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EPRI

Field Chemical Emissions Monitoring Project: Site 20 Emissions Report

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March 21, 1994

Mr. William H. Maxwell, P.E. (MD13)
Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

Dear Mr. Maxwell:

In response to the Clean Air Act Amendments of 1990, the Electric Power Research Institute (EPRI) initiated the PISCES (Power Plant Integrated Systems: Chemical Emissions Studies) program to better characterize the source, distribution, and fate of trace elements from utility fossil-fuel-fired power plants. As part of the PISCES program, the Field Chemical Emissions Monitoring (FCEM) program has sampled extensively at a number of utility sites, encompassing a range of fuels, boiler configurations, and particulate, SO_2, and NO_x control technologies. EPRI is actively pursuing additional FCEM sampling programs, with 29 sites either completed or planned.

This site report presents a preliminary summary of data gathered during a sampling program conducted at one of the FCEM sampling programs - Site 20. Site 20 consists of a 680 MW pulverized coal-fired boiler burning a lignite coal, with an electrostatic precipitator (ESP) for particulate control and a wet-limestone flue gas desulfurization (FGD) system for SO_2 control. In the Site 20 sampling and analytical program, mercury speciation measurements were conducted using the Nick Bloom/Frontier Geoscience's solid sorbent speciation train. Recently, it was determined that the analytical recovery procedure could lead to the formation of methyl Hg. This recent finding affected the methyl Hg results at Site 20 as well as all previous field sites by EPRI and other organizations. The methyl Hg measurements are considered invalid and are not included in this report. The methyl Hg and the Hg^{+2} are summed together to obtain a total oxidized Hg. At this time, EPRI is not able to quantify methyl Hg in flue gas. EPRI is following up with additional studies to evaluate this analytical artifact.

It should be noted that the results presented in this report are considered PRELIMINARY. The results are believed to be essentially correct except as noted. As additional data from other sites are collected and evaluated, however, EPRI may conduct verification tests at this site. If this is done, the new data will be made available to the Environmental Protection Agency (EPA).

The primary objective of this report is to transmit the preliminary results from Site 20 to the EPA for use in evaluating select trace chemical emissions from fossil-fuel-fired steam generating plants. In addition to the raw data in the Appendix, the report provides an assessment of the trace metals material balances, discusses the data quality, identifies suspect data, and offers possible explanations for the questionable data. Because the discussion only focuses upon the suspect or invalidated data, please keep in mind that most of the data meet the standards of quality established for this study. This report does not compare the results from Site 20 with the results from previous utility sites. Generic conclusions and recommendations were not drawn concerning the effectiveness of an ESP or wet FGD system as potential control technologies for trace elements; however, removal efficiencies were calculated where possible. Nor does this site report attempt to address the environmental and health risk impacts associated with the trace chemical emissions.

EPRI hopes that this site report is of assistance to the EPA in evaluating utility trace chemical emissions as well as the associated health risk impacts.

Sincerely,

Paul Chu
Manager, Toxic Substances Characterization
Environment Division

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INTRODUCTION

This report summarizes field data gathered during June 1993, by Radian Corporation at a power plant designated as Site 20. The Electric Power Research Institute (EPRI) sponsored this effort as part of its Field Chemical Emissions Monitoring Project (FCEM, RP-3177). The primary objective of this project is to measure the concentrations of selected inorganic and organic substances in power plant process and discharge streams. The data are being used to determine the fate and control of these substances.

The primary objectives of this report are to summarize fuel and gas concentration data for Site 20 and to evaluate these data according to the criteria outlined below. The concentration data are in a format suitable for use by the U.S. Environmental Protection Agency to study emissions from fossil-fuel-fired power plants, as mandated by the Clean Air Act Amendments of 1990.

Site 20 has an opposed wall-fired boiler and burns medium-sulfur lignite coal. Emissions are controlled by an electrostatic precipitator (ESP) and a wet-limestone flue gas desulfurization (FGD) system.

Test Objectives

The four major objectives for testing at Site 20 were to:

- Measure the emissions from a lignite-fired power plant equipped with an ESP/wet FGD system.

- Measure the emission control efficiency of a wet FGD system on a lignite-fired power plant.

- Collect size-fractionated fly ash from a lignite-fired power plant. The various size fractions may be analyzed for trace element concentrations in the future.

- Compare two methods for determining mercury concentrations in flue gas. This effort compared the EPA multi-metals method (Draft Method 29) with the mercury speciation method developed by Frontier Geosciences.

The first three objectives address the lack of information available for describing trace element emissions from lignite-fired power plants. These objectives are important because a significant number of plants burn lignite coal. The last objective is important because previous comparisons at different sites have shown fairly good agreement between the total flue gas mercury concentrations as determined by EPA Method 29 and by the mercury speciation method. The data from Site 20 augments the comparison between these two methods.

Table 1-1 lists the substances of interest to the FCEM project. A subset of these substances was chosen for study at Site 20 (i.e., no organic substance concentrations were determined).

Process Operation

The unit operated at high, steady load during each test run, although an upset in the ESP operation occurred during the second day of testing. Also, for all tests, the FGD system treated more gas than it does in normal operation because no flue gas was bypassed around the FGD system when FCEM testing was in progress (the normal operation is to bypass some gas around the FGD system while still complying with the SO_2 emission regulations). The impact of these operations is minimal with respect to the measurement results. Both of these topics and the impacts on test data are discussed in Section 4. By all other indicators, process operation during testing was representative of normal operation for this unit.

Sampling and Analysis Protocol

Appendix A describes the sampling and analysis protocol for Site 20. The methods used are comparable to those used at other FCEM sites sampled by Radian, with the following exceptions:

- In addition to ICP-AES analysis (employed at other FCEM sites), flue gas samples were analyzed for chromium and nickel using GFAAS.
- The reported selenium concentrations in the flue gas streams were based on samples analyzed by ICP-MS, instead of GFAAS (as was employed at other FCEM sites).

Quality Assurance/Quality Control (QA/QC)

The completeness of the quality assurance data was reviewed to judge whether the quality of the measurement data could be evaluated with the available information. In general, the results of the QC checks available for Site 20 indicate that the samples are well characterized. An evaluation of the accuracy, precision, and bias of the data, even if only on a qualitative level, is considered to be an important part of the data evaluation. A full discussion of each of these components can be found in Section 4.

Standard QA/QC checks for this type of sampling program involve the use of: 1) replicate tests, duplicate field samples and lab analyses, and matrix spike and lab control duplicates to determine precision; 2) matrix spikes, surrogate spikes, and laboratory control samples to determine accuracy; and 3) field blanks, trip blanks, method blanks, and reagent blanks to determine if any of the samples were

Table 1-1
FCEM Substances of Interest

Elements	Organic Compounds
Arsenic	Benzene[a]
Barium	Toluene[a]
Beryllium	Formaldehyde[a]
Cadmium	Polycyclic Organic Matter (POM)[a,b]
Chlorine (as chloride)	
Chromium	
Cobalt	
Fluorine (as fluoride)	
Lead	
Manganese	
Mercury	
Molybdenum	
Nickel	
Phosphorus	
Selenium	
Vanadium	

[a] Not measured at Site 20.

[b] Also referred to as semivolatile organic compounds. Includes polynuclear aromatic hydrocarbons (PAHs).

contaminated during collection or analysis. Most of these standard QA/QC checks were used on samples from Site 20, except for surrogate spikes (which do not apply to metals and anions analyses) and the duplicate analysis of samples. The absence of any of these "standard" quality control checks does not necessarily reflect poorly on the quality of the data but does limit the ability to measure the various components of measurement error.

Data Quality

The QA/QC results were compared to the data quality objectives shown in Section 4. QA/QC results outside the data quality objectives are noted and discussed, other quality assurance values are evaluated, and the potential effect on data quality is noted. Based on the detailed information presented in Section 4, the following conclusions can be made:

- Arsenic concentrations in the coal may be biased low. The measured concentration for a certified coal was only 43% of the certified value.
- Cobalt concentrations in the coal may be biased high. The measured concentration for a certified coal was 193% of the certified value.
- Selenium QA/QC data for impinger solutions exhibited poor precision and accuracy for GFAAS analyses. Therefore, the ICP-MS data were used to report selenium concentrations in the gas streams and to calculate the selenium emission factors and material balance closures.
- The validity of the mercury speciation data is suspect. Frontier Geosciences has stated that all reported values for methyl mercury are biased high and may in fact be ionic mercury.

Report Organization

Section 2 of this report describes the plant and the sample locations. Section 3 presents the concentration data for the coal and gas streams. Section 4 discusses the QA/QC and engineering evaluations of the data. Section 5 presents additional data. Section 6 presents example calculations, and a glossary of terms is provided in Section 7. The appendices contain information on sampling and analytical methods, stream concentrations, sampling data, error propagation equations, and detailed QA/QC data.

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SITE DESCRIPTION

The FCEM project has a policy of assigning a site code to each plant sampled. The plant discussed in this report was designated Site 20. This section describes the test site and the sampling locations.

Facility Information

Site 20 has one lignite-fired boiler with a normal full-load value of 680 MWe. The wall-fired, sub-critical, drum-type boiler was designed by Babcock and Wilcox and began operation in the mid-1980s. The furnace consists of a single chamber with no partition. Table 2-1 summarizes the unit design values.

Figure 2-1 presents a process flow diagram of Site 20. The plant burns lignite coal from a nearby mine. The lignite has typical ash, moisture, and sulfur levels of 11, 33, and 1.3%, respectively. Bottom ash is removed from the boiler by an ash sluicing system, and electrostatic precipitators (ESPs) remove fly ash from the flue gas. The plant has a FGD system that removes approximately 90% of the sulfur dioxide (SO_2) from the flue gas. The flue gas treatment and solids handling facilities are described in greater detail below.

Flue Gas Treatment Facilities

Site 20 is equipped with two parallel cold-side ESPs that have weighted-wire discharge electrodes. The collection plate spacing is 9 inches and the plate height is 36 feet. The design specific collection area (SCA) is 544 $ft^2/10^3$ acfm. The ESP outlet gas streams pass through induced draft fans (two fans total) before they are combined, just downstream of the fans. Most of the combined gas enters the FGD system, but a fraction is bypassed around the FGD system. The normal operation of the plant is to bypass as much gas as possible, while still meeting its required SO_2 emission limits. During FCEM testing, the plant operated with essentially no flue gas bypass, based on energy and material balance calculations using flue gas temperatures and SO_2 concentrations. The impacts of operating without flue gas bypass are discussed in Section 4.

The FGD system (Figure 2-2) is a dual-loop system that uses limestone slurry for reagent. Emulsified sulfur and ethylene-diamine-tetra-acetic acid (EDTA) are added to the system to control the sulfite oxidation to about 15 percent. The emulsified sulfur is added to the limestone slurry storage tank once every one to three days at a rate required to maintain a dissolved thiosulfate ($S_2O_3^-$) concentration of about 2,000 ppm in the FGD liquor. The EDTA is normally added once a month, at a rate calculated to

Table 2-1
Site 20 Summary

Maximum gross electrical output (MW)	720 (normal full load = 680)
Boiler type	Opposed wall-fired, subcritical
Boiler additives	None
Fuel type	Lignite
Fuel sulfur content (avg. % S, as received)	1.3
Fuel ash content (avg. %, as received)	11
Fuel heating value (avg. Btu/lb, as received)	6,760
Particulate controls	Cold-side ESPs
ESP design efficiency (%)	99.75
ESP design SCA (ft^2/kacfm)	544
SO_3 flue gas conditioning	None
SO_2 emission limits (lb/10^6 Btu)	1.2
SO_2 controls[b]	Wet limestone FGD Dual-loop, UOP designed Inhibited oxidation Sulfur addition to form S_2O_3 EDTA addition[a]
Number of FGD modules	4[b]
Gas/module (acfm)	660,000
Module design efficiency (%)	90
Fly ash disposal	Landfill
Bottom ash disposal	Pond
Bottom ash sluice water source	Bottom ash pond
Cooling water system	Once through
Cooling water source	Lake

[a] No EDTA was added to the system during FCEM testing.

[b] Dibasic acid (DBA) was present in all FGD system modules during FCEM testing.

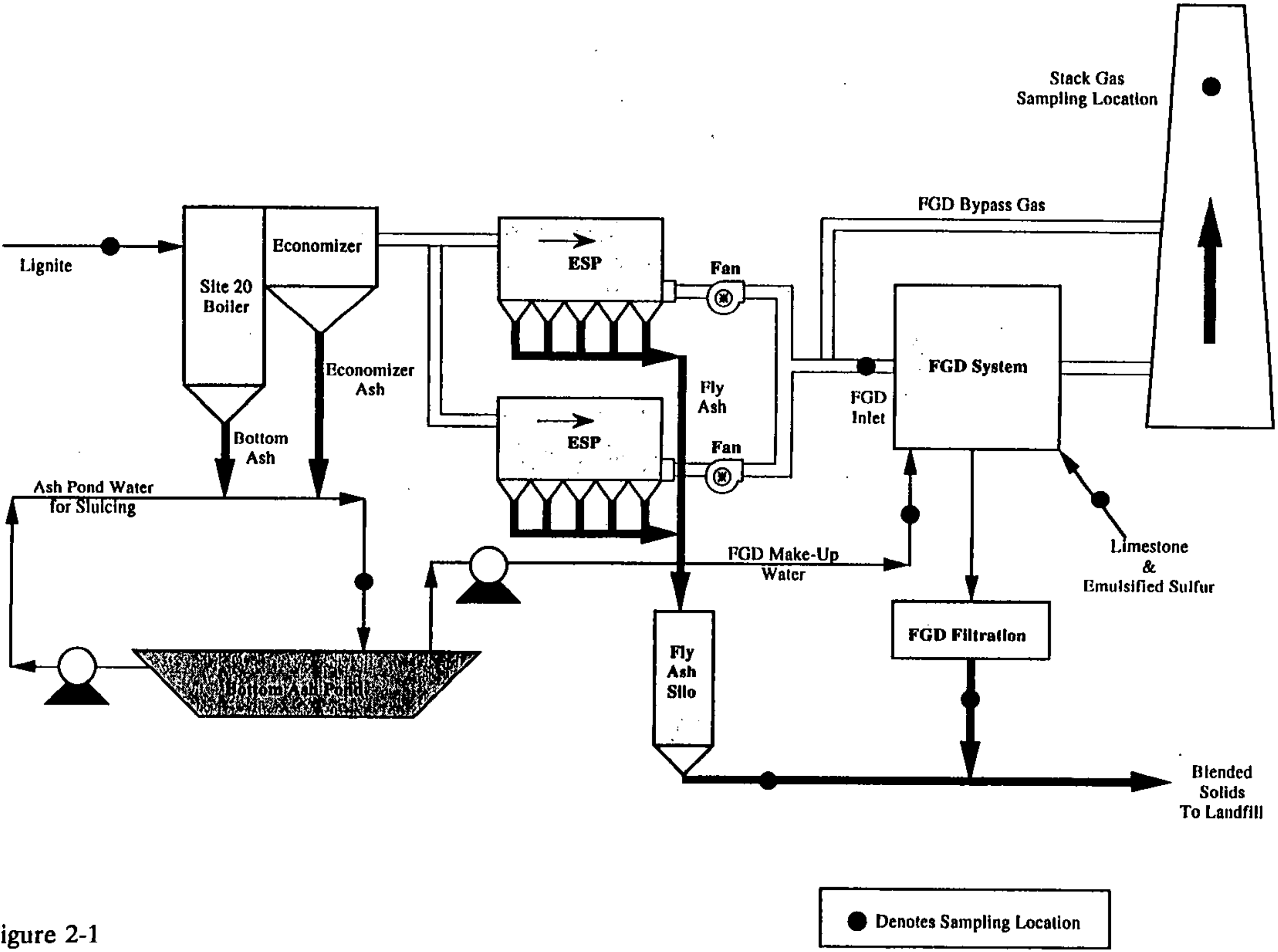

Figure 2-1
Process Flow Diagram for Site 20

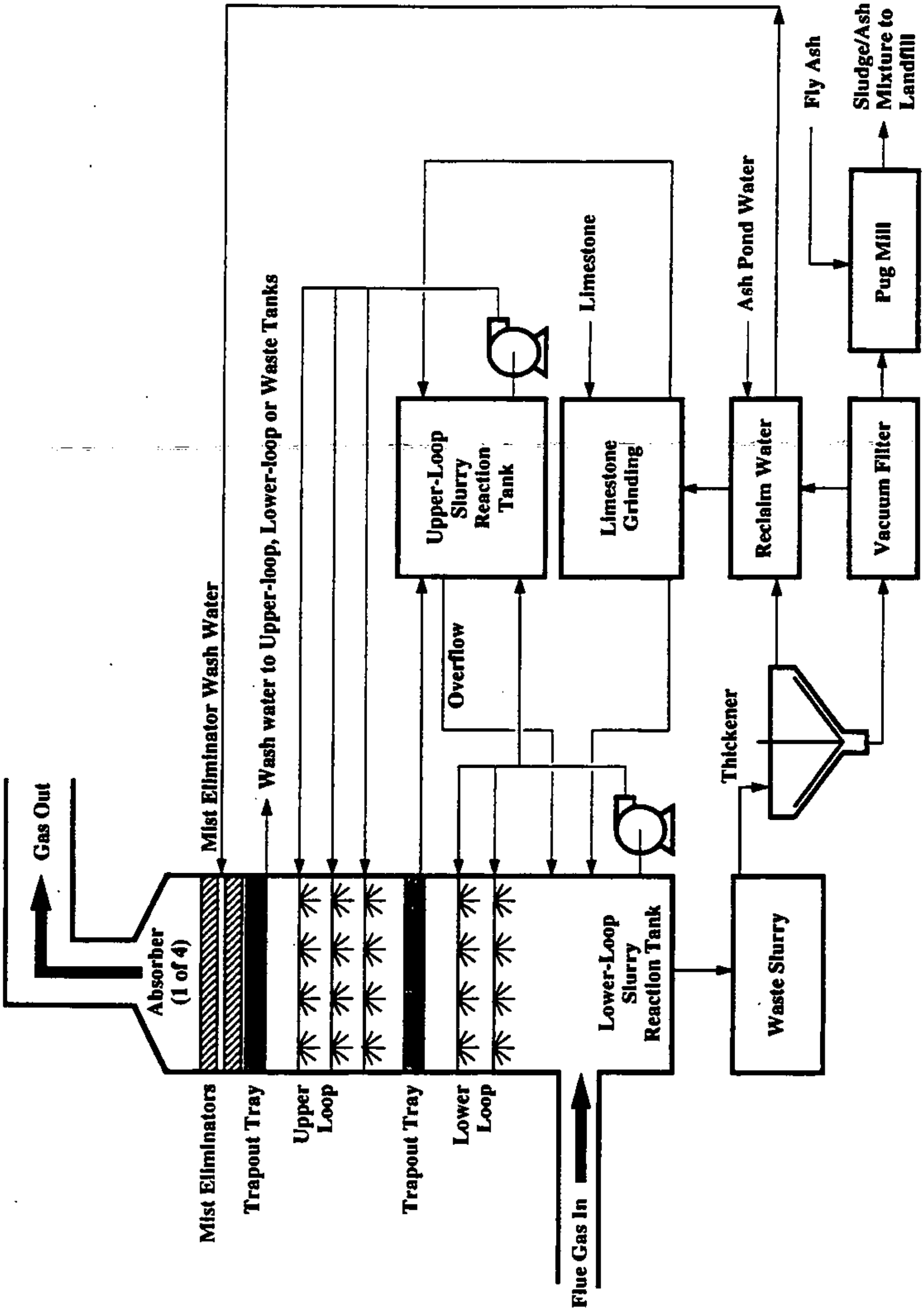

Figure 2-2
Simplified Process Flow Diagram for Site 20's FGD System

maintain 20 ppm in the FGD liquor. No EDTA was added to the system during FCEM sampling activities. Dibasic acid (DBA), an additive for improving SO_2 removal, was present at significant concentrations during FCEM testing. The presence of DBA in the FGD system was not typical for Site 20, but it is doubtful that the DBA affected the test data because the removal of trace metals by the FGD system should not be affected by the increased liquid-phase alkalinity caused by the presence of DBA (i.e., the removal of trace metals does not depend on the liquid-phase alkalinity). Radian sampled the FGD system to document the DBA levels during the field testing effort. The DBA concentration during the test period was nominally 400 ppm (Section 5).

The FGD system consists of four absorber modules, each of which treats 660,000 acfm of flue gas. All four modules are required when the boiler operates at full-load conditions. The design L/G's are 17 and 35 gal/kacfm for the upper and lower loops, respectively. The system has 4 reaction tanks: two for the upper loops and two for the lower loops. Each reaction tank is shared by two absorber modules.

Solids Handling Facilities

Lignite coal is excavated from a nearby mine and trucked to the plant site. The plant has seven coal bunkers, which feed the coal pulverizers. The residence time downstream of the bunkers is insignificant (seconds) compared to the residence time in the coal bunkers (12 hours).

FGD solids are dewatered only at night. That is, the FGD system's thickener underflow is stored during the day shift and dewatered at night using rotary vacuum filters.

Dry fly ash collected by the ESPs is pneumatically conveyed to an ash silo on a continual basis. At night, the ash is removed from the silo and blended with the FGD filter cake solids. The blended material is sent to an on-site landfill for disposal.

Bottom ash is removed from the boiler and intermittently sluiced to a bottom ash disposal pond. The sluicing water supply comes from the bottom ash pond. The ash pond water is made up with rainwater or with water from a nearby lake when necessary.

Sampling Locations

Samples were collected at several locations in the plant. These locations are identified on the process flow diagram, Figure 2-1. Brief descriptions of each sampling location are given below:

- Coal composite samples were collected from four of the seven coal bunkers at a location near the coal mills. The coal obtained at this location was considered to be more representative than the daily composite coal sample obtained by the plant because of the long residence time in the coal bunkers. Individual coal samples were collected every two hours during each test. These samples were combined to form the composite coal sample for the test.

- Flue gas exiting the ESPs was sampled from a horizontal duct located just downstream of the induced draft fans and FGD bypass takeoff ductwork.
- Flue gas was also sampled at the stack at a location well downstream of where the FGD outlet gas combines with the FGD bypass gas.
- Bottom ash that had accumulated in the boiler during testing was sampled from the discharge of the sluice pipe during the evening sluicing event.
- Fly ash samples were collected from the outlet of the screw conveyor that empties the fly ash silo.
- FGD solids were collected as the FGD filter cake fell off the rotary drum vacuum filters onto a conveyor belt, during the FGD solids dewatering procedure.
- FGD makeup water samples were collected from a tap at a location near the entrance to the FGD system.
- FGD liquor samples were collected from each of the two lower loop-reaction tanks.

All of the above sampling locations should have allowed for representative sampling of the Site 20 process streams. Appendix A presents the procedures for collecting, pretreating, and analyzing the samples.

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RESULTS

This section presents the trace substance concentration data for the process streams sampled at Site 20. Additional data for mercury speciation and particle size distribution appears in Section 5. Sampling, preparation, and analytical methods are summarized in Appendix A and detailed analytical data can be found in Appendices B and C.

Before the concentration data are discussed, the sampling schedule and the data treatment conventions are described.

Sampling Schedule

Site 20 was sampled in June 1993. Four types of flue gas sampling trains were used to collect samples from the ESP outlet and stack gas streams. The gas streams were traversed with the multi-metals and anions sampling trains; single-point sampling was used for the mercury speciation and particle size distribution (PSD) sampling trains. All trains were operated within acceptable limits for isokinetic conditions.

Figures 3-1 and 3-2 present the collection time periods for the flue gas and process samples, respectively. Bad weather interrupted gas and process sampling for about three hours on 6/10/93 (Run 3) and interfered with bottom ash sampling on 6/11/93 (Run 4). Test data were not adversely affected by these two weather episodes.

Data Treatment

Several conventions were developed for treating FCEM test data and developing average concentrations of the target species in the various streams. To determine the total gas concentration for each run, both the solid and vapor phase contributions were considered. However, the absence of some detectable (above the method detection limit) concentrations in either (or both) phase(s) required conventions for dealing with these data. These conventions are summarized below.

For each substance, there are three possible combinations of vapor and solid phase concentrations in the gas stream. These are:

Case 1: The concentrations in both the solid and vapor phases are above the detection limits.

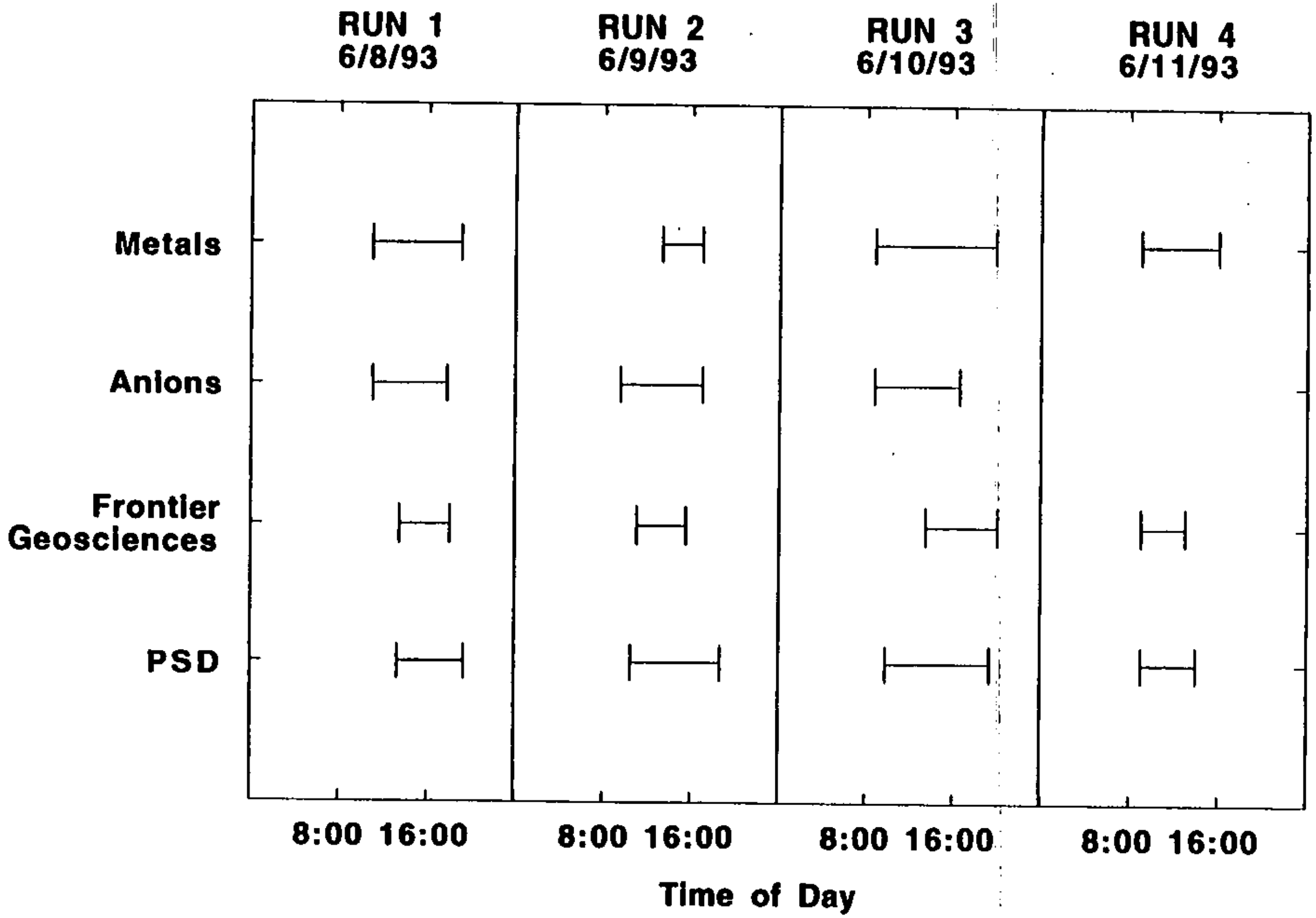

Figure 3-1
Sampling Schedule for Flue Gas Samples
All gas samples were collected simultaneously at the ESP outlet and stack locations.

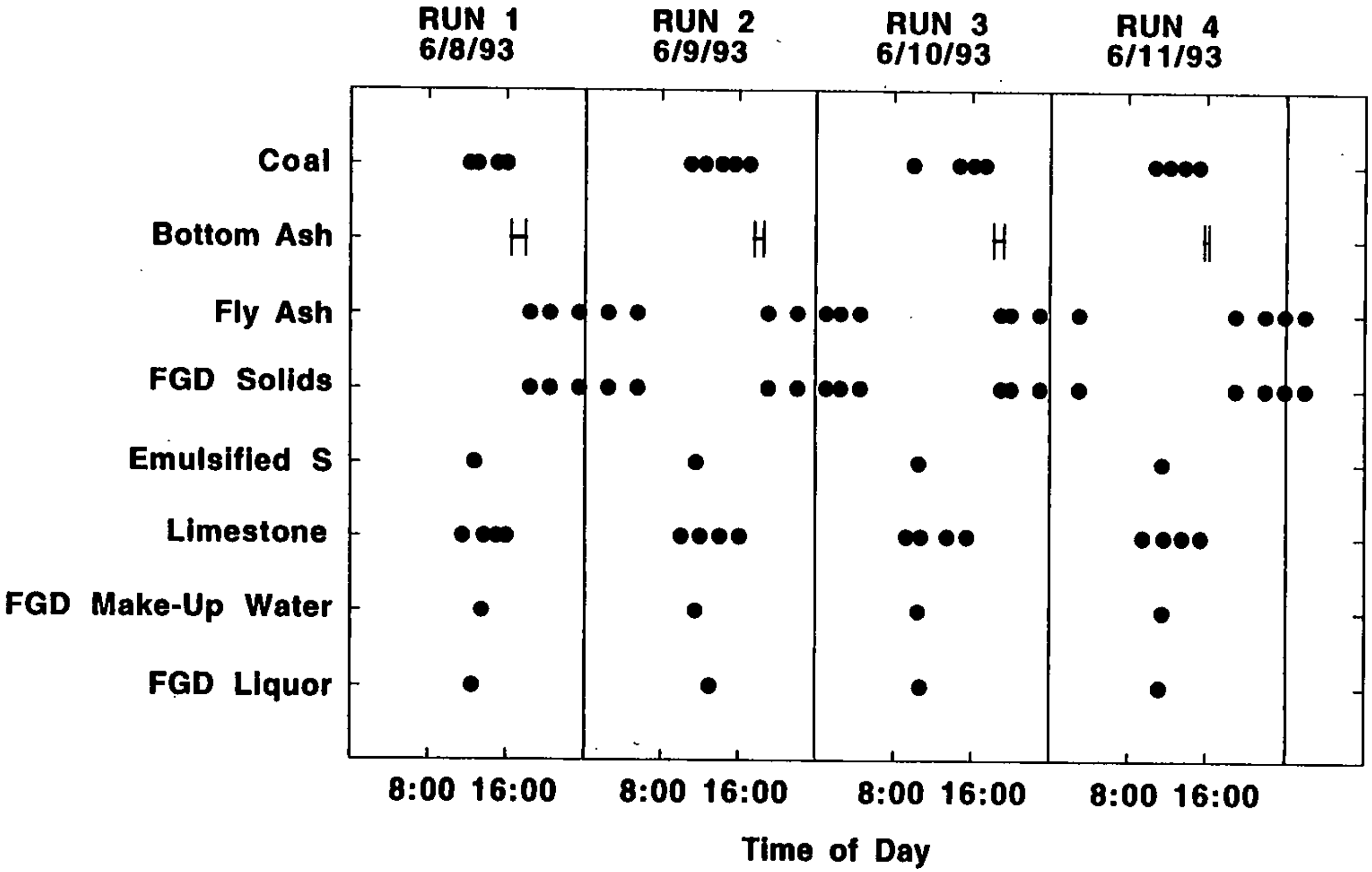

Figure 3-2
Sampling Schedule for Process Samples

Case 2: The concentrations in both the solid and vapor phases are below the detection limits.

Case 3: The concentration in one phase is above the detection limit, and the concentration in the other phase is below the detection limit.

For constituents of interest other than HCl, HF, and mercury, the flue gas stream data from previous studies of coal-fired power plants have indicated that most of the material is present in the solid phase and that only a small fraction is generally found in the vapor phase. Thus, the following conventions were selected for defining the total gas stream concentrations for analytes other than HCl, HF, and mercury:

For Case 1, the total concentration is the sum of the concentrations in the vapor and solid phases.

For example, the total selenium concentration in the ESP outlet gas for Run 3 is calculated as follows:

Se in solid phase = 77 $\mu g/Nm^3$

Se in vapor phase = 723 $\mu g/Nm^3$

Total Se in the ESP outlet gas = 800 $\mu g/Nm^3$

For Case 2, the total concentration is considered to be the detection limit in the solid phase. (This case is not represented by the data for the FCEM target species at Site 20).

For Case 3, the total concentration is considered to be the one above the detection limit, regardless of which phase this represents.

For example, the arsenic concentration in the stack gas is calculated as follows for Run 2:

As in solid phase = 0.86 $\mu g/Nm^3$

As in vapor phase = ND (0.13 $\mu g/Nm^3$)

where ND(0.13) $\mu g/Nm^3$ indicates that the analytical result was below the detection limit of 0.13 $\mu g/Nm^3$[1].

Total As in the stack gas = 0.86 $\mu g/Nm^3$

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[1] Detection limit is defined as: Method detection limit (per 40 CFR Part 136, Appendix B) multiplied by sample specific dilution and digestion factors.

The above conventions agree with guidance provided by EPA (*Technical Implementation Document for EPA's Boiler and Industrial Furnace Regulations*, U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C., March 1992).

Testing at several previous sites has indicated that HCl, HF, and mercury are present primarily in the vapor phase. For Case 2, then, the total concentration is considered to be the detection limit in the vapor phase. For Cases 1 and 3, the methodologies are unchanged from those described above.

The following criteria were used when averaging the results of different runs:

- When all values for a given variable were above the detection limit, the mean concentration was calculated as the true arithmetic mean.
- For results that include values both above and below the detection limit, one half the detection limit was used to calculate the mean. For example:

Analytical Values	Calculation	Mean Value
10, 12, ND(8)	[10+12+(8/2)]/3	8.7

  By convention, the calculated mean is not allowed to be smaller than the largest detection limit value. In the following example, using one-half the detection limit would yield a calculated mean of 2.8. This is less than the highest detection level obtained, so the reported mean is ND(4).

Analytical Values	Calculation	Mean Value
5, ND(4), ND(3)	[5+(4/2)+(3/2)]/3 = 2.8	ND(4)

- When all the analytical results for a given variable are below the detection limit, the mean is reported as ND(x), where x is the largest detection limit. The bias estimate (used to calculate confidence intervals about the mean) is one-half of the detection limit, and no confidence interval is reported.

None of the data in this report have been corrected for the blank results. Blank values were very low compared with the concentrations found in actual samples; therefore, correction for the blank results was not warranted. Detailed information on blank samples can be found in Appendix F.

Process Solids Results

Coal

Table 3-1 shows the analytical results for the coal samples. For each substance, a mean concentration has been calculated, along with the 95% confidence interval about the mean. The mean, plus and minus the confidence interval, represents the range where the probability is 95% that the true mean lies. For example, there is 95% confidence

Table 3-1
Coal Composition (mg/kg dry basis, unless noted)

Measurement	Run 1	Run 2	Run 3	Run 4	Mean	95% CI[a]
Gross Load (MWe)	652	655	657	656	655	3
Coal Rate (lb/hr, dry)	630,000	614,000	619,000	618,000	620,250	10,900
HHV (Btu/lb, dry)[b]	9,996	10,142	10,067	9,861	10,017	190
Ash (%, dry)	21.1	20.0	20.1	22.6	21.0	1.9
Moisture (%)	33.5	34.2	33.6	34.4	33.9	0.7
Sulfur (%, dry)	2.15	2.03	2.32	2.47	2.24	0.31
FCEM Substances						
Arsenic	4	3	3	1	2.8	2.0
Barium	210	180	160	210	190	39
Beryllium	5.1	6.4	10.0	4.6	6.5	3.9
Cadmium	0.3	0.2	ND(0.1)[c]	ND(0.1)	0.14	0.15
Chloride	90	165	180	77	128	83
Chromium	16	14	17	17	16	2.3
Cobalt	6	6	9	4	6.3	3.3
Fluoride	50	57	79	56	61	20
Lead	21	11	9	7	12	9.9
Manganese	54	68	98	69	72	29
Mercury (DGA/CVAAS)[d]	0.28	0.22	0.27	0.26	0.26	0.04
Mercury (CVAFS)[e]	0.27	0.24	0.28	0.29	0.27	0.03
Molybdenum	ND(4)	ND(4)	ND(3)	ND(4)	ND(4)	
Nickel	15	11	18	13	14.3	4.8
Phosphorus	95	60	35	50	60	41
Selenium	6	5	4	3	4.5	2.1
Vanadium	28	32	37	36	33	6.5

[a] CI = Confidence interval.
[b] HHV = Higher heating value.
[c] ND = Concentration was less than the method detection limit. Detection limit shown in parentheses.
[d] DGA/CVAAS = Double gold amalgamation followed by cold vapor atomic absorption spectroscopy.
[e] CVAFS = Cold vapor atomic fluorescence spectroscopy.

that the mean coal beryllium concentration was between 2.6 mg/kg and 10.4 mg/kg. The calculation of this confidence interval is presented in Appendix E.

