√

DoE/P /79798-T19

DOE/PC/79798--T19

DE92 014631

Environmental Monitoring for the DOE Coolside and LIMB Demonstration Extension Projects

Final Report for the Period of May, June, July, and August 1991

March 1992

Prepared for:
Babcock and Wilcox
20 S. Van Buren Avenue
Post Office Box 351
Barberton, Ohio 44203

Prepared by:
Radian Corporation
3200 East Chapel Hill Road/Nelson Highway
Post Office Box 13000
Research Triangle Park, North Carolina 27709

RADIAN
CORPORATION

MASTER

DISTRIBUTION OF THIS DOC MENT IS UNLIMITED

DCN: 91-209-026-07

ENVIRONMENTAL MONITORING FOR THE DOE COOLSIDE AND LIMB DEMONSTRATION EXTENSION PROJECTS

FINAL REPORT FOR THE PERIOD OF MAY, JUNE, JULY, AND AUGUST 1991

Prepared For:

Paul Nolan
Project Manager
Babcock and Wilcox
20 S. Van Buren Avenue
Post Office Box 351
Barberton, Ohio 44203

Prepared by:

Ted White
Luke Contos
Lucy Adams
Radian Corporation
Progress Center
3200 East Chapel Hill Road/Nelson Highway
Post Office Box 13000
Research Triangle Park, NC 27709

March 1992

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

LIST OF TABLES

Table Page

LIST OF TABLES (Continued)

LIST OF FIGURES

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 seventh EMP status report to be published and presents the data generated during May, June, July, and August 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's) 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 (SO_2) 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 that occurred during the May, June, July, and August 1991 reporting period.

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

- lignosulfonated lime/nominal 3.8 percent sulfur coal, 05/06 - 05/15/91;
- lignosulfonated lime/nominal 1.6 percent sulfur coal, 05/16 - 07/05/91;
- lignosulfonated lime/nominal 3.8 percent sulfur coal, 07/06 - 07/07/91;
- calcitic lime/nominal 3.8 percent sulfur coal, 07/08 - 07/19/91;
- calcitic lime/nominal 1.6 percent sulfur coal, 07/20 - 08/26/91; and
- calcitic lime/nominal 3.8 percent sulfur coal, 08/27 - 08/30/91.

The monitoring data and air quality modeling data presented in this report are based on emissions 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 the 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 FGI and Dry SO_2 Control Symposium held 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 SO_2 and NO_x emission averages should not be taken as representative of long-term, optimized operations. Ranges of SO_2 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 SO_2 mass emission rate was highest during the calcitic lime/nominal 3.8 percent sulfur coal injection period and lowest during the calcitic lime/nominal 1.6 percent sulfur coal injection period. The SO_2 mass emission rate during this reporting period varied from 1,100 to 3,600 lb/hr. The average SO_2 mass emission rate for each injection period, which ranged from 1,600 to 2,700 lb/hr, was higher than average SO_2 mass emission rate during the Baseline period of 932 lb/hr. Sulfur dioxide removal efficiencies for the reporting period varied widely, from 3 to 56 percent. The median SO_2 removal efficiency was the highest (37 percent) during the calcitic lime/nominal 3.8 percent sulfur coal injection period, and the lowest (20 percent) during the lignosulfonated lime/nominal 1.6 percent sulfur coal injection period.

The NO_x mass emission rates for the four sorbent/coal combinations ranged from 190 to 480 lb/hr during this reporting period. The average NO_x mass emission rates for each injection period, which ranged from 300 to 340 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 decreased when compared with emissions that occurred prior to the EPA LIMB Demonstration. 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 phosphorous (P) analyses were performed during this reporting period. Additional information on wastewater monitoring is presented in Section 4.2.

Fly ash samples were composited during four sorbent/coal combinations. Results are reported for the lignosulfonated lime/nominal 3.8 percent sulfur coal, lignosulfonated lime/nominal 1.6 percent sulfur coal, calcitic lime/nominal 3.8 percent sulfur coal, and calcitic lime/nominal 1.6 percent sulfur coal injection periods during the February, March, and April 1991 reporting period, and for this reporting period. 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 allow for the analyses of the targeted parameters listed in the EMP. The results from each of these analyses are summarized in Tables 2-2 and 2-3. The corrosivities of the lignosulfonated lime/nominal 3.8 percent sulfur coal, lignosulfonated lime/nominal 1.6 percent sulfur coal, calcitic lime/nominal 3.8 percent sulfur coal, and calcitic lime/nominal 1.6 percent sulfur coal injection period ash samples were below the detection limit. All ash samples were less permeable than Baseline ash. The metal concentrations of the TCLP and DI leachates for all ash samples were below the TCLP maximum contaminant levels (MCL's) and below the detection limit for arsenic (As), 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 for the three sorbent/coal combinations of the previous reporting period and the four sorbent/coal combinations of this reporting period. Modeled injection periods included the following five sorbent/coal combinations: (1) dolomitic lime/nominal 3.8 percent sulfur coal,

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

	SORBENT/COAL COMBINATION			
Parameter[a]	Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal	Calcitic Lime/Nominal 3.8 Percent Sulfur Coal	Calcitic Lime/Nominal 1.6 Percent Sulfur Coal
Average As	NA[b]	-[c]	NA	-
Average Ca	+[d]	-	+	+
Average TSS	-	-	-	-
Average O&G	-	+	+	+
Max pH	+	+	+	+
Min pH	+	+	+	+

[a] As = arsenic, Ca = calcium, TSS = total suspended solids, and O&G = oil and grease.
[b] NA = no analysis during test period.
[c] - = decrease from Baseline concentration.
[d] + = increase from Baseline concentration.

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

	CONCENTRATION COMPARED TO BASELINE			
	SORBENT/COAL COMBINATION			
Parameter	**Lignosulfonated Lime/3.8% Sulfur Coal**	**Lignosulfonated Lime/1.6% Sulfur Coal**	**Calcitic Lime/3.8% Sulfur Coal**	**Calcitic Lime/1.6% Sulfur Coal**
Total phenolics	+[a]	-[b]	-	-
pH	+	+	+	+
Calcium	+	-	+	+
Chloride	+	+	+	+
Fluoride	-	-	-	-
Potassium	+	+	+	+
Sodium	-	-	-	-
Nitrate	+	-	+	+
Sulfate	+	-	+	+
Total hardness	NA[c]	NA	NA	NA
Total alkalinity as CaCo$_3$	+	+	+	+
Acidity	NA	NA	NA	NA
Bicarbonate	BDL[d]	BDL	BDL	BDL
Carbonate	+	+	+	+
Chemical oxygen demand (COD)	+	+	+	+
Total organic carbon (TOC)	-	-	-	+
Total dissolved solids (TDS)	+	-	+	+
Iron	NA	NA	NA	NA
Copper	NA	NA	NA	NA
Magnesium	-	-	-	-
Silver	BDL	BDL	BDL	BDL
Arsenic	-	+	-	-
Barium	+	+	+	+
Cadmium	-	-	-	-
Chromium	-	-	-	-
Mercury	BDL	BDL	BDL	BDL
Lead	-	-	-	-
Selenium	-	-	+	+

[a] + = Higher than Baseline concentration.
[b] - = Lower than Baseline concentration.
[c] NA = No analysis during test period.
[d] BDL = Both Baseline and test period concentrations are below detection limits.

TABLE 2-3. COMPARISON OF ASH TCLP LEACHATE COMPOSITION DATA DURING BASELINE AND EXTENSION PERIODS OF OPERATION

	CONCENTRATION COMPARED TO BASELINE			
	SORBENT/COAL COMBINATION			
Parameter	**Lignosulfonated Lime/3.8% Sulfur Coal**	**Lignosulfonated Lime/1.6% Sulfur Coal**	**Calcitic Lime/3.8% Sulfur Coal**	**Calcitic Lime/1.6% Sulfur Coal**
Silver	NC[a]	NC	NC	NC
Arsenic	NC	-[b]	NC	NC
Barium	+[c]	+	+	+
Cadmium	NC	NC	NC	NC
Chromium	-	+	NC	NC
Mercury	BDL[d]	BDL	BDL	BDL
Lead	NC	NC	NC	NC
Selenium	-	+	-	-

[a] NC = Test period concentration is below detection limit. No comparison between Baseline and test period concentrations is possible, because the detection limit of the test period is greater than the Baseline concentration.

[b] - = Lower than Baseline concentration.

[c] + = Higher than Baseline concentration.

[d] BDL = Both Baseline and test period concentrations are below detection limits.

(2) lignosulfonated lime/nominal 1.6 percent sulfur coal, (3) lignosulfonated lime nominal 3.8 percent sulfur coal, (4) calcitic lime/nominal 1.6 percent sulfur coal, and (5) calcitic lime/nominal 3.8 percent sulfur coal. The modeling demonstrated that SO_2 and NO_x concentrations increased over Baseline period concentrations. The model predicted concentrations for NO_x were below the ambient air significance levels, as defined in the Prevention of Significant Deterioration (PSD) air regulations (40 CFR 51.165(b)(2)). The 24-hour (5 $\mu g/m^3$) and annual (1.0 $\mu g/m^3$) significance levels for SO_2 were exceeded during all the injection periods. The three-hour (25 $\mu g/m^3$) significance levels for SO_2 were exceeded during all the injection periods, with the exception of the calcitic lime/nominal 1.6 percent sulfur coal injection period. Additional modeling, using the Industrial Source Complex Short Term (ISCST) dispersion model, was conducted to determine compliance with the National Ambient Air Quality Standards (NAAQS) for SO_2 during the February through August 1991 reporting periods. 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 5.0.

The employee health and safety monitoring specified in the EMP has been completed. 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

This 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 SO_2 and NO_x emissions from existing utility power generation plants using cost-effective retrofit technologies. Specific goals of the EPA program were to demonstrate a 50 to 60 percent reduction of SO_2 emissions based on incoming coals containing a nominal 3 percent sulfur. Emissions of NO_x were expected to be less than 0.5 lb/million Btu heat input. LIMB has the potential to reduce SO_2 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.

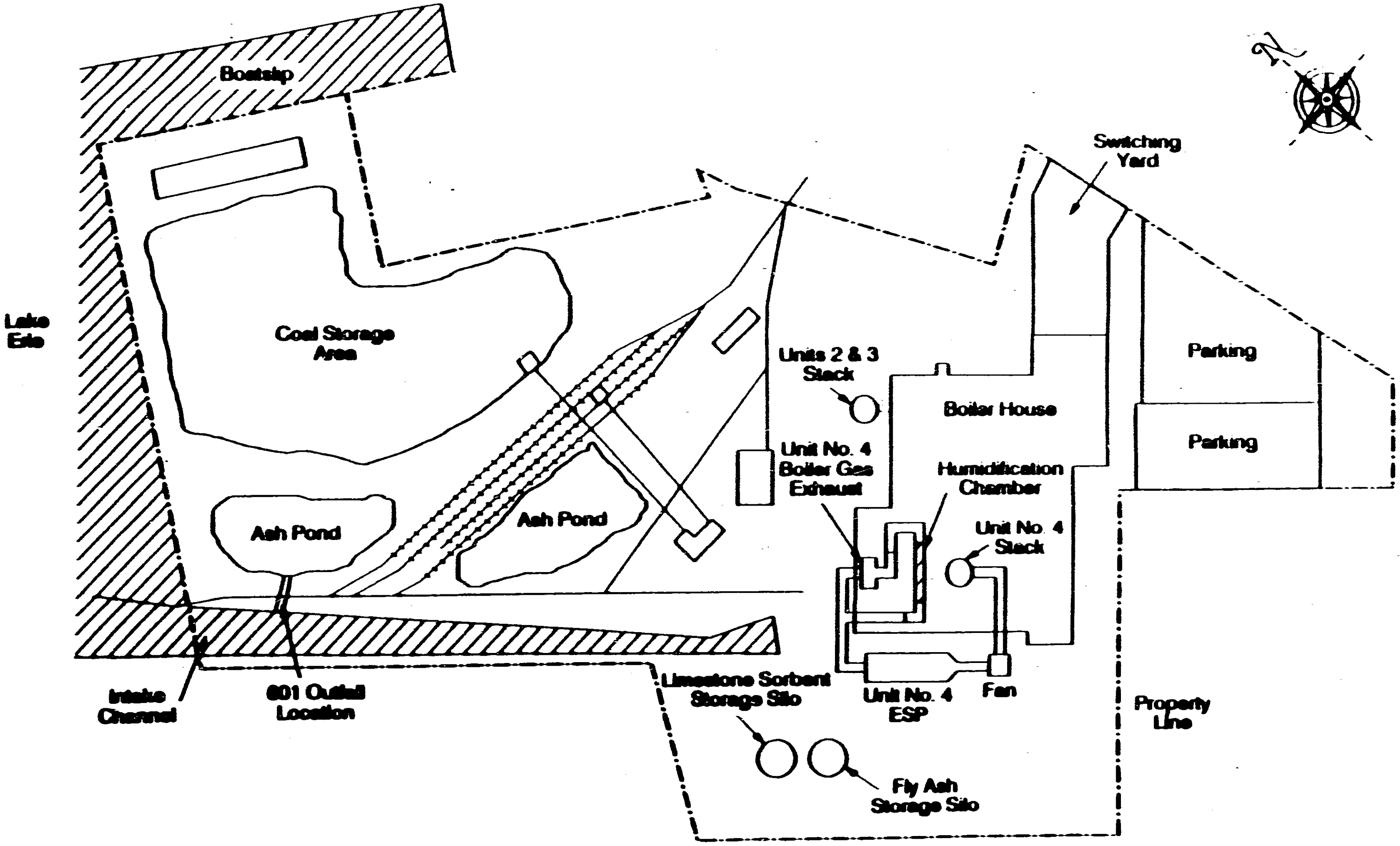


FIGURE 3-1. EDGEWATER PLANT SITE LAYOUT

The DOE LIMB Demonstration Project Extension adds a sorbent injection point located upstream of the flue gas humidification to the original boiler. The additional 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 SO_2 removal. Figure 3-2 presents a generalized schematic of the current sorbent injection configurations present at the Edgewater facility.

To accurately document and analyze SO_2 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 SO_2, NO_x, oxygen (O_2), carbon dioxide (CO_2), 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 -- SO_2, NO_x, CO, O_2, and CO_2 -- are monitored at the ESP outlet. Radian also monitors and records component concentrations with a separate personal computer-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

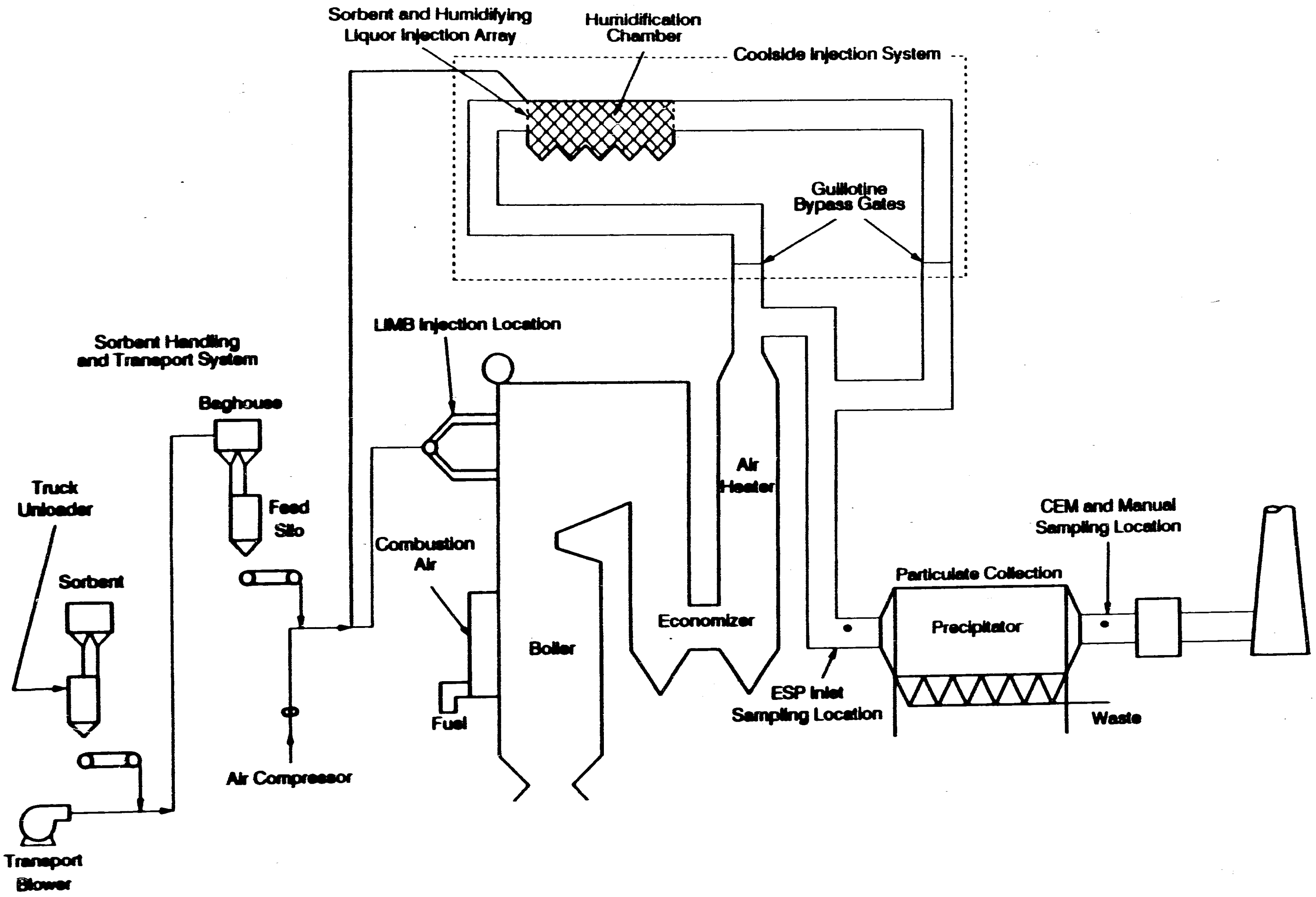


FIGURE 3-2. LIMB INJECTION AND COOLSIDE INJECTION LOCATIONS AND FLUE GAS SAMPLING LOCATIONS FOR THE DOE LIMB EXTENSION PROJECT

monitoring throughout the Demonstration. The following testing phases were conducted during the EPA Demonstration:

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 began 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 involved sorbent injection into the boiler, in conjunction with the humidification chamber operation to maintain ESP performance. When load conditions permitted, tests were run close at a 20°F approach to adiabatic 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 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 coat was burned, a number of short sorbent/coal injection test periods (each less than eight hours per day) followed; this was a format that continued to the end of the Demonstration. 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/nominal 3.0 percent sulfur coal; (3) dolomitic lime/nominal 3.8 percent sulfur coal; and (4) limestone/nominal 1.6 percent sulfur coal. The coal and sorbent combinations tested during the February, March, and April 1991 reporting period were: (1) dolomitic lime/nominal 3.8 percent sulfur coal; (2) lignosulfonated lime/nominal 1.6 percent sulfur coal; and (3) lignosulfonated lime/nominal 3.8 percent sulfur coal.

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

- lignosulfonated lime/nominal 3.8 percent sulfur coal, 05/06 - 05/15/91;
- lignosulfonated lime/nominal 1.6 percent sulfur coal, 05/16 - 07/05/91;
- lignosulfonated lime/nominal 3.8 percent sulfur coal, 07/06 - 07/07/91;

- calcitic lime/nominal 3.8 percent sulfur coal, 07/08 - 07/19/91;
- calcitic lime/nominal 1.6 percent sulfur coal, 07/20 - 08/26/91; and
- calcitic lime/nominal 3.8 percent sulfur coal, 08/27 - 08/30/91.

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 were 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 during this reporting period.

