximum or average emission rate and exit velocity) and corresponding emission rate and exit velocity producing the largest impacts for each test period and pollutant. The maximum impacts are predicted to occur using the maximum emission rate and exit velocity for all cases

TABLE 5-2. WORST-CASE OPERATING SCENARIOS DETERMINED BY SCREEN

Injection Period	SO ₂		NO _x		Scenario	Exit Velocity (m/s)	Emission Rate (g/s)	Exit Velocity (m/s)	Emission Rate (g/s)	Scenario	Exit Velocity (m/s)	Emission Rate (g/s)	Exit Velocity (m/s)	Scenario
	Emission Rate (g/s)	Exit Velocity (m/s)	Emission Rate (g/s)	Exit Velocity (m/s)										
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/01/90 - 11/12/90)	72.7	13.8	59.2	16.9	Average	16.9	59.2	16.9	59.2	Average	16.9	59.2	16.9	Maximum
DOLOMITIC LIME/NOMINAL 3.0 PERCENT SULFUR COAL (11/13/90 - 11/16/90)	421.7	15.2	54.2	15.2	Maximum	15.2	54.2	15.2	54.2	Maximum	15.2	54.2	15.2	Maximum
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/17/90 - 11/27/90)	234.0	13.0	58.4	18.2	Average	18.2	58.4	18.2	58.4	Average	18.2	58.4	18.2	Maximum
DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL (11/28/90 - 12/04/90)	273.5	15.0	50.4	15.0	Maximum	15.0	50.4	15.0	50.4	Maximum	15.0	50.4	15.0	Maximum
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (12/05/90 - 12/21/90)	262.0	14.0	45.3	14.0	Maximum	14.0	45.3	14.0	45.3	Maximum	14.0	45.3	14.0	Maximum
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (12/22/90 - 01/06/91)	287.6	15.8	50.3	15.8	Maximum	15.8	50.3	15.8	50.3	Maximum	15.8	50.3	15.8	Maximum
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (01/07/91 - 01/23/91)	293.1	15.4	50.3	15.4	Maximum	15.4	50.3	15.4	50.3	Maximum	15.4	50.3	15.4	Maximum

except two dolomitic lime/nominal 1.6 percent sulfur coal test periods (11/01 to 11/12/90, and 11/17 to 11/27/90), for SO₂.

The worst-case emission rates and exit velocities, which were input to the more refined ISCST dispersion model, were based on daily averages reported during each injection period. A representative exit temperature of 350°K was obtained from B&W and input to the ISCST model. The total suspended particulates (TSP) and CO emission rates, and therefore, their ambient impacts, are assumed not to change substantially during any portion of the LIMB Demonstration Project Extension. As a result, no comparison is made for these pollutants between the sorbent/coal injection periods and the Baseline period.

5.1.2 Air Quality Modeling Procedure

The modeling was performed as outlined in the protocol discussed in the report covering the November 1989 to January 1990 period. A five-year analysis (1981-1985) was performed for each sorbent/coal injection period. The receptor grid used in the analysis, shown in Figure 5-1, is identical to the one described in previous reports. The 5 part analysis for the dolomitic lime/nominal 3.8 percent sulfur coal, worst-case model predicted impacts for SO₂ that were greater than the NAAQS for the 3-hour and 24-hour averaging periods. Therefore, further refinement was included in the analysis of this injection period in order to better represent the actual ambient impacts that occurred.

Since the objective of the EMP is to assess the environmental impact due to the DOE LIMB Demonstration Project Extension, the refined analysis used representative meteorological data recorded at Cleveland-Hopkins and Buffalo International Airports from 11/27 to 12/07/90. The unprocessed data required for this analysis was obtained from the National Climatic Data Center and processed by Radian into the format necessary to run the ISCST model. The meteorological data consisted of hourly surface observations of wind speed and direction, temperature, ceiling height, cloud cover and

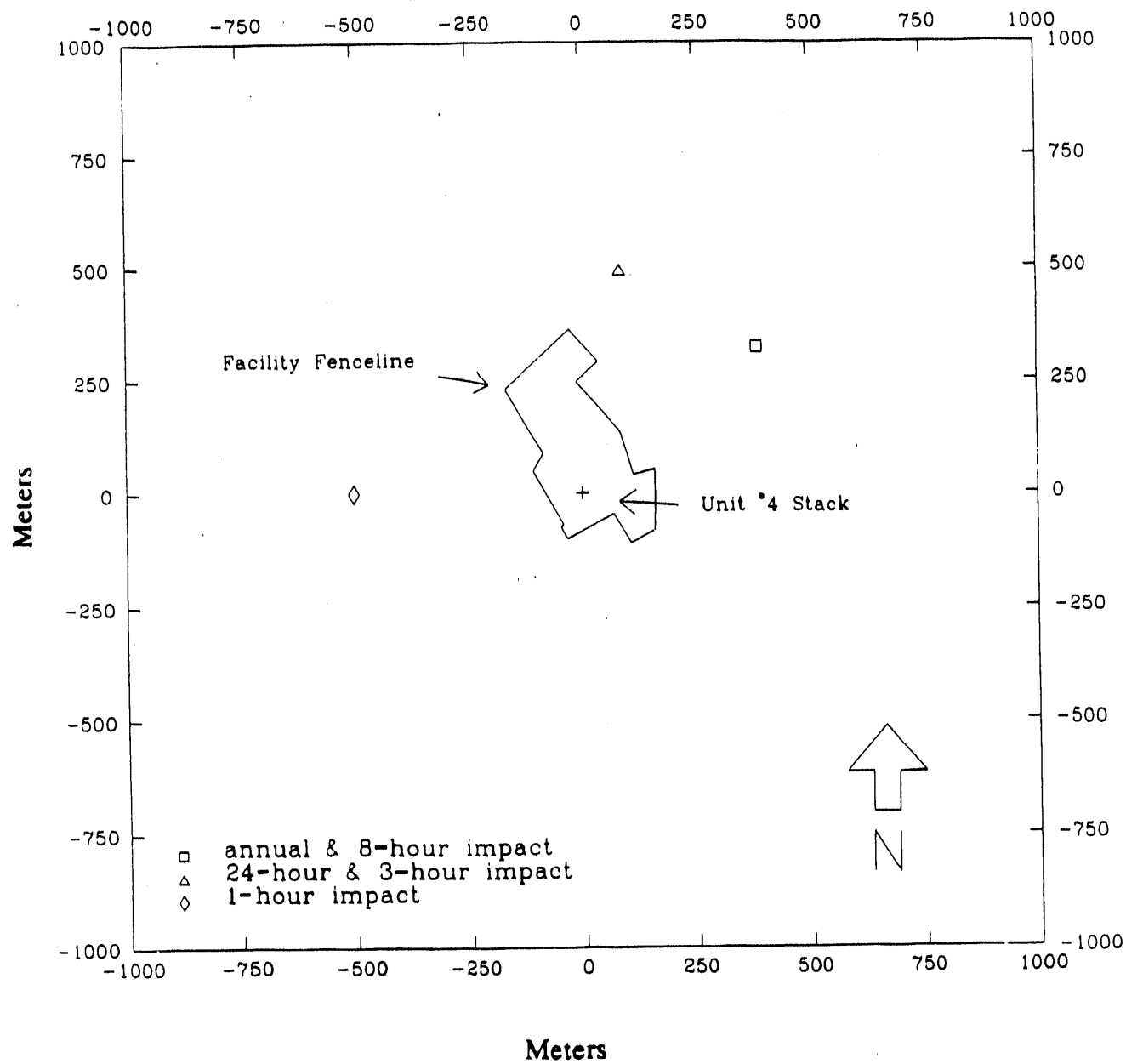


Figure 5-1. Modeled Impact Locations

total opaque cloud cover, taken at Cleveland-Hopkins Airport. Mixing height data were derived from twice-daily radiosonde observations and hourly surface data recorded at Buffalo International Airport. The mixing heights were determined using the methodology outlined by Holzworth.¹ Appendix I contains the hourly surface observations and the mixing height data input to the EPA PCRAMMET meteorological pre-processor program, which generates a file used by the ISCST model. Except for the use of real-time meteorological data, the refined modeling analysis conforms to the 5-part methodology described above.

5.1.3 Air Quality Modeling Results

The difference between the Baseline impacts and the impacts for each sorbent/coal injection period are shown in Table 5-3. Positive values indicate an increase in modeled impacts over Baseline impacts. The change in NO_x concentrations varies only slightly between the seven injection periods, from $0.4 \mu\text{g}/\text{m}^3$ to $0.1 \mu\text{g}/\text{m}^3$. The change in SO_2 concentrations is positive for all SO_2 averaging periods for each sorbent/coal injection period except the dolomitic lime/nominal 1.6 percent sulfur coal injection period from 11/17 to 11/27/90.

As outlined in the report covering the period of October 1989 to January 1990, increases in predicted air quality impacts over Baseline conditions were compared to the ambient air significance levels as defined in the PSD air regulations. For SO_2 and NO_x , these values are $1.0 \mu\text{g}/\text{m}^3$ for the annual average, $5 \mu\text{g}/\text{m}^3$ for the 24-hour average, and $25 \mu\text{g}/\text{m}^3$ for the 3-hour average. For NO_x , no increases exceeded $1.0 \mu\text{g}/\text{m}^3$ for the annual average, therefore, no further evaluation was necessary for this pollutant. For SO_2 , two averaging periods have increases less than their respective significance levels (dolomitic lime/nominal 1.6 percent sulfur coal and limestone/nominal 1.6 percent sulfur coal); therefore, these two periods require no further evaluation.

TABLE 5-3. SCENARIO IMPACTS MINUS BASELINE RESULTS

Scenario	Averaging Period	SO ₂ Scenario minus Baseline (µg/m ³)	Greater Than Significance Level	NO _x Scenario minus Baseline (µg/m ³)	Greater Than Significance Level
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/01 - 11/12/90)	Annual	2	Yes	0.4	No
	24 Hour	15.2	Yes	NA	No
	3 Hour	38.8	Yes	NA	No
DOLOMITIC LIME/NOMINAL 3.0 PERCENT SULFUR COAL (11/13 - 11/16/90)	Annual	10.4	Yes	0.4	No
	24 Hour	94	Yes	NA	No
	3 Hour	240.4	Yes	NA	No
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/17 - 11/27/90)	Annual	0	No	0.2	No
	24 Hour	-3.6	No	NA	No
	3 Hour	-9.7	No	NA	No
DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL (11/28 - 12/04/90)	Annual	15.6	Yes	0.4	No
	24 Hour	138.9	Yes	NA	No
	3 Hour	354.8	Yes	NA	No
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (12/5 - 12/21/90)	Annual	1	Yes	0.1	No
	24 Hour	6.5	Yes	NA	No
	3 Hour	16.5	No	NA	No
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (12/22/90 - 01/06/91)	Annual	0.8	No	0.1	No
	24 Hour	7.3	Yes	NA	No
	3 Hour	18.4	No	NA	No
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (1/7 - 1/23/91)	Annual	1.6	Yes	0.1	No
	24 Hour	13.8	Yes	NA	No
	3 Hour	35.5	Yes	NA	No

The October 1989 to January 1990 report presents a methodology to address situations when the difference between a test period impact and the Baseline impact is greater than PSD air significance levels. The methodology requires an evaluation of existing air monitoring data. Monitoring data were obtained from Ohio Environmental Protection Agency (OEPA) for two sites in Lorain, Ohio. The monitoring site best representing ambient background concentrations is located 6 km southeast of the Edgewater facility at 2270 East 42nd Street. The 42nd Street monitoring site is close enough to be representative of background concentrations for the Lorain area while its location is such that the monitored values include only a small contribution from the Unit 4 stack. Two years (1986 and 1987) of monitoring data were available from the East 42nd Street monitor. Table 5-4 summarizes the monitoring data provided by the OEPA. The maximum annual value for the two years of data used is shown in this table. To determine background concentrations for an air quality analysis conducted using five years of meteorological data, the second highest monitored concentration is selected to determine compliance with the short term NAAQS (24-hour and 3-hour).

Table 5-5 presents the maximum predicted impacts (i.e., ISCST predicted impacts plus monitored values) for each of the injection periods and averaging periods with increases greater than PSD significance levels. The results show that a potential exceedance of the 24-hour SO₂ NAAQS occurred during the dolomitic lime/3.8 percent sulfur coal combination. However, no other potential NAAQS exceedances are predicted for the injection periods modeled for this report.

As discussed in Section 5.1.2, a further refinement of the modeling analysis was performed in order to more accurately predict impacts during the dolomitic lime/3.8 percent sulfur coal injection period. Refined modeling shows that the 24-hour impact (using representative meteorological data) is 19.0 $\mu\text{g}/\text{m}^3$ lower than the Baseline concentration. Thus, as shown in Table 5-6, the use of representative meteorological data produces a model predicted impact which is less than the Baseline predicted impact.

TABLE 5-4. AMBIENT AIR QUALITY MONITORING DATA

Averaging Period	SO ₂ Ambient Monitored ¹ Value (µg/m ³)	Monitored Year
Annual	23	1987
24-Hour ²	83	1986
3-Hour ²	411	1986

¹ Monitor location is 2270 East 42nd Street, Lorain, Ohio.

² Second-highest impacts from 1986 and 1987 data.

TABLE 5-5. TOTAL SCENARIO SO₂ IMPACTS

Scenario	Averaging Period	Scenario Impact ¹ + Monitored SO ₂ Value (µg/m ³)	Percent Of NAAQS
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/01 - 11/12/90)	ANNUAL	42.9	54%
	24 HOUR	262	72%
	3 HOUR	867	67%
DOLOMITIC LIME/NOMINAL 3.0 PERCENT SULFUR COAL (11/13 - 11/16/90)	ANNUAL	51.3	64%
	24 HOUR	341	93%
	3 HOUR	1070	82%
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (11/17 - 11/27/90)	ANNUAL	NA	NA
	24 HOUR	NA	NA
	3 HOUR	NA	NA
DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL (11/28 - 12/04/90)	ANNUAL	56.5	71%
	24 HOUR	386	106%
	3 HOUR	1180	91%
DOLOMITIC LIME/NOMINAL 1.6 PERCENT SULFUR COAL (12/5 - 12/21/90)	ANNUAL	41.9	52%
	24 HOUR	253	69%
	3 HOUR	NA	NA
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (12/22/90 - 01/06/91)	ANNUAL	NA	NA
	24 HOUR	254	70%
	3 HOUR	NA	NA
LIMESTONE/NOMINAL 1.6 PERCENT SULFUR COAL (1/7 - 1/23/91)	ANNUAL	42.5	12%
	24 HOUR	261	71%
	3 HOUR	864	66%

¹ 24-hour and 3 hour scenario impacts are second-highest impacts.

TABLE 5-6. SCENARIO IMPACTS MINUS BASELINE RESULTS
FOR THE REFINED MODELING ANALYSIS

Scenario	Averaging Period	SO ₂ Scenario minus Baseline ($\mu\text{g}/\text{m}^3$)	Greater Than Significance Level
DOLOMITIC LIME/NOMINAL 3.8 PERCENT SULFUR COAL (11/28 - 12/04/90)	24 Hour	-19.0	No

In summary, it should be emphasized that the intent of the EMP is to assess the environmental impact due to the DOE LIMB Demonstration Project Extension. The use of 5 years of meteorological data to determine the worst-case ambient impact during a 1 to 2 week test period is a very conservative methodology. However, the use of real-time meteorological data for each injection period is not always practical due to the difficulty of obtaining and processing the data in time for each report.

5.2 Groundwater

No further definition of the environmental impact of fly ash disposal at the Kimble Landfill or Ohio Edison Ash Disposal Facility was possible during this period, since no additional ground-water monitoring data were generated.

5.3 References

1. Holzworth, G. C., Mixing Heights, Wind Speed, and Potential For Urban Air Pollution Throughout Contiguous United States. U. S. Environmental Protection Agency, Research Triangle Park, North Carolina. January 1972.

6.0 HEALTH AND SAFETY

All employee air and noise monitoring was completed in the previous reporting periods. No further employee exposure monitoring is planned for future reporting periods.

7.0 COMPLIANCE MONITORING STATUS

Compliance monitoring is required for both gaseous and aqueous discharges. Opacity measurements, particulate emissions and SO₂ emissions estimates are required to meet source permit operation requirements. Particulate emission measurements are required once every 3 years. Opacity measurements are monitored continuously, and SO₂ emissions are to be estimated daily by using a coal sulfur content estimation method approved by the Ohio EPA. Table 7-1 presents the air compliance monitoring requirements for point sources at the facility. No compliance violations occurred for SO₂, opacity, or particulate loading during the February, March and April 1991 reporting period.

Tables 4-2 and 4-3 provide wastewater compliance monitoring limitations and measured pollutant parameters. No NPDES permit values were exceeded in wastewater samples collected by Radian or Ohio Edison personnel during this reporting period.

**TABLE 7-1. SUMMARY OF CURRENTLY REQUIRED AIR
COMPLIANCE MONITORING**

Substance	Permit Level ^a (lb/MMBtu)	30-Day Weighted Rolling Average (lb/MMBtu)	Frequency of Monitoring	Averaging Approach	Monitoring Method	Duration of Monitoring ^b (years)
SO ₂	3.4 ^c	January 21-31 1991 (2.6) February 1991 (2.9) March 1991 (2.2) April 1991 (3.1)	Daily	30-day weighted rolling average	Analysis of a daily coal sample ^d	3
Opacity	20% ^e	January 21-31, 1991 (1.8) February 1991 (3.3) March 1991 (2.7) April 1991 (1.4)	Continuously	6-minute block average	In-situ opacity monitor	3
Particulate Loading	0.1	---	---	Average of 3 1-hour test runs	EPA Reference Method 5	3

^a lb/MMBtu = pounds of pollutant emitted per 10⁶ Btu actual heat input.

^b The air permit from Ohio EPA is granted for 3 years.

^c 30-day weighted, rolling average for entire plant, not just for boiler #13, Unit #4.

^d A daily composite sample of the coal to be burned each day is collected according to ASTM D2234. The sample is analyzed for ash, sulfur, and heat content according to ASTM D3174, D3177, and D3015, respectively. The SO₂ emission rate is calculated from this analysis for each day, and a 30-day rolling weighted average SO₂ emission rate is calculated for each day.

^e Up to 60 percent opacity is allowed for a duration of up to 6 minutes per hour. This regulation is applicable during start-up, once the flue gas temperature reaches 250 °F at the ESP inlet.

8.0 QUALITY ASSURANCE/QUALITY CONTROL RESULTS

All air quality monitoring data utilized in this report were collected by the DAS and are statistically summarized in Table 4-1 and in Appendix A. All sampling and analytical procedures, sample custody, calibration procedures, data reduction and validation, reporting procedures, internal quality control checks, performance and system audits, preventative maintenance, assessment of precision, accuracy and completeness, and corrective action are detailed in the LIMB Demonstration Extension Quality Assurance Project Plan, August 1990.

All NPDES water quality data for Outfall 601 utilized in Tables 4-2 and 4-3 and Appendix B are collected by Ohio Edison as a part of its permit requirements. QA/QC data for the pH, TSS, Flow, O&G, Total P, and As parameters are maintained by Ohio Edison personnel.

9.0 MONITORING PROBLEMS AND RECOMMENDATIONS FOR CHANGE

No monitoring equipment problems were encountered during the February, March and April 1991 reporting period. The data provided by the System 140 was extensively reviewed this reporting period to correct for highly repetitive data and erroneously high SO₂ and NO_x values (lb/MMBtu). The highly repetitive data points were identified when the boiler was taken off-line (a period of minimal heat production). The System 140 would lock on to the last value while the system was placed off-line and repeat this value until the boiler and System were placed back on-line. In addition, erroneously high SO₂ and NO_x concentration values were recorded when CEM equipment would automatically blow down to clear the intake lines. Both the repetitive and erroneously high values were removed from the data used to calculate the daily averages.

A wastewater monitoring equipment problem was encountered during this reporting period. The probe used by Radian personnel to monitor wastewater pH broke on March 17, 1991 and was replaced on March 22, 1991.

