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FIELD CHEMICAL EMISSIONS MONITORING PROJECT: SITE 22 EMISSIONS REPORT

PRELIMINARY DRAFT REPORT

Prepared for:

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7 February 1994

EPRI
Electric Power
Research Institute *Leadership in Science and Technology*

February 7, 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 22. Site 22 consists of a 700 MW pulverized coal-fired boiler burning a Powder River Basin coal, with an electrostatic precipitator (ESP). Site 22 did not have an suitable location for sampling at the ESP inlet, thus the sampling and analytical test program focused on the stack emissions. In the Site 22 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 22 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).

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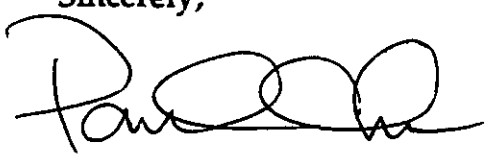
The primary objective of this report is to transmit the preliminary results from Site 22 to the EPA for use in evaluating select trace chemical emissions from fossil-fuel-fired steam generating plants. In addition to the raw data in the Appendix, the report provides an assessment of the trace metals 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 22 with the results from previous utility sites. Generic conclusions and recommendations were not drawn concerning the effectiveness of an electrostatic precipitator as a potential control technology 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 presents data gathered during a sampling program sponsored by the Electric Power Research Institute (EPRI) and the host utility. The analytical and process information discussed in this report was obtained during the sampling effort at Site 22 in July of 1993. The concentrations of selected organic and inorganic substances were measured in the process streams of a pulverized coal-fired, wall-fired utility boiler burning low-sulfur subbituminous Powder River coal. ESPs were used to control particulate emissions.

This report is one of a series being produced under the Field Chemical Emissions Monitoring (FCEM) project (RP-3177-1) sponsored by EPRI. The objective of the FCEM project is to measure selected substances in the process and discharge streams of power plants in order to determine the fate and control of these substances.

Objectives

The specific objectives of the Site 22 study were:

- To quantify emissions of target species from the stack.
- To collect sufficient data to estimate the efficiency of the ESP for removing target species.
- To determine the fate of target species in the various discharge streams associated with Site 22.
- To collect size-fractionated fly ash from a subbituminous-fired power plant. The various size fractions may be analyzed for trace element concentrations in the future.
- To compare two methods for determining mercury concentrations in flue gas. This effort compared the EPA multi-metals train method (Draft Method 29) with the mercury speciation method developed by Frontier Geosciences.

Table 1-1 lists the substances of interest to the FCEM project. All of the substances listed in Table 1-1 were measured at Site 22, except for benzene, toluene, and formaldehyde. In addition, the stack gas was sampled for dioxin and furan compounds.

Table 1-1
FCEM Substances of Interest

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

[a]Not measured at Site 22.

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

[c]Dioxin and furan compounds were also measured at Site 22.

Process Operation

The unit operated at full load during each test run. No unusual process upsets were encountered, and particulate emissions were well below compliance limits. By all indications, process operation during testing was representative of normal operation for this unit.

Sampling and Analysis Protocol

The sampling and analysis protocol for Site 22 is described in Appendix A. The methods used are comparable to those used at other FCEM sites sampled by Radian, with the following exceptions:

- In addition to INAA analysis (employed at other FCEM sites), coal samples were analyzed for metals by ICP-AES, GFAAS, and XRF.
- In addition to ICP-AES analysis (employed at other FCEM sites), flue gas samples were analyzed for arsenic, lead, nickel, and selenium using GFAAS.
- Detection limits for PAH and dioxin/furan compounds in stack gas samples were lower at Site 22 because high resolution GC/MS was used instead of the less sensitive GC/MS technique used at previous FCEM sites.

Quality Assurance/Quality Control (QA/QC) Data Completeness

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 22 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 contaminated during collection or analysis. Each of these standard QA/QC checks were used on samples from Site 22, as appropriate. 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 quality of the results reported in this document is sufficient to meet the study objectives. Established sampling and analytical procedures were employed as far as possible.

The results have been subjected to an extensive QA/QC evaluation, which is presented in Appendix F. The emission results obtained for Site 22 are considered representative of performance under normal operating conditions.

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 statements should be considered when these data are used:

- The methyl mercury data from the Frontier Geosciences' mercury speciation technique are considered invalid and have not been presented in this report. Frontier Geosciences recently discovered that a reaction occurs between acetate, sulfite, and ionic mercury during dissolution of the sorbent traps which produces methyl mercury.

- For measurements by ICP-AES, GFAAS, and CVAAS, most blank samples showed either no contamination, contamination in concentrations less than five times the detection limit, or contamination in concentrations significantly below that found in the corresponding samples. The levels of silicon found in the ash method blank and the stack solids laboratory blank are an artifact of the extraction process; therefore, the silicon levels in these samples are biased high. The stack gas trip blank concentration for silicon is high because the digestion of a quartz filter and similar levels of silicon were found in the samples associated with this blank. Levels of aluminum, barium, calcium, iron, manganese, silicon, sodium, and zinc, were found in the field blank associated with the stack gas vapor-phase stream in concentrations similar to those found in the associated samples; however, all sample levels were extremely low and within five times the detection limit, with the exception of sodium. The trip blank filter associated with ICP-AES metals contained molybdenum at levels similar to those observed in the stack gas solid samples.

- The matrix spike results for selenium in the stack gas stream vapor phase measured by GFAAS showed an extremely poor recovery (0% compared to 75-125% specification). ICP-AES matrix spike/matrix spike duplicate (MS/MSD) analytical results showed a recovery within the specification (105%) and good precision (4% RPD compared to a project specification of 20%). However, the detection limit of ICP-AES is much higher than the GFAAS detection limit, and selenium was not detected in the samples by ICP-AES analysis; therefore, the GFAAS data are used for selenium and should be used with caution.

- The recovery of arsenic (135%) in the spiked fly ash measured by GFAAS was outside the project objectives (75-125%). This indicates the possibility of a slightly high bias for arsenic in the fly ash; however, the precision of the alternate measurement (ICP-AES) of arsenic in fly ash was poor (38% RPD compared to 20% RPD objective). Therefore, although possibly biased high, the GFAAS data were reported for arsenic.

- The GFAAS MS/MSD analytical results for lead in the stack gas solid-phase stream showed slightly low recovery (72%) compared to the project specification. The precision was acceptable (1.1% RPD compared to the 20% RPD specification). This indicates the possibility of a slightly low bias in the results for lead in the stack gas solid phase.

- For PAH measurements by HRGCMS, the method, trip, and field blanks contained nearly all of the analytes at levels that would be expected to bias the results slightly high.

- For PCDD/PCDF measurements by HRGCMS, the method, trip, and field blanks contained anywhere from two to four analytes at levels near their detection limits. These results should not significantly affect the data set.

- The recovery of copper from the SARM 20 coal standard measured by INAA could not be determined. In addition, detection limits for copper by INAA were higher than those for ICP-AES, resulting in concentrations near the detection limit. Therefore, the ICP-AES data for copper were chosen as the primary values.

- The recovery of arsenic in the SARM 20 coal was 43% which is outside the 75-125% accuracy objective. This indicates a possible low bias for arsenic in the coal when measured by GFAAS; therefore, INAA data were selected as the primary values for arsenic.

- The ICP-AES recoveries of nickel (71%) and selenium (58%) in the stack gas solids were below the project specifications (75-125%); therefore, the GFAAS data for these metals were selected as the primary values.

Report Organization

Section 2 of this report presents a brief description of the plant and the sampling locations. Section 3 discusses the chemical analyses of coal, ash, and flue gas samples. Section 4 discusses the results in terms of both analytical and engineering quality assurance considerations. Section 5 presents mercury speciation and particle size distribution data. Section 6 presents example calculations, and a glossary of terms is provided in Section 7. The appendices contain material on sampling and analytical methods, stream concentrations, measured and calculated stream flow rates, particulate matter measurement results, and QA/QC. In particular, Appendix B contains the complete analytical results for the target substances, as well as for major elements. The results reported in this document were obtained using the preferred analytical methods. Appendix C includes the results of analyses for other substances, as well as results obtained by alternate methods.

2

SITE DESCRIPTION

The FCEM project has a policy of giving a site code to each plant sampled. This plant has been designated Site 22. The test site and the sampling locations are described in this section.

Facility Information

A single coal-fired unit is located at Site 22; the configuration of the unit is summarized in Table 2-1. The wall-fired, radiant boiler was designed by Babcock and Wilcox. The furnace consists of a single chamber with no partition.

Figure 2-1 is a process flow diagram of the unit. The plant burns subbituminous coal from the Powder River region. The delivered coal has a typical ash content of less than 7% and a typical sulfur content of less than 0.5 percent. Coal burned during the test was mined from the Smith and Roland Seam of the Black Thunder mine.

Bottom ash is removed from the boiler by an ash sluicing system, and electrostatic precipitators (ESPs) remove fly ash from the flue gas. The flue gas treatment and ash removal facilities are described in greater detail below.

Flue Gas Treatment Facilities

The Site 22 unit is equipped with two cold-side ESPs, with a design specific collection area (SCA) of 654 $ft^2/10^3$ acfm. Each ESP has two outlet ducts through which flue gas flows to induced draft fans (four fans in all) and into the stack.

Ash Removal Facilities

Dry fly ash collected by the ESPs is pneumatically conveyed to an ash silo, from which it is periodically transferred to trucks and sold as a by-product. River water is used to sluice bottom ash from the boiler to an ash basin. Ash collected in the economizer and primary air heater is also sluiced to the ash basin.

Sampling Locations

Samples of four streams (coal, bottom ash, ESP ash, and stack gas) were collected. The sampling locations are identified on the process flow diagram, Figure 2-1.

Table 2-1
Site 22 Summary

Maximum Gross Electrical Output (MW)	700
Startup Date	May 1980
Boiler Type	B & W Radiant Wall-Fired
Boiler Additives	None
Fuel Source	Powder River, Black Thunder Mine, Smith and Roland Seam
Fuel Type	Subbituminous
Fuel Sulfur Content (avg. % S, as received)	0.3
Fuel Ash Content (avg. %, as received)	5
Fuel Heating Value (avg. Btu/lb, dry)	12,000
NO_x Control	None
Particulate Controls	Two Parallel Cold-Side ESPs
ESP Design Efficiency (%)	99.4
ESP Design SCA (ft^2/kacfm)	654
Flue Gas Conditioning	None
Particulate Emission Limit (lb/10^6 Btu)	0.10
SO_2 Emission Limit (lb/10^6 Btu)	1.2
Design Fuel Feed Rate (tons/hr, as received)	400
Fly Ash Disposal	Sold
Bottom Ash Disposal	Pond
Bottom Ash Sluice Water Source	River
Cooling Water System	Once Through
Cooling Water Source	River

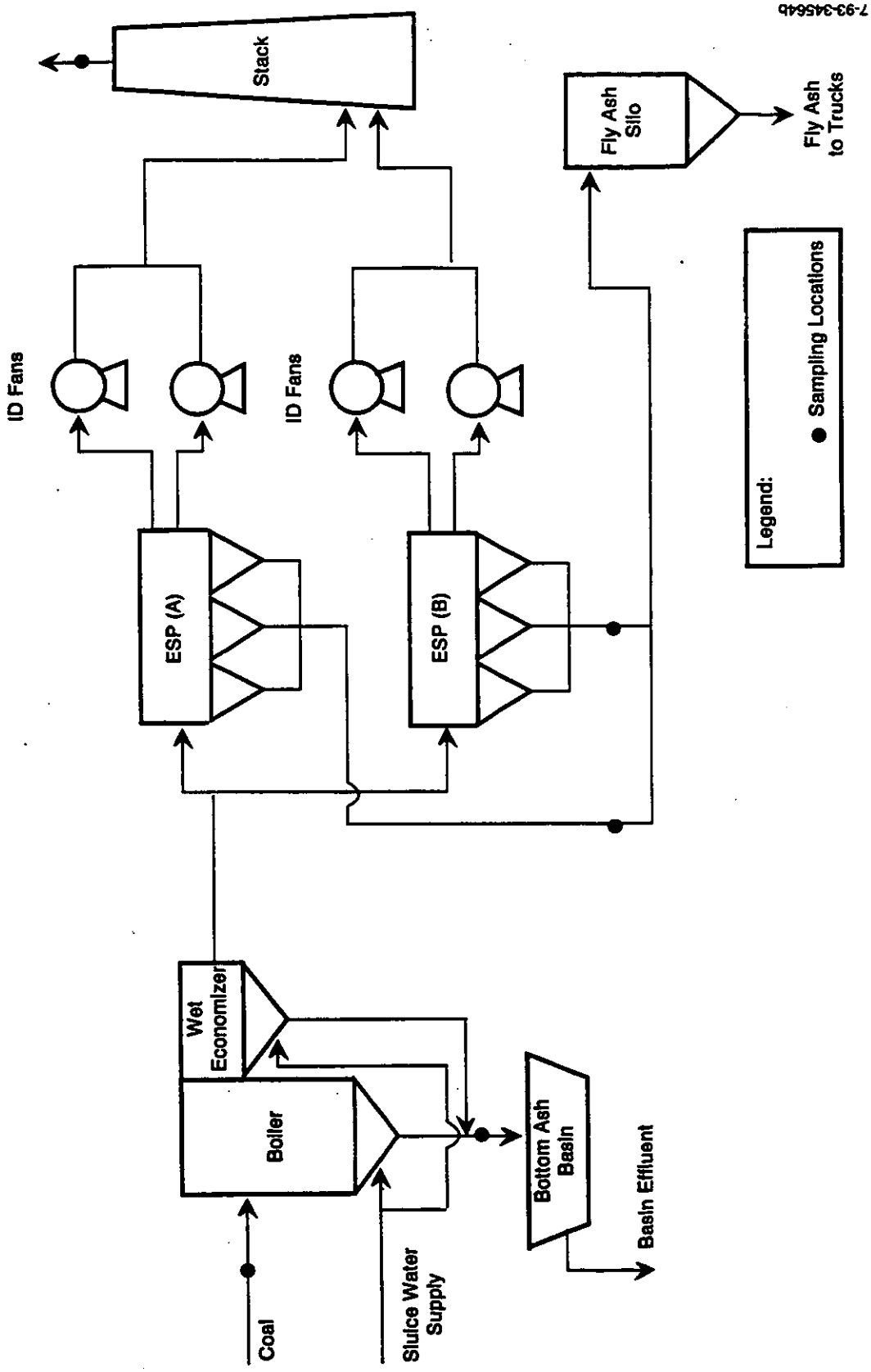


Figure 2-1
Site 22 Process Flow Diagram

- Coal samples were collected with the plant's dedicated automatic sampling system from the six-foot-wide belt that transfers coal to the storage bunkers. Because the samples were collected upstream of the mills, they were taken before the rejection of pyrites. This is not expected to significantly impact the representativeness of the coal samples.

- Bottom ash samples were collected from the sluice pipe leading to the ash basin. High water levels in the ash basin, caused by severe flooding in the area, prevented the planned collection of the bottom ash sample from the end of the sluice pipe. Instead, plant personnel unflanged the sluice pipe upstream of the ash basin to prevent the sluice pipe from plugging and to give the sampling team access to the bottom ash. The new bottom ash sampling point was partially submerged in the flood waters.

- ESP ash samples were collected with an automatic sampling system from the A- and B-side pneumatic transfer lines leading from the ESP ash hoppers to the ash silo. A- and B-side samples were combined to obtain the final composite sample for analysis each day.

- Samples of the flue gas exiting the ESPs were collected from four horizontal ports located on the 300-foot level of the stack.

The procedures for collecting, pretreating, and analyzing the samples are discussed in Appendix A. Table 2-2 presents an overview of the types of analyses performed on samples from these streams.

Table 2-2
Process Stream Analyses Performed

Stream	Metals[a]	Anions[b]	PAHs[c]	Dioxins/Furans	Mercury Speciation	PSD[d]
Coal	✓	✓				
Bottom Ash	✓	✓				
ESP Ash	✓	✓				
Stack Gas	✓	✓	✓	✓	✓	✓

[a]"Metals" include the target species: arsenic, beryllium, cadmium, chromium, copper, lead, manganese, mercury, nickel, phosphorus, and selenium. Data for other species are also available because of the multi-element techniques employed.

[b]"Anions" include the target analytes: chloride and fluoride.

[c]Polycyclic aromatic hydrocarbons (PAHs).

[d]Particle size distribution (PSD).

3

RESULTS

This section summarizes the results of the coal characterization and gas stream analyses. In addition, the performance of the ESP is evaluated, and the enrichment of target inorganic species in the ash and flue gas particulate matter samples in relation to the coal composition is discussed. Sampling, preparation, and analytical methods are summarized in Appendix A. Detailed analytical data can be found in Appendices B and C.

Sampling Schedule

Site 22 was sampled in July 1993 coincident with heavy rainfall in the mid-west. Five types of sampling trains were used to collect flue gas samples from the stack ports. Multi-metals, PAH, dioxin/furan, and anions trains were used to traverse each of the four ports during each sampling run. The mercury speciation sampling system was used to collect flue gas samples from a fixed point in one of the four ports.

Figure 3-1 presents the sampling schedule as executed. As shown in the figure, three valid runs were completed for each of the gas sampling trains. Run 1 anion samples were voided because of sampling problems; therefore, two anion samples were collected on Day 2. The Run 3 coal sampling period was terminated mid-way through the planned period because of plugging problems in the auto sampler caused by excessive moisture levels in the coal.

Data Treatment

Several conventions have been developed for treating the test data and developing average concentrations of substances in the various streams.

To determine the total gas concentration for each run, the solid- and vapor-phase contributions were considered. However, the absence of some reportable concentrations in either (or both) phases required that conventions be developed for dealing with these data and formulating emission factors. These conventions are summarized below.