A summary of average air emissions data is presented in Table 4-1, by test period. Average values in the table 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]
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal: 05/06 through 05/15, and 07/06 through 07/07.								
Average	59	12,381	1.0	3.6	2,600	30	0.42	340
Maximum				4.4	3,300	56	0.47	480
Minimum				2.9	1,800	3	0.37	220
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal: 05/16 through 07/05.								
Average	58	12,413	1.8	2.4	1,700	25	0.42	300
Maximum				3.4	2,500	50	0.49	470
Minimum				1.7	1,100	9	0.36	190
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal: 07/08 through 07/19, and 08/27 through 08/30.								
Average	55	12,491	2.1	4.0	2,700	37	0.44	300
Maximum				4.8	3,600	52	0.48	400
Minimum				2.5	1,900	16	0.35	180
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal: 07/20 through 08/26.								
Average	52	12,418	1.3	2.4	1,600	20	0.47	300
Maximum				2.8	2,100	55	0.49	400
Minimum				2.1	1,100	5	0.42	200
Overall Reporting Period Average 05/06/91 through 08/30/91.								
Average	57	12,426	1.7	3.0	2,200	29	0.43	310
Maximum				4.8	3,600	56	0.49	480
Minimum				1.7	1,100	3	0.36	190
Baseline Period: 02/17 through 04/22/90.[d]								
Average	53	11,680	1.3	1.4	932	NA[e]	0.28[f]	181[f]
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] These values calculated as: lbs/hr=[(lbs/MMBtu)*(Klb/hr)*(Btu/lb)*(1000lb/Klb)/(10E6Btu/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 from the February, March and April 1990 Report.

[e] NA = Not Applicable.

[f] 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 may be lower than actual emissions.

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 SO_2 Control Symposium, held 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 O_2 and CO_2 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 therefore is also not included in the summary tables. Monitoring data for O_2, CO_2, and CO would only be evaluated and reported if modeling results indicated that ambient concentrations of SO_2 or NO_x may be higher than the NAAQS. The results from total hydrocarbon (THC) testing, which were below 1 ppmv for the EPA portion of the base LIMB testing project (Baseline Report, 1988), demonstrated that no further THC monitoring was required.

The testing that occurred during this reporting period was performed during short time periods (two to six hours per day) when nominal 1.6 percent sulfur coal was burned. However, when high sulfur coal was burned, testing took place continuously.

During this reporting period, the average SO_2 mass emission rate was highest during the calcitic lime/nominal 3.8 percent sulfur coal injection period and lowest during the calcitic lime/nominal 1.6 percent sulfur coal injection period. The SO_2 mass emission rate during this reporting period varied from 1,100 to 3,600 lb/hr. The average SO_2 mass emission rate for each injection period, which ranged from 1,600 to 2,700 lb/hr, was higher than average SO_2 mass emission rate during the Baseline period of 932 lb/hr. Removal efficiencies for SO_2 for the reporting period varied widely, from 5 to 56 percent. The median SO_2 removal efficiency was the highest during the calcitic lime/nominal 3.8 percent sulfur coal injection period (37 percent) and was lowest during calcitic lime/nominal 1.6 percent sulfur coal injection period (20 percent).

The NO_x mass emission rates for the four sorbent/coal combinations ranged from 190 to 480 lb/hr this reporting period. The average NO_x mass emission rates for each injection period, which ranged from 300 to 340 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 that occurred prior to the EPA LIMB Demonstration. Modeled ambient air impacts from SO_2 and NO_x emissions during these six injection periods will be presented in Section 5.0 of this report.

The average opacity during each injection period ranged from 1.0 to 2.1 percent, as compared to average opacity during the Baseline period of 1.3 percent. As demonstrated in previous reporting periods, the opacity remained well below the state of Ohio permit limit of 20 percent. Injection periods when lignosulfonated lime/nominal 3.8 percent sulfur coal was used had average opacities below the Baseline period average opacity. The average higher heating value 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 the LIMB Demonstration Extension: Emission Test Report Calcium Chloride Injection Study and the Draft LIMB Demonstration Extension Quality Assurance Project Plan. Testing for PM and PM_{10} 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 four to eight hour periods precluded the PM or PM_{10} 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 as required by the NPDES permit was conducted. Monthly discharge reports are submitted by Ohio Edison to the Ohio EPA for Outfalls 601, 606, 001, and 002 for the following parameters: pH, TSS (referred to in the permit as nonfilterable residue), flow, O&G, and As.

Ohio Edison measures pH at Outfall 601 daily. Samples are collected between the primary and secondary setting ponds. TSS and flow are measured twice a week; O&G and As are measured once a month. All discharge parameters monitored during this reporting period were within NPDES permit requirements. Appendix B provides NPDES analytical data for the months of May, June, July, and August 1991. Table 4-2 shows a comparison of the Baseline values versus the average, maximum, and minimum values for each parameter recorded by Ohio Edison during the month indicated.

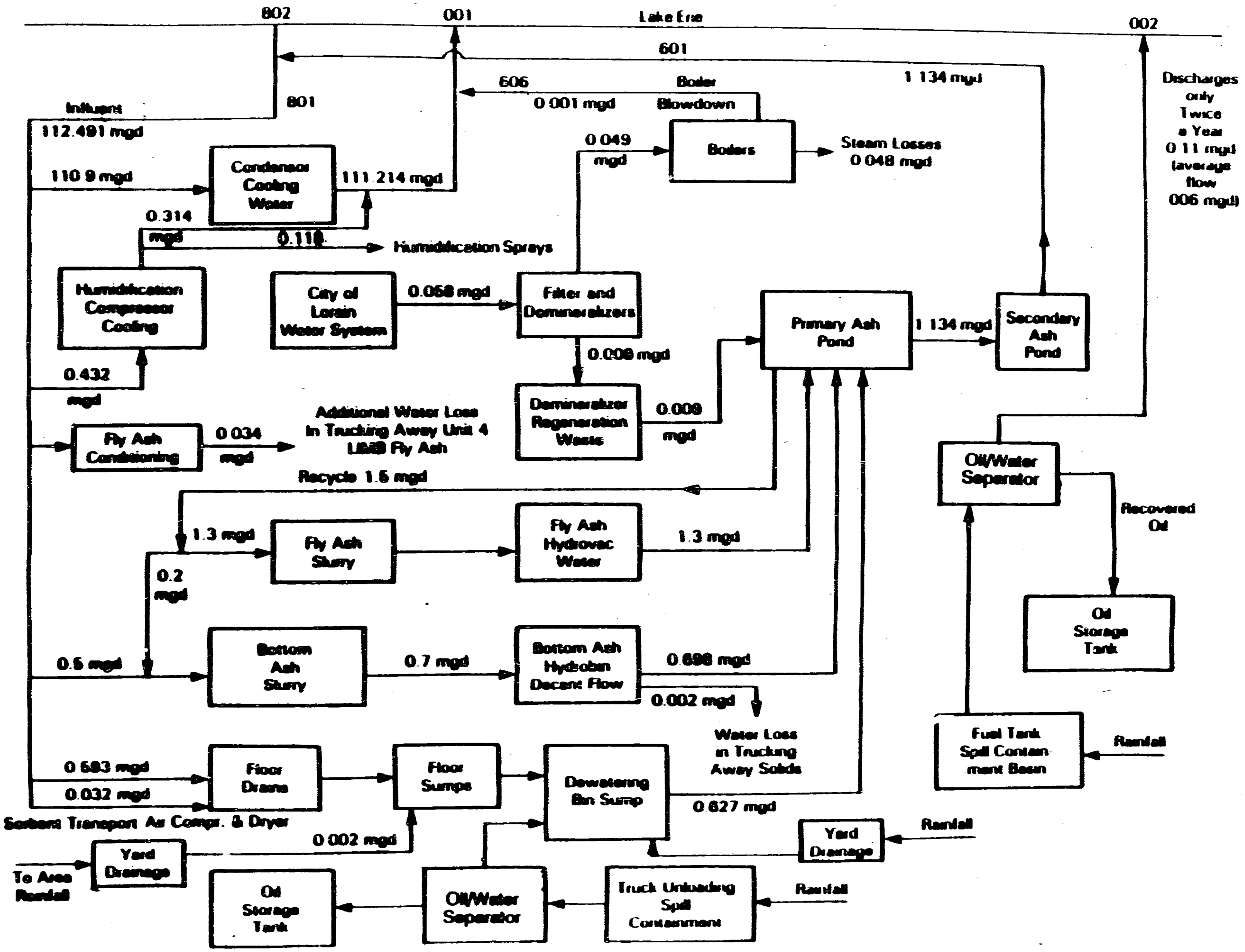


FIGURE 4-1. FLOW DIAGRAM OF EDGEWATER PLANT WATER FLOWS AND OUTFALLS

TABLE 4-2. NPDES OUTFALL 501 MONITORING DATA MAY, JUNE, JULY, AND AUGUST 1991

Reporting Period	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	--	--
May 1991[a]						
Average	--	6	1.5	BDL[b]	NA[c]	NA
Maximum	7.9	13	2.1	BDL	NA	NA
Minimum	6.8	1	0.7	BDL	NA	NA
June 1991[a]						
Average	--	6	1.4	3[d]	NA	BDL[d]
Maximum	7.8	16	2.1	3[d]	NA	BDL[d]
Minimum	7.2	1	0.7	3[d]	NA	BDL[d]
July 1991[a]						
Average	--	3	1.6	2[d]	NA	NA
Maximum	8.3	6	2.1	2[d]	NA	NA
Minimum	7.1	1	1.1	2[d]	NA	NA
August 1991[a]						
Average	--	4	1.3	2[d]	NA	7[d]
Maximum	8.0	8	2.4	2[d]	NA	7[d]
Minimum	6.5	2	0.2	2[d]	NA	7[d]
Baseline[e]						
(2/17-4/2290)						
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] BDL = Below detection limits.
[c] NA = Not analyzed during test period.
[d] Single data point for the month.
[e] Analytical data from February, March, and April 1990 Report.

Table 4-3 contains the same Ohio Edison monitoring data, categorized by sorbent/coal combination.

Radian monitored pH and temperature daily at Outfall 601 at the discharge into Lake Erie. Radian also composited daily wastewater samples for Ca analysis during the four sorbent/coal combinations. Radian's measurements were made independently from Ohio Edison's monitoring program. Radian and Ohio Edison took samples at different locations and at different times of the day, therefore, there is no direct correlation between the two sets of measurements. Radian's temperature and pH data are shown in Appendix C for the period of May, June, July, and August 1991. The Outfall 601 Ca analysis results are shown in Appendix D. Table 4-4 presents Radian's supplemental pH and Ca concentration data for Outfall 601 with the data categorized by sorbent/coal combination.

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. The bottom ash was not analyzed, since LIMB Extension activities were not expected to impact bottom ash generation, and the quantity of bottom ash generated was a small percentage of the total ash produced. Only fly ash was sampled and analyzed during the LIMB Extension periods of operation. Samples were collected for compositing daily at the bottom of the ash storage site.

Four fly ash samples were composited and analyzed from ash collected during the reporting period. Ash was composited from samples taken during each of the four test conditions as shown in Table 4-5. The Baseline ash sample was composited from samples taken from February 7 through April 22, 1990.

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

Reporting Period	Parameters					
Average, Maximum, and Minimum	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	--	--
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal: 5/6 through 5/15/91, and 7/6 through 7/7/91[a]						
Average	--	4.3	1.5	BDL[d]	NA[c]	NA
Maximum	7.8	6	2.1	BDL	NA	NA
Minimum	7.4	2	0.7	BDL	NA	NA
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal: 5/16 through 7/5/91[a]						
Average	--	5.9	1.5	3[d]	NA	BDL
Maximum	7.9	16	2.1	3[d]	NA	BDL
Minimum	7.2	1	0.7	3[d]	NA	BDL
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal: 7/8 through 7/19/91, and 8/27 through 8/30/91[a]						
Average	--	4.3	1.7	2[d]	NA	NA
Maximum	8.3	8	2.4	2[d]	NA	NA
Minimum	7.0	2	1.4	2[d]	NA	NA
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal: 7/20 through 8/26/91[a]						
Average	--	3.8	1.3	2[d]	NA	7[d]
Maximum	8.0	8	1.7	2[d]	NA	7[d]
Minimum	6.5	2	0.2	2[d]	NA	7[d]
Overall Reporting Period Average: 05/01 through 08/31/91[a]						
Average	--	4.9	1.5	2.3	NA	7[d]
Maximum	8.3	16	2.4	3	NA	7[d]
Minimum	6.5	1	0.2	2	NA	7[d]
Baseline Period: 02/17 through 4/22/90[e]						
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	BDL	0.05	48

[a] Analytical data shown in Appendix B.

[b] Below detection limits.

[c] NA = Not analyzed during test period.

[d] Single data point for the period.

[e] Analytical data from February, March, and April 1990 Report.

TABLE 4-4. OUTFALL 601 pH AND Ca CONTENT[a,b]

Sorbent/Coal Combination	Date of Sampling	Maximum pH (s.u.)	Minimum pH (s.u.)	Average Ca (mg/L)
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	5/14-5/16/91	7.48	7.31	65
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal	5/22-6/28/91	7.45	7.01	40
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	7/1-7/12/91	7.42	7.26	57
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal	7/15-7/19/91	7.63	7.37	59
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal	7/22-8/28/91	7.42	6.85	46
Baseline[c]	2/17-4/22/90	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 Report.

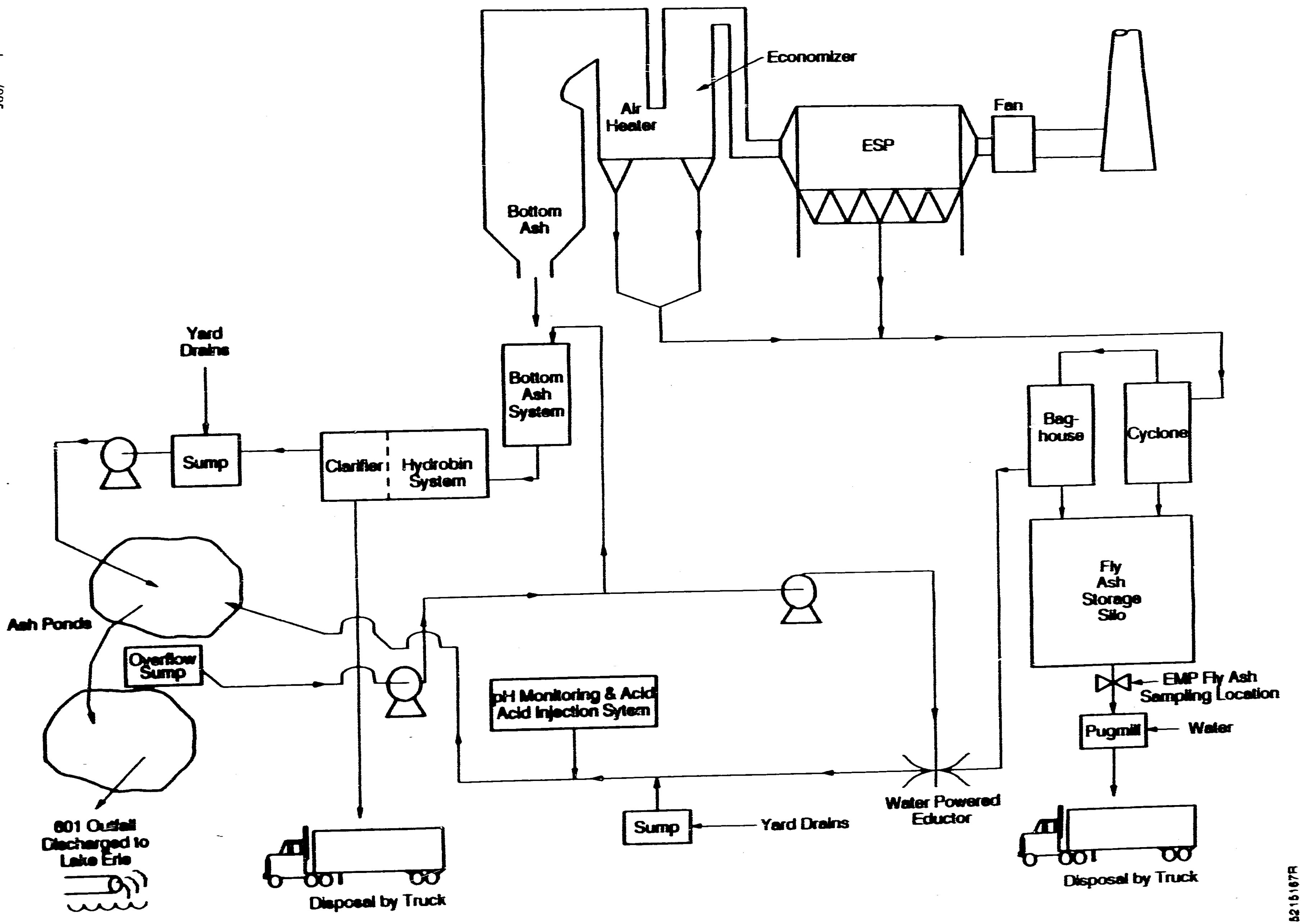


FIGURE 4-2. EDGEWATER ASH HANDLING SYSTEM

TABLE 4-5. SUMMARY OF ASH SAMPLES COLLECTED

Ash Sample	Collection Period	Test Condition
EMPASH-12	April 15 - May 16	Lignosulfonated Lime/ 3.8 Percent Sulfur Coal
EMPASH-13	May 22 - July 1	Lignosulfonated Lime/ 1.6 Percent Sulfur Coal
EMPASH-14	July 8 - July 19	Calcitic Lime/3.8 Percent Sulfur Coal
EMPASH-15	July 22 - July 31	Calcitic Lime/1.6 Percent Sulfur Coal
BASELINE	February 17 - April 22, 1990	No Sorbent Injection

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 ESP during these injection and noninjection 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 23 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 Procedures 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 is an acid extraction (pH < 5.0) and is not suitable for many of the non-metal parameters. The neutral DI leaching procedures (ASTM D3987) was selected to produce a leachate suitable for analysis for the following secondary parameters: total phenolics, pH, Ca, chloride (Cl), fluoride (Fl), 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 two sorbent/coal injection periods are compared to the Baseline in Table 4-6. Tests of the Baseline ash demonstrated a corrosivity of 1.3 mm/yr. Corrosivities of all other ash samples analyzed during this reporting period were below the detection limits for the test. The permeability of the ash from each of the four sorbent/coal injection periods was lower than that of the Baseline ash sample. This decrease in permeability 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 DI leachates from the four sorbent/coal combinations are compared to the Baseline values in Table 4-7.

TABLE 4-6. ESP ASH CORROSIVITY AND PERMEABILITY RESULTS

Condition	Corrosivity[a] (mm/yr)	Permeability[b] (cm/sec)
Lignosulfonated Lime/3.8 Percent Sulfur Coal	BDL[c]	9.9×10^{-6}
Lignosulfonated Lime/1.6 Percent Sulfur Coal	BDL	3.1×10^{-6}
Calcitic Lime/3.8 Percent Sulfur Coal	BDL	7.8×10^{-6}
Calcitic Lime/1.6 Percent Sulfur Coal	BDL	3.2×10^{-5}
Baseline	1.3	5.3×10^{-4}

[a] Conducted on DI leachate sample.
[b] Analytical data shown in Appendix G.
[c] BDL = Below detection limits.

TABLE 4-7. ANALYSIS OF FLY ASH LEACHATES FROM THE BASELINE AND EXTENSION PERIODS OF OPERATION[a]

Test Condition	Baseline Ash[b]		Calcitic Lime/ 3.8% Sulfur Coal		Calcitic Lime/ 1.6% Sulfur Coal	
Analyte	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)
Total Phenolics	0.023[c]	0.0050	BDL[d]	0.010	BDL	0.010
pH	4.0	–	12	–	12	–
Calcium	460	1.0	1700	4.0	1700	4.0
Chloride	3.2[c]	1.0	210	10	330	10
Fluoride	3.3	0.10	2.1	0.20	2.1	0.20
Potassium	BDL	3.0	7.8[c]	3.0	15	3.0
Sodium	43	1.0	3.7[c]	1.0	7.6	1.0
Nitrate	0.2	0.020	0.21	0.020	0.55	0.020
Sulfate	1700	100	2000	100	1800	100
Total Hardness	1268	66	NA[e]	–	NA	–
Total Alkalinity as $CaCO_3$	BDL	1.0	2398	1.0	2372	1.0
Acidity	250	1.0	NA	–	NA	–
Bicarbonate	BDL	1.0	BDL	1.0	BDL	1.0
Carbonate	BDL	1.0	100	1.0	123	1.0
Chemical Oxygen Deman (COD)	BDL	5.0	7.9[c]	6.0	7.9[c]	6.0
Total Organic Carbon (TOC)	2.2[c]	1.0	1.5[c]	1.0	9.6	1.0
Total Dissolved Solids (TDS)	2700	9.0	5000	1.0	5000	10
Iron	3.8	0.040	NA	–	NA	–
Copper	1.1	0.020	NA	–	NA	–
Magnesium	29	1.0	BDL	1.0	BDL	1.0
Silver	BDL	0.010	BDL	0.040	BDL	0.040
Arsenic	0.0042[c]	0.0040	BDL	0.040	BDL	0.0040
Barium	0.062	0.010	0.25	0.010	0.60	0.010
Cadmium	0.069	0.0050	BDL	0.020	BDL	0.020
Chromium	0.27	0.010	BDL	0.040	BDL	0.040
Mercury	BDL	0.0002	BDL	0.0002	BDL	0.0002
Lead	0.0097[c]	0.0030	BDL	0.0030	BDL	0.0030
Selenium	0.059	0.0050	0.073	0.010	0.100	0.010

[a] Analytical data can be found in Appendix F.