APPENDIX A

DETAILED DAILY AVERAGE AIR EMISSIONS

DETAILED DAILY AVERAGE AIR EMISSIONS DATA

Date	Coal Rate Klb/hr	HHV (1) Btu/lb	% S in Coal	Opacity %	SO ₂ Emissions		SO ₂ Removal %	NO _x Emissions	
					Bt/MMBtu	Bt/hr (2)		Bt/MMBtu	Bt/hr (2)
910201	69	12137	1.54	1.18	2.21	1845.62	23.54	0.40	334.76
910204	37	12509	1.67	0.14	2.08	960.70	30.64	0.36	166.03
910205	54	12524	2.08	0.05	2.53	1713.46	31.21	0.45	307.24
910206	62	12442	2.42	0.26	3.49	2694.48	22.75	0.45	346.56
910207	73	12526	2.92	3.26	3.75	3432.97	30.81	0.47	433.95
910214	48	12308	3.06	5.60	3.70	2191.41	34.26	0.45	267.11
910215	51	12073	2.95	6.52	4.32	2636.81	21.34	0.39	238.79
910216	40	12113	2.95	5.40	4.19	1996.95	21.45	0.42	199.59
910217	40	12356	2.97	5.44	4.35	2148.15	21.63	0.42	207.24
910218	39	12342	2.88	5.25	3.53	1682.14	21.17	0.41	193.87
910221	44	12427	3.02	2.56	3.31	1791.23	46.38	0.44	238.34
910222	47	12429	2.25	3.09	1.71	994.91	73.10	0.44	255.19
910226	54	12353	1.47	3.31	1.86	1233.11	30.62	0.38	249.20
910227	56	12526	1.28	3.13	1.89	1318.57	15.44	0.40	281.76
910228	54	12073	1.15	2.77	1.59	1036.84	23.30	0.39	251.39
Average	51	12343	2.31	3.20	2.97	1845.16	29.84	0.42	264.73
Maximum	73	12526	3.06	6.52	4.35	3432.97	73.10	0.47	433.95
Minimum	37	12073	1.15	0.05	1.59	960.70	15.44	0.36	166.03
910301	47	12555	1.07	2.42	1.50	876.59	23.44	0.35	207.21
910304	59	12356	1.19	3.70	1.92	1397.54	21.32	0.34	244.67
910305	59	12130	1.22	2.50	1.80	1286.11	22.94	0.37	263.81
910306	51	12328	1.21	2.40	1.99	1260.73	23.24	0.38	239.40
910307	61	12425	1.28	2.67	2.19	1668.45	29.20	0.40	304.33
910308	53	12580	1.47	2.77	2.67	1770.19	9.36	0.36	239.99
910311	50	12482	1.49	2.49	2.24	1396.43	19.69	0.38	239.63
910312	56	12334	1.47	2.80	2.11	1468.07	22.85	0.37	258.24
910313	52	12263	1.41	2.99	2.08	1330.89	29.12	0.37	234.55
910314	57	12490	1.36	2.74	2.59	1830.81	11.25	0.30	211.64
910315	43	12304	1.36	2.74	2.56	1356.16	23.58	0.30	159.92
910322	47	12341	1.59	2.00	2.58	1498.94	21.46	0.36	209.65
Average	53	12382	1.34	2.68	2.19	1428.41	21.45	0.36	234.42
Maximum	61	12580	1.59	3.70	2.67	1830.81	29.20	0.40	304.33
Minimum	43	12130	1.07	2.00	1.50	876.59	9.36	0.30	159.92

DETAILED DAILY AVERAGE AIR EMISSIONS DATA

Date	Coal Rate KLb/hr	HHV (1) Btu/lb	% S in Coal	Opacity %	SO ₂ Emissions		SO ₂ Removal %	NO _x Emissions	
					lb/MMBtu	lb/hr (2)		lb/MMBtu	lb/hr (2)
910401	41	12428	1.85	1.91	2.58	1300.59	22.56	0.36	181.28
910402	60	12620	1.59	1.62	2.46	1863.90	28.51	0.35	269.23
910403	66	12489	1.59	1.32	2.65	2187.32	35.25	0.38	311.15
910404	67	12592	1.61	0.85	2.57	2160.90	21.78	0.39	327.88
910405	52	12966	1.55	0.37	2.09	1408.27	25.67	0.40	268.02
910411	38	12342	2.46	2.84	3.17	1494.40	55.54	0.38	177.96
910412	50	12214	2.66	2.90	2.99	1826.55	37.71	0.42	253.96
910415	53	12234	2.58	2.66	3.15	2058.07	39.26	0.40	262.94
910416	46	12265	2.57	1.85	2.91	1653.09	36.36	0.40	225.05
910417	55	12075	2.92	1.11	3.42	2256.01	38.62	0.41	269.16
910418	70	12083	3.33	0.57	3.70	3123.06	41.54	0.43	363.96
910422	67	12222	3.58	1.04	3.56	2896.39	43.94	0.43	347.26
910423	52	12447	3.41	0.49	4.12	2661.97	33.40	0.42	273.33
910424	40	12535	3.43	0.44	3.61	1792.30	41.21	0.41	202.41
Average	54	12394	2.51	1.43	3.07	2048.77	35.81	0.40	266.69
Maximum	70	12966	3.58	2.90	4.12	3123.06	55.54	0.43	363.96
Minimum	38	12075	1.55	0.37	2.09	1300.59	21.78	0.35	177.96
LIMB Extension:									
Average	55	12388	2.02	2.31	2.77	1874.70	27.23	0.40	275.67
Baseline Period: February 17, 1990 through April 23, 1990									
Average	53	11680	1.30	1.30	1.40	932.00	NA	0.28	181.00

1. HHV = Higher Heating Value

2. These values calculated as: $\text{lbs/hr} = ((\text{lbs/mmBtu})(\text{Klb/hr}) * (\text{Btu/lb})(1000\text{lb/Klb}) / (10\text{E}6\text{Btu/mmBtu}))$

APPENDIX B

601 OUTFALL COMPLIANCE REPORTS

02/12/91 10:02 2216 384 8433

ENV & SPEC PROJ

0004

0510 M 9008 850926

4500

OHIO EPI

MONTHLY REPORT FORM

31800005601

JAN-91

1 1

2-5-91 OH005130

OHIO EDISON COMPANY

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON, OH 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

S. HILL

EDGEWATER

	3 1 PH S.U.	3 1 RESIDUE T. NFL MG/L GROSS	3 1 CONDUIT FLOW MGD	3 1 O&G FREN-G MG/L	3 1 ARSENIC AS, TOT UG/L
	00400	00530	50050	00536	01002
01			1.1		
02			1.1		
03	7.1	10	1.1		
04	7.4	10	1.5		
05			1.3		
06			1.1		
07			1.3		
08	7.0	20	1.3		
09			1.1		
10	7.2	4	1.1		
11			0.9		
12			1.1		
13			1.3		
14			1.1		
15	7.4	6	0.9		
16			1.1	2	53
17	7.6	8	1.1		
18			1.3		
19			1.5		
20			1.3		
21			1.3		
22			1.1		
23	7.2	29	0.7		
24	7.6	16	0.7		
25			0.9		
26			1.1		
27			1.1		
28			1.3		
29	7.5	11	1.3		
30	7.4	5	1.1		
31			1.1		
		119	35.3	2	53
		12	1.1	2	53
	7.6	29	1.5	2	53
	7.0	4	0.7	2	53

AILY REPORT FORM

HIO EDISON COMPANY

3IB00005601

FEB-91

1 1 3-5-91 OH0051306

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON, OH 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

EDGEWATER

S. HILL

	3 1 PH S.U.	3 1 RESIDUE MG/L GROSS	3 1 CONDUIT FLOW MGD	3 1 O&G FREN-G MG/L	
	00400	00530	50050	00556	AS
01			1.3		
02			1.3		
03			1.3		
04	7.4	4	1.3		
05			1.2		
06			1.2		
07	7.6	3	0.9		
08			1.1		
09			1.1		
10			0.9		
11			0.9		
12			1.1		
13	7.5	7	0.9	1	25
14			0.9		
15	7.5	8	0.7		
16			0.7		
17			0.9		
18			1.1		
19			1.1		
20			1.1		
21	8.5	6	1.1		
22	8.2	8	1.1		
23			0.9		
24			0.9		
25			1.1		
26	7.1	16	1.1		
27			1.1		
28			1.1		
29					
30					
31					
		52	29.4	1	
		7	1.1	1	
	8.5	16	1.3	1	
	7.1	3	0.7	1	

AGENCY

B-2

3-5-91

SC

Plant Superintendent

4500

HLY REPORT FORM

OHIO EDISON COMPANY

3IB00005601

MAR-91

1 2

OH0051306

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

EDGEWATER

S. HILL

	3 1	3 1	3 1	3 1	3 1	3 1	3 1	3 1	3 1	3 1
	PH S.U.	RESIDU T. NFL MG/L GROSS	CONDUIT FLOW MGD	O&G FREN-G MG/L	ARSENI TOT, RE UG/L	CYANID FREE MG/L	SELENI TOT, RE UG/L	BERYL BE, TOT UG/L	CAD CD, TOT UG/L	CHROM HEX-VA UG/L
	00400	00530	50050	00556	00978	00719	00981	01012	01027	0103
01	7.5	7	1.1							
02			1.1							
03			1.7							
04			1.6							
05			2.1							
06	8.0+8.2	10	2.6		43	0.005	AA	0.1	0.3	5
07	2 samples collected		2.1	AA	33					
08	7.5	16	1.6							
09			1.6							
10			1.6							
11			1.3							
12	7.4	8	1.3							
13			1.4							
14	7.4	14	1.6							
15			1.4							
16			1.7							
17			1.1							
18			1.1							
19			1.1							
20	7.4	6	0.8							
21			1.3							
22	7.6	24	1.8							
23			1.7							
24			0.9							
25			1.1							
26			1.6							
27	7.6	4	0.9							
28	7.7	5	1.6							
29			1.6							
30			1.6							
31			0.9							
		94	44.9	AA	76	0.005	AA	0.1	0.3	5
		10	1.4	AA	38	0.005	AA	0.1	0.3	5
	8.0	24	2.6	AA	43	0.005	AA	0.1	0.3	5
	7.4	4	0.8	AA	33	0.005	AA	0.1	0.3	5

AA: Below Detectable Limits

* Hex-Val Cr - < 5

Cyanide < 0.005

AGENCY

B-3

4-11-91

Plant Superintendent

4500

MONTHLY REPORT FORM

OHIO EDISON COMPANY

31B00005601

APR-91

1 1

OH00513C

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

EDGEWATER

S. HILL

	3 1 PH S.U.	3 1 RESIDU T. NFL MG/L GROSS	3 1 CONDUIT FLOW MGD	3 1 O&G FREN-G MG/L
	00400	00530	50050	00556
01			1.6	
02	7.3	41	1.6	
03			1.6	AA
04	7.3	6	1.6	
05			1.4	
06			1.3	
07			1.3	
08	7.7	2	0.7	
09			0.7	
10			1.1	
11	7.5	7	1.1	
12			1.1	
13			1.3	
14			1.1	
15			1.1	
16	8.1	24	1.4	
17			1.3	
18	7.8	4	1.3	
19			1.3	
20			1.5	
21			1.5	
22			1.5	
23	7.3	14	1.5	
24	7.4	6	1.3	
25			1.3	
26			1.1	
27			0.7	
28			0.7	
29			0.7	
30	7.0	11	0.7	
31				
		115	32.6	AA
		13	1.1	AA
	8.1	41	1.6	AA
	7.0	2	0.7	AA

AA: Below Detectable Limits

B-4

AGENCY

5-9-91

Plant Superintendent

APPENDIX C

601 OUTFALL DAILY pH AND SAMPLE LOG

DATE	LOAD (MWe)	COAL/SORBENT COMBINATION	INJECTION/ HUMIDIFICATION STATUS	Ca/S RATIO	pH	SAMPLES TAKEN Ca ASH	Temp °C	INITIALS
1-7-91		L/S, Limestone		7.24	✓	✓	9.6	1300
1-8-91		"		6.97	✓	✓	10.3	1400
1-9-91		"		7.12	✓		8.3	1400
1-10-91		"		7.26	✓	✓	7.5	1400
1-11-91		"		7.15	✓	✓	6.9	1400
1-14-91		"		7.13	✓	✓	5.8	1000
1-15-91		"		7.45	✓		10.1	1330
1-17-91		"		7.15	✓	✓	9.6	1400
1-18-91		"		7.07	✓	✓	5.8	1100
1-21-91				7.10	✓	✓	7.5	1300
1-22-91		L/S / Fine limestone		7.13	✓	✓	8.0	1400
1-23-91		"		7.07	✓	✓	7.2	1430
1-24-91		"		7.49	✓		4.8	1345
1-25		"		7.32	✓		6.9	1400
1-28-91		L/S Limestone		7.57	✓	✓	4.8	1430
1-29-91		"		7.62	✓	✓	5.7	1330
1-30-91		"		7.13	✓		6.2	1300
1-31-91		"		7.35	✓	✓	4.9	1400
2-1-91		"		7.47	✓		5.9	1400
2-4-91		L/S, Limestone		7.57	✓	✓	9.4	1400
2-5-91		H/S, Lime		7.64	✓		9.7	1400
2-6		H/S, Lime		7.72	✓		8.7	1400
2-7-91		"		7.58	✓	✓	8.9	1400
2-11-91		30% S, Dol. lime		7.43	✓	✓	4.8	1300
2-12-91		30% S, D lime		7.2	✓	✓	5.4	1500
2-13-91		"		7.36	✓	✓	6.8	1030
2-14		"		7.47	✓	✓	6.3	1300

Witnessed & Understood by me,

Date

C-1

Date

y

DATE	LOAD (MWe)	COAL/SORBENT COMBINATION	INJECTION/ HUMIDIFICATION STATUS	Ca/S RATIO	pH	SAMPLES TAKEN Ca ASH	Temp °C	INITIALS
2-18		3.0% S, Dlime			7.53	✓	9.6	1300
2-19-91		3.0% S, Dlime			7.72	✓	10.5	1300
2-20-91		3.0% S, Dlime			8.31	✓	6.0	1330
2-21-91		3.0% S, Dlime			8.37	✓	9.4	1400
2-22-91		4% S, Dlime			7.63	✓	8.5	1300 R
2-25-91		4% S, Ligno lime			7.92	✓	6.9	1400
2-26		"			7.55	✓	8.5	1400
2-27		"			7.21	✓	7.2	1400
2-28		"			7.53	✓	8.9	1400
3-6		"			7.81	✓	10.4	1100
3-7		"			7.94	✓	10.3	1100
3-8-91		"			7.90	✓	10.9	1400
3-11 To 3-17	NO	SAMPLES	COLLECTED	DUE	TO	BROKEN	PH	PROBE
4-1-91		4% S, Ligno lime			7.12	✓	14.3	1300 R
4-2					7.21	✓	15.7	1300 R
4-3					7.34	✓	16.2	1400 R
4-4					7.11	✓	18.4	1400 R
4-5					7.30	✓	18.5	1400 R
4-15-91		4% S, Ligno lime			7.79	✓	16.0	1400 R
4-16-91					7.87	✓	16.3	1400 R
4-17-91					7.22	✓	18.9	1500 R
4-18-91					7.29	✓	13.7	1500 R
4-22-91		3% S, Ligno lime			7.09	✓	13.1	1400 R
4-23-91		"			7.15	✓	15.8	1400 R
4-24-91		"			7.14	✓	16.1	1400 R
5-6-91		"			7.31	✓	17.5	1400 R
5-7-91		"			7.23	✓	17.9	1500 R

APPENDIX D

601 CALCIUM ANALYSIS

Radian Work Order 91-03-010

Analytical Report
03/26/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

Customer Work Identification LIMB PROJECT
Purchase Order Number 209-026-05-00

Contents:

- 1 Analytical Data Summary
- 2 Sample History
- 3 Comments Summary
- 4 Notes and Definitions

Radian Analytical Services
8501 Mo-Pac Boulevard
P. O. Box 201088
Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:



Babcock and Wilcox Co.
Radian Work Order: 91-03-010

Method/Analyte	Sample Identifications					
	LIMB-EMP601-23		LIMB-EMP601-24		LIMB-EMP601-25	
	01		02		03	
Matrix	water		water		water	
	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Calcium by ICPEs SW6010						
Calcium	54 mg/L	1.0	61 mg/L	1.0	56 mg/L	1.0
(1) For a detailed description of flags and technical terms in this report refer to the glossary.						

Babcock and Wilcox Co.
 Radian Work Order: 91-03-010

Method/Analyte		Sample Identifications	
	LIMB-EMP601-26		METHOD BLANK
	04		05
Matrix	water		water
	Result	Det. Limit	Result
Calcium by ICPEs SW6010			Det. Limit
Calcium	62 mg/L	1.0	ND mg/L 1.0
ND Not detected at specified detection limit			
(1) For a detailed description of flags and technical terms in this report refer to the glossary.			

Babcock and Wilcox Co.
 Radian Work Order: 91-03-010

Sample Identifications and Dates

Sample ID	LIMB-EMP601-23	LIMB-EMP601-24	LIMB-EMP601-25	LIMB-EMP601-26	METHOD BLANK
Date Sampled	02/28/91	02/28/91	02/28/91	02/28/91	
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix	water	water	water	water	water
	01	02	03	04	05

Calcium by ICPE SW6010

Prepared	03/04/91	03/04/91	03/04/91	03/04/91	03/04/91
Analyzed	03/06/91	03/06/91	03/06/91	03/06/91	03/06/91
Analyst	DRW	DRW	DRW	DRW	DRW
File ID					
Blank ID					
Instrument	JA61	JA61	JA61	JA61	JA61
Report as	received	received	received	received	received

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-03-010

ND ALL METHODS EXCEPT CLP

This flag is used to denote analytes which are not detected at or above the specified detection limit.

EXPLANATION

The value to the right of the < symbol is the method specified detection limit for the analyte.

Babcock and Wilcox Co.
Radian Work Order: 91-03-010

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

Radian Work Order 91-04-180

Analytical Report
05/01/91

Babcock and Wilcox Co.
Radian RTP NC
Luke Contos

Customer Work Identification Edgewater Purchase Order Number 209-026-05-00


Contents:	
1	Analytical Data Summary
2	Sample History
3	Comments Summary
4	Notes and Definitions

Radian Analytical Services
8501 Mo-Pac Boulevard
P. O. Box 201088
Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:



Babcock and Wilcox Co.
Radian Work Order: 91-04-180

Method/Analyte	Sample Identifications					
	LIMB-EMP601-27		LIMB-EMP601-28		LIMB-EMP601-29	
	01		02		03	
Matrix	Water		Water		Water	
	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Calcium by ICPEs SW6010						
Calcium	47	mg/L 1.0	56	mg/L 1.0	47	mg/L 1.0
(1) For a detailed description of flags and technical terms in this report refer to the glossary.						

Babcock and Wilcox Co.
Radian Work Order: 91-04-180

Method/Analyte		Sample Identifications	
METHOD BLANK			
Matrix	04 water		
Calcium by ICPES SW6010	Result	Det. Limit	
Calcium	ND	mg/L 1.0	
<p>ND Not detected at specified detection limit</p> <p>(1) For a detailed description of flags and technical terms in this report refer to the glossary.</p>			

Babcock and Wilcox Co.
 Radian Work Order: 91-04-180

Sample Identifications and Dates

Sample ID LIMB-EMP601-27 LIMB-EMP601-28 LIMB-EMP601-29 METHOD BLANK

Date Sampled	04/15/91	04/15/91	04/15/91	
Date Received	04/17/91	04/17/91	04/17/91	04/17/91
Matrix	water	water	water	water
	01	02	03	04

Calcium by ICPE SW6010

Prepared	04/23/91	04/23/91	04/23/91	04/23/91
Analyzed	04/25/91	04/25/91	04/25/91	04/25/91
Analyst	DES	DES	DES	DES
File ID				
Blank ID				
Instrument	JA61	JA61	JA61	JA61
Report as	received	received	received	received

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-04-180

ND ALL METHODS EXCEPT CLP

This flag is used to denote analytes which are not detected at or above the specified detection limit.

EXPLANATION

The value to the right of the < symbol is the method specified detection limit for the analyte.