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

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

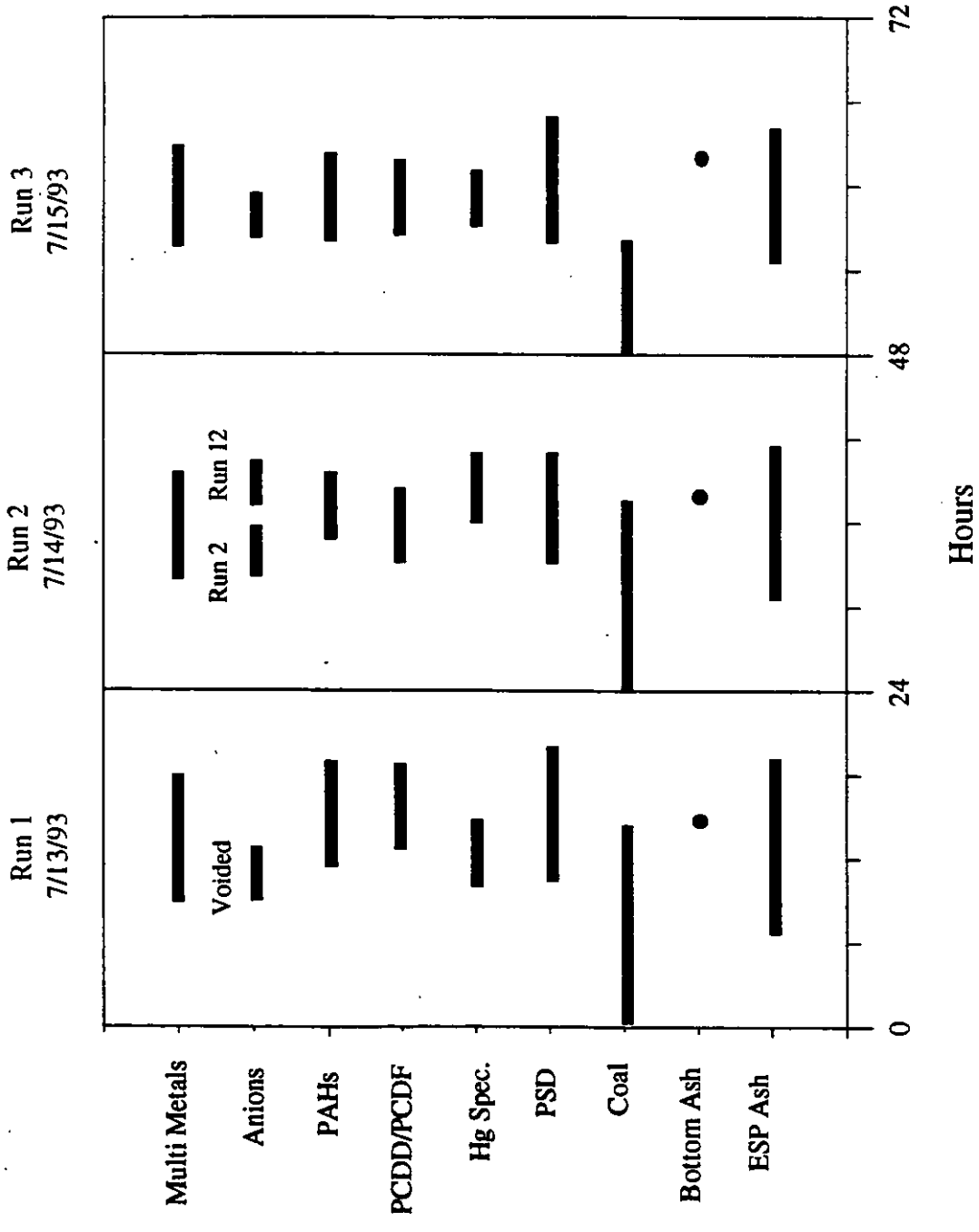


Figure 3-1
Site 22 Sampling Schedule

Case 2: The concentrations of both the solid and vapor phase 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 Case 1, the total concentration is the sum of the concentrations in the vapor and solid phases.

For example, the total chloride concentration in the stack gas for Run 1 is calculated as follows:

Cl in solid phase = 66 $\mu g/Nm^3$

Cl in vapor phase = 683 $\mu g/Nm^3$

Total Cl in stack gas = 749 $\mu g/Nm^3$

For Case 2, the total concentration is considered to be the detection limit in the solid phase.

For example, the total beryllium concentration in the stack gas for Run 1 is calculated as follows:

Be in the solid phase = ND(0.023) $\mu g/Nm^3$

Be in the vapor phase = ND(0.091) $\mu g/Nm^3$

Total Be in the stack gas = ND(0.023) $\mu g/Nm^3$

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 1:

As in solid phase = 0.11 $\mu g/Nm^3$

As in vapor phase = ND(0.11) $\mu g/Nm^3$

Total As in stack gas = 0.11 $\mu g/Nm^3$

The above conventions also are in accordance 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 sites has shown that HCl, HF, and mercury are present primarily in the vapor phase. For Case 2, then, the total concentration is considered to be the reporting limit in the vapor phase. For Cases 1 and 3, the methods are unchanged from those described above.

The following criteria were used to average the results of different runs:

- When all values for a given variable were above the method detection limit, the mean concentration was calculated as the true arithmetic mean.

- For results that include values both above and below the reporting 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 mean value 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 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 for other parameters) is one-half the detection limit, and no confidence interval is reported.

None of the data contained in this report have been corrected for the blank results. Generally, blank values were very low compared with the concentrations found in actual samples; therefore, blank correction was not warranted. Blank levels for some elements were similar to levels found in some Method 29 multi-metals train samples. These cases are noted in the data tables.

Blank levels were significant compared to sample results for some PAHs and dioxin/furan compounds measured in the stack gas, since extremely sensitive analytical methods were used for these analyses. In these cases, the blank levels are reported

along with the uncorrected sample results for comparison. Detailed information on blank sample results can be found in Appendix F.

Coal

Table 3-1 shows the analytical results for the coal samples. Appendix A presents the analytical method used for each combination of substance and stream. The concentrations reported here were measured using what Radian considered to be the best method for each matrix. Typically, the method with the lowest reporting limit was chosen, except when QA/QC data indicated significant problems with precision or bias for a particular technique. For each substance, a mean concentration has been calculated, along with the 95% confidence interval about the mean. The confidence interval is the range about the mean wherein the probability is 95% that the true mean lies. For example, it can be said, with 95% certainty, that the true mean arsenic concentration in the coal is between 0.67 and 1.0 mg/kg, according to the three results shown in Table 3-1. The calculation of this confidence interval is discussed in Section 6 and in Appendix E.

Arsenic, barium, chromium, cobalt, molybdenum, nickel, selenium, and vanadium concentrations in the coal were measured using instrumental neutron activation analysis (INAA). Lead concentrations were measured using graphite furnace atomic absorption spectrophotometry (GFAAS). Beryllium, manganese, copper, phosphorus, and cadmium concentrations were measured using inductively coupled plasma atomic emission spectroscopy (ICP-AES). Chloride concentrations were measured by ion chromatography (IC) and fluorine concentrations were measured by selective ion electrode (SIE).

Mercury concentrations were measured using double gold amalgamation (DGA) with cold vapor atomic absorption spectrophotometry (CVAAS). Mercury concentrations, determined by Frontier Geosciences using cold vapor atomic fluorescence (CVAFS), are also reported.

For those substances that could not be quantified, the notation "ND(x)" is used. This term means "not detected at a concentration of x." The detection limit is calculated by multiplying the instrument-specific method detection limit (MDL) by any dilution or concentration factors for the sample being analyzed. Thus, for a given method, the DL can vary with each individual analysis. The MDL is laboratory specific, instrument specific and matrix specific. It includes the variability arising from both sample preparation and analysis.

Stack Gas

Table 3-2 presents the concentration of the target inorganic analytes 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.

Arsenic, cadmium, chromium, lead, nickel, and selenium concentrations in the flue gas were measured using GFAAS. Beryllium, copper and manganese concentrations were

Table 3-1
Coal Composition for Site 22 (mg/kg, dry)

Substance	Run 1	Run 2	Run 3	Mean	95% CI
Gross Load (MW)	700	703	702	702	4
Coal Rate (kg/hr, dry)	251,000	256,000	246,000	251,000	12,000
HHV (Btu/lb, dry)	12,075	11,977	11,892	11,981	227
Ash (%, dry)	6.46	6.66	7.27	6.80	1.05
Moisture (%)	27.6	29.3	31.7	29.5	5.2
Sulfur (%, dry)	0.33	0.42	0.41	0.39	0.12
Target Species					
Arsenic	0.77	0.86	0.91	0.85	0.18
Barium	347	366	429	380	107
Beryllium	0.20	0.20	0.20	0.20	0
Cadmium	ND(0.1)	ND(0.1)	ND(0.1)	ND(0.1)	--
Chloride	ND(100)	ND(100)	ND(100)	ND(100)	--
Chromium	4.0	4.0	4.0	4.0	0
Cobalt	1.3	1.5	1.5	1.4	0.3
Copper	11	11	12	11	1
Fluoride	50	52	61	54	14
Lead	2.0	2.0	2.0	2.0	0
Manganese	7.0	8.0	10	8.3	3.8
Mercury (DGA/CVAAS)	0.14	0.14	0.14	0.14	0.0008
Mercury (CVAFS)	0.069	0.086	0.078	0.08	0.02
Molybdenum	1.6	2.3	2.5	2.1	1.2
Nickel	3.1	3.8	4.7	3.9	2.0
Phosphorus	300	310	320	310	25
Selenium	0.92	0.70	0.73	0.78	0.30
Vanadium	12	12	13	12	1
Other Species					
Antimony	0.11	0.14	0.15	0.13	0.06

CI = Confidence interval.

Table 3-2
Stack Gas Composition Data for Site 22 ($\mu g/Nm^3$ unless noted)

Substance	Particulate Phase			Vapor Phase			Mean	95% CI
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3		
Stack Flow Rate (Nm^3/hr)	2,500,000	2,550,000	2,550,000				2,530,000	70,000
Particulate (mg/Nm^3)	1.35	2.09	2.07				1.8	1.0
Target Species								
Arsenic	0.11	0.11	0.09	ND(0.11)	ND(0.11)	ND(0.11)	0.10	0.02
Barium	16	21	20	0.32@B	0.22@B	0.31@B	19	7
Beryllium	ND(0.023)	ND(0.036)	ND(0.023)	ND(0.091)	ND(0.10)	ND(0.091)	ND(0.036)	--
Cadmium	0.31	0.21@	0.053@	ND(0.051)	ND(0.054)	ND(0.051)	0.19	0.32
Chloride	66[a,b]	74[b]	34[b]	683	867	860	861	248
Chromium	0.56@	ND(0.63)	ND(0.39)	0.42@B	ND(0.43)	ND(0.41)	ND(0.63)	--
Cobalt	ND(0.54)	ND(0.83)	ND(0.52)	ND(0.56)	ND(0.59)	ND(0.56)	ND(0.83)	--
Copper	0.83@	0.69@	0.62@	0.81@B	0.69@B	ND(0.62)	1.2	1.3
Fluoride	16[a,b]	13[b]	13[b]	898	1,052	1,049	1,014	215
Lead	0.14	0.13	0.14	ND(0.17)	ND(0.18)	ND(0.17)	0.13	0.02
Manganese	0.46@	0.37@	0.41@	0.54B	0.77B	1.5B	1.3	1.1
Mercury	0.0083B	0.0089B	0.0056@B	4.6	4.2	4.6	4.5	0.5
Molybdenum	2.1B	2.4@B	2.0B	ND(0.76)	ND(0.81)	ND(0.76)	2.2	0.5
Nickel	0.84	0.40	0.54	0.49@	ND(0.32)	ND(0.30)	0.76	1.2
Phosphorus	14	11@	13	ND(10)	ND(11)	ND(10)	13	4
Selenium	0.081@	0.061@	0.047@	ND(0.14)[c]	ND(0.15)[c]	ND(0.14)[c]	0.063	0.042
Vanadium	0.71@	ND(0.92)	0.60@	ND(0.39)	ND(0.41)	ND(0.39)	ND(0.92)	--

Table 3-2 (Continued)

Substance	Particulate Phase			Vapor Phase			Mean	95% CI
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3		
Other Species								
Antimony	ND(2.9)	ND(4.5)	ND(2.8)	ND(4.0)	ND(4.2)	ND(3.9)	ND(4.5)	--

[a]Reported data are from Run 12 collected on Day 2. Data are shown as Run 1 for simplicity.

[b]Results may be biased high because of suspected condensation of acid gases (HCl, HF, and SO_2) within the sampling probe.

[c]Results for vapor-phase selenium may be biased low based on matrix spike recovery data.

CI = Confidence interval.

@ = Reported concentration is less than five times the detection limit.

ND = Not detected; the detection limit is shown in parentheses.

B = Background levels in the blank were $\geq$ 50% of the sample value.

measured using ICP-AES. Chloride concentrations were measured using ion chromatography, and fluoride levels were measured using an ion-selective electrode. Mercury concentrations were measured by CVAAS.

Six of the target species (barium, chromium, copper, manganese, mercury, and nickel) show measurable concentrations in the vapor phase of one or more test runs. However, background levels associated with the impinger field blanks were similar to the levels found in the samples, except for mercury and nickel. The presence of barium, chromium, copper, and manganese in the vapor phase is unlikely since these are relatively nonvolatile species. The results for molybdenum in the particulate phase are considered to be biased high because of high background levels associated with the quartz filters used to collect the particulate matter. Note that the total particulate loading of 1.8 mg/Nm^3 is very low; therefore, all of the substances present in the stack gas solid phase are at very low levels. Concentrations measured at other FCEM sites have been as high as 100 mg/Nm^3 of particulate. Low levels of particulate increase the uncertainty and make the background levels more significant when quantifying results.

Note that the results for particulate-phase anions in the stack gas are considered to be biased high because of suspected condensation of acid gases (HCl, HF, and SO_2) within the sampling probe. This problem does not affect the reported total concentration (particulate plus vapor) of chloride, fluoride, and sulfur in the stack gas.

Visual inspection of the acetone probe and nozzle rinse (PNR) fractions from the three anion trains (after evaporation of the acetone) revealed an oily, yellow/brown discoloration consistent with the presence of sulfuric acid. In addition, the weight gains for the PNR fraction of the anions trains were all an order of magnitude higher than the weight gains associated with the PNR fraction of the Method 29 multi-metals train. The procedure used at Site 22 for recovery of material from the anions train probe included an initial rinse with acetone to remove residual material, followed by a rinse with the impinger solution (carbonate/bicarbonate solution). It is suspected that residual salts remaining in the anions probe after the probe was rinsed with the impinger solution caused the acid gases present in the flue gas to condense on the inside surface of the probe during subsequent tests. This resulted in abnormally high levels of chloride, fluoride, and sulfate in the front-half fraction of the train (i.e., particulate) and abnormally high overall weight gain within the anions train PNR samples.

The results for polynuclear aromatic hydrocarbons (PAHs) and dioxin/furan compounds are presented in Tables 3-3 and 3-4, respectively. The PAH analyses were conducted using high resolution GC/MS to provide the lowest possible detection limits. PAH compounds were detected in the samples at levels ranging from 0.28 ng/Nm^3 (dibenz[a,i] acridine) to 140 ng/Nm^3 (phenanthrene); however, background levels associated with the sampling media were found to be significant in some cases. The mean background levels detected in the three blank samples (field, trip, and lab method blanks) are shown in Table 3-5 for comparison. Field blanks are resin cartridges that come from a sampling train that has been prepared, leak checked, and deprepped in the same way as the sample trains. A trip blank is an unopened resin cartridge that is brought into the

Table 3-3
Stack Gas PAH Results for Site 22 (ng/Nm 3)

Substance	Run 1	Run 2	Run 3	Sample Mean	95% CI	Blank Mean[a]
Stack Flow Rate (Nm^3/hr)	2,500,000	2,550,000	2,550,000	2,530,000	70,000	
Acenaphthalene	8.3	2.0	1.8	4.0	9.2	0.89
Acenaphthene	10	5.4	6.1	7.2	6.3	2.4
Anthracene	7.7	5.2	3.5	5.5	5.2	1.5
Benzo[a]pyrene	2.4	0.82	0.56	1.3	2.5	0.50
Benzo[b,j&k]fluoranthenes	5.5	2.4	1.8	3.2	4.9	0.81
Benzo[g,h,i]perylene	3.3	2.2	2.3	2.6	1.6	3.2
Benz[a]anthracene	2.2	0.92	0.56	1.2	2.1	0.22
Chrysene	5.1	2.7	1.1	3.0	5.1	0.43
Dibenzo[a,e]pyrene	0.35	0.33	ND(0.35)	ND(0.35)	--	0.85
Dibenzo[a,h]pyrene	ND(0.52)	ND(0.76)	ND(0.63)	ND(0.76)	--	0.82
Dibenzo[a,i]pyrene	ND(0.43)	ND(0.20)	ND(0.63)	ND(0.63)	--	0.69
Dibenz[a,h]acridine	0.41	ND(1.2)	ND(0.95)	ND(1.2)	--	1.2
Dibenz[a,h]anthracene	0.41	0.36	ND(0.39)	ND(0.39)	--	0.66
Dibenz[a,i]acridine	0.98	0.96	0.28	0.74	1.0	2.2
7H-Dibenzo[c,g]carbazole	0.38	ND(0.86)	ND(1.6)	ND(1.6)	--	2.2
Fluoranthene	47	28	10	29	45	4.5
Fluorene	25	13	4.7	14	26	4.1
Indeno[1,2,3-cd]pyrene	1.7	0.59	0.81	1.0	1.4	0.74
5-Methyl chrysene	ND(0.22)	ND(0.56)	ND(0.18)	ND(0.56)	--	ND(0.50)
Phenanthrene	140	82	23	82	146	15
Pyrene	36	17	5.3	19	39	3.0

[a] Calculation based on the average mass detected in the three blanks (field, trip, and lab) and the average gas sample volume for the three samples (3.17 Nm^3).

CI = Confidence interval.