[b] Analytical data from May, June, and July 1990 Report.

[c] Estimated results less than five times the detection limit.

[d] Below detectable limits.

[e] NA = Not analyzed.

Analytical results for metals from the TCLP leachates are shown in Table 4-8. The concentration of the metals for the four injection periods, in both DI and TCLP leachates, were below detection limits for silver, Cd, and Hg. All four ash samples had barium concentrations higher than the baseline sample concentration. The concentrations of other metals in the samples were near or below the baseline concentrations.

All DI leachates from the samples had a higher pH than the Baseline sample and showed greater concentrations of the chloride, potassium, total alkalinity, COD, and carbonate. The higher concentrations were expected, due to the presence of spent sorbent in the fly ash. All samples except the lignosulfonated lime/1.6 percent sulfur coal combination had higher levels of Ca, sulfates, and TDS than the Baseline sample. Concentrations of these three analytes were noticeably lower in the DI leachate from the lignosulfonated lime/1.6 percent sulfur coal ash sample than in the DI leachates from the other three ash samples. Spent sorbent in the fly ash would be expected to cause higher concentrations of Ca and TDS than in the Baseline samples. Concentrations of fluoride, sodium, and magnesium in the DI leachates from all four fly ash samples were lower than the Baseline concentrations. 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 May, June, July, and August 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 4-9. In May 1991, the system operated for a total of 154 hours of formal testing. During June 1991, the system was off-line for 22 days. The system operated for 69.5 hours of formal

TABLE 4-8. ANALYSIS OF FLY ASH TCLP LEACHATES FROM THE BASELINE AND EXTENSION PERIODS OF OPERATION[a]

Test Condition	Baseline[b]		Lignosulfonated Lime 3.8% Sulfur Coal		Lignosulfonated Lime 1.6% Sulfur Coal		Calcitic Lime 3.8% Sulfur Coal		Calcitic Lime 1.6% Sulfur Coal	
Analyte	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)	Measured Conc. (mg/L)	Detection Limit (mg/L)
Silver	0.013[c]	0.010	BDL[d]	0.20	BDL	0.040	BDL	0.10	BDL	0.10
Arsenic	1.5	0.30	BDL	6.0	BDL	1.2	BDL	3.0	BDL	3.0
Barium	0.053	0.010	0.73	0.010	0.69	0.010	0.53	0.10	0.44[c]	0.10
Cadmium	0.013[c]	0.0050	BDL	0.10	BDL	0.020	BDL	0.050	BDL	0.050
Chromium	0.016[c]	0.010	0.010[c]	0.010	0.16	0.010	BDL	0.10	BDL	0.10
Mercury	BDL	0.0002	BDL	0.0002	BDL	0.0002	BDL	0.0002	BDL	0.0002
Lead	BDL	0.050	BDL	1.0	BDL	0.20	BDL	0.50	BDL	0.50
Selenium	0.058	0.0050	BDL	0.0050	0.075	0.0050	0.019[c]	0.0050	0.045	0.0050

[a] Analytical data from May, June, and July 1990 Report.
[b] Analytical data can be found in Appendix F.
[c] Estimated results less than five times the detection limit.
[d] BDL = Below detectable limits.

TABLE 4-9. LIMB OPERATIONS LOG FOR MAY, JUNE, JULY AND AUGUST 1991

Date	Hours of Lime Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Stoichiometry (average)	Humidifier Outlet (°F)	Daily Hours Out of Service
	From	To	From	To				
5-1-91	UNIT OFF-LINE							
5-2-91	UNIT OFF-LINE							
5-3-91	UNIT OFF-LINE							
5-4-91	UNIT OFF-LINE							
5-5-91	1800	2400	1800	2400	6	1.6	275	18
5-6-91	0000	2400	0000	2400	24	2.2	275	0
5-7-91	0000	2400	0000	2400	24	1.6	275	0
5-8-91	0000	2000	0000	2000	20	1.2-1.95	275	4
5-9-91	UNIT OFF-LINE							
5-10-91	UNIT OFF-LINE							
5-11-91	UNIT OFF-LINE							
5-12-91	UNIT OFF-LINE							
5-13-91	UNIT OFF-LINE							
5-14-91	1100	2400	1100	2400	13	2.2	275	11
5-15-91	0000	2400	0000	2400	24	1.15-1.9	275	0
5-16-91	0000	2330	0000	2330	23.5	1.2-1.5	275	0.5
5-17-91	UNIT OFF-LINE							
5-18-91	UNIT OFF-LINE							
5-19-91	UNIT OFF-LINE							
5-20-91	UNIT OFF-LINE							
5-21-91	UNIT OFF-LINE							
5-22-91	UNIT OFF-LINE							
5-23-91	1800	2200	1800	2200	4	1.6	145	20
5-24-91	0600	1000	0600	1000	4	1.3	145	20
5-25-91	UNIT OFF-LINE							
5-26-91	UNIT OFF-LINE							
5-27-91	UNIT OFF-LINE							
5-28-91	UNIT OFF-LINE							
5-29-91	0300	0600	0300	0600	3	2	145	21
5-30-91	0230	0600	0230	0600	3.5	1.8	145	20.5
5-31-91	0200	0700	0200	0700	5	1.4	145	19
6-1-91	UNIT OFF-LINE							
6-2-91	UNIT OFF-LINE							
6-3-91	UNIT OFF-LINE							

TABLE 4-9. (Continued)

Date	Hours of Lime Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Stoichiometry (average)	Humidifier Outlet (°F)	Daily Hours Out of Service
	From	To	From	To				
6-4-91	UNIT OFF-LINE							
6-5-91	UNIT OFF-LINE							
6-6-91	UNIT OFF-LINE							
6-7-91	UNIT OFF-LINE							
6-8-91	UNIT OFF-LINE							
6-9-91	UNIT OFF-LINE							
6-10-91	UNIT OFF-LINE							
6-11-91	UNIT OFF-LINE							
6-12-91	UNIT OFF-LINE							
6-13-91	200/600	1400/17	200/600	1400/17	7	.85-22	145	17
6-14-91	0100	0400	0100	0400	3	2.2	145	21
6-15-91	UNIT OFF-LINE							
6-16-91	UNIT OFF-LINE							
6-17-91	0900	1300	0900	1300	4	2.05	145	20
6-18-91	0100	1100	0100	1100	10	1.3-1.6	145	14
6-19-91	UNIT OFF-LINE							
6-20-91	UNIT OFF-LINE							
6-21-91	UNIT OFF-LINE							
6-22-91	UNIT OFF-LINE							
6-23-91	UNIT OFF-LINE							
6-24-91	1330	2400	1330	2400	10.5	1.0-2.0	275	13.5
6-25-91	0000	1900	0000	1900	19	1.6	275	5
6-26-91	1100	2200	1100	1600	5	1.0-1.8	275	19
6-27-91	UNIT OFF-LINE							
6-28-91	1100	2200	1100	2200	11	1.0-1.85	275	19
6-29-91	UNIT OFF-LINE							
6-30-91	UNIT OFF-LINE							
7-1-91	1015	2345	1015	2345	13.5	2	275	10.5
7-2-91	UNIT OFF-LINE							
7-3-91	UNIT OFF-LINE							
7-4-91	UNIT OFF-LINE							
7-5-91	1500	2400	1500	2400	9	1	275	15
7-6-91	0000	2400	0000	2400	24	1	275	0
7-7-91	0000	2400	0000	2400	24	1	275	0
7-8-91	0000	2400	0000	2400	24	1.5	275	0

TABLE 4-9. (Continued)

	Hours of Lime Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Stoichiometry (average)	Humidifier Outlet (°F)	Daily Hours Out of Service
Date	From	To	From	To				
7-9-91	0000	2400	0000	2400	24	2	275	0
7-10-91	0000	2400	0000	2400	24	0.8-1.8	275	0
7-11-91	0000	2400	0000	2400	24	1.6	275	0
7-12-91	0000	2300	0000	2300	23	1.2-1.8	275	1
7-13-91	1300	2400	1300	2400	11	1.8	275	13
7-14-91	UNIT OFF-LINE							
7-15-91	1430	2400	1430	2400	9.5	1.8-2.2	275	14.5
7-16-91	0000	2400	0000	2400	24	1.0-2.1	275	0
7-17-91	0000	2400	0000	2400	24	1.0-1.8	275	0
7-18-91	0000	2400	0000	2400	24	1.5-1.8	275	0
7-19-91	1300	2400	1300	2400	11	1.8	275	13
7-20-91	UNIT OFF-LINE							
7-21-91	UNIT OFF-LINE							
7-22-91	1400	1830	1400	1830	4.5	2	275	19.5
7-23-91	0930	1330	–	–	4	2	–	20
7-24-91	1015	1345	–	–	3.5	0.8	–	20.5
7-25-91	0930	1500	0930	1500	5.5	1.6-1.8	275	18.5
7-26-91	UNIT OFF-LINE							
7-27-91	UNIT OFF-LINE							
7-28-91	UNIT OFF-LINE							
7-29-91	UNIT OFF-LINE							
7-30-91	UNIT OFF-LINE							
7-31-91	UNIT OFF-LINE							
8-1-91	945	1815	945	1815	8.5	1.0-1.2	275	15.5
8-2-91	1130	1700	1130	1700	5.5	1.4-2.0	275	18.5
8-3-91	UNIT OFF-LINE							
8-4-91	UNIT OFF-LINE							
8-5-91	UNIT OFF-LINE							
8-6-91	1030	1600	1030	1600	5.5	2.2	275	18.5
8-7-91	1130	2400	1130	2400	12.5	1.0-2.4	275	11.5
8-8-91	1100	1600	1100	1600	5	1.4-1.7	275	19
8-9-91	1100	1630	1100	1630	5.5	.9-1.2	275	18.5
8-10-91	UNIT OFF-LINE							
8-11-91	UNIT OFF-LINE							
8-12-91	UNIT OFF-LINE							

TABLE 4-9. (Continued)

Date	Hours of Lime Injection		Hours of Humidification		Daily Hours of Formal Testing	Ca/S Stoichiometry (average)	Humidifier Outlet (°F)	Daily Hours Out of Service
	From	To	From	To				
8-13-91	UNIT OFF-LINE							
8-14-91	UNIT OFF-LINE							
8-15-91	UNIT OFF-LINE							
8-16-91	UNIT OFF-LINE							
8-17-91	UNIT OFF-LINE							
8-18-91	UNIT OFF-LINE							
8-19-91	UNIT OFF-LINE							
8-20-91	UNIT OFF-LINE							
8-21-91	UNIT OFF-LINE							
8-22-91	UNIT OFF-LINE							
8-23-91	UNIT OFF-LINE							
8-24-91	UNIT OFF-LINE							
8-25-91	UNIT OFF-LINE							
8-26-91	1400	1530	1400	1530	0	1.6	275	24
8-27-91	1500	2400	1500	2400	9	1.5	275	15
8-28-91	0000	2400	0000	2400	24	1.6	275	0
8-29-91	0000	2400	0000	2400	24	1.7	275	0
8-30-91	0000	2400	0000	2400	24	--	--	0

testing during the month. In July 1991, the system operated for a total of 213 hours of formal testing. August operations took place over 11 days, for a total of 113.5 hours. Occasional shutdowns were reported to "zero," the System 140 percent SO_2 removal efficiency.

5.0 AMBIENT MONITORING

This section presents the results of ambient air dispersion modeling and groundwater monitoring. Section 5.1 discusses ambient air impacts predicted by using dispersion models with data from the February, March, and April 1991 reporting period, and the May, June, July, and August 1991 reporting period. Section 5.2 discusses groundwater 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 $\mu g/m^3$ 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,

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

Sorbent/Coal Combination	Injection Period
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal	02/04/91 - 02/20/91
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal	02/21/91 - 04/05/91
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	04/06/91 - 04/30/91
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	05/06/91 - 05/15/91
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal	05/16/91 - 07/05/91
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal	07/06/91 - 07/07/91
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal	07/08/91 - 07/19/91
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal	07/20/91 - 08/26/91
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal	08/27/91 - 08/30/91

results for SO_2 indicated an increase in impacts over the Baseline values that were greater than the PSD significance criteria for the 3-hour, 24-hour and annual averaging periods. Therefore, additional modeling of SO_2 impacts was required. As shown in this section, additional modeling demonstrated that the SO_2 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 SO_2 and NO_x emission rates with the maximum stack exit velocity for each injection period, and the mean SO_2 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 FEBRUARY 12, 1992

Injection Period	Pollutant	Emission Rate (g/s)	Exit Velocity (m/s)	Scenario
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal 02/04/91 - 02/20/91	SO_2 NO_x	432.9 54.7	10.5 10.5	Maximum
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal 02/21/91 - 04/05/91	SO_2 NO_x	275.8 41.3	14.2 14.2	Maximum
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 04/06/91 - 04/30/91	SO_2 NO_x	393.9 45.9	14.9 14.9	Maximum
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 05/06/91 - 05/15/91	SO_2 NO_x	494.7 60.9	17.7 17.7	Maximum
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal 05/16/91 - 07/05/91	SO_2 NO_x	320.4 58.9	16.1 16.1	Maximum
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 07/06/91 - 07/07/91	SO_2 NO_x	412.9 39.2	12.6 12.6	Maximum
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal 07/08/91 - 07/19/91	SO_2 NO_x	422.9 48.3	13.8 13.8	Maximum
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal 07/20/91 - 08/26/91	SO_2 NO_x	265.2 50.0	14.2 14.2	Maximum
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal 08/27/91 - 08/30/91	SO_2 NO_x	448.8 52.7	16.0 16.0	Maximum

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 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 is identical to the one described in previous reports. The five-year analysis for the lignosulfonated lime/nominal 3.8 percent sulfur coal (7/6-7/7/91), worst-case model predicted impacts for SO_2 that were greater than the NAAQS for the 24-hour averaging period. 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 07/05 to 07/08/91. 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 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 five-part methodology described in the November 1989 to January 1990 report.

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 nine injection periods, from -0.3 $\mu g/m^3$ to 0.6 $\mu g/m^3$. The change in SO_2 concentrations is positive for all SO_2 averaging periods for each sorbent/coal injection period.

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 $\mu g/m^3$ for the annual average, 5 $\mu g/m^3$ for the 24-hour average, and 25 $\mu g/m^3$ for the 3-hour average. For NO_x emissions from all sorbent/coal injection periods, no increases exceeded the 1.0 $\mu g/m^3$ annual average, therefore, no further evaluation was necessary for this pollutant. For SO_2, only one averaging period had an increase less than its respective significance level (calcitic lime/nominal 1.6 percent sulfur coal, three-hour average); therefore, this period required no further evaluation.

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

TABLE 5-3. SCENARIO IMPACTS MINUS BASELINE RESULTS
FEBRUARY 12, 1992

Scenario	Averaging Period	SO_2 Scenario Minus Baseline ($\mu g/m^3$)	Greater Than Significance Level
Dolomitic Lime/Nominal 3.8 Percent Sulfur Coal 02/04/91 - 02/20/91	Annual 24 Hour 3 Hour	10.8 97.3 248	Yes Yes Yes
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal 02/21/91 - 04/05/91	Annual 24 Hour 3 Hour	1.7 13.5 34.8	Yes Yes Yes
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 04/06/91 - 04/30/91	Annual 24 Hour 3 Hour	8.9 80.4 206.2	Yes Yes Yes
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 05/06/91 - 05/15/91	Annual 24 Hour 3 Hour	11.3 108.6 266.9	Yes Yes Yes
Lignosulfonated Lime/Nominal 1.6 Percent Sulfur Coal 05/16/91 - 07/05/91	Annual 24 Hour 3 Hour	2.7 24.3 61.1	Yes Yes Yes
Lignosulfonated Lime/Nominal 3.8 Percent Sulfur Coal 07/06/91 - 07/07/91	Annual 24 Hour 3 Hour	14.5 125 316.7	Yes Yes Yes
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal 07/08/91 - 07/19/91	Annual 24 Hour 3 Hour	13 113.7 289.8	Yes Yes Yes
Calcitic Lime/Nominal 1.6 Percent Sulfur Coal 07/20/91 - 08/26/91	Annual 24 Hour 3 Hour	1 6.7 17.4	Yes Yes No
Calcitic Lime/Nominal 3.8 Percent Sulfur Coal 08/27/91 - 08/30/91	Annual 24 Hour 3 Hour	11 100.8 255.9	Yes Yes Yes

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 SO_2 NAAQS occurred during the lignosulfonated lime/3.8 percent sulfur coal combination (7/6-7/7/91). 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 to more accurately predict impacts during the lignosulfonated lime/3.8 percent sulfur coal injection period. Refined modeling shows that the 24-hour impact (using representative meteorological data) is 23.1 $\mu g/m^3$ 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.

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 five years of meteorological data to determine the worst-case ambient impact during a one- to two-week test period is a very conservative methodology. However, the use of

TABLE 5-4. AMBIENT AIR QUALITY MONITORING DATA

Averaging Period	SO_2 Ambient Monitored[a] Value ($\mu g/m^3$)	Monitored Year
Annual	23	1987
24-Hour[b]	83	1986
3-Hour[b]	411	1986

[a] Monitor location is 2270 East 42nd Street, Lorain, Ohio.
[b] Second-highest impacts from 1986 and 1987 data.

TABLE 5-5. TOTAL SCENARIO SO$_2$ IMPACT
FEBRUARY 12, 1992

Scenario	Averaging Period	Scenario Impact + Monitored SO Value ($\mu g/m^3$)[a]	Percent of NAAQS
Dolomitic Lime/Nominal	Annual	51.7	65%
3.8 Percent Sulfur Coal	24 Hour	344	94%
02/04/91 - 02/20/91	3 Hour	1076	83%
Lignosulfonated Lime/Nominal	Annual	42.6	53%
1.6 Percent Sulfur Coal	24 Hour	260.2	71%
02/21/91 - 04/05/91	3 Hour	862.8	66%
Lignosulfonated Lime/Nominal	Annual	49.8	4%
3.8 Percent Sulfur Coal	24 Hour	327.1	25%
04/06/91 - 04/30/91	3 Hour	1034.2	80%
Lignosulfonated Lime/Nominal	Annual	52.2	65%
3.8 Percent Sulfur Coal	24 Hour	355.3	97%
05/06/91 - 05/15/91	3 Hour	1094.9	84%
Lignosulfonated Lime/Nominal	Annual	43.6	55%
1.6 Percent Sulfur Coal	24 Hour	271	74%
05/16/91 - 07/05/91	3 Hour	889.1	68%
Lignosulfonated Lime/Nominal	Annual	55.4	69%
3.8 Percent Sulfur Coal	24 Hour	371.7	102%
07/06/91 - 07/07/91	3 Hour	1144.7	88%
Calcitic Lime/Nominal	Annual	53.9	67%
3.8 Percent Sulfur Coal	24 Hour	360.4	99%
07/08/91 - 07/19/91	3 Hour	1117.8	86%
Calcitic Lime/Nominal	Annual	41.9	52%
1.6 Percent Sulfur Coal	24 Hour	253.4	69%
07/20/91 - 08/26/91	3 Hour	845.4	65%
Calcitic Lime/Nominal	Annual	51.9	65%
3.8 Percent Sulfur Coal	24 Hour	347.5	95%
08/27/91 - 08/30/91	3 Hour	1083.9	83%

[a] Twenty-four-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 ($\mu g/m^3$)	Greater Than Significance Level
LIGNOSULFONATED LIME/NOMINAL 3.8 PERCENT SULFUR COAL (07/06 - 07/07/90)	24 hour	-23.0	No

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 groundwater 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 was conducted.

7.0 COMPLIANCE MONITORING STATUS

Compliance monitoring is required for both gaseous and aqueous discharges. Opacity measurements, particulate emissions, and SO_2 emission estimates are required to meet source permit operation requirements. Particulate emission measurements are required once every three years. Opacity measurements are monitored continuously, and SO_2 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 SO_2, opacity, or particulate loading during the May, June, July, and August 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 (lb/MMBtu)[a]	30-Day Weighted Rolling Average (lb/MMBtu)	Frequency of Monitoring	Averaging Approach	Monitoring Method	Duration of Monitoring (years)[b]
SO_2	3.4[c]	May 1991 (3.0) June 1991 (2.2) July 1991 (3.3) August 1991 (2.9)	Daily	30-day weighted rolling average	Analysis of a daily coal sample[d]	3
Opacity	20%[e]	May 1991 (1.1%) June 1991 (2.2%) July 1991 (1.8%) August 1991 (1.7%)	Continously	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 10^6 Btu actual heat input.