Babcock and Wilcox Co.
Radian Work Order: 91-04-180

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX E

TCLP AND ASTM DI LEACHATE PROCEDURES

LEACHATE METHODS SUMMARY

Three leachate testing procedures have been discussed as viable methods to assess the chemical characteristics of leachate generated from LIMB fly ash. These three are the EP Toxicity Test, Toxicity Characteristic Leaching Procedures (TCLP), and a DI Water "shake extraction" leaching procedure. All three methods are included in this appendix and the following paragraphs generally summarize these methods as they pertain to a dry (ash) sample.

The EP Toxicity test uses a leaching procedure as follows:

- 1) extract ash with 16 times its weight in DI water;
- 2) adjust pH to 5.0 if above and do not adjust if below;
- 3) agitate for 24 hrs at approximate room temperature maintaining the pH at 5.0;
- 4) add approximately 4 times the weight of the ash of DI water; and
- 5) filter and analyze for As, Ba, Cd, Cr, Pb, Hg, Se, Ag, and various non-volatile organics.

The TCLP procedure incorporates the following general steps:

- 1) preliminary extraction
 - perform a percent solids analysis;
 - perform particle size reduction if necessary;
 - determine which of the 2 extraction fluids to use;
 - if volatiles are to be analyzed, use extract fluid #1
 - if pH of DI extract is less than 5, use extract fluid #1
 - if pH less than 5 with DI extract add small amount of acid, heat and if still less than 5, use extract fluid #1
 - otherwise use extraction fluid #2
 - extraction fluid #1 - Acetic Acid, NaOH, and water (pH \approx 4.9);

- extraction fluid #2 - Acetic acid and water (pH = 2.9);
- add 20 times the weight of the solids in the aliquot (use special digestion vessel when looking for volatiles);
- add extraction fluid and extract for 18 hours at 30 rpm (ambient temperatures); and
- filter the slurry, measure pH, and analyze the extract for metals and organics as per SW846 methods.

The DI water leveling procedure basically is completed as follows:

- perform solids analysis;
- add 700 grams aliquot of ash plus 6 times the aliquot weight in DI water;
- invert 25 times/minute for 3 minutes;
- agitate 48 hours on a piece of equipment that is equivalent to a "reciprocating platform shaker;"
- open, let settle for 5 minutes, separate solids by decanting, centrifuge, or filtering;
- filtering further by vacuum or pressure; and
- measure pH and analyze.

§ 261.33 Discarded commercial chemical products, off-specification species, container residues and spill residues thereof.

The following materials or items are hazardous wastes if and when they are discarded or intended to be discarded unless they are excluded under §§ 260.20 and 260.22 and listed in Appendix IX.

(f) . . .

Hazardous waste No.	Substance
U242.....	Pentachlorophenol.
U242.....	Phenol, pentachloro-
U212.....	Phenol, 2,3,4,6-tetrachloro-
U212.....	Phenol, 2,4,6-trichloro-
U230.....	Phenol, 2,4,6-trichloro-
U231.....	Propionic acid, 2-(2,4,6-trichlorophenyl)-
U233.....	Silver.
U232.....	2,4,5-T.
U212.....	2,3,4,6-Tetrachlorophenol.
U230.....	2,4,6-Trichlorophenol.
U231.....	2,4,6-Trichlorophenol.
U230.....	2,4,5-Trichlorophenoxyacetic acid.

APPENDIX I—REPRESENTATIVE SAMPLING METHODS

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the sampling protocols listed below, for sampling waste with properties similar to the indicated materials, will be considered by the Agency to be representative of the waste.

Extremely viscous liquid—ASTM Standard D140-70 Crushed or powdered material—ASTM Standard D346-73 Soil or rock-like material—ASTM Standard D420-69 Soil-like material—ASTM Standard D1452-65 Fly Ash-like material—ASTM Standard D2234-76 (ASTM Standards are available from ASTM, 1916 Race St., Philadelphia, PA 19103)

Containerized liquid wastes—"COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical

Methods," U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C. 20460. (Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair St., Cincinnati, Ohio 45268) Liquid waste in pits, ponds, lagoons, and similar reservoirs—"Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods,"

This manual also contains additional information on application of these protocols.

APPENDIX II—EP TOXICITY TEST PROCEDURES

A. Extraction Procedure (EP)

1. A representative sample of the waste to be tested (minimum size 100 grams) shall be obtained using the methods specified in Appendix I or any other method capable of yielding a representative sample within the meaning of Part 260. [For detailed guidance on conducting the various aspects of the EP see "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (incorporated by reference, see § 260.11).]

2. The sample shall be separated into its component liquid and solid phases using the method described in "Separation Procedure" below. If the solid residue^a obtained using this method totals less than 0.5% of the original weight of the waste, the residue can be discarded and the operator shall treat the liquid phase as the extract and proceed immediately to Step 3.

3. The solid material obtained from the Separation Procedure shall be evaluated for its particle size. If the solid material has a surface area per gram of material equal to, or greater than, 3.1 cm² or passes through a 9.5 mm (0.375 inch) standard sieve, the operator shall proceed to Step 4. If the surface area is smaller or the particle size larger than specified above, the solid material shall be prepared for extraction by crushing, cutting or grinding the material so that

^aThese methods are also described in "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA 600/2-80-018, January 1980.

^bThe percent solids is determined by drying the filter pad at 80°C until it reaches constant weight and then calculating the percent solids using the following equation:

Percent solids =

$$\frac{(\text{weight of pad} + \text{solids}) - (\text{tare weight of pad})}{\text{initial weight of sample}} \times 100$$

It passes through a 9.5 mm (0.375 inch) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described below.

4. The solid material obtained in Step 3 shall be weighed and placed in an extractor with 16 times its weight of deionized water. Do not allow the material to dry prior to weighing. For purposes of this test, an acceptable extractor is one which will impart sufficient agitation to the mixture to not only prevent stratification of the sample and extraction fluid but also insure that all sample surfaces are continuously brought into contact with well mixed extraction fluid.

5. After the solid material and deionized water are placed in the extractor, the operator shall begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 8.0, the pH of the solution shall be decreased to 8.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 8.0, no acetic acid should be added. The pH of the solution shall be monitored, as described below, during the course of the extraction and if the pH rises above 8.2, 0.5N acetic acid shall be added to bring the pH down to 8.0 ± 0.2 . However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture shall be agitated for 24 hours and maintained at 20°-40°C (68°-104°F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5N acetic acid. If such a system is not available, the following manual procedure shall be employed:

(a) A pH meter shall be calibrated in accordance with the manufacturer's specifications.

(b) The pH of the solution shall be checked and, if necessary, 0.5N acetic acid shall be manually added to the extractor until the pH reaches 8.0 ± 0.2 . The pH of the solution shall be adjusted at 15, 30 and 60 minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5N pH units.

(c) The adjustment procedure shall be continued for at least 6 hours.

(d) If at the end of the 24-hour extraction period, the pH of the solution is not below 8.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH shall be adjusted to 8.0 ± 0.2 and the extraction continued for an additional four hours, during which the pH shall be adjusted at one hour intervals.

6. At the end of the 24 hour extraction period, deionized water shall be added to

the extractor in an amount determined by the following equation:

$$V = (20 \times W) - 16(W) - A$$

V = ml deionized water to be added

W = weight in grams of solid charged to extractor

A = ml of 0.5N acetic acid added during extraction

7. The material in the extractor shall be separated into its component liquid and solid phases as described under "Separation Procedure."

8. The liquids resulting from Steps 2 and 7 shall be combined. This combined liquid (or the waste itself if it has less than 1/4 percent solids, as noted in step 2) is the extract and shall be analyzed for the presence of any of the contaminants specified in Table I of § 261.24 using the Analytical Procedures designated below.

Separation Procedure

Equipment: A filter holder, designed for filtration media having a nominal pore size of 0.45 micrometers and capable of applying a 5.3 kg/cm² (75 psi) hydrostatic pressure to the solution being filtered, shall be used. For mixtures containing nonabsorptive solids, where separation can be effected without imposing a 5.3 kg/cm² pressure differential, vacuum filters employing a 0.45 micrometers filter media can be used. (For further guidance on filtration equipment or procedures see "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" incorporated by reference, see § 260.11). Procedure:

(1) Following manufacturer's directions, the filter unit shall be assembled with a filter bed consisting of a 0.45 micrometer filter membrane. For difficult or slow to filter mixtures a prefilter bed consisting of the following prefilters in increasing pore size (0.65 micrometer membrane, fine glass

"This procedure is intended to result in separation of the "free" liquid portion of the waste from any solid matter having a particle size >0.45 μm. If the sample will not filter, various other separation techniques can be used to aid in the filtration. As described above, pressure filtration is employed to speed up the filtration process. This does not alter the nature of the separation. If liquid does not separate during filtration, the waste can be centrifuged. If separation occurs during centrifugation, the liquid portion (centrifugate) is filtered through the 0.45 μm filter prior to becoming mixed with the liquid portion of the waste obtained from the initial filtration. Any material that will not pass through the filter after centrifugation is considered a solid and is extracted.

(fiber prefilter, and coarse glass fiber prefilter) can be used.

(ii) The waste shall be poured into the filtration unit.

(iii) The reservoir shall be slowly pressurized until liquid begins to flow from the filtrate outlet at which point the pressure in the filter shall be immediately lowered to 10-15 psig. Filtration shall be continued until liquid flow ceases.

(iv) The pressure shall be increased stepwise in 10 psi increments to 75 psig and filtration continued until flow ceases or the pressurizing gas begins to exit from the filtrate outlet.

(v) The filter unit shall be depressurized, the solid material removed and weighed and then transferred to the extraction apparatus, or, in the case of final filtration prior to analysis, discarded. Do not allow the material retained on the filter pad to dry prior to weighing.

(vi) The liquid phase shall be stored at 4°C for subsequent use in Step 8.

B. Structural Integrity Procedure

Equipment: A Structural Integrity Tester having a 3.18 cm (1.25 in.) diameter hammer weighing 0.33 kg (0.73 lbs.) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 1.

Procedure

1. The sample holder shall be filled with the material to be tested. If the sample of waste is a large monolithic block, a portion shall be cut from the block having the dimensions of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder. For a fixated waste, samples may be cast in the form of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder for purposes of conducting this test. In such cases, the waste may be allowed to cure for 30 days prior to further testing.

2. The sample holder shall be placed into the Structural Integrity Tester, then the hammer shall be raised to its maximum height and dropped. This shall be repeated fifteen times.

3. The material shall be removed from the sample holder, weighed, and transferred to the extraction apparatus for extraction.

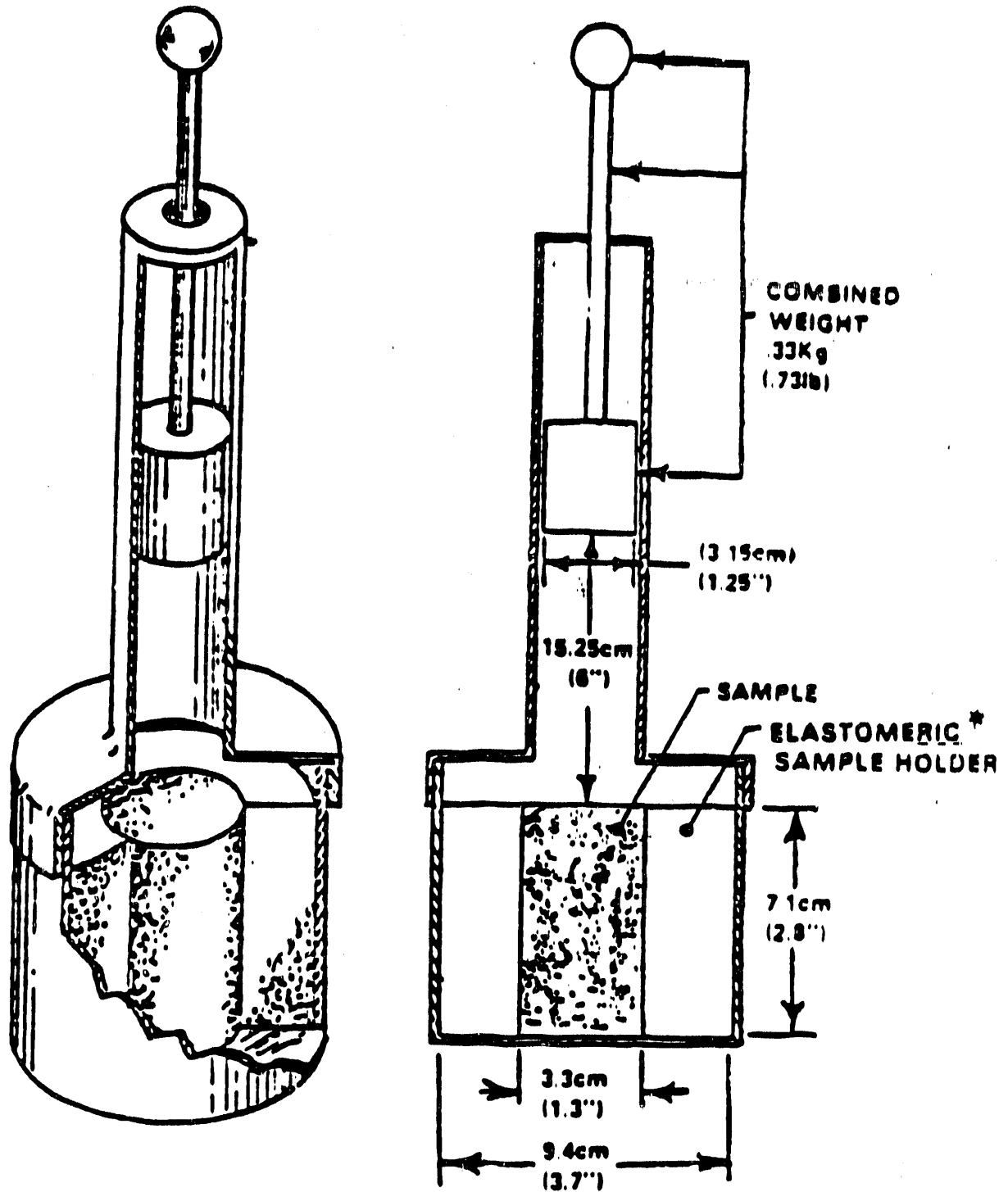
Analytical Procedures for Analyzing Extract Contaminants

The test methods for analyzing the extract are as follows:

1. For arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver, endrin, lindane, methoxychlor, toxaphene, 2,4-D[2,4-dichlorophenoxyacetic acid] or 2,4,5-TP [2,4,5-trichlorophenoxypropionic acid]: "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (incorporated by reference, see § 260.11).

2. [Reserved]

For all analyses, the methods of standard addition shall be used for quantification of species concentration.



*ELASTOMERIC SAMPLE HOLDER FABRICATED OF MATERIAL FIRM ENOUGH TO SUPPORT THE SAMPLE

Figure 1
COMPACTION TESTER

[45 FR 33119, May 19, 1980, as amended at 46 FR 35247, July 7, 1981]



Standard Test Method for SHAKE EXTRACTION OF SOLID WASTE WITH WATER¹

This standard is issued under the fixed designation D 3987; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers a procedure for leaching of solid waste to obtain an aqueous solution to be used to determine the materials leached under the specified testing conditions.

1.2 It provides for the shaking of a known weight of waste with water of specified composition and the separation of the aqueous phase for analysis.

2. Applicable Documents

2.1 ASTM Standards:

D 75 Practice for Sampling Aggregates²

D 420 Recommended Practice for Investigating and Sampling of Soil and Rock for Engineering Purposes³

D 1129 Definitions of Terms Relating to Water⁴

D 1193 Specification for Reagent Water⁵

D 1888 Test Methods for Particulate and Dissolved Matter in Water⁶

D 2216 Method for Laboratory Determination of Moisture Content of Soils⁷

D 2777 Practice for Determination of Precision and Bias of Methods of Committee D-19 on Water⁸

D 2234 Method for Collection of a Gross Sample of Coal⁹

D 3370 Practices for Sampling Water¹⁰

E 122 Recommended Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process¹¹

3. Significance and Use

3.1 This method is intended as a rapid means for obtaining an extract of solid waste. The extract may be used to estimate the release of certain constituents of the solid waste under

the laboratory conditions described in this procedure.

3.2 This method is not intended to provide an extract that is representative of the actual leachate produced from a solid waste in the field or to produce extracts to be used as the sole basis of engineering design.

3.3 This method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions.

3.4 It is intended that the final pH of the extract reflect the interaction of the extractant with the buffering capacity of the solid waste.

3.5 It is intended that the water extraction simulate conditions where the solid waste is the dominant factor in determining the pH of the extract.

3.6 The method produces an extract that is amenable to the determination of both major and minor constituents. When minor constituents are being determined, it is especially important that precautions are taken in sample storage and handling to avoid possible contamination of the samples.

3.7 This method has been tested to determine its applicability to certain inorganic components in the solid waste (see Appendix X1). The method has not been tested for applicabil-

¹ This method is under the jurisdiction of Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D 34.02 on Extraction and Leachate Testing.

Current edition approved March 26, 1981. Published June 1981.

² *Annual Book of ASTM Standards*, Vol 04.08.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.05.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

ity to organic substances and volatile matter (see 5.3).

3.8 The agitation technique and rate and the liquid-to-solid ratio specified in the procedure may not be suitable for extracting all types of solid waste. (See discussion in Appendix X2.)

4. Definitions

4.1 For definitions of terms used in this method, see Definitions D 1129.

5. Apparatus

5.1 *Agitation Equipment*—Agitation equipment of any type that will produce constant movement of the aqueous phase equivalent to that of a reciprocating platform shaker operated at 60 to 70 1-in. (25-mm) cycles per minute without incorporation of air is suitable. A cycle shall be understood to include one forward and one equal return movement. Equipment used shall be designed for continuous operation without heating the samples being agitated (see discussion of agitation in Appendix X2).

5.2 *Membrane Filter Assembly*—A borosilicate glass or stainless steel funnel with a flat, fritted base of the same material and membrane filters.

5.3 *Containers*—Round, wide-mouth bottles of composition suitable to the nature of the solid waste and the analyses to be performed, and constructed of materials that will not allow sorption of constituents of interest. One-gallon (or 4-L) bottles should be used with 700-g samples and ½-gal (or 2-L) bottles with 350-g samples. Multiples of these sizes may be used for larger samples. These sizes were selected to establish suitable geometry and provide that the sample plus liquid would occupy approximately 80 to 90 % of the container. Bottles must have a watertight closure. Containers for samples where gases may be released should be provided with a venting mechanism. (Note that the venting of the container has the potential to affect the concentration of volatile extracts in the extract.) Containers should be cleaned in a manner consistent with the analyses to be performed.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the

American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water at 18 to 27°C (Specification D 1193).

7. Sampling

7.1 Obtain a representative sample of the solid waste to be tested using ASTM sample methods developed for the specific industry where available.

7.2 Where no specific methods are available, sampling methodology for materials of similar physical form shall be used.

7.3 A minimum sample of 5000 g shall be sent to the laboratory (see Method E 122).

7.4 It is important that the sample of the solid waste be representative with respect to surface area, as variations in surface area would directly affect the leaching characteristics of the sample. Solid waste samples should contain a representative distribution of particles sizes.

7.5 Keep samples in closed containers appropriate to the sample type prior to the extraction in order to prevent sample contamination or constituent loss. Where it is desired to extract biologically or chemically active samples in their existing state, store the samples at 4°C (Practices D 3370) and start the extraction within 8 h. Where it is desired to extract such samples in a state representative of the results of biological or chemical activities, the samples may be specifically handled to simulate such activities. Record the storage conditions and handling procedures in the report.

8. Sample Preparation

8.1 For free-flowing particulate solid wastes, obtain a sample of the approximate size required in the test by quartering the sample (Section 7) received for testing on an impermeable sheet of glazed paper, oil cloth, or other

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopoeia."

flexible material as follows:

8.1.1 Empty the sample container into the center of the sheet.

8.1.2 Flatten out the sample gently with a suitable straightedge until it is spread uniformly to a depth at least twice the maximum particle diameter particle size.

8.1.3 Remix the sample by lifting a corner of the sheet and drawing it across, low down, to the opposite corner in a manner that the material is made to roll over and over and does not merely slide along. Continue operation with each corner, proceeding in a clockwise direction. Repeat this operation ten times.