As will be discussed in Section 4, the quality of the coal analytical data is good, except for arsenic and cobalt. The analytical result for arsenic in a standard reference material was less than 45% of the certified concentration. The analytical result for cobalt in a standard reference material was about 95% higher than the certified concentration. This suggests that the coal analytical results may be biased low for arsenic and high for cobalt.

Gas Stream Results

Electrostatic Precipitator Outlet Gas

Table 3-2 presents concentration data for the flue gas exiting the ESP at Site 20. The data are presented as solid and vapor compositions, along with the mean concentrations and confidence intervals of the combined phases. The particulate concentration data presented in Table 3-2 are averages of the values obtained from the metals and anions sampling trains at the ESP outlet.

Concentrations in both vapor phase and solid phase blank samples were insignificant when compared with the measured concentrations; therefore, no blank corrections were applied.

Arsenic, beryllium, cadmium, chromium, lead, and nickel concentrations were determined using GFAAS (a glossary of terms is presented in Section 7). The reported value for selenium was determined using ICP-MS instead of GFAAS. Additional discussion on this topic is presented in Section 4. Chloride was determined using ion chromatography; fluoride was determined using an ion-specific electrode. Mercury concentrations were determined using CVAAS.

As at the FCEM sites tested previously, most of the target elements were found primarily in the solid phase. The exceptions were chloride, fluoride, mercury, and selenium. Because of their high volatility at ESP outlet temperatures (≈ 300°F), these substances are primarily found in the vapor phase.

Stack Gas

Table 3-3 presents the metal and anion concentrations in the stack gas. The data are presented as solid and vapor compositions, along with the mean concentrations and confidence intervals of the combined phases. The analytical methods chosen for each analyte are identical to those mentioned for the ESP outlet gas.

The particulate concentration data presented in Table 3-3 do not include the measurements obtained with the anions trains. The anions results were excluded because the probe and nozzle rinses (PNR) for Runs 1 and 3 contained a large amount of solids that appeared to be stack wall deposits. The solids are not believed to be representative of

Table 3-2
Site 20 ESP Outlet Gas Composition ($\mu g/Nm^3$)

	Solid Phase				Vapor Phase					
Measurement	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4	Mean	95% CI
Gross Load (MWe)	652	655	657	656					655	3
Gas Flow (dscfm) [a,b]	1,960,000	1,980,000	1,960,000	1,920,000					1,960,000	40,000
Gas Flow (Nm^3/hr) [c]	3,100,000	3,140,000	3,100,000	3,040,000					3,100,000	66,000
Particulate (lb/hr)	227	449	350	231					315	170
Particulate (mg/Nm^3)	33	65 [d]	51	35					46	24
FCEM Substances										
Antimony	ND(0.99)		ND(1.18)	ND(1.43)	ND(4.31)		ND(4.90)	ND(5.20)	ND(5.20)	
Arsenic	2.9		4.3	2.2	ND(0.12)		ND(0.13)	ND(0.14)	3.1	2.7
Barium	129		202	135	2.2		0.6	0.6	156	101
Beryllium	1.4		3.3	1.3	ND(0.10)		ND(0.11)	ND(0.12)	2.0	2.8
Cadmium	0.2		0.3	0.4	0.21		0.12	0.20	0.47	0.22
Chloride	32.4	*65.1*	23.2		2,500	*8,800*	2,400		4,600	9,000
Chromium	4.6		6.8	4.5	ND(0.44)		ND(0.51)	ND(0.54)	5.3	3.2
Cobalt	2.0		3.7	1.8	ND(0.61)		ND(0.69)	ND(0.73)	2.5	2.5
Fluoride	9	14	19		4,400	4,200	4,100		4,200	440
Lead	5.3		3.0	2.6	3.3		1.7	2.1	6.0	4.2
Manganese	16.6		33.8	16.5	4.3		1.1	0.7	24.3	25.2
Mercury	0.01		0.01	0.01	13.1		10.9	16.1	13.4	6.5
Nickel	3.6		7.5	4.0	ND(1.76)		ND(2.00)	ND(2.13)	5.0	5.3
Phosphorus	23.7		21.4	15.3	ND(10.9)		15.9	ND(13.2)	29.4	17.8
Selenium [e]	42		77	76	573		723	637	709	193
Vanadium	10.5		13.5	9.6	ND(0.42)		ND(0.48)	ND(0.51)	11.2	5.1

[a] 1 atm, 68°F (dry).

[b] Gas flow rate measured at stack. FGD inlet gas flow considered biased due to sample location that did not meet EPA Method 2 requirements.

[c] 1 atm, 0°C (dry).

[d] Mass loading for Run 2 could be high because of ESP problems (see Section 4, Process Operations).

[e] ICP-MS data.

ND = Concentration was less than the method detection limit. Detection limit in parentheses.

Table 3-3
Site 20 Stack Gas Composition ($\mu g/Nm^3$)

	Solid Phase				Vapor Phase					
Measurement	Run 1	Run 2	Run 3	Run 4					Mean	95% CI
Gross Load (MWe)	652	655	657	656					655	3
Gas Flow (dscfm)	1,960,000	1,980,000	1,960,000	1,920,000					1,960,000	40,000
Gas Flow (Nm^3/hr)	3,100,000	3,140,000	3,100,000	3,040,000					3,100,000	66,000
Particulate (lb/hr)	59	175	95	123					113	78
Particulate (mg/Nm^3)	9	25[a]	14	18					17	11
FCEM Substances										
Antimony	ND(1.31)	ND(1.07)	ND(1.13)	ND(1.29)	ND(4.65)	ND(4.73)	ND(5.09)	ND(5.09)	ND(5.09)	
Arsenic	0.46	0.86	0.50	0.49	ND(0.13)	ND(0.13)	ND(0.14)	ND(0.14)	0.6	0.3
Barium	27.3	65.6	27.8	29.1	3.9	0.4	0.3	0.3	39	30
Beryllium	0.26	0.46	0.29	0.28	ND(0.11)	ND(0.11)	ND(0.12)	ND(0.12)	0.3	0.1
Cadmium	0.90	0.07	0.64	0.26	0.31	0.08	0.20	0.08	0.64	0.62
Chloride	127	133	500		52.1	68.6	174		351	555
Chromium	2.8	3.7	1.3	2.2	ND(0.48)	ND(0.49)	ND(0.53)	ND(0.52)	2.5	1.6
Cobalt	0.50	0.89	0.46	0.66	ND(0.66)	ND(0.67)	ND(0.72)	ND(0.72)	0.6	0.3
Fluoride	153	149	637		91.9	69.4	71.5		390	700
Lead	0.65	1.15	0.38	0.60	3.6	0.5	2.0	1.8	2.7	2.1
Manganese	3.9	6.5	3.9	4.9	0.2	6.1	2.6	92.5[c]	7.7	5.1
Mercury	0.04	0.07	0.01	0.05	12.7	10.4	9.7	9.7	10.7	2.2
Nickel	3.5	4.7	2.7	4.7	ND(1.90)	ND(1.93)	ND(2.08)	ND(2.08)	3.9	1.6
Phosphorus	6.1	7.8	3.2	5.6	12.2	9.1	15.4	18.9	15.7	6.9
Selenium[b]	15.0	24.0	16.5	16.1	91	123	178	122	147	58
Vanadium	2.5	3.7	2.3	2.8	ND(0.46)	ND(0.46)	ND(0.50)	ND(0.50)	2.8	1.0

[a] Mass loading for Run 2 could be high because of ESP problems (see Section 4, Process Operations).
[b] ICP-MS data.
[c] Value is suspect; not used in the calculation of the mean or the 95% CI.
ND = Concentration was less than the method detection limit. Detection limit in parentheses.
CI = Confidence interval.

the particulate matter present in the gas stream. The filter weight gains for all of the anions runs were similar to those for the multi-metals runs, indicating that both trains collected a similar number of small particles and that the difference in PNR weights was due to material scraped off the sampling port walls when the anions probes were removed from the stack.

Emission Factors

Table 3-4 presents mean emission factors for both the ESP outlet and stack gas streams. Emission factors are presented for each of the substances on a mass-per-unit-energy basis.

For both gas streams, chloride, fluoride, and selenium had the highest emission factors of all the target species. All three of these species showed much lower concentrations at the stack compared to the ESP outlet, indicating that these species were effectively removed by the FGD system, as discussed below.

FGD System Control Efficiency

Table 3-5 presents the removal efficiencies for the FGD system, listed by species. The average particulate removal was calculated to be 63 percent. The calculated particulate removal is lower than the fly ash removal levels due to re-entrainment of scrubber solids and acid mist formation. Based on the removal of other species that were primarily present in the particulate phase at the ESP outlet (e.g., arsenic, beryllium, aluminum, barium, iron, and vanadium), the average fly ash removal was about 75 percent.

The removal efficiencies for chloride and fluoride were based on vapor phase concentrations only because both of these species should be present in the vapor phase at both locations. The calculated removal efficiency would have been lower if the solid phase results had been included, due to the relatively high concentrations of chloride and fluoride in the stack gas particulate. Most of this particulate is probably scrubber generated material, which contains significant levels of chloride and fluoride.

When calculating the FGD system removal efficiency, the actual measured ESP outlet gas flow was not used. Rather, it was assumed that the gas flow measured at the stack was the same as the FGD inlet gas flow (i.e., it was assumed that no flue gas bypassed the FGD system). This assumption was based on SO_2 and energy balances around the FGD system. (Section 4 provides additional discussion.) Also, the ESP outlet sampling location did not meet the requirements of EPA Method 2, due to the close proximity of this sampling location to the induced draft fans and FGD system bypass ductwork.

ESP System Control Efficiency

Estimates of the ESP system control efficiency are also shown in Table 3-5. The ESP inlet ash loading was estimated using coal flow rates and analyses and using the

Table 3-4
Emission Factors (lb/10^{12} Btu)

	ESP Outlet		Stack	
Substance	Combined Mean	95% CI About Mean	Combined Mean	95% CI About Mean
Gas Flow (dscfm)	1,960,000	40,000	1,960,000	40,000
Gas Flow (Nm^3/hr)	3,100,000	66,000	3,100,000	66,000
Coal Flow (lb/hr, dry)	620,000	11,000		
Heating Value (Btu/lb, dry)	10,000	190		
Particulate (lb/10^6 Btu)	0.051	0.026	0.019	0.012
FCEM Substances				
Antimony	ND	--	ND	--
Arsenic	3.4	2.9	0.63	0.34
Barium	170	110	42	33
Beryllium	2.2	3.1	0.35	0.18
Cadmium	0.52	0.18	0.70	0.68
Chloride	5,000	9,900	390	610
Chromium	5.8	3.6	2.8	1.8
Cobalt	2.7	2.8	0.69	0.54
Fluoride	4,600	620	430	770
Lead	7.7	3.5	3.8	2.9
Manganese	27	28	8.5	8.6
Mercury	15	7.2	12	2.6
Nickel	5.5	6.0	4.3	2.1
Phosphorus	22	10	21	7.2
Selenium	780	220	160	65
Vanadium	12	5.7	3.08	1.15

CI = Confidence interval.

Table 3-5
Removal Efficiencies for ESP[a] and FGD Systems at Site 20

	FGD Systems		ESP Performance	
	% Reduction	95% CI	% Reduction	95% CI
Particulate	63	25	99.7	0.1
Target Species				
Antimony	ND	ND	--	--
Arsenic	82	13	99.5	0.4
Barium	75	20	99.6	0.2
Beryllium	84	24	99.6	0.6
Cadmium	-36	140	94.6	7.0
Chloride[b]	98	4	-0.54	200
Chromium	53	32	99.6	0.3
Cobalt	75	27	99.6	0.5
Fluoride[b]	98	1	2.2	9.2
Lead	55	36	98.7	0.8
Manganese	68	28	99.6	0.4
Mercury	20	33	1.3	--
Molybdenum	3	44	98.7	0.9
Nickel	22	72	99.7	0.4
Phosphorous	47	32	99.3	0.5
Selenium	79	8	18.2	19.5
Vanadium	75	11	99.6	0.2
Other Elements				
Aluminum	83	9	99.7	0.2
Calcium	62	22	99.7	0.1
Copper	63	39	99.5	0.3
Iron	84	14	99.8	0.3
Magnesium	0	47	99.7	0.1
Potassium	66	46	99.8	0.1
Sodium	32	17	99.4	0.2
Sulfur	95	2	2.8	17.2

[a] ESP removal efficiencies estimated using an assumed fly ash collection rate.

[b] Removal of vapor phase anions only for FGD system.

CI = Confidence interval.

ND = Concentration was below method detection limit. Removal calculation not performed.

assumption that 80% of the coal ash is transformed into fly ash. The ESP outlet ash loading used was the average value shown in Table 3-2.

4

DATA EVALUATION

Several procedures can be used to evaluate the information developed during a field sampling program. In the case of Site 20, three methods were used to evaluate data quality. First, the process data were examined to determine if the unit operated at normal, steady-state conditions during the sampling periods. Second, the quality assurance/quality control (QA/QC) protocol for sampling and analytical procedures used at Site 20 (i.e., equipment calibration and leak checks, duplicates, blanks, spikes, standards, etc.) were evaluated. Site 20 QA/QC data were compared with FCEM project objectives. Third, material balances were calculated around the entire plant, the boiler/ESP combination, and the FGD system. Material balances involve the summation and comparison of mass flow rates in several streams, often sampled and analyzed by different methods. Closure within an acceptable range can be used as an indicator of accurate results for streams that contribute significantly to the overall inlet or outlet mass rates, such as the coal and ash streams.

Process Operation

A major objective of this project was to estimate the emission rates of FCEM species from the Site 20 power plant. Therefore, it was important that the plant operate under representative and stable conditions throughout each test day. To ensure that the desired conditions were met and maintained, the plant's control room operators set up special logs to record relevant data from the plant's data acquisition system.

The logged data show that the boiler and FGD system operations were relatively consistent and stable during sample collection periods. Table 4-1 and Figures 4-1 and 4-2 demonstrate this conclusion. An upset in the ESP operation occurred on the second test day. The upset and its impacts on test results are described below. Note that the large variability in the ESP outlet opacity data is commonly observed for ESPs and results from ESP rapper cycling.

The logged data also showed that the boiler and ESP operations were representative of "typical" plant operation except for the problems caused by the ESP opacity monitor malfunction. However, the FGD system treated more gas than it usually does under "typical" plant operation. The impacts of the higher gas flow to the FGD system are discussed below.

Table 4-1
Site 20 Process Stability Summary[a]

Date	Sampling Period	Period Averaged		Unit Load (MW)	Econ. Out Avg. O_2 (%)	Stack CO_2 (%)	SO_2 Removal (%)	ESP "B" Opacity (%)	ESP "A"[b] Opacity (%)
6/8/93	1100-1907	1035-1935	Average	652	2.9	10.0	99.5	10.9	9.8
			Std. Deviation	2	0.1	0.2	0.2	3.8	2.5
			Maximum	656	3.1	10.4	100.3	31.7	23.7
			Minimum	648	2.7	9.7	99.3	0.1	0.2
6/9/93	0915-1803	0900-1835	Average	654	3.0	9.9	99.5	9.9	b
			Std. Deviation	1	0.1	0.1	0.7	2.9	b
			Maximum	658	3.2	10.1	100.4	27.7	b
			Minimum	650	2.9	9.7	98.1	0.1	b
6/10/93	0842-1944	0820-2004	Average	657	3.0	10.1	99.0	9.5	b
			Std. Deviation	2	0.1	0.1	0.5	3.3	b
			Maximum	663	3.2	10.5	99.7	27.8	b
			Minimum	652	2.7	9.9	97.2	0.1	
6/11/93	0855-1555	0840-1610	Average	656	2.9	10.3	98.6	8.4	b
			Std. Deviation	1	0.1	0.2	0.4	2.3	b
			Maximum	659	3.1	10.8	99.4	15.8	b
			Minimum	652	2.6	9.9	96.9	0.0	b

[a] Data from plant's data acquisition system.

[b] The opacity monitor for the "A" side ESP malfunctioned on 6/9/93 and gave faulty signals on 6/10 and 6/11.

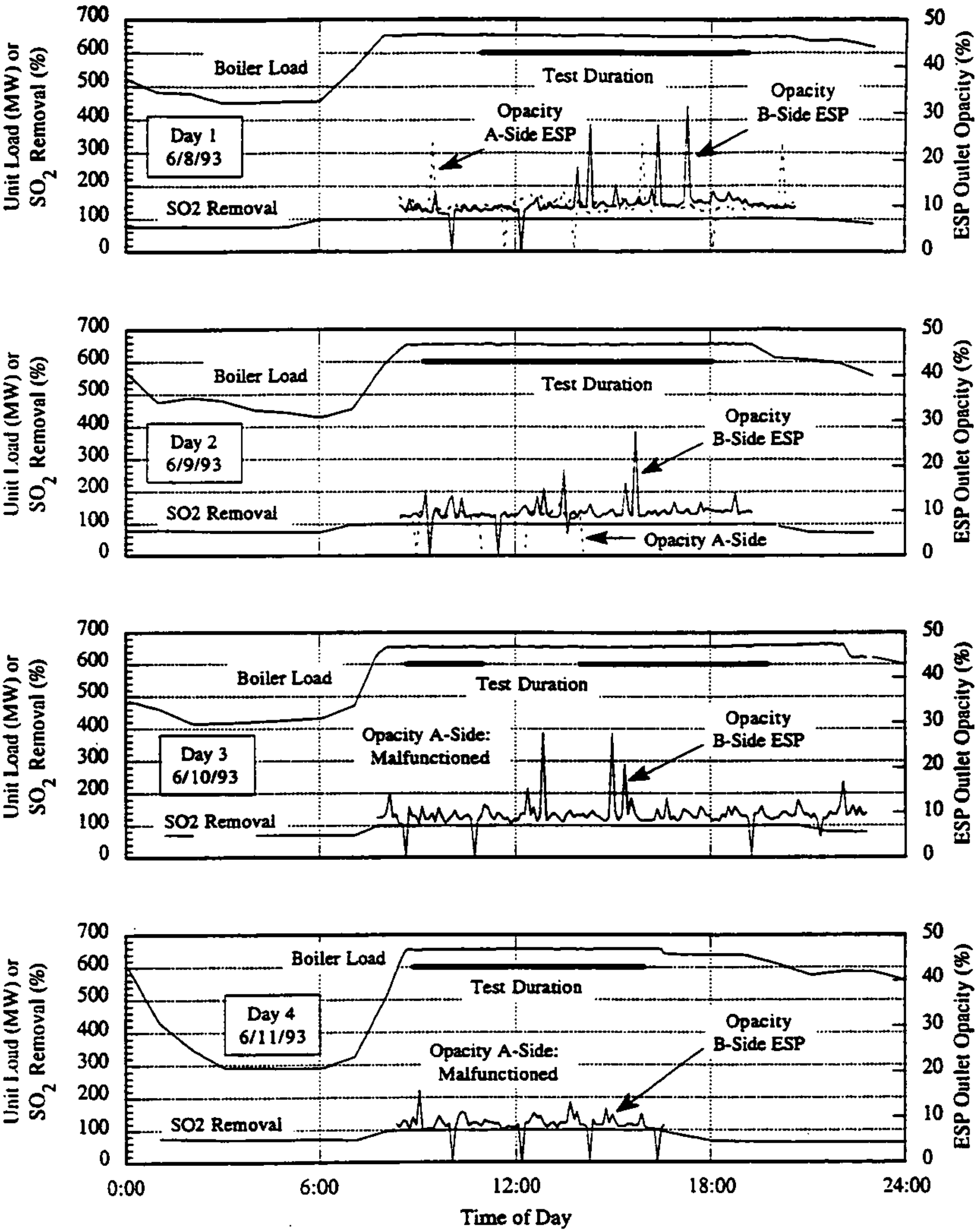

Figure 4-1
Site 20 Process Data for Days 1-4

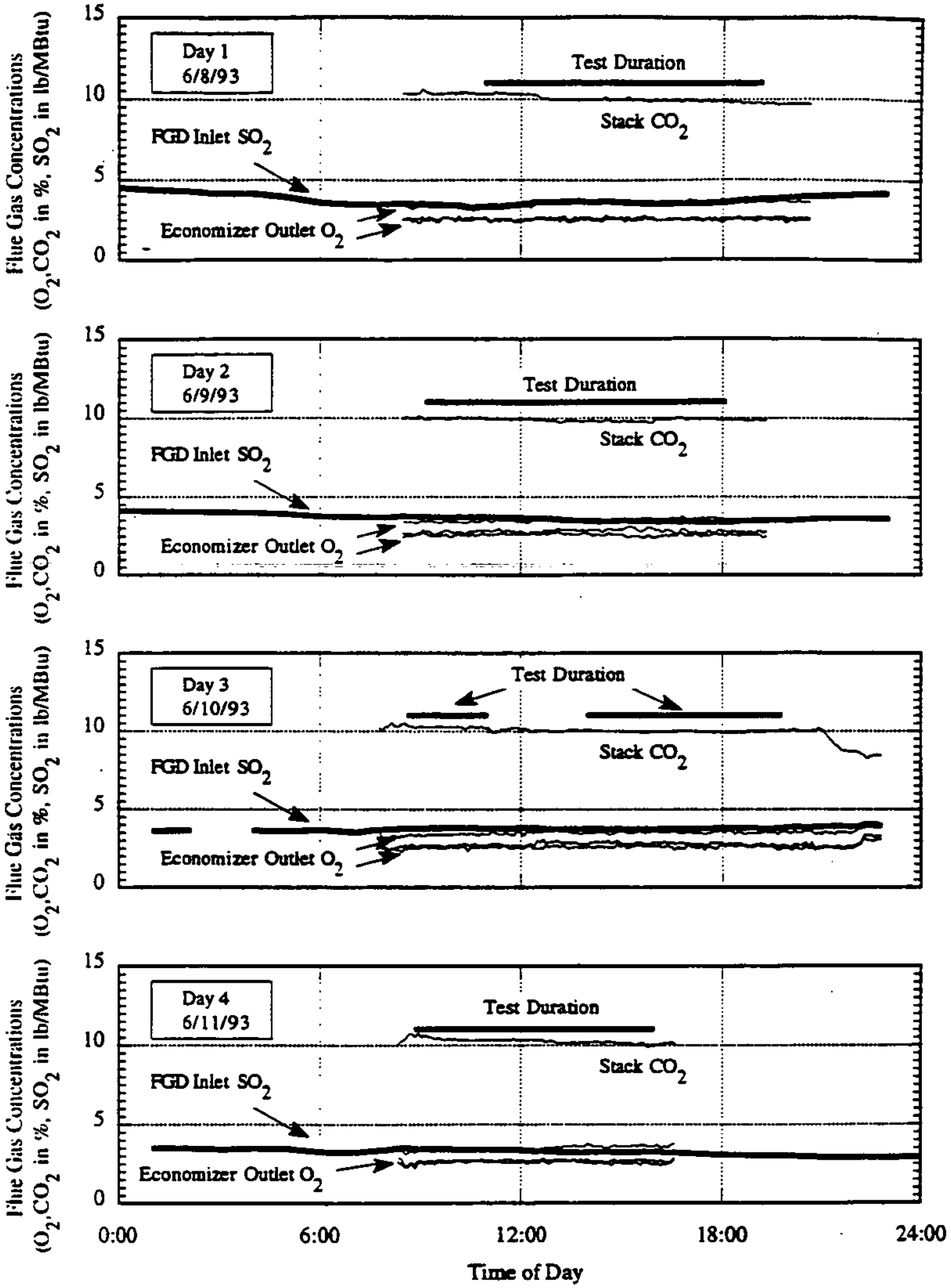

Figure 4-2
Gas Concentration Data for Days 1-4

Plant Operational Changes

Three changes were made to the "typical" plant operating procedures. These changes have the potential to affect the test data, so they are described and discussed below.

Flue Gas Bypass Around FGD System

The plant can bypass flue gas around its FGD system. Typical plant operation is to bypass as much gas as possible, while still meeting the required SO_2 emission limit (1.2 lb/MMBtu). The amount of bypass depends on the sulfur level in the coal, which is highly variable. To ensure that the plant's emissions were in compliance and that the amount of bypass was constant during testing, the plant operated its bypass dampers at an abnormally low value of only 10% open. This appeared to cause all of the flue gas to enter the FGD system, based on gas temperature and SO_2 concentration measurements at the scrubber inlet, scrubber outlet, and stack locations. The gas velocity measurements from the sampling trains suggest that approximately 15% of the flue gas bypassed the scrubbers, but the scrubber inlet velocity measurements were questionable because of the close proximity of the sampling ports to the induced draft fans and flue gas bypass ductwork.

The lower-than-typical amount of bypass might affect the estimated trace element emission rates for "typical" plant operation. For example, the measured stack gas concentrations might be lower than typical for species that are effectively removed by the FGD system. Species that are not effectively removed by the FGD system are probably not affected by the lower-than-typical amount of bypass. In any case, "typical" stack gas emissions can be estimated if the amount of flue gas bypass is known. For example, the bypass gas concentrations should be the same as those measured for the FGD inlet flue gas. Scrubber outlet flue gas concentrations can be approximated by the measured stack gas concentrations (assuming that 100 percent of the flue gas entered the scrubbers during the tests). Knowing these concentrations and the amount of flue gas bypass, the "typical" emission rates from the plant can be calculated.

Bottom Ash Sluicing

Bottom ash and economizer ash are sluiced to an ash pond. Plant personnel report that very little economizer ash is produced, so only bottom ash was sampled at Site 20. The normal plant procedure is to sluice the bottom ash every 24 hours, usually at night. This procedure was modified to collect a bottom ash sample more representative of the gas sampling periods. That is, the plant sluiced bottom ash early in the morning to remove most of the ash before gas sampling activities started. After gas sampling was completed, the plant sluiced bottom ash again, and the sluicing stream was sampled to obtain a bottom ash sample that corresponded to the daily gas sampling activities.

The change in the bottom ash sluicing procedure should make the bottom ash sample more representative.

Soot Blowing

The plant normally operates soot blowers on an "as-needed" basis (about every other day, according to plant personnel). During testing, however, one set of soot blowers, designated as "3 IKS sequence 9" was operated for one hour every day while gas sampling activities were in progress. No additional soot blowing occurred during the day, but plant personnel operated soot blowers at night on an "as-needed" basis.

The change in soot blowing schedule may represent a "worst-case" scenario for emissions from Site 20. That is, particulate emissions from the ESP should increase when the ESP inlet particulate loading increases. If all other parameters are held constant, the ESP inlet particulate loading should increase during soot blowing operations. Therefore, higher ESP outlet emissions would be expected. Since gas sampling activities were in progress during soot blowing operations, the measured emissions from Site 20 may be higher than the average values for "typical" plant operation.

Plant Operational Problems

The only plant-related problem occurred on 6/9/93 (Run 2) when the ESP energy management system reduced the electrical power supplied to the A-side precipitator. The power was reduced because the energy management system received a faulty signal from the opacity monitor installed on the A-side ESP outlet duct work. Since the energy management system uses the opacity monitor signal to control the power supplied to the ESP transformer/rectifier (T/R) sets, the artificially low opacity signal resulted in less power supplied to the A-side T/R sets. The B-side ESP was not affected by these events.

The opacity monitor failure was not discovered until 8:00 a.m. on 6/10/93. At that time, the energy management system was set to manual control for the A-side precipitator. The system remained in this mode for the last two test days (6/10/93 and 6/11/93).

The reduced power supply to the A-side T/R sets probably caused an increase in the particulate grain loading at the ESP outlet during Run 2. The field data seem to support this conclusion. That is, the highest particulate concentrations were measured during Run 2 (for both the ESP outlet and stack gas locations). Even though the particulate data suggest that the ESP problems occurred during Run 2, the test data from Run 2 were used in the calculation of all concentration and emission factors.

One other item to note concerning the ESP at Site 20 was the large number of broken discharge electrodes. According to plant personnel, Site 20 commonly experiences broken electrodes and needs to repair broken electrodes every time the plant is shut down. Plant personnel also indicated that the number of broken electrodes was high relative to normal plant operation. Although the number of broken electrodes might have been higher than during "typical" plant operation, the plant continued to operate without exceeding its opacity limits, indicating that the broken electrodes had an insignificant effect on outlet mass emissions. Also, because the number of broken electrodes stayed constant during the field testing effort, any effect they caused should have been constant over the four days of testing.

At this time, it appears that the large number of broken electrodes did not affect the data from the field testing program.

Sample Collection

Appendix A describes the sampling procedures used at Site 20. Following are some significant observations about sample collection:

- The multi-metals sample collected at the ESP outlet on 6/9/93 (Run 2) was voided because of an accident that caused the sample to be collected at nonisokinetic conditions for a substantial amount of time. The completeness objectives for the project were still met in spite of this accident.

- A red precipitate coated the hot box exit glass pieces for the anions and multi-metals trains at both sampling locations on every test day. The precipitate could not be recovered from the glassware using the rinses described by the sampling methods. The precipitate was removed by concentrated sulfuric acid during cleaning. This acid was not used for sample recovery since it was not specified in the sampling method.

- The above observations suggest that the anions and multi-metals sampling methods did not recover the elements in the red precipitate. If these elements were FCEM target species, the field test data may underestimate their concentrations in the flue gas streams.

- A mercury speciation sample was not collected at the stack on 6/8/93 (Run 1) because of sampling problems. The completeness objectives for the project were still met in spite of the sampling problems.

- Although the test plan called for one particle size distribution (PSD) sample to be collected each day, only a small amount of solids were collected on the first day; therefore, three-day composite samples were collected at the ESP outlet and stack locations (2 samples total). This limits the ability to generate average values and to estimate some of the uncertainties for the PSD data.

Analytical Quality Control Results

Generally, the type of quality control information obtained pertains to measurement precision, accuracy, and blank effects, determined using various types of replicate, spiked, and blank samples. The specific characteristics evaluated depend on the type of quality control checks performed. For example, blanks may be prepared at different stages in the sampling and analysis process to isolate the source of a blank effect. Similarly, replicate samples may be generated at different stages to isolate and measure sources of variability. Table 4-2 summarizes the QA/QC measures commonly used as part of the FCEM data evaluation protocol, and the characteristic information obtained. The absence of any of these types of quality control checks from the data does not necessarily reflect poorly on the quality of the data but does limit the ability to estimate the magnitude of the measurement error and, hence, prevents estimating the confidence that can be placed in the results.

Table 4-2
Types of Quality Control Samples

QC Activity	Characteristic Measured
Precision	
Replicate samples collected over time under the same conditions	Total variability, including process or temporal, sampling, and analytical, but not bias.
Duplicate field samples collected simultaneously	Sampling plus analytical variability at the actual sample concentrations.
Duplicate analyses of a single sample	Analytical variability at the actual sample concentrations.
Matrix- or media-spiked duplicates	Sampling plus analytical variability at an established concentration.
Laboratory control sample duplicates	Analytical variability in the absence of sample matrix effects.
Accuracy (Includes Bias and Precision)	
Matrix-spiked samples	Analyte recovery in the sample matrix, indicating possible matrix interferences and other effects. In a single sample, includes both random error (imprecision) and systematic error (bias).
Media-spiked samples	Same as matrix-spiked samples. Used where a matrix-spiked sample is not feasible, such as certain stack sampling methods.
Laboratory control samples (LCS)	Analyte recovery in the absence of actual sample matrix effects. Used as an indicator of analytical control.
Standard Reference Material	Analyte recovery in a matrix similar to the actual samples.
Blank Effects	
Field Blank	Total sampling plus analytical blank effect, including sampling equipment and reagents, sample transport and storage, and analytical reagents and equipment.
Method Blank	Blank effects inherent in analytical method, including reagents and equipment.
Reagent Blank	Blank effects from reagents used.

As shown in Table 4-2, different QC checks provide different types of information, particularly pertaining to the sources of inaccuracy, imprecision, and blank effects. As part of the FCEM project, measurement precision and accuracy are typically estimated from QC indicators that cover as much of the total sampling and analytical process as feasible. Precision and accuracy measurements are based primarily on the actual sample matrix. The precision and accuracy estimates obtained experimentally during the test programs are compared with data quality objectives (DQOs) established for the FCEM project.

These DQOs are not intended to be used as validation criteria, but they can be used as empirical estimates of the precision and accuracy that would be expected from existing reference measurement methods and that would be considered acceptable. The precision and accuracy objectives are not necessarily derived from analyses of the same types of samples being investigated. Although analytical precision and accuracy are relatively easy to quantify and control, sampling precision and accuracy are unique to each site and each sample matrix. Data that do not meet these objectives are not necessarily unacceptable. Rather, the intent is to document the precision and accuracy actually obtained, and the objectives serve as benchmarks for comparison. The effects of not meeting the objectives should be considered in light of the intended use of the data.

Table 4-3 shows the types of quality control data reported for this site. The results of these analyses can be found in Appendix F. Table 4-4 presents a summary of precision and accuracy estimates. Most of the quality control results met the project objectives.

The quality control data show that the following information should be considered when the analytical data are evaluated:

- The recovery of arsenic in the coal by GFAAS was low (43% compared to a 75-125% objective), suggesting a low bias for arsenic in the coal.

- The recovery for cobalt in the coal by ICP-AES was high (193% compared to a 75-125% objective), suggesting a high bias for cobalt in the coal.

- The recovery of lead in the stack gas and ESP outlet solid phases measured by GFAAS (70%) was slightly below the project objectives (75-125%). The recovery of lead in the ICP-MS samples was higher (104%); however, significant levels of lead were found in the blanks for this method. The high blank levels could account for the higher recovery. The GFAAS values were used in the mass balance. In the flue gas samples, lead was contained primarily in the solid phase; therefore, the results for lead in the flue gas may be biased slightly low.

- The recoveries for all the metals (except for selenium and lead), as determined by ICP-MS, were outside the specifications for metals in flue gas (solid phase). ICP-MS is an evolving analytical technique, and the results seem to confirm that the technique needs further refinement before it will produce consistently useable results. However, for selenium, the recovery was 122% (within the 75-125% specification). In addition, no selenium was detected in the method blanks.