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

[c] Thirty-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 SO_2 emission rate is calculated from this analysis for each day, and a 30-day rolling weighted average SO_2 emission rate is calculated for each day.

[e] Up to 60 percent opacity is allowed for a duration of up to six 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. Quality Assurance/QC data for the pH, TSS, flow, O&G, 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 May, June, July, and August 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 SO_2 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 SO_2 and NO_x concentration values were recorded when continuous emissions monitoring 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.

APPENDIX A

DETAILED DAILY AVERAGE AIR EMISSIONS

APPENDIX A: DETAILED DAILY AVERAGE AIR EMISSIONS DATA

Date	Coal Firing Rate (Klb/hr)	HHV[a]	Opacity %	Outlet SO_2		SO_2 Removal %	Outlet NO_x	
				lb/MMBtu	lb/hr[b]		lb/MMBtu	lb/hr[b]
50691	57	12260	0.83	2.91	2044.52	56.11	0.43	301.44
50791	77	12363	0.56	4.11	3922.66	37.41	0.47	452.07
50891	45	12556	0.15	3.97	2267.51	41.07	0.38	215.35
51491	83	12310	0.70	2.91	2966.62	55.76	0.47	483.03
51591	57	12363	1.96	3.26	2295.91	53.08	0.37	259.99
51691	56	12400	1.44	3.35	2337.51	50.51	0.42	291.80
52391	46	12583	1.08	2.89	1659.86	19.96	0.42	242.97
52491	53	12561	1.12	2.81	1879.22	19.39	0.44	293.34
52991	76	12539	1.45	2.66	2540.56	9.08	0.49	466.77
53091	71	12466	1.14	2.25	2001.07	24.82	0.43	380.61
53191	57	12538	1.78	2.21	1580.96	18.30	0.39	278.73
average	62	12449	1.11	3.03	2317.86	35.05	0.43	333.28
maximum	83	12583	1.96	4.11	3922.66	56.11	0.49	483.03
minimum	45	12260	0.15	2.21	1580.96	9.08	0.37	215.35
61391	47	12648	3.58	1.77	1060.43	32.15	0.38	228.56
61491	62	12416	2.83	2.43	1866.44	45.87	0.41	317.44
61791	52	12414	2.54	2.29	1482.90	36.85	0.38	245.48
61891	59	12361	2.31	2.12	1546.63	23.31	0.41	300.52
62491	49	12249	2.14	2.22	1345.22	18.69	0.40	244.07
62591	60	12244	1.49	2.20	1619.77	21.41	0.42	306.72
62691	62	12151	1.13	2.35	1761.27	15.49	0.42	316.85
62891	73	12293	1.27	2.14	1918.58	23.78	0.44	396.84
average	58	12347	2.16	2.19	1575.15	27.19	0.41	294.56
maximum	73	12648	3.58	2.43	1918.58	45.87	0.44	396.84
minimum	47	12151	1.13	1.77	1060.43	15.49	0.38	228.56
70191	68	12500	2.22	1.73	1481.64	30.97	0.42	361.46
70591	43	12247	2.04	2.16	1136.46	16.72	0.36	190.89
70691	47	12393	1.10	3.09	1808.85	20.39	0.37	215.84
70791	59	12393	1.28	4.44	3274.02	3.20	0.42	310.83
70891	55	12539	1.66	3.61	2470.82	17.20	0.42	284.36
70991	42	12366	2.32	4.31	2216.68	30.17	0.35	180.91

---

[a] HHV = Higher Heating Value

[b] These values calculate as: lbs/hr = ((lbs/mmBtu)(Klb/hr)*(Btu/lb)(1000 lb/Klb)/(10E6Btu/mmBtu))

APPENDIX A: DETAILED DAILY AVERAGE AIR EMISSIONS DATA

				Outlet SO_2			Outlet NO_x	
Date	Coal Firing Rate (Klb/hr)	HHV[a]	Opacity %	lb/MMBtu	lb/hr[b]	SO_2 Removal %	lb/MMBtu	lb/hr[b]
71091	52	12306	2.34	4.53	2898.21	30.71	0.43	273.97
71191	65	12477	2.00	4.16	3353.04	37.34	0.42	336.01
71291	56	12611	2.48	4.21	2976.33	35.42	0.41	286.92
71591	44	12447	2.46	3.52	1914.80	52.79	0.47	255.42
71691	54	12477	2.52	4.80	3207.03	33.20	0.48	319.85
71791	59	12543	2.38	3.91	2898.30	43.41	0.47	350.55
71891	57	12557	1.85	4.06	2904.33	40.24	0.47	333.25
71991	63	12585	1.22	2.53	2012.36	51.31	0.48	382.97
72291	61	12411	1.10	2.28	1724.19	38.31	0.49	372.11
72391	57	12438	1.93	2.42	1721.89	8.90	0.48	344.89
72491	41	12161	1.82	2.58	1287.85	5.41	0.45	225.86
72591	49	12722	0.87	2.27	1423.94	17.03	0.49	306.12
72691	35	12834	0.84	2.45	1097.85	8.69	0.45	201.36
average	53	12474	1.81	3.32	2200.45	27.44	0.44	291.24
maximum	68	12834	2.52	4.80	3353.04	52.79	0.49	382.97
minimum	35	12161	0.84	1.73	1097.85	3.20	0.35	180.91
80191	67	12328	0.93	2.54	2102.94	18.15	0.48	396.65
80291	50	12629	1.20	2.65	1683.54	14.99	0.48	302.01
80691	43	12240	0.95	2.14	1134.19	24.76	0.42	225.02
80791	52	12257	0.55	2.14	1370.82	17.21	0.46	297.68
80891	57	12241	0.41	2.22	1542.03	19.45	0.47	323.64
80991	56	12365	2.30	2.30	1588.38	55.79	0.47	322.93
82691	58	12389	2.87	2.83	2037.68	12.03	0.45	326.36
82791	56	12588	2.74	3.35	2365.98	16.06	0.40	280.45
82891	69	12488	2.51	4.12	3558.38	30.30	0.43	374.23
82991	75	12336	2.09	3.75	3473.28	36.02	0.45	418.10
83091	71	12282	1.69	3.40	2962.15	42.15	0.46	401.65
average	60	12377	1.66	2.86	2165.40	26.08	0.45	333.52
maximum	75	12629	2.87	4.12	3558.38	55.79	0.48	418.10
minimum	43	12240	0.41	2.14	1134.19	12.03	0.40	225.02

---

[a] HHV = Higher Heating Value

[b] These values calculate as: lbs/hr = ((lbs/mmBtu)(Klb/hr)*(Btu/lb)(1000 lb/Klb)/(10E6Btu/mmBtu))

APPENDIX B

601 OUTFALL COMPLIANCE REPORTS

N F-OPER : EO

~~KSR~~ 7/5/91
~~DAM~~

OHIOEDISON
The Energy Makers

Edgewater Plant
200 Oberlin Ave.
Lorain, Ohio 44052

June 5, 1991

Ohio EPA
Division Water Pollution Control
Enforcement Section, ES-MOR
P.O. Box 1049
Columbus, OH 43266-0149

Monthly Operation Report Forms

As required by Rule 3745-33-05 of the Ohio Administrative Code, enclosed are the Monthly Operating Report Forms EPA 4500 (08-86) for the Edgewater Plant, 3IB00005, and the West Lorain Plant, 3IB00008, covering the month of May, 1991.

Very truly yours,

S Douglas Koch

S. Douglas Koch
Superintendent, Edgewater Plant

jrc
enc.

8501 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005801 MAY-91 1 1 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

801 AT CONDENSER COOLING WATER INTAKE

FORM

EDGEWATER S. HILL

2
24

WATER
TEMP
F

00011

01	61
02	61
03	61
04	61
05	61
06	62
07	61
08	63
09	61
10	61
11	61
12	61
13	61
14	73
15	77
16	65
17	70
18	68
19	68
20	68
21	68
22	67
23	71
24	71
25	73
26	73
27	73
28	73
29	72
30	75
31	77

2078
67
77
61

AGENCY

6-5-91

Plant Superintend

8501 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005802 MAY-91 1 1 OH00513C

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

802 AT PLANT WATER INTAKE

FORM

EDGEWATER S. HILL

3
1

RESIDUE
T,NFLT
MG/L

00530

01	18
02	
03	
04	
05	
06	
07	41
08	13
09	
10	
11	
12	
13	
14	
15	7
16	
17	4
18	
19	
20	
21	11
22	
23	16
24	
25	
26	
27	
28	
29	11
30	7
31	

128
14
41
4

B-3

AGENCY

6-5-91

Plant Superintendent

8501 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005001 MAY-91 1 1 OH0051306

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

001 CONDENSER COOLING WATER OUTFALL PRIOR TO LAKE ERIE

FORM

EDGEWATER S. HILL

	2 24 WATER TEMP F	2 24 CONDUIT FLOW MGD	3 1 CHLOR TOT RES MG/L	3 1 PH S.U.
	00011	50050	50060	00400
01	61	152.6	AH	
02	61	152.6	AH	
03	61	152.6	AH	
04	61	152.6	AH	
05	64	152.6	AH	
06	70	152.6	AH	
07	73	152.6	AH	8.1
08	71	152.6	AH	
09	61	152.6	AH	
10	61	152.6	AH	
11	61	152.6	AH	
12	61	152.6	AH	
13	61	86.4	AH	
14	82	86.4	AH	
15	93	86.4	0.02	
16	75	122.4	AH	
17	75	122.4	AH	
18	68	152.6	AH	
19	68	152.6	AH	
20	68	152.6	AH	
21	68	152.6	AH	
22	75	152.6	AH	
23	87	152.6	AH	
24	90	152.6	AH	
25	73	152.6	AH	
26	73	152.6	AH	
27	73	152.6	AH	
28	93	152.6	AH	
29	87	152.6	AH	
30	86	152.6	AH	
31	96	152.6	AH	
	2257	4471.6	0.02	
	73	144.2	0.02	
	96	152.6	0.02	8.1
	61	86.4	AH	8.1

AH: Did not chlorinate, so no sample was taken

AGENCY

6-5-91

Plant Superintendent

8501 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY

3IB00005002 MAY-91 1 1 OH005130

EDGEWATER PLANT
76 SOUTH MAIN STRET
AKRON 44308 LORAIN

002 DISCHARGE FROM OIL SEPARATOR PRIOR TO LAKE ERIE

FORM

EDGEWATER S. HILL

	3	3	2
	1	1	24
	PH S.U.	O&G FREN-G MG/L	CONDUIT FLOW MGD
	00400	00556	50050
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH

AH: No Flow for the Entire Month

AGENCY

6-5-91

Plant Superintendent

NTHLY REPORT FORM

4500

OHIO EDISON COMPANY — 3IB00005601 — MAY-91 — 1 1 — 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 PH S.U.	3 1 RESIDU T. NFL MG/L GROSS	3 1 CONDUI FLOW MGD	3 1 O&G FREN-G MG/L
	00400	00530	50050	00556
01	6.8	1	0.7	
02			0.7	
03			0.7	
04			1.1	
05			0.7	
06			1.1	
07	7.7	2	1.3	AA
08	7.8	6	2.1	
09			1.7	
10			1.7	
11			2.1	
12			1.7	
13			1.6	
14			1.6	
15	7.4	5	1.6	
16			1.6	
17	7.7	2	1.6	
18			1.8	
19			1.8	
20			1.8	
21	7.9	4	1.8	
22			1.8	
23	7.4	13	1.8	
24			1.8	
25			1.7	
26			1.7	
27			1.7	
28			1.7	
29	7.4	8	1.7	
30	7.7	12	1.3	
31			1.3	
		53	47.3	AA
		6	1.5	AA
	7.9	13	2.1	AA
	6.8	1	0.7	AA

AA: Below detectable limits

AGENCY

6-5-91

Plant Superintendent

8501 M 9008 850926

NTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005606 MAY-91 1 1 OH005130

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

606 AT BOILER BLOWDOWN PRIOR MIXING WITH COOLWATER

FORM

EDGEWATER S. HILL

why do we bother

Required 1/mo.

	2 24 CONDUIT FLOW MGD	3 1 PH S.U.	3 1 RESIDU T,NFLT MG/L	3 1 O&G TOTAL MG/L	3 1 PHOS-1 P-WET MG/L
	50050	00400	00530	00550	00665
01	0.0007				
02	0.0007				
03	0.0007				
04	0.0007				
05	0.0007				
06	0.0007				
07	0.0086	10.0	AA	1	1.49
08	0.0033				
09	0.0007				
10	0.0007				
11	0.0007				
12	0.0007				
13	0.0007				
14	0.0007				
15	0.0007	9.9	AA		
16	0.0007				
17	0.0007				
18	0.0007				
19	0.0007				
20	0.0007				
21	0.0007				
22	0.0007				
23	0.0112	AH	AH		
24	0.0033				
25	0.0007				
26	0.0007				
27	0.0007				
28	0.0152				
29	0.0033	10.5	AA		
30	0.0007				
31	0.0007				
	0.0624		AA	1	1.49
	0.0020		AA	1	1.49
	0.0152	10.5	AA	1	1.49
	0.0007	9.9	AA	1	1.49

AA: Below Detectable Limits
AH: Plant did not reach stable operating conditions, therefore no sample was taken

AGENCY

6-5-91

Plant Superintendent

~~DEM~~ 7/13/
~~KSK~~

N F-OPER ED

OHIO**EDISON**
The Energy Makers

Edgewater Plant
200 Oberlin Ave
Lorain, Ohio 44052

July 9, 1991

Ohio EPA
Division Water Pollution Control
Enforcement Section, ES-MOR
P.O. Box 1049
Columbus, OH 43266-0149

Monthly Operation Report Forms

As required by Rule 3745-33-05 of the Ohio Administrative Code, enclosed are the Monthly Operating Report Forms EPA 4500 (08-86) for the Edgewater Plant, 3IB00005, and the West Lorain Plant, 3IB00008, covering the month of June, 1991.

Very truly yours,

S. Douglas Koch

S. Douglas Koch
Superintendent, Edgewater Plant

jrc
enc.

8501 M 9008 850926

4500

MONTHLY REPORT FORM

OHIO EDISON COMPANY

3IB00005801

JUN-91

1 1

OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

801 AT CONDENSER COOLING WATER INTAKE

FORM

EDGEWATER

S. HILL

2
24

WATER
TEMP
F

	00011
01	78
02	78
03	78
04	76
05	76
06	76
07	75
08	75
09	74
10	74
11	75
12	76
13	77
14	74
15	78
16	79
17	80
18	79
19	75
20	79
21	80
22	78
23	74
24	76
25	72
26	78
27	78
28	85
29	78
30	82
31	
	2313
	77
	85
	72

AGENCY

7-8-91

Plant Superintendent

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005802 JUNJ-91 1 1 OHOC5130

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

802 AT PLANT WATER INTAKE

FORM

EDGEWATER S. HILL

	3 1 RESIDUE T,NFLT MG/L 00530
01	
02	
03	
04	24
05	
06	10
07	
08	
09	
10	
11	6
12	4
13	
14	
15	
16	
17	
18	12
19	
20	
21	6
22	
23	
24	
25	13
26	
27	5
28	
29	
30	
31	
	80
	10
	24
	4

B-10

AGENCY

7-8-91 SDKoch Plant Superintendent

8501 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY

3IB00005001 JUN-91 1 1 OH005130

EDGEWATER Plant
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

001 CONDENSER COOLING WATER OUTFALL PRIOR LAKE ERIE

FORM

EDGEWATER S. HILL

	2	2	3	3
	24	24	1	1
	WATER TEMP F	CONDUIT FLOW MGD	CHLOR TOT RES MG/L	PH S.U.
	00011	50050	50060	00400
01	96	76.3	AH	
02	78	76.3	AH	
03	78	46.1	AH	
04	76	46.1	AH	7.7
05	76	46.1	AH	
06	76	46.1	AH	
07	75	46.1	AH	
08	75	46.1	AH	
09	74	76.3	AH	
10	97	76.3	AH	
11	97	76.3	AH	
12	98	76.3	AH	
13	89	76.3	AH	
14	96	76.3	AH	
15	100	76.3	AH	
16	79	76.3	AH	
17	101	76.3	AH	
18	97	152.6	AH	
19	93	152.6	AH	
20	87	152.6	AH	
21	97	152.6	AH	
22	87	152.6	AH	
23	78	152.6	AH	
24	85	152.6	AH	
25	86	152.6	AH	
26	94	152.6	AH	
27	93	152.6	0.01	
28	95	152.6	0.01	
29	88	152.6	AH	
30	92	152.6	AH	
31				
	2633	3099.7	0.02	
	88	103.3	0.01	
	101	152.6	0.01	7.7
	74	46.1	0.01	7.7

AH: Did not chlorinate, so no sample was taken.

AGENCY

7-8-91

Plant Superintendent

[illegible]

IONTHLY REPORT FORM 4500

OHIO EDISON COMPANY 3IB00005002 JUN-91 1 1 OHD00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

002 DISCHARGE FROM OIL SEPARATOR PRIOR TO LAKE ERIE

FORM

EDGEWATER S. HILL

	3 1	3 1	2 24
	PH S.U.	O&G FREN-G MG/L	CONDUIT FLOW MGD
	00400	00556	50050
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH

AH: No flow for the entire month

B-12

AGENCY

7-8-91 SDKoch Plant Superintendent

8501 M 9008 850926

4500

[illegible]

IONTHLY REPORT FORM

OHIO EDISON COMPANY 3IB00005601 JUN-91 1 2 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

						EDGEWATER			S. HILL	
	3	3	3	3	3	3	3	3	3	3
	1	1	1	1	1	1	1	1	1	1
	PH S.U.	RESIDU T. NFL MG/L GROSS	CONDUI 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	CHRO HEX-V UG/L
	00400	00530	50050	00556	00978	00719	00981	01012	01027	0103
01			0.9							
02			0.9							
03			0.9							
04	7.8	1	0.7	3						
05			0.7							
06	7.8	1	0.9		AA	0.005	AA	AA	AA	3
07			0.9							
08			0.7							
09			1.5							
10			1.6							
11	7.7	16	1.5							
12	7.4	7	1.6							
13			1.6							
14			1.6							
15			1.7							
16			1.7							
17			1.6							
18	7.3	1	2.1							
19			1.9							
20			1.9							
21	7.3	8	1.6							
22			1.6							
23			1.6							
24			1.6							
25	7.2	7	1.6							
26			1.3							
27	7.5	6	1.7							
28			1.6							
29			1.6							
30			1.6							
31										
		47	42.7	3	AA	0.005	AA	AA	AA	3
		6	1.4	3	AA	0.005	AA	AA	AA	3
	7.8	16	2.1	3	AA	0.005	AA	AA	AA	3
	7.2	1	0.7	3	AA	0.005	AA	AA	AA	3

AA: Below detectable limits.

The Cyanide and Chromium Hex VA actual result less than reported value.

B-13

AGENCY

7-8-91 SDKah Plant Superintendent

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005601 JUN-91 2 2 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

EDGEWATER S. HILL

	3 1 CHROM CR,TOT UG/L 01034	3 1 NICKEL TOT,RE UG/L 01074	3 1 SILVER TOT,RE UG/L 01079	3 1 ZINC TOT,RE UG/L 01094	3 1 LEAD CP TOT,RE UG/L 01114	3 1 MERCUR TOT,RE UG/L 71901
01						
02						
03						
04						
05						
06	20	10	AA	10	AA	0.5
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
	20	10	AA	10	AA	0.5
	20	10	AA	10	AA	0.5
	20	10	AA	10	AA	0.5
	20	10	AA	10	AA	0.5

AA: Below detectable limits
The Chromium Cr, Nickel, Zinc and Mercury actual result less than reported value.