8.1.4 Lift all four corners of the sheet towards the center and holding all four corners together, raise the entire sheet into the air to form a pocket for the sample.

8.1.5 Repeat Step 8.1.2.

8.1.6 With a straightedge at least as long as the flattened mound of sample (such as a thin-edged yard stick), gently divide the sample into quarters. An effort should be made to avoid using pressure on the straightedge sufficient to cause damage to the particles.

8.1.7 Discard alternate quarters.

8.1.8 If further reduction of sample size is necessary, repeat Steps 8.1.3 through 8.1.7. A minimum sample size of 350 g is recommended for each extraction. Additional samples should be provided for determination of solids content. If smaller samples are used in the test, report this fact.

8.2 For field-cored solid wastes or castings produced in the laboratory, cut a representative section weighing approximately 350 or 700 g for testing, plus samples for determination of solids content. Shape the sample so that the leaching solution will cover the material to be tested.

8.3 For fluid solid wastes, mix thoroughly in a manner that does not incorporate air to assure uniformity before withdrawing a 350 or 700-g sample for test. Take samples for determination of solids content at the same time as the test sample.

9. Procedure

9.1 Record the physical description of the sample to be tested including particle size so far as it is known.

9.2 *Solids Content*—Determine the solids content of separate portions of the sample as

follows:

9.2.1 Dry to constant weight two dishes or pans of size suitable to the solid waste being tested at $104 \pm 2^\circ\text{C}$. Cool in a desiccator and weigh. Record the value to ± 0.1 g.

9.2.2 Put an appropriately sized portion of sample of the solid waste to be tested into each pan. Scale the weight used to the physical form of the solid waste tested. Use a minimum of 50 g but use larger samples where particles larger than 10-mm in average diameter are being tested. Weigh. Record the weight to ± 0.1 g.

9.2.3 Dry 16 to 20 h at $104 \pm 2^\circ\text{C}$. Certain solid wastes, such as scrubber sludges, may contain compounds that are subject to calcination at the specified drying temperature. Dry these compounds at lower temperatures. For example, gypsum may be successfully dried at 45°C (Method C 471) and $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ wastes at 85°C . Record the actual temperature and time of the drying period.

9.2.4 Cool to room temperature in a desiccator and reweigh. Record the weight to ± 0.1 g.

9.3 *Shake Procedure*—Weigh or tare the container to be used in the shake test to the nearest or within 1 g.

9.4 Add the container approximately 700 g of solid waste (Section 8) and determine and record the weight of sample used to 1 g. If weights other than 700 g are used, note in the report.

9.5 Add to the container a volume of test water (6.2) equal in millilitres to four times the weight in grams of the sample used in 9.4. See discussion of dilution ratio in Appendix X2.

9.6 Close the container. Invert the container approximately 25 times per minute for 3 min. Place the container upright on the agitation equipment.

9.7 Agitate continuously for $48 \text{ h} \pm 0.5 \text{ h}$ at 18 to 27°C .

9.8 Open the container. Observe and record any physical changes in the sample and leaching solution.

9.9 Shake the container to mix the entire sample thoroughly. Let the sample settle for 5 min; then separate the bulk of the aqueous phase from solid phase by decantation, centrifugation, or filtration through filter paper as appropriate. Then vacuum or pressure filter the liquid through a $0.45\text{-}\mu\text{m}$ filter. If these separation means result in prolonged filtering time,

a 8- μ m filter or other device may be used. Record any such deviations in the report.

9.10 The filtrate obtained in 9.9 is the extract mentioned elsewhere in this method. Measure the pH of the extract immediately, then preserve the extract in a manner consistent with the chemical analysis or biological testing procedures to be performed (Practices D 3370). If sufficient liquid phase is not available for the analyses, so indicate in the report and do not continue the procedure; or alternatively, perform the extraction procedure on additional samples of the solid waste to obtain sufficient liquid phase. Where phase separation occurs during the storage of the extract, appropriate mixing should be used to ensure the homogeneity of the extract prior to its use in such analysis or testing.

9.11 Analyze the extract for specific constituents or properties or use the extract for biological testing procedures as desired using appropriate ASTM standard methods. Where no appropriate ASTM methods exist, other methods may be used and recorded in the report.

10. Calculation

10.1 Calculate the solids content of the individual samples from the data obtained in 9.2 as follows:

$$S = A/B$$

where:

A = weight in grams of sample after drying,
g = original weight in grams of sample, and
S = solid content, g/g.

Average the two values obtained. Record as the solids content.

11. Report

11.1 The report shall include the following:

11.1.1 Source of the solid waste, date of

sampling, and sample preservation used.

11.1.2 Description of the solid waste including physical characteristics and particle size, if known (9.1),

11.1.3 Solids content (9.2),

11.1.4 Sample weight if other than 700 g,

11.1.5 Drying time and temperature if other than 16 to 20 h at $104 \pm 2^\circ\text{C}$,

11.1.6 pH and results of specific analyses calculated in appropriate units. State analytical procedures used, and filter used if other than 0.45 μ m,

11.1.7 Observation of changes in test material or leaching solution recorded in 9.8.

11.1.8 Date leach testing started, preservation used for extract, and date of analysis.

12. Precision and Accuracy

12.1 No information is presently available as to the precision or accuracy of the analysis of specific constituents in the extract. It is recommended that users of this test validate the applicability of their chosen methods of detection by spiking portions of the extract, before using these methods for the analysis of the extract.

12.2 Based on a collaborative series of tests on six solid wastes including fly ash, scrubber sludge, API separator sludge, metal finishing waste, textile waste, and soil, the precision of iron and calcium determinations for these specific solid wastes was measured. Information on the test program is provided in Appendix X1.

12.3 The precision of this method may vary depending on the solid waste being tested and on the element being extracted.

12.4 Determination of the accuracy of this method is not possible, as no standard reference material exists.

APPENDIXES

XI. COLLABORATIVE TEST PROGRAM

X1.1 Based on a collaborative series of tests on six solid wastes including fly ash, scrubber sludge, API separator sludge, metal finishing waste, textile waste, and soil, the precision of this method for these specific materials, including variability of the extraction test and the analytical procedure, may be expressed as shown below. Twenty one laboratories participated in the collaborative test program, and each of the six solid wastes was tested by at least five of the laboratories, with a single operator performing three extraction replicates.¹ The collaborative test program was conducted with both an unclear definition of whether a stroke constituted forward-return movement (see 5.1) and without the inversion instruction (see 9.6). It has not been determined how this contributed to the observed deviation.

X1.1.1 For calcium in concentrations ranging between 2.8 and 220 mg/L:

$$S_r = 0.311X + 9.26$$

$$S_o = 0.192X - 1.59$$

where:

S_r = overall precision,

S_o = single-operator precision, and

X = determined concentration of Ca, mg/L

X1.1.2 For iron, in concentrations ranging from 0.06 and 1.4 mg/L:

$$S_r = 0.792X - 0.013$$

$$S_o = 0.543X - 0.023$$

where:

S_r = overall precision,

S_o = single-operator precision, and

X = determined concentration of Fe, mg/L

¹ The collaborative data are on file at ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103 and may be obtained on loan by requesting RR: D 19-1000

X2. AGITATION TECHNIQUE AND RATE, AND LIQUID/SOLID RATIOS

X2.1 While the major effort relative to development of the test method has been undertaken at the agitation rate and liquid/solid ratios specified in the method, it is recognized that these variables may significantly influence the results on certain solid wastes, and that they may not be adequate for certain solid wastes.

X2.1.1 The possible effects of varying the agitation technique and rate include degree of mixing, rate of release of constituents, and particle abrasion ef-

fects. The precision of the method may also be influenced.

X2.1.2 The possible effects of varying the dilution ratio include degree of mixing, rate of release of constituents (and possible concentration effects, depending on availability), and particle abrasion effects.

X2.2 The agitation techniques and ratio and dilution ratio used by other proposed extraction methods differ from those used in this method.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.

APPENDIX I—REPRESENTATIVE SAMPLING METHODS

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the sampling protocols listed below, for sampling waste with properties similar to the indicated materials, will be considered by the Agency to be representative of the waste.

Extremely viscous liquid—ASTM Standard D140-70 Crushed or powdered material—ASTM Standard D346-75 Soil or rock-like material—ASTM Standard D420-69 Soil-like material—ASTM Standard D1452-65

Fly Ash-like material—ASTM Standard D2234-76 [ASTM Standards are available from ASTM, 1916 Race St., Philadelphia, PA 19103]

Containerized liquid wastes—"COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical

Methods," U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C. 20460. [Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair St., Cincinnati, Ohio 45268]

Liquid waste in pits, ponds, lagoons, and similar reservoirs—"Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods,"

This manual also contains additional information on application of these protocols.

Appendix II—Method 1311 Toxicity Characteristic Leaching Procedure (TCLP)

[Revised by 55 FR 11862, March 29, 1990]

1.0 Scope and Application

1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphase wastes.

1.2 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high levels that even after accounting for dilution from the other fractions of the extract the concentration would be above the regulatory threshold for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

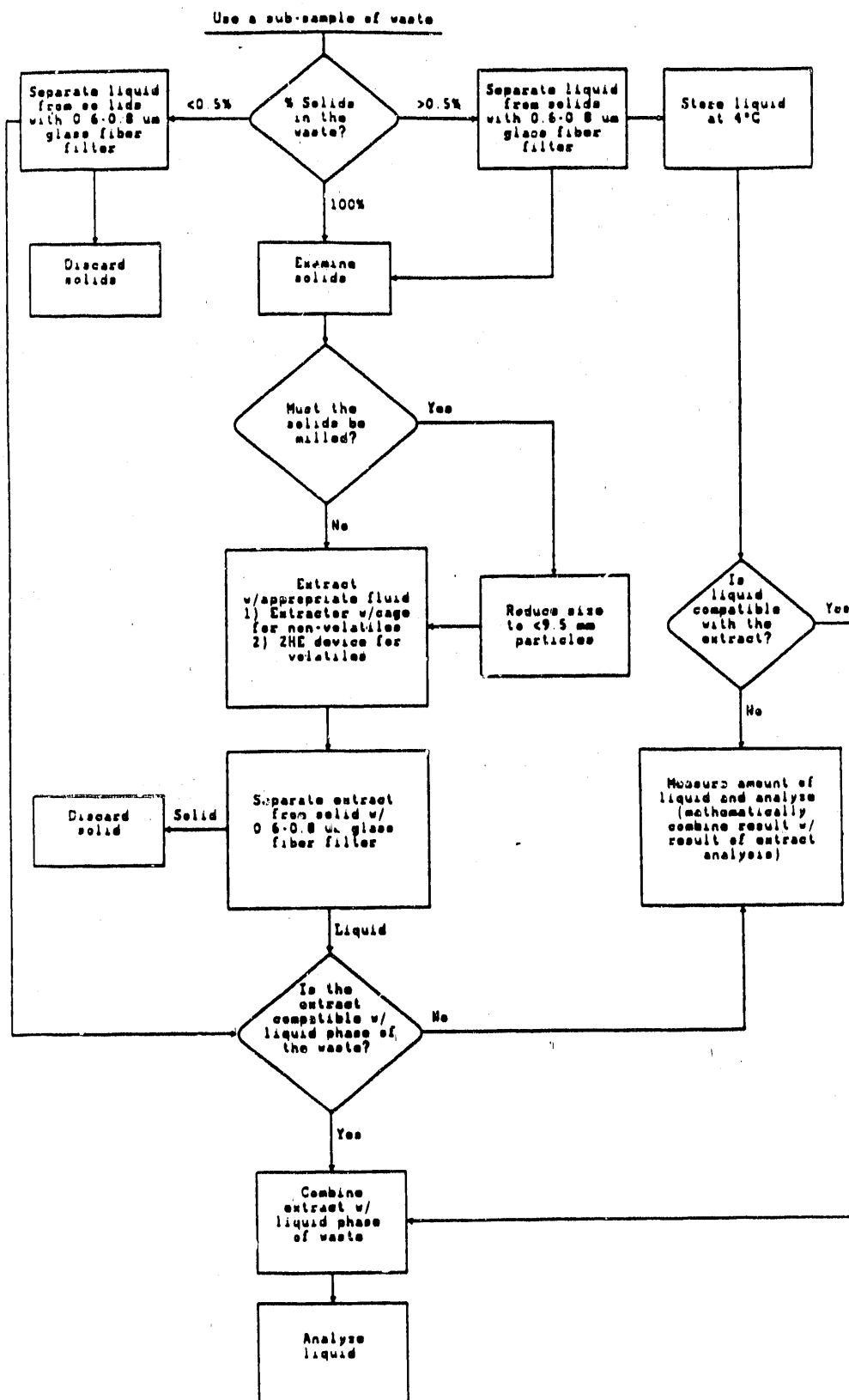
1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile contaminant exceeds the regulatory threshold for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory threshold.

2.0 Summary of Method (see Figure 1)

2.1 For liquid wastes (i.e., those containing less than 0.5 percent dry solid material), the waste, after filtration through a 0.6 to 0.8-um glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the solid phase, if necessary, is reduced in particle size. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile contaminants (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8-um glass fiber filter.

Figure 1 Method 1311 Flowchart



[Appendix II]

TABLE 1.—VOLATILE CONTAMINANTS¹

Compound	CAS no.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methyl ethyl chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichloroethane	75-80-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4

TABLE 1.—VOLATILE CONTAMINANTS¹—
Continued

Compound	CAS no.
Xylene	1330-20-7

¹ When testing for any or all of these contaminants, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 Apparatus and Materials

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel¹ in an end-over-end fashion (see Figure 2) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessel:

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile constituents (i.e., those listed in Table 1). The ZHE (depicted in Figure 3) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see step 4.3.1). The vessels shall have an internal volume of 500–600 mL and be equipped to accommodate a 80–110 mm filter. The devices contain VITON[®] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

¹ VITON[®] is a trademark of Du Pont.

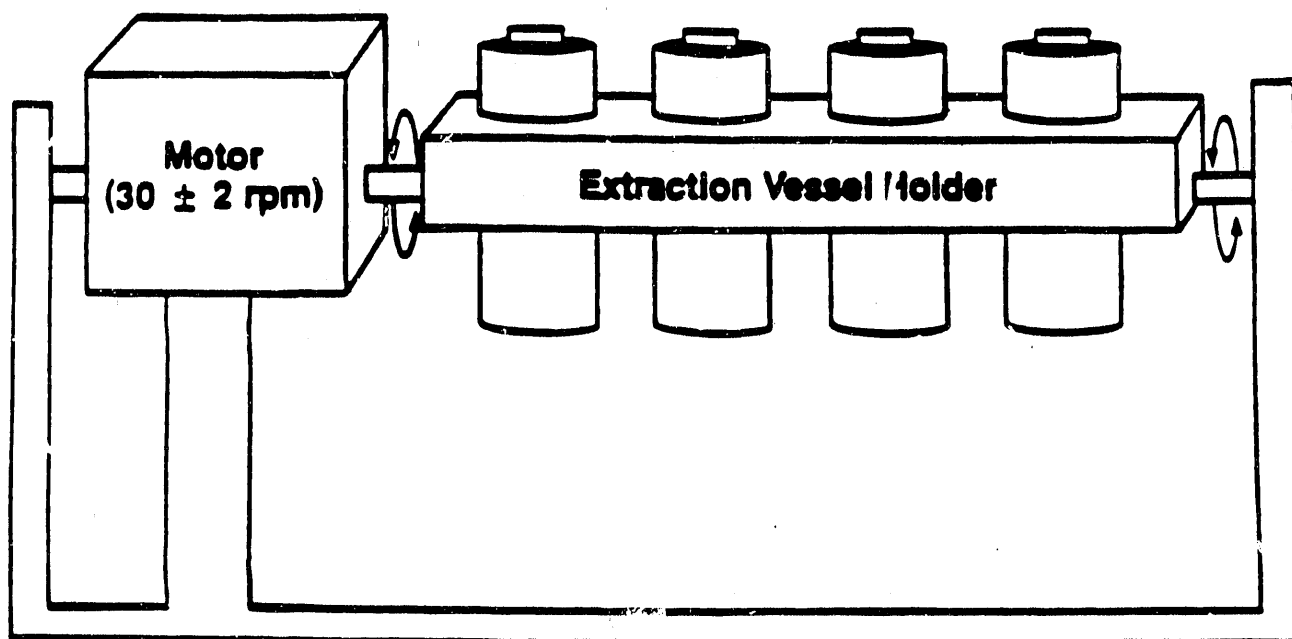


Figure 2. Rotary Agitation Apparatus

[Appendix II]

TABLE 2.—SUITABLE ROTARY AGITATION APPARATUS ¹

Company	Location	Model no.
Analytical Testing and Consulting Services, Inc.....	Warrington, PA (215) 343-4480..	2-ZHE or 4-bottle extractor (DC20S); 4-ZHE or 6-bottle extractor (DC20); 6-ZHE or 12-bottle extractor (DC20S).
Associated Design and Manufacturing Company.....	Alexandria, VA (703) 549-5999..	2-vessel (3740-2), 4-vessel (3740-4), 6-vessel (3740-6), 8-vessel (3740-8), 12-vessel (3740-12), 24-vessel (3740-24).
Environmental Machine and Design, Inc.....	Lynchburg, VA (804) 845-8424..	8-vessel (08-00-00), 4-vessel (04-00-00).
IRA Machine Shop and Laboratory.....	Santurce, PR (809) 752-4004.....	8-vessel (011001).
Lars Lande Manufacturing.....	Whitmore Lake, MI (313) 449-4116.	10-vessel (10VRE), 5-vessel (5 VRE).
Millipore Corp.....	Bedford, MA (800) 225-3384.....	4-ZHE or 4 1-liter bottle extractor (YT300RAHW).

¹ Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

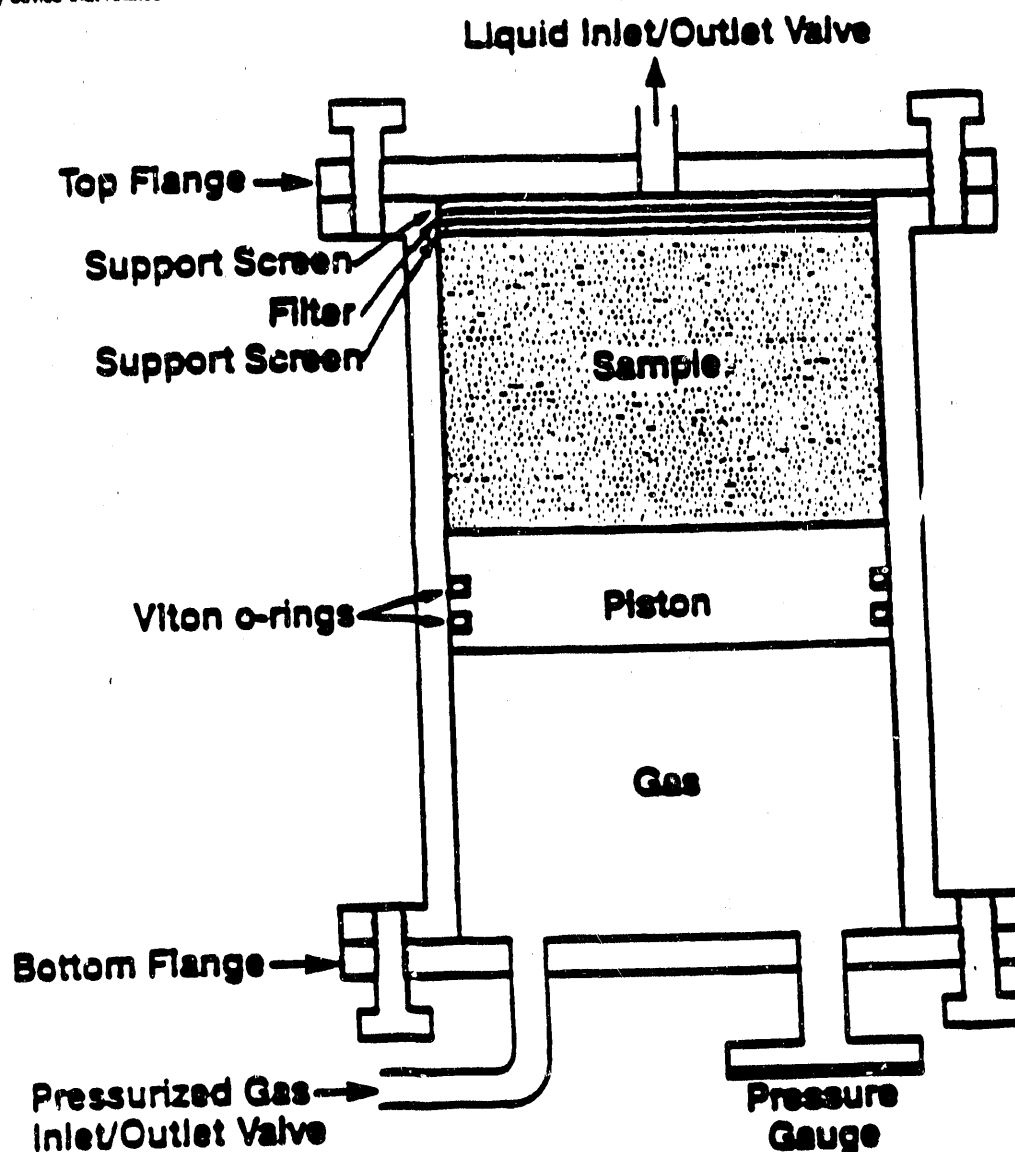


Figure 3. Zero-Headspace Extractor (ZHE)

[Appendix II]

TABLE 3.—SUITABLE ZERO-HEADSPACE EXTRACTOR VESSELS ¹

Company	Location	Model no.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device.
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device.
Lars Lande Manufacturing ²	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device.
Millipore Corporation	Bedford, MA (800) 225-3384	YT3009OHW, Gas Pressure Device.
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Gas Pressure Device.