Table 3-4
Stack Gas Dioxin and Furan Results for Site 22 (pg/Nm^3)

Substance	Run 1	Run 2	Run 3	Sample Mean	95% CI	Blank Mean[a]
Stack Flow Rate (Nm^3/hr)	2,500,000	2,550,000	2,550,000	2,530,000	70,000	
Dioxin Compounds						
2,3,7,8-TCDD	ND(2.9)	ND(3.9)	ND(2.1)	ND(3.9)	--	ND
Total TCDD	12.7 ND[b]	6.5 ND	5.6	5.6	--	3
1,2,3,7,8-PeCDD	ND(1.5)	ND(4.6)	ND(2.5)	ND(4.6)	--	ND
Total PeCDD	ND	ND	ND	ND	--	ND
1,2,3,4,7,8-HxCDD	ND(3.4)	ND(5.7)	ND(3.1)	ND(5.7)	--	ND
1,2,3,6,7,8-HxCDD	ND(5.0)	ND(2.8)	ND(2.2)	ND(5.0)	--	ND
1,2,3,7,8,9-HxCDD	ND(2.9)	ND(5.0)	ND(2.5)	ND(5.0)	--	ND
Total HxCDD	ND	ND	ND	ND	--	ND
1,2,3,4,6,7,8-HpCDD	4.5	9.2	ND(9.1)	6.1	6.9	6
Total HpCDD	9.7	18.1	7.0	12	14	11
OCDD	47.2	74.7	62.8	62	34	38
Furan Compounds						
2,3,7,8-TCDF	ND(1.5)	ND(4.3)	ND(2.6)	ND(4.3)	--	ND
Total TCDF	2.1	ND	12.6	7.3	67	ND
1,2,3,7,8-PeCDF	ND(1.4)	ND(2.6)	1.8	ND(2.6)	--	ND
2,3,4,7,8-PeCDFe	ND(1.4)	ND(5.0)	ND(1.8)	ND(5.0)	--	ND
Total PeCDF	10.0	11.4	4.5	8.6	9.0	ND
1,2,3,4,7,8-HxCDF	ND(3.4)	ND(7.8)	ND(4.2)	ND(7.8)	--	ND
1,2,3,6,7,8-HxCDF	ND(9.4)	ND(14)	ND(10)	ND(14)	--	ND
1,2,3,7,8,9-HxCDF	ND(3.7)	ND(3.0)	ND(4.9)	ND(4.9)	--	ND
2,3,4,6,7,8-HxCDF	ND(3.4)	ND(5.3)	ND(4.2)	ND(5.3)	--	2
Total HxCDF	2.9	5.3	4.2	4.1	3.0	5
1,2,3,4,6,7,8-HpCDF	2.4	ND(5.0)	3.0	ND(5.0)	--	4
1,2,3,4,7,8,9-HpCDF	ND(2.2)	ND(5.3)	ND(3.8)	ND(5.3)	--	ND
Total HpCDF	2.4	ND	3.0	2.7	3.8	4
OCDF	3.7	6.8	4.5	5.0	4.0	4

[a] Calculated based on the average mass detected in the three blanks (field, trip, and lab) and the average gas sample volume for the three samples (3.17 Nm^3).

[b] ND = Not detected; Method 23 does not specify how to determine detection limits for the total congener class. Detection limits can only be calculated for individual congeners within each class.

CI = Confidence interval.

Table 3-5
Emission Factors for Inorganic Substances (lb/trillion Btu unless noted)

Substance	Mean	95% CI
Stack Flow Rate (Nm^3/hr)	2,530,000	70,000
Coal Flow Rate (lb/hr, dry)	553,000	26,000
Heating Value (Btu/lb)	11981	227
Particulate (lb/Million Btu)	0.0015	0.0009
Target Species		
Arsenic	0.087	0.019
Barium	16	6
Beryllium	ND(0.031)	--
Cadmium	0.16	0.27
Chloride	726	215
Chromium	ND(0.53)	--
Cobalt	ND(0.70)	--
Copper	1.0B	1.1
Fluoride	855	192
Lead	0.11	0.01
Manganese	1.1B	0.9
Mercury (CVAAS)	3.8	0.4
Mercury (CVAFS)	6.7	2.2
Molybdenum	1.9B	0.4
Nickel	0.64	1.1
Phosphorus	11	3
Selenium	0.053	0.036
Vanadium	ND(0.78)	--
Other Species		
Aluminum	136	17
Antimony	ND(3.8)	--
Calcium	325	37
Iron	52	16
Magnesium	47	14
Potassium	ND(82)	--
Sodium	86B	15
Sulfur (lb/million Btu)	0.43	0.04
Titanium	12	2

CI = Confidence interval.

B = Background levels were ≥ 50% of the total (front half and back half) sample value.

field and returned with the samples to the laboratory. Given the variability in the measured concentrations and the measured blank levels, the quantitation of the compounds detected in the flue gas samples is highly uncertain (refer to Section 4 and Appendix F for a detailed discussion of the blank sample results for organic compounds). Eleven of the 21 PAH compounds were detected in all three samples at levels greater than the mean blank value. This means that the compounds were present; however, the quantitation is suspect.

Some dioxin and furan compounds and congeners were detected in the stack gas samples at low levels (near the detection limit); however, for most of the compounds detected, the background levels associated with the blanks (field, trip, and laboratory) were similar to the levels found in the samples (refer to Section 4 and Appendix F for a detailed discussion of the blank results for organic compounds). Compounds or congeners detected in one or more stack gas samples but not detected in the blank samples include: tetrachlorodibenzofuran compounds; 1,2,3,7,8-pentachlorodibenzofuran; and total pentachlorodibenzofuran compounds. 2,3,7,8-tetrachlorodibenzo-p-dioxin was not detected at a level of 3.9 pg/Nm^3.

Tables 3-5, 3-6, and 3-7 present the emission factors, on a unit energy basis, for the inorganic, PAH and dioxin/furan analytes, respectively. Mean particulate matter emissions were 0.0015 lb/million Btu. Chloride (726 lb/trillion Btu) and fluoride (855 lb/trillion Btu) have the highest emission factors, which is expected because the vapor-phase species (HCl and HF) are not effectively removed by the ESP, and because the concentrations of chloride and fluoride in the coal are higher than those of the other target species. The lowest emission factors were obtained for the dioxin/furan compounds (0.00001 to 0.0001 lb/trillion Btu), when detected.

ESP Performance

Table 3-8 shows the estimated ESP removal efficiencies for removing the target species. Measurements were not made on the ESP inlet gas; therefore, the mass rates of substances entering the ESP were assumed to be equal to the mass rates of the substances in the coal for chloride, fluoride, mercury, and sulfur. For the other (less volatile) elements and total ash, 80% of the mass rate in the coal was assumed for the ESP inlet mass rate, which is consistent with an 80:20 fly ash-to-bottom ash ratio. This is a typical design value for wall-fired boilers.

Table 3-6
Emission Factors for PAH Compounds (lb/trillion Btu unless noted)

Substance	Sample Mean	Sample 95% CI	Blank Mean	Blank 95% CI
Stack Flow Rate (Nm^3/hr)	2,530,000	70,000		
Coal Flow Rate (lb/hr, dry)	553,000	26,000		
Heating Value (Btu/lb)	11,981	227		
PAH Compounds				
Acenaphthalene	0.0034	0.0078	0.00075	0.00091
Acenaphthene	0.0060	0.0053	0.0020	0.0010
Anthracene	0.0046	0.0044	0.0012	0.0008
Benzo[a]pyrene	0.0011	0.0021	0.00043	0.00063
Benzo[b,j&k]fluoranthenes	0.0027	0.0042	0.00068	0.00053
Benzo[g,h,i]perylene	0.0022	0.0013	0.0027	0.0020
Benz[a]anthracene	0.0010	0.0018	0.00019	0.00028
Chrysene	0.0025	0.0042	0.00036	0.00016
Dibenzo[a,e]pyrene	ND(0.00030)	NC	0.00072	0.00004
Dibenzo[a,h]pyrene	ND(0.00064)	NC	0.00069	0.00004
Dibenzo[a,i]pyrene	ND(0.00053)	NC	0.00059	0.00003
Dibenz[a,h]acridine	ND(0.0010)	NC	0.0010	0.0012
Dibenz[a,h]anthracene	ND(0.00033)	NC	0.00056	0.0008
Dibenz[a,i]acridine	0.00062	0.00083	0.0019	0.0013
7H-Dibenzo[c,g]carbazole	ND(0.0013)	NC	0.0018	0.0001
Fluoranthene	0.024	0.038	0.0038	0.0037
Fluorene	0.012	0.022	0.0034	0.0027
Indeno[1,2,3-cd]pyrene	0.0086	0.00	0.00062	0.0005
5-Methyl chrysene	ND(0.00047)	NC	ND(0.00042)	NC
Phenanthrene	0.069	0.12	0.013	0.015
Pyrene	0.016	0.033	0.0025	0.0036

CI = Confidence interval.

Table 3-7
Emission Factors for Dioxin/Furan Compounds (lb/trillion Btu unless noted)

Substance	Sample Mean	Sample 95% CI	Blank Mean	Blank 95% CI
Stack Flow Rate (Nm^3/hr)	2,530,000	70,000		
Coal Flow Rate (lb/hr, dry)	553,000	26,000		
Heating Value (Btu/lb)	11,981	227		
Dioxin Compounds				
2,3,7,8-TCDD	ND(3.3E-06)	--	ND	--
Total TCDD	4.7E-06	NC	2.4E-06	1.4E-07
1,2,3,7,8-PeCDD	ND(3.9E-06)	--	ND	--
Total PeCDD	ND	--	ND	--
1,2,3,4,7,8-HxCDD	ND(4.8E-06)	--	ND	--
1,2,3,6,7,8-HxCDD	ND(4.2E-06)	--	ND	--
1,2,3,7,8,9-HxCDD	ND(4.2E-06)	--	ND	--
Total HxCDD	ND	--	ND	--
1,2,3,6,7,8-HpCDD	ND(7.7E-06)	--	4.8E-06	5.7E-07
Total HpCDD	9.8E-06	3.0E-05	9.5E-06	2.1E-06
OCDD	5.2E-05	7.2E-05	3.2E-05	1.9E-06
Furan Compounds				
2,3,7,8-TCDF	ND(3.6E-06)	--	ND	--
Total TCDF	6.2E-06	1.4E-04	ND	--
1,2,3,7,8-PeCDF	ND(2.2E-06)	--	ND	--
2,3,4,7,8-PeCDF	ND(4.2E-06)	--	ND	--
Total PeCDF	7.3E-06	1.9E-05	ND	--
1,2,3,4,7,8-HxCDF	ND(6.6E-06)	--	ND	--
1,2,3,6,7,8-HxCDF	ND(1.2E-05)	--	ND	--
1,2,3,7,8,9-HxCDF	ND(4.1E-06)	--	ND	--
2,3,4,6,7,8-HxCDF	ND(4.5E-06)	--	1.8E-06	1.1E-07
Total HxCDF	3.5E-06	6.4E-06	4.1E-06	4.2E-06
1,2,3,4,6,7,8-HpCDF	ND(4.2E-06)	--	3.5E-06	4.4E-06
1,2,3,4,7,8,9-HpCDF	ND(4.5E-06)	--	ND	--
Total HpCDF	2.2E-06	8.1E-06	3.5E-06	4.4E-06
OCDF	4.2E-06	8.3E-06	3.6E-06	4.1E-07

CI = Confidence interval.

Table 3-8
Estimated ESP Removal Efficiency at Site 22[a]

Substance	Removal (%)	95% CI About Mean
Particulate Matter	99.97	0.02
Target Species		
Arsenic	99.85	0.03
Barium	99.94	0.02
Beryllium	>99.9	--
Cadmium	NC[b]	--
Chloride	NC	--
Chromium	>99.8	--
Cobalt	>99.3	--
Copper	99.9[c]	0.1
Fluoride	81	4
Lead	99.99	0.01
Manganese	99.7[c]	0.4
Mercury (DGA/CVAAS)	67	6
Mercury (CVAFS)	0[d]	28
Molybdenum	98.7[c]	0.6
Nickel	99.8	0.4
Phosphorus	99.94	0.02
Selenium	99.90	0.07
Vanadium	>99.9	--
Other Species		
Aluminum	99.96	0.01
Antimony	>56	--
Calcium	99.94	0.01
Iron	99.97	0.01
Magnesium	99.88	0.03
Potassium	>99.9	--
Sodium	99.78[c]	0.04
Sulfur	0[d]	14
Titanium	99.96	0.01

[a]No measurements were made at the ESP inlet. Instead, the ESP inlet flow rates were estimated from the coal measurements. For total ash and the less volatile elements (all except Cl F, S, and Hg), the ESP inlet rate was assumed to be 80% of the mass rate in the coal. For Cl, F, S, and Hg, the ESP inlet rate was assumed to be 100% of the mass rate in the coal.

[b]NC = Not calculated. Substance not detected in the coal.

[c]Removal is considered to be biased slightly low because of high background levels associated with the stack gas sample.

[d]Calculated control efficiencies are negative but are shown as zero.

CI = Confidence interval.

4

DATA EVALUATION

Several procedures can be used to evaluate the information developed during a field sampling program. In the case of Site 22, three methods were used to evaluate data quality. First, the process data were examined to determine if the unit was operating at normal, steady-state conditions during the sampling periods. Second, the QA/QC protocol for the sampling and analytical procedures used at Site 22 (i.e., equipment calibration and leak checks, duplicates, blanks, spikes, standards, etc.) were evaluated. Site 22 QA/QC data were compared with FCEM project objectives. The third data evaluation tool involves calculating material balances for various substances around the entire plant. Since material balances involve the summation and comparison of mass flow rates in several streams (often sampled and analyzed by different methods), good agreement, i.e., closure within an acceptable range, can be used as an indicator of accurate results for streams that contribute significantly to the overall inlet and outlet mass rates (e.g., coal, bottom ash, ESP ash, etc.).

Process Operation

Process data were examined to ensure that unit operation was stable during the sampling periods. Measurements were available from three sources: 1) the plant's continuous emission monitoring system (CEM); 2) flue gas sampling data sheets; and 3) plant process flow meters. The key parameters are shown in Table 4-1. The coefficients of variation (CV, the standard deviation divided by the mean) were calculated to determine process variability. In addition, Appendix G contains process trend plots.

It was originally planned to collect process data using the plant's computerized data acquisition system (DAS); however, an electrical problem in the DAS prevented the automated collection of process data. Instead, the values for most key process parameters were logged manually each hour from the CEM system. Coal feed rates were logged hourly from the flow meters on each of the seven gravimetric coal feeders, and economizer outlet oxygen data were monitored hourly from the digital meters on the A- and B-side outlet ducts.

No major process upsets were encountered during the sampling effort. The unit maintained steady, full-load operation throughout each of the test runs. Boiler operation was stable, as indicated by the low CVs for the gross load, coal feed rate, and economizer outlet O_2 concentration. The ESPs were performing well, maintaining opacities in the stack well below permit levels, and opacity CVs were less than 20 percent indicating minimal variation in performance. Stack gas SO_2 levels measured by the plant's CEM system (average = 309 ppmv) agree with the anions train measurement data within 15

Table 4-1
Process Data Summary for Site 22

	7/13/93		7/14/93		7/15/93	
	Mean	CV (%)	Mean	CV (%)	Mean	CV (%)
Unit Load (MW)[a]	700.3	0.2	703.1	0.2	701.5	0.2
Coal Feed Rate (tons/hr, wet)[b]	382	2	398	1	397	1
Economizer Outlet O_2 (%)[a]	2.2	7.9	2.6	5.9	2.4	6.7
Stack Opacity (%)[c]	6.0	2.9	6.3	5.1	6.0	6.5
Stack Temperature (°F)[d]	292	2	302	1	299	1
Stack SO_2 (ppmv)[c]	281	5	327	3	319	3
Stack NO_x (ppmv)[c]	160	7	165	4	159	7
Stack CO_2 (%)[c]	11.3	9.4	10.9	1.0	11.2	11.5
Stack Flow Rate (kscfm)[c]	1,630	1	1,648	1	1,640	1

[a]Data collected from plant control room meters.

[b]Data collected from digital flow meters on the gravimetric feeders.

[c]Data collected from the plant's CEM system.

[d]Data are from stack gas sampling data sheets.

CV = Coefficient of variation.

percent. Stack gas flow rates measured by the CEM system (average = 1,640 kscfm) agree with the average flow rate determined from the sampling trains within 3 percent.

Sample Collection

Several factors indicate acceptable sample collection. Key components of the sampling equipment--pitot tubes, thermocouples, orifice meters, dry gas meters, and sampling nozzles--were calibrated before use in the field, and those calibrations were checked at the end of sampling. These calibrations are on file at Radian Corporation. The methods used to collect metal and anion samples were comparable to those used at other FCEM sites sampled by Radian. The sampling runs were well documented, and all flue gas samples were collected at rates of between 90 and 110% of the isokinetic rates, except for the Run 1 anions sample, which was considered to be invalid and was not analyzed. Sufficient data were collected using standard sampling and analysis methods to ensure acceptable data completeness and the comparability of the measurements.

Coal samples are considered to be representative of the coal fired during flue gas sampling. Coal samples were collected from the six-foot-wide belt that conveys coal into the top of the storage bunkers for each of the seven coal mills. The residence time is approximately eight hours from the sample point to the boiler; therefore, coal sampling was started approximately eight hours before flue gas sampling to account for this lag time. Samples were collected using the plant's dedicated auto sampling system, which is designed to collect samples according to standard industry ASTM specifications.

The measured flow rates of flue gas and coal agree with a combustion calculation that uses the mean coal composition, the mean coal flow rate, and the mean oxygen concentration in the stack gas to predict a "theoretical" flue gas flow rate. This calculated flow rate agreed with the measured stack flow rate within approximately 10 percent.

Analytical Quality Control Results

Generally, the type of quality control information obtained pertains to measurement precision, accuracy (which included precision and bias), 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 the 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 placing an estimate of confidence 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.
Surrogate-spiked sample sets	Analytical variability in the sample matrix but at an established concentration.
Accuracy (including 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.
Surrogate-spiked samples	Analyte recovery in the sample matrix, to the extent that the surrogate compounds are chemically similar to the compounds of interest. Primarily used as indicator of analytical efficacy.
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.
Trip blank	Blank effects arising from sample transport and storage. Typically used only for volatile organic compound analyses.
Method blank	Blank effects inherent in the 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 presents 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 the precision and accuracy estimates. Most of the quality control results met the project objectives.