AGENCY

7-8-91 SD Karl Plant Superintendent

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY

3IB00005606 JUN-91 1 1 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

606 AT BOILER BLOWDOWN PRIOR MIXING WITH COOL WATER

FORM

EDGEWATER S. HILL

	2 24	3 1	3 1	3 1	3 1
	CONDUIT FLOW MGD	PH S.U.	RESIDUE T,NFLT MG/L	O&G TOTAL MG/L	PHOS-1 P-WET MG/L
	50050	00400	00530	00550	00665
01	0.0007				
02	0.0007				
03	0.0007				
04	0.0007				
05	0.0007				
06	0.0007				
07	0.0007				
08	0.0007				
09	0.0007				
10	0.0020				
11	0.0086	9.5	1	AA	1.90
12	0.0007				
13	0.0033				
14	0.0007				
15	0.0007				
16	0.0007				
17	0.0007				
18	0.0007				
19	0.0086				
20	0.0350				
21	0.0007				
22	0.0086				
23	0.0007				
24	0.0007				
25	0.0007				
26	0.0007				
27	0.0007				
28	0.0007				
29	0.0007				
30	0.0007				
31					
	0.0829		1	AA	1.90
	0.0028		1	AA	1.90
	0.0350	9.5	1	AA	1.90
	0.0007	9.5	1	AA	1.90

pH is not a parameter requiring monitoring

AA: Below detectable limits

B-15

AGENCY

7-8-91

SOKah

Plant Superintendent

NF-OPER ED

~~KSR~~
DRM

OHIOEDISON
The Energy Makers

Edgewater Plant
200 Oberlin Ave
Lorain, Ohio 44052

August 13, 1991

Ohio EPA
Division Water Pollution Control
Enforcement Section, ES-MOR
P.O. Box 1049
Columbus, OH 43266-0149

Monthly Operation Report Forms

As required by Rule 3745-33-05 of the Ohio Administrative Code, enclosed are the Monthly Operating Report Forms EPA 4500 (08-86) for the Edgewater Plant, 3IB00005, and the West Lorain Plant, 3IB00008, covering the month of July, 1991.

Very truly yours,

S D Koch / J Hill

S. Douglas Koch
Superintendent, Edgewater Plant

jrc
enc.

8510 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY

3IB00005801 JUL-91 1 1 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

801 AT CONDENSER COOLING WATER INTAKE

FORM

EDGEWATER S. HILL

	2 24 WATER TEMP F 00011
01	81
02	80
03	82
04	77
05	80
06	82
07	82
08	80
09	84
10	79
11	84
12	78
13	77
14	79
15	81
16	78
17	82
18	81
19	80
20	86
21	82
22	83
23	85
24	84
25	88
26	87
27	83
28	81
29	80
30	80
31	80
	2526
	81
	87 88
	77

AGENCY

8-13-91 JD Koch / S Hill Plant Superintendent

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005802 JUL-91 1 1 OH00513

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

802 AT PLANT WATER INTAKE

FORM

EDGEWATER S. HILL

	3 1 RESIDUE T,NFLT MG/L 00530
01	
02	
03	4
04	
05	5
06	
07	
08	
09	9
10	31
11	
12	
13	
14	
15	
16	
17	9
18	
19	10
20	
21	
22	
23	14
24	11
25	
26	
27	
28	
29	
30	4
31	6

103
10
31
4

B-18

AGENCY

8-13-91 JD Koch/S Hill Plant Superintendent

8501 M 9008 850926

4500

MONTHLY REPORT FORM

OHIO EDISON COMPANY 3IB00005001 JUL-91 1 1 OH00513C

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

001 CONDENSER COOLING WATER OUTFALL PRIOR LAKE ERIE

FORM

EDGEWATER S. HILL

	2 24	2 24	3 1	3 1
	WATER TEMP F	CONDUIT FLOW MGD	CHLOR TOT RES MG/L	PH S.U.
	00011	50050	50060	00400
01	99	116.6	AH	
02	99	116.6	AH	
03	102	116.6	AH	
04	82	116.6	AH	
05	89	116.6	AH	
06	95	116.6	AH	
07	95	116.6	AH	
08	97	116.6	AH	
09	92	116.6	AH	
10	96	116.6	AH	7.7
11	97	116.6	AH	
12	94	116.6	AH	
13	84	116.6	AH	
14	79	116.6	AH	
15	89	116.6	AH	
16	88	116.6	AH	
17	98	152.6	AH	
18	96	152.6	AH	
19	96	152.6	AH	
20	104	152.6	AH	
21	94	152.6	AH	
22	99	152.6	AH	
23	102	152.6	AH	
24	96	152.6	AH	
25	101	152.6	AH	
26	95	152.6	AH	
27	94	152.6	AH	
28	84	152.6	AH	
29	83	152.6	AH	
30	80	123.1	AH	
31	90	86.4	AH	
	2889	4058.9	AH	
	93	130.9	AH	
	104	152.6	AH	7.7
	79	86.4	AH	7.7

AH: Did not chlorinate, so no sample was taken.

AGENCY

8-13-91 SDKoch/JHill Plant Superintendent

8510 M 9008 850926

NTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005002 JUL-91 1 1 OH 0051306

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

002 DISCHARGE FROM OIL SEPARATOR PRIOR TO LAKE ERIE

FORM

EDGEWATER S. HILL

	3	3	2
	1	1	24
	PH S.U.	O&G FREN-G MG/L	CONDUIT FLOW MGD
	00400	00556	50050
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH

AH: No Flow For The Entire Month

AGENCY

8-13-91 J D Koch / Stall Plant Superintendent

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005601 JUL-91 1 1 OH005130

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE

FORM

EDGEWATER S. HILL

	3	3	3	3
	1	1	1	1
	PH S.U.	RESIDU T. NFL MG/L GROSS	CONDUI FLOW MGD	O&G FREN-G MG/L
	00400	00530	50050	0055~~6~~0
01			1.6	
02			1.7	
03	7.4	1	1.6	
04			1.6	
05	7.2	1	1.3	
06			1.3	
07			1.6	
08			1.6	
09	7.6	4	2.1	
10	7.5	4	1.4	2
11			1.4	
12			1.4	
13			1.4	
14			1.6	
15			1.7	
16			1.6	
17	7.7	2	1.7	
18			1.7	
19	8.3	5	2.1	
20			1.6	
21			1.6	
22			1.6	
23	7.5	4	1.7	
24	7.4	6	1.7	
25			1.7	
26			1.6	
27			1.6	
28			1.3	
29			1.3	
30	7.1	3	1.1	
31	7.6	2	1.1	
		32	48.3	2
		3	1.6	2
	8.3	6	2.1	2
	7.1	1	1.1	2

B-21

AGENCY

8-13-91 SD Koch/JHall Plant Superintendent

8510 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005606 JUL-91 1 1 OH005130

EDGEWATER PLANT
76 SOUTH MAIN STREET
AKRON 44308 LORAIN

606 AT BOILER BLOWDOWN PRIOR MIXING WITH COOL WATER

FORM

EDGEWATER S. HILL

	2 24	3 1	3 1	3 1
	CONDUIT FLOW MGD	RESIDUE T,NFLT MG/L	O&G TOTAL MG/L	PHOS-1 P-WET MG/L
	50050	00530	00550	00665
01	0.0007			
02	0.0007			
03	0.0007			
04	0.0007			
05	0.0007			
06	0.0007			
07	0.0007			
08	0.0099			
09	0.0047			
10	0.0060	AA	1	3.16
11	0.0047			
12	0.0060			
13	0.0007			
14	0.0007			
15	0.0007			
16	0.0033			
17	0.0152			
18	0.0112			
19	0.0060			
20	0.0086			
21	0.0007			
22	0.0086			
23	0.0047			
24	0.0007			
25	0.0007			
26	0.0007			
27	0.0007			
28	0.0007			
29	0.0007			
30	0.0007			
31	0.0060			
	0.1075	AA	1	3.16
	0.0035	AA	1	3.16
	0.0152	AA	1	3.16
	0.0007	AA	1	3.16

AA: Below Detectable Limits

AGENCY

8-13-91

SDKoch/[illegible]

Plant Superintendent

6510 M 9008 850926

NTHLY REPORT FORM

4500

HIO EDISON COMPANY

3IB00005801 AUG-91 1 1 OH005130

DGEWATER PLANT
6 SOUTH MAIN ST.
KRON 44308 LORAIN

801 AT CONDENSER COOLING WATER INTAKE

FORM

EDGEWATER

S. HILL

2
24

	WATER TEMP F 00011
01	84
02	83
03	82
04	82
05	81
06	80
07	80
08	80
09	82
10	80
11	80
12	80
13	80
14	80
15	80
16	80
17	80
18	80
19	80
20	80
21	80
22	80
23	80
24	80
25	80
26	80
27	83
28	79
29	81
30	83
31	81

2501
81
84
79

AGENCY

9-13-91

NTHLY REPORT FORM

4500

HIO EDISON COMPANY

3IB00005802 AUG-91 1 1 OH005130

DGEWATER PLANT
76 SOUTH MAIN ST.
KRON 44308 LORAIN

802 AT PLANT WATER INTAKE

FORM

EDGEWATER S. HILL

3
1

RESIDUE
T,NFLT
MG/L

00530

01	
02	
03	
04	
05	
06	6
07	
08	9
09	
10	
11	
12	6
13	
14	1
15	
16	
17	
18	
19	
20	
21	4
22	4
23	
24	
25	
26	
27	12
28	8
29	
30	
31	

50
6
12
1

AGENCY

9-13-91

Plant Superintendent

8501 M 9008 850926

4500

NTHLY REPORT FORM

)HIO EDISON COMPANY 3IB00005001 AUG-91 1 1 OH005130

ĐDGEWATER PLANT
76 SOUTH MAIN ST.
KRON 44308 LORAIN

001 CONDENSER COOLING WATER OUTFALL PRIOR LAKE ERIE

FORM

EDGEWATER S. HILL

	2 24	2 24	3 1	3 1
	WATER TEMP F	CONDUIT FLOW MGD	CHLOR TOT RES MG/L	PH S.U.
	00011	50050	50060	00400
01	100	189.3	AH	
02	100	189.3	AH	
03	82	159.1	AH	
04	82	82.8	AH	
05	81	82.8	AH	
06	81	159.1	AH	8.0
07	84	159.1	AH	
08	85	159.1	AH	
09	93	159.1	AH	
10	90	46.1	AH	
11	80	46.1	AH	
12	80	46.1	AH	
13	80	46.1	AH	
14	80	46.1	AH	
15	80	46.1	AH	
16	80	46.1	AH	
17	80	46.1	AH	
18	80	46.1	AH	
19	80	46.1	AH	
20	80	46.1	AH	
21	80	46.1	AH	
22	80	46.1	AH	
23	80	46.1	AH	
24	80	46.1	AH	
25	80	46.1	AH	
26	96	46.1	AH	
27	117	46.1	AH	
28	99	46.1	AH	
29	114	46.1	AH	
30	105	152.6	AH	
31	93	152.6	AH	
	2702	2566.9	AH	
	87	82.8	AH	
	117	189.3	AH	8.0
	80	46.1	AH	8.0

AH: Did not chlorinate, so no sample was taken.

AGENCY

9-13-91 Plant Superintendent

8510 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005002 AUG-91 1 1 OH005130

EDGEWATER PLANT
76 SOUTH MAIN ST.
AKRON 44308 LORAIN

002 DISCHARGE FROM OIL SEPARATOR PRIOR TO LAKE ERIE

FORM

EDGEWATER S. HILL

	3	3	2
	1	1	24
	PH S.U.	O&G FREN-G MG/L	CONDUIT FLOW MGD
	00400	00556	50050
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH
	AH	AH	AH

AH: No Flow For The Entire Month

B-26

AGENCY

9-13-91

Plant SUperintendent

6510 M 9008 850926

NTHLY REPORT FORM

4500

OHIO EDISON COMPANY　3IB00005601　AUG-91　1 2　OH00513

EDGEWATER PLANT
76 SOUTH MAIN ST.
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	3 1 CONDUI FLOW MGD	3 1 O&G FREN-G MG/L	3 1 ARSENI TOT,RE UG/L	3 1 CYANID FREE MG/L	3 1 SELENI TOT,RE UG/L	3 1 BERYL BE,TOT UG/L	3 1 CAD CD,TOT UG/L	3 1 CHRO HEX- UG/L
	00400	00530	50050	00550	00978	00719	00981	01012	01027	01032
01			1.1							
02			1.1							
03			1.7							
04			1.6							
05			1.6							
06	7.9	3	1.6	2						
07			1.6							
08	8.0	8	1.6							
09			1.1							
10			1.3							
11			1.3							
12	7.9	5	1.3							
13			1.1							
14	7.6	2	0.7							
15	7.4		0.7		7	0.005	AA	AA	AA	3
16			1.1							
17			1.1							
18			1.3							
19			1.1							
20			1.1							
21	6.5	2	0.2							
22	7.1	3	0.2							
23			1.1							
24			1.7							
25			1.6							
26			1.6							
27	7.2	8	2.4							
28	7.0	3	2.1							
29			1.7							
30			1.7							
31			1.7							
		34	41.1	2	7	0.005	AA	AA	AA	3
		4	1.3	2	7	0.005	AA	AA	AA	3
	8.0	8	2.4	2	7	0.005	AA	AA	AA	3
	6.5	2	0.2	2	7	0.005	AA	AA	AA	3

AA: Below detectable limits

he Cyanide and Chromium Hex VA actual results less than reported value

AGENCY

9-13-91

8510 M 9008 850926

ONTHLY REPORT FORM

4500

OHIO EDISON COMPANY 3IB00005601 AUG-91 2 2 OH005130

EDGEWATER PLANT
76 SOUTH MAIN ST. 601 ASH POND DISCHARGE PRIOR TO ENTERING LAKE ERIE
AKRON 44308 LORAIN

FORM

EDGEWATER S. HILL

	3 1	3 1	3 1	3 1	3 1	3 1
	CHROM CR,TOT UG/L	NICKEL TOT,RE UG/L	SILVER TOT,RE UG/L	ZINC TOT,RE ug/L	LEAD CP TOT,RE UG/L	MERCUR TOT,RE UG/L
	01034	10174	01079	01094	01114	71901
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15	20	20	AA	30	AA	0.1
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
	20	20	AA	30	AA	0.1
	20	20	AA	30	AA	0.1
	20	20	AA	30	AA	0.1
	20	20	AA	30	AA	0.1

AA: Below detectable limits

The Chromium Crand Mercury actual result less than reported value

AGENCY

9-13-91

Plant SUperintendent

[illegible] M 9008 [illegible]

NTHLY REPORT FORM

4500

HIO EDISON COMPANY	3IB00005606	AUG-91	1	1	OH0051306

DGEWATER PLANT
6 SOUTH MAIN ST.
KRON 44308 LORAIN

606 AT BOILER BLOWDOWN PRIOR MIXING WITH COOL WATER

FORM

	2 24 CONDUIT FLOW MGD	3 1 RESIDU T,NFLT MG/L	3 1 O&G TOTAL MG/L	3 1 PHOS-1 P-WET MG/1
	50050	00530	00550	00665
01	0.0033			
02	0.0007			
03	0.0007			
04	0.0007			
05	0.0007			
06	0.0007	1	3	2.76
07	0.0033			
08	0.0007			
09	0.0007			
10	0.0007			
11	0.0007			
12	0.0007			
13	0.0007			
14	0.0007			
15	0.0007			
16	0.0007			
17	0.0007			
18	0.0007			
19	0.0007			
20	0.0007			
21	0.0007			
22	0.0007			
23	0.0007			
24	0.0007			
25	0.0007			
26	0.0007			
27	0.0033			
28	0.0060			
29	0.0007			
30	0.0086			
31	0.0007			
	0.0427	1	3	2.76
	0.0014	1	3	2.76
	0.0086	1	3	2.76
	0.0007	1	3	2.76

AGENCY

9-13-91

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	C	INITIALS
2-18		3.0%S, Dlime			7.53	✓	~~9.6~~	9.6	1300 RL
2-19-91		3.0%S, Dlime			7.72	✓	~~10.5~~	12.5	1300 RL
2-20-91		3.0%S, D.lime			8.31	✓	✓	6.0	1330 RL
2-21-91		3.0%S, Dlime			8.37	✓	✓	9.4	1400 RL
2-22-91		L%S, Dlime			7.63	✓		8.5	1300 RL
2-25-91		L%S, Ligno lime			7.92	✓	✓	6.9	1400 RL
2-26		"			7.55	✓	✓	8.5	1400 RL
2-27		"			7.21	✓	✓	7.2	1400 RL
2-28		"			7.53	✓	✓	8.9	1400 RL
3-6		"			7.81	✓		10.4	1100 61
3-7		"			7.94	✓		10.3	1100 61
3-8-91		"			7.94	✓		10.9	1400 61
3-11 To 3-17	NO	SAMPLES	COLLECTED	DUE	TO BROKEN	Ph		PROBE	
4-1-91		L%S, Lignolime			7.12	✓	✓	14.3	1300 RL
4-2					7.21	✓	✓	15.7	1300 RL
4-3					7.34	✓	✓	16.2	1400 RL
4-4					7.11	✓	✓	18.4	1400 RL
4-5					7.30	✓	✓	18.5	1400 RL
4-15-91		H%S, Lignolime			7.79	✓	✓	16.0	1400 RL
4-16-91					7.87	✓	✓	16.3	1400 RL
4-17-91					7.22	✓	-	18.9	1500 RL
4-18-91					7.29	✓	✓	13.7	1500 RL
	↑ LIMB	-EMP601-30							
4-22-91		3%S, Lignolime			7.09	✓	-	13.1	1400 RL
4-23-91		"			7.15	✓	✓	15.8	1400 RL
4-24-91		"			7.14	✓	-	16.1	1400 RL
5-6-91		"			7.31	✓	✓	17.5	1400 RL
5-7-91					7.23	✓	✓	17.9	1500 RL

Page No.

Witnessed & Understood by me,

Date

1 by

Recorded by

Date

LE

m Page No.

		SAMPLES TAKEN					
DATE	COAL/SORBENT COMBINATION	pH	Ca	Ash	°C	Initials	
5-8-91		7.61	✓	✓	18.2	1100 RQ	
	↑ LIME EMP601-31						
5-14-91	H/S, LL	7.31	✓	✓	21.3	1200 RQ	
5-15		7.38	✓	✓	23.7	1300 RQ	
5-16		7.48	✓	–	23.3	1100 RQ	
5-22-91	L/S, LL	7.21	✓	✓	24.7	1400 RQ	EM 601.
-23	〃	7.09	✓	–	23.9	1400 RQ	
-24	〃	7.12	✓	✓	25.3	1400 RQ	
5-28	〃	7.01	✓	✓	26.7	1430 RQ	
5-29		7.17	✓	–	25.9	1400 RQ	
5-30		7.21	✓	–	26.2	1200 RQ	
5-31		7.28	✓	✓	26.8	1100 RQ	
6-10-91	L/S, LL	7.06	✓	✓	27.1	1300 RQ	EMP 3 601 -
6-11-91		7.11	✓		26.9	1300 RQ	
6-12-91		7.09	✓		26.7	1300 RQ	
6-13-91		7.10	✓	✓	26.2	1300 RQ	
6-14-91		7.08	✓	✓	26.8	1300 RQ	
6-17-91		7.18	✓		26.2	1230 RQ	EMP 3 601-
6-18-91		7.16	✓		25.7	1300 RQ	
6-19-91		7.32	✓		25.6	1230 RQ	
6-21-91		7.29	✓	✓	26.3	1200 RQ	
-24-91		7.14	✓		25.2	1200 RQ	EMP 3 601-
-25-91		7.38	✓	✓	25.7	1200 RQ	
-26-91		7.27	✓	✓	27.9	1200 RQ	
6-27-91		7.45	✓	✓	29.1	1330 RQ	
6-28-91		7.32	✓	✓	29.1	1200 RQ	
7-1-91	L/S, LL	7.31	✓	–	27.5	1200 RQ	
7-2-91	L/S	7.32	✓	-	27.3	1200 RQ	
7-3-91	L/S, Calc/lime	7.27	✓		31.0	1500 RQ	
7-8-91	H/S, C.L	7.32	✓		27.9	1300 RQ	
7-9-91	H/S, C.Lime	7.39	✓	✓	27.3	1300 RQ	
7-10-91	H/S, CLime	7.26	✓	✓	25.5	1100 RQ	

To Page No.

itnessed & Understood by me, | Date | by | Date

Recorded by

From Page No. DATE	COAL/SORBENT COMBINATION	pH	Ca	Ash	°C	INITIALS
7-11-91	H% S, Lime	7.37	✓	✓	26.2	1300
7-12-91	4.0% S, Lime	7.42	✓	✓	26.5	1400
7-15-91	4.0% S, Lime	7.5	✓	✓	26.7	1500
7-16-91	" "	7.47	✓	✓	25.8	1200
7-17-91	"	7.38	✓	✓	27.9	1400
7-18-91	"	7.63	✓	✓	28.1	1300
7-19-91	"	7.37	✓		28.3	1230
7-22-91	L% S, Lime	7.27	✓		28.7	1300
7-23	L% S, Lime	7.22	✓	✓	28.1	1300
7-24	L% S, Lime	7.13	✓	✓	29.0	1315
7-25	"	7.42			28.0	1145
7-26		plant down				
7-29-91		plant down				
7-30-91		plant down				
7-31-91		plant down				
8-1-91	"	7.25	✓		26.7	1400
8-2-91	"	7.19	✓		27.2	1400
8-6-91	"	7.13	✓		27.0	1100
8-26-91	"	6.85	✓		26.6	1400
8-27-91		7.11	✓		26.2	1100
8-28-91		7.08	✓		26.5	1000

C-3

To Page

Witnessed & Understood by me. | Date | Invented by | Date

APPENDIX D

601 CALCIUM ANALYSIS

RADIAN
CORPORATION

Radian Work Order 91-05-097

Analytical Report
06/06/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: John Stowe

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-05-097

Method/Analyte	Sample Identifications LIMB-EMP601-30 01			LIMB-EMP601-31 02			METHOD BLANK 03		
Matrix	water			water			water		
	Result		Det. Limit	Result		Det. Limit	Result		Det. Limit
Calcium by ICPES SW6010									
Calcium	39	mg/L	1.0	56	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.