Table 4-3
Types of Quality Control Data Reported

	Precision					Accuracy				Blank			
Analysis (Grouped by Source/Matrix)	Replicate Runs	Dup Field Samples	Dup Lab Analysis	Matrix or Media Spiked Dup	Lab Control Sample Dup	Matrix or Media Spike	Surrogate Spike	Lab Control Sample	Standard Reference Material	Field Blank	Trip Blank	Method Blank	Reagent Blank
ESP Outlet Gas													
Metals - Vapor Phase	✓			✓	✓	✓		✓		✓	✓	✓	
Metals - Solid Phase	✓				✓			✓	✓	✓		✓	
Anions - Vapor Phase	✓			✓	✓	✓		✓		✓	✓	✓	
Anions - Solid Phase	✓				✓			✓				✓	
Coal													
Metals	✓	✓							✓				
Anions	✓	✓		✓	✓	✓		✓				✓	

Table 4-4
Summary of Precision and Accuracy Estimates for Site 20

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Coal - GFAAS[a]	Precision - Replicate Samples Accuracy - Standard Reference Material				
Arsenic		20	75-125	46*	43
Cadmium		20	75-125	70*	Not Defined[b]
Selenium		20	75-125	29*	125
Metals in Coal by CVAFS[c]	Precision - Replicate Samples Accuracy - Standard Reference Material				
Mercury		20	75-125	0	85
Metals in Coal by DGAA[d]	Precision - Replicate Samples Accuracy - Standard Reference Material				
Mercury		20	75-125	10	116
Metals in Coal by ICP-AES	Precision - Replicate Samples Accuracy - Standard Reference Material				
Barium		20	75-125	13*	97
Beryllium		20	75-125	37*	104
Cobalt		20	75-125	33*	193
Chromium		20	75-125	9*	98
Lead		20	75-125	52*	85
Manganese		20	75-125	26*	101
Nickel		20	75-125	21*	96
Phosphorus		20	75-125	42*	110
Vanadium		20	75-125	12*	98
Metals in Stack Gas & ESP Outlet Solid Phase - ICP-AES[e]	Precision - Analytical Spike Replicates Accuracy - Analytical Spike				
Beryllium		20	75-125	1	88
Chromium		20	75-125	1	90
Nickel		20	75-125	1	85
Metals in Stack Gas & ESP Outlet Solid Phase - GFAAS and CVAAS[f]	Precision - Analytical Spike Replicate Accuracy - Analytical Spike				
Arsenic		20	75-125	2	92
Cadmium		20	75-125	1	89
Lead		20	75-125	3	71
Mercury		20	75-125	1	121
Selenium		20	75-125	0	100

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Stack Gas & ESP Outlet Solid Phase - ICP-MS [g]	Precision - Reference Sample Replicates Accuracy - Reference Sample Recovery				
Arsenic		20	75-125	5	53
Beryllium		20	75-125	13	56
Cadmium		20	75-125	1	147
Chromium		20	75-125	1	38
Lead		20	75-125	6	104
Mercury		20	75-125	18	3100
Nickel		20	75-125	4	40
Selenium		20	75-125	24	122
Metals in Stack Gas & ESP Outlet Vapor Phase - ICP-AES	Precision - Matrix Spike Replicate Accuracy - Matrix Spike				
Beryllium		20	75-125	0	100
Chromium		20	75-125	0	97
Nickel		20	75-125	1	96
Metals in Stack Gas & ESP Outlet Vapor Phase - GFAAS and CVAAS					
Arsenic		20	75-125	1.1	94
Cadmium		20	75-125	2.9	104
Lead		20	75-125	13	95
Mercury		20	75-125	2 [h]	95 [h]
Selenium		20	75-125	>500	8 [h]
Metals in Stack Gas & ESP Outlet Vapor Phase - ICP-MS [i]	Precision - Laboratory Control Spike Replicate (3020 Digestion) Accuracy - Laboratory Control Spike				
Arsenic		20	75-125	2	87
Beryllium		20	75-125	--	--
Cadmium		20	75-125	3	71
Chromium		20	75-125	1	61
Lead		20	75-125	1	92
Mercury		20	75-125	--	--
Nickel		20	75-125	0	73
Selenium		20	75-125	1	61

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Anions in Stack Gas & ESP Outlet	Precision - Matrix Spike Replicate Accuracy - Matrix Spike				
Chloride		20	80-120	9 [h]	88 [h]
Fluoride		20	80-120	4 [h]	9 [h]

[a] GFAAS = Graphite furnace atomic absorption spectrophotometry. These values were measured by CT&E Laboratories.

[b] Not defined; this coal has no certified value for cadmium.

[c] CVAFS = Cold vapor atomic fluorescence spectroscopy. These values were measured by Frontier Geosciences.

[d] DGAA = Double gold amalgam atomic absorption spectrophotometry. These values were measured by CT&E Laboratories.

[e] ICP-AES = Inductively coupled plasma emission spectroscopy.

[f] CVAAS = Cold vapor atomic absorptions spectrophotometry.

[g] ICP-MS = Inductively coupled plasma emission spectroscopy coupled with mass spectrometry. The concentrations measured by Harvard University.

[h] These are averages of recoveries from several MS/MSD pairs.

[i] Matrix spike duplicate not available. This precision was estimated from the laboratory control spike duplicate results for the gas metals nitric impinger fraction.

*Precision is expressed as coefficient of variance (CV) based on replicate samples. Precision objective was based on duplicate not replicate samples. However, there was not enough sample available to conduct duplicate analyses.

- Selenium in the vapor phase measured by GFAAS showed poor precision (>500% RPD compared to the 20% specification), and accuracy (8% compared to the 75% to 125% specification), indicating a severe low bias in these data. The GFAAS data were not used. The ICP-MS data were used to report selenium concentrations in the gas streams and to calculate the emission factors in the selenium mass balance closure levels.

- The recovery of all the metals (except for arsenic and lead), as determined by ICP-MS, were outside the specifications for metals in flue gas (vapor phase). In addition, the blanks showed high levels of contamination for most analytes. The recovery for selenium (61%), however, was well above the recovery by GFAAS. In addition, the precision was well within the specification. For this reason, the ICP-MS data for selenium in the vapor phase was used to calculate selenium concentrations in the gas streams.

- For measurements by ICP-AES, GFAAS, CVAAS, CVAFS, all blanks, except for one field blank, showed either no contamination, contamination less than five times the detection limit, or contamination at concentrations significantly below those found in the corresponding samples.

- For measurements by ICP-MS, the digestion blanks contained all of the analytes (except for selenium) at levels that would be expected to bias the results high.

Detailed QC Results

Precision is a measure of the reproducibility of measurements under a given set of conditions. It is expressed in terms of the distribution, or scatter, of the data, calculated as the standard deviation or coefficient of variation (CV, standard deviation divided by the mean). For duplicates, precision is expressed as the relative percent difference (RPD).

Accuracy is a measure of the degree of conformity of a value generated by a specific procedure to the assumed or accepted true value; it includes both precision and bias. Bias is the persistent positive or negative deviation of the method average value from the assumed or accepted true value.

The efficiency of the analytical procedure for a given sample matrix is quantified by the analysis of spiked samples containing target or indicator analytes or other quality assurance measures, as necessary. However, all spikes, unless made to the flowing stream ahead of the sampling, produce only estimates of recovery of the analyte through all of the measurement steps occurring after the addition of the spike. A good spike recovery tells little about the true value of the sample before spiking.

Representativeness expresses how well the sampling data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. The representativeness criterion is based on making certain that sampling locations are properly selected and that a sufficient number of samples are collected.

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sampling data should be comparable with other measurement data for similar samples collected under similar conditions. This goal is achieved using standard techniques to collect and analyze representative samples and by reporting analytical results in appropriate units. Data sets can be compared with confidence when the precision and accuracy are known.

Completeness is an expression of the number of valid measurements obtained compared with the number planned for a given study. The goal is to generate a sufficient quantity of valid data.

A discussion of the overall measurement precision, accuracy, and blank effects appears below for each measurement type.

Metals

Precision. The precision of metals analyses was estimated for coal samples using replicate samples, which include a component of sampling variability.

For the flue gas metals in the vapor phase analyzed by ICP-AES, GFAAS, and CVAAS, precision was estimated using matrix spike replicate analyses; seven out of eight met the precision objectives. The exception was selenium (>500% RPD), for which the variability was greater than the objective. For flue gas metals in the vapor phase analyzed by ICP-MS precision was estimated by analyzing replicate laboratory control samples. The results for all metals were found to meet the precision objectives.

For the flue gas metals in the solid phase analyzed by ICP-AES, GFAAS, and CVAAS, precision was estimated using analytical spike replicate results, and all the metals were found to meet precision objectives. For flue gas metals in the solid phase analyzed by ICP-MS, precision was estimated using the analysis of replicate standard samples. Seven of the eight metals met precision objectives. The exception was selenium (24% RPD), for which the variability was slightly greater than the objective.

Accuracy. The accuracy of metals analyses was estimated for coal samples using standard reference coal samples. Of the metals analyzed by ICP-AES, GFAAS, CVAFS, and DGAA, 10 of the 13 met the accuracy objective. Recoveries outside the objectives were identified for chromium (193% recovery) and arsenic (43% recovery). The standard reference coal sample was not certified for cadmium, so a recovery could not be calculated.

Matrix spikes were used to estimate the accuracy of metals analyses of flue gas vapor-phase samples. Seven of the eight metals analyzed by ICP-AES, GFAAS, and CVAAS met the accuracy objective. The recovery of selenium (8%) was well below the specification. Of the metals analyzed by ICP-MS, two of the eight met the accuracy objective. Low recoveries for cadmium (71%), chromium (61%), nickel (73%), and selenium (61%) were slightly outside the accuracy objective. However, the accuracy of the selenium measurement for

ICP-MS was significantly above that for GFAAS; therefore, the ICP-MS selenium value was chosen as the primary value.

The accuracy of metals analyses was estimated for flue gas particulate phase samples using analytical spike recoveries. The results show that the recoveries of seven of the eight metals analyzed by ICP-AES, GFAAS, and CVAAS met the 75-125% accuracy objective. For lead, the recovery of 71% was slightly below the objective.

The accuracy of metals analyses was estimated for flue gas particulate-phase samples measured by ICP-MS using standard reference material (NIST 1633a fly ash). The matrix of the standard is not identical to that of the samples, especially since flue gas particulate samples are digested along with the filters; however, no better estimates of accuracy are available for these samples. Except for selenium (122%) and lead (102%), the results for no metal showed a recovery within the accuracy objective [arsenic (53%), beryllium (56%), cadmium (147%), chromium (38%), mercury (3100%), and nickel (40%)]. For that reason, ICP-AES, GFAAS, and CVAAS were chosen as the primary values for the flue gas samples (vapor phase + solid phase), with the exceptions noted above.

Blank Effects. The only significant blank effect found for ICP-AES, GFAAS, or CVAAS analyses was a field blank that contained significant concentrations of silicon. This was probably the result of the digestion of an inappropriate filter.

Because of the increased sensitivity of ICP-MS, blank effects are more significant, although compared with measurements at much lower levels than by ICP-AES, GFAAS, or CVAAS. Nearly all of the ICP-MS digestion blanks contained significant quantities of seven of the eight analytes. Both of the digestion blanks were devoid of selenium.

Anions

Precision. The precision of anion analyses was estimated for coal and ash samples using matrix-spiked duplicates. The precision estimates for both chloride and fluoride met the objective of 20% RPD. Replicate runs were used to estimate the precision of anions analysis of flue gas samples. The CVs for both chloride and fluoride met the precision objective.

Accuracy. Matrix spikes were used to estimate the accuracy of anion analyses of coal, ash, and flue gas samples. The 75-125% recovery objective was met for chloride and fluoride in all of the samples.

Blank Effects. Field blank and trip blank impinger solutions were analyzed for chloride and fluoride, and the concentrations were below detection limits in all of the blanks. No blank contamination problems were identified.

Material Balances

Evaluating data consistency can be another overall data quality evaluation tool, especially the evaluation of coal, ash, and flue gas flow rates. Material balances for ash and major elements can be used to verify the internal consistency of stream flow rates. Material balance closures for trace species can be used to indicate whether the samples collected were representative with respect to the trace element concentrations and can help identify analytical biases in one or more types of samples.

The results of material balances performed around the boiler/ESP, the FGD system, and the entire plant are shown in Table 4-5. Closure is defined as the ratio of outlet to inlet mass rates for a particular substance. A 100% closure indicates perfect agreement. When trace substances are analyzed, a closure of between 70% and 130% has been set as a goal for the FCEM project. This range reflects the typical level of uncertainty in the measurements and, therefore, allows one to interpret the inlet and outlet mass flow rates as being equivalent. The 95% confidence intervals about the closures have been calculated using an error propagation analysis, discussed in detail in Appendix E.

The material balances will be discussed for each of the three types of balances. Figure 4-3 illustrates the systems used to calculate material balance closures at Site 20. Table 4-6 lists the steam flow rates used in the material balance calculations.

Boiler/Process ESP System

Good material balance closures were obtained around the boiler/ESP for most of the target species for Site 20. Material balance closures between 70% and 130% were obtained for beryllium, cadmium, chromium, cobalt, fluoride, manganese, phosphorus, and vanadium. When the 95% confidence intervals (CI) were considered, the closures met the target criteria for all the FCEM species except for barium (290 ± 81). The large values for the calculated 95% CIs are primarily due to run-to-run concentration variability in the ESP outlet gas stream and in the coal. The 95% CIs do not strongly depend on the standard deviations or biases for the process stream flow rates.

FGD System

Material balance closures between 70% and 130% were obtained around the FGD system for cadmium, cobalt, manganese, molybdenum, nickel, and phosphorus. When the 95% confidence intervals were considered, the closures for barium, beryllium, chromium, mercury, selenium, and vanadium met, or almost met, the FCEM target criteria. The poor closures for arsenic and lead are not explainable, given the QC data reported in Table 4-4.

The closures for chloride (36% ± 10%) and fluoride (13% ± 16%) may have been low because of the accumulation of these species in the FGD scrubber slurry. That is, these species accumulate in the FGD scrubber slurry, making it difficult to obtain acceptable material balance closures for these species when the fuel concentration varies.

Table 4-5
Site 20 Material Balances

	Boiler/ESP		FGD System		Entire Plant	
FCEM Substances	**Out/In, %**	**95% CI, %**	**Out/In, %**	**95% CI, %**	**Out/In, %**	**95% CI, %**
Arsenic	276	210	32	14	229	144
Barium	290	81	69	28	281	76
Beryllium	95	56	61	29	95	56
Cadmium	84	100	77	25	81	78
Chloride	40	85	36	10	17	10
Chromium	109	25	183	50	110	25
Cobalt	127	69	103	38	127	68
Fluoride	79	27	13	16	12	14
Lead	46	37	29	16	46	37
Manganese	110	46	101	31	108	44
Mercury	58	31	148	22	86	16
Molybdenum	--	--	105	42	--	--
Nickel	143	54	111	58	143	53
Phosphorus	95	68	77	28	93	60
Selenium	228	112	61	24	154	65
Vanadium	104	27	142	39	105	27
Other Species						
Aluminum	98	25	117	28	98	25
Calcium	102	41	100	29	100	24
Copper	--	--	124	33	--	--
Iron	107	34	121	31	107	37
Magnesium	103	35	88	26	101	32
Potassium	--	--	116	33	--	--
Sulfur	84	16	124	25	104	24
Sodium	89	63	69	18	90	61

CI = Confidence interval.

NC = Not calculated. The beryllium content of the coal was less than the method detection limit.

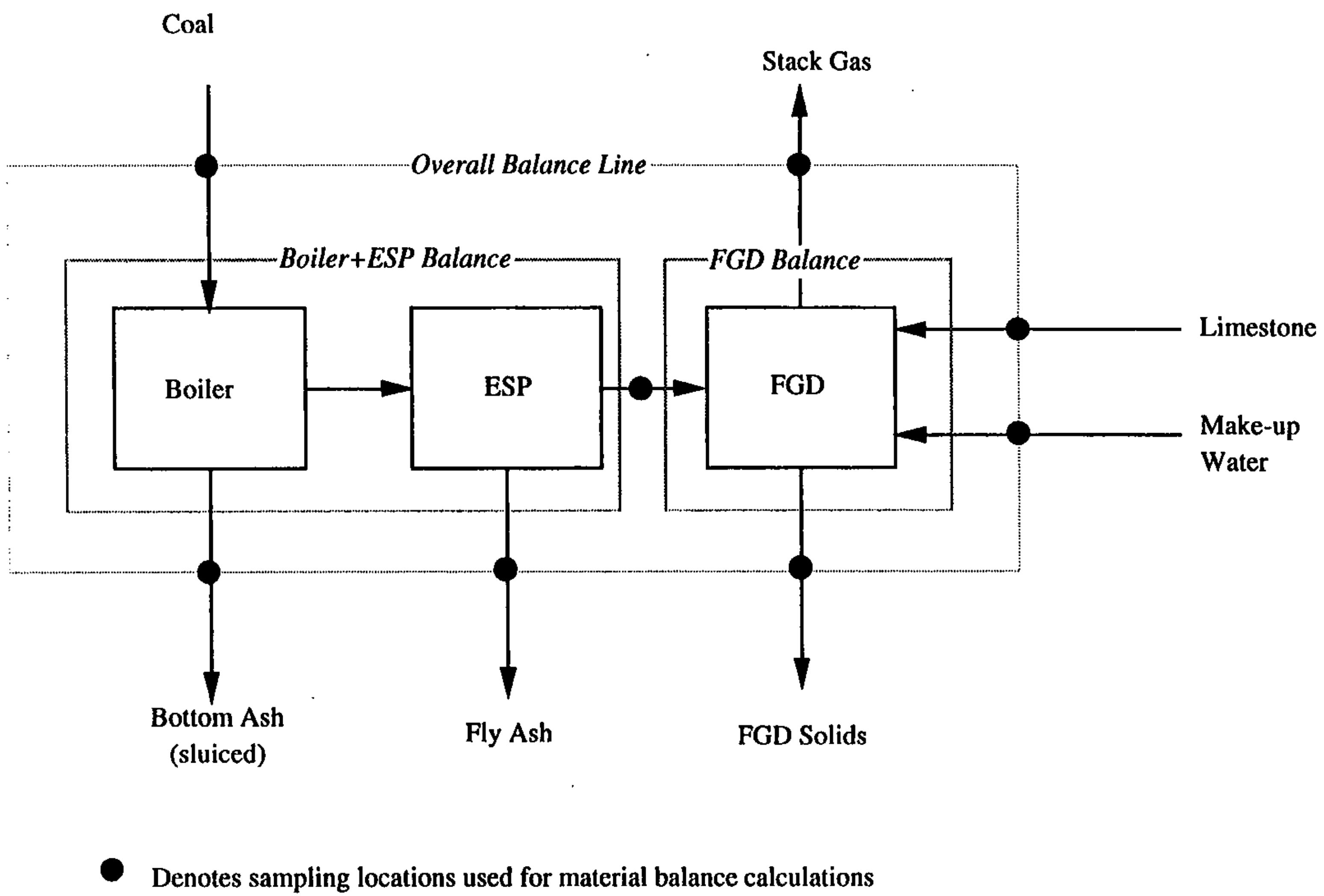

Figure 4-3
Systems for Material Balance Closure Calculations for Site 20

Table 4-6
Process Stream Flows at Site 20

Stream	Mean Flow Rate	Std. Dev.	Source
Coal (lb/hr, dry)	620,250	6850	Measured[a]
Economizer Ash[b]	–	–	--
Bottom Ash (lb/hr, dry)	26,000	300[c]	Calculated[d]
Fly Ash (lb/hr, dry)	104,000	1150[c]	Calculated[d]
FGD Makeup Water (lb/hr)	342,000	3800[c]	Calculated[e]
Limestone Flow (lb/hr)	60,300	670[c]	Calculated[f]
FGD Solids Flow (lb/hr, dry)	76,400	840[c]	Calculated[g]
Flue Gas Flow (dry Nm^3/h)	3,095,000	41,200	Measured[h]

[a] "As-received" coal flow rate determined from plant's data acquisition system. Dry coal flow rate calculated using measured coal moisture values.

[b] Not measured. Flow rate assumed to be insignificant compared to ESP fly ash.

[c] Standard deviation assumed to be proportional to standard deviation for coal flow rate.

[d] Calculated from the dry coal flow rate, the measured coal ash content, and the assumption that 80% of the coal ash is transformed into fly ash and 20% is transformed into bottom ash.

[e] Calculated from measured stack gas flow rate and moisture content and assuming a flue gas moisture content of 13% of the FGD inlet. Also assumes that the wet FGD solids contain 35 wt % water.

[f] Calculations assume no flue gas bypass, 95% SO_2 removal, and that limestone is 90% $CaCO_3$.

[g] Same assumptions as f, and that FGD solids oxidation is 15 percent.

[h] Measured at the stack. Assumes that the ESP outlet gas flow equals the stack gas flow.

Entire Plant

Table 4-5 shows that good closures around the entire Site 20 plant were obtained for most of the target elements. Poor closures were obtained for chloride and fluoride, presumably because of the accumulation of these species in Site 20's FGD system.

5

ADDITIONAL DATA

This section presents miscellaneous data from Site 20. These data are presented separately because they are not direct measurements of trace substances. The methods employed also have less stringent QA requirements.

Specifically, this section presents the results from mercury speciation tests, the results of the particle size distribution tests, and the measured concentrations of dibasic acid in the FGD liquor.

Mercury Speciation Tests

The solid sorbent method developed by Frontier Geosciences was used to determine the speciation of mercury in the flue gas. This method collects vapor phase mercury on two KCl-impregnated soda lime traps followed by two iodated carbon traps. The traps are installed in a quartz tube which is placed in a heated probe (maintained at 100-120° C). All of the sampling is performed at a single point in the gas stream. At Site 20, approximately 100 L of flue gas was collected at a rate of 0.5 L/min. The sorbent traps were then removed by Radian personnel, packaged, and shipped to Frontier Geosciences for analysis.

Oxidized mercury (Hg^{2+}) and methyl mercury (CH_3-Hg) were determined by dissolving the KCl-impregnated traps in an acetic acid/HCl mixture, followed by aqueous ethylation, separation by GC, and detection by cold vapor atomic fluorescence spectrometry (CVAFS). Oxidized mercury was detected as diethyl mercury and methyl mercury as methyl ethyl mercury. Recently, the validity of the methyl mercury determination has been questioned by Frontier Geosciences. Therefore, the distinction between methyl and ionic mercury is questionable.

Elemental mercury (Hg^0) was determined by digesting the carbon traps in 10 mL of 7:3 HNO_3/H_2SO_4 at 70° C for two to three hours and then diluting them to 100 mL with 0.05 N BrCl. The mercury in the resulting digestate was reduced using $SnCl_2$, trapped on a gold surface, then detected by CVAFS.

The speciation procedure assumes that all the oxidized and methyl mercury is collected on the KCl/soda lime trap and that all the mercury on the carbon trap is elemental (i.e., Hg^0).

Table 5-1 shows that the ESP outlet gas contained about 3.2 $\mu g/Nm^3$ elemental mercury; the stack gas contained about 12.4 $\mu g/Nm^3$. These data suggest that a large

Table 5-1
Mercury Speciation Data ($\mu g/Nm^3$)

Location	Run	Frontier Geosciences Hg^0	Method 29 Permanganate Impinger	Frontier Geosciences Hg^{2+}[a]	Method 29 Peroxide Impinger
ESP Outlet	1	3.23	9.3	20.4	3.8
	2	5.46	NC	21.0	NC
	3	1.77	5.8	14.7	5.1
	4	2.41	7.0	22.7	9.1
	Avg:	3.22	7.4	19.7	6.0
Stack	1	NC	11.6	NC	1.1
	2	14.2	9.6	0.44	0.8
	3	10.4	8.0	1.88	1.7
	4	12.7	8.8	0.52	0.9
	Avg:	12.4	9.5	0.95	1.1

[a] Frontier Geosciences Hg^{2+} values include the data that was originally reported as methyl mercury.

NC = Sample not collected.

fraction of the oxidized mercury (Hg^{2+}) or methyl mercury was converted to elemental mercury in the FGD system.

Table 5-2 compares the total mercury concentrations measured by the multi-metals method (EPA Method 29) with the results from the mercury speciation method. At both locations, the concentrations measured by Method 29 were lower than those determined by the mercury speciation method. The difference was greatest at the ESP outlet, ranging from 52-80%. The stack concentrations differed by 26-40%. Also, note that the average mercury removal differs for the methods.

The theoretical flue gas mercury content in the ESP outlet gas is 23 $\mu g/Nm^3$, based on the levels of mercury in the fly ash, bottom ash, and coal at Site 20 (using the DGAA values for mercury in the coal). The average value obtained by Method 29 was 13.4 ± 6.5 $\mu g/Nm^3$; the average obtained using the mercury speciation method was 22.9 ± 7.1 $\mu g/Nm^3$.

The method comparison results are surprising because data from previous sites have shown good agreement between the two methods. Note that the low values for the field blanks (Table 5-1) indicate that the solid sorbent traps were not contaminated.

Frontier Geosciences reported that an unexpected precipitate formed in three of the soda lime traps for the ESP outlet location (first trap only). Frontier Geosciences dissolved the precipitate by adding an extra 10 mL HCl to the digestion. Initially, Frontier Geosciences attributed the precipitate to particulate on the glass wool or to contamination by a flue gas species associated with the lignite coal. Further investigation revealed that the initial acetic acid solution was too dilute. The extra HCl added solubilized the soda lime by lowering the pH.

Dibasic Acid Concentrations

Radian collected FGD liquor samples from both of the lower loop reaction tanks on each test day. Radian analyzed these samples for dibasic acid (DBA) to document the level of this additive in the FGD system. The DBA results are presented in Table 5-3.

Particle Size Distribution Tests

Table 5-4 presents the results of the particle size distribution tests for the ESP outlet and stack locations. Samples from the various size fractions were archived for possible elemental analyses in the future.

Fly Ash Enrichment Data

Although the particle size distribution samples were not chemically analyzed, the existing test data were used to estimate whether certain trace species were enriched in the smaller particle sizes at Site 20. For species that were enriched in the smaller particle sizes, their concentration should be highest in the solids collected on the ESP outlet

Table 5-2
Comparison of Method 29 with the Mercury Speciation Method (Total Hg $\mu g/Nm^3$)

Location	Run	Method 29	Solid Sorbent
ESP Outlet	1	13.13	23.66
	2	NC	26.48
	3	10.89	16.50
	4	16.07	25.11
	Days 1,3,4 Average:	13.36	21.76
Stack	1	12.70	NC
	2	10.43	14.63
	3	9.72	12.28
	4	9.74	13.23
	Days 2,3,4 Average:	9.96	13.38
	Average Hg Removal:	25%	39%
Average Hg Removal by FGD	Days 3,4	28%	39%
	Days 1,3,4	20%	--
	Days 2,3,4	--	41%

NC = Sample not collected.

Table 5-3
Dibasic Acid Concentrations at Site 20

	Reaction Tank A		Reaction Tank B	
Run	**(mmol/l)**	**(ppm)**	**(mmol/l)**	**(ppm)**
1	3.31	430	3.41	443
2	3.22	419	3.12	406
3	2.68	348	3.04	395
4	2.48	322	2.97	386
Average	2.92	380	3.14	408

Table 5-4
Particle Size Distribution Data for Site 20

ESP Outlet Location				
Gas Sampled	865.1 dscf 26.3 Nm^3			
Percent Isokinetic	79			
Stage Number	**Cut Size (μm)**	**Collected Mass (g)**	**Grain Loading (gr/dscf)**	**Grain Loading (mg/Nm^3)**
1	7.2	0.2337	0.0042	8.89
2	2.4	0.0258	0.0005	0.98
3	0.54	0.0034	0.0001	0.13
Filter		0.0062	0.0001	0.24
		Total:	0.0048	10.23
Stack Location				
Gas Sampled	788.4 dscf 24.0 Nm^3			
Percent Isokinetic	97			
Stage Number	**Cut Size (μm)**	**Collected Mass (g)**	**Grain Loading (gr/dscf)**	**Grain Loading (mg/Nm^3)**
1	7.2	0.1994	0.0039	8.32
2	2.4	0.3201	0.0063	13.36
3	0.54	0.0645	0.0013	2.69
Filter		0.0498	0.0010	2.08
		Total:	0.0124	26.45

sample filers and lowest in the bottom ash solids. The concentrations in the bulk fly ash and the fly ash collected in the last field of the ESP should be in between the other two *concentrations* (the concentration should be lower in the bulk fly ash because the ash collected in the last ESP field should have a smaller particle size than the bulk fly ash.

Figure 5-1 shows that arsenic, cadmium, lead, selenium, and phosphorus are the primary species enriched in the finer particle sizes. These results were expected, because of the relatively high vapor pressure of these elements at boiler temperatures. The concentrations of arsenic and phosphorus were about twice as high in the last field ash compared to the bulk fly ash. Selenium was about four times more concentrated in the last field ash.

Comparison of Analytical Methods

Historically, three analytical techniques have been used to analyze for the FCEM target metals. Because of its greater sensitivity, GFAAS is used to analyze for arsenic, cadmium, lead, nickel, and selenium; CVAAS is used for mercury; and ICP-AES is used for the others. ICP-MS, which is sensitive enough for all of the target elements, was investigated as an alternative analytical method at Site 20. ICP-MS analyses were done on both the front half (solid phase) and back half (vapor phase) of the three multi-metals trains at the absorber outlet and the four multi-metals trains at the stack. Also, ICP-MS analyses were done on digested and undigested fractions of the vapor phase samples and the results were compared.

Table 5-5 compares GFAAS and ICP-AES with ICP-MS analytical results for the solid phase fraction. These data show that the ICP-MS results are generally the same as standard GFAAS or ICP-AES results. Although the mean values may vary slightly between the methods, the 95% confidence intervals of these values overlap for most analytes. The ICP-MS QA spike recoveries for a laboratory control sample (LCS) were generally below the data quality objectives, indicating that a method (sample preparation/analysis) bias may exist. The LCS is a blank filter prepared in a standardized aqueous solution. Since the entire sample filters were digested, it was not possible to prepare matrix spike/duplicate QA samples for analysis. The matrix spike and matrix spike duplicate (MS/MSD) samples might have been able to indicate if interferences or method biases were responsible for these poor results. For these reasons, except for selenium, the ICP-MS results were not presented in Section 3. ICP-MS data for selenium were chosen over the GFAAS data since the QC results for GFAAS were outside the control limits. Because the selenium concentrations in the samples were high, the spectral interferences common to the ICP-MS determination of selenium are minimized.

The impingers (vapor phase fraction) were analyzed by ICP-MS (digested and undigested samples) and GFAAS or ICP-AES. The results for the vapor-phase fractions which appear in Table 5-6, show that, in general, the analytes not detected by standard methods (arsenic, beryllium, and chromium) were detected by ICP-MS at levels at or below the standard method detection limits. Two analytes, arsenic in the undigested

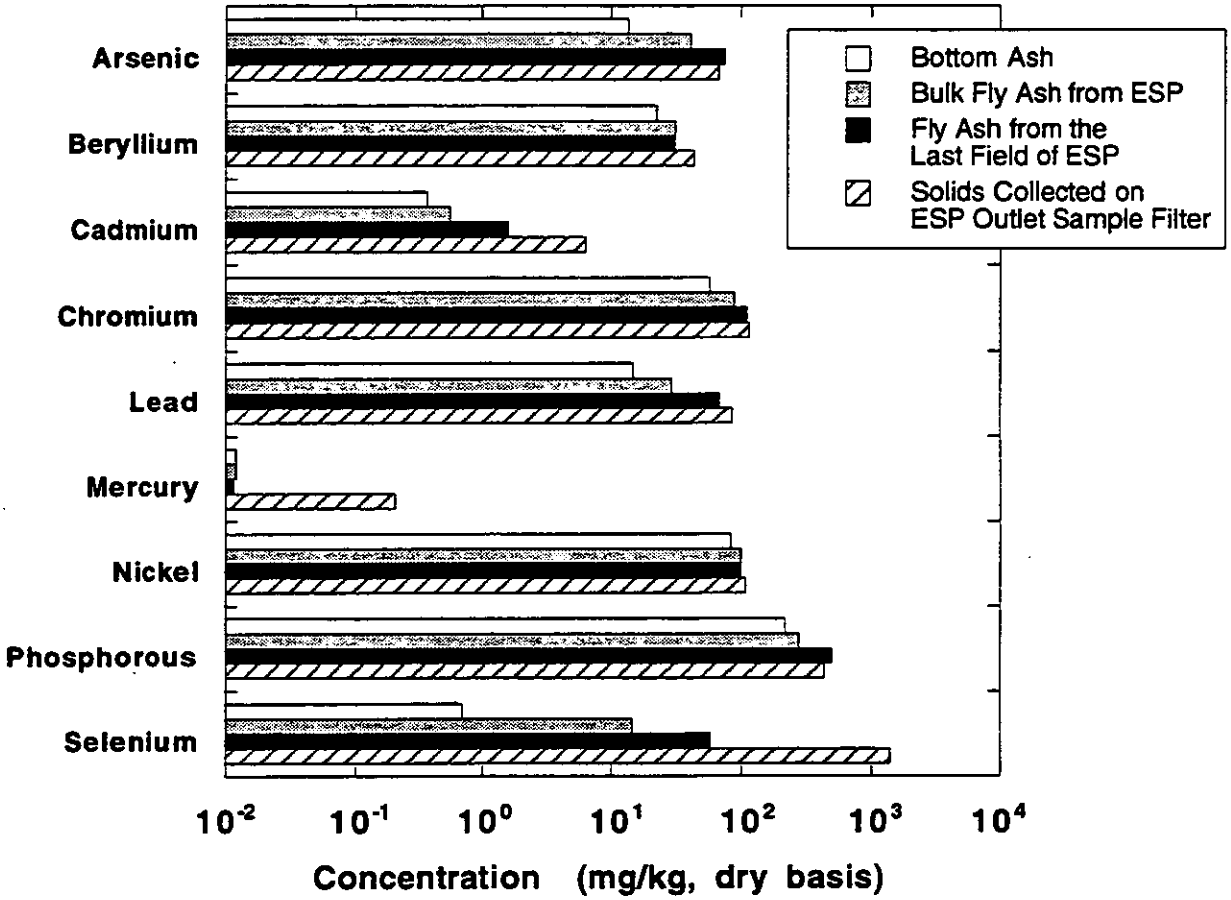

Figure 5-1
FCEM Target Species Concentrations in Site 20 Ash Streams

Table 5-5
Comparison of ICP-MS to Standard Methods for ESP Outlet and Stack Gas Solid Phase ($\mu g/Nm^3$)

Method	Substance	ESP Outlet Mean[a]	95% CI[b]	Stack Mean[c]	95% CI[b]
GFAAS	Arsenic	3.1	2.7	0.58	0.3
ICP-MS	Arsenic	1.6	1.2	0.27	0.12
ICP-AES	Beryllium	2.0	2.8	0.32	0.15
ICP-MS	Beryllium	1.1	1.5	0.15	0.07
GFAAS	Cadmium	0.3	0.2	0.48	0.59
ICP-MS	Cadmium	0.24	0.16	0.28	0.29
ICP-AES	Chromium	5.3	3.2	2.5	1.6
ICP-MS	Chromium	2.6	1.8	0.90	0.54
GFAAS	Lead	3.6	3.6	0.7	0.52
ICP-MS	Lead	3.92	3.32	0.96	0.53
GFAAS	Nickel	5.0	5.3	3.9	1.6
ICP-MS	Nickel	16.1	16.94	2.67	1.09
GFAAS	Selenium	125	117	45.1	25.5
ICP-MS	Selenium	64.6	49.47	17.9	6.57
CVAAS	Mercury	ND	--	0.04	0.04
ICP-MS	Mercury	0.06	0.11	0.06	0.05

[a] The mean was calculated using three data points.

[b] CI = Confidence interval.

[c] The mean was calculated using four data points.

Table 5-6
Comparison of ICP-MS to Standard Methods for ESP Outlet and Stack Gas Vapor Phase ($\mu g/Nm^3$)

Method	Substance	ESP Outlet Mean[a]	95% CI[b]	Stack Mean[c]	95% CI[b]
GFAAS	Arsenic	ND(0.14)	--	ND(0.12)	--
undig[d]/ICP-MS	Arsenic	0.26	0.15	0.008	0.008
dig[d]/ICP-MS	Arsenic	0.06	0.06	0.008	0.008
ICP-AES	Beryllium	ND(0.12)	--	ND(0.12)	--
undig/ICP-MS	Beryllium	0.01	0.02	ND	--
dig/ICP-MS	Beryllium	0.01	0.02	0.008	0.008
GFAAS	Cadmium	0.18	0.12	0.17	0.18
undig/ICP-MS	Cadmium	0.05	0.04	0.05	0.03
dig/ICP-MS	Cadmium	0.17	0.04	0.14	0.12
ICP-AES	Chromium	ND(0.54)	--	ND(0.53)	--
undig/ICP-MS	Chromium	0.40	0.30	0.14	0.02
dig/ICP-MS	Chromium	0.76	0.15	0.47	0.04
GFAAS	Lead	2.4	2.1	2.0	2.0
undig/ICP-MS	Lead	0.17	0.06	0.07	0.03
dig/ICP-MS	Lead	3.1	2.6	2.45	2.54
GFAAS	Nickel	ND(2.13)	--	ND(2.08)	--
undig/ICP-MS	Nickel	0.89	0.46	0.26	0.1
dig/ICP-MS	Nickel	0.85	0.49	0.44	0.14
GFAAS	Selenium	90.5	334.5	140	32
undig/ICP-MS	Selenium	659	196	132	38
dig/ICP-MS	Selenium	644	186	129	58
CVAAS	Mercury	13.4	6.5	10.6	2.3
undig/ICP-MS	Mercury	11.07	10.45	2.19	1.6
dig/ICP-MS	Mercury	10.45	10.77	1.68	1.59

[a] The mean was calculated using three data points.