¹ Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

² This device uses a 110 mm filter.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see section 9.0) refers to pounds-per-square-inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the

extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the contaminants to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

Note: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile compounds, any filter holder capable of supporting a glass

fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Step 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater and equipped to accommodate a 142 mm diameter filter are recommended). Vacuum filtration can only be used for wastes with low solids content (< 10 percent) and for highly granular liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are constituents of concern.

TABLE 4.—SUITABLE FILTER HOLDERS ¹

Company	Location	Model/Catalogue no.	Size (um)
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

¹ Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern. The 142 mm size filter holder is recommended.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8-um or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-

filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1-L per rinse is recommended). Glass fiber

filters are fragile and should be handled with care.

4.5 pH meters: The meter should be accurate to +0.05 units at 25 °C.

[Appendix II]

TABLE 5.—SUITABLE FILTER MEDIA¹

Company	Location	Model	Pore size
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 483-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-8010	GF75	0.7

¹ Any filter that meets the specifications in Section 4.4 of the Method is suitable.

4.6 ZHE extract collection devices: TEDLAR[®] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1 percent of total waste), the TEDLAR[®] bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1 percent of total waste), the syringe or the TEDLAR[®] bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100 percent solid) or has no significant solid phase (is 100 percent liquid), either the TEDLAR[®] bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE extraction fluid transfer devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas-tight syringe, pressure filtration unit (See Step 4.3.2), or other ZHE device).

4.8 Laboratory balance: Any laboratory balance accurate to within +0.01 grams may be used (all weight measurements are to be within +0.1 grams).

5.0 Reagents

5.1 Reagent water. Reagent water is defined as water in which an interferent is not observed at or above the methods detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.1.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.1.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 99 ± 5 °C, bubble a contaminant-free inert gas (e.g., nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.2 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.3 Nitric acid (1N), HNO₃, made from ACS reagent grade.

5.4 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.5 Glacial acetic acid, HOAc, ACS reagent grade.

5.6 Extraction fluid.

5.6.1 Extraction fluid #1: Add 5.7 mL glacial HOAc to 500 mL of the appropriate water (See Step 5.1), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.99 ± 0.05.

5.6.2 Extraction fluid #2: Dilute 5.7 mL glacial HOAc with ASTM Type II water (See Step 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.98 ± 0.05.

Note: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.7 Analytical standards prepared according to the appropriate analytical method.

6.0 Sample Collection, Preservation, and Handling

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample depending upon the physical state or states of the waste and the contaminants of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile contaminant extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile contaminants, care shall be taken to minimize the loss of volatiles. Samples shall be taken and stored in a manner to prevent the loss of volatile contaminants (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C, until ready to be opened prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic contaminant determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see section 8.14 if precipitation occurs). Extracts or portions of extracts for organic contaminant determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See section 10.0 (QA requirements) for acceptable sample and extract holding times.

7.0 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; (3) determination of whether the solid portion of the waste requires particle size reduction; and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste.

7.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Step 7.3.

7.1.2 If the sample is liquid or multiphase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.1.3 through 7.1.9.

7.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

² TEDLAR[®] is a registered trademark of Du Pont.

7.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1 percent of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.5 to

determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10-psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.1.5 or 7.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.9)}}{\text{Total weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

7.2 If the percent solids determined in Step 7.1.9 is equal to or greater than 0.5%, then proceed either to Step 7.3 to determine whether the solid material requires particle size reduction or to Step 7.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Step 7.1.9 is less than 0.5%, then proceed to Step 8.9 if the

nonvolatile TCLP is to be performed and to Section 9.0 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within +1 percent. Record the final weight.

Note: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Weight of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

7.2.4 If the percent dry solids is less than 0.5 percent, then proceed to Step 8.9 if the nonvolatile TCLP is to be performed, and to Section 9.0 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.0) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Step 7.3) and determine the appropriate extraction fluid (Step 7.4). If only the volatile TCLP is to be performed, see the note in Step 7.4.

7.3 Determination of whether the waste requires particle-size reduction (particle-size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle-size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm², or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding

the waste to a surface area or particle-size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken, see Step 8.6.

Note: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5 percent and if TCLP extraction for nonvolatile constituents will take place (Section 8.0), perform the determination of the appropriate fluid (Step 5.8) to use for the nonvolatiles extraction as follows:

Note: TCLP extraction for volatile constituents uses only extraction fluid #1 (Step 5.8.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 9.0.

7.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle-size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500-mL beaker or Erlenmeyer flask.

7.4.2 Add 96.5 mL of reagent water (ASTM Type II) to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 8.0.

7.4.3 If the pH from Step 7.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 8.0.

7.5 If the aliquot of the waste used for the preliminary evaluation (Steps 7.1-7.4) was determined to be 100% solid at Step 7.1.1, then it can be used for the Section 8.0 extraction (assuming at least 100 grams

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remain), and the section 9.0 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Step 7.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 9.0. The aliquot of the waste subjected to the procedure in Step 7.1.7 might be appropriate for use for the section 8.0 extraction if an adequate amount of solid (as determined by Step 7.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 8.10 of the nonvolatile TCLP extraction.

8.0 Procedure When Volatiles Are Not Involved

A minimum sample size of 100 grams (solid and liquid phases) is required. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Step 7.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

8.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100 percent solid, see Step 7.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Step 8.9.

8.2 If the sample is liquid or multiphase, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 8.3 to 8.8.

8.3 Pre-weigh the container that will receive the filtrate.

8.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Step 4.4).

Note: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

8.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5 percent dry solids (Step 7.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5 percent dry solids (Step 7.1 or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

8.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

8.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Step 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1 percent of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 8.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased

at 50 psi (i.e., filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Step 8.12) or stored at 4 °C until time of analysis.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 8.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

8.9 If the waste contains <0.5 percent dry solids (see Step 7.2), proceed to Step 8.13. If the waste contains >0.5 percent dry solids (see Step 7.1 or 7.2), and if particle-size reduction of the solid was needed in Step 7.3, proceed to Step 8.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Step 8.11.

8.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Step 7.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

Note: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

8.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Step 7.1)} \times \text{weight of waste filtered (Step 8.5 or 8.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Step 7.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 22 ± 3 °C during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., lined or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

8.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by

filtering through a new glass fiber filter, as outlined in Step 8.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

8.13 Prepare the TCLP extract as follows:

8.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. Proceed to Step 8.14.

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8.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 8.12 with the initial liquid phase of the waste obtained in Step 8.7. This combined liquid is defined as the TCLP extract. Proceed to Step 8.14.

8.13.3 If the initial liquid phase of the waste, as obtained from Step 8.7, is not or may not be compatible with the filtered liquid resulting from Step 8.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Step 8.14.

8.14 Following collection of the TCLP extract, the pH of the extract should be

recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH < 2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic contaminants. If an analysis of the

undigested extract shows that the concentration of any regulated metallic contaminant exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to +0.5 percent), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).

C_1 = The concentration of the contaminant of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the contaminant of concern in the second phase (mg/L).

8.15 Compare the contaminant concentrations in the TCLP extract with the thresholds identified in the appropriate regulations. Refer to § 10.0 for quality assurance requirements.

9.0 Procedure When Volatiles Are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500-mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 p due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase).

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

9.1 Pre-weigh the (evacuated) filtrate collection container (See Step 4.6) and set aside. If using a TEDLAR[®] bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Step 4.6 are recommended for use under the conditions stated in 4.6.1-4.6.3.

9.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to loosen the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 9.0, Step 7.1 and/or 7.2). Secure the gas inlet/

outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

9.3 If the waste is 100 percent solid (see Step 7.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Step 9.5.

9.4 If the waste contains <0.5 percent dry solids (Step 7.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >0.5 percent dry solids (Steps 7.1 and/or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

9.4.1 For wastes containing <0.5 percent solids (see Step 7.1), weigh out a 500-gram subsample of waste and record the weight.

9.4.2 For wastes containing >0.5 percent solids (see Step 7.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{Percent solids (Step 7.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

9.5 If particle-size reduction of the solid portion of the waste was required in Step 7.3, proceed to Step 8.6. If particle-size reduction was not required in Step 7.3, proceed to Step 9.7.

9.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle-size as described in Step 7.3.1. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle-size reduction. The means used to

affect particle-size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

Note: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle-size has been appropriately altered, proceed to Step 9.7.

9.7 Waste slurry need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

9.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (see

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inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 9.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before

filtering. If the waste is 100 percent solid (see Step 7.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Step 9.12.

9.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

9.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5 percent dry solids (see Step 7.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Step 9.15.

9.11 The liquid phase may now be either analyzed immediately (See Steps 9.13 through 9.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Step 7.1)} \times \text{weight of waste filtered (Step 9.4 or 9.8)}}{100}$$

9.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Step 5.6).

9.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

9.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This

bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

9.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 22 ± 3 °C during agitation.

9.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Step 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR[®] bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough

volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Step 9.9. All extract shall be filtered and collected if the TEDLAR[®] bag is used. If the extract is multiphase, or if the waste contained an initial liquid phase (see Steps 4.6 and 9.1).

Note: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

9.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from step 9.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 9.13 and the initial liquid phase (Step 9.9) are collectively defined as the TCLP extract.

9.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

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where:

- V_1 = The volume of the first phase (l).
 C_1 = The concentration of the contaminant of concern in the first phase (mg/l).
 V_2 = The volume of the second phase (l).
 C_2 = The concentration of the contaminant of concern in the second phase (mg/l).

9.16 Compare the contaminant concentrations in the TCLP extract with the thresholds identified in the appropriate regulations. Refer to section 10.0 for quality assurance requirements.

10.0 Quality Assurance Requirements

10.1 Maintain all data, including quality assurance data, and keep it available for reference or inspection.

10.2 A minimum of one blank (extraction fluid ≈ 1) for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring.

10.3 A matrix spike shall be performed for each waste unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. If more than one sample of the same waste is being tested, a matrix spike needs to be performed for every twenty samples and the average percent recovery applied to the waste characterization.

10.3.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

10.3.2 Matrix spike levels should be made at the appropriate regulatory threshold limits. However, if the extract contaminant concentration is less than one half the threshold limit, the spike level may be one half the contaminant concentration but not less than the quantitation limit or a fifth of the threshold limit.

10.3.3 The purpose of the matrix spike is to monitor the adequacy of the analytical

methods used on the TCLP extract and to determine whether matrix interferences exist in analyte detection. If the matrix spike recoveries are less than 50%, then the analytical methods are not performing adequately or use of the methods is inadequate. Use of internal calibration quantitation methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the contaminant concentration in the TCLP extract.

10.3.4 Use of internal quantitation methods is also required when the contaminant concentration is within 20% of the regulatory level. (See section 10.5 concerning the use of internal calibration methods.)

10.3.5 Matrix spike recoveries are calculated by the following formula:

$$\text{Percent recovery} = \frac{A-B}{C} \times 100\%$$

where A = the concentration of the spiked sample.

B = the concentration of the unspiked sample, and

C = the spike level

10.4 All quality control measures described in the appropriate analytical methods shall be followed.

10.5 The use of internal calibration quantitation methods shall be employed for a contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

10.5.1 The method of standard additions shall be employed as the internal calibration

quantitation method for each metallic contaminant.

10.5.1.1 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrumental technique. All four aliquots are analyzed.

10.5.1.2 Prepare a plot, or subject data to linear regression, of instrumental signals or external-calibration-derived concentrations as the dependent variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

10.5.1.3 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject data to linear regression of the corrected instrumental signals or external-calibration-derived concentrations as the dependent variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

10.6 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES

(Days)

	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles.....	14	NA	14	28
Semi-volatiles.....	7	7	40	54
Mercury.....	28	NA	28	56
Metals, except mercury.....	180	NA	180	360

NA = Not applicable.

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is

not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not

invalidate characterization if the waste exceeds the regulatory level.

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Standard Additions

and particularly the higher absorption values are more closely spaced. This presentation closely imitates the logarithmic response of the atomic absorption spectrophotometer as it appears on the strip-chart of a recorder. Concentration values obtained between 60% and 70% absorption are considerably more difficult to resolve than those between 10% to 20% or 30% absorption.

Any of these calibration curves can be used for the purpose of estimating calcium in serum because only a small segment of each curve is needed to contain the entire range of the normal values, as well as most abnormally low or high concentrations. This segment of the curve is shown in expanded form in figure 6-11. The concentration scale is in apparent units—equivalent to 50 times the true concentration—in order to present a direct result. The response of the instrument is given in % absorption as obtained on the strip-chart recorder. Note that the calibration function is nearly a straight line between concentrations of 5 and 12.5 $\mu\text{g}/100\text{ ml}$. Because of this property, the unknown concentration may be entered into the working curve as % absorption rather than absorbance, thereby eliminating the need to transpose absorption into absorbance. (This transposition is cumbersome, and should be avoided when analytical conditions permit.) Results of comparable accuracy and precision are obtained by using either direct % absorption readings on a linear plot (figure 6-9, curve A), or % absorption plotted against concentration on a log-log scale (figure 6-10), or transposing % absorption to absorbance (figure 6-9, curve B).

Similar working curves and the same general approach also apply to the determination of Na, K, and Mg in serum, urine and other fluids with predictable and relatively narrow concentration ranges.

METHOD OF STANDARD ADDITIONS

The determination of an unknown concentration by the method of standard additions is used in several instrumental procedures including atomic absorption spectroscopy. In clinical chemistry, this method has limited application and does not usually serve as a basis for routine analysis. Its chief usefulness is in those determinations where appropriate comparative standard solutions are not readily available, or where more accurate methods have not been worked out or are unknown to the analyst. The principal advantage of this method is that, within certain concentration limits, it is self-

compensatory for various interferences which may not be measurable and need not be known or corrected for.

*The standard additions technique is an extrapolative measurement based on two assumptions:

(1) That the working curve is linear or predictable and remains so within the concentration range of the analysis;

(2) That a known amount of a metal added to the specimen will behave, in a spectrochemical sense, in a manner similar to the naturally bound metal in the native tissue.

Both assumptions may be correct only in relation to certain elements, in near-aqueous or aqueous solutions, and within relatively narrow concentration limits.

The Procedure:

(a) Estimate (or test) the concentration of the element under analysis to determine the approximate instrument range at which the analysis should be performed. (You must know whether to dilute, concentrate, or analyze the specimen "as-is" in its native state.)

(b) From the original specimen withdraw at least 2 (and preferably 3) equal aliquots, and transfer these to volumetric flasks of the same volume. The flasks should be only partially filled.

(c) To aliquot #1, add water to volume.

(d) To aliquot #2, add about 25% of the estimated amount of the element under analysis (step a).

(e) To aliquot #3, add about 50% of the amount originally estimated (step a). Additional aliquots may be used, each containing an increasingly larger quantity of the element under analysis. Be sure the final volume or weight of all the aliquots is the same.

(f) Regardless of the sample preparation method used prior to analysis (dilution, concentration, extraction, etc.), treat all aliquots of the specimen in exactly the same manner.

(g) Aspirate the original specimen (aliquot #1) and the additional aliquots, each containing a known added amount, and record the absorption signal. Convert % absorption to absorbance

(h) The concentration of the unknown can be calculated or it can be determined graphically as shown in figure 6-12. This working curve is based on linear proportionality between concentration and absorbance. Therefore, the concentration of the unknown (x) is proportional to its absorbance A_x

as the concentration of the unknown (x) plus the added amount (a_1) is proportional to its absorbance, A_1 .

Or (equation 1) $X = \frac{a_1 A_x}{A_1 - A_x}$

Note: a_1 is known; A_x and A_1 are measured.

(i) When more than a single "added amount" is used, the additional values may serve to verify the assumption that the working curve is indeed linear. In accordance with equation 1, the calculated concentration of the unknown (x) should be the same regardless *which* aliquots (#2, #3, or others) are used in its determination.

(j) The graphic presentation (figure 6-12) is frequently more convenient in verifying the linearity of the working curve. Note that the concentration of the unknown is determined at the intercept of the zero-absorbance axis and a line drawn through points A_x , A_1 , A_2 , etc. The units of concentration used in measuring the added values also establish the concentration of the unknown.

(k) For best results, the added quantities should be fairly close to the true concentration. Additions of from 1/4 to twice the originally estimated amount are suitable for most clinical analyses, within the general limitations of this method.

Problem:

Determine an unknown concentration of calcium in an aqueous solution. (**Note:** the values obtained below represent the actual readings in this experiment.)

Step 1

With the AA instrument programmed for calcium, the unknown specimen was aspirated. The resulting signal of about 13% absorption was estimated (from the typical calcium calibration curve in figure 6-1) to represent a concentration of over 2 $\mu\text{g/ml}$ but no more than 5 $\mu\text{g/ml}$ (say 3 $\mu\text{g/ml}$). Aliquots of 5 ml of this unknown solution were added to each of four 10-ml volumetric flasks.

Step 2

(a) To aliquot #1, water was added to volume, resulting in a 1:2 dilution.

(b) To aliquot #2, 1 ml of a 2 $\mu\text{g/ml}$ aqueous calcium standard was added. The volumetric flask was filled to volume with water. (Added amount: $a_1 = 0.2 \mu\text{g/ml}$ of Ca)

(c) To aliquot #3, 2 ml of a 2 $\mu\text{g/ml}$ Ca standard

were added and made up to volume. (Added amount: $a_2 = 0.4 \mu\text{g/ml}$)

(d) To aliquot #4, 5 ml of a 2 $\mu\text{g/ml}$ Ca standard were added. This filled the volumetric flask to volume. (Added amount: $a_3 = 1.0 \mu\text{g/ml}$)

Step 3

All 4 aliquots were aspirated, and the % absorption measured and transposed to absorbance. The data was plotted as shown in figure 6-13. The following results were obtained:

Aliquot no.	Amount added, a ($\mu\text{g/ml}$)	Absorbance A
1	$a_0 = 0$	$A_x = 0.065$
2	$a_1 = 0.2$	$A_1 = 0.076$
3	$a_2 = 0.4$	$A_2 = 0.087$
4	$a_3 = 1.0$	$A_3 = 0.115$

Step 4

Concentration was determined from equation 1.

$$X = \frac{0.2 \times 0.065}{0.076 - 0.065} = 1.18$$

This value is multiplied by 2 because of the previous 1:2 dilution (step 2).

$$1.18 \times 2 = 2.36 \mu\text{g/ml.}$$

Step 5

From figure 6-13, the concentration of the unknown was obtained graphically by connecting points A_x , A_1 , A_2 , A_3 , through the intercept of the zero absorbance line. The value thus obtained was $1.22 \times 2 = 2.44 \mu\text{g/ml}$. Note that point A_3 fell below the straight line that connected A_x , A_1 , and A_2 .