Sample Identifications and Dates

Sample ID	LIMB-EMP601-30	LIMB-EMP601-31	METHOD BLANK			
Date Sampled	05/09/91	05/09/91				
Date Received	05/10/91	05/10/91	05/10/91			
Matrix	water	water	water			
	01	02	03			
Calcium by ICPES SW6010						
Prepared	05/31/91	05/31/91	05/31/91			
Analyzed	06/03/91	06/03/91	06/03/91			
Analyst	DRW	DRW	DRW			
File ID	JA610603-37	JA610603-39				
Blank ID						
Instrument	JA61	JA61	JA61			
Report as	received	received	received			

Appendix A

Comments, Notes and Definitions

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: 91-05-097

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; microSiemans/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
CORPORATION

Radian Work Order 91-05-160

Analytical Report
06/10/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: Linda Carter

Babcock and Wilcox Co.
Radian Work Order: 91-05-160

Method/Analyte	Sample Identifications					
	LIMB-EMP601-32			METHOD BLANK		
	01			02		
Matrix	water			water		
	Result		Det. Limit	Result		Det. Limit
Calcium by ICPES SW6010						
Calcium	65	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: 91-05-160

Sample Identifications and Dates

Sample ID	LIMB-EMP601-32 METHOD BLANK	
Date Sampled	05/16/91	
Date Received	05/20/91	05/20/91
Matrix	water	water
	01	02

Calcium by ICPES SW6010							
Prepared	06/04/91	06/04/91					
Analyzed	06/05/91	06/05/91					
Analyst	DRW	DRW					
File ID	JA610605-26						
Blank ID							
Instrument	JA61	JA61					
Report as	received	received					

Appendix A

Comments, Notes and Definitions

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.

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; microSiemans/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
CORPORATION

Radian Work Order 91-07-032

Analytical Report
07/15/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: Andy Ch

Babcock and Wilcox Co.
Radian Work Order: 91-07-032

Method/Analyte	Sample Identifications LIMB-EMP601-33 01 water		LIMB-EMP601-34 02 water		LIMB-EMP602-35 03 water	
Matrix	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Calcium by ICPES SW6010						
Calcium	40 mg/L	1.0	40 mg/L	1.0	37 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: 91-07-032

Method/Analyte	Sample Identifications			
	LIMB-EMP603-36		METHOD BLANK	
	04		05	
Matrix	water		water	
	Result	Det. Limit	Result	Det. Limit
Calcium by ICPES SW6010				
Calcium	45 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: 91-07-032

Sample Identifications and Dates

Sample ID	LIMB-EMP601-33	LIMB-EMP601-34	LIMB-EMP602-35	LIMB-EMP603-36	METHOD BLANK
Date Sampled	07/02/91	07/02/91	07/02/91	07/02/91	
Date Received	07/03/91	07/03/91	07/03/91	07/03/91	07/03/91
Matrix	water	water	water	water	water
	01	02	03	04	05
Calcium by ICPES SW6010					
Prepared	07/05/91	07/05/91	07/05/91	07/05/91	07/05/91
Analyzed	07/08/91	07/08/91	07/08/91	07/08/91	07/08/91
Analyst	DRW	DRW	DRW	DRW	DRW
File ID			JA610708-39	JA610708-40	
Blank ID					
Instrument	JA61	JA61	JA61	JA61	JA61
Report as	received	received	received	received	received

Appendix A

Comments, Notes and Definitions

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: 91-07-032

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; microSiemans/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
CORPORATION

Radian Work Order 91-08-164

Analytical Report
09/12/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-08-164

Method/Analyte	Sample Identifications					
	LIMB-EMP601-37		LIMB-EMP601-38		LIMB-EMP601-39	
	01		02		03	
Matrix	water		water		water	
	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Calcium by ICPES SW6010						
Calcium	57 mg/L	1.0	59 mg/L	1.0	53 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: 91-08-164

Method/Analyte	Sample Identifications				
	METHOD BLANK 04				
Matrix	water				
	Result		Det. Limit		
Calcium by ICPES SW6010					
Calcium	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: 91-08-164

Sample Identifications and Dates

Sample ID	LIMB-EMP601-37	LIMB-EMP601-38	LIMB-EMP601-39	METHOD BLANK
Date Sampled	08/16/91	08/16/91	08/16/91	
Date Received	08/17/91	08/17/91	08/17/91	08/17/91
Matrix	water	water	water	water
	01	02	03	04

Calcium by ICPES SW6010						
Prepared	08/29/91	08/29/91	08/29/91	08/29/91		
Analyzed	09/05/91	09/05/91	09/05/91	09/05/91		
Analyst	DRW	DRW	DRW	DRW		
File ID	JA610905-31	JA610905-33	JA610905-34			
Blank ID						
Instrument	JA61	JA61	JA61	JA61		
Report as	received	received	received	received		

Appendix A

Comments, Notes and Definitions

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.

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; microSiemans/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
CORPORATION

Radian Work Order 91-09-101

Analytical Report
09/26/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by:

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-09-101

Method/Analyte	Sample Identifications						
	LIMB-EMP601-40			METHOD BLANK			
	01			02			
Matrix	Water			Water			
	Result		Det. Limit	Result		Det. Limit	
Calcium by ICPES SW6010							
Calcium	38	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.

Sample History

Babcock and Wilcox Co.
Radian Work Order: 91-09-101

Sample Identifications and Dates

Sample ID LIMB-EMP601-40 METHOD BLANK

Date Sampled						
Date Received	09/11/91	09/11/91				
Matrix	water	water				
	01	02				
Calcium by ICPES SW6010						
Prepared	09/18/91	09/18/91				
Analyzed	09/24/91	09/24/91				
Analyst	DES	DES				
File ID	JA610924-13	JA610924-10				
Blank ID						
Instrument	JA61	JA61				
Report as	received	received				

Appendix A

Comments, Notes and Definitions

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: 91-09-101

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; microSiemans/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 ≈ 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, 2,4,5-trichloro-.
U230	Phenol, 2,4,6-trichloro-.
	• • • • •
U231	Propionic acid, 2-(2,4,5-trichlorophenoxy)-.
	• • • • •
U233	Silvex.
	• • • • •
U232	2,4,5-T.
	• • • • •
U212	2,3,4,6-Tetrachlorophenol.
	• • • • •
U230	2,4,5-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 D346-75 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."[1] 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."[1]

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[1] 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 8.

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 cm^2 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

---

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

[1] The 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 =

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

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 5.0, the pH of the solution shall be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, 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 5.2, 0.5N acetic acid shall be added to bring the pH down to 5.0 ± 0.2. However, in no event shall the aggregrate 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 5.0 ± 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 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH shall be adjusted to 5.0 ± 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:

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

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 ½ 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/cm^2 (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/cm^2 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]

(i) 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

---

[1] 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.

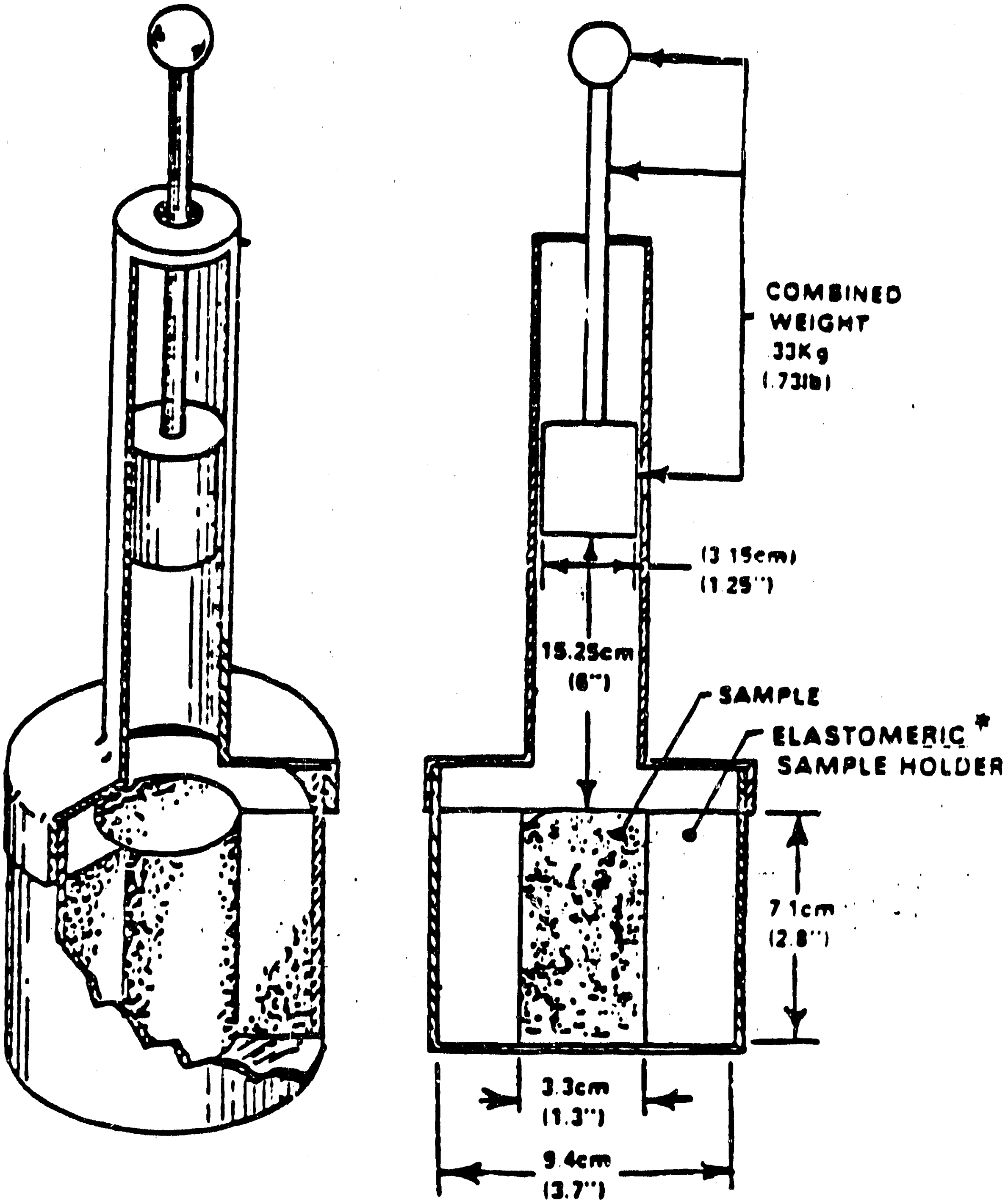
Analytical Procedures for Analyzing Extract Contaminants

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]

Designation: D 3987 - 81

Standard Test Method for
SHAKE EXTRACTION OF SOLID WASTE WITH WATER[1]

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 Aggregates[2]
D 420 Recommended Practice for Investigating and Sampling of Soil and Rock for Engineering Purposes[2]
D 1129 Definitions of Terms Relating to Water[3]
D 1193 Specification for Reagent Water[3]
D 1888 Test Methods for Particulate and Dissolved Matter in Water[3]
D 2216 Method for Laboratory Determination of Moisture Content of Soils[2]
D 2777 Practice for Determination of Precision and Bias of Methods of Committee D-19 on Water[3]
D 2234 Method for Collection of a Gross Sample of Coal[4]
D 3370 Practices for Sampling Water[3]
E 122 Recommended Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process[5]

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 Pharmacopeia."

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 ± 2°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 ± 2°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 $CaSO_3 \cdot 1/2H_2O$ 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 h ± 0.5 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-μm filter. If these separation means result in prolonged filtering time, a 8-μm 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,
B = 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 ± 2°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 μm,

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

X1. 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 foward-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:

$S_t = 0.311X + 9.26$

$S_o = 0.192X - 1.59$

where:

S_t = 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:

$S_t = 0.792X - 0.013$

$S_o = 0.543X - 0.023$

where:

S_t = 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 effects. 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.

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 multiphasic 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.

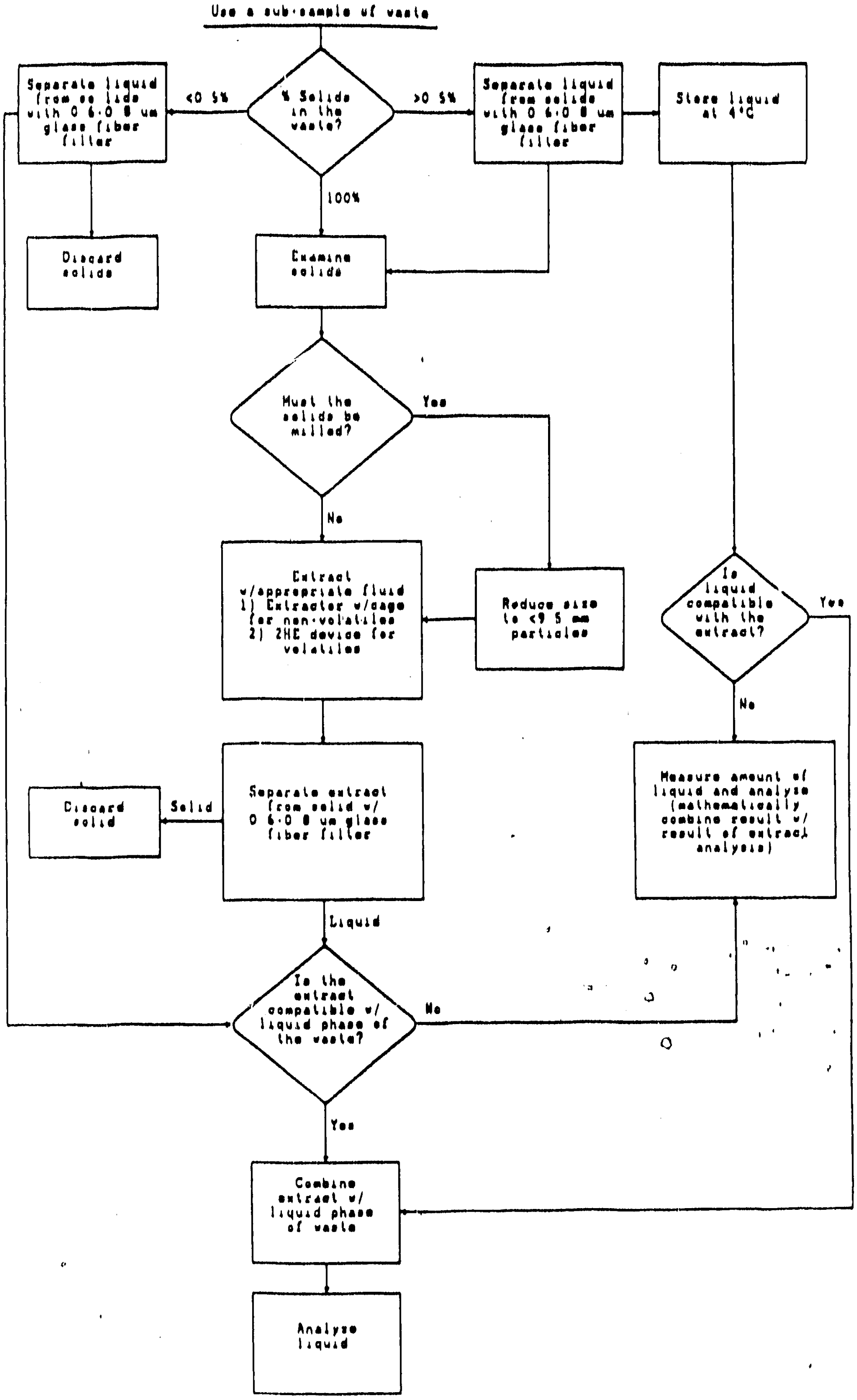
1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile contaminant exceeds the regulatory threshold for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot 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 CONTAMINANTS [1]

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
Methylene 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
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4

TABLE 1.—VOLATILE CONTAMINANTS [1]—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 vessel 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 90–110 mm filter. The devices contain VITON ®[1] 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.

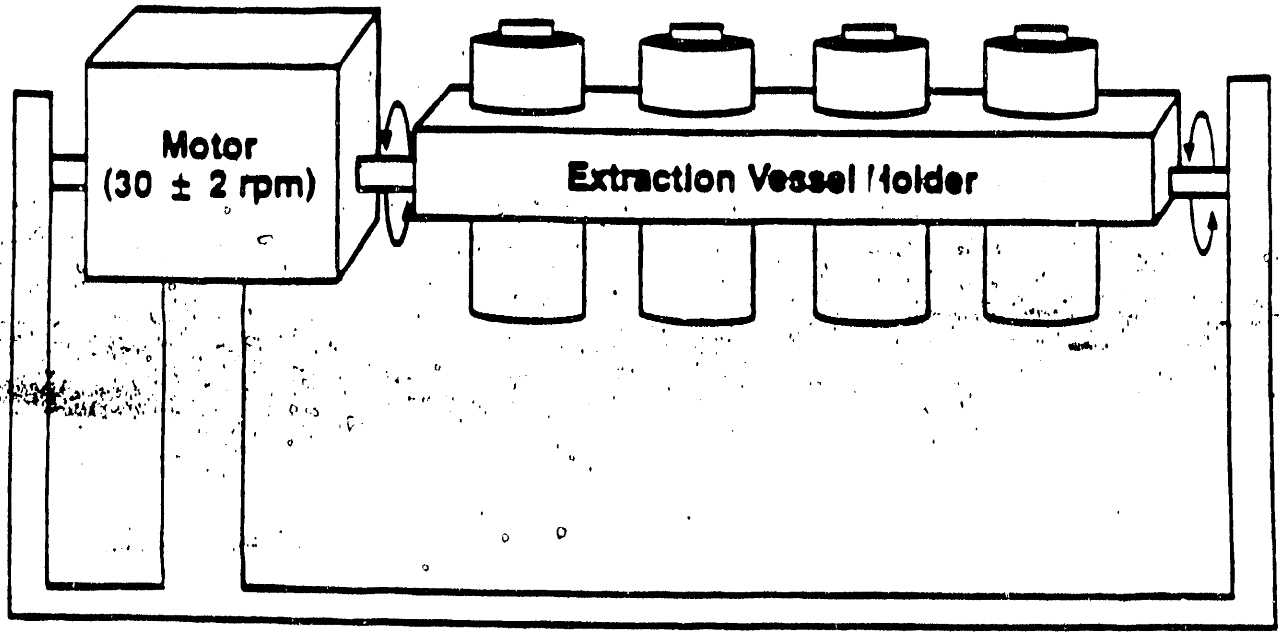


Figure 2. Rotary Agitation Apparatus

[Appendix II]

TABLE 2.—SUITABLE ROTARY AGITATION APPARATUS [1]

Company	Location	Model no.
Analytical Testing and Consulting Services, Inc	Warrington, PA (215) 343-4490	2-ZHE or 4-bottle extractor (DC20S); 4-ZHE or 8-bottle extractor (DC20); 6-ZHE or 12-bottle extractor (DC20B).
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-[illegible]). 12-vessel (3740-12). 24-vessel (3740-24).
Environmental Machine and Design, Inc	Lynchburg, VA (804) 845-6424	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 (YT30ORAHW).