However, the quality control data revealed the following potential problems:

- The recovery of copper from the SARM 20 coal standard measured by INAA could not be determined. In addition, detection limits for INAA were higher than those for ICP-AES, resulting in copper concentrations near the detection limit. Therefore, the ICP-AES data for copper were chosen as the primary values.

- The recovery of arsenic in the SARM 20 coal was 43% which is outside the 75-125% accuracy objective. This indicates a possible low bias for arsenic in the coal when measured by GFAAS; therefore, INAA data were selected as the primary values for arsenic.

- The recovery of arsenic (135%) in the spiked fly ash measured by GFAAS was outside the project objectives (75-125%). This indicates the possibility of a slightly high bias for arsenic in the fly ash; however, the precision of the alternate method (ICP-AES) for measurement of arsenic in fly ash was poor (38% RPD compared to 20% RPD objective). Therefore, although possibly biased high, the GFAAS data were reported for arsenic.

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
Stack Gas													
Metals - Vapor Phase	✓			✓	✓	✓		✓		✓	✓	✓	
Metals - Solid Phase	✓				✓			✓	✓	✓	✓	✓	
Anions - Vapor Phase	✓			✓	✓	✓		✓		✓		✓	
Anions - Solid Phase	✓				✓			✓		✓		✓	
PAHs	✓						✓	✓		✓	✓	✓	
PCDD/PCDF	✓						✓	✓		✓	✓	✓	
Coal													
Metals	✓		✓						✓				
Anions	✓		✓	✓	✓	✓		✓				✓	
Ultimate/Proximate	✓		✓										
Bottom Ash and Fly Ash													
Metals	✓		✓	✓	✓	✓		✓	✓			✓	
Anions	✓		✓	✓	✓	✓		✓				✓	

Table 4-4
Summary of Precision and Accuracy Estimates

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Coal by INAA (NC State)	Precision - Duplicate Samples Accuracy - Standard Reference Material				
Antimony		20	75-125	4.6	106
Arsenic		20	75-125	0.86	93.1
Cadmium		20	75-125	NC	NC
Chromium		20	75-125	1.7	113
Cobalt		20	75-125	0.84	104
Copper		20	75-125	18	NC
Manganese		20	75-125	5.4	96.8
Nickel		20	75-125	14	95.8
Selenium		20	75-125	15	120
Aluminum		20	75-125	4.0	97.6
Barium		20	75-125	2.4	112
Calcium		20	75-125	2.9	137
Iron		20	75-125	1.2	102
Magnesium		20	75-125	7.3	99.5
Mercury		20	75-125	32[a]	115
Molybdenum		20	75-125	5.4	108
Sodium		20	75-125	0.97	107
Vanadium		20	75-125	4.3	98.7
Metals in Coal by XRF (WAL)	Precision - Duplicate Samples Accuracy - Standard Reference Material				
Copper		20	75-125	8.5	133[a]
Lead		20	75-125	17	111
Manganese		20	75-125	1.3	86
Nickel		20	75-125	NA	104
Phosphorus		20	75-125	2.3	69
Selenium		20	75-125	NA	NA
Titanium		20	75-125	3.8	90

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Coal by ICP-AES (CT&E)	Precision - Not Determined Accuracy - Standard Reference Material				
Beryllium		20	75-125	NA	104
Chromium		20	75-125	NA	99
Copper		20	75-125	NA	89
Manganese		20	75-125	NA	101
Nickel		20	75-125	NA	96
Phosphorus		20	75-125	NA	110
Titanium		20	75-125	NA	95
Metals in Coal by ICP-AES (WAL)	Precision - Duplicate Samples Accuracy - Standard Reference Material				
Beryllium		20	75-125	17	92
Cadmium		20	75-125	NA	NA
Chromium		20	75-125	NA	97
Metals in Coal by GFAAS (CT&E)	Precision - Not Determined Accuracy - Standard Reference Material				
Arsenic		20	75-125	--	43
Cadmium		20	75-125	--	NA
Lead		20	75-125	--	85
Selenium		20	75-125	--	NC
Metals in Coal by CVAAS (CT&E)	Precision - Not Determined Accuracy - Standard Reference Material				
Mercury		20	75-125	--	116
Metals in Coal by GFAAS, CVAAS (WAL)	Precision - Duplicate Samples Accuracy - Standard Reference Material				
Arsenic		20	75-125	NA	53[a]
Mercury		20	75-125	12	92
Metals in Ashes - GFAAS/CVAAS	Precision - Duplicate Samples Accuracy - Matrix Spikes				
Arsenic		20	75-125	0.8	126[a]
Cadmium[b]		20	75-125	0.9	114

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Lead [b]		20	75-125	2.8	106
Mercury [b]		20	75-125	5.0	109
Nickel		20	75-125	0.9	106
Selenium [b]		20	75-125	6.2	96
Anions in Ashes	Precision - Duplicate Samples Accuracy - Matrix Spikes				
Chloride		20	75-125	0	84
Fluoride		20	75-125	18	86
Metals in Ashes - ICP-AES	Precision - Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Antimony		20	75-125	9.1	81
Arsenic		20	75-125	38 [a]	85
Beryllium		20	75-125	2.0	89
Chromium		20	75-125	3.3	91
Cobalt		20	75-125	1.6	92
Copper		20	75-125	15	95
Manganese		20	75-125	3.1	90
Nickel		20	75-125	9.1	89
Selenium		20	75-125	3.1	NC
Aluminum		20	75-125	38 [a]	113
Barium		20	75-125	113 [a]	65 [a]
Cadmium		20	75-125	21 [a]	94
Calcium		20	75-125	39 [a]	114
Iron		20	75-125	7.6	91
Lead		20	75-125	2.8	104
Molybdenum		20	75-125	36 [a]	116
Potassium		20	75-125	4.8	91
Sodium		20	75-125	37 [a]	115
Titanium		20	75-125	59 [a]	86
Vanadium		20	75-125	3.3	91

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Stack Gas Vapor Phase - GFAAS/CVAAS	Precision - Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Arsenic		20	75-125	6.0	101
Cadmium		20	75-125	3.0	100
Lead		20	75-125	6.0	87
Mercury		20	75-125	2.0	95
Nickel		20	75-125	2.9	102
Selenium		20	75-125	NC	NC
Metals in Stack Gas Vapor Phase - ICP-AES	Precision - Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Antimony		20	75-125	0	96
Arsenic		20	75-125	2.0	99
Beryllium		20	75-125	1.0	101
Chromium		20	75-125	1.0	97
Cobalt		20	75-125	1.0	95
Copper		20	75-125	1.0	97
Manganese		20	75-125	1.0	95
Nickel		20	75-125	2.0	97
Selenium		20	75-125	4.0	105
Aluminum		20	75-125	0	93
Barium		20	75-125	4.0	95
Cadmium		20	75-125	2.0	98
Calcium		20	75-125	1.0	99
Iron		20	75-125	1.0	98
Lead		20	75-125	4.0	93
Magnesium		20	75-125	1.0	93
Molybdenum		20	75-125	2.0	94
Potassium		20	75-125	1.0	96
Sodium		20	75-125	1.0	87
Titanium		20	75-125	1.0	97

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Vanadium		20	75-125	1.0	98
Metals in Stack Gas Outlet Solid Phase - GFAAS/CVAAS	Precision - Replicate Runs [c] Accuracy - Standard Reference Material				
Arsenic		20	75-125	4.2	85
Cadmium		20	75-125	19	86
Lead		20	75-125	1.1	72
Mercury		20	75-125	3.4	113
Nickel		20	75-125	0.7	95
Selenium		20	75-125	18	100
Metals in Stack Gas Solid Phase - ICP-AES	Precision - Spike Duplicate Accuracy - Standard Reference Material				
Antimony		20	75-125	9.4	93
Arsenic		20	75-125	13	93
Beryllium		20	75-125	1.6	93
Chromium		20	75-125	2.2	94
Cobalt		20	75-125	1.6	91
Copper		20	75-125	3.6	96
Manganese		20	75-125	2.7	94
Nickel		20	75-125	1.7	92
Selenium		20	75-125	58 [a]	232 [a]
Aluminum		20	75-125	1.0	93
Barium		20	75-125	2.2	94
Cadmium		20	75-125	64 [a]	65 [a]
Calcium		20	75-125	1.0	98
Iron		20	75-125	2.2	92
Lead		20	75-125	11	104
Magnesium		20	75-125	1.6	94
Molybdenum		20	75-125	4.6	93
Potassium		20	75-125	2.1	94
Sodium		20	75-125	2.7	93

Table 4-4 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Vanadium		20	75-125	1.6	94
Anions in Coal	Precision - Replicate Runs[b] Accuracy - Matrix Spikes				
Chloride		20% CV	75-125	20% CV	96
Fluoride		20% CV	75-125	9.2% CV	125
Anions in Stack Gas Vapor Phase	Precision - Matrix-Spiked Duplicates Accuracy - Matrix Spike				
Chloride		20	75-125	3.0	82
Fluoride		20	75-125	4.0	103
Sulfate		20	75-125	12.0	97
Anions in Stack Gas Solid Phase	Precision - Matrix-Spiked Duplicates Accuracy - Matrix Spike				
Chloride		20	75-125	9.0	104
Fluoride		20	75-125	4.0	103
Sulfate		20	75-125	2.0	100
PAH Analysis by HRGCMS (XAD)	Precision - Surrogate Spike Recovery Accuracy - CV of Surrogate Spike Recovery				
Biphenyl - d10		40 CV	50-150	14 CV	85
Hexachlorobenzene		40 CV	50-150	24 CV	67
Perylene - d12		40 CV	50-150	37 CV	99
PCDD/PCDF Analysis by HRGCMS (XAD)	Precision - Surrogate Spike Recovery Accuracy - CV of Surrogate Spike Recovery				
2,3,7,8-TCDD-$^{37}Cl_4$		25 CV	70-130	4.7 CV	91
2,3,4,7-PeCDF-^{13}C		25 CV	70-130	6.2 CV	82
1,2,3,4,7,8-HxDCF-^{13}C		25 CV	70-130	21 CV	103
1,2,3,4,7,8-HxDCC-^{13}C		25 CV	70-130	0.84 CV	95
1,2,3,4,7,8,9-HpCDF-^{13}C		25 CV	70-130	4.4 CV	75

Table 4-4 (Continued)

[a] Outside the project objectives.

[b] Matrix spike duplicate data were used to estimate precision for these analytes.

[c] Replicate runs include components of process, sampling, and analytical variability. The precision objective is based only on expected analytical variability.

NA = Not available.

NC = Not calculated; analyte concentration below reporting limit in two or more runs.

RPD = Relative percent difference.

CV = Coefficient of variance.

- The matrix spike results for selenium in the stack gas stream vapor phase measured by GFAAS showed an extremely poor recovery (0% compared to 75-125% specification). ICP-AES matrix spike/matrix-spike duplicate (MS/MSD) analytical results showed a recovery within the specification (105%) and good precision (4% RPD compared to a project specification of 20%). However, the detection limit of ICP-AES is much higher than the GFAAS detection limit, and selenium was not detected in the samples by ICP-AES analysis; therefore, the GFAAS data are used for selenium.

- The GFAAS MS/MSD analytical results for lead in the stack gas solid-phase stream showed slightly low recovery (72%) compared to the project specification. The precision was acceptable (1.1% RPD compared to the 20% RPD specification). This indicates the possibility of a slightly low bias in the results for lead in the stack gas solid phase.

- The ICP-AES recoveries of nickel (71%) and selenium (58%) in the stack gas solids were below the project specifications (75-125%); therefore, GFAAS data for these metals were selected for the material balance.

- For measurements by ICP-AES, GFAAS, and CVAAS, most blank results demonstrated either no contamination, contamination in concentrations less than five times the detection limit, or contamination in concentrations significantly below that found in the corresponding samples. The levels of silicon found in the ash method blank and the stack solids laboratory blank are an artifact of the extraction process; therefore, the silicon levels in these samples are biased high. The stack gas trip blank concentration for silicon is high because the digestion of a quartz filter and similar levels of silicon were found in the samples associated with this blank. Levels of aluminum, barium, calcium, iron, manganese, silicon, sodium, and zinc, were found in the field blank associated with the stack gas vapor-phase stream in concentrations similar to those found in the associated samples; however, all sample levels were extremely low and within five times the detection limit, with the exception of sodium. The trip blank filter associated with ICP-AES metals contained molybdenum at levels similar to those observed in the stack gas solid samples.

- For PAH measurements by HRGCMS, the method, trip, and field blanks contained nearly all of the analytes at levels that would bias the results slightly high.

- For PCDD/PCDF measurements by HRGCMS, the method, trip, and field blanks contained anywhere from two to four analytes at levels near their detection limits. These results should not significantly affect the data set.

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 how well a value generated by a specific procedure conforms to the assumed or accepted true value, and 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 sampling, produce only estimates of the 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 the degree to which 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 to 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 amount of valid data.

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

Metals

Precision. Duplicate samples were used to estimate the precision, including a component of sampling variability, of metals analyses of coal samples by INAA. The results for 16 of the 17 analytes met the precision objective of 20% RPD. The INAA results for mercury in the coal had a 31.8% RPD, compared with the project objective of 20 percent. The concentration of mercury in the coal as determined by CVAAS was used in the material balance. All precision estimates of metal concentrations in the coal as measured by XRF were within the specifications. The precision of the GFAAS analytical results for arsenic in the coal was not determined since arsenic was not detected in the coal in concentrations above the detection limit of the method.

For the stack gas metals in the vapor phase analyzed by ICP-AES and GFAAS/CVAAS, precision was estimated by analyzing matrix spike duplicate samples. The results for all 20 metals met the precision objectives for the ICP-AES analyses. The results for five of six metals determined by GFAAS/CVAAS also met the precision objectives. It was not possible to estimate the precision of the selenium results obtained by GFAAS since this metal was not detected in the matrix spike and matrix spike duplicate samples.

The precision of the stack gas metal solid-phase results obtained by ICP-AES, GFAAS, and CVAAS was estimated by analyzing spike duplicate samples. The results for 18 of the 20 metals measured by ICP-AES met precision objectives. Cadmium (RPD 64%) and selenium (58%) were the exceptions. The GFAAS concentrations of these metals were used for the material balance. The results for all six metals analyzed by GFAAS/CVAAS had a variability below the 20% precision objective.

Duplicate matrix spike samples were used to estimate the precision of analyses of the fly ash and bottom ash samples for metals by ICP-AES. The results for 17 of 20 metals had a precision within the project objectives. The exceptions were arsenic (38% RPD), aluminum (38% RPD), and sodium (37% RPD), which were higher than the precision objective of 20 percent. The GFAAS result for arsenic was reported.

Accuracy. The accuracy of the coal sample metals analyses was estimated using standard reference coal samples. Of the metals analyzed by GFAAS, CVAAS, ICP-AES, XRF, and INAA, all met the accuracy objective except for arsenic by GFAAS (53% recovery), calcium by INAA (137% recovery), and copper by XRF (133% recovery), compared to a project objective of 75-125%). The INAA data for the arsenic concentration in the coal was selected for material balance calculations.

Matrix spikes were used to estimate the accuracy of the flue gas vapor-phase metals analyses by ICP-AES, GFAAS, and CVAAS. Of the samples analyzed by GFAAS/CVAAS, five of the six metal results met the accuracy objective. For selenium, the recovery of 0% by GFAAS was, of course, below the specifications; however, the accuracy of the ICP-AES selenium measurement was within the accuracy objective of 75-125 percent. Therefore, the ICP-AES selenium value was chosen as the primary value. All samples analyzed by ICP-AES met the accuracy objectives of the project.

The accuracy of flue gas particulate-phase metals analyses was estimated using analytical spike recoveries. The results show that the recoveries of five of the six metals analyzed by GFAAS and CVAAS met the 75-125% accuracy objective. For lead, the recovery of 72% was slightly below the objective. The accuracy of the ICP-AES metals analyses for flue gas particulate-phase samples was estimated using a standard reference material (NIST 1633a fly ash). The results for 17 of the 20 metals met the accuracy objectives. The results for nickel (71% recovery), cadmium (65% recovery), and selenium (0% recovery) had accuracies below the 75-125% project objectives. GFAAS analytical results for these metals met the accuracy objectives; therefore, the GFAAS values were chosen as the primary values for these metals.

Matrix spikes were used to estimate the accuracy of fly ash and bottom ash metals analyses. The results for five of the six metals measured by GFAAS/CVAAS met the accuracy objectives, the results for arsenic (126% recovery) being the exception. The results for all metals measured by ICP-AES met the project objectives, except for barium (65% recovery, compared to a project objective of 75-125%). The spike level for barium in coal was eight times less than the barium concentration in the native sample; therefore, these recovery data may not be useful.

Blank Effects. Most blanks analyzed by ICP-AES, GFAAS, or CVAAS showed no contamination, contamination at levels less than five times the detection limit, or contamination at concentrations significantly below that found in the corresponding samples. Silicon was found in the ash and stack solids method blanks. High concentrations of silicon found in the trip blank were presumably due to the digestion of a quartz filter. Similar levels were found in the associated samples. The levels of manganese, silicon, sodium, and zinc found in the field blank associated with the stack gas vapor-phase stream were similar to those of the corresponding samples. The reagent blank filters associated with the stack gas particulate phase contained molybdenum in levels similar to those observed in the samples.

Anions

Precision. The precision of anion coal and ash sample analyses was estimated using duplicate analyses. The precision estimates for both chloride and fluoride in ash samples met the objective of 20% RPD, as did the precision estimates for chloride, fluoride, and sulfur in the coal. Anion chloride, fluoride, and sulfate analytical results for the stack gas solid- and vapor-phase streams met the precision objectives.

Accuracy. Matrix spikes were used to estimate the accuracy of anion ash and stack gas analyses. Except for the analysis of fluoride in fly ash, all analytical results met the 75-125% recovery objective. The recovery of fluoride in the ash was below the project objective (66% compared to 75-125%). Coal chloride, fluoride, and sulfur recoveries were not estimated.