Step 6

The unknown concentration was also calculated on the basis of each "added amount" separately as follows:

$a_2 A_2$, added amount 0.4 $\mu\text{g/ml}$.

$$(\text{eq. 1}) X = \frac{0.4 \times 0.065}{0.087 - 0.065} = 1.18; 1.18 \times 2 =$$

$$2.36 \mu\text{g/ml (answer)}$$

$a_3 A_3$, added amount 1.0 $\mu\text{g/ml}$.

$$(\text{eq. 1}) X = \frac{1.0 \times 0.065}{0.115 - 0.065} = 1.3; 1.3 \times 2 =$$

$$2.6 \mu\text{g/ml (answer)}$$

The results obtained on the basis of the last value (a_3, A_3) differed from the previous value by about +10%. This confirmed that the calibration curve could *not* be extrapolated to point A_3 which departed from the expected linear proportionality between concentration and absorbance.

The concentration of the unknown was found to be 2.40 ppm \pm SD (standard deviation) 0.008 ppm on the basis of triplicate analysis by conventional interpolation between the closely spaced standard solutions of 2.0 and 3.0 ppm.

The method of standard additions is prone to error because of possible inaccurate reading of % absorption or absorbance - much more so than the conventional interpolative techniques. For example, assume that the absorbance at Point A_x was read as 0.063 instead of 0.065.

The difference between these readings is equivalent to about 2 to 3 times the thickness of the stylus line of the recorder. The error that would result had the concentration of the unknown been obtained by conventional methods would be about 3%. However, in the method of standard additions, the error would be far greater, since the proportionality factors which apply to equation 1 (arithmetically or graphically) are very sensitive to A_x , the absorbance of the unknown. To illustrate: if A_x was read as 0.63 and a single added value of 0.2 ppm (a_1, A_1) used in computing the unknown, then:

$$(eq. 1) \quad X = \frac{0.2 \times 0.063}{0.076 - 0.063} = 0.97 \text{ ppm}$$

$$0.97 \times 2 = 1.94 \text{ ppm}$$

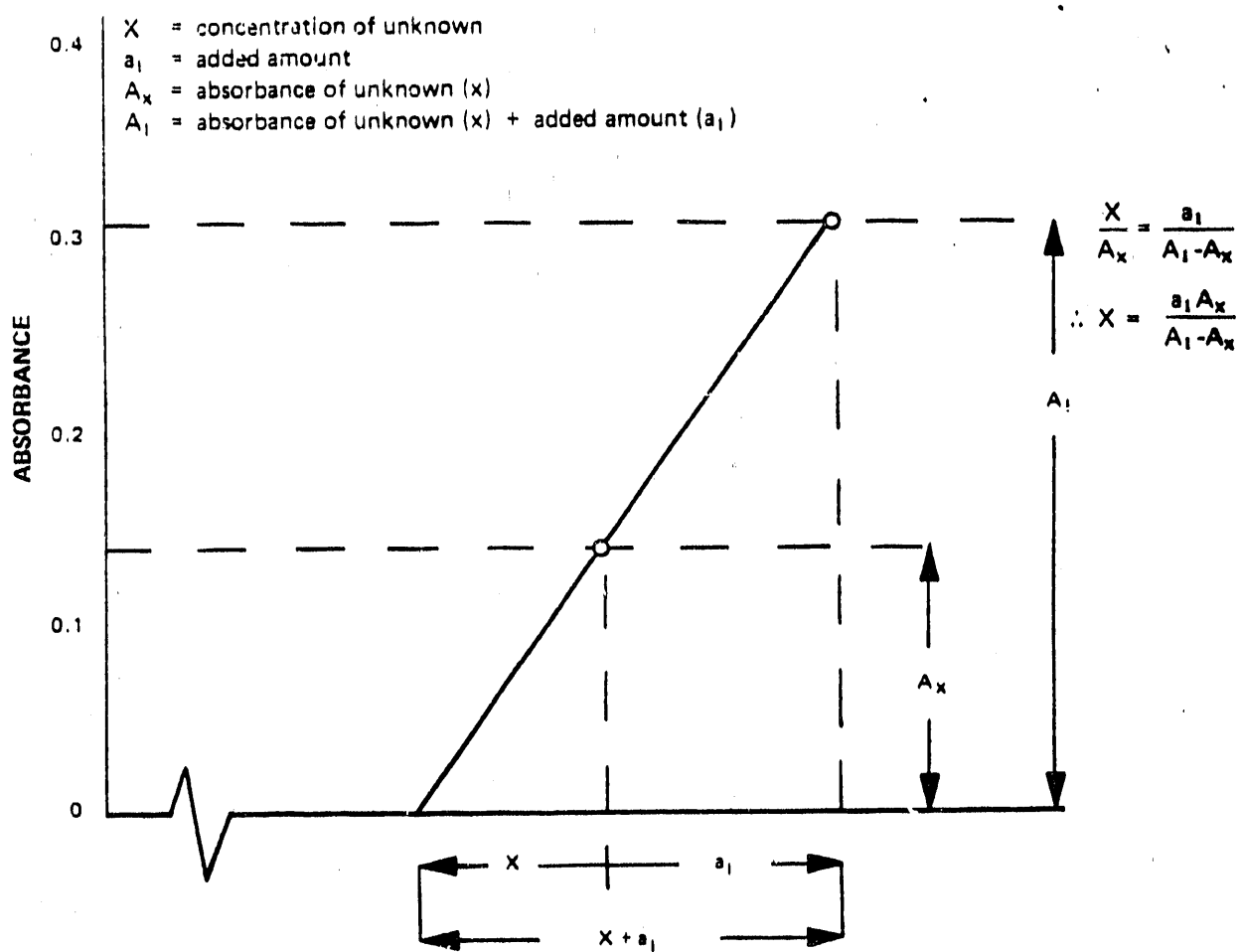
The difference between this and the "true" value is:

$$2.40 - 1.94 = 0.46 \text{ ppm or } 19\% (!).$$

Points A_2 and A_3 would yield similar errors.

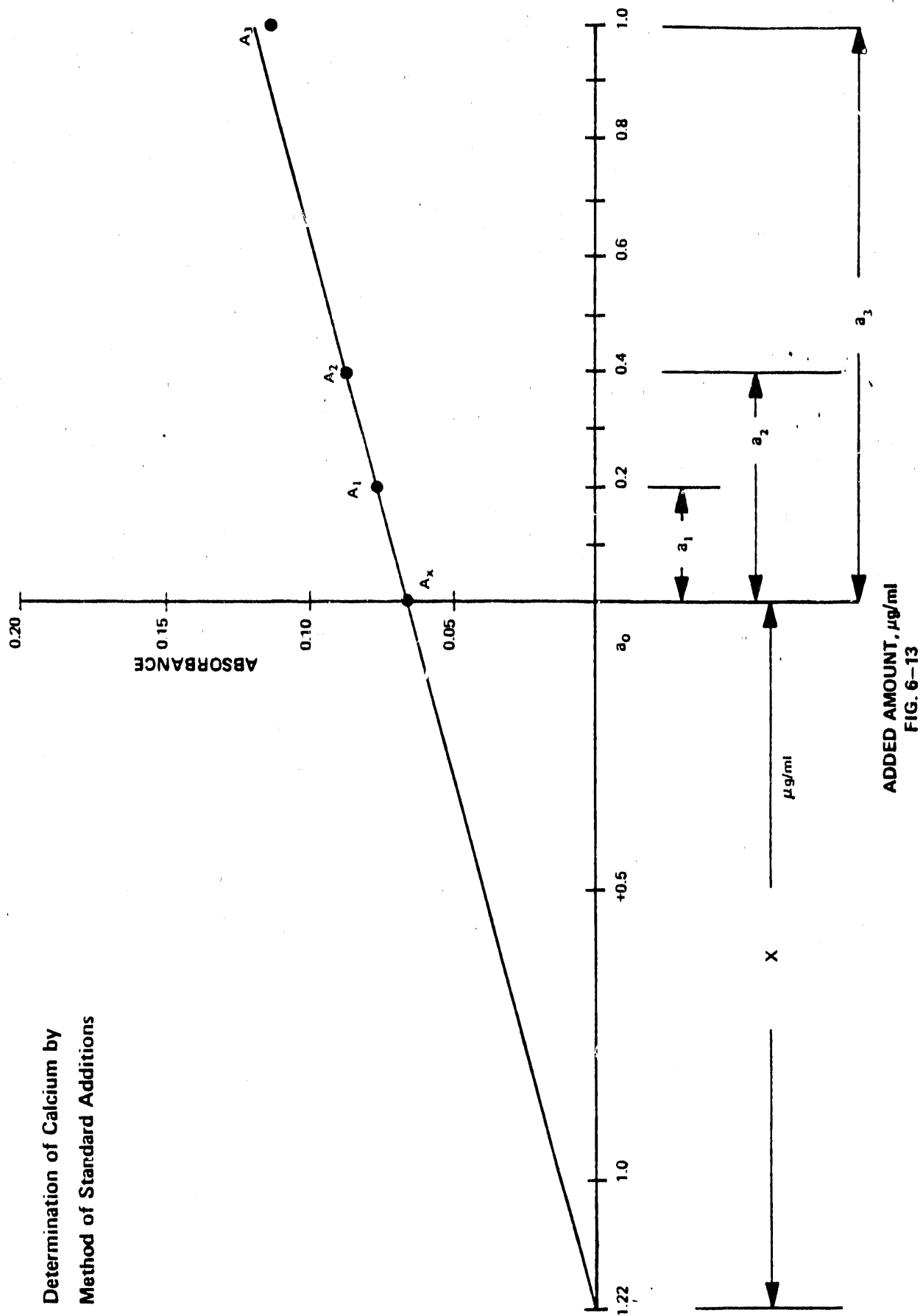
What we can conclude from all this is that the analyst should experiment with the method of standard additions *before* applying it to important analytical problems, in order to become fully aware of its advantages and its limitations. It should be considered an *emergency* procedure only. Analytical routines are usually not based on it.

Method of Standard Additions



CONCENTRATION (In Units Relative to " a_1 ")
 FIG. 6-12

Determination of Calcium by
Method of Standard Additions



ADDED AMOUNT, $\mu\text{g/ml}$
FIG. 6-13

APPENDIX F
ANALYTICAL DATA SUMMARY

Analytical Report
03/15/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

Customer Work Identification LIMB PROJECT
Purchase Order Number 209-026-05-00

Contents:

- 1 Analytical Data Summary
- 2 Sample History
- 3 Comments Summary
- 4 Notes and Definitions

Radian Analytical Services
8501 Mo-Pac Boulevard
P. O. Box 201088
Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:



Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Method: Alkalinity components (1)

List:

Sample ID:	DI LEACH LIMB	METHOD BLANK
	EMPASH-09	
Factor:	1.000000	1.000000
Results in:	mg/L	mg/L
	04A	05B
Matrix:	water	water

	Result	Det. Limit	Result	Det. Limit
Bicarbonate	ND	1.0	ND	1.0
Carbonate	110	1.0	ND	1.0
Total alkalinity	2300	1.0	ND	1.0

ND Not detected at specified detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Method: ICP 25 element scan SW6010 (1)

List: ICP, TCLP MS

Sample ID:	TCLP LEACH LI- MB EMPASH-09	TCLP BLANK	METHOD BLANK
Factor:	1	1	1
Results in:	mg/L	mg/L	mg/L
	02A	03B	05A
Matrix:	water	water	water

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Arsenic	<u>3.7 a</u>	3.0	ND	0.30	ND	0.30
Barium	<u>0.47</u>	0.010	<u>0.019 a</u>	0.010	ND	0.010
Cadmium	ND	0.050	ND	0.0050	ND	0.0050
Chromium	<u>0.19</u>	0.010	ND	0.010	ND	0.010
Lead	ND	0.50	ND	0.050	ND	0.050
Silver	ND	0.10	ND	0.010	ND	0.010

a Est. result less than 5 times detection limit

ND Not detected at specified detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Method/Analyte		Sample Identifications			
		TCLP LEACH LI- MB EMPASH-09		TCLP BLANK	
		02		03	
Matrix		water		water	
		Result	Det. Limit	Result	Det. Limit
Mercury, cold vapor E245.1					
Mercury		ND	mg/L 0.0002	ND	mg/L 0.0002
Selenium by SW7740					
Selenium		0.21	mg/L 0.025	ND	mg/L 0.0050
ND Not detected at specified detection limit					
(1) For a detailed description of flags and technical terms in this report refer to the glossary.					

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Method/Analyte		Sample Identifications			
		DI LEACH LIMB EMPASH-09 04 Matrix water		METHOD BLANK 05 water	
		Result	Det. Limit	Result	Det. Limit
Silver by ICPEs SW6010					
Silver		ND	mg/L 0.10		
Arsenic by SW7060					
Arsenic		0.011 @	mg/L 0.0040	ND	mg/L 0.0040
Barium by ICPEs SW6010					
Barium		0.60	mg/L 0.010		
Calcium by ICPEs SW6010					
Calcium		1500	mg/L 10	ND	mg/L 1.0
Cadmium by ICPEs SW6010					
Cadmium		ND	mg/L 0.050		
Chloride, titration. E325.3					
Chloride		98	mg/L 1.0	ND	mg/L 1.0
COD by SM5088					
Chemical Oxygen Demand		8.3 @	mg/L 5.0	ND	mg/L 5.0
Chromium by ICPEs SW6010					
Chromium		0.030 @	mg/L 0.010		
Fluoride by EPA 340.2					
Fluoride		3.8	mg/L 0.20	ND	mg/L 0.10
Mercury, cold vapor E245.1					
Mercury		ND	mg/L 0.0002	ND	mg/L 0.0002
Potassium by ICPEs SW6010					
Potassium		23	mg/L 3.0	ND	mg/L 3.0
Magnesium by ICPEs SW6010					
Magnesium		ND	mg/L 10	ND	mg/L 1.0
Nitrate-nitrite, EPA 353.2					
Nitrate-Nitrite as N		ND	mg/L 0.020	ND	mg/L 0.020
NACE corrosivity by SW1110					
Corrosivity		ND	mm/yr N/A	ND	mm/yr N/A
Sodium by ICPEs, SW6010					
Sodium		9.6	mg/L 1.0	ND	mg/L 1.0
Lead by SW7421					
Lead		0.027	mg/L 0.0030	ND	mg/L 0.0030
Total phenolics by SW9065					
Total phenolics		ND	mg/L 0.0050	ND	mg/L 0.0050
pH, SW846					
pH		12	pH units	6.2	pH units
Selenium by SW7740					
Selenium		0.13	mg/L 0.025	ND	mg/L 0.0050

ND Not detected at specified detection limit

@ Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to the glossary.

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Method/Analyte		Sample Identifications			
		D1 LEACH LIMB EMPASH-09 04 Matrix water		METHOD BLANK 05 water	
		Result	Det. Limit	Result	Det. Limit
Sulfate, SW9038					
sulfate		1000 mg/L	100	ND	mg/L 1.0
Tot. dissolv. solid E160.1					
Total dissolved solids		4000 mg/L	9.0	ND	mg/L 9.0
TOC by EPA 415.2					
Total organic carbon		ND	mg/L 1.0	ND	mg/L 1.0
ND Not detected at specified detection limit					
(1) For a detailed description of flags and technical terms in this report refer to the glossary.					

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Sample Identifications and Dates						
Sample ID	LIMB EMPASH-09 TCLP LEACH LI- TCLP BLANK MB EMPASH-09			DI LEACH LIMB EMPASH-09	METHOD BLANK	
Date Sampled	02/05/91	02/11/91		02/11/91		
Date Received	02/06/91	02/06/91	02/06/91	02/06/91	02/06/91	
Matrix	01	02	03	04 water	05 water	
Silver by ICPEs SW6010						
Prepared				02/12/91		
Analyzed				02/14/91		
Analyst				DRW		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Alkalinity components						
Prepared				02/25/91	02/25/91	
Analyzed				02/25/91	02/25/91	
Analyst				RDO	RDO	
File ID				6360225-1	6360225-1	
Blank ID						
Instrument				636	636	
Report as				received	received	
Arsenic by SW7060						
Prepared				03/07/91	03/07/91	
Analyzed				03/09/91	03/09/91	
Analyst				MXZ	MXZ	
File ID						
Blank ID						
Instrument				3030S	3030S	
Report as				received	received	
Barium by ICPEs SW6010						
Prepared				02/12/91		
Analyzed				02/13/91		
Analyst				DRW		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Calcium by ICPEs SW6010						
Prepared				02/12/91	02/12/91	
Analyzed				02/14/91	02/13/91	
Analyst				DRW	DRW	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Sample Identifications and Dates						
Sample ID	LIMB EMPASH-09	TCLP LEACH LI- MB EMPASH-09	TCLP BLANK	DI LEACH LIMB EMPASH-09	METHOD BLANK	
Date Sampled	02/05/91	02/11/91		02/11/91		
Date Received	02/06/91	02/06/91	02/06/91	02/06/91	02/06/91	
Matrix				water	water	
	01	02	03	04	05	
Cadmium by ICPEs SW6010						
Prepared				02/12/91		
Analyzed				02/14/91		
Analyst				DRW		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Chloride, titration E325.3						
Prepared				02/26/91	02/26/91	
Analyzed				02/26/91	02/26/91	
Analyst				TRR	TRR	
File ID						
Blank ID						
Instrument				CLT	CLT	
Report as				received	received	
COD by SM508B						
Prepared				03/06/91	03/06/91	
Analyzed				03/07/91	03/07/91	
Analyst				TRR	TRR	
File ID				COD0307-1		
Blank ID						
Instrument				COD	COD	
Report as				received	received	
Chromium by ICPEs SW6010						
Prepared				02/12/91		
Analyzed				02/13/91		
Analyst				DRW		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Fluoride by EPA 340.2						
Prepared				03/07/91	03/07/91	
Analyzed				03/07/91	03/07/91	
Analyst				EAT	EAT	
File ID				9250307-1	9250307-1	
Blank ID						
Instrument				925	925	
Report as				received	received	

Babcock and Wilcox Co.
 Radfan Work Order: 91-02-047

Sample Identifications and Dates						
Sample ID	LIMB EMPASH-09	TCLP LEACH LI- MB EMPASH-09	TCLP BLANK	DI LEACH LIMB EMPASH-09	METHOD BLANK	
Date Sampled	02/05/91	02/11/91		02/11/91		
Date Received	02/06/91	02/06/91	02/06/91	02/06/91	02/06/91	
Matrix		water	water	water	water	
	01	02	03	04	05	
Mercury, cold vapor E245.1						
Prepared		02/13/91	02/13/91	02/13/91	02/13/91	
Analyzed		02/13/91	02/13/91	02/13/91	02/13/91	
Analyst		MXZ	MXZ	MXZ	MXZ	
File ID						
Blank ID						
Instrument		403	403	403	403	
Report as		received	received	received	received	
ICP 25 element scan SW6010						
Prepared		02/12/91	02/12/91		02/12/91	
Analyzed		02/13/91	02/13/91		02/13/91	
Analyst		DRW	DRW		DRW	
File ID						
Blank ID						
Instrument		JA61	JA61		JA61	
Report as		received	received		received	
Potassium by ICPES SW6010						
Prepared				02/12/91	02/12/91	
Analyzed				02/13/91	02/13/91	
Analyst				DRW	DRW	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Magnesium by ICPES SW6010						
Prepared				02/12/91	02/12/91	
Analyzed				02/14/91	02/13/91	
Analyst				DRW	DRW	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Nitrate-nitrite, EPA 353.2						
Prepared				02/15/91	02/15/91	
Analyzed				02/15/91	02/15/91	
Analyst				RDO	RDO	
File ID				AA10215-1	AA10215-1	
Blank ID						
Instrument				AA1	AA1	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Sample Identifications and Dates						
Sample ID	LIMB EMPASH-09	TCLP LEACH LI- MB EMPASH-09	TCLP BLANK	D1 LEACH LIMB EMPASH-09	METHOD BLANK	
Date Sampled	02/05/91	02/11/91		02/11/91		
Date Received	02/06/91	02/06/91	02/06/91	02/06/91	02/06/91	
Matrix	01	02	03	water 04	water 05	
NACE corrosivity by SW1110						
Prepared				02/27/91	02/27/91	
Analyzed				02/27/91	02/27/91	
Analyst				EAT	EAT	
File ID				COR0227-1	COR0227-1	
Blank ID						
Instrument				COR	COR	
Report as				received	received	
Sodium by ICPES, SW6010						
Prepared				02/12/91	02/12/91	
Analyzed				02/13/91	02/13/91	
Analyst				DRW	DRW	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Lead by SW7421						
Prepared				02/12/91	02/12/91	
Analyzed				03/06/91	03/06/91	
Analyst				DLC	DLC	
File ID						
Blank ID						
Instrument				3030Z	3030Z	
Report as				received	received	
Total phenolics by SW9065						
Prepared				03/08/91	03/08/91	
Analyzed				03/08/91	03/08/91	
Analyst				MJS	MJS	
File ID						
Blank ID						
Instrument				AA 11	AA 11	
Report as				received	received	
pH, SW846						
Prepared				02/11/91	02/11/91	
Analyzed				02/11/91	02/11/91	
Analyst				MH	MH	
File ID						
Blank ID						
Instrument				925	925	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-02-047

Sample Identifications and Dates						
Sample ID	LIMB EMPASH-09	TCLP LEACH LI- MB EMPASH-09	TCLP BLANK	DI LEACH LIMB EMPASH-09	METHOD BLANK	
Date Sampled	02/05/91	02/11/91		02/11/91		
Date Received	02/06/91	02/06/91	02/06/91	02/06/91	02/06/91	
Matrix	solid	water	water	water	water	
	01	02	03	04	05	
Selenium by SW7740						
Prepared		02/12/91	02/12/91	02/12/91	02/12/91	
Analyzed		02/13/91	02/13/91	02/13/91	02/13/91	
Analyst		RAA	RAA	RAA	RAA	
File ID						
Blank ID						
Instrument		3030Z	3030Z	3030Z	3030Z	
Report as		received	received	received	received	
Sulfate, SW9038						
Prepared				03/01/91	03/01/91	
Analyzed				03/01/91	03/01/91	
Analyst				TRR	TRR	
File ID				2100A0301-1		
Blank ID						
Instrument				2100A	2100A	
Report as				received	received	
TCLP leaching						
Prepared	02/11/91		02/11/91			
Analyzed	02/12/91		02/12/91			
Analyst	HD		HD			
File ID						
Blank ID						
Instrument						
Report as	received		received			
Tot. dissolv. solid E160.1						
Prepared				02/13/91	02/13/91	
Analyzed				02/13/91	02/13/91	
Analyst				EAT	EAT	
File ID				TDS0213-1	TDS0213-1	
Blank ID						
Instrument				TDS	TDS	
Report as				received	received	
TOC by EPA 415.2						
Prepared				02/14/91	02/14/91	
Analyzed				02/14/91	02/14/91	
Analyst				MH	MH	
File ID						
Blank ID						
Instrument				DC-80	DC-80	
Report as				received	received	

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-02-047

Q ALL METHODS EXCEPT CLP

The results which are less than five times the method specified detection limit.