---

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

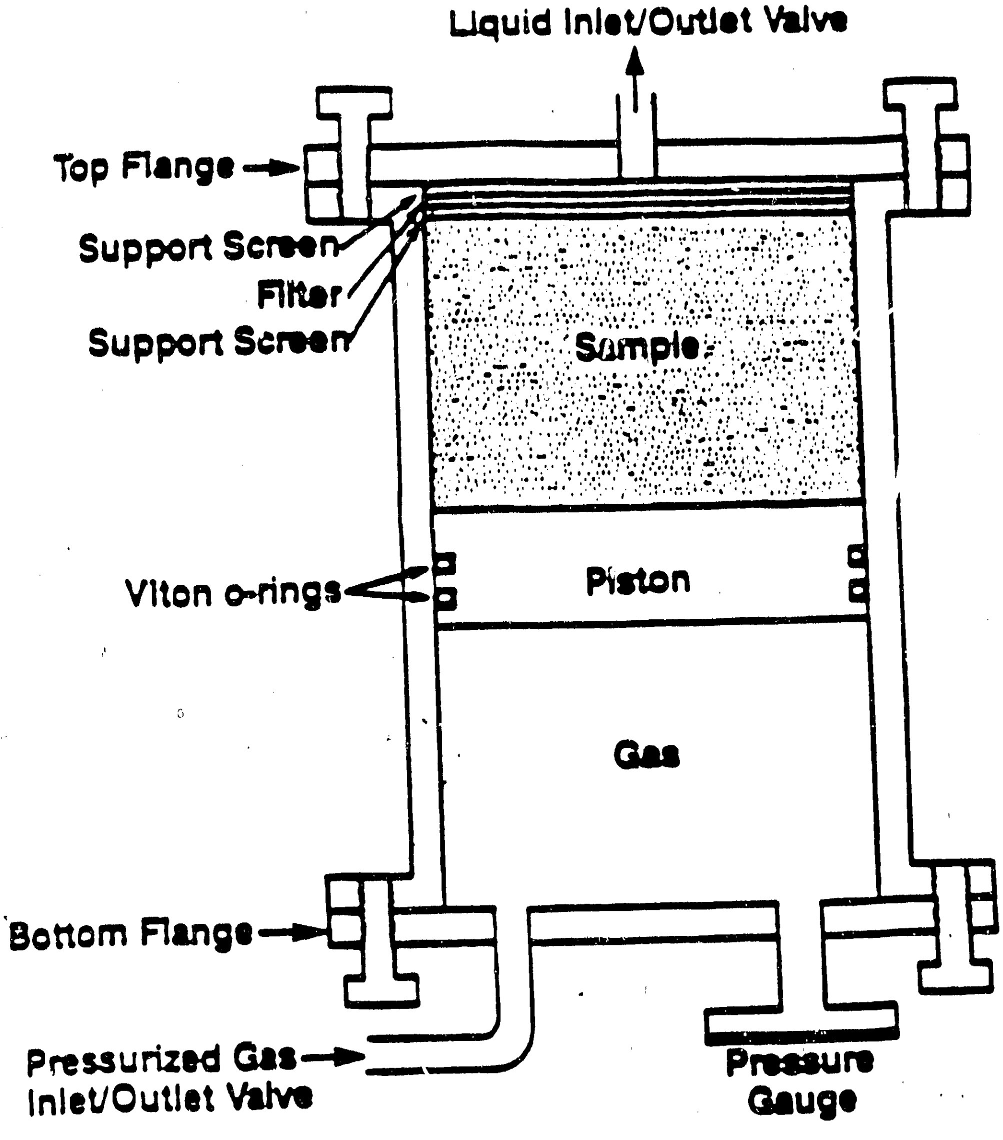


Figure 3. Zero-Headspace Extractor (ZHE)

[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	YT30090HW, 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.

TABLE 5.—SUITABLE FILTER MEDIA [1]

Company	Location	Model	Pore size
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	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®[2] 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 interferant 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 90 +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), HNO_3, 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 4.93 +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 2.88 +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 aliqout 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) procced to Step 7.3.

7.1.2 If the sample is liquid or multiphasic, 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:

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

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 cm^2, 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 9.6.

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 then 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 5.6) to use for the nonvolatiles extraction as follows:

Note: TCLP extraction for volatile constituents uses only extraction fluid #1 (Step 5.6.1). 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 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 multiphasic, 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., limed 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.

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 psi), 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 moisten 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 9.6. 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 effect 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 slurries 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 (gas 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 multiphasic, 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:

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

[Appendix II]

where:

V_1 = The volume of the first phases (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.

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 µg/100 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 μg/ml but no more than 5 μg/ml (say 3 μg/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 μg/ml aqueous calcium standard was added. The volumetric flask was filled to volume with water. (Added amount: a_1 = 0.2 μg/ml of Ca)

(c) To aliquot #3, 2 ml of a 2 μg/ml Ca standard were added and made up to volume. (Added amount: a_2 = 0.4 μg/ml)

(d) To aliquot #4, 5 ml of a 2 μg/ml Ca standard were added. This filled the volumetric flask to volume. (Added amount: a_3 = 1.0 μg/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 (μg/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	$a_3 = 1.0$	$A_3 = 0.116$

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).

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

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 x 2 = 2.44 μg/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 μg/ml.

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

2.36 μg/ml (answer)

$a_3 A_3$, added amount 1.0 μg/ml.

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

2.6 μg/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 ± SD (standard deviation) 0.008 ppm on the basis of quintuplicate 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:

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

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

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

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

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

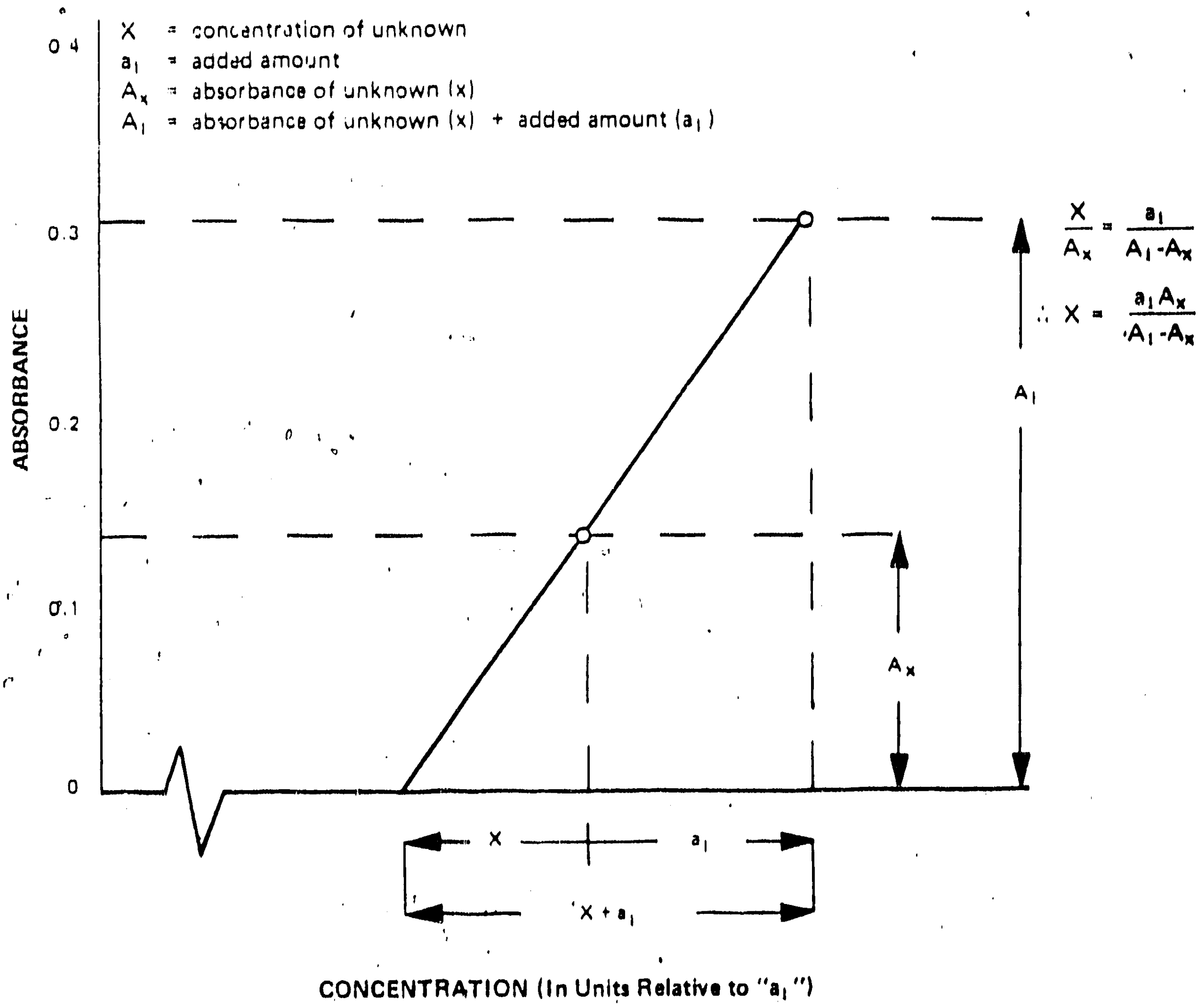


FIG. 6–12

Determination of Calcium by
Method of Standard Additions

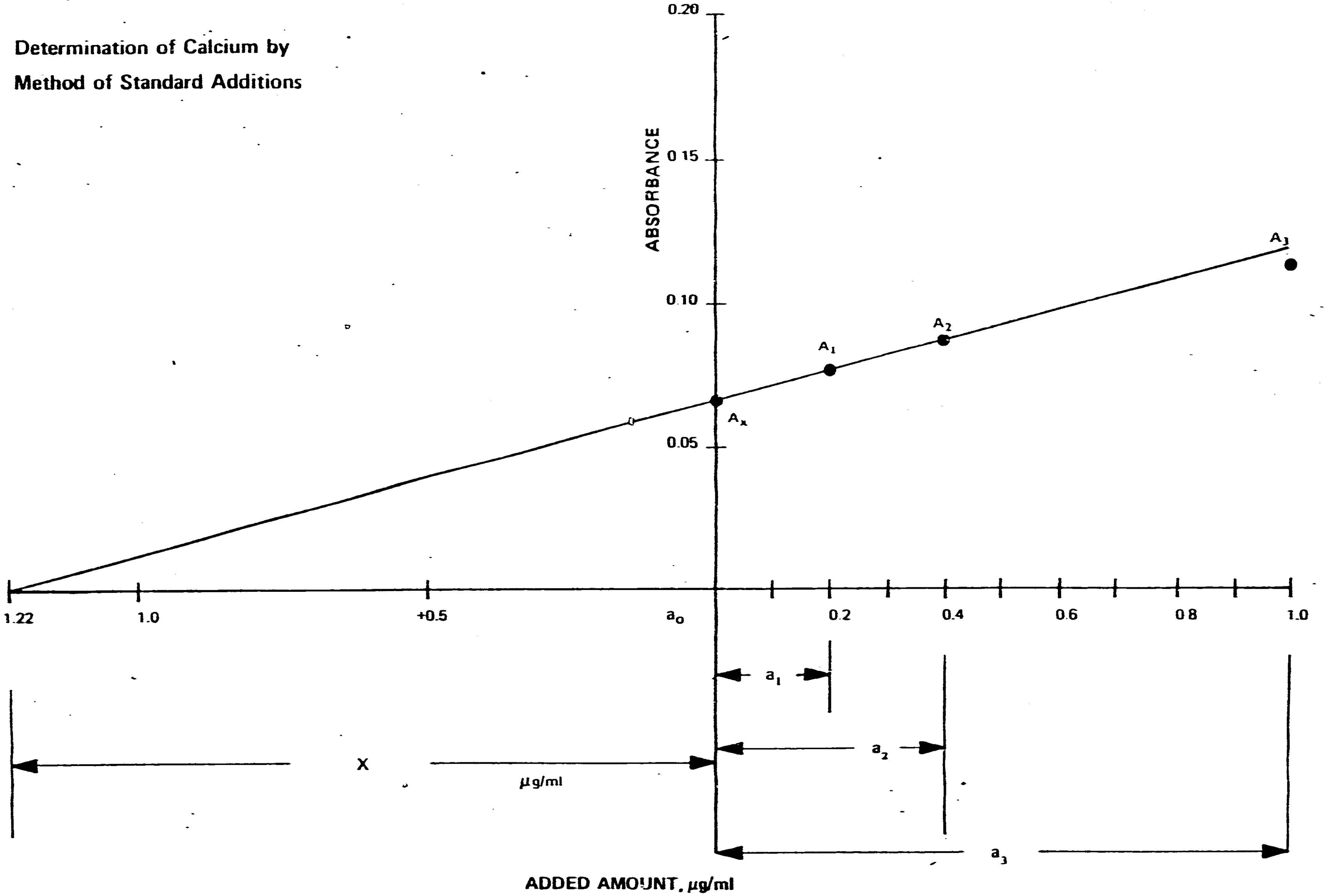


FIG. 6–13

APPENDIX F

ANALYTICAL DATA SUMMARY

RADIAN
CORPORATION

Radian Work Order 91-05-166

Analytical Report
06/28/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: Andy Ch

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Method:Alkalinity components (1)
List:

Sample ID:	DI LEACH LIMB- -EMPASH-12	METHOD BLANK
Factor:	1	1
Results in:	mg/L 04A	mg/L 05B
Matrix:	water	water

	Result	Det. Limit	Result	Det. Limit
Bicarbonate	ND	1.0	ND	1.0
Carbonate	112	1.0	ND	1.0
Total alkalinity	2335	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.

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Method:ICP 25 element scan SW6010 (1)
List:ICP, TCLP MS

Sample ID:	TCLP LEACH LI- MB-EMPASH-12	TCLP BLANK	METHOD BLANK
Factor:	20.00000	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	ND	6.0	ND	0.30	ND	0.30
Barium	0.73	0.010	ND	0.010	ND	0.010
Cadmium	ND	0.10	ND	0.0050	ND	0.0050
Chromium	0.010 @	0.010	ND	0.010	ND	0.010
Lead	ND	1.0	ND	0.050	ND	0.050
Silver	ND	0.20	ND	0.010	ND	0.010

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 Appendix A in this report.

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Method/Analyte	Sample Identifications					
	TCLP LEACH LI-MB-EMPASH-12			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	ND	mg/L	0.0050	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: 91-05-166

Method/Analyte	Sample Identifications					
	DI LEACH LIMB- -EMPASH-12 04			METHOD BLANK 05		
Matrix	water			water		
	Result		Det. Limit	Result		Det. Limit
Silver by ICPES SW6010						
Silver	ND	mg/L	0.040			
Arsenic by SW7060						
Arsenic	ND	mg/L	0.0040	ND	mg/L	0.0040
Barium by ICPES SW6010						
Barium	0.55	mg/L	0.010			
Calcium by ICPES SW6010						
Calcium	1600	mg/L	4.0	ND	mg/L	1.0
Cadmium by ICPES SW6010						
Cadmium	ND	mg/L	0.020			
Chloride, titration E325.3						
Chloride	240	mg/L	10	ND	mg/L	1.0
COD by SM508B						
Chemical Oxygen Demand	12 @	mg/L	6.0			
Chromium by ICPES SW6010						
Chromium	0.058	mg/L	0.010			
Fluoride by EPA 340.2						
Fluoride	2.3	mg/L	0.10	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	6.3 @	mg/L	3.0	ND	mg/L	3.0
Magnesium by ICPES SW6010						
Magnesium	ND	mg/L	1.0	ND	mg/L	1.0
Nitrate-nitrite, EPA 353.1						
Nitrate-Nitrite as N	0.38	mg/L	0.040	ND	mg/L	0.020
NACE corrosivity by SW1110						
Corrosivity	ND	mm/yr	N\A			
Sodium by ICPES, SW6010						
Sodium	4.0 @	mg/L	1.0	ND	mg/L	1.0
Lead by SW7421						
Lead	0.0035 @	mg/L	0.0030	ND	mg/L	0.0030
Total phenolics by SW9065						
Total phenolics	0.033 @	mg/L	0.010	ND	mg/L	0.010
pH, SW846						
pH	12	pH units				
Selenium by SW7740						
Selenium	0.024 @	mg/L	0.0050	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.

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Method/Analyte	DI LEACH LIMB- -EMPASH-12 04			METHOD BLANK 05		
Matrix	water			water		
	Result		Det. Limit	Result		Det. Limit
Sulfate, SW9038						
Sulfate	1900	mg/L	100	ND	mg/L	1.0
Tot. dissolv. solid E160.1						
Total dissolved solids	4900	mg/L	10	ND	mg/L	10
TOC by EPA 415.2						
Total organic carbon	1.9 @	mg/L	1.0	ND	mg/L	1.0

@ 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 the glossary.

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12	TCLP LEACH LI-MB-EMPASH-12	TCLP BLANK	DI LEACH LIMB--EMPASH-12	METHOD BLANK
Date Sampled	05/16/91	05/28/91		05/30/91	
Date Received	05/21/91	05/21/91	05/21/91	05/21/91	05/21/91
Matrix				water	water
	01	02	03	04	05
Silver by ICPES SW6010					
Prepared				06/04/91	
Analyzed				06/06/91	
Analyst				DRW	
File ID				JA610606-100	
Blank ID					
Instrument				JA61	
Report as				received	
Alkalinity components					
Prepared				06/12/91	06/12/91
Analyzed				06/12/91	06/12/91
Analyst				RDO	RDO
File ID					
Blank ID					
Instrument				636	636
Report as				received	received
Arsenic by SW7060					
Prepared				06/04/91	06/04/91
Analyzed				06/05/91	06/05/91
Analyst				RAA	RAA
File ID					
Blank ID					
Instrument				3030E	3030E
Report as				received	received
Barium by ICPES SW6010					
Prepared				06/04/91	
Analyzed				06/05/91	
Analyst				DRW	
File ID				JA610606-100	
Blank ID					
Instrument				JA61	
Report as				received	
Calcium by ICPES SW6010					
Prepared				06/04/91	06/04/91
Analyzed				06/06/91	06/05/91
Analyst				DRW	DRW
File ID				JA610606-100	
Blank ID					
Instrument				JA61	JA61
Report as				received	received

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12	TCLP LEACH LIMB-EMPASH-12	TCLP BLANK	DI LEACH LIMB-EMPASH-12	METHOD BLANK
Date Sampled	05/16/91	05/28/91		05/30/91	
Date Received	05/21/91	05/21/91	05/21/91	05/21/91	05/21/91
Matrix				water	water
	01	02	03	04	05

	01	02	03	04	05	
Cadmium by ICPES SW6010						
Prepared				06/04/91		
Analyzed				06/06/91		
Analyst				DRW		
File ID				JA610606-100		
Blank ID						
Instrument				JA61		
Report as				received		
Chloride, titration E325.3						
Prepared				06/03/91	06/03/91	
Analyzed				06/03/91	06/03/91	
Analyst				TRR	TRR	
File ID						
Blank ID						
Instrument				CLT	CLT	
Report as				received	received	
COD by SM508B						
Prepared				06/21/91		
Analyzed				06/21/91		
Analyst				TRR		
File ID						
Blank ID						
Instrument				COD		
Report as				received		
Chromium by ICPES SW6010						
Prepared				06/04/91		
Analyzed				06/05/91		
Analyst				DRW		
File ID				JA610606-100		
Blank ID						
Instrument				JA61		
Report as				received		
Fluoride by EPA 340.2						
Prepared				06/20/91	06/20/91	
Analyzed				06/20/91	06/20/91	
Analyst				EAT	EAT	
File ID						
Blank ID						
Instrument				925	925	
Report as				received	received	

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12	TCLP LEACH LIMB-EMPASH-12	TCLP BLANK	DI LEACH LIMB-EMPASH-12	METHOD BLANK	
Date Sampled	05/16/91	05/28/91		05/30/91		
Date Received	05/21/91	05/21/91	05/21/91	05/21/91	05/21/91	
Matrix		water	water	water	water	
	01	02	03	04	05	
Mercury, cold vapor E245.1						
Prepared		06/11/91	06/11/91	06/11/91	06/11/91	
Analyzed		06/11/91	06/11/91	06/11/91	06/11/91	
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		06/04/91	06/04/91		06/04/91	
Analyzed		06/11/91	06/05/91		06/05/91	
Analyst		DRW	DRW		DRW	
File ID		JA610611-64				
Blank ID						
Instrument		JA61	JA61		JA61	
Report as		received	received		received	
Potassium by ICPES SW6010						
Prepared				06/04/91	06/04/91	
Analyzed				06/05/91	06/05/91	
Analyst				DRW	DRW	
File ID				JA610606-100		
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Magnesium by ICPES SW6010						
Prepared				06/04/91	06/04/91	
Analyzed				06/05/91	06/05/91	
Analyst				DRW	DRW	
File ID				JA610606-100		
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Nitrate-nitrite, EPA 353.1						
Prepared				05/31/91	05/31/91	
Analyzed				05/31/91	05/31/91	
Analyst				RDO	RDO	
File ID				AA10531-26	AA10531-27	
Blank ID						
Instrument				AAI	AAI	
Report as				received	received	