[b] CI = Confidence interval.

[c] The mean was calculated using four data points.

[d] "Undig" = undigested sample; "dig" = digested sample.

fraction and chromium in the digested fraction, were detected by ICP-MS above the standard method detection limits. Arsenic results may be biased high because of the formation of ArCl in the argon plasma. Chromium was detected in the method blank at levels similar to that observed in the digested sample, suggesting that a method (sample preparation) bias may exist.

A comparison of the ICP-MS results for digested and undigested vapor-phase fractions indicates that the results were essentially the same. While there is some variation between the mean values for the digested and undigested fractions, the 95% confidence intervals of these values overlap. Since the vapor-phase samples are trapped in aqueous solution (i.e., the analytes are soluble) it is not unexpected that digestion of these samples has little or no effect on the observed concentrations.

Further study is needed to address the apparent bias problem associated with the analysis of solid-phase fractions of the metals train by ICP-MS before this method can be used as the primary means of analysis. The digestion of vapor-phase fractions was shown to have virtually no effect on the observed analyte concentrations. We recommend that, in the future, only undigested vapor-phase fractions be analyzed.

6

EXAMPLE CALCULATIONS

This section presents selected examples of the calculations used to develop the results discussed in Sections 3 and 4. Specifically, the calculation of stream flow rates, mean concentration values and confidence intervals, and emission factors are presented here.

Stream Flow Rates

Coal flow rates were determined from the plant's calibrated coal feeders. Bottom ash and fly ash flow rates were calculated based on the ash content of the coal and assuming that 20% of the total ash was bottom ash and the rest was fly ash. This assumption was based on the boiler design information for Site 20.

Flue gas flow rates were measured directly during sampling at the stack location. The flow rate measurements at the ESP outlet location were considered to be unreliable, due to the nonideal gas flow characteristics at this location. Since material (SO_2) and energy balances indicated that no flue gas bypassed the FGD system, the ESP outlet flue gas flow rate was assumed to be the same as the stack flow rate (both flows on a dry basis). A combustion calculation using data for the Site 20 coal showed the calculated gas flow rate to be very close to the measured stack gas flow rate.

Estimates for the FGD system flow rates (i.e., limestone and FGD solids) were based on the amount of SO_2 removed from the system (as determined by gas-phase analyses and gas flow rates). The makeup water flow rate was estimated by a water balance around the FGD system and was corrected for the amount of water lost with the FGD solids (assuming the FGD solids contained 35% water).

Means and Confidence Intervals for Stream Concentrations

The mean concentrations and 95% confidence intervals (CIs) about the mean were calculated for each target substance in the streams sampled. The means were calculated according to the conventions listed in Section 3. The equations used to calculate the 95% confidence intervals are presented in Appendix E. Example calculations for arsenic in the ESP outlet gas follow here; these results were shown in Table 3-10.

The concentration data (in $\mu g/Nm^3$) given for arsenic in Table 3-10 are:

	Run 1	Run 3	Run 4
Solid Phase	2.9	4.3	2.2
Vapor Phase	ND(0.12)	ND(0.13)	ND(0.14)
Total	2.9	4.3	2.2

The mean is calculated from the individual run totals:

$$\text{Mean} = (2.9 + 4.3 + 2.2)/3$$

$$= 3.1$$

The sample standard deviation of the individual run totals is calculated:

$$S_p = \sqrt{\left[(2.9-3.1)^2 + (4.3-3.1)^2 + (2.2-3.1)^2\right] /2}$$

$$= 1.07$$

The standard deviation of the average is calculated according to Equation 6 in Appendix E for N = 3:

$$S_{\bar{p}} = 1.07/\sqrt{3}$$

$$= 0.62$$

The bias error is found by root-sum-squaring the product of the bias error and the sensitivity from each run (see Equation 2 in Appendix E). According to the conventions listed in Section 3, no bias error is assigned to values above reporting limits, whereas a bias error of one-half the detection limit is assigned to values below detection limits. The sensitivity of the mean to each run in this case is 1/3.

$$\beta_r = \sqrt{(1/3 \times 0)^2 + (1/3 \times 0)^2 + (1/3 \times 0)^2}$$

$$= 0$$

The total uncertainty in the result is found from Equation 1 in Appendix E:

$$U_r = \sqrt{\beta_r^2 + (t \times S_p)^2}$$

$$= \sqrt{0^2 + (4.3 \times 0.62)^2}$$

$$= 2.7$$

Thus, the result is reported as 3.1 ± 2.7 $\mu g/Nm^3$.

Unit Energy Emission Factors

In addition to the gas phase concentrations, unit energy-based emission factors have been developed for each target substance. These values were determined by calculating the mass flow rate of a substance in the flue gas (mean concentration times mean flow rate) and dividing by the mean heat input to the boiler during testing. The mean heat input is the product of the mean coal flow rate and the mean higher heating value (HHV) of the coal.

For example, note the calculation of the emission factor for arsenic at the ESP outlet. The mean coal flow rate is 620,000 lb/hr on a dry basis. The mean HHV of the coal is 10,017 Btu/lb on a dry basis. Multiplying the coal flow rate by the HHV gives a mean heat input of 6.2×10^9 Btu/hr. The mean arsenic mass flow through the stack (the product of the mean concentration, 0.57 $\mu g/Nm^3$, and the mean gas flow rate, 3,095,000 Nm^3/hr) is 1.8×10^6 $\mu g/hr$ or 0.004 lb/hr. When the mean mass flow rate is divided by the mean heat input, an emission factor of 0.63 $lb/10^{12}$ Btu is obtained, as shown in Table 3-12.

The 95% confidence intervals for emission factors were calculated according to the equations presented in Appendix E. For each parameter (flue gas flow rate, concentration, coal flow rate, and HHV) the mean, standard deviation, number of points, and bias estimates were used to calculate the combined uncertainty in the mean emission factors.

7

GLOSSARY

AAS	Atomic Absorption Spectrometry
Btu	British Thermal Unit
CI	Confidence Interval
CVAAS	Cold Vapor Atomic Absorption Spectrophotometry
DGAA	Double Gold Amalgamation
DQO	Data Quality Objective
dscfm	Dry Standard Cubic Feet per Minute (1 atm, 68° F)
ESP	Electrostatic Precipitator
FCEM	Field Chemical Emissions Monitoring
GFAAS	Graphite Furnace Atomic Absorption Spectrophotometry
HHV	Higher Heating Value
IC	Ion Chromatography
ICP (ICAP, ICP-AES, ICAPES)	Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP-MS, ICPES-MS	Inductively Coupled Argon Plasma Mass Spectroscopy
INAA	Instrument Neutron Activation Analysis
ISE	Ion Selective Electrode
MDL	Method Detection Limit
MSD	Matrix Spike Duplicate
MW	Megawatt
NBS	National Bureau of Standards
ND	Not Detected (below detection limit)
Nm^3	Dry Normal Cubic Meter (0° C, 1 atm)
PAH	Polynuclear Aromatic Hydrocarbons
POM	Polycyclic Organic Matter
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
RSD	Relative Standard Deviation

APPENDIX A: SAMPLING AND ANALYTICAL SUMMARY

This appendix presents details of the sampling and analytical activities performed at Site 20.

Sampling Summary

Sampling was performed from June 8 to June 11, 1993. Samples from several process streams were collected during each day of sampling. These streams included:

- ESP outlet flue gas;
- Stack flue gas;
- Coal fed to the power plant boiler;
- ESP collected fly ash;
- Sluiced boiler bottom ash;
- Fly ash from one of the last ESP fields;
- Limestone;
- FGD makeup water;
- FGD solids; and
- FGD liquor.

Flue Gas Sampling

Flue gas samples were collected at the ESP outlet and at the stack to determine the concentration of the following substances:

- Trace metals;
- Mercury (speciated); and
- Anions.

Information about the sampling methods used for these substances is presented below.

Multi-Metals Collection

The sampling methodology specified in Section 3.1 of the 40 CFR Part 266, Appendix IX was used to determine the particulate mass loading and simultaneously collect solid and vapor phase samples of the stack flue gas for trace metals analysis. This method was modified for sampling at the ESP outlet location.

The first modification consisted of using Teflon® tubing to transfer flue gas from the filter holder to the impinger train. This tubing was necessary because vertical sampling was conducted using a very long sampling probe (20 feet). A three-inch filter holder was attached directly to the exit of this probe and a small oven was used to keep the filter at 250°F. The Teflon® tubing was needed because it was not practical or safe to attach the impinger train directly to the exit of the filter holder as is specified by the published method. At the end of each test day, the Teflon® tubing was soaked for a minimum of 15 minutes with nitric acid solution to recover any trace metals that might have adsorbed during sampling. The resulting rinse was added to the first nitric acid impinger sample.

The second modification consisted of not monitoring the flue gas flow rate during actual sample collection at the ESP outlet. A velocity profile was performed just prior to testing and the flow rate data from the velocity profile was used to determine the sampling rate needed to collect the sample at isokinetic conditions. The approach of using velocity profile data to calculate isokinetic sampling rates was also used during the collection of anions samples and Cyclade PSD samples at the ESP outlet.

Twelve sampling ports were present at the ESP outlet sampling location. A detailed velocity profile was performed on June 7 at the ESP outlet to determine the distribution of flue gas flow across the entire duct. Results of the 96 point (12 X 8) velocity profile traverse are presented in Table A-1. The velocity profile data were used to select suitable sampling ports for collecting the ESP outlet samples. On the first day of testing (June 8), ports 2, 4, 8, and 10 were used to collect metals samples while ports 3, 5, 9, and 11 were used to collect the anions sample. The metals and anions samples were collected from 32 sampling points (4 X 8). Ports 3, 5, 8, and 10 were use to collect both the metals and anions samples at the ESP outlet on June 9,10, and 11.

Horizontal sampling was performed at the stack. The stack sampling ports were far enough upstream and downstream from any disturbances to allow for the use of twelve sampling points (4 x 3) to collect composite metals and anions samples on the stack.

Table A-1
FCEM Site 20 - ESP Outlet Detailed Velocity profile Data

	Port											
Depth	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**
1	59.1 318	59.4 324	60.2 315	57.5 327	54.7 320	45.9 323	54.9 325	59.4 326	61.3 324	63.0 310	58.3 307	65.2 311
2	61.1 319	65.0 323	58.5 313	61.8 327	59.2 320	49.4 323	57.3 323	59.8 326	61.3 324	60.6 305	58.6 305	64.3 307
3	64.8 319	67.4 322	55.1 308	58.3 328	58.8 320	50.3 323	55.2 322	62.1 325	60.1 324	60.4 301	62.3 303	64.9 305
4	60.3 319	62.7 321	53.7 305	57.5 329	56.8 321	53.1 323	52.5 320	60.5 324	61.3 325	60.4 300	64.1 301	66.0 305
5	58.0 319	61.1 320	52.4 303	53.3 330	50.7 321	50.8 322	50.6 318	56.9 323	60.6 326	61.7 297	62.9 299	67.7 306
6	59.2 320	55.1 321	52.9 294	53.3 330	52.1 322	49.3 321	45.2 316	56.5 323	60.6 326	62.4 296	61.0 297	65.8 308
7	55.6 276	56.5 324	56.6 283	59.6 330	57.3 324	48.4 323	41.0 318	53.2 326	59.9 327	62.5 299	61.6 304	63.9 315
8	53.5 254	54.8 322	60.5 286	61.5 330	56.6 327	45.3 320	36.1 318	42.8 325	53.2 327	63.3 318	65.1 310	63.0 320

	Average	Range
Temperature:	315.6	254-330
Velocity:	57.68	36.1-67.7

The top entry in each cell is the gas velocity in feet per second; the second entry is the gas temperature in °F. Temperatures are from the velocity traverse performed on 06/07/93. Velocities are calculated using the measured temperatures and ΔPs from 06/07/94 and average molecular weight and absolute pressure of 28.4 and 30.3, respectively.

A summary of the ESP outlet and stack trace metals source sampling data is presented in Table A-2. A total of four sets of ESP outlet and absorber outlet trace metal samples were collected. The trace metals sample collected at the ESP outlet on the second day of testing was voided because the isokinetic rate could not be maintained. The three sets of ESP outlet and four sets of stack trace metal samples were submitted for analysis.

Anions Collection

A modification to the procedures specified in EPA Method 5 was used to collect solid and vapor phase samples of the ESP outlet and stack flue gas for anions analysis. The anions sampling trains were also used to determine the particulate mass loading. Horizontal sampling was performed at the stack using a typical EPA Method 5-style sampling train. Vertical sampling was performed at the ESP outlet using a modified EPA Method 5 train similar to the one described for the collection of trace metals. A Teflon® sample line was used to transfer the flue gas from the filter holder to the impingers. At the end of each test day, the Teflon® sample line was soaked for a minimum of 15 minutes with a carbonate/bicarbonate solution to recover any anions that may have adsorbed onto the walls of the tubing during sampling. The resulting rinse solution was added to the first impinger sample for analysis.

Two impingers containing 6% hydrogen peroxide were used to collect the vapor phase anions. These impingers were combined into a single sample container. Upon completion of sampling, the probe liner and sampling nozzle (P&N) were rinsed with deionized water and acetone to recover any solids that were present for mass loading determination. The probe liner and nozzle were then rinsed with a carbonate/bicarbonate solution to recover any anions that may have adsorbed onto the walls of the tubing during sampling. The deionized water/acetone P&N rinse was evaporated to determine the mass of solids present. These solids were combined with the carbonate/bicarbonate P&N rinse and the filter to generate the solid phase anions sample.

A summary of the ESP outlet and stack anions sampling data is presented in Table A-4. A total of three sets of ESP outlet and stack anions samples were collected over the period of June 8, 9, and 10. All three sets of anion samples were submitted to the lab for analysis.

Mercury Speciation

Samples of the ESP outlet and stack flue gas were collected for mercury speciation using the Frontier Geosciences' solid sorbent system. The sampling configuration consists of two KCl-saturated soda-lime traps and two iodated carbon traps. The mercury speciation samples were collected at a single point approximately two to three feet from the wall. A total of four sets of mercury speciation samples were collected at the ESP outlet over the period of June 8, 9, 10, and 11. Attempts at collecting a mercury speciation sample on the stack on June 8 were unsuccessful. This resulted in a total of three sets of mercury speciation samples being collected at the stack over the period of June 9, 10, and 11. A summary of the ESP outlet and stack mercury speciation sampling data is

Table A-2
FCEM Site 20 - Trace Metals Source Sampling Data

ESP Outlet

		Flue Gas Composition			
Run No.	Sample Gas Volume (dscf)	Moisture (%)	O_2[a] (%)	CO_2[a] (%)	% Isokinetic
1	288.33	13.1	6.5	10.0	100.3
2[b]					
3	243.08	13.3	7.5	10.5	103.1
4	200.40	13.4	7	10.0	103.7

Stack

		Flue Gas Composition			
Run No.	Sample Gas Volume (dscf)	Moisture[c] (%)	O_2[a] (%)	CO_2[a] (%)	% Isokinetic
1	217.90	16.4/18.1	10	10.5	97.7
2	266.67	16.4/18.2	10	10.5	99.3
3	253.89	16.8/18.0	10	11.0	101.0
4	221.79	17.3/18.2	8.5	10.7	101.1

[a] Two composite bag samples were collected during multi-metals sample collection at the ESP outlet and stack for CO_2 and O_2 analysis by Fyrite.

[b] ESP outlet metals Run 2 was voided.

[c] The stack flue gas moisture was supersaturated with respect to moisture. The first number represents the saturation moisture content at the average stack temperature. The second number represents the measured flue gas moisture content.

presented in Table A-3. The three sets of ESP outlet and stack mercury speciation samples collected over the period of June 9, 10, and 11 were submitted to Frontier Geosciences for analysis.

Particle Size Distribution

Size-fractionated samples of the particulate matter were collected from the ESP outlet and stack using a Cyclade Model 283-2 cascade cyclone sampler which consists of three cyclones and a final filter. The aerodynamic cut-points of the cyclones are approximately 7.5, 2.7, and 0.57 microns for a flow rate of 1.0 acfm and temperature of 300 degrees F. A point of average velocity was selected for use in collecting the size-fractionated sample at both the ESP outlet and stack.

The ESP outlet and stack Cyclades were allowed to preheat for a minimum of 30 minutes and 60 minutes, respectively, before initiating sampling. The Cyclade final filter was heated using heat tape and a variable voltage transformer to prevent blinding of the filter. A thermocouple monitored the skin temperature of the filter holder during preheating and sample collection. The skin temperature of the stack Cyclade was maintained at between 190 to 230°F during sample collection.

Observations made during the recovery of the PSD samples are documented below:

ESP Outlet

- A very small portion of the filter could not be recovered because parts of the filter were stuck in the holes around the outside perimeter of the support screen. This did not affect the recovery of the filter solids.
- The center of the filter was lightly coated with beige colored solids.
- A small amount of solids were recovered from the No. 5 cyclone. Some of the solids were in the cyclone body, but the majority of solids were recovered from the collection cup. The solids adhered to each other and to the cyclone. The solids had to be scraped from the cyclone. The solids had the sweet aroma of sulfuric acid samples collected at another site.
- There was a small amount of solids in the catch bulbs of cyclones No. 1 and 3. The solids were easily recovered using a camel hair brush.

Stack

- The filter stuck to the support screen in a manner very similar to that encountered at the ESP outlet. Some of the filter media was not recovered, but this should not have affected the recovery of the collected solids.
- The solids on the stack filter were a lighter in color than the ESP outlet solids.

Table A-3
FCEM Site 20 - Mercury Speciation Data

Run No.	Integrator Volume (liters)	
	ESP Outlet	Stack
1	101.6	NC
2	101.4	100
3	103.2	61.9
4	102.7	32.3

NC = Not collected.

- There were no solids in the catch cup of the No. 5 cyclone. There were a small amount of very fine red colored solids in the inlet throat of the No. 5 cyclone. The solids did not appear to be rust. An attempt was made to recover these solids into the sample bottle.

- Cyclone No. 3 contained a small amount of solids that were easily recovered using a camel hair brush.

- Cyclone No. 1 had a layer of solids along the walls of the cyclone chamber, but there were no solids present in the collection cup. The solids appeared to have been wetted at some point and were left behind after the water evaporated.

- It was difficult to move the Cyclade into and out of the port. The Cyclade was generally dirty when it was removed from the stack and contamination of the nozzle area with wet solids is a possibility.

- Amber glass bottles with Teflon® lid inserts were used to store all the cyclone samples.

Flue Gas Flow Rate

The flow rate of flue gas entering the FGD system and exiting the stack were determined using the procedures specified in EPA Methods 1, 2, 3, and 4. Two velocity profiles were performed during each day of testing at the ESP outlet. An initial velocity profile was performed in the morning to determine the conditions necessary to achieve the isokinetic sampling rate at the meter for both the metals and anions samples. A second ESP outlet velocity profile was performed at the end of the day to verify that the flow rate had not changed appreciably during the day. The velocity data showed that the flue gas temperature increased from about 310°F in the morning to about 330°F in the afternoon. The increase in flue gas temperature was attributed to a combination of increasing ambient temperature during the day and heat loss to duct work structure and fans early in the morning while the boiler load was being increased. The higher, end of the day, ESP outlet flue gas temperatures were used to calculate the flue gas flow rate each day because the plant's process data indicated that these temperatures were more representative of the average gas temperature for the entire run.A summary of the flue gas flow rate data obtained at the ESP outlet and stack is presented in Table A-5.

Process Stream Sampling

Coal

Coal samples were collected every day from samplers on four of the six operating coal feed chutes. The individual samples (approximately 500 ml each) were taken about every two hours during the gas sampling activities and added to a plastic collection bucket. At the end of the test day, the material in the bucket was well-mixed using a

Table A-4
FCEM Site 20 - Anions Source Sampling Data

ESP Outlet					
		Flue Gas Composition			
Run No.	Sample Gas Volume (dscf)	Moisture (%)	O_2 [a] (%)	CO_2 [a] (%)	% Isokinetic
1	72.98	13.5	6.5	10.0	100.5
2	63.45	13.3	8.0	11.0	102.4
3	62.21	13.9	7.5	10.5	102.4
Stack					
		Flue Gas Composition			
Run No.	Sample Gas Volume (dscf)	Moisture [b] (%)	O_2 [a] (%)	CO_2 [a] (%)	% Isokinetic
1	73.43	17.7/18.6	10	10.5	96.3
2	74.81	17.7/18.9	10	10.5	98.0
3	72.32	17.7/21.0	10	11.0	99.6

[a] Two composite bag samples were collected during multi-metals sample collection at the ESP outlet and stack for CO_2 and O_2 analysis by Fyrite.

[b] The stack flue gas moisture was supersaturated with respect to moisture. The first number represents the saturation moisture content at the average stack temperature. The second number represents the measured flue gas moisture content.

Table A-5
FCEM Site 20 - Flue Gas Flow Rate Data

ESP Outlet				
	Average Flue Gas			
Run No.	ΔP^a (inches H_2O)	Temperature[a] (°F)	Velocity (ft/sec)	Flow Rate (dscfm E+06)
1	0.96	312	66.8	1.73
2	0.93	332	66.4	1.68
3	0.92	332	66.0	1.68
4	0.89	335	65.1	1.65

Stack						
		Average Flue Gas				
Run No.	Train	ΔP (inches H_2O)	Temperature (°F)	Velocity (ft/sec)	Flow Rate (dscfm E+06)	Average Flow Rate of Metals & Anions (dscfm E+06)
1	Metals Anions	1.48 1.61	133 136	74.4 77.9	1.93 1.98	1.96
2	Metals Anions	1.56 1.61	133 136	76.1 77.8	1.98 1.98	1.98
3	Metals Anions	1.51 1.49	134 136	74.9 74.5	1.94 1.98	1.92
4	Metals	1.49	135	74.5	1.92	N/A

[a] The ΔPs and flue gas temperatures for Day 1 are from the pre-test velocity traverse. The ΔPs and flue gas temperature for Days 2, 3, and 4 are from the post-test velocity.

large plastic spoon. A daily composite sample was then obtained by scooping the well-mixed material into a composite sample bottle.

There were two different types of samplers: continuous and batch. The continuous samplers used a slow turning screw to extract coal from the feed chute. The coal accumulated in a stainless steel collection pipe, which was emptied each time an individual sample was collected.

The batch samplers consisted of a knife-gate valve and collection pipe mounted on the wall of the feed chute. The collection pipe was constructed out of a combination of carbon steel pipe and PVC pipe. The knife-gate valves did not close properly, so coal accumulated in the collection pipe in between the collection of the individual samples. The accumulated coal was purged from the collection pipe before each of the individual samples were collected, so the valve problem should have no impact on the collected coal samples.

Bottom Ash

Bottom ash samples were collected using a PVC pipe that was manually inserted into the bottom ash slurry stream as the slurry entered the bottom ash pond. The slurry samples flowed through the pipe into a five-gallon bucket. The slurry was allowed to settle before decanting most of the liquid. The concentrated slurry was then stirred and added to another bucket, which accumulated the bottom ash sample for the test. Multiple slurry samples were collected and processed in an identical manner. At the end of the bottom ash sluicing process, the bucket containing the accumulated bottom ash slurry sample was stirred and then allowed to settle. Most of the water was then decanted. The composite bottom ash sample for the test was obtained by scooping a sample from the settled bottom ash bucket.

Bottom ash samples were collected within one hour after the flue gas sampling was completed. In order to provide a sample of the boiler bottom ash that was representative of the gas sampling period, bottom ash sluicing was suspended approximately one hour before gas testing began and resumed after gas testing was completed. Grab samples of the sluice were collected after bottom ash solids appeared in the sluice water. Sampling continued until it became apparent by the amount of solids present in the sluice water that the boiler bottom ash transfer was complete.

Fly Ash

Fly ash samples were collected during Site 20's daily fly ash silo emptying procedure. Four or five grab samples were collected each day using a plastic scoop to collect the ash as it fell off the end of the fly ash silo's screw conveyor. The grab samples were then combined to form the composite sample for the test day.

FGD Solids

FGD solids samples were collected during Site 20's daily FGD sludge dewatering procedure. Four or five grab samples were collected each day using a plastic scoop to collect the solids as they fell off the rotary vacuum drum filter. The grab samples were then combined to form the composite sample for the test day.

FGD Makeup Water

Radian collected two FGD makeup water samples each day. The samples were collected from a tap near the entrance to the FGD system. The tap was opened and water was allowed to flow at a high rate for at least 15 seconds before collecting the water samples. One sample was cooled to 4°C and then analyzed for anions (in Austin). The other sample was acidified using nitric acid. The acidified sample was analyzed for metals (also in Austin).

Limestone

Radian collected limestone samples from the conveyor belts that feed the limestone ball mills. Multiple samples were collected each day using plastic scoops. These samples were composited to form the sample for the test day.

FGD Liquor

Radian collected daily FGD liquor samples from both of the lower loop reaction tanks. These samples were immediately filtered into a sample bottle containing a known amount of distilled water. The resulting liquid was then analyzed (in Austin) for dibasic acid (DBA), an FGD system performance additive, using ion chromatography.

Detailed Sample Collection/Preparation/Analysis Tables

Table A-6 lists the techniques used to collect, preserve, and handle the samples at Site 20. Analytical methods applied to the coal samples are listed in Table A-7. Analytical methods for all other samples are listed in Table A-8.

Table A-6
Sample Collection, Preservation, and Handling Techniques for Site 20

Stream	Collection Method	Fraction Description	Sample Handling & Preservation	Comments
Flue Gas Samples				
ESP Outlet Gas Stack Gas	Specified in Section 3.1 of 40 CFR, Part 266, Appendix IX[a]	Metals Probe and Nozzle Rinse	Acetone portion dried and weighed; nitric acid portion sealed and kept at ambient temperature.	Digested probe and nozzle rinses combined with digested filters prior to analysis.
		Metals Filter	Desiccated at room temperature to constant weight.	
		Metals Impingers 1 & 2	Sealed and kept at ambient temperature.	
		Metals Impingers 3 & 4	Sealed and kept at ambient temperature.	Analyzed for mercury only.
	Radian Method	Anions Train	Sealed and kept at ambient temperature.	Analyzed for chloride, fluoride, and sulfate.
Water Samples				
FGD Makeup Water	Grab	Metals	Nitric acid to pH <2; kept in a sealed container.	One grab sample during each test run.
		Cl, F	Kept in a sealed container. Cooled to 4°C.	
Solid Samples				
Coal	Grab/Composite	Ultimate, Proximate Metals; Cl, F	Kept in sealed container; submitted "as collected."	Multiple grab samples collected during the test period were composited directly into buckets, coned and quartered to reduce sample size.
Bottom Ash Solids	Grab/Composite	Metals; Cl, F	Placed in sealed container in the field; dried at 105°C and ground prior to analysis.	Multiple grab samples taken during bottom ash sluicing were composited in a large plastic container. Solids were allowed to settle and the water was decanted.

Table A-6 (Continued)

Stream	Collection Method	Fraction Description	Sample Handling & Preservation	Comments
ESP Ash	Grab/Composite	Metals; Cl, F	Kept in sealed container; submitted "as collected."	Multiple grab samples taken from screw feeder with plastic scoop; composited in plastic bucket.
FGD Solids	Grab/Composite	Metals; Cl, F	Kept in sealed container; submitted "as collected."	Multiple grab samples from rotary drum filter; composited in a plastic bucket.
Limestone	Grab/Composite	Metals; Cl, F	Kept in sealed container; submitted "as collected."	Multiple grab samples from limestone conveyor belts; composited in a plastic bucket.

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[a] 40 CFR, Part 266, Appendix IX, Section 3.1, "Methodology for the Determination of Metals Emissions from Hazardous Waste Incineration and Similar Combustion Processes," 1991.

Table A-7
Preparation Procedures and Chemical Analysis Methods Applied to Coal at Site 20

Component	Method Reference
Ultimate Analysis of Coal	
Ash	ASTM D 3174
Carbon	ASTM D 3178
Hydrogen	ASTM D 3178
Nitrogen	ASTM D 3179
Sulfur	ASTM D 4239
Heating Value	ASTM D 2015
Chlorine and Fluorine Analysis in Coal	
Preparation	
Oxygen Bomb Digestion	ASTM D 4208/ASTM D 3761
Analysis by Potentiometric Titration	
Chloride	SM 407C
Analysis by Ion Selective Electrode	
Fluoride	ASTM D 3761
Other FCEM Species in Coal	
Preparation	
Ashing at 500°C/Acid Digestion	ASTM 3683
Analysis by GFAAS	
Cadmium	SW 7131
Arsenic	SW 7131
Selenium	SW 7740
Analysis by ICP-AES	
Ba, Be, Co, Cr, Pb, Ni, P, V	SW 6010
Selenium	ASTM D 4326-84
Mercury Analysis in Coal	
Preparation	
Double Gold Amalgamation	Karr, Chapter 14
Analysis by CVAAS	
Mercury	Karr, Chapter 14

Karr, C. Jr., (ed)., "Analytical Methods for Coal and Coal Products."

SW is EPA SW-846, "Test Methods for Evaluating Solid Waste".

SM is "Standard Methods for the Examination of Water and Wastewater," 16th ed.

Table A-8
Preparation Procedures and Chemical Analysis Methods for Inorganic Chemical Components in Non-Coal Solids, Water Samples, and Flue Gas at Site 20

Component	Method Reference	Non-Coal Solids	Water Samples	Flue Gas Solids	Metals Impingers 1 & 2	Metals Impingers 3 & 4	Anions Impingers
Target Elements by ICP-AES							
Preparation							
MW Digestion for Filters	CEM-F			X			
MW Digestion for Solids	CEM-FA	X					
Analysis by ICP-AES							
Beryllium	SW 6010	X	X	X	X		
Chromium	SW 6010	X	X	X	X		
Nickel	SW 6010	X	X	X	X		
Target Elements by GFAAS							
Preparation							
MW Digestion for Filters	CEM-F			X			
MW Digestion for Solids	CEM-FA	X					
Analysis by GFAAS							
Arsenic	SW 7060	X	X	X	X		
Cadmium	SW 7131	X	X	X	X		
Lead	SW 7421	X	X	X	X		
Selenium	SW 7740	X	X	X	X		

Table A-8 (Continued)

Component	Method Reference	Non-Coal Solids	Water Samples	Flue Gas Solids	Metals Impingers 1 & 2	Metals Impingers 3 & 4	Anions Impingers
Mercury by CVAAS							
Preparation							
MW Digestion for Filters	CEM-F			X			
MW Digestion for Solids	CEM-FA	X					
Analysis by CVAAS							
Mercury	SW 7470	X	X	X	X	X	
Acid-Forming Anions							
Preparation							
Aqueous Extraction of Solids (Cl,F)	Radian	X		X			
Sodium Hydroxide Fusion (F)	McQuaker and Gurney	X					
Chloride by IC	EPA 300.0	X	X	X			X
Fluoride by ISE	EPA 340.2	X	X	X			X
Additional Inorganic Analytes by ICP-AES							
Preparation							
MW Digestion for Filters	CEM-F			X			
MW Digestion for Solids	CEM-FA	X					
Analysis by ICP-AES							
Aluminum	SW 6010	X	X	X	X		
Antimony	SW 6010	X	X	X	X		
Barium	SW 6010	X	X	X	X		
Calcium	SW 6010	X	X	X	X		
Cobalt	SW 6010	X	X	X	X		

Table A-8 (Continued)

Component	Method Reference	Non-Coal Solids	Water Samples	Flue Gas Solids	Metals Impingers 1 & 2	Metals Impingers 3 & 4	Anions Impingers
Copper	SW 6010	X	X	X	X		
Iron	SW 6010	X	X	X	X		
Magnesium	SW 6010	X	X	X	X		
Manganese	SW 6010	X	X	X	X		
Molybdenum	SW 6010	X	X	X	X		
Phosphorus	SW 6010	X	X	X	X		
Potassium	SW 6010	X	X	X	X		
Sodium	SW 6010	X	X	X	X		
Vanadium	SW 6010	X	X	X	X		

ASTM is American Society for Testing and Materials.

EPA is EPA Methods for Chemical Analysis of Water and Wastes, 1983.

SW is EPA SW-846, "Test Methods for Evaluating Solid Waste", 3rd ed.

CEM-FA CEM Corporation, Matthews, NC Procedure for microwave digestion of coal fly ash.

CEM-F CEM Corporation, Matthews, NC Procedure for microwave digestion of glass or quartz filters.

McQuaker, N. R., and M. Gurney. "Determination of Total Fluoride in Soil and Vegetation Using An Alkali Fusion-Selective Ion Electrode Technique", *Analytical Chemistry*, Vol. 49, No. 1, January 1977, pp. 53-56.