EXPLANATION

Uncertainty of the analysis will increase as the method detection limit is approached. These results should be considered approximate.

ND ALL METHODS EXCEPT CLP

This flag is used to denote analytes which are not detected at or above the specified detection limit.

EXPLANATION

The value to the right of the < symbol is the method specified detection limit for the analyte.

Babcock and Wilcox Co.
Radian Work Order: 91-02-047

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

Radian Work Order 91-03-011

Analytical Report
04/10/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

Customer Work Identification LIMB PROJECT
Purchase Order Number 209-026-05-00

Contents:

- 1 Analytical Data Summary
- 2 Sample History
- 3 Comments Summary
- 4 Notes and Definitions

Radian Analytical Services
8501 Mo-Pac Boulevard
P. O. Box 201088
Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:



Babcock and Wilcox Co.

Radian Work Order: 91-03-011

Method: Alkalinity components (1)

List:

Sample ID:

 DI LEACH LIMB-
 -EMPASH-10

METHOD BLANK

Factor:

1

1

Results in:

mg/L

mg/L

04A

05B

Matrix:

water

water

Bicarbonate

Result Det. Limit

ND 1.0

Result Det. Limit

ND 1.0

Carbonate

120 1.0

ND 1.0

Total alkalinity

2300 1.0

ND 1.0

ND Not detected at specified detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Method: ICP 25 element scan SW6010 (1)

List: ICP, TCLP MS

Sample ID:	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	METHOD BLANK	TCLP LEACH MS
Factor:	1	1	1	0
Results in:	mg/L	mg/L	mg/L	%recvry
	02A	03B	05A	06A
Matrix:	water	water	water	water

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Arsenic	ND	1.2	ND	0.30	ND	0.30	112	
Barium	1.1	0.040	0.96	0.010	ND	0.010	64 q	
Cadmium	ND	0.020	ND	0.0050	ND	0.0050	98	
Chromium	0.018 q	0.010	ND	0.010	ND	0.010	85	
Lead	ND	0.20	ND	0.050	ND	0.050	48 q	
Silver	ND	0.040	ND	0.010	ND	0.010	94	

 ND Not detected at specified detection limit
 q Outside control limits

q Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

Method: ICP 25 element scan SW6010 (1)
List: ICP, TCLP MS
Sample ID: TCLP LEACH MSD
Factor: 0
Results in: %recvry
07A
Matrix: water

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Arsenic	108							
Barium	66 Q							
Cadmium	99							
Chromium	82							
Lead	63 Q							
Silver	97							

Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Method/Analyte		Sample Identifications	
	TCLP LEACH LI- MB-EMPASH-10 02	TCLP BLANK 03	
Matrix	Water	Water	

	Result	Det. Limit	Result	Det. Limit	
Mercury, cold vapor E245.1					
Mercury	ND	mg/L 0.0002	ND	mg/L 0.0002	
Selenium by SW7740					
Selenium	0.024 SD	mg/L 0.0050	ND S	mg/L 0.0050	

ND Not detected at specified detection limit

S Determined by Method of Standard Addition

SD Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to the glossary.

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Method/Analyte	Sample Identifications					
	DI LEACH LIMB- -EMPASH-10		METHOD BLANK		TCLP LEACH MS	
	04 Matrix water		05 water		06 water	
	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Silver by ICPE SW6010						
Silver	ND	mg/L 0.040				
Arsenic by SW7060						
Arsenic	0.0040 a	mg/L 0.0040	ND	mg/L 0.0040		
Barium by ICPE SW6010						
Barium	0.48	mg/L 0.040				
Calcium by ICPE SW6010						
Calcium	1700	mg/L 4.0	ND	mg/L 1.0		
Cadmium by ICPE SW6010						
Cadmium	ND	mg/L 0.020				
Chloride, titration E325.3						
Chloride	460	mg/L 50	ND	mg/L 1.0		
COD by SM5088						
Chemical Oxygen Demand	11 a	mg/L 5.0				
Chromium by ICPE SW6010						
Chromium	0.29	mg/L 0.040				
Fluoride by EPA 340.2						
Fluoride	2.3	mg/L 0.20	ND	mg/L 0.10		
Mercury, cold vapor E245.1						
Mercury	ND	mg/L 0.0002	ND	mg/L 0.0002	96	%recvry
Potassium by ICPE SW6010						
Potassium	12 a	mg/L 3.0	ND	mg/L 3.0		
Magnesium by ICPE SW6010						
Magnesium	ND	mg/L 1.0	ND	mg/L 1.0		
Nitrate-nitrite, EPA 353.2						
Nitrate-Nitrite as N	0.26	mg/L 0.020	ND	mg/L 0.020		
NACE corrosivity by SW1110						
Corrosivity	ND	mm/yr N/A				
Sodium by ICPE SW6010						
Sodium	4.9 a	mg/L 1.0	ND	mg/L 1.0		
Lead by SW7421						
Lead	0.012 a	mg/L 0.0030	ND	mg/L 0.0030		
Total phenolics by SW9065						
Total phenolics	0.012 a	mg/L 0.0050	0.013 a	mg/L 0.0050		
pH, SW846						
pH	12	pH units				
Selenium by SW7740						
Selenium	0.071 S	mg/L 0.0050	ND	mg/L 0.0050	41 Q	%recvry

 ND Not detected at specified detection limit
 S Determined by Method of Standard Addition

 a Est. result less than 5 times detection limit
 Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to the glossary.

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Method/Analyte		Sample Identifications			
		DI LEACH LIMB- -EMPASH-10 04 Matrix		METHOD BLANK 05 water	
		Result	Det. Limit	Result	Det. Limit
Sulfate, SW9038					
Sulfate		1300 mg/L	100	ND	1.0
Tot. dissolv. solid E160.1					
Total dissolved solids		5000 mg/L	9.0	ND	9.0
TOC by EPA 415.2					
Total organic carbon		ND	1.0	ND	1.0
ND Not detected at specified detection limit (1) For a detailed description of flags and technical terms in this report refer to the glossary.					

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

Method/Analyte		Sample Identifications	
		TCLP LEACH MSD	
		07	
Matrix		water	
	Result	Det. Limit	
Mercury, cold vapor E245.1			
Mercury	103	%recvry	
Selenium by SW7740			
Selenium	38 Q	%recvry	
Q Outside control limits.			
(1) For a detailed description of flags and technical terms in this report refer to the glossary.			

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Sample Identifications and Dates						
Sample ID	LIMB-EMPASH-10	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	DI LEACH LIMB- -EMPASH-10	METHOD BLANK	TCLP LEACH MS
Date Sampled	02/28/91	03/04/91		03/06/91		03/04/91
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix	01	02	03	water 04	water 05	06
Silver by ICPES SW6010						
Prepared				03/11/91		
Analyzed				03/12/91		
Analyst				DES		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Alkalinity components						
Prepared				03/19/91	03/19/91	
Analyzed				03/19/91	03/19/91	
Analyst				RDO	RDO	
File ID						
Blank ID						
Instrument				636	636	
Report as				received	received	
Arsenic by SW7060						
Prepared				03/11/91	03/11/91	
Analyzed				04/03/91	04/03/91	
Analyst				DLC	DLC	
File ID						
Blank ID						
Instrument				3030s	3030s	
Report as				received	received	
Barium by ICPES SW6010						
Prepared				03/11/91		
Analyzed				03/12/91		
Analyst				DES		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Calcium by ICPES SW6010						
Prepared				03/11/91	03/11/91	
Analyzed				03/12/91	03/12/91	
Analyst				DES	DES	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Sample Identifications and Dates						
Sample ID	LIMB-EMPASH-10	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	DI LEACH LIMB- -EMPASH-10	METHOD BLANK	TCLP LEACH MS
Date Sampled	02/28/91	03/04/91		03/06/91		03/04/91
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix	01	02	03	water 04	water 05	06
Cadmium by ICPEs SW6010						
Prepared				03/11/91		
Analyzed				03/12/91		
Analyst				DES		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Chloride, titration E325.3						
Prepared				03/11/91	03/11/91	
Analyzed				03/11/91	03/11/91	
Analyst				TRR	TRR	
File ID						
Blank ID						
Instrument				CLT	CLT	
Report as				received	received	
COD by SM508B						
Prepared				03/22/91		
Analyzed				03/22/91		
Analyst				TRR		
File ID						
Blank ID						
Instrument				COD		
Report as				received		
Chromium by ICPEs SW6010						
Prepared				03/11/91		
Analyzed				03/12/91		
Analyst				DES		
File ID						
Blank ID						
Instrument				JA61		
Report as				received		
Fluoride by EPA 340.2						
Prepared				03/07/91	03/07/91	
Analyzed				03/07/91	03/07/91	
Analyst				EAT	EAT	
File ID				9250307-1	9250307-1	
Blank ID						
Instrument				925	925	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Sample Identifications and Dates						
Sample ID	LIMB-EMPASH-10	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	DI LEACH LIMB- -EMPASH-10	METHOD BLANK	TCLP LEACH MS
Date Sampled	02/28/91	03/04/91		03/06/91		03/04/91
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix		water	water	water	water	water
	01	02	03	04	05	06
Mercury, cold vapor E245.1						
Prepared		03/07/91	03/07/91	03/07/91	03/07/91	03/07/91
Analyzed		03/07/91	03/07/91	03/07/91	03/07/91	03/07/91
Analyst		MXZ	MXZ	MXZ	MXZ	MXZ
File ID						
Blank ID						
Instrument		403	403	403	403	403
Report as		received	received	received	received	received
ICP 25 element scan SW6010						
Prepared		03/11/91	03/11/91		03/11/91	03/11/91
Analyzed		03/12/91	03/12/91		03/12/91	03/12/91
Analyst		DES	DES		DES	DES
File ID						
Blank ID						
Instrument		JA61	JA61		JA61	JA61
Report as		received	received		received	received
Potassium by ICPEs SW6010						
Prepared				03/11/91	03/11/91	
Analyzed				03/12/91	03/12/91	
Analyst				DES	DES	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Magnesium by ICPEs SW6010						
Prepared				03/11/91	03/11/91	
Analyzed				03/12/91	03/12/91	
Analyst				DES	DES	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Nitrate-nitrite, EPA 353.2						
Prepared				03/14/91	03/14/91	
Analyzed				03/14/91	03/14/91	
Analyst				MJS	MJS	
File ID						
Blank ID						
Instrument				AA I	AA I	
Report as				received	received	

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Sample Identifications and Dates						
Sample ID	LIMB-EMPASH-10	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	DI LEACH LIMB- -EMPASH-10	METHOD BLANK	TCLP LEACH MS
Date Sampled	02/28/91	03/04/91		03/06/91		03/04/91
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix	01	02	03	water 04	water 05	06
NACE corrosivity by SW1110						
Prepared				04/05/91		
Analyzed				04/07/91		
Analyst				EAT		
File ID						
Blank ID						
Instrument				NA		
Report as				received		
Sodium by ICPES, SW6010						
Prepared				03/11/91	03/11/91	
Analyzed				03/12/91	03/12/91	
Analyst				DES	DES	
File ID						
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Lead by SW7421						
Prepared				03/11/91	03/11/91	
Analyzed				04/03/91	04/03/91	
Analyst				RAA	RAA	
File ID						
Blank ID						
Instrument				3030E	3030E	
Report as				received	received	
Total phenolics by SW9065						
Prepared				03/27/91	03/27/91	
Analyzed				03/27/91	03/27/91	
Analyst				MJS	MJS	
File ID						
Blank ID						
Instrument				AA II	AA II	
Report as				received	received	
pH, SW846						
Prepared				03/07/91		
Analyzed				03/07/91		
Analyst				EAT		
File ID				9250307-1		
Blank ID						
Instrument				925		
Report as				received		

Babcock and Wilcox Co.
 Radian Work Order: 91-03-011

Sample Identifications and Dates						
Sample ID	LIMB-EMPASH-10	TCLP LEACH LI- MB-EMPASH-10	TCLP BLANK	DI LEACH LIMB- -EMPASH-10	METHOD BLANK	TCLP LEACH MS
Date Sampled	02/28/91	03/04/91		03/06/91		03/04/91
Date Received	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91	03/01/91
Matrix	solid	water	water	water	water	water
	01	02	03	04	05	06
Selenium by SW7740						
Prepared		03/11/91	03/11/91	03/11/91	03/11/91	03/11/91
Analyzed		03/15/91	03/15/91	03/15/91	03/15/91	03/15/91
Analyst		DLC	DLC	DLC	DLC	DLC
File ID						
Blank ID						
Instrument		3030Z	3030Z	3030Z	3030Z	3030Z
Report as		received	received	received	received	received
Sulfate, SW9038						
Prepared				03/14/91	03/14/91	
Analyzed				03/14/91	03/14/91	
Analyst				TRR	TRR	
File ID						
Blank ID						
Instrument				2100A	2100A	
Report as				received	received	
TCLP leaching						
Prepared	03/04/91		03/04/91			
Analyzed	03/05/91		03/05/91			
Analyst	HD		HD			
File ID						
Blank ID						
Instrument						
Report as	received		received			
Tot. dissolv. solid E160.1						
Prepared				03/13/91	03/13/91	
Analyzed				03/13/91	03/13/91	
Analyst				EAT	EAT	
File ID						
Blank ID						
Instrument				TDS	TDS	
Report as				received	received	
TOC by EPA 415.2						
Prepared				03/28/91	03/28/91	
Analyzed				03/28/91	03/28/91	
Analyst				MH	MH	
File ID						
Blank ID						
Instrument				DC-80	DC-80	
Report as				received	received	

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

Sample Identifications and Dates

Sample ID TCLP LEACH MSD

Date Sampled 03/04/91

Date Received 03/01/91

Matrix water
 07

Mercury, cold vapor E245.1

Prepared 03/07/91

Analyzed 03/07/91

Analyst MXZ

File ID

Blank ID

Instrument 403

Report as received

ICP 25 element scan SW6010

Prepared 03/11/91

Analyzed 03/12/91

Analyst DES

File ID

Blank ID

Instrument JA61

Report as received

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

Sample Identifications and Dates

Sample ID TCLP LEACH MSD

Date Sampled 03/04/91
Date Received 03/01/91
Matrix water
 07

Selenium by SW7740

Prepared 03/11/91
Analyzed 03/15/91
Analyst DLC
File ID
Blank ID
Instrument 3030Z
Report as received

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

General Comments

Samples 9103011-02A, -06A MS, and -07A MSD had to be diluted by a factor of four to eliminate interferences from calcium. The low recovery for lead may be due to the dilution of the spike to within five times the IDL.

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

Q ALL METHODS EXCEPT CLP

The results which are less than five times the method specified detection limit.

EXPLANATION

Uncertainty of the analysis will increase as the method detection limit is approached. These results should be considered approximate.

ND ALL METHODS EXCEPT CLP

This flag is used to denote analytes which are not detected at or above the specified detection limit.

EXPLANATION

The value to the right of the < symbol is the method specified detection limit for the analyte.

Q ALL METHODS EXCEPT CLP

This quality control standard is outside method or laboratory specified control limits.

EXPLANATION

This flag is applied to matrix spike, analytical QC spike, and surrogate recoveries; and to RPD(relative percent difference) values for duplicate analyses and matrix spike/matrix spike duplicate result.

S INORGANIC METHODS INCLUDING CLP

This flag indicates that a specific result from analysis has been obtained using the Method of Standard Addition.

Babcock and Wilcox Co.
Radian Work Order: 91-03-011

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX G
PERMEABILITY DATA SUMMARY

Radian Work Order 91-02-048

Analytical Report

03/15/91

Babcock and Wilcox Co.

Radian

RTP

NC

Luke Contos

Customer Work Identification LIMB PROJECT

Purchase Order Number 209-026-05-00

Contents:

- 1 Analytical Data Summary
- 2 Sample History
- 3 Comments Summary
- 4 Notes and Definitions

Radian Analytical Services

8501 Mo-Pac Boulevard

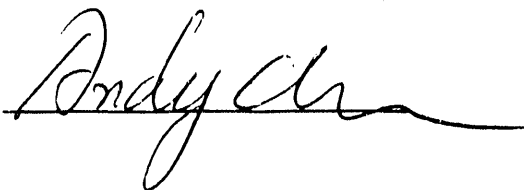
P. O. Box 201088

Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:



Babcock and Wilcox Co.
Radian Work Order: 91-02-048

Method/Analyte		Sample Identifications	
	LIMB EMPASH-09		
	01		
Matrix			
Permeability SW9100	Result	Det. Limit	
Permeability	0.000013 cm/sec	N/A	

(1) For a detailed description of flags and technical terms in this report refer to the glossary.

Babcock and Wilcox Co.
Radian Work Order: 91-02-048

Sample Identifications and Dates

Sample ID LIMB EMPASH-09

Date Sampled 02/05/91
Date Received 02/06/91
Matrix

 01

Permeability SW9100

Prepared

Analyzed

Analyst

File ID

Blank ID

Instrument

Report as

03/08/91

GST

received

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-02-048

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

Analytical Report
04/04/91

REVISED 04/14/91

Babcock and Wilcox Co.
Radian RTP NC
Luke Contos

Customer Work Identification LIMB PROJECT Purchase Order Number 209-026-05-00
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Contents:	
1	Analytical Data Summary
2	Sample History
3	Comments Summary
4	Notes and Definitions

Radian Analytical Services
8501 Mo-Pac Boulevard
P. O. Box 201088
Austin, TX 78720-1088

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: 

Previously Reported on 03/25/91.