Blank Effects. Field blank impinger solutions and probe and nozzle rinses were analyzed for chloride, fluoride, and sulfate. The concentrations of these anions were above reporting limits in all of the blanks, and well below the levels observed in some of the samples. The levels of chloride, fluoride, and sulfate found in the field blank for the stack gas solid phase were 3-4%, 0.6-0.7%, and 0.006% of the concentrations in the samples, respectively; therefore, these data should not be affected by the blank levels. The field blank associated with the vapor-phase samples contained all of the anions at levels much lower than those found in the vapor-phase samples. The method blanks associated with the stack gas samples showed fluoride at levels less than five times the detection limit; much higher levels of fluoride were observed in the associated samples. Fluoride was also observed in the method blank associated with the ash samples at a level near the detection limit, but at a higher concentration than those observed in the samples. Therefore, the results for fluoride in ash samples may be biased high.

Polycyclic Aromatic Hydrocarbons (PAHs)

Precision. The precision of the PAH analytical results was evaluated using the coefficients of variation for the surrogate spiked samples, which were below the project limits of 40 percent.

Accuracy. The accuracy of the PAH analytical results was evaluated using analytical spike and surrogate spike recoveries. All recoveries were within the project specification of 50-150 percent.

Blank Effects. Table F-1 contains the blank results for the PAH analyses. Many low-level measurements were reported for the trip, field, and method blanks. Concentrations ranged from 0.4-44.5 ng for the compounds in the method blank and 1.2-72.2 ng for the compounds in the trip blank. Field blank results ranged from 1.0-27.7 ng. For the compounds found in the blanks, the results of the PAH analyses of stack gas samples are biased high.

Dioxins and Furans

Precision. The precision of the PCDD/PCDF analyses was estimated using the coefficients of variation of the surrogate spikes. The coefficients of variation for the analyses of all surrogate spikes were below the 25% project specification.

Accuracy. The accuracy of the analyses was estimated using surrogate spike recoveries. All spike recoveries were within the 70-130% project objectives.

Blank Effects. Low concentrations of two to four compounds in the method, trip, and field blanks associated with these samples were observed. All concentrations reported for the blank results were near the analytical detection limits. Sample concentrations, which were also near the detection limits of many compounds, may be biased slightly high for these compounds.

Material Balances

Table 4-5 shows the results of the material balance around the entire plant. 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 error propagation analysis, which is discussed in detail in Appendix E.

The coal was considered the only inlet stream for the material balance around the entire plant. Three outlet streams were included: bottom ash, ESP ash, and stack gas. The economizer ash was not considered in the material balance because its flow rate could

Table 4-5
Material Balance Results for Site 22

Substance	Closure (%)	95% CI
Target Species		
Arsenic	161	28
Barium	87	21
Beryllium	95	13
Cadmium	NC	--
Chloride	NC	--
Chromium	107	21
Cobalt	117	57
Copper	92	11
Fluoride	67	29
Lead	71	8
Manganese	95	41
Mercury (DGA/CVAAS)	38	4
Mercury (CVAFS)	110	29
Molybdenum	<46	--
Nickel	104	38
Phosphorus	76	5
Selenium	61	45
Vanadium	118	13
Other Species		
Ash	100[a]	--
Aluminum	112	15
Antimony	NC	--
Calcium	151	16
Iron	112	17
Magnesium	329	86
Potassium	15	7
Sodium	114	22
Sulfur	135	14
Titanium	111	16

[a]The ash balance closure was set at 100% assuming an 80:20 split of the coal ash to fly ash and bottom ash.

NC = Not calculated.

not be accurately measured, but it is assumed to be insignificant in relation to the ESP ash flow rate.

Of the 17 target species shown in Table 4-5, ten have closures around the entire plant that meet the project goal. The others, whose closures do not meet the goals are arsenic, fluoride, mercury, molybdenum, and selenium. Because cadmium and chlorine concentrations were below the detection limit in the coal, material balance calculations could not be performed. Material balances for major species (aluminum, iron, sodium, and titanium) met the project goal, which validates the stream flow rates used in the material balance and the assumption of an 80/20 split of coal ash between the bottom ash and entrained fly ash in the flue gas.

The QA/QC results for arsenic indicate a slightly high bias in the ash sample results (135% recovery), which may account for the somewhat high closure for this element. Likewise, the recovery of selenium (120%) from standard coal samples was slightly high, resulting in a closure slightly below the target range. Spike recovery data for fluoride for one of the two spiked ash samples were slightly low (74%) which may account for the 67% closure for fluoride, since the majority of the fluoride is present in the collected fly ash. The QA/QC data for mercury (CVAAS) and molybdenum were within the data quality objectives and do not provide any additional insight into these low material balance closures.

5

ADDITIONAL TEST RESULTS

This section presents miscellaneous data from Site 22. These data are presented in a separate section 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 and the results of the particle size distribution tests.

Mercury Speciation Tests

Mercury speciation tests were conducted at the stack during the same sampling periods used for the Method 29 multi-metals train. The following is a discussion of the sampling and analytical methods, a comparison of speciation train results with the Method 29 results, and the material balance results.

Sampling and Analytical Method

The solid sorbent method developed by Frontier Geosciences [1] 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). At Site 22, approximately 100 L of flue gas was collected at a rate of 0.5 L/minute from a single point in the stack (the sampling procedure is nonisokinetic and does not specify traversing the stack). The sorbent traps were then removed by Radian personnel and packaged and shipped to Frontier Geosciences for analysis.

Oxidized mercury (Hg^{2+}) and methyl-mercury (species such as CH_3HgCl) concentrations 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. Subsequent investigation by Frontier Geosciences has revealed that the method produces invalid results for methyl mercury; therefore, methyl mercury results are not presented in this report. Frontier Geosciences discovered that there is a reaction among acetate, sulfite, and oxidized mercury that occurs during dissolution of the soda-lime traps. This reaction has been shown to produce methyl mercury.

[1] Nicolas S. Bloom. "Mercury Speciation in Flue Gases: Overcoming the Analytical Difficulties." Proceedings of the Conference on Managing Air Toxics: State of the Art. November 4-6, 1991. Washington, D.C.

The elemental mercury (Hg^0) concentration 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 the solution to 100 ml with 0.05 N BrCl. The mercury in the resulting digestate was reduced using $SnCl_2$ and then trapped on a gold surface. The elemental mercury was 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).

Results of Speciation Measurements

Table 5-1 shows that the stack gas contained about 8.0 $\mu g/Nm^3$ of elemental mercury and 0.02 $\mu g/Nm^3$ of oxidized mercury. Table 5-1 also compares total mercury concentrations measured by the multi-metals method (EPA Method 29) with the results from the Frontier Geosciences method. For all three runs, the concentrations measured by Method 29 were 40% to 50% lower than those determined by the Frontier Geosciences method. The average value obtained by Method 29 was 4.5 $\pm$ 0.6 $\mu g/Nm^3$, and the average obtained using the Frontier Geosciences method was 8.0 $\pm$ 2.6 $\mu g/Nm^3$.

The method comparison results are surprising because data from previous FCEM sites have typically shown good agreement between the two methods for total mercury. The low field blank values show that the solid sorbent traps were not contaminated. Flue gas temperature at the sample point was approximately 300° F, and the gas velocity through the stack was about 100 feet per second.

Material Balance Results

Table 5-2 summarizes the results of the mercury material balances. The material balance closure for mercury is approximately 110 $\pm$ 29% when the total stack gas mercury data from the sorbent trap method are used along with the CVAFS coal data. This value is higher than the closure obtained using the Method 29 stack gas mercury data and the DGA/CVAAS coal data (38 $\pm$ 4 percent). QA/QC data for the coal samples analyzed by DGA/CVAAS and the Method 29 stack gas samples analyzed by CVAAS all met the project data quality objectives. The reason for the difference between the two data sets is not apparent.

Particle Size Distribution Tests

Table 5-3 presents the results of the particle size distribution tests for the stack location. Approximately 50% of the material collected in the PSD sample had a diameter less than 2.2 μm; 70% was less than 6.9 μm. This result is not surprising given the high overall removal efficiency of the ESP (99.97%).

The particulate loading measured by the PSD test (0.98 mg/Nm^3) is somewhat lower than the mean loading calculated from the Method 29 multi-metals train data

Table 5-1
Stack Gas Mercury Speciation Results

Stream/Species	Run 1	Run 2	Run 3	Mean	95% CI
Coal (mg/kg, dry)					
Hg by CVAFS (Frontier Geosciences)	0.069	0.086	0.078	0.079	0.020
Hg by CVAAS	0.14	0.14	0.14	0.14	0.0008
Sorbent Trap Speciation ($\mu g/Nm^3$)					
Ionic Mercury	ND(0.009)	0.04	ND(0.009)	0.02	0.05
Elemental Mercury	7.3	9.2	7.5	8.0	2.6
Sum of Species	7.3	9.2	7.5	8.0	2.6
Multi-Metals Trains ($\mu g/Nm^3$)					
Nitric Acid Impingers	1.5	1.2	1.6	1.4	0.5
Permanganate Impingers	3.1	3.0	3.0	3.1	0.1
Total Vapor-Phase Mercury	4.6	4.2	4.6	4.5	0.6

Table 5-2
Site 22 Mercury Material Balances

Stream	Mean Flow Rates	Method 29, CVAAS Measurements		Sorbent Trap, CVAFS Measurements	
		Mean Concentration	Mass (g/hr)	Mean Concentration	Mass (g/hr)
Coal (dry)	251,000 kg/hr	0.14 mg/kg	35.1	0.079 mg/kg	19.8
Bottom Ash (dry)	3,410 kg/hr	0.12 mg/kg	0.4	0.12 mg/kg*	0.4
Collected Ash (dry)	13,600 kg/hr	0.094 mg/kg	1.3	0.094 mg/kg*	1.3
Stack Gas	2,530,000 Nm^3/hr	4.5 $\mu g/Nm^3$	11.4	8.0 $\mu g/Nm^3$	20.2
Overall Mass Balance Closure (%)			38 ± 4		110 ± 29

*Samples were not analyzed by Frontier Geosciences. Data shown are CVAAS results.

Table 5-3
Particle Size Distribution Data for Site 22

Stack Location				
Gas Sampled:	902.3 dscf			
Percent Isokinetic:	94			
Stage Number	Cut Size (μm)	Collected Mass (g)	Grain Loading (mg/Nm^3)	Percent of Total Mass
1	>6.9	0.0060	0.25	26
2	2.2	0.0046	0.19	20
3	0.5	0.0104	0.44	44
Filter	<0.5	0.0023	0.097	10
Total:			0.98	100

(1.8 mg/Nm^3). The PSD sample was collected at a single point in the stack, whereas the Method 29 sample was collected by traversing the diameter of the stack, which may account for this difference. In addition, it is typically very difficult to recover 100% of the material from the surfaces within the PSD sampling apparatus, resulting in a low bias in the particulate loading measurement. However, data obtained about the distribution of particulate matter among the size fractions is considered valid. The particulate matter from each size fraction will be archived for possible elemental analysis in the future, although the mass available (2 to 10 mg) precludes conventional analytical techniques.

6

EXAMPLE CALCULATIONS

This section describes the methodology and sample calculations used to develop the results discussed in Section 3. Specifically, the calculation of stream flow rates, emission factors, mean values, and confidence intervals are presented.

Stream Flow Rates

Appendix D contains information about the stream flow rates measured at Site 22 during the sampling period. Coal feed rates were determined from hourly reading taken from each of the seven gravimetric coal feeders. The flow rates in the stack measured directly during sampling. Bottom ash and ESP ash rates were determined assuming a 20:80 split of the coal ash, respectively.

Means and Confidence Intervals for Stream Concentrations

The mean concentration and 95% confidence interval (CI) about the mean were calculated for each target substance in the coal, bottom ash, ESP ash and stack gas. The means were calculated according to the conventions listed in Section 3. Equations used to calculate 95% confidence intervals are presented in Appendix E. Example calculations are presented here for arsenic in the stack gas; these results were shown in Table 3-4.

The concentration data (in $\mu g/Nm^3$) given for arsenic, as shown in Table 3-4, are:

	Run 1	Run 2	Run 3
Solid Phase	0.107	0.109	0.0932
Vapor Phase	ND(0.11)	ND(0.11)	ND(0.11)
Total	0.107	0.109	0.0932

The mean is calculated from the individual run totals:

$$\text{Mean} = (0.107+0.109+0.0932)/3$$

$$= 0.103$$

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

$$S_p = \sqrt{[(0.107-0.103)^2 + (0.109-0.103)^2 + (0.0932-0.103)^2]/2}$$

$$= \quad 0.00860$$

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

$$S_{\bar{p}} = \frac{0.00860}{\sqrt{3}}$$

$$= \quad 0.00497$$

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 detection 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.

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

$$= \quad 0$$

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

$$U_r = \sqrt{\beta^2 + (t \times S_{\bar{p}})^2}$$

$$= \sqrt{0^2 + (4.3 \times 0.00497)^2}$$

$$= 0.02$$

Thus, the result is reported as 0.11 ± 0.02 $\mu g/Nm^3$.

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 of a substance in the ESP outlet 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.

As an example, the calculation of the emission factor for arsenic is presented. The mean coal flow rate is 552,940 lb/hr on a dry basis. The mean HHV of the coal is 11,981 Btu/lb on a dry basis. Multiplying the coal flow rate by the HHV gives a mean heat input of 6.62 billion Btu/hr. The mean arsenic mass flow through the stack (the product of the mean concentration, 0.103 $\mu g/Nm^3$, and the mean gas flow rate, 2,534,100 Nm^3/hr) is 0.261 g/hr or 0.000575 lb/hr. When the mean mass flow rate is divided by the mean heat input, an emission factor of 0.087 lb/trillion Btu is obtained, as shown in Table 3-7.

The 95% confidence intervals for emission factors were calculated according to the equations presented in Appendix E. For each parameter (stack 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

Btu	British Thermal Unit
CAAA	Clean Air Act Amendments
CEM	Continuous Emissions Monitoring
CVAAS	Cold Vapor Atomic Absorption Spectrophotometry
CVAFS	Cold Vapor Atomic Fluorescence Spectroscopy
DGA	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-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy
ID	Induced Draft
INAA	Instrumental Neutron Activation Analysis
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MW	Megawatt
NC	Not Calculated
NIST	National Institute of Standards and Technology
Nm^3	Dry Normal Cubic Meter (0° C, 1 atm)
ND	Not Detected (below detection limit)
PAH	Polycyclic Aromatic Hydrocarbons
PCDD	Polychlorinated Dibenzo-p-dioxin
PCDF	Polychlorinated Dibenzofuran

POM	Polycyclic Organic Matter
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SARM	South African Reference Material
SIE	Selective Ion Electrode

Appendix A: Detailed Sample Collection/ Preparation/Analysis Information

This appendix presents the methods used to collect and analyze each type of sample. Summary tables showing collection times and important observations for each of the samples are also included.

Multi-Metals Sampling Train

A modified version of the sampling methodology specified in Section 3.1 of 40 CFR, Part 266, Appendix IX (proposed EPA Method 29) was used to determine the particulate mass loading and to collect solid and vapor phase samples of the stack gas for trace metals analysis. This method provides for the collection of a flue gas sample at isokinetic conditions while traversing the duct according to EPA Method 1. Particulate matter is collected in the glass nozzle, probe liner, and on a three-inch high purity quartz filter during sample collection. Use of the three-inch filter versus the normal four-inch filter helped reduce the background concentration of certain trace elements of interest. The vapor phase species are absorbed in an impinger train consisting of:

- Two impingers containing 5% HNO_3/10% H_2O_2, which are analyzed for all metals of interests; and
- Two impingers containing 4% $KMnO_4$/10% H_2SO_4, which are analyzed for mercury only.

The multi-metals samples were collected by traversing all four quadrants of the stack. A total of approximately 250 scf of flue gas was collected over a period of six to eight hours.

Upon completion of sampling, the glass nozzle and probe liner were first rinsed with acetone to recover any solids present for determining total particulate mass loading and for trace metals analysis. The nozzle and probe were then rinsed with a nitric acid solution to recover any trace metals that may not have been recovered during the acetone rinse. The acetone rinse was evaporated to determine the mass of solids present in the sample. The residual mass was combined with the filter solids to determine the particulate mass loading. The nitric acid rinse sample was combined with the acetone probe and nozzle rinse residue and filter sample for trace metals analysis.

The multi-metals method specifies that HNO_3/H_2O_2 impinger solutions be evaporated to near dryness prior to analysis. However, due to concern over the possible loss of volatile metals, this procedure was not followed. Instead, the impinger solutions were analyzed as recovered to avoid any loss of volatile metals. The combined filter/probe and nozzle rinse samples were digested using a microwave technique.

Anions Sampling Train

Anions (HCL, HF, and SO_2) samples and particulate mass loading samples were collected simultaneously using an adaptation of the procedures specified in EPA Method 5. Particulate matter was captured in the glass nozzle, probe liner, and on the 3-inch quartz filter. The vapor phase anions were absorbed in two impingers containing 2.4 mmole Na_2CO_3, 3 mmole $NaHCO_3$, and 6% H_2O_2.

The anions samples were collected by traversing all four quadrants of the stack. A total of approximately 70 dscf were collected over a period of two to three hours.

Upon completion of sampling, the glass nozzle and probe liner were first rinsed with acetone to remove any solids present. The nozzle and liner were then rinsed with a carbonate/bicarbonate buffered solution to remove any anions that were not recovered during the acetone rinse. The acetone rinse was evaporated to determine the mass of solids present in the sample. The acetone probe and nozzle rinse mass was combined with the mass of solids on the filter to determine the particulate mass loading. The acetone probe and nozzle rinse sample and the filter were leached using the carbonate/ bicarbonate rinse sample to recovery any anions present. The resulting solution was then analyzed to determine the particulate phase concentration of anions. Vapor phase concentrations were determined from the analysis of the impinger solutions.