APPENDIX B: DATA USED IN CALCULATIONS

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Bottom ash	ICP-AES	Aluminum	mg/kg	5.28E+04	5.18E+04	5.34E+04	5.57E+04
Coal	XRF	Aluminum	percent	1.68E+00	1.54E+00	1.76E+00	1.33E+00
ESP outlet, gas phase	ICP-AES	Aluminum	µg/Nm3	1.61E+01		2.54E+01	2.59E+01
ESP outlet, solid phase	ICP-AES	Aluminum	µg/Nm3	3.40E+03		4.50E+03	3.29E+03
FGD solids	ICP-AES	Aluminum	mg/kg	7.12E+02	6.97E+02	7.78E+02	6.30E+02
Fly ash	ICP-AES	Aluminum	mg/kg	7.48E+04	7.50E+04	8.33E+04	8.08E+04
Limestone	ICP-AES	Aluminum	mg/kg	4.11E+02	3.76E+02	3.79E+02	4.24E+02
Makeup water	ICP-AES	Aluminum	mg/L	1.73E-01	1.44E-01	1.56E-01	1.61E-01
Precipitator ash	ICP-AES	Aluminum	mg/kg	9.22E+04	9.22E+04	9.30E+04	
Stack gas, gas phase	ICP-AES	Aluminum	µg/Nm3	6.43E+00	< 5.57E+00	9.32E+00	8.42E+00
Stack gas, solid phase	ICP-AES	Aluminum	µg/Nm3	5.11E+02	9.49E+02	4.65E+02	6.12E+02
Bottom ash	ICP-AES	Antimony	mg/kg	< 1.82E+02	< 7.39E+01	< 7.22E+01	< 7.34E+01
ESP outlet, gas phase	ICP-AES	Antimony	µg/Nm3	< 4.31E+00		< 4.90E+00	< 5.20E+00
ESP outlet, solid phase	ICP-AES	Antimony	µg/Nm3	< 9.92E-01		< 1.18E+00	< 1.43E+00
FGD solids	ICP-AES	Antimony	mg/kg	< 1.30E+00	< 1.62E+00	< 1.48E+00	< 1.38E+00
Fly ash	ICP-AES	Antimony	mg/kg	< 7.12E+01	< 7.44E+01	< 7.42E+01	< 7.48E+01
Limestone	ICP-AES	Antimony	mg/kg	< 1.44E+00	< 1.41E+00	< 1.45E+00	< 1.43E+00
Makeup water	ICP-AES	Antimony	mg/L	< 2.41E-02	< 2.41E-02	< 2.41E-02	< 2.41E-02
Precipitator ash	ICP-AES	Antimony	mg/kg	< 7.24E+01	< 7.32E+01	< 2.25E+02	
Stack gas, gas phase	ICP-AES	Antimony	µg/Nm3	< 4.65E+00	< 4.73E+00	< 5.09E+00	< 5.09E+00
Stack gas, solid phase	ICP-AES	Antimony	µg/Nm3	< 1.31E+00	< 1.07E+00	< 1.13E+00	< 1.29E+00
Bottom ash	GFAAS	Arsenic	mg/kg	1.40E+01	1.49E+01	1.23E+01	1.31E+01
Coal	GFAAS	Arsenic	mg/kg	4.00E+00	3.00E+00	3.00E+00	1.00E+00
ESP outlet, gas phase	GFAAS	Arsenic	µg/Nm3	< 1.18E-01		< 1.34E-01	< 1.42E-01
ESP outlet, solid phase	GFAAS	Arsenic	µg/Nm3	2.94E+00		4.29E+00	2.17E+00
FGD solids	GFAAS	Arsenic	mg/kg	1.97E+00	1.62E+00	1.85E+00	1.50E+00
Fly ash	GFAAS	Arsenic	mg/kg	3.87E+01	4.18E+01	4.67E+01	3.94E+01
Limestone	GFAAS	Arsenic	mg/kg	6.27E+00	7.19E+00	8.27E+00	4.93E+00
Makeup water	GFAAS	Arsenic	mg/L	1.20E-03	1.80E-03	2.00E-03	1.50E-03
Precipitator ash	GFAAS	Arsenic	mg/kg	7.52E+01	7.54E+01	7.46E+01	
Stack gas, gas phase	GFAAS	Arsenic	µg/Nm3	< 1.27E-01	< 1.29E-01	< 1.39E-01	< 1.39E-01
Stack gas, solid phase	GFAAS	Arsenic	µg/Nm3	4.59E-01	8.57E-01	4.96E-01	4.85E-01
Coal	Proximate	Ash	percent	2.11E+01	2.00E+01	2.01E+01	2.26E+01
Bottom ash	ICP-AES	Barium	mg/kg	2.25E+03	2.53E+03	4.00E+03	2.25E+03
Coal	ICP-AES	Barium	mg/kg	2.10E+02	1.80E+02	1.60E+02	2.10E+02
ESP outlet, gas phase	ICP-AES	Barium	µg/Nm3	2.15E+00		6.16E-01	6.26E-01
ESP outlet, solid phase	ICP-AES	Barium	µg/Nm3	1.29E+02		2.02E+02	1.35E+02
FGD solids	ICP-AES	Barium	mg/kg	4.91E+01	5.41E+01	5.80E+01	5.15E+01
Fly ash	ICP-AES	Barium	mg/kg	2.25E+03	2.41E+03	3.10E+03	2.60E+03
Limestone	ICP-AES	Barium	mg/kg	8.67E+01	1.12E+02	7.31E+01	6.75E+01
Makeup water	ICP-AES	Barium	mg/L	1.50E-01	1.70E-01	1.51E-01	1.64E-01

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Precipitator ash	ICP-AES	Barium	mg/kg	2.63E+03	2.63E+03	2.68E+03	
Stack gas, gas phase	ICP-AES	Barium	µg/Nm3	3.92E+00	3.71E-01	2.66E-01	3.46E-01
Stack gas, solid phase	ICP-AES	Barium	µg/Nm3	2.73E+01	6.56E+01	2.78E+01	2.91E+01
Bottom ash	ICP-AES	Beryllium	mg/kg	2.03E+01	2.38E+01	3.34E+01	1.25E+01
Coal	ICP-AES	Beryllium	mg/kg	5.10E+00	6.40E+00	1.00E+01	4.60E+00
ESP outlet, gas phase	ICP-AES	Beryllium	µg/Nm3	< 9.92E-02		< 1.13E-01	< 1.20E-01
ESP outlet, solid phase	ICP-AES	Beryllium	µg/Nm3	1.43E+00		3.34E+00	1.31E+00
FGD solids	ICP-AES	Beryllium	mg/kg	9.64E-02	6.67E-02	1.48E-01	1.42E-01
Fly ash	ICP-AES	Beryllium	mg/kg	2.59E+01	2.84E+01	4.38E+01	2.69E+01
Limestone	ICP-AES	Beryllium	mg/kg	5.54E-02	7.48E-02	5.34E-02	5.62E-02
Makeup water	ICP-AES	Beryllium	mg/L	< 5.54E-04	< 5.54E-04	6.10E-04	< 5.54E-04
Precipitator ash	ICP-AES	Beryllium	mg/kg	3.23E+01	2.94E+01	3.05E+01	
Stack gas, gas phase	ICP-AES	Beryllium	µg/Nm3	< 1.07E-01	< 1.09E-01	< 1.17E-01	< 1.17E-01
Stack gas, solid phase	ICP-AES	Beryllium	µg/Nm3	2.56E-01	4.61E-01	2.88E-01	2.81E-01
Bottom ash	GFAAS	Cadmium	mg/kg	< 3.61E-01	< 3.66E-01	< 3.58E-01	< 3.63E-01
Coal	GFAAS	Cadmium	mg/kg	3.00E-01	2.00E-01	1.00E-01	1.00E-01
ESP outlet, gas phase	GFAAS	Cadmium	µg/Nm3	2.11E-01		1.16E-01	2.03E-01
ESP outlet, solid phase	GFAAS	Cadmium	µg/Nm3	2.18E-01		3.01E-01	3.60E-01
FGD solids	GFAAS	Cadmium	mg/kg	1.92E-01	2.02E-01	2.61E-01	1.82E-01
Fly ash	GFAAS	Cadmium	mg/kg	1.10E+00	7.78E-01	< 3.67E-01	< 3.71E-01
Limestone	GFAAS	Cadmium	mg/kg	3.52E-01	3.90E-01	4.11E-01	3.81E-01
Makeup water	GFAAS	Cadmium	mg/L	< 3.10E-04	< 3.10E-04	< 3.10E-04	3.30E-04
Precipitator ash	GFAAS	Cadmium	mg/kg	1.67E+00	1.83E+00	1.28E+00	
Stack gas, gas phase	GFAAS	Cadmium	µg/Nm3	3.07E-01	8.44E-02	2.01E-01	7.81E-02
Stack gas, solid phase	GFAAS	Cadmium	µg/Nm3	9.04E-01	7.29E-02	6.39E-01	2.63E-01
Bottom ash	ICP-AES	Calcium	mg/kg	4.85E+04	4.52E+04	3.46E+04	6.04E+04
Coal	XRF	Calcium	percent	1.31E+00	1.21E+00	7.50E-01	1.30E+00
ESP outlet, gas phase	ICP-AES	Calcium	µg/Nm3	5.55E+01		1.05E+02	1.08E+02
ESP outlet, solid phase	ICP-AES	Calcium	µg/Nm3	2.42E+03		2.81E+03	2.53E+03
FGD solids	ICP-AES	Calcium	mg/kg	2.61E+05	2.78E+05	2.82E+05	2.63E+05
Fly ash	ICP-AES	Calcium	mg/kg	5.00E+04	8.73E+04	4.39E+04	6.85E+04
Limestone	GFAAS	Calcium	mg/kg	3.49E+05	3.43E+05	3.45E+05	3.37E+05
Makeup water	ICP-AES	Calcium	mg/L	5.17E+01	5.31E+01	5.04E+01	5.11E+01
Precipitator ash	ICP-AES	Calcium	mg/kg	6.84E+04	6.42E+04	6.69E+04	
Stack gas, gas phase	ICP-AES	Calcium	µg/Nm3	< 2.86E+01	< 2.90E+01	< 3.13E+01	< 3.12E+01
Stack gas, solid phase	ICP-AES	Calcium	µg/Nm3	7.13E+02	1.49E+03	7.52E+02	1.15E+03
Coal	Ultimate	Carbon	percent	6.12E+01	6.23E+01	6.04E+01	5.98E+01
Bottom ash	IC	Chloride	mg/kg	3.62E+00	5.35E+00	3.26E+00	5.64E+00
Coal	IC	Chloride	mg/kg	9.02E+01	1.65E+02	1.80E+02	7.66E+01
ESP outlet, gas phase	IC	Chloride	µg/Nm3	2.53E+03	8.75E+03	2.37E+03	
ESP outlet, solid phase	IC	Chloride	µg/Nm3	3.24E+01	6.51E+01	2.32E+01	

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
FGD solids	IC	Chloride	mg/kg	1.61E+02	1.50E+02	1.77E+02	1.92E+02
Fly ash	IC	Chloride	mg/kg	< 2.56E-01	1.57E+00	< 2.53E-01	2.24E+00
Limestone	IC	Chloride	mg/kg	2.24E+01	2.48E+01	2.69E+01	6.45E+01
Makeup water	IC	Chloride	mg/L	1.71E+01	2.15E+01	8.25E+00	1.98E+01
Precipitator ash	IC	Chloride	mg/kg	1.14E+00			
Stack gas, gas phase	IC	Chloride	µg/Nm3	5.21E+01	6.86E+01	1.74E+02	
Stack gas, solid phase	IC	Chloride	µg/Nm3	1.27E+02	1.33E+02	5.00E+02	
Bottom ash	ICP-AES	Chromium	mg/kg	5.81E+01	5.58E+01	6.18E+01	5.45E+01
Coal	ICP-AES	Chromium	mg/kg	1.60E+01	1.40E+01	1.70E+01	1.70E+01
ESP outlet, gas phase	ICP-AES	Chromium	µg/Nm3	< 4.46E-01		< 5.07E-01	< 5.37E-01
ESP outlet, solid phase	ICP-AES	Chromium	µg/Nm3	4.61E+00		6.81E+00	4.55E+00
FGD solids	ICP-AES	Chromium	mg/kg	5.26E+00	5.49E+00	5.32E+00	4.64E+00
Fly ash	ICP-AES	Chromium	mg/kg	8.64E+01	8.36E+01	9.51E+01	9.00E+01
Limestone	ICP-AES	Chromium	mg/kg	3.05E+00	3.36E+00	3.01E+00	3.07E+00
Makeup water	ICP-AES	Chromium	mg/L	< 2.49E-03	< 2.49E-03	< 2.49E-03	< 2.49E-03
Precipitator ash	ICP-AES	Chromium	mg/kg	1.10E+02	1.10E+02	1.14E+02	
Stack gas, gas phase	ICP-AES	Chromium	µg/Nm3	< 4.81E-01	< 4.89E-01	< 5.26E-01	< 5.26E-01
Stack gas, solid phase	ICP-AES	Chromium	µg/Nm3	2.79E+00	3.70E+00	1.34E+00	2.24E+00
Bottom ash	ICP-AES	Cobalt	mg/kg	3.00E+01	2.94E+01	4.20E+01	1.80E+01
Coal	ICP-AES	Cobalt	mg/kg	6.00E+00	6.00E+00	9.00E+00	4.00E+00
ESP outlet, gas phase	ICP-AES	Cobalt	µg/Nm3	< 6.09E-01		< 6.92E-01	< 7.34E-01
ESP outlet, solid phase	ICP-AES	Cobalt	µg/Nm3	1.99E+00		3.67E+00	1.82E+00
FGD solids	ICP-AES	Cobalt	mg/kg	4.75E-01	7.16E-01	6.62E-01	5.16E-01
Fly ash	ICP-AES	Cobalt	mg/kg	3.49E+01	3.54E+01	5.10E+01	3.81E+01
Limestone	ICP-AES	Cobalt	mg/kg	6.49E-01	4.21E-01	4.45E-01	4.18E-01
Makeup water	ICP-AES	Cobalt	mg/L	< 3.40E-03	< 3.40E-03	< 3.40E-03	< 3.40E-03
Precipitator ash	ICP-AES	Cobalt	mg/kg	4.28E+01	4.26E+01	4.59E+01	
Stack gas, gas phase	ICP-AES	Cobalt	µg/Nm3	< 6.57E-01	< 6.67E-01	< 7.19E-01	< 7.18E-01
Stack gas, solid phase	ICP-AES	Cobalt	µg/Nm3	4.98E-01	8.93E-01	4.55E-01	6.65E-01
Bottom ash	ICP-AES	Copper	mg/kg	9.44E+01	8.77E+01	9.42E+01	8.69E+01
ESP outlet, gas phase	ICP-AES	Copper	µg/Nm3	2.51E+00		1.39E+00	< 8.22E-01
ESP outlet, solid phase	ICP-AES	Copper	µg/Nm3	7.47E+00		1.03E+01	7.72E+00
FGD solids	ICP-AES	Copper	mg/kg	2.57E+00	2.68E+00	2.73E+00	2.39E+00
Fly ash	ICP-AES	Copper	mg/kg	1.16E+02	1.30E+02	1.39E+02	1.37E+02
Limestone	ICP-AES	Copper	mg/kg	1.84E+00	2.12E+00	1.46E+00	1.81E+00
Makeup water	ICP-AES	Copper	mg/L	< 3.81E-03	< 3.81E-03	< 3.81E-03	< 3.81E-03
Precipitator ash	ICP-AES	Copper	mg/kg	1.66E+02	1.68E+02	2.27E+02	
Stack gas, gas phase	ICP-AES	Copper	µg/Nm3	< 7.36E-01	< 7.48E-01	< 8.05E-01	9.63E-01
Stack gas, solid phase	ICP-AES	Copper	µg/Nm3	6.43E+00	2.64E+00	1.50E+00	1.87E+00
Bottom ash	ISE	Fluoride	mg/kg	B 7.53E-01	B 7.09E-01	B 8.56E-01	B 8.72E-01
Coal	ISE	Fluoride	mg/kg	4.00E+01	3.00E+01	4.00E+01	4.00E+01

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
ESP outlet, gas phase	ISE	Fluoride	µg/Nm3	4.42E+03	4.16E+03	4.08E+03	
ESP outlet, solid phase	ISE	Fluoride	µg/Nm3	9.40E+00	1.45E+01	1.89E+01	
FGD solids	ISE	Fluoride	mg/kg	1.73E+01	1.55E+01	1.50E+01	1.76E+01
Fly ash	ISE	Fluoride	mg/kg	7.96E+00	6.51E+00	8.55E+00	6.20E+00
Limestone	ISE	Fluoride	mg/kg	B 3.25E+00	B 2.99E+00	B 5.22E+00	6.36E+00
Makeup water	ISE	Fluoride	mg/L	3.11E-01	3.25E-01	3.30E-01	3.14E-01
Precipitator ash	ISE	Fluoride	mg/kg	6.95E+01			
Stack gas, gas phase	ISE	Fluoride	µg/Nm3	B 9.19E+01	B 6.94E+01	B 7.15E+01	
Stack gas, solid phase	ISE	Fluoride	µg/Nm3	B 1.53E+02	B 1.49E+02	B 6.37E+02	
Coal	Calorimetry	HHV	Btu/lb	1.00E+04	1.01E+04	1.01E+04	9.86E+03
Coal	Ultimate	Hydrogen	percent	4.17E+00	4.29E+00	4.22E+00	3.93E+00
Bottom ash	ICP-AES	Iron	mg/kg	9.50E+04	9.11E+04	1.09E+05	7.96E+04
Coal	XRF	Iron	percent	1.58E+00	1.68E+00	2.28E+00	1.65E+00
ESP outlet, gas phase	ICP-AES	Iron	µg/Nm3	1.22E+01		2.40E+01	1.90E+01
ESP outlet, solid phase	ICP-AES	Iron	µg/Nm3	2.67E+03		5.26E+03	2.57E+03
FGD solids	ICP-AES	Iron	mg/kg	1.17E+03	1.20E+03	1.29E+03	1.10E+03
Fly ash	ICP-AES	Iron	mg/kg	9.27E+04	8.92E+04	9.79E+04	8.35E+04
Limestone	ICP-AES	Iron	mg/kg	8.80E+02	8.93E+02	8.78E+02	9.32E+02
Makeup water	ICP-AES	Iron	mg/L	1.03E-01	8.76E-02	9.05E-02	2.26E-01
Precipitator ash	ICP-AES	Iron	mg/kg	8.99E+04	9.80E+04	9.45E+04	
Stack gas, gas phase	ICP-AES	Iron	µg/Nm3	4.13E+00	3.24E+00	5.33E+00	5.02E+00
Stack gas, solid phase	ICP-AES	Iron	µg/Nm3	4.06E+02	8.68E+02	4.38E+02	4.77E+02
Bottom ash	GFAAS	Lead	mg/kg	2.84E+01	1.33E+01	1.00E+01	7.48E+00
Coal	ICP-AES	Lead	mg/kg	2.10E+01	1.10E+01	9.00E+00	7.00E+00
ESP outlet, gas phase	GFAAS	Lead	µg/Nm3	3.29E+00		1.70E+00	2.05E+00
ESP outlet, solid phase	GFAAS	Lead	µg/Nm3	5.30E+00		3.02E+00	2.57E+00
FGD solids	GFAAS	Lead	mg/kg	< 7.28E-02	< 7.75E-02	< 8.21E-02	< 1.24E-01
Fly ash	GFAAS	Lead	mg/kg	3.58E+01	3.48E+01	2.32E+01	2.27E+01
Limestone	GFAAS	Lead	mg/kg	9.02E-01	8.29E-01	7.40E-01	9.53E-01
Makeup water	GFAAS	Lead	mg/L	5.26E-03	6.73E-03	6.86E-03	9.22E-03
Precipitator ash	GFAAS	Lead	mg/kg	3.86E+01	1.25E+02	4.24E+01	
Stack gas, gas phase	GFAAS	Lead	µg/Nm3	3.59E+00	5.38E-01	2.01E+00	1.81E+00
Stack gas, solid phase	GFAAS	Lead	µg/Nm3	6.54E-01	1.15E+00	3.84E-01	6.02E-01
Bottom ash	ICP-AES	Magnesium	mg/kg	1.06E+04	1.05E+04	8.19E+03	1.25E+04
Coal	XRF	Magnesium	percent	2.80E-01	2.70E-01	1.80E-01	2.80E-01
ESP outlet, gas phase	ICP-AES	Magnesium	µg/Nm3	5.46E+00		6.79E+00	< 4.92E+00
ESP outlet, solid phase	ICP-AES	Magnesium	µg/Nm3	5.38E+02		6.38E+02	5.45E+02
FGD solids	ICP-AES	Magnesium	mg/kg	2.45E+03	2.35E+03	2.40E+03	2.38E+03
Fly ash	ICP-AES	Magnesium	mg/kg	1.15E+04	1.48E+04	1.07E+04	1.47E+04
Limestone	ICP-AES	Magnesium	mg/kg	3.08E+03	3.57E+03	3.38E+03	3.53E+03
Makeup water	ICP-AES	Magnesium	mg/L	7.81E+00	8.05E+00	7.68E+00	7.95E+00

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Precipitator ash	ICP-AES	Magnesium	mg/kg	1.55E+04	1.47E+04	1.51E+04	
Stack gas, gas phase	ICP-AES	Magnesium	µg/Nm3	< 4.40E+00	< 4.47E+00	< 4.82E+00	< 4.81E+00
Stack gas, solid phase	ICP-AES	Magnesium	µg/Nm3	4.63E+02	6.67E+02	4.26E+02	7.52E+02
Bottom ash	ICP-AES	Manganese	mg/kg	2.45E+02	2.65E+02	3.61E+02	2.70E+02
Coal	ICP-AES	Manganese	mg/kg	5.40E+01	6.80E+01	9.80E+01	6.90E+01
ESP outlet, gas phase	ICP-AES	Manganese	µg/Nm3	4.35E+00		1.08E+00	7.10E-01
ESP outlet, solid phase	ICP-AES	Manganese	µg/Nm3	1.66E+01		3.38E+01	1.65E+01
FGD solids	ICP-AES	Manganese	mg/kg	9.58E+01	9.25E+01	8.90E+01	7.84E+01
Fly ash	ICP-AES	Manganese	mg/kg	4.22E+02	3.62E+02	4.46E+02	3.67E+02
Limestone	ICP-AES	Manganese	mg/kg	1.12E+02	1.11E+02	1.12E+02	1.13E+02
Makeup water	ICP-AES	Manganese	mg/L	1.17E-02	7.70E-03	6.80E-03	1.34E-02
Precipitator ash	ICP-AES	Manganese	mg/kg	5.13E+02	5.66E+02	5.46E+02	
Stack gas, gas phase	ICP-AES	Manganese	µg/Nm3	2.11E-01	6.08E+00	2.58E+00	9.25E+01
Stack gas, solid phase	ICP-AES	Manganese	µg/Nm3	3.90E+00	6.51E+00	3.85E+00	4.90E+00
Bottom ash	CVAAS	Mercury	mg/kg	< 1.17E-02	< 1.17E-02	< 1.20E-02	< 1.15E-02
Coal	DGAA	Mercury	mg/kg	2.80E-01	2.20E-01	2.70E-01	2.60E-01
Coal	CVAFS	Mercury	µg/g	2.74E-01	2.45E-01	2.81E-01	2.88E-01
ESP outlet, gas phase	CVAAS	Mercury	µg/Nm3	1.31E+01		1.09E+01	1.61E+01
ESP outlet, solid phase	CVAAS	Mercury	µg/Nm3	9.99E-03		9.04E-03	9.08E-03
FGD solids	CVAAS	Mercury	mg/kg	9.53E-01	7.24E-01	8.20E-01	8.29E-01
Fly ash	CVAAS	Mercury	mg/kg	< 1.20E-02	< 1.09E-02	< 1.20E-02	< 1.20E-02
Limestone	CVAAS	Mercury	mg/kg	< 1.20E-02	< 1.20E-02	< 1.20E-02	< 1.20E-02
Makeup water	CVAAS	Mercury	mg/L	< 4.80E-05	< 4.80E-05	< 4.80E-05	< 4.80E-05
Precipitator ash	CVAAS	Mercury	mg/kg	< 1.20E-02	< 1.20E-02	2.25E-02	
Stack gas, gas phase	CVAAS	Mercury	µg/Nm3	1.27E+01	1.04E+01	9.72E+00	9.74E+00
Stack gas, solid phase	CVAAS	Mercury	µg/Nm3	4.35E-02	6.59E-02	1.07E-02	4.99E-02
Coal	Proximate	Moisture	percent	3.35E+01	3.42E+01	3.36E+01	3.44E+01
Bottom ash	ICP-AES	Molybdenum	mg/kg	< 1.41E+01	< 1.43E+01	< 1.40E+01	< 1.42E+01
Coal	ICP-AES	Molybdenum	mg/kg	4.00E+00	4.00E+00	3.00E+00	4.00E+00
ESP outlet, gas phase	ICP-AES	Molybdenum	µg/Nm3	< 8.29E-01		< 9.42E-01	< 9.99E-01
ESP outlet, solid phase	ICP-AES	Molybdenum	µg/Nm3	2.35E+00		2.99E+00	3.11E+00
FGD solids	ICP-AES	Molybdenum	mg/kg	2.83E-01	3.04E-01	4.83E-01	< 1.87E-01
Fly ash	ICP-AES	Molybdenum	mg/kg	< 1.38E+01	< 1.44E+01	< 1.43E+01	< 1.45E+01
Limestone	ICP-AES	Molybdenum	mg/kg	2.67E-01	2.50E-01	2.54E-01	< 1.94E-01
Makeup water	ICP-AES	Molybdenum	mg/L	1.16E-02	1.08E-02	8.95E-03	1.25E-02
Precipitator ash	ICP-AES	Molybdenum	mg/kg	1.48E+01	< 1.41E+01	< 4.36E+01	
Stack gas, gas phase	ICP-AES	Molybdenum	µg/Nm3	< 8.94E-01	< 9.09E-01	< 9.79E-01	< 9.77E-01
Stack gas, solid phase	ICP-AES	Molybdenum	µg/Nm3	2.57E+00	3.52E+00	2.11E+00	2.69E+00
Bottom ash	ICP-AES	Nickel	mg/kg	6.95E+01	7.85E+01	1.28E+02	5.84E+01
Coal	ICP-AES	Nickel	mg/kg	1.50E+01	1.10E+01	1.80E+01	1.30E+01
ESP outlet, gas phase	ICP-AES	Nickel	µg/Nm3	< 1.76E+00		< 2.01E+00	< 2.13E+00

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
ESP outlet, solid phase	ICP-AES	Nickel	µg/Nm3	3.58E+00		7.45E+00	4.03E+00
FGD solids	ICP-AES	Nickel	mg/kg	3.33E+00	1.48E+00	2.33E+00	3.27E+00
Fly ash	ICP-AES	Nickel	mg/kg	7.71E+01	9.84E+01	1.26E+02	1.01E+02
Limestone	ICP-AES	Nickel	mg/kg	2.42E+00	3.40E+00	2.18E+00	2.85E+00
Makeup water	ICP-AES	Nickel	mg/L	< 9.86E-03	< 9.86E-03	< 9.86E-03	< 9.86E-03
Precipitator ash	ICP-AES	Nickel	mg/kg	1.01E+02	1.04E+02	9.82E+01	
Stack gas, gas phase	ICP-AES	Nickel	µg/Nm3	< 1.90E+00	< 1.93E+00	< 2.08E+00	< 2.08E+00
Stack gas, solid phase	ICP-AES	Nickel	µg/Nm3	3.46E+00	4.73E+00	2.70E+00	4.69E+00
Coal	Ultimate	Nitrogen	percent	1.25E+00	1.21E+00	1.16E+00	1.48E+00
Coal	Ultimate	Oxygen	percent	1.02E+01	1.02E+01	1.18E+01	9.68E+00
Bottom ash	ICP-AES	Phosphorous	mg/kg	2.74E+02	2.25E+02	2.53E+02	1.22E+02
Coal	ICP-AES	Phosphorous	mg/kg	9.50E+01	6.00E+01	3.50E+01	5.00E+01
ESP outlet, gas phase	ICP-AES	Phosphorous	µg/Nm3	< 1.09E+01		1.59E+01	< 1.32E+01
ESP outlet, solid phase	ICP-AES	Phosphorous	µg/Nm3	2.37E+01		2.14E+01	1.53E+01
FGD solids	ICP-AES	Phosphorous	mg/kg	4.38E+01	4.28E+01	4.05E+01	3.09E+01
Fly ash	ICP-AES	Phosphorous	mg/kg	3.25E+02	2.89E+02	2.55E+02	2.61E+02
Limestone	GFAAS	Phosphorous	mg/kg	6.89E+01	5.18E+01	6.94E+01	6.46E+01
Makeup water	ICP-AES	Phosphorous	mg/L	7.10E-02	< 6.10E-02	< 6.10E-02	< 6.10E-02
Precipitator ash	ICP-AES	Phosphorous	mg/kg	4.85E+02	5.07E+02	4.62E+02	
Stack gas, gas phase	ICP-AES	Phosphorous	µg/Nm3	1.22E+01	< 1.20E+01	1.54E+01	1.89E+01
Stack gas, solid phase	ICP-AES	Phosphorous	µg/Nm3	6.11E+00	7.80E+00	3.22E+00	5.55E+00
Bottom ash	ICP-AES	Potassium	mg/kg	5.18E+03	5.46E+03	5.70E+03	5.24E+03
ESP outlet, gas phase	ICP-AES	Potassium	µg/Nm3	< 5.14E-01		< 5.84E-01	< 6.19E-01
ESP outlet, solid phase	ICP-AES	Potassium	µg/Nm3	2.73E+02		3.79E+02	2.82E+02
FGD solids	ICP-AES	Potassium	mg/kg	1.65E+02	2.08E+02	2.17E+02	1.85E+02
Fly ash	ICP-AES	Potassium	mg/kg	7.79E+03	7.55E+03	9.82E+03	9.13E+03
Limestone	ICP-AES	Potassium	mg/kg	1.38E+02	1.20E+02	1.28E+02	1.28E+02
Makeup water	ICP-AES	Potassium	mg/L	5.94E+00	6.17E+00	5.73E+00	5.99E+00
Precipitator ash	ICP-AES	Potassium	mg/kg	8.03E+03	8.11E+03	8.30E+03	
Stack gas, gas phase	ICP-AES	Potassium	µg/Nm3	< 5.54E-01	< 5.63E-01	6.55E+00	1.68E+02
Stack gas, solid phase	ICP-AES	Potassium	µg/Nm3	5.79E+01	9.39E+01	3.82E+01	5.86E+01
Bottom ash	GFAAS	Selenium	mg/kg	< 6.82E-01	< 6.91E-01	< 6.76E-01	< 6.86E-01
Coal	GFAAS	Selenium	mg/kg	6.00E+00	5.00E+00	4.00E+00	3.00E+00
ESP outlet, gas phase	GFAAS	Selenium	µg/Nm3	1.66E+01		8.79E+00	2.46E+02
ESP outlet, solid phase	GFAAS	Selenium	µg/Nm3	7.41E+01		1.36E+02	1.66E+02
FGD solids	GFAAS	Selenium	mg/kg	2.16E+01	2.15E+01	3.95E+01	2.48E+01
Fly ash	GFAAS	Selenium	mg/kg	1.32E+01	1.46E+01	8.97E+00	1.15E+01
Limestone	GFAAS	Selenium	mg/kg	3.08E+00	3.44E+00	2.87E+00	2.72E+00
Makeup water	GFAAS	Selenium	mg/L	3.02E-03	4.29E-03	2.70E-03	3.29E-03
Precipitator ash	GFAAS	Selenium	mg/kg	5.78E+01	5.79E+01	6.02E+01	
Stack gas, gas phase	GFAAS	Selenium	µg/Nm3	1.23E+02	1.58E+02	1.23E+02	1.57E+02

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Stack gas, solid phase	GFAAS	Selenium	µg/Nm3	4.21E+01	6.85E+01	3.24E+01	3.76E+01
Bottom ash	ICP-AES	Sodium	mg/kg	2.44E+03	2.37E+03	1.41E+03	2.47E+03
Coal	XRF	Sodium	percent	1.10E-01	8.00E-02	3.00E-02	7.00E-02
ESP outlet, gas phase	ICP-AES	Sodium	µg/Nm3	5.58E+01		8.38E+01	1.03E+02
ESP outlet, solid phase	ICP-AES	Sodium	µg/Nm3	2.10E+02		1.93E+02	2.24E+02
FGD solids	ICP-AES	Sodium	mg/kg	3.20E+02	3.05E+02	3.14E+02	3.34E+02
Fly ash	ICP-AES	Sodium	mg/kg	2.78E+03	3.94E+03	2.55E+03	3.92E+03
Limestone	ICP-AES	Sodium	mg/kg	5.02E+01	4.99E+01	5.06E+01	4.98E+01
Makeup water	ICP-AES	Sodium	mg/L	5.69E+01	5.65E+01	5.13E+01	5.49E+01
Precipitator ash	ICP-AES	Sodium	mg/kg	4.21E+03	3.99E+03	5.00E+03	
Stack gas, gas phase	ICP-AES	Sodium	µg/Nm3	5.50E+01	3.92E+01	5.03E+01	4.39E+01
Stack gas, solid phase	ICP-AES	Sodium	µg/Nm3	1.65E+02	1.56E+02	1.09E+02	1.66E+02
ESP outlet, gas phase	S04	Sulfate	µg/Nm3	5.15E+06		4.51E+06	
ESP outlet, solid phase	S04	Sulfate	µg/Nm3	2.23E+03	3.94E+03	6.64E+03	
Makeup water	S04	Sulfate	mg/L	2.08E+02	2.30E+02	1.19E+02	2.19E+02
Stack gas, gas phase	S04	Sulfate	µg/Nm3	2.55E+05	1.90E+05	2.40E+05	
Stack gas, solid phase	S04	Sulfate	µg/Nm3	5.86E+03	6.10E+03	2.51E+04	
Coal	Ultimate	Sulfur	percent	2.15E+00	2.03E+00	2.32E+00	2.47E+00
Bottom ash	ICP-AES	Vanadium	mg/kg	1.19E+02	1.07E+02	1.20E+02	1.04E+02
Coal	ICP-AES	Vanadium	mg/kg	2.80E+01	3.20E+01	3.70E+01	3.60E+01
ESP outlet, gas phase	ICP-AES	Vanadium	µg/Nm3	< 4.22E-01		< 4.80E-01	< 5.09E-01
ESP outlet, solid phase	ICP-AES	Vanadium	µg/Nm3	1.05E+01		1.35E+01	9.59E+00
FGD solids	ICP-AES	Vanadium	mg/kg	6.40E+00	7.49E+00	7.24E+00	6.58E+00
Fly ash	ICP-AES	Vanadium	mg/kg	1.64E+02	1.74E+02	1.91E+02	1.83E+02
Limestone	ICP-AES	Vanadium	mg/kg	5.28E+00	4.97E+00	4.76E+00	4.99E+00
Makeup water	ICP-AES	Vanadium	mg/L	1.57E-02	1.44E-02	1.20E-02	1.41E-02
Precipitator ash	ICP-AES	Vanadium	mg/kg	2.45E+02	2.37E+02	2.35E+02	
Stack gas, gas phase	ICP-AES	Vanadium	µg/Nm3	< 4.56E-01	< 4.63E-01	< 4.99E-01	< 4.98E-01
Stack gas, solid phase	ICP-AES	Vanadium	µg/Nm3	2.45E+00	3.67E+00	2.27E+00	2.85E+00

APPENDIX C: DATA NOT USED IN CALCULATIONS

The following data in this appendix were not used in any calculations for this report. Note that this section contains trace substance concentrations for the coal at Site 20. The analytical methods used to obtain these data were not the methods specified in the test plan; therefore, these coal data were not used in any calculations for Site 20.