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12	TCLP LEACH LIMB-EMPASH-12	TCLP BLANK	DI LEACH LIMB-EMPASH-12	METHOD BLANK	
Date Sampled	05/16/91	05/28/91		05/30/91		
Date Received	05/21/91	05/21/91	05/21/91	05/21/91	05/21/91	
Matrix				water	water	
	01	02	03	04	05	
NACE corrosivity by SW1110						
Prepared				06/17/91		
Analyzed				06/18/91		
Analyst				EAT		
File ID						
Blank ID						
Instrument				COR		
Report as				received		
Sodium by ICPES, SW6010						
Prepared				06/04/91	06/04/91	
Analyzed				06/05/91	06/05/91	
Analyst				DRW	DRW	
File ID				JA610606-100		
Blank ID						
Instrument				JA61	JA61	
Report as				received	received	
Lead by SW7421						
Prepared				06/04/91	06/04/91	
Analyzed				06/05/91	06/05/91	
Analyst				RAA	RAA	
File ID						
Blank ID						
Instrument				3030Z	3030Z	
Report as				received	received	
Total phenolics by SW9065						
Prepared				06/07/91	06/07/91	
Analyzed				06/07/91	06/07/91	
Analyst				MJS	MJS	
File ID						
Blank ID						
Instrument				AA II	AA II	
Report as				received	received	
pH, SW846						
Prepared				05/30/91		
Analyzed				05/30/91		
Analyst				MH		
File ID						
Blank ID						
Instrument				636		
Report as				received		

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12	TCLP LEACH LIMB-EMPASH-12	TCLP BLANK	DI LEACH LIMB-EMPASH-12	METHOD BLANK	
Date Sampled	05/16/91	05/28/91		05/30/91		
Date Received	05/21/91	05/21/91	05/21/91	05/21/91	05/21/91	
Matrix	solid	water	water	water	water	
	01	02	03	04	05	
Selenium by SW7740						
Prepared		06/04/91	06/04/91	06/04/91	06/04/91	
Analyzed		06/04/91	06/04/91	06/04/91	06/04/91	
Analyst		HD	HD	HD	HD	
File ID						
Blank ID						
Instrument		3030Z	3030Z	3030Z	3030Z	
Report as		received	received	received	received	
Sulfate, SW9038						
Prepared				06/11/91	06/11/91	
Analyzed				06/11/91	06/11/91	
Analyst				GMC	GMC	
File ID						
Blank ID						
Instrument				DC-80	DC-80	
Report as				received	received	
TCLP leaching						
Prepared	05/28/91		05/28/91			
Analyzed	05/29/91		05/29/91			
Analyst	SFM		SFM			
File ID						
Blank ID						
Instrument						
Report as	received		received			
Tot. dissolv. solid E160.1						
Prepared				06/04/91	06/06/91	
Analyzed				06/04/91	06/06/91	
Analyst				MH	MH	
File ID						
Blank ID						
Instrument						
Report as				received	received	
TOC by EPA 415.2						
Prepared				06/04/91	06/04/91	
Analyzed				06/04/91	06/04/91	
Analyst				GMC	GMC	
File ID						
Blank ID						
Instrument				DC-80	DC-80	
Report as				received	received	

Appendix A

Comments, Notes and Definitions

General Comments
The alkalinity for fraction -04A was due in part to caustic hydroxide.

Babcock and Wilcox Co.
Radian Work Order: 91-05-166

@ 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.

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; microSiemans/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
CORPORATION

Radian Work Order 91-07-035

Analytical Report
08/08/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: Andy Ca

Babcock and Wilcox Co.
Radian Work Order: 91-07-035

Method:Alkalinity components (1)		
List:		
Sample ID:	DI LEACH LIMB- -EMPASH-13	METHOD BLANK
Factor:	1	1
Results in:	mg/L 04A	mg/L 05B
Matrix:	water	water

	Result	Det. Limit	Result	Det. Limit		
Bicarbonate	ND	1.0	ND	1.0		
Carbonate	35	1.0	ND	1.0		
Total alkalinity	681	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: 91-07-035

Method:ICP 25 element scan SW6010 (1)
List:ICP, TCLP

Sample ID:	METHOD BLANK
Factor:	1
Results in:	mg/L 05A
Matrix:	water

	Result	Det. Limit			
Arsenic	ND	0.30			
Barium	ND	0.010			
Cadmium	ND	0.0050			
Calcium	ND	1.0			
Chromium	ND	0.010			
Lead	ND	0.050			
Magnesium	ND	1.0			
Potassium	ND	3.0			
Silver	ND	0.010			
Sodium	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: 91-07-035

Method:ICP 25 element scan SW6010 (1)
List:ICP, TCLP
Sample ID: DI LEACH LIMB-
-EMPASH-13
Factor: 1
Results in: mg/L
04B
Matrix: water

	Result	Det. Limit
Barium	0.20	0.010
Cadmium	ND	0.0050
Calcium	360	1.0
Chromium	ND	0.010
Magnesium	ND	1.0
Potassium	120	3.0
Silver	ND	0.010
Sodium	17	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: 91-07-035

Method:ICP 25 element scan SW6010 (1)		
List:ICP, TCLP MS		
Sample ID:	TCLP LIMB-EMP-ASH-13	TCLP BLANK
Factor:	4	1
Results in:	mg/L 02A	mg/L 03B
Matrix:	water	water

	Result	Det. Limit	Result	Det. Limit
Arsenic	ND	1.2	ND	0.30
Barium	0.69	0.010	ND	0.010
Cadmium	ND	0.020	ND	0.0050
Chromium	0.16	0.010	ND	0.010
Lead	ND	0.20	ND	0.050
Silver	ND	0.040	ND	0.010

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: 91-07-035

Method/Analyte	Sample Identifications					
	TCLP LIMB-EMP-ASH-13 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	0.075	mg/L	0.0050	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: 91-07-035

Method/Analyte	Sample Identifications					
	DI LEACH LIMB- -EMPASH-13			METHOD BLANK		
	04			05		
Matrix	water			water		
	Result		Det. Limit	Result		Det. Limit
Arsenic by SW7060						
Arsenic	0.0047	mg/L	0.0040	ND	mg/L	0.0040
Chloride, titration E325.3						
Chloride	66	mg/L	1.0	ND	mg/L	1.0
COD by SM508B						
Chemical Oxygen Demand	9.4	mg/L	6.0			
Fluoride by EPA 340.2						
Fluoride	0.95	mg/L	0.10	ND	mg/L	0.10
Mercury, cold vapor E245.1						
Mercury	ND	mg/L	0.0002	ND	mg/L	0.0002
Nitrate-nitrite, EPA 353.1						
Nitrate-Nitrite as N	ND	mg/L	0.020	ND	mg/L	0.020
NACE corrosivity by SW1110						
Corrosivity	ND	mm/yr	N\A			
Lead by SW7421						
Lead	ND	mg/L	0.0030	ND	mg/L	0.0030
Total phenolics by SW9065						
Total phenolics	ND	mg/L	0.010	ND	mg/L	0.010
pH, SW846						
pH	12	pH units		ND	pH units	
Selenium by SW7740						
Selenium	0.0053	mg/L	0.0050	ND	mg/L	0.0050
Sulfate, SW9038						
Sulfate	330	mg/L	20	1.8	mg/L	1.0
Tot. dissolv. solid E160.1						
Total dissolved solids	1400	mg/L	10	ND	mg/L	10
TOC by EPA 415.2						
Total organic carbon	2.1	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: 91-07-035

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-13	TCLP LIMB-EMP-ASH-13	TCLP BLANK	DI LEACH LIMB-EMPASH-13	METHOD BLANK
Date Sampled	07/02/91	07/08/91		07/11/91	
Date Received	07/03/91	07/03/91	07/03/91	07/03/91	07/03/91
Matrix				water	water
	01	02	03	04	05
Alkalinity components					
Prepared				07/24/91	07/24/91
Analyzed				07/24/91	07/24/91
Analyst				RDO	RDO
File ID					
Blank ID					
Instrument				636	636
Report as				received	received
Arsenic by SW7060					
Prepared				07/12/91	07/12/91
Analyzed				07/16/91	07/16/91
Analyst				HD	HD
File ID					
Blank ID					
Instrument				3030Z	3030Z
Report as				received	received
Chloride, titration E325.3					
Prepared				07/29/91	07/29/91
Analyzed				07/29/91	07/29/91
Analyst				TRR	TRR
File ID				CLT0729-4	
Blank ID					
Instrument				CLT	CLT
Report as				received	received
COD by SM508B					
Prepared				07/19/91	
Analyzed				07/22/91	
Analyst				TRR	
File ID				COD0722-5	
Blank ID					
Instrument				COD	
Report as				received	
Fluoride by EPA 340.2					
Prepared				07/22/91	07/22/91
Analyzed				07/22/91	07/22/91
Analyst				EAT	EAT
File ID				9250722-1	
Blank ID					
Instrument				925	925
Report as				received	received

Babcock and Wilcox Co.
Radian Work Order: 91-07-035

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-13	TCLP LIMB-EMP-ASH-13	TCLP BLANK	DI LEACH LIMB--EMPASH-13	METHOD BLANK
Date Sampled	07/02/91	07/08/91		07/11/91	
Date Received	07/03/91	07/03/91	07/03/91	07/03/91	07/03/91
Matrix		water	water	water	water
	01	02	03	04	05
Mercury, cold vapor E245.1					
Prepared		07/11/91	07/11/91	07/11/91	07/11/91
Analyzed		07/11/91	07/11/91	07/11/91	07/11/91
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					07/12/91
Analyzed					07/15/91
Analyst					DRW
File ID					
Blank ID					
Instrument					JA61
Report as					received
ICP 25 element scan SW6010					
Prepared				07/12/91	
Analyzed				07/15/91	
Analyst				DRW	
File ID					
Blank ID					
Instrument				JA61	
Report as				received	
ICP 25 element scan SW6010					
Prepared		07/12/91	07/12/91		
Analyzed		07/24/91	07/15/91		
Analyst		DRW	DRW		
File ID		JA610724-39			
Blank ID					
Instrument		JA61	JA61		
Report as		received	received		
Nitrate-nitrite, EPA 353.1					
Prepared				08/06/91	08/06/91
Analyzed				08/06/91	08/06/91
Analyst				RDO	RDO
File ID				AA10806-13	AA10806-14
Blank ID					
Instrument				AAI	AAI
Report as				received	received

Babcock and Wilcox Co.
Radian Work Order: 91-07-035

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-13	TCLP LIMB-EMP-ASH-13	TCLP BLANK	DI LEACH LIMB--EMPASH-13	METHOD BLANK
Date Sampled	07/02/91	07/08/91		07/11/91	
Date Received	07/03/91	07/03/91	07/03/91	07/03/91	07/03/91
Matrix		water	water	water	water
	01	02	03	04	05

	01	02	03	04	05
NACE corrosivity by SW1110					
Prepared				07/25/91	
Analyzed				07/25/91	
Analyst				EAT	
File ID				COR0725-1	
Blank ID					
Instrument				COR	
Report as				received	
Lead by SW7421					
Prepared				07/12/91	07/12/91
Analyzed				07/15/91	07/15/91
Analyst				HD	HD
File ID					
Blank ID					
Instrument				3030Z	3030Z
Report as				received	received
Total phenolics by SW9065					
Prepared				08/01/91	08/01/91
Analyzed				08/01/91	08/01/91
Analyst				MJS	MJS
File ID				AAI10801-	AAI10801-
Blank ID					
Instrument				AAII	AAII
Report as				received	received
pH, SW846					
Prepared				07/12/91	07/12/91
Analyzed				07/12/91	07/12/91
Analyst				EAT	EAT
File ID					
Blank ID					
Instrument				925	925
Report as				received	received
Selenium by SW7740					
Prepared		07/12/91	07/12/91	07/12/91	07/12/91
Analyzed		07/29/91	07/29/91	07/29/91	07/29/91
Analyst		HD	HD	HD	HD
File ID					
Blank ID					
Instrument		3030Z	3030Z	3030Z	3030Z
Report as		received	received	received	received

Babcock and Wilcox Co.
Radian Work Order: 91-07-035

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-13	TCLP LIMB-EMPASH-13	TCLP BLANK	DI LEACH LIMB--EMPASH-13	METHOD BLANK
Date Sampled	07/02/91	07/08/91		07/11/91	
Date Received	07/03/91	07/03/91	07/03/91	07/03/91	07/03/91
Matrix	solid		solid	water	water
	01	02	03	04	05

Sulfate, SW9038						
Prepared				07/29/91	07/29/91	
Analyzed				07/29/91	07/29/91	
Analyst				GMC	GMC	
File ID				21000729-9		
Blank ID						
Instrument				2100A	2100A	
Report as				received	received	
TCLP leaching						
Prepared	07/08/91		07/08/91			
Analyzed	07/09/91		07/09/91			
Analyst	SFM		SFM			
File ID						
Blank ID						
Instrument						
Report as	received		received			
Tot. dissolv. solid E160.1						
Prepared				07/16/91	07/16/91	
Analyzed				07/16/91	07/16/91	
Analyst				EAT	EAT	
File ID				TDS0716-1	TDS0716-1	
Blank ID						
Instrument				TDS	TDS	
Report as				received	received	
TOC by EPA 415.2						
Prepared				07/15/91	07/15/91	
Analyzed				07/15/91	07/15/91	
Analyst				GMC	GMC	
File ID				DC-80715-33		
Blank ID						
Instrument				DC-80	DC-80	
Report as				received	received	

Appendix A

Comments, Notes and Definitions

General Comments

For alkalinity, 9207035-04A was determined to be caustic.

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.

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; microSiemans/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

For Arsenic by SW7060 the following should be noted:

The results for this analyte are less than five times the method specified detection limit.

DI LEACH LIMB-EMPASH-13	Arsenic

For COD by SM508B the following should be noted:

DI LEACH LIMB-EMPASH-13	Chemical Oxygen Demand

For Selenium by SW7740 the following should be noted:

DI LEACH LIMB-EMPASH-13	Selenium

For Sulfate, SW9038 the following should be noted:

METHOD BLANK	Sulfate

For TOC by EPA 415.2 the following should be noted:

DI LEACH LIMB-EMPASH-13	Total organic carbon

RADIAN
CORPORATION

Radian Work Order 91-08-166

Analytical Report

09/18/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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

512/454-4797

Client Services Coordinator: KAYOUNG

Certified by: ____________________

APPENDIX G

PERMEABILITY DATA SUMMARY

RADIAN
CORPORATION

Radian Work Order 91-04-177

Analytical Report
05/23/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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: 91-04-177

Method/Analyte	Sample Identifications LIMB-EMPASH-11 01			
Matrix	solid			
	Result	Det. Limit		
Permeability SW9100				
Permeability	0.000039 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: 91-04-177

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-11					
Date Sampled	04/15/91					
Date Received	04/16/91					
Matrix	solid					
	01					
Permeability SW9100						
Prepared	05/21/91					
Analyzed	05/21/91					
Analyst	GAT					
File ID						
Blank ID						
Instrument						
Report as	received					

Appendix A

Comments, Notes and Definitions

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; microSiemans/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
CORPORATION

Radian Work Order 91-05-167

Analytical Report
06/25/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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: 91-05-167

Sample Identifications

Method/Analyte	LIMB-EMPASH-12 01			
Matrix	solid			
	Result	Det. Limit		
Permeability SW9100				
Permeability	0.0000099 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: 91-05-167

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-12
Date Sampled	05/16/91
Date Received	05/21/91
Matrix	solid
	01

Permeability SW9100						
Prepared						
Analyzed	06/21/91					
Analyst	GST					
File ID						
Blank ID						
Instrument						
Report as	received					

Appendix A

Comments, Notes and Definitions

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; microSiemans/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
CORPORATION

Radian Work Order 91-07-034

Analytical Report
08/09/91

Babcock and Wilcox Co.

Radian
RTP
NC

Luke Contos

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

Contents:

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: 91-07-034

Method/Analyte	Sample Identifications			
	LIMB-EMPASH-13			
	01			
Matrix	solid			
	Result	Det. Limit		
Permeability SW9100				
Permeability	0.0000031 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: 91-07-034

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-13
Date Sampled	07/02/91
Date Received	07/03/91
Matrix	solid
	01

Permeability SW9100						
Prepared						
Analyzed	08/06/91					
Analyst	GST					
File ID						
Blank ID						
Instrument						
Report as	received					

Appendix A

Comments, Notes and Definitions

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; microSiemans/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
CORPORATION

Radian Work Order 91-08-165

Analytical Report
09/20/91

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: Linda Carter

Analytical Data Summary

Babcock and Wilcox Co.
Radian Work Order: 91-08-165

Method/Analyte	Sample Identifications			
	LIMB-EMPASH-14		LIMB-EMPASH-15	
	01		02	
Matrix	solid		solid	
	Result	Det. Limit	Result	Det. Limit
Permeability SW9100				
Permeability	0.0000078 cm/sec	N\A	0.000032 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: 91-08-165

Sample Identifications and Dates

Sample ID	LIMB-EMPASH-14	LIMB-EMPASH-15
Date Sampled	08/16/91	08/16/91
Date Received	08/17/91	08/17/91
Matrix	solid	solid
	01	02

Permeability SW9100						
Prepared						
Analyzed	09/19/91	09/19/91				
Analyst	GST	GST				
File ID						
Blank ID						
Instrument						
Report as	received	received				

Appendix A

Comments, Notes and Definitions

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; microSiemans/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 SO_2, NO_x, PM/PM_{10}, 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 demonstation 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 SO_2, NO_x, CO, and PM/PM_{10} 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 methodolgy 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 $\mu g/m^3$ (SO_2, PM/PM_{10}, NO_x) for the annual average, 5 $\mu g/m^3$ (SO_2, PM/PM_{10}) for the 24-hour average, 25 $\mu g/m^3$ (SO_2) for the 3-hour average, 500 $\mu g/m^3$ (CO) for the 1-hour average, and 2000 $\mu g/m^3$ (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

HOURLY SURFACE DATA
JULY 1991
CLEVELAND/HOPKINS AIRPORT

DAY	HOUR	CEILING (100's ft)	WIND DIRECTION (degrees)	WIND SPEED (knots)	TEMPERATURE (F)	CLOUD COVER (tenths)
6	1	0	230	6	71	0
6	2	90	220	5	71	6
6	3	0	230	4	72	5
6	4	0	250	5	71	3
6	5	0	200	4	67	4
6	6	0	230	5	70	2
6	7	0	220	8	75	3
6	8	250	250	11	78	10
6	9	250	230	12	81	10
6	10	250	240	10	84	10
6	11	250	290	12	86	8
6	12	250	240	11	89	8
6	13	250	300	11	92	7
6	14	250	360	9	92	7
6	15	0	360	7	92	5
6	16	0	120	4	90	4
6	17	0	230	4	93	4
6	18	0	220	8	91	3
6	19	0	210	10	90	2
6	20	0	210	8	86	2
6	21	0	220	8	83	1
6	22	0	220	10	83	1
6	23	0	210	10	82	4
6	24	130	210	11	82	10
7	1	130	220	10	81	9
7	2	250	230	8	80	10
7	3	130	220	9	82	9
7	4	250	240	11	82	9
7	5	75	240	11	81	9
7	6	0	240	12	81	4
7	7	0	240	11	82	3
7	8	250	250	14	82	7
7	9	50	270	14	84	8
7	10	55	250	12	87	8
7	11	55	240	14	87	8
7	12	60	240	17	90	8
7	13	250	290	12	92	8
7	14	250	310	11	94	8
7	15	250	360	10	94	9
7	16	0	240	10	93	3
7	17	0	250	8	94	3
7	18	0	230	10	91	3
7	19	130	250	10	88	10
7	20	130	220	8	85	8
7	21	130	290	8	82	10
7	22	33	310	19	75	10
7	23	85	200	6	71	10
7	24	75	200	9	71	10

MIXING HEIGHT DATA
BUFFALO,NY

STATION ID	YEAR	MONTH	DAY	MORNING MIXING HEIGHT (m)	AFTERNOON MIXING HEIGHT (m)
14733	91	7	5	NA	546
14733	91	7	6	620	730
14733	91	7	7	544	2019
14733	91	7	8	1551	NA

END

DATE
FILMED

7 / 21 / 92