Babcock and Wilcox Co.
Radian Work Order: 91-03-009

Method/Analyte		Sample Identifications	
LIMB-EMPASH-10			
Matrix		01	
Permeability SW9100	Result	Det. Limit	
Permeability	0.0000037 cm/sec	N/A	
(1) For a detailed description of flags and technical terms in this report refer to the glossary.			

Babcock and Wilcox Co.
Radian Work Order: 91-03-009

Sample Identifications and Dates

Sample ID LIMB-EMPASH-10

Date Sampled 02/28/91

Date Received 03/01/91

Matrix 01

Permeability SW9100

Prepared 03/15/91

Analyzed 03/19/91

Analyst GST

File ID

Blank ID

Instrument

Report as received

Appendix A

Comments, Notes and Definitions

Babcock and Wilcox Co.
Radian Work Order: 91-03-009

TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units - ug/L	micrograms per liter (parts per billion); liquids/water
ug/kg	micrograms per kilogram (parts per billion); soils/solids
ug/M3	micrograms per cubic meter; air samples
mg/L	milligrams per liter (parts per million); liquids/water
mg/kg	milligrams per kilogram (parts per million); soils/solids
%	percent; usually used for percent recovery of QC standards
uS/cm	conductance unit; microSiemens/centimeter
mL/hr	milliliters per hour; rate of settlement of matter in water
NTU	turbidity unit; nephelometric turbidity unit
CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX H
AMBIENT AIR MODELING PROTOCOL

5.0 AMBIENT MONITORING

5.1 Air

Air dispersion modeling will be used to assess the relative change in maximum ground level pollutant concentrations for Unit 4. The maximum predicted ground level concentration will be determined from the baseline operating conditions for Unit 4 during normal firing conditions with no sorbent injection (firing coal with a 1.8% sulfur content) and for each of the coal/sorbent scenarios that will be evaluated in the Coolside and LIMB Extension studies. The pollutant emissions evaluated will include SO₂, NO_x, PM/PM₁₀, and CO. The averaging periods that will be predicted for each pollutant will correspond with those for which a National Ambient Air Quality Standard (NAAQS) has been established.

In most cases, reductions in predicted maximum ground level pollutant concentrations are expected to occur since Unit 4 emissions levels will decrease compared to baseline levels. If increases in maximum concentrations over the baseline case are predicted, a further evaluation will be conducted to determine if the NAAQS will be exceeded. No additional ambient air monitoring will be conducted during the demonstration study.

The following methodology will be used in this study:

1. Define the baseline emissions case. AP-42 emissions factors will be used to determine emissions of SO₂, NO_x, CO, and PM/PM₁₀ from Unit 4 firing 1.8% sulfur coal if stack data are not available. Representative stack parameters (stack exit temperature and flowrate) for the maximum firing rate of Unit 4 will be derived from existing stack test data for the period when Unit 4 was firing 1.8% sulfur coal.

2. Determine the maximum baseline ground level concentration. The maximum predicted annual average and short term average off-property concentrations from Unit 4 will be determined for the baseline emissions case. The modeling analysis will be conducted using five years of meteorological

data and an EPA approved air dispersion model ISCST Version 88348. Additional discussion of the model methodology is presented below.

3. Define the emissions case for each coal/sorbent scenario. New stack parameter and emissions data for Unit 4 will be developed from the actual data collected from CEM monitoring and from Method 5 testing during the demonstration project.

4. Determine the maximum ground level concentration for a new scenario. The maximum predicted annual average and short term average off property concentrations from Unit 4 will be determined for each coal/sorbent emissions case. The modeling methodology and model inputs used to determine the maximum concentrations will be identical to those used in (2) and discussed below.

5. Compare the maximum concentrations predicted in (2) and (4). The results of the modeling analyses conducted in (2) and (4) will be compared to determine the increase (or decrease) in the predicted maximum ground level concentration for each pollutant and averaging period. In some cases, the maximum predicted concentration for the baseline and coal/sorbent case will occur at different receptors for the same pollutant and averaging period because of the differences in stack exit temperature or flowrate. For these cases, the maximum predicted concentration for the baseline case and the coal/sorbent cases will be determined at the maximum receptor location determined for each case, and the maximum difference reported.

If the difference in maximum predicted concentration from the new coal/sorbent case compared to the baseline case that was determined in (5) for all pollutants and averaging periods result in concentration decreases, no further evaluation will be necessary. Otherwise, the following analysis will be performed:

6. The magnitude of the increase for each pollutant and averaging period predicted in (5) will be compared to the ambient air significance levels as defined in the Prevention of Significant Deterioration air regulations (40 CFR 51.165 b(2)). For the pollutants evaluated in this study,

these values are $1 \mu\text{g}/\text{m}^3$ (SO_2 , PM/PM_{10} , NO_x) for the annual average, $5 \mu\text{g}/\text{m}^3$ (SO_2 , PM/PM_{10}) for the 24-hour average, $25 \mu\text{g}/\text{m}^3$ (SO_2) for the 3-hour average, $500 \mu\text{g}/\text{m}^3$ (CO) for the 1-hour average, and $2000 \mu\text{g}/\text{m}^3$ (CO) for the 8-hour averaging period. By definition, if the concentration is less than the significance level, a source is not considered to cause or contribute to a violation of the national air quality standard. If the increase in concentration predicted in (5) for a given pollutant and averaging period is significant, the existing ambient air monitoring will be reviewed and the need for collection of additional monitoring data will be evaluated.

5.1.1 Model Selection

The estimates of ambient air quality concentrations will be based on the applicable air quality model and techniques as specified in the EPA Guideline on Air Quality Models. The EPA approved version of the Industrial Source Complex model (ISCST version 88348) will be used in the modeling analysis.

5.1.2 Meteorological Data

Five years (1981-1985) of meteorological data will be used in the analysis. The surface data were recorded at Hopkins International Airport in Cleveland, Ohio, and the upper-air data were recorded at Buffalo International Airport in Buffalo, New York. These data were obtained from Ohio Edison in preprocessed format.

5.1.3 Stack Height Analysis

A Good Engineering Practice (GEP) stack height analysis will be conducted. The purpose of the GEP stack height analysis is to evaluate the potential influence of building wake effects from the existing structures on ground level concentrations. Building dimensions will be input to the ISCST model. The worst-case building dimension inputs will be calculated using guidance in the Industrial Source Complex (ISC) User's Guide and the Bowman Environmental Engineering GEP computer program.

5.1.4 Receptor Grid

A regularly spaced cartesian grid, with a spacing of 250 to 500 meters, surrounding the facility will be developed. Additional receptors will be located along the plant fenceline.

5.2 Future Ambient Air Quality Work

A plant visit was conducted on January 23, 1990. During this visit, Ohio Edison personnel provided the following items:

- Plot plan showing property and fenceline positions
- Building orientation and dimensions
- Stack dimensions

In addition to the above information, photographs were taken during a tour of the facility, and a survey of the local area provided needed information for future modeling work.

APPENDIX I

HOURLY SURFACE OBSERVATIONS AND MIXING HEIGHT DATA

APPENDIX K-1. CLEVELAND-HOPKINS AIRPORT HOURLY SURFACE OBSERVATIONS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	HOUR	CEILING (100s FT)	WIND DIR. (10s DEG.)	WIND SPEED (KTS)	DRY BULB (F)	CLOUD COVER (TENTHS)	TOTAL OPAQUE CLOUD COVER (TENTHS)
14820	11	27	0	50	19	17	61	10	10
14820	11	27	1	42	19	14	62	10	10
14820	11	27	2	55	19	15	63	10	10
14820	11	27	3	70	19	15	63	10	10
14820	11	27	4	120	20	16	62	10	10
14820	11	27	5	110	20	16	62	10	10
14820	11	27	6	30	22	17	63	10	10
14820	11	27	7	130	20	15	63	10	10
14820	11	27	8	130	21	24	63	10	7
14820	11	27	9	100	22	18	63	10	9
14820	11	27	10	95	21	15	64	10	10
14820	11	27	11	150	21	19	66	9	8
14820	11	27	12	30	22	22	69	10	8
14820	11	27	13	30	21	21	80	10	9
14820	11	27	14	30	21	16	69	10	10
14820	11	27	15	80	21	14	69	10	10
14820	11	27	16	110	21	15	81	8	7
14820	11	27	17	55	23	13	81	9	9
14820	11	27	18	110	21	12	81	10	9
14820	11	27	19	110	20	14	81	10	9
14820	11	27	20	110	19	13	69	7	7
14820	11	27	21		19	11	68	8	2
14820	11	27	22		19	12	68	4	2
14820	11	27	23		21	14	68	3	0
14820	11	28	0		20	14	68	1	1
14820	11	28	1		20	16	67	3	0
14820	11	28	2		20	21	68	3	0
14820	11	28	3		19	18	68	4	2
14820	11	28	4	250	21	22	68	8	6
14820	11	28	5	70	19	16	67	10	10
14820	11	28	6	40	22	20	67	10	10
14820	11	28	7	41	21	17	67	10	10
14820	11	28	8	40	23	10	64	10	10
14820	11	28	9	250	22	19	66	10	10
14820	11	28	10	29	27	22	65	10	10
14820	11	28	11	12	27	15	54	10	10
14820	11	28	12	10	28	12	51	10	10
14820	11	28	13	14	25	16	50	10	10
14820	11	28	14	16	30	18	49	10	10
14820	11	28	15	16	27	16	45	10	10
14820	11	28	16	28	25	18	43	10	10
14820	11	28	17	20	24	14	40	10	10
14820	11	28	18	17	26	10	38	10	10
14820	11	28	19	20	24	13	36	10	10
14820	11	28	20	18	27	11	37	10	10
14820	11	28	21	20	28	8	36	10	10
14820	11	28	22	21	25	9	35	10	10
14820	11	28	23	26	25	8	35	10	10
14820	11	29	0	20	26	8	35	10	10
14820	11	29	1	27	24	8	34	10	10
14820	11	29	2	25	29	7	33	10	10
14820	11	29	3	26	28	6	33	10	10

APPENDIX K-1. CLEVELAND-HOPKINS AIRPORT HOURLY SURFACE OBSERVATIONS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	HOUR	CEILING (100s FT)	WIND DIR. (10s DEG.)	WIND SPEED (KTS)	DRY BULB (F)	CLOUD COVER (TENTHS)	TOTAL OPAQUE CLOUD COVER (TENTHS)
14820	11	29	4	28	25	8	33	10	10
14820	11	29	5	30	23	7	33	10	10
14820	11	29	6	30	23	7	33	10	10
14820	11	29	7	30	23	5	33	10	10
14820	11	29	8	29	24	6	33	10	10
14820	11	29	9	29	24	7	32	10	10
14820	11	29	10	26	24	7	33	10	10
14820	11	29	11	26	25	7	34	8	8
14820	11	29	12	29	27	11	35	9	9
14820	11	29	13	29	25	8	34	10	10
14820	11	29	14	26	26	7	35	10	10
14820	11	29	15	28	27	6	35	10	10
14820	11	29	16	37	25	7	34	10	10
14820	11	29	17	40	26	6	35	10	10
14820	11	29	18	36	27	5	35	10	10
14820	11	29	19	33	25	6	35	10	10
14820	11	29	20	33	25	6	35	10	10
14820	11	29	21	40	25	7	34	9	9
14820	11	29	22	36	25	7	33	7	7
14820	11	29	23	30	26	5	32	7	7
14820	11	30	0	31	25	5	32	7	7
14820	11	30	1	31	23	5	30	6	6
14820	11	30	2		24	4	30	5	5
14820	11	30	3		22	5	29	0	0
14820	11	30	4		22	4	27	0	0
14820	11	30	5		22	4	25	0	0
14820	11	30	6		21	4	25	0	0
14820	11	30	7		22	4	25	1	0
14820	11	30	8		22	4	25	0	0
14820	11	30	9		22	9	29	0	0
14820	11	30	10		22	9	34	0	0
14820	11	30	11		23	11	37	0	0
14820	11	30	12		24	11	40	0	0
14820	11	30	13		22	13	42	1	0
14820	11	30	14		21	12	44	1	0
14820	11	30	15		20	17	43	2	1
14820	11	30	16		21	12	43	5	1
14820	11	30	17		21	18	40	9	4
14820	11	30	18		22	13	39	7	3
14820	11	30	19		19	14	38	0	0
14820	11	30	20		21	17	37	0	0
14820	11	30	21		20	21	38	0	0
14820	11	30	22		21	19	38	0	0
14820	11	30	23		21	17	37	0	0
14820	12	1	0		21	13	36	0	0
14820	12	1	1		21	11	37	5	5
14820	12	1	2		21	9	35	0	0
14820	12	1	3		21	6	34	0	0
14820	12	1	4		21	6	33	0	0
14820	12	1	5		20	6	32	0	0
14820	12	1	6		21	6	35	3	1
14820	12	1	7		22	7	36	8	3

APPENDIX K-1. CLEVELAND-HOPKINS AIRPORT HOURLY SURFACE OBSERVATIONS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	HOUR	CEILING (100s FT)	WIND DIR. (10s DEG.)	WIND SPEED (KTS)	DRY BULB (F)	CLOUD COVER (TENTHS)	TOTAL OPAQUE CLOUD COVER (TENTHS)
14820	12	1	8		20	6	35	9	3
14820	12	1	9		21	11	40	9	3
14820	12	1	10		23	11	46	9	3
14820	12	1	11		23	17	49	9	3
14820	12	1	12	250	23	16	48	9	6
14820	12	1	13	100	23	16	48	10	7
14820	12	1	14	100	23	11	48	10	10
14820	12	1	15	110	22	10	48	10	10
14820	12	1	16	110	22	10	49	10	10
14820	12	1	17	65	22	9	49	10	10
14820	12	1	18	60	23	13	49	10	10
14820	12	1	19	120	25	9	47	10	10
14820	12	1	20	130	22	7	46	10	10
14820	12	1	21	65	24	6	46	10	10
14820	12	1	22	250	24	7	46	9	7
14820	12	1	23	130	25	9	45	10	10
14820	12	2	0	250	25	6	44	10	9
14820	12	2	1	130	22	5	42	10	8
14820	12	2	2	130	24	5	41	9	9
14820	12	2	3		24	5	40	4	4
14820	12	2	4	130	26	4	40	10	9
14820	12	2	5	130	34	6	40	10	9
14820	12	2	6	130	34	5	40	9	8
14820	12	2	7	130	34	8	40	9	8
14820	12	2	8		35	6	38	10	4
14820	12	2	9		35	7	39	10	4
14820	12	2	10		2	11	41	8	4
14820	12	2	11	24	36	8	42	8	8
14820	12	2	12	25	4	9	44	7	7
14820	12	2	13	130	3	10	42	7	7
14820	12	2	14	130	3	9	41	8	8
14820	12	2	15	130	5	6	40	9	8
14820	12	2	16	130	4	6	39	9	8
14820	12	2	17	110	4	11	38	10	10
14820	12	2	18	95	4	6	38	10	10
14820	12	2	19	110	6	11	37	10	10
14820	12	2	20	95	7	14	37	10	10
14820	12	2	21	90	7	7	37	10	10
14820	12	2	22	85	6	6	37	10	10
14820	12	2	23	90	7	15	37	10	10
14820	12	3	0	90	7	11	38	10	10
14820	12	3	1	70	10	12	38	10	10
14820	12	3	2	30	10	18	36	10	10
14820	12	3	3	36	8	12	32	10	10
14820	12	3	4	11	7	16	33	10	10
14820	12	3	5	13	9	16	35	10	10
14820	12	3	6	13	12	13	35	10	10
14820	12	3	7	12	12	17	36	10	10
14820	12	3	8	12	13	18	38	10	10
14820	12	3	9	14	13	24	40	10	10
14820	12	3	10	17	14	18	42	10	10
14820	12	3	11	17	15	18	45	10	10

APPENDIX K-1. CLEVELAND-HOPKINS AIRPORT HOURLY SURFACE OBSERVATIONS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	HOUR	CEILING (100s FT)	WIND DIR. (10s DEG.)	WIND SPEED (KTS)	DRY BULB (F)	CLOUD COVER (TENTHS)	TOTAL OPAQUE CLOUD COVER (TENTHS)
14820	12	3	12	15	17	19	47	10	10
14820	12	3	13	13	17	17	49	10	10
14820	12	3	14	14	18	20	51	10	10
14820	12	3	15	15	19	20	54	10	10
14820	12	3	16	26	18	21	56	10	10
14820	12	3	17	28	19	25	57	10	10
14820	12	3	18	16	29	18	53	10	10
14820	12	3	19	26	24	7	50	10	10
14820	12	3	20	26	31	11	50	10	10
14820	12	3	21	70	29	10	48	10	10
14820	12	3	22	28	29	12	45	10	10
14820	12	3	23	33	24	19	41	10	10
14820	12	4	0	32	24	20	41	10	10
14820	12	4	1	31	25	18	38	10	10
14820	12	4	2	34	24	22	37	10	10
14820	12	4	3	32	25	21	35	10	10
14820	12	4	4	31	23	20	34	10	10
14820	12	4	5	24	23	18	33	10	10
14820	12	4	6	26	23	19	32	10	10
14820	12	4	7	26	23	21	32	10	10
14820	12	4	8	23	23	22	31	10	10
14820	12	4	9	26	24	18	30	10	10
14820	12	4	10	20	24	18	30	10	10
14820	12	4	11	20	25	14	30	10	10
14820	12	4	12	20	25	14	29	10	10
14820	12	4	13	20	25	14	29	10	10
14820	12	4	14	16	24	16	29	10	10
14820	12	4	15	13	26	12	29	10	10
14820	12	4	16	55	25	10	29	10	10
14820	12	4	17	80	25	10	29	10	10
14820	12	4	18	12	27	8	27	10	10
14820	12	4	19	14	28	13	27	10	10
14820	12	4	20	29	30	13	27	10	10
14820	12	4	21	28	31	10	26	10	10
14820	12	4	22	26	29	13	27	10	10
14820	12	4	23	29	31	14	26	10	10
14820	12	5	1	29	31	14	27	10	10
14820	12	5	2	29	32	13	28	10	10
14820	12	5	3	26	30	12	28	10	10
14820	12	5	4	26	30	10	28	10	10
14820	12	5	5	29	31	9	28	10	10
14820	12	5	6	29	30	8	29	10	10
14820	12	5	7	29	30	7	29	10	10
14820	12	5	8	28	33	8	29	10	10
14820	12	5	9	29	31	9	30	10	10
14820	12	5	10	28	29	8	30	10	10
14820	12	5	11	26	25	8	28	10	10
14820	12	5	12	33	24	10	29	10	10
14820	12	5	13	32	26	10	29	10	10
14820	12	5	14	26	24	8	29	10	10
14820	12	5	15		25	10	29	8	3
14820	12	5	16		23	12	31	5	2

APPENDIX K-1. CLEVELAND-HOPKINS AIRPORT HOURLY SURFACE OBSERVATIONS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	HOUR	CEILING (100s FT)	WIND DIR. (10s DEG.)	WIND SPEED (KTS)	DRY BULB (F)	CLOUD COVER (TENTHS)	TOTAL OPAQUE CLOUD COVER (TENTHS)
14820	12	5	17		22	14	31	10	4
14820	12	5	18		22	9	31	9	1
14820	12	5	19		19	8	30	8	1
14820	12	5	20		19	16	31	7	0
14820	12	5	21		21	14	31	7	2
14820	12	5	22		21	14	31	8	2
14820	12	5	23		21	12	31	8	2
14820	12	6	0		21	13	32	8	2

APPENDIX K-2. BUFFALO INTERNATIONAL AIRPORT MIXING HEIGHTS
(11/27/90 - 12/05/90)

STATION ID	MONTH	DAY	MORNING MIXING HEIGHT (m)	AFTERNOON MIXING HEIGHT (m)
14733	11	27	546	
14733	11	28	272	1447
14733	11	29	1860	1957
14733	11	30	1208	1162
14733	12	01	787	1499
14733	12	02	684	1313
14733	12	03	927	313
14733	12	04	1305	1110
14733	12	05	739	

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