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Bottom ash	ICP-AES	Arsenic	mg/kg	< 2.01E+02	< 5.09E+01	< 4.98E+01	< 5.05E+01
Coal	GFAAS	Arsenic	µg/g	9.00E+00	7.00E+00	1.20E+01	1.20E+01
ESP outlet, gas phase	ICP-AES	Arsenic	µg/Nm3	< 4.03E+00		6.22E+00	< 4.86E+00
ESP outlet, solid phase	ICP-AES	Arsenic	µg/Nm3	< 6.84E-01		< 8.11E-01	< 9.83E-01
FGD solids	ICP-AES	Arsenic	mg/kg	2.80E+00	1.80E+00	2.45E+00	1.58E+00
Fly ash	ICP-AES	Arsenic	mg/kg	< 4.91E+01	< 5.12E+01	< 5.11E+01	< 5.15E+01
Limestone	ICP-AES	Arsenic	mg/kg	< 1.18E+00	1.70E+00	2.12E+00	2.68E+00
Makeup water	ICP-AES	Arsenic	mg/L	2.54E-02	2.86E-02	< 2.25E-02	< 2.25E-02
Precipitator ash	ICP-AES	Arsenic	mg/kg	< 4.99E+01	< 5.04E+01	< 1.55E+02	
Stack gas, gas phase	ICP-AES	Arsenic	µg/Nm3	< 4.35E+00	< 4.42E+00	< 4.76E+00	< 4.75E+00
Stack gas, solid phase	ICP-AES	Arsenic	µg/Nm3	< 9.04E-01	< 7.39E-01	< 7.76E-01	< 8.89E-01
Coal	AAS	Beryllium	µg/g	< 1.00E+00	< 1.00E+00	< 1.00E+00	< 1.00E+00
ESP outlet, gas phase	ICP-AES	Boron	µg/Nm3	7.93E+02		2.00E+03	4.19E+02
FGD solids	ICP-AES	Boron	mg/kg	9.47E+01	8.95E+01	9.70E+01	1.20E+02
Limestone	ICP-AES	Boron	mg/kg	1.11E+00	1.23E+00	9.12E-01	1.24E+00
Makeup water	ICP-AES	Boron	mg/L	2.10E+00	2.11E+00	1.98E+00	2.01E+00
Stack gas, gas phase	ICP-AES	Boron	µg/Nm3	9.37E+01	8.44E+01	1.36E+02	9.06E+01
Bottom ash	ICP-AES	Cadmium	mg/kg	< 3.10E+00	< 3.14E+00	3.27E+00	< 3.12E+00
Coal	XRF	Cadmium	µg/g	1.50E+00	2.00E+00	2.00E+00	< 1.00E+00
ESP outlet, gas phase	ICP-AES	Cadmium	µg/Nm3	< 3.08E-01		< 3.50E-01	< 3.71E-01
ESP outlet, solid phase	ICP-AES	Cadmium	µg/Nm3	2.20E-01		2.78E-01	3.74E-01
FGD solids	ICP-AES	Cadmium	mg/kg	3.25E-01	< 2.41E-01	3.23E-01	
Fly ash	ICP-AES	Cadmium	mg/kg	3.11E+00	< 3.16E+00	4.14E+00	5.04E+00
Limestone	ICP-AES	Cadmium	mg/kg	2.51E-01	3.22E-01	3.28E-01	3.62E-01
Makeup water	ICP-AES	Cadmium	mg/L	< 1.72E-03	< 1.72E-03	< 1.72E-03	< 1.72E-03
Precipitator ash	ICP-AES	Cadmium	mg/kg	4.53E+00	6.11E+00	< 9.58E+00	
Stack gas, gas phase	ICP-AES	Cadmium	µg/Nm3	1.31E+00	< 3.38E-01	< 3.64E-01	6.04E-01
Stack gas, solid phase	ICP-AES	Cadmium	µg/Nm3	8.36E-01	1.34E-01	5.81E-01	2.55E-01
Coal	XRF	Chloride	percent	< 1.00E-02	< 1.00E-02	< 1.00E-02	@ 1.00E-02
Coal	XRF	Chromium	µg/g	1.30E+01	1.20E+01	1.50E+01	1.80E+01
Coal	ISE	Fluoride	mg/kg	5.04E+01	5.71E+01	7.93E+01	5.62E+01
Bottom ash	ICP-AES	Lead	mg/kg	6.54E+01	< 6.59E+01	< 6.44E+01	< 6.54E+01
Coal	XRF	Lead	µg/g	1.70E+01	@ 7.00E+00	@ 6.00E+00	@ 7.00E+00
ESP outlet, gas phase	ICP-AES	Lead	µg/Nm3	< 4.83E+00		< 5.49E+00	< 5.83E+00
ESP outlet, solid phase	ICP-AES	Lead	µg/Nm3	7.05E+00		5.40E+00	5.12E+00
FGD solids	ICP-AES	Lead	mg/kg	< 1.67E+00	< 2.08E+00	< 1.90E+00	< 1.76E+00
Fly ash	ICP-AES	Lead	mg/kg	9.33E+01	8.84E+01	8.70E+01	7.54E+01
Limestone	ICP-AES	Lead	mg/kg	< 1.84E+00	< 1.80E+00	< 1.86E+00	< 1.83E+00
Makeup water	ICP-AES	Lead	mg/L	< 2.70E-02	< 2.70E-02	< 2.70E-02	< 2.70E-02
Precipitator ash	ICP-AES	Lead	mg/kg	1.00E+02	7.49E+01	< 2.01E+02	
Stack gas, gas phase	ICP-AES	Lead	µg/Nm3	1.75E+01	< 5.30E+00	< 5.71E+00	< 5.70E+00

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Stack gas, solid phase	ICP-AES	Lead	µg/Nm3	2.78E+00	3.20E+00	2.10E+00	2.79E+00
Coal	DGAA	Mercury	µg/g	2.11E-01	2.13E-01	2.26E-01	2.13E-01
Coal	XRF	Nickel	µg/g	1.80E+01	1.10E+01	1.20E+01	1.90E+01
Bottom ash	SW846	Percent moisture	%	1.43E+01	1.29E+01	1.32E+01	1.38E+01
FGD solids	SW846	Percent moisture	%	3.20E+01	3.69E+01	3.54E+01	3.87E+01
Limestone	SW846	Percent moisture	%	1.55E+00	2.15E+00	1.75E+00	1.57E+00
Bottom ash	ICP-AES	Selenium	mg/kg	< 1.50E+02	< 1.52E+02	< 1.48E+02	< 1.51E+02
Coal	XRF	Selenium	µg/g	7.50E+00	7.00E+00	7.00E+00	7.00E+00
ESP outlet, gas phase	ICP-AES	Selenium	µg/Nm3	3.87E+02		5.27E+02	4.88E+02
ESP outlet, solid phase	ICP-AES	Selenium	µg/Nm3	8.61E+01		1.51E+02	1.70E+02
FGD solids	ICP-AES	Selenium	mg/kg	2.38E+01	2.84E+01	3.20E+01	3.43E+01
Fly ash	ICP-AES	Selenium	mg/kg	< 1.46E+02	< 1.53E+02	< 1.52E+02	< 1.54E+02
Limestone	ICP-AES	Selenium	mg/kg	4.39E+00		< 3.32E+00	< 3.27E+00
Makeup water	ICP-AES	Selenium	mg/L	< 4.17E-02	< 4.17E-02	5.33E-02	< 4.17E-02
Precipitator ash	ICP-AES	Selenium	mg/kg	< 1.49E+02	< 1.50E+02	< 4.63E+02	
Stack gas, gas phase	ICP-AES	Selenium	µg/Nm3	1.12E+02	1.49E+02	2.05E+02	1.48E+02
Stack gas, solid phase	ICP-AES	Selenium	µg/Nm3	3.95E+01	7.56E+01	4.58E+01	4.85E+01
Bottom ash	ICP-AES	Silicon	mg/kg	2.08E+05	2.14E+05	2.12E+05	2.05E+05
ESP outlet, gas phase	ICP-AES	Silicon	µg/Nm3	3.79E+02		5.29E+02	4.10E+02
ESP outlet, solid phase	ICP-AES	Silicon	µg/Nm3	B 1.21E+04		B 1.92E+04	B 2.65E+04
FGD solids	ICP-AES	Silicon	mg/kg	6.10E+02	6.41E+02	6.33E+02	5.86E+02
Fly ash	ICP-AES	Silicon	mg/kg	2.25E+05	2.22E+05	2.27E+05	2.24E+05
Limestone	ICP-AES	Silicon	mg/kg	3.30E+02	2.75E+02	2.69E+02	3.11E+02
Makeup water	ICP-AES	Silicon	mg/L	3.61E+00	3.49E+00	3.34E+00	3.31E+00
Precipitator ash	ICP-AES	Silicon	mg/kg	1.78E+05	1.88E+05	1.94E+05	
Stack gas, gas phase	ICP-AES	Silicon	µg/Nm3	9.41E+01	7.42E+01	5.33E+01	5.53E+01
Stack gas, solid phase	ICP-AES	Silicon	µg/Nm3	B 2.50E+04	B 2.00E+04	B 2.12E+04	B 2.39E+04
Bottom ash	ICP-AES	Silver	mg/kg	< 1.08E+01	< 1.10E+01	< 1.07E+01	< 1.09E+01
ESP outlet, gas phase	ICP-AES	Silver	µg/Nm3	< 8.81E-01		< 1.00E+00	< 1.06E+00
ESP outlet, solid phase	ICP-AES	Silver	µg/Nm3	< 1.47E-01		< 1.75E-01	< 2.12E-01
FGD solids	ICP-AES	Silver	mg/kg	< 1.24E-01	< 1.54E-01	1.44E-01	< 1.31E-01
Fly ash	ICP-AES	Silver	mg/kg	< 1.06E+01	< 1.10E+01	< 1.10E+01	< 1.11E+01
Limestone	ICP-AES	Silver	mg/kg	< 1.37E-01	< 1.34E-01	< 1.38E-01	< 1.36E-01
Makeup water	ICP-AES	Silver	mg/L	< 4.92E-03	< 4.92E-03	< 4.92E-03	< 4.92E-03
Precipitator ash	ICP-AES	Silver	mg/kg	< 1.07E+01	< 1.09E+01	< 3.34E+01	
Stack gas, gas phase	ICP-AES	Silver	µg/Nm3	< 9.50E-01	< 9.65E-01	< 1.04E+00	< 1.04E+00
Stack gas, solid phase	ICP-AES	Silver	µg/Nm3	< 1.95E-01	< 1.59E-01	< 1.67E-01	< 1.91E-01
Bottom ash	ICP-AES	Strontium	mg/kg	1.32E+03	1.21E+03	8.98E+02	1.61E+03
ESP outlet, gas phase	ICP-AES	Strontium	µg/Nm3	2.06E-01		4.41E-01	4.40E-01
ESP outlet, solid phase	ICP-AES	Strontium	µg/Nm3	6.87E+01		7.23E+01	6.92E+01
FGD solids	ICP-AES	Strontium	mg/kg	8.43E+02	8.19E+02	7.92E+02	7.04E+02

Stream	Test code	Analyte	Units	Run 1	Run 2	Run 3	Run 4
Fly ash	ICP-AES	Strontium	mg/kg	1.28E+03	1.77E+03	1.11E+03	1.83E+03
Limestone	ICP-AES	Strontium	mg/kg	1.06E+03	1.06E+03	1.10E+03	1.04E+03
Makeup water	ICP-AES	Strontium	mg/L	1.44E+00	1.47E+00	1.37E+00	1.38E+00
Precipitator ash	ICP-AES	Strontium	mg/kg	1.76E+03	1.67E+03	1.74E+03	
Stack gas, gas phase	ICP-AES	Strontium	µg/Nm3	4.83E-02	1.00E-01	1.35E-01	1.35E-01
Stack gas, solid phase	ICP-AES	Strontium	µg/Nm3	1.28E+01	2.37E+01	1.07E+01	1.64E+01
Bottom ash	ICP-AES	Thallium	mg/kg	< 1.00E+02	< 1.02E+02	< 9.95E+01	< 1.01E+02
ESP outlet, gas phase	ICP-AES	Thallium	µg/Nm3	3.56E+00		< 3.50E+00	< 3.71E+00
ESP outlet, solid phase	ICP-AES	Thallium	µg/Nm3	< 1.37E+00		< 1.62E+00	< 1.97E+00
FGD solids	ICP-AES	Thallium	mg/kg	< 4.69E+00	< 5.83E+00	< 5.34E+00	< 4.96E+00
Fly ash	ICP-AES	Thallium	mg/kg	< 9.81E+01	< 1.02E+02	< 1.02E+02	< 1.03E+02
Limestone	ICP-AES	Thallium	mg/kg	< 5.18E+00	< 5.07E+00	< 5.22E+00	< 5.14E+00
Makeup water	ICP-AES	Thallium	mg/L	< 1.72E-02	< 1.72E-02	< 1.72E-02	< 1.72E-02
Precipitator ash	ICP-AES	Thallium	mg/kg	< 9.97E+01	< 1.01E+02	< 3.10E+02	
Stack gas, gas phase	ICP-AES	Thallium	µg/Nm3	< 3.32E+00	< 3.38E+00	< 3.64E+00	< 3.63E+00
Stack gas, solid phase	ICP-AES	Thallium	µg/Nm3	< 1.81E+00	< 1.48E+00	< 1.55E+00	< 1.78E+00
Bottom ash	ICP-AES	Zinc	mg/kg	3.31E+02	2.98E+02	2.95E+02	5.90E+01
ESP outlet, gas phase	ICP-AES	Zinc	µg/Nm3	3.06E+00		6.29E+00	4.47E+00
ESP outlet, solid phase	ICP-AES	Zinc	µg/Nm3	5.11E+01		6.07E+01	3.55E+01
FGD solids	ICP-AES	Zinc	mg/kg	7.90E+00	8.61E+00	9.51E+00	7.47E+00
Fly ash	ICP-AES	Zinc	mg/kg	7.67E+02	5.50E+02	4.81E+02	2.96E+02
Limestone	ICP-AES	Zinc	mg/kg	5.60E+00	5.96E+00	5.56E+00	5.76E+00
Makeup water	ICP-AES	Zinc	mg/L	5.91E-03	5.80E-03	4.93E-03	1.21E-02
Precipitator ash	ICP-AES	Zinc	mg/kg	1.07E+03	1.30E+03	1.26E+03	
Stack gas, gas phase	ICP-AES	Zinc	µg/Nm3	4.33E+00	1.14E+00	2.79E+00	2.98E+00
Stack gas, solid phase	ICP-AES	Zinc	µg/Nm3	1.71E+01	2.12E+01	1.27E+01	2.74E+01

APPENDIX D: FLUE GAS SAMPLING DATA SHEETS

(On File at Radian Corporation)

APPENDIX E: ERROR PROPAGATION AND UNCERTAINTY CALCULATIONS

An error propagation analysis was performed on calculated results to determine the contribution of process, sampling, and analytical variability, and measurement bias, to the overall uncertainty in the result. This uncertainty was determined by propagating the bias and precision error of individual parameters into the calculation of the results. This uncertainty does not represent the total uncertainty in the result since many important bias errors are unknown and have been assigned a value of zero for this analysis. Also, this uncertainty is only the uncertainty in the result for the period of time that the measurements were taken.

The procedure described below is based on ANSI/ASME PTC 19.1-1985, "Measurement Uncertainty."

Nomenclature

r = Calculated result;

S_{pi} = Sample standard deviation of parameter i;

θ_i = Sensitivity of the result to parameter i;

β_{pi} = Bias error estimate for parameter i;

v_i = Degrees of freedom in parameter i;

v_r = Degrees of freedom in result;

S_r = Precision component of result uncertainty;

β_r = Bias component of result uncertainty;

t = Student "t" factor (two-tailed distribution at 95%);

U_r = Uncertainty in r; and

N_i = Number of measurements of parameter i.

For a result, r, the uncertainty in r is calculated as:

$$U_r = \sqrt{\beta_r^2 + (S_r * t)^2} \tag{1}$$

The components are calculated by combining the errors in the parameters used in the result calculation.

$$\beta_r = \sqrt{\sum_{i=1}^{j} (\theta_i * \beta_{\overline{pi}})^2} \tag{2}$$

$$S_r = \sqrt{\sum_{i=1}^{j} (\theta_i S_{\overline{pi}})^2} \tag{3}$$

The sensitivity of the result to each parameter is found from a Taylor series estimation method:

$$\theta_i = \frac{\partial r}{\partial pi} \tag{4}$$

Or using a perturbation method (useful in computer applications):

$$\theta_i = \frac{r(P_i + \Delta P_i) - r(P_i)}{\Delta P_i} \tag{5}$$

The standard deviation of the average for each parameter is calculated as:

$$S_{\overline{pi}} = \frac{S_{pi}}{\sqrt{N}} \qquad (6)$$

The degrees of freedom for each parameter is found from

$$v_i = N_i - 1 \qquad (7)$$

and the degrees of freedom for the result if found by weighing the sensitivity and precision error in each parameter.

$$v_r = \frac{S_r^4}{\sum_{i=1}^{j} \left[\frac{(S_{\overline{pi}} \times \theta_i)^4}{v_i} \right]} \qquad (8)$$

The student "t" in Equation 1 is associated with the degrees of freedom in the result.

The precision error terms are easily generated using collected data. The bias error terms are more difficult to quantify. The following conventions were used for this report:

- 5% bias in coal flow rates.
- 20% bias in limestone and FGD flow rates.
- 5% bias in gas flow rates.
- No bias in analytical results unless the result is less than reporting limit. Then one-half the reporting limit is used for both the parameter value and its bias in calculations.

The flow rate bias values are assigned using engineering judgment. No bias is assigned to the analytical results (above the reporting limit) or gas flow rate since a good estimate for magnitude of these terms is unknown. These bias terms may be very large (relative to the mean values of the parameters) and may represent a large amount of unaccounted uncertainty in each result. Analytical bias near the instrument reporting limit may be especially large. Therefore, the uncertainty values calculated for this report should be used with care.

In addition to the assumptions about bias errors referred to above, the calculations also assume that the population distribution of each measurement is normally distributed and that the samples collected reflect the true population.

Also, the uncertainty calculated is only for the average value over the sampling period. The uncertainty does not represent long-term process variations. In other words, the calculated uncertainty does not include a bias term to reflect the fact that the sampled system was probably not operating (and emitting) at conditions equivalent to the average conditions for that system over a longer period (in other words, autocorrelation may be important). An example of the confidence interval calculation is provided below.

Confidence Interval Calculations

Confidence intervals (CIs) were calculated for the mean particulate phase concentrations, the mean vapor phase concentrations, and the total concentrations in all gas streams. In addition, confidence intervals were determined for the stack gas emission factors presented in Table 3-12.

The following example shows an example calculation for the 95% confidence interval around the emission factor. This procedure utilizes the same method outlined earlier in this appendix. The example uses concentration data for mercury in the stack gas.

$$E = \frac{(g * s) + (g * v)}{HHV * Coal} * 2204.6 \qquad (5\text{-}3)$$

where:

g = Gas flow rate, Nm^3/hr;

s = Solid phase conc., $\mu g/Nm^3$;

v = Vapor phase conc., $\mu g/Nm^3$;

HHV = Coal higher heating value, Btu/lb; and

$Coal$ = Coal feed rate, lb/hr.

The values used to calculate the emission factor and the confidence interval are as follows:

	Parameter				
	g Nm^3/hr	s mg/Nm^3	v mg/Nm^3	HHV Btu/lb	Coal Klb/hr
Mean	3,095,000	0.0425	10.647	10,016	620.16
S_p	41,231	0.0232	1.407	120	6.85
$S_{\bar{p}}$	20,616	0.0116	0.703	59.8	3.42
N	4	4	4	4	4
β_p	154,750	0	0	0	31.0
θ	$3.8\text{x}10^{6-}$	1,098	1,098	$-1.16\text{x}10^{5-}$	$-1.87\text{x}10^{5-}$
v_p	3	3	3	3	3

The calculation for the solid phase values is included for reference.

Solid phase analytical: 0.043 μg/Nm^3

0.066 μg/Nm^3

0.011 μg/Nm^3

0.050 μg/Nm^3

$$N = 4$$

$$\text{Mean} = 0.043$$

$$S_p = 0.037$$

$$S_{\bar{p}} = \frac{0.037}{\sqrt{4}} = 0.012$$

As explained above, the β for analytical results is assigned as zero.

$$\beta_p = 0$$

Next, calculate the sensitivity using perturbation method and a 10% perturbation:

$$\theta_i = \frac{r\ (0.047) - r\ (0.043)}{0.0042}$$

$$= 1.098$$

Similar calculations can be done for each parameter.

The precision component is then found by root-sum-squaring the product of the parameter $S_{\bar{p}}$s and their sensitivities.

$$S_r = \sqrt{\left(\theta_g\ S_{\bar{g}}\right)^2 + \left(\theta_s\ S_{\bar{s}}\right)^2 + \left(\theta_v\ S_{\bar{v}}\right)^2 + \left(\theta_{HHV}\ S_{HHV}\right)^2 + \left(\theta_{coal}\ S_{coal}\right)^2}$$

$$S_r = 0.781$$

The bias component is found using the same equation substituting β_p for the $S_{\bar{p}}$ term.

$$\beta_r = \sqrt{\left(\theta_g\ \beta_g\right)^2 + \left(\theta_s\ \beta_s\right)^2 + \left(\theta_v\ \beta_v\right)^2 + \left(\theta_{HHV}\ \beta_{HHV}\right)^2 + \left(\theta_{coal}\ \beta_{coal}\right)^2}$$

$$\beta_r = 0.793$$

The uncertainty in the result is then

$$U_r = \sqrt{\beta_r^2 + \left(t\ x\ S_r\right)^2}$$

The degrees of freedom is found from:

$$v_r = \frac{S_r^4}{\sum_{i=1}^{j} \frac{\left(S_{\bar{pi}}\ \theta_i\right)^4}{v_{pi}}}$$

$$= \frac{0.373}{0.119} = 3$$

Therefore, "t" = 3.182.

$$U_r = \sqrt{(0.793)^2 + (3.182 \times 0.781)^2}$$

$$= 2.6$$

The emission rate is calculated as 11.7 lb/10^{12} Btu.

The value is reported as 11.7 ± 2.6 lb/10^{12} Btu.

APPENDIX F:
QUALITY ASSESSMENT/QUALITY CONTROL RESULTS

This appendix presents the detailed quality assessment/quality control results for the coal, flue gas, limestone, makeup water, and ash samples. The tables included in this section are:

- Table F-1: Summary of Blank Sample Results for Site 20;
- Table F-2: Detailed Blank Sample Results for Site 20;
- Table F-3: Summary of Laboratory Control Spike (LCS) Results for Site 20;
- Table F-4: Detailed Laboratory Control Spike Results for Site 20;
- Table F-5: Summary of Matrix Spike Results for Site 20;
- Table F-6: Detailed Matrix Spike Results for Site 20;
- Table F-7: Summary of Analytical Spike Results for Site 20;
- Table F-8: Detailed Analytical Spike Results for Site 20;
- Table F-9: Coal QA/QC for Site 20;
- Table F-10: Detailed Blank Results for Metals Measured by ICP-MS, Site 20;
- Table F-11: Detailed Spike Results for Metals Measured by ICP-MS, Site 20; and
- Table F-12: Detailed Reference Sample Results for Metals Measured by ICP-MS for Site 20.

The first eight tables (F-1 through F-8) correspond to the flue gas, ash, limestone, and makeup water samples analyzed using ICPES[a], GFAA[b], CVAA[c], IC[d], or SIE[e]. Table F-9 contains QA/QC information for coal analyzed by XRF[f], GFAA[g], DGAA[h], or CVAFS[i]. Finally, Tables F-10, F-11, and F-12 contain information pertaining to the

the determination of metals on flue gas streams by ICP-MS[j]. This information was used to derive the data evaluation summary presented in Section 4.

[a] ICPES = Inductively couple plasma emission spectroscopy.

[b] GFAA = Graphite furnace atomic absorption spectrophotometry.

[c] CVAA = Cold vapor atomic absorption spectrophotometry.

[d] IC = Ion chromatography.

[e] SIE = Ion selective electrode.

[f] XRF = X-Ray fluorescence, measured by Wyoming Analytical Laboratory.

[g] GFAA = Graphite furnace atomic absorption spectrophotometry, measured by Wyoming Analytical Laboratories.

[h] DGAA = Double gold amalgam atomic absorption spectrophotometry, measured by Wyoming Analytical Laboratories.

[i] CVAFS = Cold vapor atomic fluorescence spectroscopy measured by Frontier Geosciences.

[j] ICPES-MS = Inductively coupled plasma emission spectroscopy coupled with mass spectroscopy, measured by Harvard University.

Table F-1
Summary of Blank Sample Results for Site 20

Parameter	No. of Blanks Analyzed	No. of Detects[a]	Range of Compounds Detected[b]	Highest Detection Limit[c]
Laboratory (Method) Blanks - Solids[d]: Metals determined by ICPES[e]				
Aluminum	4	0	NA[f]	135 mg/kg
Antimony	4	0	NA	75.5 mg/kg
Barium	5	0	NA	2.15 mg/kg
Beryllium	4	0	NA	0.61 mg/kg
Boron	2	0	NA	0.90 mg/kg
Calcium	5	0	NA	225 mg/kg
Chromium	4	0	NA	10.5 mg/kg
Cobalt	4	0	NA	14 mg/kg
Copper	4	1	0.00341 (0.00247) mg/kg	10.5 mg/kg
Iron	4	0	NA	309 mg/kg
Magnesium	4	0	NA	92.1 mg/kg
Manganese	4	1	0.00019 (0.000118) mg/kg	2.72 mg/kg
Molybdenum	4	0	NA	14.6 mg/kg
Nickel	4	0	NA	24.4 mg/kg
Phosphorus	3	1	13.3 (7.29) mg/kg	61 mg/kg
Potassium	4	0	NA	1640 mg/kg
Silicon	4	2	0.102 (0.0371) mg/kg 774 (132) mg/kg	132 mg/kg
Sodium	4	3	10.4 (6.12) mg/kg 331 (6.12) mg/kg 330 (61.2) mg/kg	61.2 mg/kg
Vanadium	4	0	NA	15.5 mg/kg
Zinc	4	1	0.00293 (0.00291) mg/kg	2.73 mg/kg
Laboratory (Method) Blanks - Solids: Metals determined by GFAA[g]				
Arsenic	3	1	3.1 (0.933) mg/kg	0.933 mg/kg
Cadmium	2	0	NA	0.374 mg/kg
Lead	3	0	NA	1.1 mg/kg
Selenium	4	0	NA	0.116 mg/kg
Laboratory (Method) Blanks - Solids: Metals determined by CVAA[h]				
Mercury	3	0	NA	0.012 mg/kg
Laboratory (Method) Blanks - Solids: Anions				
Chloride	9	2	0.953 (0.252) mg/kg 0.130 (0.0126) mg/kg	0.63 mg/kg
Fluoride	4	4	0.57 (0.47) mg/kg 3.48 (2.35) mg/kg 2.99 (2.35) mg/kg 0.535 (0.47) mg/kg	2.35 mg/kg
Sulfate	2	0	NA	0.05 mg/kg

Table F-1 (Continued)

Parameter	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Highest Detection Limit
Laboratory (Method) Blanks - Filters[i]: Metals determined by ICPES				
Aluminum	1	0	NA	13.5 µg
Antimony	1	0	NA	7.55 µg
Barium	1	0	NA	0.215 µg
Beryllium	1	0	NA	0.061 µg
Cadmium	1	0	NA	0.10 µg
Calcium	1	0	NA	22.5 µg
Chromium	1	0	NA	1.05 µg
Cobalt	1	0	NA	1.4 µg
Copper	1	0	NA	1.05 µg
Iron	1	0	NA	30.9 µg
Magnesium	1	0	NA	9.21 µg
Manganese	1	0	NA	0.272 µg
Molybdenum	1	0	NA	1.46 µg
Nickel	1	0	NA	2.44 µg
Phosphorus	1	0	NA	7.29 µg
Potassium	1	0	NA	164 µg
Silicon	1	1	114 µg[j]	13.2 µg
Sodium	1	1	10.4 µg	6.12 µg
Vanadium	1	0	NA	1.55 µg
Zinc	1	0	NA	0.273 µg
Laboratory (Method) Blanks - Filters: Metals determined by GFAA				
Arsenic	1	0	NA	0.0933 µg
Cadmium	1	0	NA	0.10 µg
Lead	1	0	NA	0.11 µg
Selenium	1	0	NA	0.116 µg
Laboratory (Method) Blanks - Filters: Metals determined by CVAA				
Mercury	1	1	0.010 µg	0.0096 µg
Laboratory (Method) Blanks - Liquids: Metals determined by ICPES				
Aluminum	1	0	NA	0.0284 mg/L
Antimony	1	0	NA	0.0241 mg/L
Barium	1	0	NA	0.00053 mg/L
Beryllium	1	0	NA	0.000554 mg/L
Boron	1	0	NA	0.015 mg/L
Calcium	1	0	NA	0.148 mg/L
Chromium	1	0	NA	0.000249 mg/kg
Cobalt	1	0	NA	0.0034 mg/L
Copper	1	0	NA	0.00381 mg/L
Iron	1	0	NA	0.00596 mg/L
Magnesium	1	0	NA	0.0228 mg/L
Manganese	1	0	NA	0.000395 mg/L

Table F-1 (Continued)

Parameter	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Highest Detection Limit
Molybdenum	1	0	NA	0.00463 mg/L
Nickel	1	0	NA	0.00986 mg/L
Phosphorus	1	0	NA	0.0061 mg/L
Potassium	1	0	NA	0.00287 mg/L
Silicon	1	1	0.102 (0.0371) mg/L	0.0371 mg/L
Sodium	1	0	NA	0.0397 mg/L
Vanadium	1	0	NA	0.00236 mg/L
Zinc	1	0	NA	0.00153 mg/L
Laboratory (Method) Blanks - Liquids: Metals determined by GFAA				
Arsenic	1	0	NA	0.0933 mg/L
Cadmium	2	0	NA	0.031 mg/L
Lead	1	1	0.00255 (0.00105) mg/L	0.00105 mg/L
Selenium	2	0	NA	0.000843 mg/L
Laboratory (Method) Blanks - Liquids: Metals determined by CVAA				
Mercury	5	0	NA	0.000048 mg/L
Laboratory (Method) Blanks - Liquids: Anions				
Chloride	6	0	NA	0.026 mg/L
Fluoride	4	3	0.037 (0.0235) mg/L 0.0295 (0.0235) mg/L 0.0207 (0.0200) mg/L	0.0235 mg/L
Sulfate	5	0	NA	0.060 mg/L
Field Blanks[k]: Metals determined by ICPES				
Aluminum	3	3	0.104 (0.0284) mg/L 110 (13.5) µg 129 (54) µg	0.0284 mg/L 54µg
Antimony	2	0	NA	0.0241 mg/L 7.55µg
Barium	3	3	0.00812 (0.00053) mg/L 5.48 (0.86) µg 5.09 (0.215) µg	0.00053 mg/L 0.86 µg
Beryllium	2	0	NA	0.000554 mg/L 0.061 µg
Boron	1	1	0.0289 (0.0150) mg/L	0.0150 mg/L
Calcium	2	2	0.536 (0.148) mg/L 163 (22.5) µg	0.148 mg/L 22.5 µg
Chromium	2	1	1.91 (1.05) µg	0.00249 mg/L 1.05 µg
Cobalt	2	0	NA	0.0034 mg/L 1.4 µg
Copper	2	2	0.00061 (0.00381) mg/L 1.76 (1.05) µg	0.000381 mg/L 1.05 µg
Iron	2	2	0.13 (0.000596) mg/L 36.8 (30.9) µg	0.000596 mg/L 30.9 µg

Table F-1 (Continued)

Parameter	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Highest Detection Limit
Magnesium	2	2	0.0301 (0.0228) mg/L 26.8 (9.21) μg	0.0288 mg/L 9.21 μg
Manganese	3	3	0.034 (0.000395) mg/L 0.738 (0.272) μg 1.49 (1.09) μg	0.000395 mg/L 1.09 μg
Molybdenum	3	2	12.9 (5.84) μg 12.8 (1.46) μg	0.00463 mg/L 5.84 μg
Nickel	2	0	NA	0.00986 mg/L 2.44 μg
Phosphorus	2	1	0.0918 (0.0610) mg/L	0.610 mg/L 7.29 μg
Potassium	2	0	NA	0.00287 mg/L 7.29 μg
Silicon	2	2	0.541 (0.0371) mg/L 151000 (52.8) μg	0.0371 mg/L 52.8 μg
Sodium	3	3	0.468 (0.0397) mg/L 252 (6.12) μg 283 (24.5) μg	0.0397 mg/L 24.5 μg
Vanadium	2	0	NA	0.00236 mg/L 1.155 μg
Zinc	3	3	0.0333 (0.00153) mg/L 6.09 (0.273) μg 11.3 (1.09) μg	0.00153 mg/L 1.09 μg
Field Blanks: Metals determined by GFAA				
Arsenic	2	0	NA	0.000657 mg/L 0.0933 μg
Cadmium	2	2	0.00034 (0.00031) mg/L 0.114 (0.100) μg	0.00031 ,g/L 0.100 μg
Lead	2	2	0.00524 (0.00105) mg/L 0.300 (0.110) μg	0.00105 mg/L 0.110 μg
Selenium	2	2	0.0112 (0.000843) mg/L 1.05 (0.116) μg	0.000843 mg/L 0.116 μg
Field Blanks: Metals determined by CVAA				
Mercury	2	2	0.0015 (0.00048) mg/L 0.018 (0.0096) μg	0.00048 mg/L 0.0096 μg
Field Blanks[1]: Anions				
Chloride	6	4	0.041 - 1.03 mg/L	0.0200 mg/L
Fluoride	6	6	0.0383 - 0.101 mg/L	0.0235 mg/L
Sulfate	7	3	2.51 - 5.96 mg/L	0.0600 mg/L

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a. Only those compounds detected above the detection limit are reported in this summary table. Table F-2 (Detailed Blank Sample Results for Site 20) contain all blank results.

b. Analyte concentration detected in the sample is shown followed by the corresponding detection limit for that sample (in parentheses).

c. Detection Limit = Method detection limit as defined in 40 CFR, Part 136, Appendix B, multiplied by a sample specific dilution and digestion factor. As a result of multiplication by the sample specific dilution/digestion factor, there may be a range of detection limits for a single analyte for a single matrix. The highest detection limit is shown here. Table F-2 (Detailed Blank Sample Results for Site 20) contains the detection limit for each blank sample.

d. These are actually liquid samples of the digestion fluid. They are reported here in mg/kg units so that they can be easily compared to the corresponding solid samples that they were batched with.

e. ICPES = Inductively coupled plasma emissions spectroscopy.

f. NA = Not applicable. This term is used when there were no "hits" for that analyte in that matrix.

g. GFAA = Graphite furnace atomic absorption spectrophotometry.

h. CVAA = Cold vapor atomic absorption spectrophotometry.

i. These are filter samples that are digested and analyzed.

j. Silicon contamination on filters is common.

k. Field blanks with units in mg/L correspond to nitric acid impinger fractions. Field blanks with units in μg correspond to nitric and acetone probe and nozzle rinse + filter fractions. For these entries, the largest detection limit for each type of blank is listed.

l. All field blanks for the anions train were carbonate impinger solution.