Mercury Speciation Sampling Train

Mercury speciation samples were collected using a solid sorbent procedure developed by Frontier Geosciences. A pair of KCl-saturated soda-lime traps were used to sorb oxidized forms of mercury while a pair of iodated carbon traps were used to sorb elemental mercury from the flue gas stream. The samples were collected at a single point approximately two feet from the stack wall. A sampling rate of 0.5 liters per minute was used to collect the 100 liter samples over a period of about three to four hours. After sampling, the sample traps were sealed and then shipped back to Frontier Geosciences for analysis by cold vapor fluorescence detection. Additional details for this method are provided in Appendix H.

Semivolatiles - Polycyclic Aromatic Hydrocarbons (PAHs) Sampling Train

PAHs samples were collected using the sampling methodology specified in Method 0010 of SW-846. During sampling, particulate phase PAHs were captured in the glass nozzle, probe liner, and three-inch high purity quartz filter. The vapor phase PAHs were absorbed in a chilled XAD resin cartridge.

The semivolatiles samples were collected by traversing all four quadrants of the stack. A total of approximately 105 dscf were collected over a period of three to four hours.

Upon completion of sampling, the glass nozzle and probe liner were rinsed with acetone and methylene chloride to remove any solids and PAHs present. The filter and XAD resin cartridge were recovered and sent along with the probe and nozzle rinse sample to Twin Cities Testing for analysis.

PCDD/PCDF Sampling Train

PCDD/PCDF samples were collected using a modified version of the sampling methodology specified in EPA Method 23. During sampling, particulate phase PCDD/PCDFs were captured in the glass nozzle, probe liner, and three-inch high purity quartz filter. The vapor phase PCDD/PCDFs were absorbed in a chilled XAD resin cartridge.

The PCDD/PCDF samples were collected by traversing all four quadrants of the stack. A total of approximately 106 dscf were collected over a period of three to four hours.

Upon completion of sampling, the glass nozzle and probe liner were rinsed with acetone, methylene chloride, and toluene to remove any solids and PCDD/PCDFs present. The filter and XAD resin cartridge were recovered and sent along with the probe and nozzle rinse sample to Twin Cities Testing for analysis.

Particle Size Distribution Sampling Train

A Cyclade Model 283-2 cascade cyclone sampler was used to collect a size fractionated sample of the stack flue gas particulate solids. The Cyclade consisted of three cyclones and a final filter. Sampling was conducted at a single point in the stack. A single composite sample of approximately 902 dscf was collected over a three day period. The cut-points of the three cyclones were 6.9, 2.2, and 0.5 microns at the test conditions. Upon completion of testing, the solids present in each of the three cyclones and the final

filter were recovered into individual sample storage containers. The samples were archived for possible future analysis.

Flue Gas Flow Rate

The flow rate of flue gas exiting the stack was measured using the procedures specified in EPA Methods 1, 2, 3, and 4. EPA Method 1 was used to determine the number and location of the sampling points used to collect a representative flue gas sample. Twelve sampling points (4 x 3) were used to collect the stack samples.

EPA Method 2 was used to determine the velocity and flow rate of the flue gas. A K-type thermocouple was used to measure the flue gas temperature and a S-type pitot tube was used to measure the velocity head at each sampling point during the collection of PAHs, PCDD/PCDFs, multi-metals, and anions samples.

EPA Method 3 was used to determine the molecular weight of the flue gas. An integrated bag sample was collected during the collection of each multi-metals sample. The integrated sample was analyzed for O_2 and CO_2 using a Fyrite gas analyzer.

EPA Method 4 was used to determine the moisture content of the flue gas. The moisture content of the stack gas was determined during the collection of anions, multi-metals, PAHs, and PCDD/PCDF samples. The impingers used during the collection of these samples were weighed before and after sampling to determine the mass of water condensed during sample collection. The mass of condensed water was related to the volume of flue gas sampled to determine the fraction of water vapor present in the flue gas.

Process Samples

The collection of coal, bottom ash, and ESP fly ash at Site 22 is discussed in the following paragraphs. The sampling procedures outlined in the Site 22 test plan were used to collect all the solid samples, except for bottom ash.

Coal. No problems were encountered during the collection of coal samples during Runs 1 and 2. The plant's dedicated autosampler was set to begin collecting coal at midnight before the next day's test and was allowed to collect samples until approximately 2:00 p.m. (e.g., Run 1 collection occurred from midnight on 7/12/93 to 2:00 p.m. on 7/13/93).

Plant personnel were forced to shut down the coal autosampler midway through the day during Run 3 (7/15/93) because excessive moisture in the coal caused plugging problems in the autosampler. Therefore, the sample obtained for Run 3 may not be representative of the coal burned during the entire gas sampling period.

Bottom Ash. Severe flooding in the bottom ash pond prevented collecting the bottom ash samples as specified in the Site 22 test plan. The procedure outlined in the test plan specified the collection of bottom ash from the end of the discharge line using PVC U-tube to divert a portion of the stream to a sample container. Subsamples were to be collected periodically during the ash sluicing period to obtain a representative composite.

On 7/13/93 (Run 1) plant personnel unflanged the ash line upstream of the ash pond to allow access to the bottom ash and prevent the ash line from plugging. The new sampling point was still half under water, and access to the pipe was somewhat restricted. On 7/14/93 and 7/15/93, plant personnel provided access to the discharge point by boat. For safety reasons, samples had to be collected from the material that was accumulating at the end of the pipe rather than directly from the discharge stream. A PVC scoop was used to collect ash samples over a period of approximately 10 minutes at the beginning of each three-hour sluicing period. The short sampling period could

potentially affect the representativeness of the bottom ash samples. In addition, floodwaters mixing with the ash could potentially contaminate the bottom ash samples.

ESP Fly Ash. No problems were encountered during the collection of fly ash samples. Samples were collected using the plant's dedicated autosampler, which collects ash from both the A- and B-side ESP discharge lines upstream of the ash silo. The sampling system was emptied at 7:00 a.m. each test day and the system was allowed to collect ash until approximately 5:00 p.m. The A- and B-side samples were combined at the end of each day to obtain a representative composite.

Table A-1
Sample Collection, Preservation, and Handling Techniques for Site 22

Stream	Collection Method	Fraction Description	Sample Handling & Preservation	Comments
Flue Gas Samples				
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.	Separate filters used for A/B and C/D ESP outlet ducts; combined prior to analysis.
		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.
Solid Samples				
Coal	Grab/Composite	Ultimate, Proximate, 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.
		Mercury	Kept in sealed container; submitted "as collected".	
		Metals	Placed in sealed container in the field; air dried, ground prior to analysis.	
Fly Ash, Bottom Ash	Grab	Metals, Cl, SO_4, F	Kept in sealed container in the field; air dried, ground prior to analysis.	

[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-2
Preparation Procedures and Chemical Analysis Methods Applied to Coal at Site 22

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
Proximate Analysis of Coal	
Moisture	ASTM D 3173
Ash	ASTM D 3174
Volatiles	ASTM D 3175
Fixed Carbon	Calculated
Target Elements by INAA	
Preparation	
Analysis by INAA	
Arsenic	Karr, Chapters 12 and 46
Cadmium	Karr, Chapters 12 and 46
Chromium	Karr, Chapters 12 and 46
Chlorine	Karr, Chapters 12 and 46
Copper	Karr, Chapters 12 and 46
Manganese	Karr, Chapters 12 and 46
Mercury	Karr, Chapters 12 and 46
Nickel	Karr, Chapters 12 and 46
Selenium	Karr, Chapters 12 and 46
Chlorine and Fluorine Analysis in Coal	
Preparation	
Oxygen Bomb Digestion	ASTM D 2361/D 3761
Analysis by Potentiometric Titration	
Chloride	SM 407C
Analysis by BIF Method	
Chloride	56 CFR 136, July 17, 1991
Analysis by Ion Selective Electrode	
Fluoride	ASTM D 3761

Table A-2 (Continued)

Component	**Method Reference**
Be, Cr, Cu, Mn, P, Ti, and Ni in Coal	
Preparation	
Ashing at 500° C/Acid Digestion	EPA 340.2, ASTM D 3683
Analysis by ICP-AES	
Beryllium	SW 6010
Chromium	SW 6010
Copper	SW 6010
Manganese	SW 6010
Nickel	SW 6010
Phosphorus	SW 6010
Titanium	SW 6010
Arsenic, Cadmium, Lead, and Selenium in Coal	
Preparation	
Oxygen Bomb Combustion/Acid Digestion	ASTM D 3684
Analysis by GFAAS	
Arsenic	SW 7060
Cadmium	SW 7131
Lead	SW 7421
Selenium	SW 7740
Mercury Analysis in Coal	
Preparation	
Double Gold Amalgamation	Karr, Chapter 14
Analysis by CVAAS	
Mercury	Karr, Chapter 14, SW 7471
Additional Inorganic Analytes in Coal	
Preparation	
Analysis by INAA	
Aluminum	Karr, Chapters 12 and 46
Antimony	Karr, Chapters 12 and 46
Arsenic	Karr, Chapters 12 and 46
Barium	Karr, Chapters 12 and 46
Cadmium	Karr, Chapters 12 and 46
Calcium	Karr, Chapters 12 and 46

Table A-2 (Continued)

Component	Method Reference
Chromium	Karr, Chapters 12 and 46
Cobalt	Karr, Chapters 12 and 46
Copper	Karr, Chapters 12 and 46
Iron	Karr, Chapters 12 and 46
Magnesium	Karr, Chapters 12 and 46
Manganese	Karr, Chapters 12 and 46
Mercury	Karr, Chapters 12 and 46
Molybdenum	Karr, Chapters 12 and 46
Nickel	Karr, Chapters 12 and 46
Potassium	Karr, Chapters 12 and 46
Selenium	Karr, Chapters 12 and 46
Sodium	Karr, Chapters 12 and 46
Titanium	Karr, Chapters 12 and 46
Zinc	Karr, Chapters 12 and 46

C. Karr 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 Edition.

ASTM is the American Society for Testing and Materials.

Table A-3
Preparation Procedures and Chemical Analysis Methods for Inorganic and Organic Chemical Components in Flue Gas at Site 22

Component	Method Reference	Non-coal Solids	Stack Solids	Metals Impingers 1 & 2	Metals Impingers 3 & 4	Anions Impingers	XAD Resin
Target Elements 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			
Antimony	SW 6010	X	X	X			
Barium	SW 6010	X	X	X			
Beryllium	SW 6010	X	X	X			
Calcium	SW 6010	X	X	X			
Cobalt	SW 6010	X	X	X			
Copper	SW 6010	X	X	X			
Chromium	SW 6010	X	X	X			
Iron	SW 6010	X	X	X			
Lead	SW 6010	X	X	X			
Magnesium	SW 6010	X	X	X			
Manganese	SW 6010	X	X	X			
Molybdenum	SW 6010	X	X	X			
Nickel	SW 6010	X	X	X			
Phosphorus	SW 6010	X	X	X			
Potassium	SW 6010	X	X	X			
Sodium	SW 6010	X	X	X			
Titanium	SW 6010	X	X	X			
Vanadium	SW 6010	X	X	X			
Target Elements by GFAAS							
Preparation							
MW Digestion for Filters	CEM-F		X				
MW Digestion for Solids	CEM-FA	X					

Table A-3 (Continued)

Component	Method Reference	Non-coal Solids	Stack Solids	Metals Impingers 1 & 2	Metals Impingers 3 & 4	Anions Impingers	XAD Resin
Analysis by GFAAS							
Arsenic	SW 7060	X	X	X			
Cadmium	SW 7131	X	X	X			
Lead	SW 7421	X	X	X			
Nickel	EPA 249.2	X	X	X			
Selenium	SW 7740	X	X	X			
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		
Acid-Forming Anions							
Aqueous Extraction of Solids	Radian	X	X				
Chloride and Sulfate by IC	EPA 300.0	X	X			X	
Fluoride by ISE	EPA 340.2	X	X			X	
Fluoride by ISE	McQuaker and Gurney	X					
Semivolatiles by HRGCMS	SW 846 Method 0010		X				X
PCDD/PCDF by HRGCMS	Mod. EPA Method 23		X				X

ASTM is American Society for Testing and Materials.
EPA is *EPA Methods for Chemical Analysis of Water and Wastes*, 1983.
SW EPA is SW-846, *Test Methods for Evaluating Solid Waste*, 3rd ed.
CEM-F CEM Corporation, Matthews, NC, "Procedure for Microwave Digestion of Glass or Quartz Filters."
CEM-FA CEM Corporation, Matthews, NC, "Procedure for Microwave Digestion of Coal Fly Ash."
N.R. McQuaker 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.

Table A-4
FCEM Site 22 - Flue Gas Samples Collection Time Periods

	Parameter	Stack Start-Stop	Comments
RUN 1 7/13/93	PCDD/PCDF	1252-1812	1) The start-stop times include the time required to change sampling ports.
	Semivolatile Compounds/PAHs	1137-1817	2) Stack velocity data were measured during the collection of PCDD/ PCDF, semivolatile compounds, multi-metals and anions samples on each test day.
	Multi-Metals	0854-1725	3) The stack anions train was voided because isokenetics were 113% which is above the QC limit of 110%
	Anions	0916-1215	
	Hg Speciation	1008-1418	
	PSD	1026-1930	
RUN 2 7/14/93	PCDD/PCDF	0925-1356	1) No problems were encountered.
	Semivolatile Compounds/PAHs	1107-1515	2) Two sets of Anions samples were collected on the second day of testing.
	Multi-Metals	0826-1547	
	Anions	0832-1122	
	Anions (Run 1-2)	1339-1606	
	Hg Speciation	1225-1626	
	PSD	0935-1640	
RUN 3 7/15/93	PCDD/PCDF	0911-1350	1) No problems were encountered.
	Semivolatile Compounds/PAHs	0850-1406	
	Multi-Metals	0824-1444	
	Anions	0904-1129	
	Hg Speciation	0942-1312	
	PSD	0836-1700	

Table A-5
FCEM Site 22 - Process Stream Sample Collection Periods

	Stream	Start-Stop	Comments
Run 1 7/13/93	Coal	2400 (7/12)-1400	1) Bottom ash collected from material accumulated at end of pipe using sample scoop
	Bottom Ash	1420-1440	
	Fly Ash	0700-1900	
Run 2 7/14/93	Coal	2400 (7/13)-1320	1) See comment 1 for Run 1 above
	Bottom Ash	1410-1420	
	Fly Ash	0700-1715	
Run 3 7/15/93	Coal	2400 (7/14)-0800	1) Coal autosampler shut down because of plugging problems caused by excessive coal moisture.
	Bottom Ash	1420-1430	
	Fly Ash	0700-1600	2) See comment 1 for Run 1 above

Appendix B:
Data Used In Calculations

Site 22: Data Used in Calculations

Bottom Ash

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		ICAP	mg/kg	81400 B	76600 B	74500 B
Antimony		ICAP	mg/kg	ND (74)	ND (73)	ND (73)
Arsenic		GFAA	mg/kg	8.41 B	9.63 B	13.7 B
Beryllium		ICAP	mg/kg	0.988 B	1.11 B	1.17 B
Cadmium		GFAA	mg/kg	ND (0.41) BJ	ND (0.41) BJ	0.491 B
Calcium		ICAP	mg/kg	141000 B	135000 B	123000 B
Chloride		SIE	mg/kg	ND (100)	ND (100)	ND (100)
Chromium		ICAP	mg/kg	56.9 B	54.4 B	55.0 B
Cobalt		ICAP	mg/kg	22.2 B	20.1 B	22.1 B
Copper		ICAP	mg/kg	109 B	99.2 B	99.5 B
Fluoride	NaOH digestion	SIE	mg/kg	20.3	24.9	27.3
Iron		ICAP	mg/kg	40900 B	38000 B	50600 B
Lead		GFAA	mg/kg	5.67	6.35	7.99
Magnesium		ICAP	mg/kg	23200	22300	20600
Manganese		ICAP	mg/kg	109 B	104 B	104 B
Mercury		CVAA	mg/kg	ND (0.012)	ND (0.012)	0.353
Mercury	redigestion	CVAA	mg/kg			0.452
Mercury	reanalysis	CVAA	mg/kg			0.380 B
Mercury	second split	CVAA	mg/kg			0.435 B
Molybdenum		ICAP	mg/kg	ND (14)	ND (14)	ND (14)
Nickel		GFAA	mg/kg	54.1	53.4	78.5
Percent moisture		oven dry	%	0.598	0.199	0.400
Potassium		ICAP	mg/kg	2570	2560	2490
Selenium		GFAA	mg/kg	ND (1.1)	ND (1.1)	ND (1.1) J
Sodium		ICAP	mg/kg	8920 B	6810 B	6170 B
Sulfur		tot S	% Sulf	ND (0.0050)	ND (0.0050)	0.323
Titanium		ICAP	mg/kg	7410	6920	6530
Vanadium		ICAP	mg/kg	160 B	154 B	144 B

Site 22: Data Used in Calculations

Coal

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		NAA	mg/kg	5070	5400	5730
Antimony		NAA	mg/kg	0.107	0.142	0.151
Arsenic		NAA	mg/kg	0.768	0.859	0.908
Ash		Proximate	wt. %	6.46	6.66	7.27
Barium		NAA	mg/kg	347	365	429
Beryllium		D3683/ICAP	mg/kg	0.200	0.200	0.200
Cadmium		Parr/GFAA	mg/kg	ND (0.10)	ND (0.10)	ND (0.10)
Calcium		NAA	mg/kg	7610	7680	7720
Carbon		Ultimate	wt. %	69.9	69.5	69.2
Chloride	reanalysis	D4208	mg/kg	ND (100)	ND (100)	ND (100)
Chlorine		IC	mg/kg	ND (100)	ND (100)	ND (100)
Chromium		D3683/ICAP	mg/kg	4.00	4.00	4.00
Cobalt		NAA	mg/kg	1.25	1.47	1.46
Copper		D3683/ICAP	mg/kg	11.0	11.0	12.0
Fixed carbon		Proximate	wt. %	47.1	46.7	46.9
Fluoride	reanalysis	D3761	mg/kg	50.0	50.0	60.0
Fluoride		IC	mg/kg	50.0	52.0	60.5
HHV		Proximate	Btu/lb	12100	12000	11900
Hydrogen		Ultimate	wt. %	4.87	4.91	4.90
Iron		NAA	mg/kg	2200	2310	2550
Lead		Parr/GFAA	mg/kg	2.00	2.00	2.00
Magnesium		NAA	mg/kg	478	617	610
Manganese		D3683/ICAP	mg/kg	7.00	8.00	10.0
Mercury		CVAFS	ng/g	69.0	85.6	77.7
Mercury	reanalysis	DGA/CVAA	mg/kg	0.220	0.130	0.120
Mercury		DGA/CVAA	ug/g	0.138	0.138	0.138
Moisture		Proximate	wt. %	27.5	29.3	31.7
Molybdenum		NAA	mg/kg	1.59	2.33	2.51
Nickel		NAA	mg/kg	3.06	3.80	4.71
Nitrogen		Ultimate	wt. %	1.01	1.04	1.02

Site 22: Data Used in Calculations

Coal

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Oxygen		Ultimate	wt. %	17.4	17.5	17.2
Phosphorus		D3683/ICAP	mg/kg	300	310	320
Potassium		NAA	mg/kg	1190	1020	1620
Selenium		NAA	mg/kg	0.921	0.697	0.728
Sodium		NAA	mg/kg	644	540	549
Sulfur		Ultimate	wt. %	0.330	0.420	0.410
Titanium		NAA	mg/kg	535	489	471
Vanadium		NAA	mg/kg	12.3	12.0	13.2
Volatile		Proximate	wt. %	46.4	46.6	45.8

Collected Flyash

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		ICAP	mg/kg	91200 B	86800 B	86100 B
Antimony		ICAP	mg/kg	ND (72)	ND (75)	ND (73)
Arsenic		GFAA	mg/kg	21.6 B	23.6 B	21.9 B
Barium		ICAP	mg/kg	5500	5290	5020
Beryllium		ICAP	mg/kg	3.30 B	3.37 B	3.00 B
Cadmium		GFAA	mg/kg	0.759 B	1.01 B	0.810 B
Calcium		ICAP	mg/kg	178000 B	172000 B	167000 B
Chloride		IC	mg/kg			13.1
Chloride		SIE	mg/kg	ND (100)	ND (100)	835
Chromium		ICAP	mg/kg	64.4 B	63.4 B	67.2 B
Cobalt		ICAP	mg/kg	25.7 B	27.8 B	24.4 B
Copper		ICAP	mg/kg	171 B	168 B	161 B
Fluoride	NaOH digestion	SIE	mg/kg	462	348	623
Iron		ICAP	mg/kg	36900 B	34000 B	34500 B
Lead		GFAA	mg/kg	25.5	24.1	23.4
Magnesium		ICAP	mg/kg	29800	28900	28000
Manganese		ICAP	mg/kg	120 B	119 B	116 B

Site 22: Data Used in Calculations

Collected Flyash

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Mercury		CVAA	mg/kg	0.0852	0.110	0.0877
Molybdenum		ICAP	mg/kg	ND (14)	ND (14) J	ND (14)
Nickel		GFAA	mg/kg	60.9	58.4	58.0
Percent moisture		oven dry	%	0.197	0.199	0.197
Potassium		ICAP	mg/kg	2940	2730	3270
Selenium		GFAA	mg/kg	8.36	5.64	12.1
Sodium		ICAP	mg/kg	10300 B	10700 B	9350 B
Strontium		ICAP	mg/kg	2870	2710	2590
Sulfur		tot S	% Sulf	ND (0.0050)	ND (0.0050)	ND (0.0050)
Titanium		ICAP	mg/kg	8510	7980	7870
Vanadium		ICAP	mg/kg	215 B	209 B	206 B

Stack Gas Dioxins and Furans

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
1234678-HpCDD		Method 2	ug/Nm3	0.00000445	0.00000925	ND (0.0000091)
1234678-HpCDF		Method 2	ug/Nm3	0.00000236	ND (0.0000050)	0.00000296
123478-HxCDD		Method 2	ug/Nm3	ND (0.0000034)	ND (0.0000057)	ND (0.0000031)
123478-HxCDF		Method 2	ug/Nm3	ND (0.0000034)	ND (0.0000078)	ND (0.0000042)
1234789-HpCDF		Method 2	ug/Nm3	ND (0.0000022)	ND (0.0000053)	ND (0.0000038)
123678-HxCDD		Method 2	ug/Nm3	ND (0.0000050)	ND (0.0000028)	ND (0.0000022)
123678-HxCDF		Method 2	ug/Nm3	ND (0.0000094)	ND (0.000014)	ND (0.000010)
12378-PeCDD		Method 2	ug/Nm3	ND (0.0000015)	ND (0.0000046)	ND (0.0000025)
12378-PeCDF		Method 2	ug/Nm3	ND (0.0000014)	ND (0.0000026)	0.00000181
123789-HxCDD		Method 2	ug/Nm3	ND (0.0000029)	ND (0.0000050)	ND (0.0000025)
123789-HxCDF		Method 2	ug/Nm3	ND (0.0000037)	ND (0.0000030)	ND (0.0000049)
234678-HxCDF		Method 2	ug/Nm3	ND (0.0000034)	ND (0.0000053)	ND (0.0000042)
23478-PeCDF		Method 2	ug/Nm3	ND (0.0000014)	ND (0.0000050)	ND (0.0000018)
2378-TCDD		Method 2	ug/Nm3	ND (0.0000029)	ND (0.0000039)	ND (0.0000021)
2378-TCDF		Method 2	ug/Nm3	ND (0.0000015)	ND (0.0000043)	ND (0.0000026)

Site 22: Data Used in Calculations

Stack Gas Dioxins and Furans

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
OCDD		Method 2	ug/Nm3	0.0000472	0.0000747	0.0000628
OCDF		Method 2	ug/Nm3	0.00000367	0.00000676	0.00000453
Total HpCDD		Method 2	ug/Nm3	0.00000970	0.0000181	0.00000698
Total HpCDF		Method 2	ug/Nm3	0.00000236	ND	0.00000296
Total HxCDD		Method 2	ug/Nm3	ND	ND	ND
Total HxCDF		Method 2	ug/Nm3	0.00000288	0.00000534	0.00000419
Total PeCDD		Method 2	ug/Nm3	ND	ND	ND
Total PeCDF		Method 2	ug/Nm3	0.00000996	0.0000114	0.00000453
Total TCDD		Method 2	ug/Nm3	ND	ND	0.00000558
Total TCDF		Method 2	ug/Nm3	0.00000207	ND	0.0000126

Stack Gas Polynuclear Aromatic Hydrocarbons

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
5-Methyl chrysene		HRGC/HRMS	ug/Nm3	ND (0.00022)	ND (0.00056)	ND (0.00018)
7h-Dibenzo[c,g]carbazole		HRGC/HRMS	ug/Nm3	0.000380	ND (0.00086)	ND (0.0016)
Acenaphthalene		HRGC/HRMS	ug/Nm3	0.00831	0.00198	0.00176
Acenaphthene		HRGC/HRMS	ug/Nm3	0.0101	0.00537	0.00606
Anthracene		HRGC/HRMS	ug/Nm3	0.00768	0.00517	0.00353
Benz[a]anthracene		HRGC/HRMS	ug/Nm3	0.00217	0.000922	0.000564
Benzo[a]pyrene		HRGC/HRMS	ug/Nm3	0.00242	0.000823	0.000564
Benzo[b,j&k]fluoranthenes		HRGC/HRMS	ug/Nm3	0.00551	0.00244	0.00180
Benzo[ghi]perylene		HRGC/HRMS	ug/Nm3	0.00334	0.00224	0.00226
Chrysene		HRGC/HRMS	ug/Nm3	0.00508	0.00273	0.00109
Dibenz[a,h]acridine		HRGC/HRMS	ug/Nm3	0.000407	ND (0.0012)	ND (0.00095)
Dibenz[a,h]anthracene		HRGC/HRMS	ug/Nm3	0.000407	0.000362	ND (0.00039)
Dibenz[a,i]acridine		HRGC/HRMS	ug/Nm3	0.000977	0.000955	0.000282
Dibenzo[a,e]pyrene		HRGC/HRMS	ug/Nm3	0.000353	0.000329	ND (0.00035)
Dibenzo[a,h]pyrene		HRGC/HRMS	ug/Nm3	ND (0.00052)	ND (0.00076)	ND (0.00063)
Dibenzo[a,i]pyrene		HRGC/HRMS	ug/Nm3	ND (0.00043)	ND (0.00020)	ND (0.00063)

Site 22: Data Used in Calculations

Stack Gas Polynuclear Aromatic Hydrocarbons

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Fluoranthene		HRGC/HRMS	ug/Nm3	0.0470	0.0281	0.0104
Fluorene		HRGC/HRMS	ug/Nm3	0.0254	0.0131	0.00472
Indeno[1,2,3-cd]pyrene		HRGC/HRMS	ug/Nm3	0.00166	0.000593	0.000811
Phenanthrene		HRGC/HRMS	ug/Nm3	0.140	0.0820	0.0226
Pyrene		HRGC/HRMS	ug/Nm3	0.0365	0.0165	0.00532

Stack Gas, Particulates Anions

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Chloride		IC	ug/g	49000*	35200	16300
Fluoride		SIE	ug/g	11600*	6400	6340

Stack Gas, Particulates Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		ICAP	ug/g	108000	79300	72100
Antimony		ICAP	ug/g	ND (2100)	ND (2200)	ND (1300)
Arsenic		GFAA	ug/g	79.5	52.1	45.0
Barium		ICAP	ug/g	11500	10100	9430
Beryllium		ICAP	ug/g	ND (17)	ND (17)	ND (11)
Cadmium		GFAA	ug/g	230	103	25.4
Calcium		ICAP	ug/g	247000	195000	170000
Chromium		ICAP	ug/g	414	ND (300)	ND (190)
Cobalt		ICAP	ug/g	ND (400)	ND (400)	ND (250)
Copper		ICAP	ug/g	613	333	299
Iron		ICAP	ug/g	47000	24500	25000
Lead		GFAA	ug/g	105	60.0	66.2
Magnesium		ICAP	ug/g	33400	27600	24500
Manganese		ICAP	ug/g	340	175	200

Site 22: Data Used in Calculations

Stack Gas, Particulates Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Mercury		CVAA	ug/g	6.14	4.29	2.71
Molybdenum		ICAP	ug/g	1590	1150	983
Nickel		GFAA	ug/g	619	191	263
Phosphorus		ICAP	ug/g	10600	5390	6290
Potassium		ICAP	ug/g	ND (47000)	ND (47000)	ND (29000)
Selenium		GFAA	ug/g	60.2	29.3	22.9
Sodium		ICAP	ug/g	29800	15500	14700
Titanium		ICAP	ug/g	9720	7360	6550
Vanadium		ICAP	ug/g	526	ND (440)	292

Stack Gas, Permanganate Impingers Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Mercury		CVAA	ug/Nm3	3.13	3.04	3.03

Stack Gas, Solid Phase Anions

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Chloride		IC	ug/Nm3	66.2*	73.5	33.6
Fluoride		SIE	ug/Nm3	15.7*	13.4	13.1
Sulfate		IC	ug/Nm3	640*	1080	14700

Stack Gas, Solid Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		ICAP	ug/Nm3	146	165	150
Antimony		ICAP	ug/Nm3	ND (2.9)	ND (4.5)	ND (2.8)
Arsenic		GFAA	ug/Nm3	0.107	0.109	0.0933

Site 22: Data Used in Calculations

Stack Gas, Solid Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Barium		ICAP	ug/Nm3	15.5	21.0	19.5
Beryllium		ICAP	ug/Nm3	ND (0.023)	ND (0.036)	ND (0.023)
Cadmium		GFAA	ug/Nm3	0.310	0.215	0.0526
Calcium		ICAP	ug/Nm3	333	407	353
Chromium		ICAP	ug/Nm3	0.559	ND (0.63)	ND (0.39)
Cobalt		ICAP	ug/Nm3	ND (0.54)	ND (0.83)	ND (0.52)
Copper		ICAP	ug/Nm3	0.828	0.695	0.620
Iron		ICAP	ug/Nm3	63.5	51.1	51.9
Lead		GFAA	ug/Nm3	0.141	0.125	0.137
Magnesium		ICAP	ug/Nm3	45.1	57.7	50.8
Manganese		ICAP	ug/Nm3	0.460	0.365	0.415
Mercury		CVAA	ug/Nm3	0.00829	0.00894	0.00563
Molybdenum		ICAP	ug/Nm3	2.15	2.40	2.04
Nickel		GFAA	ug/Nm3	0.836	0.398	0.545
Phosphorus		ICAP	ug/Nm3	14.3	11.2	13.0
Potassium		ICAP	ug/Nm3	ND (63)	ND (98)	ND (61)
Selenium		GFAA	ug/Nm3	0.0813	0.0611	0.0474
Sodium		ICAP	ug/Nm3	40.2	32.3	30.4
Titanium		ICAP	ug/Nm3	13.1	15.4	13.6
Vanadium		ICAP	ug/Nm3	0.710	ND (0.92)	0.605

Stack Gas, Vapor Phase Anions

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Chloride		IC	ug/Nm3	683*	867	860
Fluoride		SIE	ug/Nm3	898*	1050	1050
Sulfate		IC	ug/Nm3	1590000*	1580000	1450000

Site 22: Data Used in Calculations

Stack Gas, Vapor Phase Mercury Speciation

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Elemental mercury		CVAFS	ug/Nm3	7.3	9.2	7.5
Ionic mercury		CVAFS	ug/Nm3	ND(0.009)	0.04	ND(0.009)
Total mercury		CVAFS	ug/Nm3	7.3	9.2	7.5

Stack Gas, Vapor Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Aluminum		ICAP	ug/Nm3	7.77	5.99	8.74
Antimony		ICAP	ug/Nm3	ND (4.0)	ND (4.2)	ND (3.9)
Arsenic		GFAA	ug/Nm3	ND (0.11)	ND (0.11)	ND (0.11)
Barium		ICAP	ug/Nm3	0.316	0.217	0.315
Beryllium		ICAP	ug/Nm3	ND (0.091)	ND (0.097)	ND (0.091)
Cadmium		GFAA	ug/Nm3	ND (0.051)	ND (0.054)	ND (0.051)
Calcium		ICAP	ug/Nm3	33.4	ND (26)	30.6
Chromium		ICAP	ug/Nm3	0.421	ND (0.43)	ND (0.41)
Cobalt		ICAP	ug/Nm3	ND (0.56)	ND (0.59)	ND (0.56)
Copper		ICAP	ug/Nm3	0.814	0.690	ND (0.62)
Iron		ICAP	ug/Nm3	6.58	4.78	6.69
Lead		GFAA	ug/Nm3	ND (0.17)	ND (0.18)	ND (0.17)
Magnesium		ICAP	ug/Nm3	4.08	4.45	4.61
Manganese		ICAP	ug/Nm3	0.538	0.773	1.38
Mercury		CVAA	ug/Nm3	1.45	1.20	1.54
Molybdenum		ICAP	ug/Nm3	ND (0.76)	ND (0.81)	ND (0.76)
Nickel		GFAA	ug/Nm3	0.494	ND (0.32)	ND (0.30)
Phosphorus		ICAP	ug/Nm3	ND (10)	ND (11)	ND (10)
Potassium		ICAP	ug/Nm3	ND (61)	ND (65)	ND (61)
Selenium		GFAA	ug/Nm3	ND (0.14)	ND (0.15)	ND (0.14)
Sodium		ICAP	ug/Nm3	69.1	63.9	69.8
Titanium		ICAP	ug/Nm3	0.431	0.316	0.429
Vanadium		ICAP	ug/Nm3	ND (0.39)	ND (0.41)	ND (0.39)

* = Run 1 stack gas anions were collected on Day 2 of sampling.
B = Analyte detected in method blank.
J = Result is less than detection limit but greater than or equal to 0.
ND = Not detected.