Table F-2
Detailed Blank Sample Results for Site 20

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Aluminum	W	mg/L	0.00034J	0.0284
Method Blank	Aluminum	S	μg	3.49J	13.5
Method Blank	Aluminum	S	mg/kg	0.0385J	0.0733
Method Blank	Aluminum	S	mg/kg	11.9J	135
Method Blank	Aluminum	S	mg/kg	25.8J	135
Method Blank	Aluminum	S	mg/kg	ND	7.33
Method Blank	Antimony	S	mg/kg	ND	1.93
Method Blank	Antimony	S	mg/kg	ND	75.5
Method Blank	Antimony	S	mg/kg	0.00043J	0.0193
Method Blank	Antimony	S	mg/kg	ND	75.5
Method Blank	Antimony	W	mg/L	0.00091J	0.0241
Method Blank	Antimony	S	μg	ND	7.55
Method Blank	Arsenic	S	mg/kg	ND	0.0933
Method Blank	Arsenic	W	mg/L	ND	0.000657
Method Blank	Arsenic	S	mg/kg	ND	0.0933
Method Blank	Arsenic	S	mg/L	ND	0.0933
Method Blank	Arsenic	S	mg/kg	3.1	0.933
Method Blank	Arsenic	S	μg	ND	0.0933
Method Blank	Barium	S	mg/kg	0.00012J	0.000579
Method Blank	Barium	S	mg/kg	0.31J	2.15
Method Blank	Barium	S	mg/kg	0.21J	2.15
Method Blank	Barium	S	mg/kg	ND	0.0579
Method Blank	Barium	W	mg/L	ND	0.00053
Method Blank	Barium	S	μg	0.062J	0.215
Method Blank	Barium	S	mg/kg	0J	0.666
Method Blank	Beryllium	W	mg/L	0.00007J	0.000554
Method Blank	Beryllium	S	mg/kg	ND	0.0589
Method Blank	Beryllium	S	mg/kg	ND	0.61
Method Blank	Beryllium	S	mg/kg	ND	0.61
Method Blank	Beryllium	S	mg/kg	0.00026J	0.000589
Method Blank	Beryllium	S	μg	ND	0.061
Method Blank	Boron	W	mg/L	0.0131J	0.015
Method Blank	Boron	S	mg/kg	0.00653J	0.009

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Boron	S	mg/kg	0.0274J	0.9
Method Blank	Cadmium	S	mg/kg	ND	0.374
Method Blank	Cadmium	T	μg	ND	0.1
Method Blank	Cadmium	S	mg/kg	0.01J	0.0374
Method Blank	Cadmium	W	mg/L	0.00019J	0.00031
Method Blank	Cadmium	W	mg/L	ND	0.031
Method Blank	Calcium	S	mg/kg	0.0236J	23.8
Method Blank	Calcium	S	mg/kg	13.7J	225
Method Blank	Calcium	S	mg/kg	0.0553J	0.238
Method Blank	Calcium	W	mg/L	0.0227J	0.148
Method Blank	Calcium	S	mg/kg	6.47J	225
Method Blank	Calcium	S	μg	0.803J	22.5
Method Blank	Calcium	S	mg/kg	11.2J	25.9
Method Blank	Chloride	S	mg/kg	ND	0.252
Method Blank	Chloride	S	mg/kg	ND	0.0126
Method Blank	Chloride	S	mg/kg	0.953@	0.252
Method Blank	Chloride	W	mg/L	ND	0.0126
Method Blank	Chloride	W	mg/L	ND	0.0126
Method Blank	Chloride	S	mg/kg	ND	0.63
Method Blank	Chloride	W	mg/L	ND	0.026
Method Blank	Chloride	S	mg/kg	0.00296J	0.0126
Method Blank	Chloride	W	mg/L	ND	0.026
Method Blank	Chloride	W	mg/L	ND	0.0126
Method Blank	Chloride	W	mg/L	ND	0.02
Method Blank	Chloride	S	mg/kg	0.13	0.0126
Method Blank	Chromium	S	μg	0.433J	1.05
Method Blank	Chromium	S	mg/kg	3.34J	10.5
Method Blank	Chromium	W	mg/L	ND	0.00249
Method Blank	Chromium	S	mg/kg	2.01J	10.5
Method Blank	Chromium	S	mg/kg	0.00129J	0.00273
Method Blank	Chromium	S	mg/kg	0.00424J	0.273
Method Blank	Cobalt	S	mg/kg	ND	0.00522
Method Blank	Cobalt	S	mg/kg	0.00043J	0.522
Method Blank	Cobalt	W	mg/L	0.00022J	0.0034
Method Blank	Cobalt	S	mg/kg	1.16J	14

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Cobalt	S	mg/kg	1.62J	14
Method Blank	Cobalt	S	µg	0.306J	1.4
Method Blank	Copper	S	mg/kg	ND	10.5
Method Blank	Copper	W	mg/L	0.00148J	0.00381
Method Blank	Copper	S	mg/kg	0.00111J	0.247
Method Blank	Copper	S	mg/kg	0.00341@	0.00247
Method Blank	Copper	S	µg	0.579J	1.05
Method Blank	Copper	S	mg/kg	0.88J	10.5
Method Blank	Fluoride	S	mg/kg	0.57@	0.47
Method Blank	Fluoride	W	mg/L	0.037@	0.0235
Method Blank	Fluoride	W	mg/kg	3.48@	2.35
Method Blank	Fluoride	S	mg/kg	2.99@	2.35
Method Blank	Fluoride	S	mg/kg	0.535@	0.47
Method Blank	Fluoride	W	mg/L	0.0295@	0.0235
Method Blank	Fluoride	W	mg/L	0.0194J	0.0235
Method Blank	Fluoride	W	mg/L	0.0207@	0.0200
Method Blank	Iron	S	µg	ND	30.9
Method Blank	Iron	S	mg/kg	0.0226J	31.1
Method Blank	Iron	W	mg/L	0.00116J	0.00596
Method Blank	Iron	S	mg/kg	0.0371J	0.311
Method Blank	Lead	W	mg/L	0.00255@	0.00105
Method Blank	Lead	S	mg/kg	ND	1.1
Method Blank	Lead	S	mg/kg	ND	0.11
Method Blank	Lead	S	µg	0.05J	0.11
Method Blank	Lead	S	mg/kg	ND	0.11
Method Blank	Magnesium	S	mg/kg	0.00568J	0.0273
Method Blank	Magnesium	S	µg	2.6J	9.21
Method Blank	Magnesium	S	mg/kg	0.00019J	2.73
Method Blank	Magnesium	W	mg/L	0.00094J	0.0228
Method Blank	Magnesium	S	mg/kg	10.1J	92.1
Method Blank	Magnesium	S	mg/kg	21.7J	92.1
Method Blank	Manganese	S	µg	0.015J	0.272
Method Blank	Manganese	S	mg/kg	0.00019@	0.000118
Method Blank	Manganese	S	mg/kg	ND	0.0118

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Manganese	S	mg/kg	ND	2.72
Method Blank	Manganese	S	mg/kg	ND	2.72
Method Blank	Manganese	W	mg/L	ND	0.000395
Method Blank	Mercury	W	mg/L	ND	0.000048
Method Blank	Mercury	W	mg/L	ND	0.000048
Method Blank	Mercury	W	mg/L	ND	0.000048
Method Blank	Mercury	W	μg	0.0100@	0.0096
Method Blank	Mercury	S	mg/kg	ND	0.012
Method Blank	Mercury	S	mg/kg	ND	0.012
Method Blank	Mercury	S	mg/kg	ND	0.012
Method Blank	Molybdenum	S	mg/kg	0.00114J	0.00262
Method Blank	Molybdenum	S	mg/kg	ND	14.6
Method Blank	Molybdenum	S	mg/kg	ND	0.262
Method Blank	Molybdenum	S	μg	ND	1.46
Method Blank	Molybdenum	W	mg/L	0.00154J	0.00463
Method Blank	Molybdenum	S	mg/kg	ND	14.6
Method Blank	Nickel	S	mg/kg	ND	0.0109
Method Blank	Nickel	S	mg/kg	6J	24.4
Method Blank	Nickel	S	mg/kg	ND	1.09
Method Blank	Nickel	S	mg/kg	ND	24.4
Method Blank	Nickel	S	μg	0.172J	2.44
Method Blank	Nickel	W	mg/L	0.00662J	0.00986
Method Blank	Phosphorus	S	mg/kg	13.3@	7.29
Method Blank	Phosphorus	S	mg/kg	ND	7.29
Method Blank	Phosphorus	S	mg/kg	ND	61
Method Blank	Phosphorus	S	μg	ND	7.29
Method Blank	Phosphorus	W	mg/L	0.0705@	0.061
Method Blank	Potassium	S	μg	22J	164
Method Blank	Potassium	S	mg/kg	38.6J	1,640
Method Blank	Potassium	S	mg/kg	0.246J	34.6
Method Blank	Potassium	S	mg/kg	82.8J	1,640
Method Blank	Potassium	S	mg/kg	ND	0.346
Method Blank	Potassium	W	mg/L	ND	0.00287
Method Blank	Selenium	S	mg/kg	ND	0.0706

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Selenium	S	mg/kg	ND	0.116
Method Blank	Selenium	S	µg	ND	0.116
Method Blank	Selenium	W	mg/L	ND	0.000843
Method Blank	Selenium	W	mg/L	ND	0.000843
Method Blank	Selenium	S	mg/kg	ND	0.706
Method Blank	Silicon	S	mg/kg	0.107J	0.11
Method Blank	Silicon	W	mg/L	0.102@	0.0371
Method Blank	Silicon	S	mg/kg	0.0535J	11
Method Blank	Silicon	S	µg	114	13.2
Method Blank	Silicon	S	mg/kg	13.5J	16.4
Method Blank	Silicon	W	mg/L	0.102@	0.0371
Method Blank	Silicon	S	mg/kg	774	132
Method Blank	Silicon	S	mg/kg	1,020	132
Method Blank	Sodium	S	mg/kg	ND	2.59
Method Blank	Sodium	S	mg/kg	330	61.2
Method Blank	Sodium	W	mg/L	ND	0.0397
Method Blank	Sodium	S	mg/kg	ND	0.0259
Method Blank	Sodium	S	µg	10.4@	6.12
Method Blank	Sodium	S	mg/kg	331	61.2
Method Blank	Sulfate	W	mg/kg	ND	2.5
Method Blank	Sulfate	W	mg/L	ND	0.05
Method Blank	Sulfate	W	mg/kg	ND	0.05
Method Blank	Sulfate	W	mg/L	ND	0.05
Method Blank	Sulfate	W	mg/L	ND	0.05
Method Blank	Sulfate	W	mg/L	ND	0.06
Method Blank	Sulfate	W	mg/L	ND	0.06
Method Blank	Vanadium	S	mg/kg	0.00092J	0.43
Method Blank	Vanadium	S	mg/kg	1.48J	15.5
Method Blank	Vanadium	S	mg/kg	1.71J	15.5
Method Blank	Vanadium	S	mg/kg	0.00053J	0.0043
Method Blank	Vanadium	W	mg/L	ND	0.00236
Method Blank	Vanadium	S	µg	0.325J	1.55
Method Blank	Zinc	S	µg	ND	0.273
Method Blank	Zinc	S	mg/kg	0.00293@	0.00291
Method Blank	Zinc	S	mg/kg	0.0012J	0.291

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Method Blank	Zinc	S	mg/kg	2.1J	2.73
Method Blank	Zinc	S	mg/kg	1.61J	2.73
Method Blank	Zinc	W	mg/L	0.00001J	0.00153
Nitric Impinger	Aluminum	W	mg/L	0.104@	0.0284
Nitric & Ace PNR & Filter	Aluminum	S	μg	110	13.5
Nitric & Ace PNR & Filter	Aluminum	S	μg	129@	54
Nitric Impinger	Antimony	W	mg/L	ND	0.0241
Nitric & Ace PNR & Filter	Antimony	S	μg	ND	7.55
Nitric Impinger	Arsenic	W	mg/L	ND	0.000657
Nitric & Ace PNR	Arsenic	S	μg	ND	0.0933
Nitric Impinger	Barium	W	mg/L	0.00821	0.00053
Nitric & Ace PNR & Filter	Barium	S	μg	5.48	0.86
Nitric & Ace PNR & Filter	Barium	S	μg	5.09	0.215
Nitric Impinger	Beryllium	W	mg/L	0.00006J	0.000554
Nitric & Ace PNR & Filter	Beryllium	S	μg	0.008J	0.061
Nitric Impinger	Boron	W	mg/L	0.0289@	0.015
Nitric Impinger	Cadmium	W	mg/L	0.00034@	0.00031
Nitric & Ace PNR	Cadmium	S	μg	0.114@	0.100
Nitric Impinger	Calcium	W	mg/L	0.536@	0.148
Nitric & Ace PNR & Filter	Calcium	S	μg	163	22.5
Carbonate Impinger 1	Chloride	W	mg/L	0.179	0.0126
Carbonate Impinger 2	Chloride	W	mg/L	0.0676	0.0126
Carbonate & Ace PNR	Chloride	W	mg/L	1.03	0.020
Reagent Blank, Anions Impingers	Chloride	W	mg/L	ND	0.0126
Reagent Blank, Anions Impingers	Chloride	W	mg/L	0.041@	0.0126
Reagent Blank, Anions Impingers	Chloride	W	mg/L	ND	0.0126
Nitric Impinger	Chromium	W	mg/L	0.00116J	0.00249
Nitric & Ace PNR & Filter	Chromium	S	μg	1.91@	1.05
Nitric Impinger	Cobalt	W	mg/L	ND	0.0034
Nitric & Ace PNR & Filter	Cobalt	S	μg	0.177J	1.4
Nitric Impinger	Copper	W	mg/L	0.0061@	0.00381
Nitric & Ace PNR & Filter	Copper	S	μg	1.76@	1.05
Carbonate Impinger 1	Fluoride	W	mg/L	0.101@	0.0235
Carbonate & Ace PNR	Fluoride	W	mg/L	0.053@	0.0235

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Reagent Blank, Anions Impingers	Fluoride	W	mg/L	0.0383@	0.0235
Reagent Blank, Anions Impingers	Fluoride	W	mg/L	0.0658@	0.0235
Reagent Blank, Anions Impingers	Fluoride	W	mg/L	0.0535@	0.0235
Reagent Blank, Anions Impingers	Fluoride	W	mg/L	0.0394@	0.0235
Nitric Impinger	Iron	W	mg/L	0.13	0.00596
Nitric & Ace PNR & Filter	Iron	S	μg	36.8@	30.9
Nitric Impinger	Lead	W	mg/L	0.00524@	0.00105
Nitric & Ace PNR	Lead	S	μg	0.3@	0.11
Nitric Impinger	Magnesium	W	mg/L	0.0301@	0.0228
Nitric & Ace PNR & Filter	Magnesium	S	μg	26.8@	9.21
Nitric Impinger	Manganese	W	mg/L	0.034	0.000395
Nitric & Ace PNR & Filter	Manganese	S	μg	0.738@	0.272
Nitric & Ace PNR & Filter	Manganese	S	μg	1.49@	1.09
Nitric Impinger	Mercury	W	mg/L	0.0015	0.00048
Nitric & Ace PNR	Mercury	W	μg	0.018@	0.0096
Nitric Impinger	Molybdenum	W	mg/L	0.00254J	0.00463
Nitric & Ace PNR & Filter	Molybdenum	S	μg	12.9@	5.84
Nitric & Ace PNR & Filter	Molybdenum	S	μg	12.8	1.46
Nitric Impinger	Nickel	W	mg/L	0.00267J	0.00986
Nitric & Ace PNR & Filter	Nickel	S	μg	1.27J	2.44
Nitric Impinger	Phosphorus	W	mg/L	0.0918@	0.061
Nitric & Ace PNR & Filter	Phosphorus	S	μg	ND	7.29
Nitric Impinger	Potassium	W	mg/L	ND	0.00287
Nitric & Ace PNR & Filter	Potassium	S	μg	16.6J	164
Nitric Impinger	Selenium	W	mg/L	0.0112	0.000843
Nitric & Ace PNR	Selenium	S	μg	1.05	0.116
Nitric Impinger	Silicon	W	mg/L	0.541	0.0371
Nitric & Ace PNR & Filter	Silicon	S	μg	151,000	52.8
Nitric Impinger	Silver	W	mg/L	0.00195J	0.00492
Nitric & Ace PNR & Filter	Silver	S	μg	ND	1.12
Nitric Impinger	Sodium	W	mg/L	0.468	0.0397
Nitric & Ace PNR & Filter	Sodium	S	μg	252	6.12
Nitric & Ace PNR & Filter	Sodium	S	μg	283	24.5
Carbonate Impinger 1	Sulfate	W	mg/L	5.96	0.06

Table F-2 (Continued)

Sample Type	Analyte	Matrix	Units	Result	Detection Limit
Carbonate Impinger 2	Sulfate	W	mg/L	5.96	0.06
Carbonate & Ace PNR	Sulfate	W	mg/L	2.51	0.06
Reagent Blank, Anions Impingers	Sulfate	W	mg/L	ND	0.06
Reagent Blank, Anions Impingers	Sulfate	W	mg/L	ND	0.06
Reagent Blank, Anions Impingers	Sulfate	W	mg/L	ND	0.06
Reagent Blank, anions Impingers	Sulfate	W	mg/L	ND	0.06
Nitric Impinger	Vanadium	W	mg/L	0.00088J	0.00236
Nitric & Ace PNR & Filter	Vanadium	S	μg	0.581J	1.55
Nitric Impinger	Zinc	W	mg/L	0.0333	0.00153
Nitric & Ace PNR & Filter	Zinc	S	μg	6.09	0.273
Nitric & Ace PNR & Filter	Zinc	S	μg	11.3	1.09

ND = Not detected at stated concentration.

J = Results less than detection limit.

S = Solid

W = Water

T = Train

@ = Concentration is less than five times detection limit.

Table F-3
Summary of Laboratory Control Spike (LCS) Results for Site 20

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO[a] for Recovery
Solid (ERA Soil[b]): Metals Determined by ICPES[c]						
Aluminum	4	102	7	0	0	75-125%
Antimony	4	129	5	0	2	75-125%
Barium	4	91	2	0	0	75-125%
Beryllium	4	93	2	0	0	75-125%
Calcium	6	95	2	0	0	75-125%
Chromium	4	108	4	0	2	75-125%
Cobalt	4	91	2	0	0	75-125%
Copper	4	94	3	0	0	75-125%
Iron	4	107	6	0	0	75-125%
Magnesium	4	92	2	0	0	75-125%
Manganese	4	92	1	0	0	75-125%
Molybdenum	4	97	4	0	0	75-125%
Nickel	4	96	2	0	0	75-125%
Phosphorus						75-125%
Potassium	4	95	4	0	0	75-125%
Silicon						75-125%
Sodium	4	110	2	0	2	75-125%
Vanadium	4	102	2	0	0	75-125%
Zinc	4	97	9	0	0	75-125%
Solids (ERA Soil): Metals Determined by GFAA[d]						
Arsenic	4	110	3	0	0	75-125%
Cadmium	4	107	3	0	0	75-125%
Lead	4	93	1	0	0	75-125%
Selenium	6	106	2	0	0	75-125%
Solids (ERA Soil): Metals Determined by CVAA[e]						
Mercury	4	95	3	0	0	75-125%
Solid (NBS 1633A[f]): Metals Determined by ICPES						
Aluminum	8	77	17	0	0	75-125%
Barium	10	77	4	0	0	75-125%

Table F-3 (Continued)

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO for Recovery
Beryllium	8	98	1	0	0	75-125%
Calcium	8	86	10	0	0	75-125%
Chromium	8	91	2	0	0	75-125%
Cobalt	8	98	20	0	1	75-125%
Copper	8	94	2	0	0	75-125%
Iron	8	88	2	0	0	75-125%
Magnesium	8	77	21	0	0	75-125%
Manganese	8	90	5	0	0	75-125%
Nickel	8	108	4	0	0	75-125%
Potassium	8	85	2	0	0	75-125%
Silicon	8	94	8	0	0	75-125%
Sodium	8	111	6	0	2	75-125%
Vanadium	8	93	1	0	0	75-125%
Zinc	8	100	1	0	0	75-125%
Solids (NBS 1633A): Metals Determined by GFAA						
Arsenic	4	112	2	0	0	75-125%
Cadmium	4	28	84	4	0	75-125%
Lead	6	77	1	4	0	75-125%
Selenium	4	81	5	1	0	75-125%
Solids (NBS 1633A): Metals Determined by CVAA						
Mercury	6	105	3	0	0	75-125%
Gas Metals Nitric Impingers: Metals Determined by ICPES						
Aluminum	10	93	1	0	0	75-125%
Antimony	10	89	4	0	0	75-125%
Barium	12	95	1	0	0	75-125%125%
Beryllium	10	92	1	0	0	75-125%
Boron	6	99	2	0	0	75-125%
Calcium	12	94	1	0	0	75-125%
Chromium	10	94	1	0	0	75-125%
Cobalt	10	92	1	0	0	75-125%
Copper	10	93	1	0	0	75-125%
Iron	10	91	1	0	0	75-125%

Table F-3 (Continued)

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO for Recovery
Magnesium	10	92	1	0	0	75-125%
Manganese	10	92	1	0	0	75-125%
Molybdenum	10	92	2	0	0	75-125%
Nickel	10	93	1	0	0	75-125%
Phosphorous	10	98	2	0	0	75-125%
Potassium	10	91	2	0	0	75-125%
Silicon	12	97	1	0	0	75-125%
Sodium	10	94	1	0	0	75-125%
Vanadium	10	94	1	0	0	75-125%
Zinc	10	92	1	0	0	75-125%
Cadmium	10	108	3	0	0	75-125%
Lead	10	103	2	0	0	75-125%
Selenium	12	94	1	0	0	75-125%
Gas Metals Nitric Impingers: Metals determined by CVAA						
Mercury	12	108	2	0	0	75-125%
Gas Metals Nitric Impingers: Anions						
Chloride	26	101	4	0	0	80-120%
Fluoride	16	95	3	0	0	80-120%
Sulfate	12	95	5	0	0	80-120%

[a] DQO = Data quality objective.

[b] ERA soil is a standard soil which has been spiked with an appropriate concentration of the designated analyte. The soil is then digested and analyzed per the method.

[c] ICPES = Inductively coupled plasma emission spectroscopy.

[d] GFAA = Graphite furnace atomic absorption spectrophotometry.

[e] CVAA = Cold vapor atomic absorption spectrophotometry.

[f] NBS 1633A is a standard fly ash (National Bureau of Standards standard reference coal fly ash). Recoveries are based on the comparison of analyzed concentration to certified value.

Table F-4
Laboratory Control Spike Results (%)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Arsenic	GFAA		ERA	110	111	111	1
Arsenic	GFAA		Lab Control Sample	91	93	92	2
Chloride	IC		Lab Control Sample	97	98	98	1
Chloride	IC		Lab Control Sample	100	100	100	0
Fluoride	SIE		Lab Control Sample	96	97	97	1
Mercury	CVAA		Lab Control Sample	102	103	103	1
Chloride	IC	Gas Anions Impingers	Lab Control Sample	95	97	96	2
Chloride	IC	Gas Anions Impingers	Lab Control Sample	97	108	103	11
Chloride	IC	Gas Anions Impingers	Lab Control Sample	99	102	101	3
Fluoride	SIE	Gas Anions Impingers	Lab Control Sample	96	98	97	2
Fluoride	SIE	Gas Anions Impingers	Lab Control Sample	94	102	98	8
Sulfate	IC	Gas Anions Impingers	Lab Control Sample	97	97	97	0
Chloride	IC	Gas Anions Solids	Lab Control Sample	92	105	99	13
Chloride	IC	Gas Anions Solids	Lab Control Sample	101	102	102	1
Fluoride	SIE	Gas Anions Solids	Lab Control Sample	100	104	102	4
Fluoride	SIE	Gas Anions Solids	Lab Control Sample	100	100	100	0
Sulfate	IC	Gas Anions Solids	Lab Control Sample	99	102	100	3
Sulfate	IC	Gas Anions Solids	Lab Control Sample	79	100	90	24
Sulfate	IC	Gas Anions Solids	Lab Control Sample	90	91	90	0
Sulfate	IC	Gas Anions Solids	Lab Control Sample	96	96	96	0
Sulfate	IC	Gas Anions Solids	Lab Control Sample	100	100	100	0
Aluminum	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	97	98	98	1
Antimony	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	96	98	97	2
Arsenic	GFAA	Gas Metals Nitric Impingers	Lab Control Sample	97	100	98	3
Barium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	100	100	100	0
Beryllium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	102	102	102	0
Boron	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	103	105	104	2
Cadmium	GFAA	Gas Metals Nitric Impingers	Lab Control Sample	108	108	108	0
Calcium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	100	101	101	1
Chromium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	100	101	101	1
Cobalt	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	98	99	98	1
Copper	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	99	100	99	1
Iron	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	98	98	98	1
Lead	GFAA	Gas Metals Nitric Impingers	Lab Control Sample	112	116	114	3
Magnesium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	97	98	98	1
Manganese	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	99	99	99	1
Mercury	CVAA	Gas Metals Nitric Impingers	Lab Control Sample	104	106	105	2
Mercury	CVAA	Gas Metals Nitric Impingers	Lab Control Sample	102	102	102	0
Molybdenum	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	97	98	98	1
Nickel	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	100	100	100	0
Phosphorus	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	98	100	99	2

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Potassium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	95	98	96	4
Selenium	GFAA	Gas Metals Nitric Impingers	Lab Control Sample	99	103	101	4
Selenium	GFAA	Gas Metals Nitric Impingers	Lab Control Sample	95	96	96	1
Silicon	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	105	106	106	1
Sodium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	98	99	99	2
Vanadium	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	101	102	102	1
Zinc	ICAP	Gas Metals Nitric Impingers	Lab Control Sample	97	98	97	1
Mercury	CVAA	Gas Metals Permanganate Impingers	Lab Control Sample	109	112	111	3
Mercury	CVAA	Gas Metals Permanganate Impingers	Lab Control Sample	102	104	103	2
Aluminum	ICAP	Solids	ERA	102	108	105	6
Aluminum	ICAP	Solids	ERA	94	103	99	8
Aluminum	ICAP	Solids	Lab Control Sample	94	94	94	0
Aluminum	ICAP	Solids	Lab Control Sample	95	95	95	1
Aluminum	ICAP	Solids	Lab Control Sample	90	90	90	0
Aluminum	ICAP	Solids	Lab Control Sample	93	93	93	0
Aluminum	ICAP	Solids	Lab Control Sample	87	89	88	3
Aluminum	ICAP	Solids	NBS 1633A	76	93	84	21
Aluminum	ICAP	Solids	NBS 1633A	76	90	83	17
Aluminum	ICAP	Solids	NBS 1633A	68	78	73	14
Aluminum	ICAP	Solids	NBS 1633A	63	73	68	14
Antimony	ICAP	Solids	ERA	127	138	133	8
Antimony	ICAP	Solids	ERA	124	125	124	1
Antimony	ICAP	Solids	Lab Control Sample	87	89	88	2
Antimony	ICAP	Solids	Lab Control Sample	89	91	90	2
Antimony	ICAP	Solids	Lab Control Sample	87	89	88	3
Antimony	ICAP	Solids	Lab Control Sample	82	82	82	0
Antimony	ICAP	Solids	Lab Control Sample	76	85	81	12
Antimony	ICAP	Solids	NBS 1633A				
Antimony	ICAP	Solids	NBS 1633A				
Arsenic	GFAA	Solids	ERA	102	106	104	4
Arsenic	GFAA	Solids	ERA	112	117	114	4
Arsenic	GFAA	Solids	Lab Control Sample	116	116	116	1
Arsenic	GFAA	Solids	Lab Control Sample	91	92	91	1
Arsenic	GFAA	Solids	Lab Control Sample	93	93	93	1
Arsenic	GFAA	Solids	Lab Control Sample	123	124	123	1
Arsenic	GFAA	Solids	NBS 1633A	102	103	102	1
Arsenic	GFAA	Solids	NBS 1633A	119	123	121	3
Arsenic	ICAP	Solids	Lab Control Sample	116	116	116	1
Arsenic	ICAP	Solids	NBS 1633A	102	103	102	1

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Barium	ICAP	Solids	ERA	90	93	91	2
Barium	ICAP	Solids	ERA	90	92	91	2
Barium	ICAP	Solids	Lab Control Sample	97	97	97	0
Barium	ICAP	Solids	Lab Control Sample	93	93	93	0
Barium	ICAP	Solids	Lab Control Sample	98	98	98	0
Barium	ICAP	Solids	Lab Control Sample	95	96	95	1
Barium	ICAP	Solids	Lab Control Sample	90	90	90	0
Barium	ICAP	Solids	Lab Control Sample	85	87	86	3
Barium	ICAP	Solids	NBS 1633A	77	81	79	5
Barium	ICAP	Solids	NBS 1633A	81	83	82	2
Barium	ICAP	Solids	NBS 1633A	83	83	83	0
Barium	ICAP	Solids	NBS 1633A	71	77	74	7
Barium	ICAP	Solids	NBS 1633A	66	70	68	6
Beryllium	ICAP	Solids	ERA	93	94	94	1
Beryllium	ICAP	Solids	ERA	91	94	93	3
Beryllium	ICAP	Solids	Lab Control Sample	92	92	92	1
Beryllium	ICAP	Solids	Lab Control Sample	94	94	94	0
Beryllium	ICAP	Solids	Lab Control Sample	88	88	88	1
Beryllium	ICAP	Solids	Lab Control Sample	84	84	84	0
Beryllium	ICAP	Solids	Lab Control Sample	82	83	83	2
Beryllium	ICAP	Solids	NBS 1633A	109	109	109	0
Beryllium	ICAP	Solids	NBS 1633A	97	97	97	0
Beryllium	ICAP	Solids	NBS 1633A	97	103	100	6
Beryllium	ICAP	Solids	NBS 1633A	88	88	88	0
Boron	ICAP	Solids	ERA				
Boron	ICAP	Solids	Lab Control Sample	97	99	98	2
Boron	ICAP	Solids	Lab Control Sample	95	96	95	1
Cadmium	GFAA	Solids	ERA	109	113	111	3
Cadmium	GFAA	Solids	ERA	101	104	102	3
Cadmium	GFAA	Solids	Lab Control Sample	99	102	100	3
Cadmium	GFAA	Solids	Lab Control Sample	112	120	116	7
Cadmium	GFAA	Solids	Lab Control Sample	111	112	112	1
Cadmium	GFAA	Solids	Lab Control Sample	101	105	103	4
Cadmium	GFAA	Solids	NBS 1633A	3	21	12	151
Cadmium	GFAA	Solids	NBS 1633A	40	47	43	16
Calcium	ICAP	Solids	ERA	93	94	93	2
Calcium	ICAP	Solids	ERA	95	98	96	3
Calcium	ICAP	Solids	ERA	96	97	97	1
Calcium	ICAP	Solids	Lab Control Sample	91	92	91	1
Calcium	ICAP	Solids	Lab Control Sample	95	95	95	0
Calcium	ICAP	Solids	Lab Control Sample	95	95	95	1

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Calcium	ICAP	Solids	Lab Control Sample	91	92	92	1
Calcium	ICAP	Solids	Lab Control Sample	96	96	96	0
Calcium	ICAP	Solids	Lab Control Sample	91	94	92	4
Calcium	ICAP	Solids	NBS 1633A	93	102	97	9
Calcium	ICAP	Solids	NBS 1633A	88	95	92	9
Calcium	ICAP	Solids	NBS 1633A	76	86	81	12
Calcium	ICAP	Solids	NBS 1633A	70	78	74	11
Chloride	IC	Solids	Lab Control Sample	101	109	105	7
Chloride	IC	Solids	Lab Control Sample	101	109	105	7
Chloride	IC	Solids	Lab Control Sample	101	103	102	3
Chloride	IC	Solids	Lab Control Sample				
Chloride	IC	Solids	Lab Control Sample	102	105	104	2
Chloride	IC	Solids	Lab Control Sample	100	101	101	1
Chloride	IC	Solids	Lab Control Sample	98	103	101	5
Chromium	ICAP	Solids	ERA	130	135	132	4
Chromium	ICAP	Solids	ERA	83	85	84	3
Chromium	ICAP	Solids	Lab Control Sample	92	93	93	1
Chromium	ICAP	Solids	Lab Control Sample	95	96	96	1
Chromium	ICAP	Solids	Lab Control Sample	93	94	93	0
Chromium	ICAP	Solids	Lab Control Sample	89	89	89	0
Chromium	ICAP	Solids	Lab Control Sample	86	88	87	2
Chromium	ICAP	Solids	NBS 1633A	94	96	95	2
Chromium	ICAP	Solids	NBS 1633A	90	92	91	2
Chromium	ICAP	Solids	NBS 1633A	93	94	94	1
Chromium	ICAP	Solids	NBS 1633A	84	85	85	1
Cobalt	ICAP	Solids	ERA	90	91	91	1
Cobalt	ICAP	Solids	ERA	89	92	91	3
Cobalt	ICAP	Solids	Lab Control Sample	90	90	90	0
Cobalt	ICAP	Solids	Lab Control Sample	94	94	94	0
Cobalt	ICAP	Solids	Lab Control Sample	92	92	92	1
Cobalt	ICAP	Solids	Lab Control Sample	87	87	87	0
Cobalt	ICAP	Solids	Lab Control Sample	84	86	85	2
Cobalt	ICAP	Solids	NBS 1633A	94	139	116	39
Cobalt	ICAP	Solids	NBS 1633A	90	97	93	7
Cobalt	ICAP	Solids	NBS 1633A	82	107	94	26
Cobalt	ICAP	Solids	NBS 1633A	84	92	88	8
Copper	ICAP	Solids	ERA	99	100	100	1
Copper	ICAP	Solids	ERA	87	90	88	4
Copper	ICAP	Solids	Lab Control Sample	91	91	91	0
Copper	ICAP	Solids	Lab Control Sample	96	96	96	0
Copper	ICAP	Solids	Lab Control Sample	92	95	94	2

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Copper	ICAP	Solids	Lab Control Sample	88	88	88	0
Copper	ICAP	Solids	Lab Control Sample	84	87	86	3
Copper	ICAP	Solids	NBS 1633A	96	97	96	1
Copper	ICAP	Solids	NBS 1633A	97	103	100	5
Copper	ICAP	Solids	NBS 1633A	96	96	96	0
Copper	ICAP	Solids	NBS 1633A	83	83	83	1
Fluoride	SIE	Solids	Lab Control Sample	91	94	92	3
Fluoride	SIE	Solids	Lab Control Sample	80	83	81	4
Fluoride	SIE	Solids	Lab Control Sample	88	90	89	2
Iron	ICAP	Solids	ERA	111	114	113	3
Iron	ICAP	Solids	ERA	96	105	100	8
Iron	ICAP	Solids	Lab Control Sample	92	92	92	0
Iron	ICAP	Solids	Lab Control Sample	92	93	93	0
Iron	ICAP	Solids	Lab Control Sample	86	88	87	2
Iron	ICAP	Solids	Lab Control Sample	86	86	86	0
Iron	ICAP	Solids	Lab Control Sample	83	85	84	2
Iron	ICAP	Solids	NBS 1633A	89	91	90	2
Iron	ICAP	Solids	NBS 1633A	87	88	87	2
Iron	ICAP	Solids	NBS 1633A	89	92	91	3
Iron	ICAP	Solids	NBS 1633A	83	84	83	1
Lead	GFAA	Solids	ERA	100	101	101	1
Lead	GFAA	Solids	ERA	85	86	85	1
Lead	GFAA	Solids	Lab Control Sample	94	95	95	1
Lead	GFAA	Solids	Lab Control Sample	106	109	108	3
Lead	GFAA	Solids	Lab Control Sample	95	97	96	2
Lead	GFAA	Solids	Lab Control Sample	99	102	100	2
Lead	GFAA	Solids	NBS 1633A	85	85	85	0
Lead	GFAA	Solids	NBS 1633A	72	73	73	1
Lead	GFAA	Solids	NBS 1633A	72	73	73	0
Lead	GFAA	Solids	unknown	111	111	111	0
Magnesium	ICAP	Solids	ERA	99	102	101	3
Magnesium	ICAP	Solids	ERA	82	83	83	1
Magnesium	ICAP	Solids	Lab Control Sample	92	93	93	0
Magnesium	ICAP	Solids	Lab Control Sample	93	94	93	1
Magnesium	ICAP	Solids	Lab Control Sample	89	90	90	0
Magnesium	ICAP	Solids	Lab Control Sample	93	93	93	0
Magnesium	ICAP	Solids	Lab Control Sample	87	90	89	4
Magnesium	ICAP	Solids	NBS 1633A	76	97	87	24
Magnesium	ICAP	Solids	NBS 1633A	73	93	83	24
Magnesium	ICAP	Solids	NBS 1633A	66	80	73	19
Magnesium	ICAP	Solids	NBS 1633A	61	73	67	17

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Manganese	ICAP	Solids	ERA	90	93	91	2
Manganese	ICAP	Solids	ERA	92	92	92	0
Manganese	ICAP	Solids	Lab Control Sample	91	91	91	0
Manganese	ICAP	Solids	Lab Control Sample	95	95	95	0
Manganese	ICAP	Solids	Lab Control Sample	91	92	92	1
Manganese	ICAP	Solids	Lab Control Sample	87	87	87	0
Manganese	ICAP	Solids	Lab Control Sample	84	86	85	2
Manganese	ICAP	Solids	NBS 1633A	93	95	94	2
Manganese	ICAP	Solids	NBS 1633A	90	91	91	1
Manganese	ICAP	Solids	NBS 1633A	87	97	92	10
Manganese	ICAP	Solids	NBS 1633A	79	86	83	8
Mercury	CVAA	Solids	ERA	94	94	94	1
Mercury	CVAA	Solids	ERA	95	98	96	4
Mercury	CVAA	Solids	Lab Control Sample	122	124	123	2
Mercury	CVAA	Solids	NBS 1633A	94	99	97	5
Mercury	CVAA	Solids	NBS 1633A	104	106	105	2
Mercury	CVAA	Solids	NBS 1633A	114	116	115	2
Molybdenum	ICAP	Solids	ERA	96	99	98	4
Molybdenum	ICAP	Solids	ERA	94	97	96	3
Molybdenum	ICAP	Solids	Lab Control Sample	89	89	89	0
Molybdenum	ICAP	Solids	Lab Control Sample	94	94	94	1
Molybdenum	ICAP	Solids	Lab Control Sample	86	88	87	2
Molybdenum	ICAP	Solids	Lab Control Sample	86	86	86	0
Molybdenum	ICAP	Solids	Lab Control Sample	87	91	89	4
Molybdenum	ICAP	Solids	NBS 1633A				
Molybdenum	ICAP	Solids	NBS 1633A				
Nickel	ICAP	Solids	ERA	98	100	99	2
Nickel	ICAP	Solids	ERA	91	93	92	2
Nickel	ICAP	Solids	Lab Control Sample	90	91	90	1
Nickel	ICAP	Solids	Lab Control Sample	94	95	94	0
Nickel	ICAP	Solids	Lab Control Sample	94	94	94	0
Nickel	ICAP	Solids	Lab Control Sample	88	88	88	0
Nickel	ICAP	Solids	Lab Control Sample	87	91	89	4
Nickel	ICAP	Solids	NBS 1633A	120	120	120	1
Nickel	ICAP	Solids	NBS 1633A	97	100	98	3
Nickel	ICAP	Solids	NBS 1633A	113	119	116	5
Nickel	ICAP	Solids	NBS 1633A	95	100	98	5
Phosphorus	ICAP	Solids	Lab Control Sample	100	101	101	1
Phosphorus	ICAP	Solids	Lab Control Sample	101	104	103	3
Phosphorus	ICAP	Solids	Lab Control Sample	95	99	97	4
Phosphorus	ICAP	Solids	Lab Control Sample	88	89	89	0

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Potassium	ICAP	Solids	ERA	94	97	96	2
Potassium	ICAP	Solids	ERA	92	98	95	6
Potassium	ICAP	Solids	Lab Control Sample	92	93	93	0
Potassium	ICAP	Solids	Lab Control Sample	93	94	93	1
Potassium	ICAP	Solids	Lab Control Sample	89	90	90	2
Potassium	ICAP	Solids	Lab Control Sample	88	88	88	0
Potassium	ICAP	Solids	Lab Control Sample	84	85	84	1
Potassium	ICAP	Solids	NBS 1633A	87	89	88	2
Potassium	ICAP	Solids	NBS 1633A	82	86	84	5
Potassium	ICAP	Solids	NBS 1633A	90	90	90	1
Potassium	ICAP	Solids	NBS 1633A	78	80	79	2
Selenium	GFAA	Solids	ERA	104	104	104	0
Selenium	GFAA	Solids	ERA	102	107	105	4
Selenium	GFAA	Solids	ERA	110	111	110	1
Selenium	GFAA	Solids	Lab Control Sample	104	104	104	0
Selenium	GFAA	Solids	Lab Control Sample	103	103	103	0
Selenium	GFAA	Solids	Lab Control Sample	96	96	96	0
Selenium	GFAA	Solids	Lab Control Sample	87	87	87	0
Selenium	GFAA	Solids	Lab Control Sample	99	100	100	1
Selenium	GFAA	Solids	Lab Control Sample	83	83	83	1
Selenium	GFAA	Solids	NBS 1633A	74	81	77	9
Selenium	GFAA	Solids	NBS 1633A	84	84	84	1
Silicon	ICAP	Solids	ERA				
Silicon	ICAP	Solids	ERA				
Silicon	ICAP	Solids	Lab Control Sample	98	99	99	1
Silicon	ICAP	Solids	Lab Control Sample	93	94	93	1
Silicon	ICAP	Solids	Lab Control Sample	98	98	98	0
Silicon	ICAP	Solids	Lab Control Sample	98	99	99	1
Silicon	ICAP	Solids	Lab Control Sample	91	91	91	0
Silicon	ICAP	Solids	Lab Control Sample	90	92	91	2
Silicon	ICAP	Solids	NBS 1633A	99	100	100	2
Silicon	ICAP	Solids	NBS 1633A	95	98	97	3
Silicon	ICAP	Solids	NBS 1633A	88	97	93	9
Silicon	ICAP	Solids	NBS 1633A	82	93	88	12
Sodium	ICAP	Solids	ERA	130	134	132	3
Sodium	ICAP	Solids	ERA	87	88	88	0
Sodium	ICAP	Solids	Lab Control Sample	95	95	95	0
Sodium	ICAP	Solids	Lab Control Sample	96	96	96	1
Sodium	ICAP	Solids	Lab Control Sample	91	92	92	1
Sodium	ICAP	Solids	Lab Control Sample	95	95	95	0
Sodium	ICAP	Solids	Lab Control Sample	88	91	90	3

RPD = relative percent difference   NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-4 (Continued)

Analyte	Method	Sample Type	LCS Type	Minimum Recovery	Maximum Recovery	Average Recovery	Duplicate RPD
Sodium	ICAP	Solids	NBS 1633A	99	104	101	5
Sodium	ICAP	Solids	NBS 1633A	88	89	89	1
Sodium	ICAP	Solids	NBS 1633A	132	143	137	8
Sodium	ICAP	Solids	NBS 1633A	111	121	116	8
Vanadium	ICAP	Solids	ERA	110	112	111	2
Vanadium	ICAP	Solids	ERA	93	94	93	1
Vanadium	ICAP	Solids	Lab Control Sample	94	95	95	1
Vanadium	ICAP	Solids	Lab Control Sample	96	96	96	1
Vanadium	ICAP	Solids	Lab Control Sample	92	93	93	1
Vanadium	ICAP	Solids	Lab Control Sample	87	87	87	0
Vanadium	ICAP	Solids	Lab Control Sample	84	86	85	2
Vanadium	ICAP	Solids	NBS 1633A	95	96	95	0
Vanadium	ICAP	Solids	NBS 1633A	93	93	93	1
Vanadium	ICAP	Solids	NBS 1633A	95	95	95	1
Vanadium	ICAP	Solids	NBS 1633A	87	87	87	0
Zinc	ICAP	Solids	ERA	100	117	108	16
Zinc	ICAP	Solids	ERA	85	87	86	2
Zinc	ICAP	Solids	Lab Control Sample	87	87	87	0
Zinc	ICAP	Solids	Lab Control Sample	92	93	92	1
Zinc	ICAP	Solids	Lab Control Sample	92	93	92	1
Zinc	ICAP	Solids	Lab Control Sample	90	90	90	0
Zinc	ICAP	Solids	Lab Control Sample	93	94	93	1
Zinc	ICAP	Solids	NBS 1633A	113	114	114	1
Zinc	ICAP	Solids	NBS 1633A	88	89	88	1
Zinc	ICAP	Solids	NBS 1633A	103	105	104	2
Zinc	ICAP	Solids	NBS 1633A	95	95	95	0

RPD = relative percent difference NBS 1633A = National Bureau of Standards standard reference coal fly ash

Table F-5
Summary of Matrix Spike Results for Site 20

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO[a] for Recovery
Solid: Metals Determined by ICPES						
Aluminum	10	113	2	0	4	75-125%
Antimony	10	100	15	1	4	75-125%
Barium	12	105	9	2	4	75-125%
Beryllium	14	101	2	0	1	75-125%
Calcium	4	89	3	0	0	75-125%
Chromium	12	100	1	0	3	75-125%
Cobalt	12	87	2	2	0	75-125%
Copper	12	104	4	0	4	75-125%
Iron	12	97	1	1	3	75-125%
Magnesium	10	93	2	1	3	75-125%
Manganese	10	105	4	0	4	75-125%
Molybdenum	10	103	4	0	1	75-125%
Nickel	12	98	2	0	1	75-125%
Phosphorus	8	90	3	0	0	75-125%
Potassium	12	96	3	2	4	75-125%
Silicon	12	151	6	0	7	75-125%
Sodium	10	113	5	0	4	75-125%
Vanadium	10	104	2	0	4	75-125%
Zinc	12	102	2	0	0	75-125%
Solids: Metals Determined by GFAA						
Arsenic	10	97	2	2	1	75-125%
Cadmium	9	111	5	0	0	75-125%
Lead	6	98	9	0	0	75-125%
Selenium	6	91	11	1	0	75-125%
Solids: Metals Determined by CVAA						
Mercury	14	102	8	0	0	75-125%
Solids: Anions						
Chloride	12	62	9	6	0	80-120%
Fluoride	8	93	2	0	0	80-120%
Sulfate	2	91	4	0	0	80-120%
Liquids: Metals Determined by ICPES						
Aluminum	4	94	0	0	0	75-125%
Antimony	4	92	2	0	0	75-125%
Barium	4	62	6	2	0	75-125%

Table F-5 (Continued)

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO for Recovery
Beryllium	4	98	0	0	0	75-125%
Calcium	4	83	3	1	0	75-125%
Chromium	4	96	0	0	0	75-125%
Cobalt	4	94	0	0	0	75-125%
Copper	4	95	0	0	0	75-125%
Iron	5	79	8	1	0	75-125%
Magnesium	4	89	1	0	0	75-125%
Manganese	4	95	0	0	0	75-125%
Molybdenum	4	94	2	0	0	75-125%
Nickel	4	95	1	0	0	75-125%
Phosphorus	4	99	1	0	0	75-125%
Potassium	4	96	3	0	0	75-125%
Silicon	4	100	1	0	0	75-125%
Sodium	4	82	4	2	0	75-125%
Vanadium	4	97	0	0	0	75-125%
Zinc	4	93	0	0	0	75-125%
Liquids: Metals Determined by GFAA						
Arsenic	4	101	1	0	0	75-125%
Cadmium	3	101	3	0	0	75-125%
Lead	4	105	0	0	0	75-125%
Selenium	6	29	750	6	0	75-125%
Liquids: Metals Determined by CVAA						
Mercury	14	93	2	2	0	75-125%
Liquids: Anions						
Chloride	12	94	9	2	0	80-120%
Fluoride	12	81	10	3	0	80-120%
Sulfate	8	98	15	0	0	80-120%

[a] DQO = Data quality objective.

Table F-6
Detailed Matrix Spike Results

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Aluminum	mg/kg	6,090	152Q	146Q	4	FGD Solids	Grab
Aluminum	mg/kg	5,800	144Q	146Q	1.4	FGD Solids	Grab
Aluminum	mg/kg	160,000	83Q	88	5.8	Fly Ash	Grab
Aluminum	mg/kg	157,000	81	82	1.2	Fly Ash	Grab
Aluminum	mg/kg	4240	102	102	0	Limestone	Grab
Aluminum	mg/L	9.4	93	93	0	ESP Outlet	Nitric Impinger
Aluminum	mg/L	9.54	94	94	0	Makeup Water	Grab
Antimony	mg/kg	85.9	138Q	128Q	7.5	FGD Solids	Grab
Antimony	mg/kg	85.6	138Q	128Q	7.5	FGD Solids	Grab
Antimony	mg/kg	259J	43Q	75	54Q	Fly Ash	Grab
Antimony	mg/kg	370	82	86	4.8	Fly Ash	Grab
Antimony	mg/kg	57.2	86	86	0	Limestone	Grab
Antimony	mg/L	0.968	98	94	4.2	ESP Outlet	Nitric Impinger
Antimony	mg/L	0.888					
Antimony	mg/L	0.89	88	88	0	Makeup Water	Grab
Arsenic	mg/L	16.8	87	86	1.2	QA	MSD
Arsenic	mg/kg	14.8	64Q	65Q	1.6	Limestone	Grab
Arsenic	mg/kg	391	123	132Q	7.1	Fly Ash	Grab
Arsenic	mg/kg	12.9	109	108	0.92	FGD Solids	Grab
Arsenic	mg/kg	12.8	96	95	1	QA	MSD
Arsenic	mg/L	0.047	95	94	1.1	ESP Outlet	Nitric Impinger
Arsenic	mg/L	0.0553	108	108	0	Makeup Water	Grab
Barium	mg/kg	3,200	95	77	21Q	Fly Ash	Grab
Barium	mg/kg	162	154Q	143Q	7.4	FGD Solids	Grab
Barium	mg/kg	154	142Q	144Q	0.7	FGD Solids	Grab
Barium	mg/kg	2,940	80	84	4.9	Fly Ash	Grab
Barium	mg/kg	2,830	74Q	66Q	11	Fly Ash	Grab
Barium	mg/kg	148	108	97	11	Limestone	Grab
Barium	mg/L	0.279	28Q	31Q	10	ESP Outlet	Nitric Impinger
Barium	mg/L	1.09	93	94	1.1	Makeup Water	Grab
Beryllium	mg/kg	93.3	129Q	125	3.2	FGD Solids	Grab
Beryllium	mg/kg	88.7	123	125	1.6	FGD Solids	Grab
Beryllium	mg/kg	439	87	89	2.3	Fly Ash	Grab

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Beryllium	mg/kg	415	82	81	1.2	Fly Ash	Grab
Beryllium	mg/kg	62	83	83	0	Limestone	Grab
Beryllium	mg/L	1	100	100	0	ESP Outlet	Nitric Impinger
Beryllium	mg/L	0.979	98	98	0	Makeup Water	Grab
Cadmium	mg/kg	3.25	109	114	4.5	FGD Solids	Grab
Cadmium	mg/kg	2.47	110	105	4.6	QA	MSD
Cadmium	mg/kg	22	114	106	7.3	Fly Ash	Grab
Cadmium	mg/kg	0.0442	110	101	8.5	Makeup Water	Grab
Cadmium	mg/kg	3.96	116	117	0.86	Limestone	Grab
Cadmium	mg/L	0.043	105	102	2.9	ESP Outlet	Nitric Impinger
Calcium	mg/kg	271,000	49Qx	52Qx	5.9	FGD Solids	Grab
Calcium	mg/kg	155,000	90	94	4.4	Fly Ash	Grab
Calcium	mg/kg	148,000	85	86	1.2	Fly Ash	Grab
Calcium	mg/kg	348,000	148Qx	321Qx	129Q	Limestone	Grab
Calcium	mg/L	10.2	97	96	1	ESP Outlet	Nitric Impinger
Calcium	mg/L	57.9	68Q	72Q	5.7	Makeup Water	Grab
Chloride	mg/kg	5.52	74Q	71Q	4.1	Precipitator Ash	Grab
Chloride	mg/kg	31	-60Q	-60Q	0	Limestone	Grab
Chloride	mg/kg	6.26	62Q	67Q	7.8	Fly Ash	Grab
Chloride	mg/kg	11.1	88	99	12	Fly Ash	Grab
Chloride	mg/kg	526	109	93	16	FGD Solids	Grab
Chloride	mg/kg	115	103	97	6	Coal	Grab
Chloride	μg/filter	184	100	95	5.1	Stack Gas	Analytical Spike
Chloride	mg/L	59.9	100	99	1	Makeup Water	Grab
Chloride	mg/L	0.236	80	87	8.4	Stack Gas	Carbonate Impinger 2
Chloride	mg/L	0.622	91	108	17	Stack Gas	Carbonate Impinger 1
Chloride	mg/L	50.5	107	97	9.8	Stack Gas	Carbonate & Ace PNR
Chloride	mg/L	0.24	70Q	70Q	0	Stack Gas	Carbonate Impinger 2
Chloride	mg/L	5.19	120	103	15	ESP Outlet	Carbonate & Ace PNR & Filter
Chromium	mg/kg	99	131Q	127Q	3.1	FGD Solids	Grab
Chromium	mg/kg	94.7	125	127Q	1.6	FGD Solids	Grab
Chromium	mg/kg	82	85	87	2.3	QA	MSD
Chromium	mg/kg	516	90	90	0	Fly Ash	Grab

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Chromium	mg/kg	489	84	84	0	Fly Ash	Grab
Chromium	mg/kg	64.9	83	82	1.2	Limestone	Grab
Chromium	mg/L	0.972	97	97	0	ESP Outlet	Nitric Impinger
Chromium	mg/L	0.952	95	95	0	Makeup Water	Grab
Cobalt	mg/kg	90	124	122	1.6	FGD Solids	Grab
Cobalt	mg/kg	86.2	119	121	2.5	FGD solids	Grab
Cobalt	mg/kg		27Q	26Q	3.8	FGD Solids	Grab
Cobalt	mg/kg	469	90	88	2.2	Fly Ash	Grab
Cobalt	mg/kg	433	83	83	0	Fly Ash	Grab
Cobalt	mg/kg	60.1	80	80	0	Limestone	Grab
Cobalt	mg/L	0.944	95	95	0	ESP Outlet	Nitric Impinger
Cobalt	mg/L	0.932	93	93	0	Makeup Water	Grab
Copper	mg/kg	105	143Q	135Q	5.8	FGD Solids	Grab
Copper	mg/kg	100	136Q	135Q	0.74	FGD Solids	Grab
Copper	mg/kg	85.9	85	89	4.6	QA	MSD
Copper	mg/kg	543	85	89	4.6	Fly Ash	Grab
Copper	mg/kg	530	83	84	1.2	Fly Ash	Grab
Copper	mg/kg	74	97	92	5.3	Limestone	Grab
Copper	mg/L	0.959	96	96	0	ESP Outlet	Nitric Impinger
Copper	mg/L	0.947	94	94	0	Makeup Water	Grab
Fluoride	mg/kg	26.6	88	87	1.1	FGD Solids	Grab
Fluoride	mg/kg	14.1	94	93	1.1	ESP Outlet	Carbonate Impinger 1
Fluoride	mg/kg	14.4	102	99	3	Stack Gas	Carbonate & Ace PNR
Fluoride	mg/kg	13	87	87	0	FGD Solids	Grab
Fluoride	mg/L	16.6	88	87	1.1	Limestone	MSD
Fluoride	mg/L	17.1	65Q			Fly Ash	Grab
Fluoride	mg/L	0.821	82	83	1.2	ESP Outlet	Carbonate & Ace PNR & Filter
Fluoride	mg/L	17.2	85	93	9	Stack Gas	Carbonate Impinger 1
Fluoride	mg/L	0.811	69Q	47Q	38Q	Fly Ash	Grab
Fluoride	mg/L	0.759	83			QA	MS
Fluoride	mg/L	0.72	96	96	0	Makeup Water	Grab
Iron	mg/kg		136Q	129Q	5.3	FGD Solids	Grab
Iron	mg/kg		94	94	0	QA	MSD

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Iron	mg/kg	169,000	93	93	0	Makeup Water	Grab
Iron	mg/kg	11,700	93	93	0	QA	MSD
Iron	mg/kg	11,100R	128Q	129Q	0.78	FGD Solids	Grab
Iron	mg/kg	155,000	93			QA	MS
Iron	mg/kg	3,860	94	94	0	ESP Outlet	Nitric Impinger
Iron	mg/kg	165,000	28Qx	28Q	0	FGD Solids	Grab
Iron	mg/kg	156,000	129Qx			FGD Solids	Grab
Iron	mg/L	9.51	81	80	1.2	Limestone	Grab
Iron	mg/L	9.52	83	87	4.7	Fly Ash	Grab
Iron	mg/L	9.48	65Q	79	19	QA	MSD
Lead	mg/kg	512	101	89	13	ESP Outlet	Nitric Impinger
Lead	mg/kg	9.55	103	102	0.98	Fly Ash	Grab
Lead	mg/kg	9.61	101	89	13	ESP Outlet	MSD
Lead	mg/L	0.0892	115	115	0	Limestone	Grab
Lead	mg/L	0.104	105			Makeup Water	Grab
Lead	mg/L	0.157	85	85	0	FGD Solids	Grab
Magnesium	mg/kg	7,190	92			ESP Outlet	Nitric Impinger
Magnesium	mg/kg	101,000	29Q	28Q	3.5	FGD Solids	Grab
Magnesium	mg/kg	96,200	139Q	140Q	0.72	FGD Solids	Grab
Magnesium	mg/kg	6,820	93	92	1.1	QA	MSD
Magnesium	mg/L	9.24	89	89	0	QA	MSD
Magnesium	mg/L	16.9	89	89	0	Makeup Water	Grab
Magnesium	mg/L	16.8	88	90	2.2	Fly Ash	Grab
Manganese	mg/kg	183	145Q	138Q	5	FGD Solids	Grab
Manganese	mg/kg	174	133Q	137Q	3.7	FGD Solids	Grab
Manganese	mg/kg	779	85	88	3.5	Fly Ash	Grab
Manganese	mg/kg	738	81	81	0	Fly Ash	Grab
Manganese	mg/kg	174	82	75	8.9	Limestone	Grab
Manganese	mg/L	0.953	95	95	0	ESP Outlet	Nitric Impinger
Manganese	mg/L	0.952	94	94	0	Makeup Water	Grab
Mercury	mg/kg	1.1	111	111	0	Limestone	Grab
Mercury	mg/kg	1.68	101	99	2	FGD Solids	Grab
Mercury	mg/kg	0.941	96	105	9	QA	MSD
Mercury	mg/kg	1.49	126Q	96	27Q	FGD Solids	Grab

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Mercury	mg/kg	1.47	100	103	3	QA	MSD
Mercury	mg/kg	1.16	85	75	12	QA	MSD
Mercury	mg/kg	1.1	109	106	2.8	Fly Ash	Grab
Mercury	mg/L	0.636	72Q	71Q	1.4	QA	MSD
Mercury	mg/L	0.0835	103	102	0.98	ESP Outlet	Nitric Impinger
Mercury	mg/L	0.0019	98	92	6.3	Makeup Water	Grab
Mercury	mg/L	0.00194	101	102	0.98	Makeup Water	Grab
Mercury	mg/L	0.159	86	87	1.2	Stack Gas	$KMnO_4$ Impinger
Mercury	mg/L	0.31	98	102	4	Stack Gas	$KMnO_4$ Impinger
Mercury	mg/L	0.0788	92	91	1.1	ESP Outlet	Nitric Impinger
Molybdenum	mg/kg	92.2	128Q	124	3.2	FGD Solids	Grab
Molybdenum	mg/kg	85.4	124			FGD Solids	Grab
Molybdenum	mg/kg	88.3	122	124	1.6	FGD Solids	Grab
Molybdenum	mg/kg	204	95	88	7.6	Fly Ash	Grab
Molybdenum	mg/kg	183	90	90	0	Fly Ash	Grab
Molybdenum	mg/kg	61.6	82	82	0	Limestone	Grab
Molybdenum	mg/L	0.938	94	94	0	ESP Outlet	Nitric Impinger
Molybdenum	mg/L	0.944	93	93	0	Makeup Water	Grab
Nickel	mg/kg	93.6	126Q	123	2.4	FGD Solids	Grab
Nickel	mg/kg	88.3	118	122	4.2	FGD Solids	Grab
Nickel	mg/kg	564	94	93	1.1	Fly Ash	Grab
Nickel	mg/kg	72.4	85	86	1.2	QA	MSD
Nickel	mg/kg	490	85	83	2.4	Fly Ash	Grab
Nickel	mg/kg	61.7	79	78	1.3	Limestone	Grab
Nickel	mg/L	0.969	97	96	1	ESP Outlet	Nitric Impinger
Nickel	mg/L	0.946	95	95	0	Makeup Water	Grab
Phosphorus	mg/kg	695	92	93	1.1	FGD Solids	Grab
Phosphorus	mg/kg	46,300	97	96	1	Fly Ash	Grab
Phosphorus	mg/kg	775	94	89	5.5	Limestone	Grab
Phosphorus	mg/kg	670	81	79	2.5	Limestone	Grab
Phosphorus	mg/L	10.4	103	104	0.97	ESP Outlet	Nitric Impinger
Phosphorus	mg/L	9.51	95	94	1.1	Makeup Water	Grab

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Potassium	mg/kg	5,450	146Q	141Q	3.5	FGD Solids	Grab
Potassium	mg/kg	5,220	140Q	141Q	0.71	FGD Solids	Grab
Potassium	mg/kg		27Q	29Q	7.1	FGD Solids	Grab
Potassium	mg/kg	50,100	85	88	3.5	Fly Ash	Grab
Potassium	mg/kg	48,800	84	83	1.2	Fly Ash	Grab
Potassium	mg/kg	3,730	96	96	0	Limestone	Grab
Potassium	mg/L	18.5	93	92	1.1	ESP Outlet	Nitric Impinger
Potassium	mg/L	24.3	91	92	1.1	Makeup Water	Grab
Selenium	mg/kg	52.6	82	83	1.2	Fly Ash	Grab
Selenium	mg/kg	4.54F,R	68Q	85	22Q	Limestone	Grab
Selenium	mg/kg	6.89	108	118	8.8	Limestone	Grab
Selenium	mg/kg	19	-200Qx	-71Q	103Q	FGD Solids	Grab
Selenium	mg/L	1.49	11Q			ESP Outlet	Nitric Impinger
Selenium	mg/L	1.65	19Q		Q	ESP Outlet	Nitric Impinger
Selenium	mg/L	0.914	-5Q	6.1Q	2,240Q	ESP Outlet	Nitric Impinger
Selenium	mg/L	0.0397	73Q	71Q	2.8	Makeup Water	Grab
Silicon	mg/kg	1,460	244Q	228Q	6.8	FGD Solids	Grab
Silicon	mg/kg	1,400	227Q	228Q	0.44	FGD Solids	Grab
Silicon	mg/kg	392,000	88	93	5.5	Fly Ash	Grab
Silicon	mg/kg	387,000	87	88	1.1	Fly Ash	Grab
Silicon	mg/kg	968	132Q	145Q	9.4	Limestone	Grab
Silicon	mg/kg	747	117	135Q	14	Limestone	Grab
Silicon	mg/L	6.7	96	97	1	ESP Outlet	Nitric Impinger
Silicon	mg/L	8.53	104	102	1.9	Makeup Water	Grab
Sodium	mg/kg	5,780	151Q	146Q	3.4	FGD Solids	Grab
Sodium	mg/kg	5,510	144Q	146Q	1.4	FGD Solids	Grab
Sodium	mg/kg	86,800	87	90	3.4	Fly Ash	Grab
Sodium	mg/kg	86,100	87	87	0	Fly Ash	Grab
Sodium	mg/kg	3,700	98	97	1	Limestone	Grab
Sodium	mg/L	9.9	94	94	0	ESP Outlet	Nitric Impinger
Sodium	mg/L	61.5	66Q	72Q	8.7	Makeup Water	Grab

Table F-6 (Continued)

Analyte	Units	Result	Spike Recovery	Dup Recovery	RPD	Stream	Sample Type
Sulfate	μg/filter	15,200	89	93	4.4	Stack Gas	Analytical Spike
Sulfate	mg/L	413	96	97	1	Makeup Water	Grab
Sulfate	mg/L	1,730	85	85	0	Stack Gas	Carbonate Impinger 1
Sulfate	mg/L	641	116	107	8.1	QA	MSD
Sulfate	mg/L	337	114	86	28Q	QA	MSD
Vanadium	mg/kg	105	137Q	133Q	3	FGD Solids	Grab
Vanadium	mg/kg	100	130Q	133Q	2.3	FGD Solids	Grab
Vanadium	mg/kg	676	86	88	2.3	Fly Ash	Grab
Vanadium	mg/kg	647	83	82	1.2	Fly Ash	Grab
Vanadium	mg/kg	68.9	86	85	1.2	Limestone	Grab
Vanadium	mg/L	0.98	98	98	0	ESP Outlet	Nitric Impinger
Vanadium	mg/L	0.978	96	96	0	Makeup Water	Grab
Zinc	mg/kg	101	129	125	3.2	FGD Solids	Grab
Zinc	mg/kg	96.7	124	125	0.8	FGD Solids	Grab
Zinc	mg/kg	753	95	97	2.1	Fly Ash	Grab
Zinc	mg/kg	85.2	88	84	4.6	QA	MSD
Zinc	mg/kg	723	92	90	2.2	Fly Ash	Grab
Zinc	mg/kg	69.2	85	84	1.2	Limestone	Grab
Zinc	mg/L	0.942	92	92	0	ESP Outlet	Nitric Impinger
Zinc	mg/L	0.943	93	93	0	Makeup Water	Grab

x = Indicates inappropriate spike concentration; this value not used in calculations.

Table F-7
Summary of Analytical Spike Results for Site 20

Compound	No. of Spiked Samples	Mean % Recovery	Mean RPD	No. Below Recov. Limits	No. Above Recov. Limits	DQO[a] for Recovery
Probe and Nozzle Rinse + Filter Fraction (Metals Train, ESP Outlet): Metals Determined by ICPES						
Aluminum	4	86	1	0	0	75-125%
Antimony	4	89	5	0	0	75-125%
Barium	4	66	5	4	0	75-125%
Beryllium	4	88	1	0	0	75-125%
Calcium	4	91	1	0	0	75-125%
Chromium	4	90	1	0	0	75-125%
Cobalt	4	91	1	0	0	75-125%
Copper	4	91	1	0	0	75-125%
Iron	4	85	1	0	0	75-125%
Magnesium	4	89	0	0	0	75-125%
Manganese	4	88	1	0	0	75-125%
Molybdenum	4	85	1	0	0	75-125%
Nickel	4	90	3	0	0	75-125%
Phosphorus	2	95	1	0	0	75-125%
Potassium	4	86	0	0	0	75-125%
Silicon	4	242	1	0	0	75-125%
Sodium	4	91	1	0	0	75-125%
Vanadium	4	90	1	0	0	75-125%
Zinc	4	86	2	0	0	75-125%
Probe and Nozzle Rinse + Filter Fraction (Metals Train, ESP Outlet): Metals Determined by GFAA						
Arsenic	2	92	2	0	0	75-125%
Cadmium	4	89	1	0	0	75-125%
Lead	4	71	3	4	0	75-125%
Selenium	2	100	0	0	0	75-125%
Probe and Nozzle Rinse + Filter Fraction (Metals Train, ESP Outlet): Metals Determined by GFAA						
Mercury	2	121	1	0	0	75-125%
Solids: Anions						
Fluoride	4	99	1	0	0	80-120%

[a] DQO = Data quality objective.

Table F-8
Detailed Analytical Spike Results

Analyte	Spike Recovery	Duplicate Recovery	RPD	Stream	Sample type
Aluminum	89	88	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Aluminum	83	84	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Antimony	90	92	2.2	ESP Outlet	Nitric & Ace PNR & Filter
Antimony	83	90	8.1	ESP Outlet	Nitric & Ace PNR & Filter
Arsenic	93	91	2.2	ESP Outlet	Nitric & Ace PNR & Filter
Barium	73Q	70Q	4.2	ESP Outlet	Nitric & Ace PNR & Filter
Barium	59Q	63Q	6.6	ESP Outlet	Nitric & Ace PNR & Filter
Beryllium	92	92	0	ESP Outlet	Nitric & Ace PNR & Filter
Beryllium	84	85	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Cadmium	88	90	2.2	ESP Outlet	Nitric & Ace PNR & Filter
Cadmium					
Calcium	95	95	0	ESP Outlet	Nitric & Ace PNR & Filter
Calcium	87	88	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Chromium	93	94	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Chromium	86	87	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Cobalt	95	94	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Cobalt	87	87	0	ESP Outlet	Nitric & Ace PNR & Filter
Copper	93	93	0	ESP Outlet	Nitric & Ace PNR & Filter
Copper	88	89	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Fluoride	85	91	6.8	Stack Gas	Analytical Spike
Iron	89	89	0	ESP Outlet	Nitric & Ace PNR & Filter
Iron	81	82	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Lead	69Q	69Q	0	ESP Outlet	Nitric & Ace PNR & Filter
Lead	70Q	74Q	5.6	ESP Outlet	Nitric & Ace PNR & Filter
Magnesium	92	92	0	ESP Outlet	Nitric & Ace PNR & Filter
Magnesium	86	86	0	ESP Outlet	Nitric & Ace PNR & Filter

Table F-8 (Continued)

Analyte	Spike Recovery	Duplicate Recovery	RPD	Stream	Sample type
Manganese	91	91	0	ESP Outlet	Nitric & Ace PNR & Filter
Manganese	84	85	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Molybdenum	86	86	0	ESP Outlet	Nitric & Ace PNR & Filter
Molybdenum	85	83	2.4	ESP Outlet	Nitric & Ace PNR & Filter
Nickel	93	90	3.3	ESP Outlet	Nitric & Ace PNR & Filter
Nickel	86	89	3.4	ESP Outlet	Nitric & Ace PNR & Filter
Phosphorus	94	95	1.1	Stack Gas	Nitric & Ace PNR & Filter
Potassium	88	88	0	ESP Outlet	Nitric & Ace PNR & Filter
Potassium	84	84	0	ESP Outlet	Nitric & Ace PNR & Filter
Selenium	100	100	0	ESP Outlet	Nitric & Ace PNR & Filter
Silicon	161Q	162Q	1.62	ESP Outlet	Nitric & Ace PNR & Filter
Silicon	323Q	323Q	0	ESP Outlet	Nitric & Ace PNR & Filter
Silver	92	90	2.2	ESP Outlet	Nitric & Ace PNR & Filter
Silver	80	81	1.2	ESP Outlet	Nitric & Ace PNR & Filter
Sodium	93	93	0	ESP Outlet	Nitric & Ace PNR & Filter
Sodium	89	90	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Vanadium	93	93	0	ESP Outlet	Nitric & Ace PNR & Filter
Vanadium	87	88	1.1	ESP Outlet	Nitric & Ace PNR & Filter
Zinc	90	88	2.2	ESP Outlet	Nitric & Ace PNR & Filter
Zinc	81	83	2.4	ESP Outlet	Nitric & Ace PNR & Filter
Fluoride	112	111	0.9	Coal	Grab
Fluoride	86	87	1.2	Fly Ash	Grab
Mercury	120	121	0.83	ESP Outlet	Nitric & Ace PNR & Filter

Table F-9
Coal QA/QC

Lab	Stream	Analyte	Method	Result (dry)	Units	Det Limit	SARM Value	Certified % R	Flag
CT&E	SARM 20	Arsenic	GFAA	2	µg/g		4.70	43	
CT&E	SARM 20	Barium	ICPES	360	µg/g		372	97	
CT&E	SARM 20	Cadmium	GFAA						
CT&E	SARM 20	Chromium	ICPES	66	µg/g		67	98	
CT&E	SARM 20	Cobalt	ICPES	16	µg/g		8.3	193	
CT&E	SARM 20	Lead	ICPES	22	µg/g		26	85	
CT&E	SARM 20	Manganese	ICPES	81	µg/g		80	101	
CT&E	SARM 20	Mercury	DGAA	0.29	µg/g		0.25	116	
CT&E	SARM 20	Molybdenum							
CT&E	SARM 20	Nickel	ICPES	24	µg/g		25	96	
CT&E	SARM 20	Phosphorous	ICPES	670	µg/g		612	110	
CT&E	SARM 20	Selenium	GFAA	1	µg/g	1	0.8	125	<
CT&E	SARM 20	Vanadium	ICPES	46	µg/g		47	98	

< Flag indicates value shown is less than the detection limit.

RPD = Relative percent difference.

SARM 20 = South African Reference Material distributed by the South African Bureau of Standards.

NIST = National Institute of Standards and Testing reference material.

Table F-10
Detailed Blank Results for Metals Measured by ICP-MS, Site 20

Description	Analyte	Conc. µg/L	Detection Limit
Blank Microwave Digestion	Arsenic	5.41	0.026
Blank Microwave Digestion	Beryllium	4.84	0.009
Blank Microwave Digestion	Cadmium	0.07@	0.023
Blank Microwave Digestion	Chromium	0.52	0.033
Blank Microwave Digestion	Lead	0.25	0.056
Blank Microwave Digestion	Mercury	0.67	0.064
Blank Microwave Digestion	Nickel	0.33	0.039
Blank Microwave Digestion	Selenium	ND	0.762
Blank 3020 Digestions	Arsenic	0.04@	0.026
Blank 3020 Digestions	Beryllium	0.05	0.009
Blank 3020 Digestions	Cadmium	0.12	0.023
Blank 3020 Digestions	Chromium	2.39	0.033
Blank 3020 Digestions	Lead	3.95	0.056
Blank 3020 Digestions	Mercury	0.78	0.064
Blank 3020 Digestions	Nickel	1.31	0.039
Blank 3020 Digestions	Selenium	ND	0.762

ND = Analyte not detected at stated detection limit.

Table F-11
Detailed Spike Results for Metals Measured by ICP-MS for Site 20

Description	Conc. µg/L	Duplicate Conc. µg/L	Detection Limit	Analyte	Spiked Conc. µg/L	Percent Recovery	Duplicate Percent Recovery	Relative Percent Difference
LCS 3020 Digestions	43.28	44.02	0.026	Arsenic	50	86.56	88.04	2
LCS Microwave Digestion	597.33	640.4	0.026	Arsenic	300	199.11	213.47	7
LCS 3020 Digestions	30.96	31.11	0.009	Beryllium	0[a]			
LCS Microwave Digestion	446.31	449.04	0.009	Beryllium	500	89.26	89.81	1
LCS 3020 Digestions	35.27	36.3	0.023	Cadmium	50	70.54	72.6	3
LCS Microwave Digestion	15.08	17.52	0.023	Cadmium	20	75.40	87.6	15
LCS 3020 Digestions	30.74	30.53	0.033	Chromium	50	61.48	61.06	1
LCS Microwave Digestion	555.73	530.31	0.033	Chromium	500	111.15	106.06	5
LCS 3020 Digestions	83.91	82.91	0.056	Lead	90	93.23	92.12	1
LCS Microwave Digestion	423.21	438.54	0.056	Lead	500	84.64	97.71	14
LCS 3020 Digestions	1.11	0.42	0.064	Mercury	0[a]			
LCS Microwave Digestion	29.96	17.63	0.064	Mercury	0[b]			
LCS 3020 Digestions	65.38	65.47	0.039	Nickel	90	72.64	72.74	0
LCS Microwave Digestion	537.73	513.73	0.039	Nickel	500	107.55	102.78	5
LCS 3020 Digestions	30.49	30.14	0.762	Selenium	50	60.98	60.28	1
LCS Microwave Digestion	115.70	114.76	0.762	Selenium	50	231.40	229.52	1

[a] 3020 digestions are performed for analytes that are determined by graphite furnace atomic absorption spectrophotometry. Beryllium and mercury are not analyzed by this method.

[b] Microwave digestions are performed for analytes that are determined by inductively coupled plasma emission spectroscopy. Mercury is not analyzed by this method.

ICPES-MS = Inductively coupled plasma emission spectroscopy coupled with mass spectrometry.

Table F-12
Detailed Reference Sample Results for Metals Measured by ICP-MS for Site 20

Description	Conc. µg/L	Duplicate Conc. µg/L	Detection Limit	Analyte	NBS 1633a Conc. µg/g	Percent Recovery	Duplicate Percent Recovery	Relative Percent Difference
NBS 1633a	78.20	76.13	0.026	Arsenic	145	53.93	51.47	5
NBS 1633a	6.22	7.23	0.009	Beryllium	12	51.83	59.07	13
NBS 1633a	1.46	1.51	0.023	Cadmium	1	146.00	148.04	1
NBS 1633a	73.95	76.49	0.033	Chromium	196	37.73	38.26	1
NBS 1633a	77.50	74.49	0.056	Lead	72.4	107.04	100.87	6
NBS 1633a	4.64	5.47	0.064	Mercury	0.16	2843.14	3418.75	18
NBS 1633a	53.21	49.9	0.039	Nickel	127	41.08	39.29	4
NBS 1633a	11.05	14.28	0.762	Selenium	10.3	107.28	135.92	24

For fly ash samples, 0.100 grams were digested; for duplicate samples, 0.1020 grams were digested.

ICPES-MS = Inductively coupled plasma emission spectroscopy coupled with mass spectrometry.