Analytical Methods

CVAA = cold vapor atomic absorption spectrophotometry
CVAFS = cold vapor atomic fluorescence spectroscopy
D3683/ICAP = ASTM D3683/inductively coupled argon plasma emission spectrophotometry
D3761 = ASTM D3761
DGA/CVAA = double gold amalgamation cold vapor atomic absorption spectrophotometry
GFAA = graphite furnace atomic absorption spectrophotometry
HRGC/HRMS = high resolution gas chromatography/high resolution mass spectroscopy
IC = ion chromatography
ICAP = inductively coupled argon plasma emission spectrophotometry
Method 2 = EPA Method 23, high resolution gas chromatography/mass spectroscopy
NAA = neutron activation analysis
Parr/GFAA = Parr bomb digestion/graphite furnace atomic absorption spectrophotometry
Proximate = proximate analysis
SIE = selective ion electrode
Ultimate = ultimate analysis
XRF = Xray fluorescence spectroscopy
oven dry = oven drying
tot S = total sulfur analysis

Appendix C:
Data Not Used In Calculations

Site 22: Data Not Used in Calculations

Bottom Ash

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		ICAP	mg/kg	ND (51) BJ	ND (50) BJ	ND (50) B
Barium		ICAP	mg/kg	3730	3350	3060
Cadmium		ICAP	mg/kg	ND (3.1) BJ	ND (3.1) BJ	ND (3.1) BJ
Fluoride	sonication extraction, unacceptable	SIE	mg/kg	1.52 B	2.26 B	1.48 B
Lead		ICAP	mg/kg	ND (66) BJ	ND (65) BJ	ND (65) BJ
Nickel		ICAP	mg/kg	42.6 B	28.6 B	42.1 B
Selenium		ICAP	mg/kg	ND (150) J	ND (150)	ND (150)
Silicon		ICAP	mg/kg	219000 B	196000 B	203000 B
Silver		ICAP	mg/kg	ND (11)	ND (11)	ND (11)
Strontium		ICAP	mg/kg	2180	2050	1890
Thallium		ICAP	mg/kg	ND (100) BJ	ND (100) BJ	ND (100) BJ
Zinc		ICAP	mg/kg	42.1 B	39.3 B	46.8 B

Coal

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		GFAA	ug/g	ND (0.10)	ND (0.10)	ND (0.10)
Arsenic		Parr/GFAA	mg/kg	ND (1.0)	ND (1.0)	ND (1.0)
Beryllium		ICAP	ug/g	0.400	0.400	0.400
Bromine		NAA	mg/kg	0.687	0.754	0.842
Cadmium		ICAP	ug/g	ND (2.0)	ND (2.0)	ND (2.0)
Cadmium		NAA	mg/kg	ND (2.5)	ND (1.3)	ND (3.1)
Cerium		NAA	mg/kg	5.83	6.96	7.68
Cesium		NAA	mg/kg	0.109	0.124	0.148
Chlorine		NAA	mg/kg	22.9	ND (63)	87.0
Chromium		ICAP	ug/g	ND (5.0)	ND (5.0)	ND (5.0)
Chromium		NAA	mg/kg	3.77	4.24	4.91
Copper		NAA	mg/kg	19.3	11.6	ND (32)
Copper		XRF	ug/g	15.5	14.5	15.5

Site 22: Data Not Used In Calculations

Coal

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Europium		NAA	mg/kg	0.123	0.139	0.145
Hafnium		NAA	mg/kg	0.575	0.604	0.613
Iodine		NAA	mg/kg	0.330	2.65	4.27
Lanthanum		NAA	mg/kg	2.66	3.24	3.48
Lead		XRF	ug/g	15.0	22.0	19.5
Lutetium		NAA	mg/kg	0.0386	0.0419	0.0499
Manganese		NAA	mg/kg	2.15	3.24	9.95
Manganese		XRF	ug/g	25.0	25.5	26.0
Mercury		CVAA	ug/g	0.130	0.165	0.143
Mercury		NAA	mg/kg	0.0825	ND (0.031)	0.194
Neodymium		NAA	mg/kg	3.08	4.68	4.18
Nickel		D3683/ICAP	mg/kg	2.00	4.00	3.00
Nickel		XRF	ug/g	ND (5.0)	ND (5.0)	ND (5.0)
Phosphorus		XRF	ug/g	290	285	290
Rubidium		NAA	mg/kg	2.72	1.91	2.31
Samarium		NAA	mg/kg	0.501	0.681	0.670
Scandium		NAA	mg/kg	1.42	1.51	1.61
Selenium		Parr/GFAA	mg/kg	2.00	ND (1.0)	1.00
Selenium		XRF	ug/g	ND (1.0)	ND (1.0)	ND (1.0)
Silver		NAA	mg/kg	ND (0.31)	ND	ND (0.29)
Strontium		NAA	mg/kg	142	149	155
Tantalum		NAA	mg/kg	0.140	0.136	0.137
Terbium		NAA	mg/kg	0.0823	0.100	0.0967
Thorium		NAA	mg/kg	1.38	1.54	1.64
Tin		NAA	mg/kg	ND (13)	ND (6.3)	ND (13)
Titanium		D3683/ICAP	mg/kg	480	490	520
Titanium		XRF	ug/g	840	770	775
Tungsten		NAA	mg/kg	0.223	0.204	0.219
Uranium		NAA	mg/kg	0.474	0.566	0.568
Ytterbium		NAA	mg/kg	1.15	0.976	0.625

Site 22: Data Not Used In Calculations

Coal

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Zinc		NAA	mg/kg	8.39	8.95	8.72
Zirconium		NAA	mg/kg	25.5	26.7	24.3

Collected Flyash

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		ICAP	mg/kg	50.3 B	ND (52) BJ	ND (50) BJ
Cadmium		ICAP	mg/kg	ND (3.0) BJ	ND (3.2) B	ND (3.1) BJ
Fluoride	sonication extraction, unacceptable	SIE	mg/kg	93.5 B	79.5 B	79.1 B
Lead		ICAP	mg/kg	ND (64) BJ	ND (67) BJ	ND (65) BJ
Nickel		ICAP	mg/kg	57.3 B	40.0 B	53.9 B
Selenium		ICAP	mg/kg	ND (150)	ND (150)	ND (150)
Silicon		ICAP	mg/kg	183000 B	187000 B	179000 B
Silver		ICAP	mg/kg	ND (11)	ND (11)	ND (11)
Thallium		ICAP	mg/kg	ND (99) B	ND (100) BJ	ND (100) B
Zinc		ICAP	mg/kg	83.6 B	84.6 B	80.3 B

Stack Gas, Particulates Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		ICAP	ug/g	ND (920)	ND (490)	ND (580)
Cadmium		ICAP	ug/g	166	ND (91)	ND (57)
Lead		ICAP	ug/g	ND (1900)	ND (1900)	ND (1200)
Nickel		ICAP	ug/g	774	ND (700)	ND (440)
Selenium		ICAP	ug/g	ND (4400)	ND (4400)	ND (2800)
Silicon		ICAP	ug/g	18600000	11900000	12000000
Silver		ICAP	ug/g	781	ND (320)	609
Strontium		ICAP	ug/g	4220	3340	2940

Site 22: Data Not Used In Calculations

Stack Gas, Particulates Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Thallium		ICAP	ug/g	ND (3000)	ND (3000)	ND (1900)
Zinc		ICAP	ug/g	1580	800	703

Stack Gas, Solid Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		ICAP	ug/Nm3	ND (1.2)	ND (1.0)	ND (1.2)
Cadmium		ICAP	ug/Nm3	0.224	ND (0.19)	ND (0.12)
Lead		ICAP	ug/Nm3	ND (2.6)	ND (4.0)	ND (2.5)
Nickel		ICAP	ug/Nm3	1.05	ND (1.5)	ND (0.90)
Selenium		ICAP	ug/Nm3	ND (5.9)	ND (9.2)	ND (5.7)
Silicon		ICAP	ug/Nm3	25200	24900	24900
Silver		ICAP	ug/Nm3	1.06	ND (0.67)	1.26
Strontium		ICAP	ug/Nm3	5.70	6.97	6.08
Thallium		ICAP	ug/Nm3	ND (4.0)	ND (6.2)	ND (3.8)
Zinc		ICAP	ug/Nm3	2.13	1.67	1.46

Stack Gas, Vapor Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Arsenic		ICAP	ug/Nm3	ND (3.7)	ND (3.9)	ND (3.7)
Boron		ICAP	ug/Nm3	24.7	23.2	23.6
Cadmium		ICAP	ug/Nm3	ND (0.28)	ND (0.30)	ND (0.28)
Lead		ICAP	ug/Nm3	ND (4.4)	ND (4.7)	ND (4.4)
Nickel		ICAP	ug/Nm3	ND (1.6)	ND (1.7)	ND (1.6)
Selenium		ICAP	ug/Nm3	ND (6.9)	ND (7.3)	ND (6.8)
Silicon		ICAP	ug/Nm3	85.4	97.6	114
Silver		ICAP	ug/Nm3	ND (0.81)	ND (0.86)	ND (0.81)
Strontium		ICAP	ug/Nm3	0.293	0.155	0.244

Site 22: Data Not Used In Calculations

Stack Gas, Vapor Phase Multimetals

Analyte	Comment	Method	Units	Run 1	Run 2	Run 3
Thallium		ICAP	ug/Nm3	ND (2.8)	3.61	ND (2.8)
Zinc		ICAP	ug/Nm3	5.10	1.60	3.21

Appendix D: Flue Gas Sampling Data and Process Stream Flow Rates

FCEM Site 22 Sampling Summary - Multi-Metals Train - Stack

	Run 1 07/13 0854-1024 1045-1215 1303-1433 1555-1725	Run 2 07/14 0826-0956 1045-1215 1225-1355 1417-1547	Run 3 07/15 0824-0954 1000-1130 1134-1304 1314-1444	Average
Duct Diameter (ft)	24	24	24	
Pitot Tube Correction Factor	0.84	0.84	0.84	
Nozzle Diameter (inches)	0.193	0.193	0.193	
DGMCF	1.023	1.023	1.023	
Barometric Pressure (" Hg)	28.82	28.96	28.97	
Average Stack Temperature (F)	292	303	299	
Average DGM Temp (F)	92	92	94	
Delta H (in wc)	1.84	2	2.03	
Condensed Water (g)	N/A	849.3	856.7	
Test Duration (minutes)	360	360	360	
Static Pressure (in wc)	-2.5	-2.5	-2	
% CO				
% CO2	12.75	12.75	13	
% O2	8	7.5	7.75	
% N2	79.25	79.75	79.25	
% H2				
% CH4				
Meter Volume (acf)	260.784	267.145	269.801	
Average square root of delta p	1.41	1.44	1.47	
Volume at Meter (dscf)	246.95	254.30	256.01	
Flue Gas Moisture (%)	13.0	13.6	13.6	13.4
Gas Molecular Weight (Wet) (g/g-mole	28.75	28.66	28.70	
Absolute Stack Pressure (in Hg)	28.64	28.78	28.82	
Corrected Volume of Gas sampled (acf)	266.78	273.29	276.01	
Absolute Stack Temperature (R)	752	763	759	758
Average Gas Velocity (f/sec)	96.9	99.8	101.1	99.3
Avg Flow Rate (acfh)	1.58E+08	1.63E+08	1.65E+08	1.62E+08
Avg Flow Rate (acfm)	2.63E+06	2.71E+06	2.74E+06	2.69E+06
Avg Flow Rate (dscfh)	9.23E+07	9.34E+07	9.53E+07	9.37E+07
Avg Flow Rate (dscfm)	1.54E+06	1.56E+06	1.59E+06	1.56E+06
Isokinetic Sampling Rate (%)	99.3	101.0	99.7	

Moisture value for Run 1 is an average of the moisture determined on the PCDD and PAH trains.

FCEM Site 22 Sampling Summary - Anions Train - Stack

	Run 1 07/13 0916-0946 1002-1032 1052-1122 1145-1215	Run 2 07/14 0832-0902 0908-0938 0950-1020 1052-1122	Run 12 07/14 1339-1409 1420-1450 1459-1529 1536-1606	Run 3 07/15 0904-0934 0944-1014 1020-1050 1059-1129	Average
Duct Diameter (ft)	24	24	24	24	
Pitot Tube Correction Factor	0.84	0.84	0.84	0.84	
Nozzle Diameter (inches)	0.175	0.175	0.175	0.175	
DGMCF	1.003	1.003	1.003	1.003	
Barometric Pressure (" Hg)	28.82	28.96	28.96	28.99	
Average Stack Temperature (F)	300	304	310	302	
Average DGM Temp (F)	94	90	94	95	
Delta H (in wc)	1.64	1.31	1.26	1.32	
Condensed Water (g)	N/A	239.5	241.2	248.6	
Test Duration (minutes)	120	120	120	120	
Static Pressure (in wc)	-2.5	-2.5	-2.5	-2	
% CO					
% CO2	12.75	12.75	12.75	13	
% O2	8	7.5	7.5	7.75	
% N2	79.25	79.75	79.75	79.25	
% H2					
% CH4					
Meter Volume (acf)	85.357	76.647	75.913	77.471	
Average square root of delta p	1.45	1.49	1.47	1.51	
Volume at Meter (dscf)	78.95	71.67	70.46	71.87	
Flue Gas Moisture (%)	13.0	13.6	13.9	14.0	13.6
Gas Molecular Weight (Wet) (g/g-mole	28.75	28.66	28.62	28.65	
Absolute Stack Pressure (in Hg)	28.64	28.78	28.78	28.84	
Corrected Volume of Gas sampled (acf	85.61	76.88	76.14	77.70	
Absolute Stack Temperature (R)	760	764	770	762	763.9
Average Gas Velocity (f/sec)	100.1	103.0	101.9	104.1	102.3
Avg Flow Rate (acfh)	1.63E+08	1.68E+08	1.66E+08	1.70E+08	1.67E+08
Avg Flow Rate (acfm)	2.70E+06	2.80E+06	2.77E+06	2.83E+06	2.77E+06
Avg Flow Rate (dscfh)	9.43E+07	9.63E+07	9.42E+07	9.74E+07	9.55E+07
Avg Flow Rate (dscfm)	1.57E+06	1.60E+06	1.57E+06	1.62E+06	1.59E+06
Isokinetic Sampling Rate (%)	113.3	100.7	101.2	99.9	

FCEM Site 22 Sampling Summary - Semi-Volatiles/PAHs - Stack

	Run 1 07/13 1137-1237 1405-1505 1553-1653 1717-1817	Run 2 07/14 1107-1152 1200-1245 1306-1351 1430-1515	Run 3 07/15 0850-0935 1108-1153 1209-1254 1321-1406	Average
Duct Diameter (ft)	24	24	24	
Pitot Tube Correction Factor	0.84	0.84	0.84	
Nozzle Diameter (inches)	0.174	0.174	0.174	
DGMCF	1.017	1.007	1.007	
Barometric Pressure (" Hg)	28.82	28.96	28.97	
Average Stack Temperature (F)	294	305	301	
Average DGM Temp (F)	92	93	90	
Delta H (in wc)	1.34	1.36	1.21	
Condensed Water (g)	451.1	355.6	323.7	
Test Duration (minutes)	240	180	180	
Static Pressure (in wc)	-2.5	-2.5	-2	
% CO				
% CO2	12.75	12.75	13	
% O2	8	7.5	7.75	
% N2	79.25	79.75	79.25	
% H2				
% CH4				
Meter Volume (acf)	148.43	123.19	114.4	
Average square root of delta p	1.47	1.52	1.46	
Volume at Meter (dscf)	139.56	115.04	107.41	
Flue Gas Moisture (%)	13.2	12.7	12.5	12.8
Gas Molecular Weight (Wet) (g/g-mole	28.72	28.77	28.85	
Absolute Stack Pressure (in Hg)	28.64	28.78	28.82	
Corrected Volume of Gas sampled (acf)	150.95	124.05	115.20	
Absolute Stack Temperature (R)	754	765	761	760
Average Gas Velocity (f/sec)	101.0	104.8	100.1	102.0
Avg Flow Rate (acfh)	1.65E+08	1.71E+08	1.63E+08	1.66E+08
Avg Flow Rate (acfm)	2.74E+06	2.84E+06	2.72E+06	2.77E+06
Avg Flow Rate (dscfh)	9.57E+07	9.89E+07	9.54E+07	9.67E+07
Avg Flow Rate (dscfm)	1.59E+06	1.65E+06	1.59E+06	1.61E+06
Isokinetic Sampling Rate (%)	99.8	106.2	102.8	

FCEM Site 22 Sampling Summary - PCDD/PCDF- Method 23 - Stack

	Run 1 07/13 1252-1352 1410-1510 1548-1648 1712-1812	Run 2 07/14 0925-1010 1100-1145 1205-1250 1311-1356	Run 3 07/15 0911-0956 1027-1113 1142-1227 1305-1350	Average
Duct Diameter (ft)	24	24	24	
Pitot Tube Correction Factor	0.84	0.84	0.84	
Nozzle Diameter (inches)	0.174	0.173	0.176	
DGMCF	1.007	1.017	1.017	
Barometric Pressure (" Hg)	28.82	28.96	28.97	
Average Stack Temperature (F)	290	305	303	
Average DGM Temp (F)	91	90	90	
Delta H (in wc)	1.26	1.40	1.45	
Condensed Water (g)	445.6	350	356.3	
Test Duration (minutes)	240	180	181	
Static Pressure (in wc)	-2.5	-2.5	-2	
% CO				
% CO2	12.75	12.75	13	
% O2	8	7.5	7.75	
% N2	79.25	79.75	79.25	
% H2				
% CH4				
Meter Volume (acf)	155.1	112.34	114.53	
Average square root of delta p	1.47	1.54	1.53	
Volume at Meter (dscf)	144.63	106.54	108.67	
Flue Gas Moisture (%)	12.7	13.4	13.4	13.2
Gas Molecular Weight (Wet) (g/g-mole	28.79	28.68	28.73	
Absolute Stack Pressure (in Hg)	28.64	28.78	28.82	
Corrected Volume of Gas sampled (acf)	156.19	114.25	116.48	
Absolute Stack Temperature (R)	750	765	763	759
Average Gas Velocity (f/sec)	100.4	106.5	105.4	104.1
Avg Flow Rate (acfh)	1.64E+08	1.73E+08	1.72E+08	ff0
Avg Flow Rate (acfm)	2.73E+06	2.89E+06	2.86E+06	2.83E+06
Avg Flow Rate (dscfh)	9.61E+07	9.96E+07	9.91E+07	9.83E+07
Avg Flow Rate (dscfm)	1.60E+06	1.66E+06	1.65E+06	1.64E+06
Isokinetic Sampling Rate (%)	103.0	98.7	97.3	

Table D-5
Site 22 Stream Flow Rates

Parameter	Run 1	Run 2	Run 3	Avg	Std. Dev.	Measured (M) or Calculated (C)
Coal Rate(kg/hr as received)	346550	361066	360158	355925	8131	M
Coal Rate(kg/hr dry)	251076	255418	245952	250815	4738	M
Coal Ash Rate(kg/hr)	16219	17011	17881	17037	831	M
Bottom Ash Rate (kg/hr)	3244	3402	3576	3407	166	C
Collected Fly Ash Rate (kg/hr)	12972	13604	14299	13625	664	C
Stack Gas Rate (Nm3/hr)	2502447	2549961	2549961	2534123	27433	M
Stack Partuculate Rate (kg/hr)	3.3	5.1	5.2	4.5	1.1	M
Stack Particulate Loading (mg/Nm3)	1.35	2.09	2.07	1.8	0.4	M
ESP Inlet Particulate Loading (mg/Nm3)	5185	5337	5610	5377	215	C
ESP Inlet Particulate Rate (kg/hr)	12976	13609	14305	13630	665	C
Coal Ash (wt. %, dry)	6.46	6.66	7.27	6.80	0.42	M
Coal Moisture (wt. %)	27.6	29.3	31.7	29.51	2.09	M
Coal HHV (Btu/lb, dry)	12075	11977	11892	11981	92	M

Appendix E: Uncertainty Analysis

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 through 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.

This method 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;

B_r = Bias component of result uncertainty;

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

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:

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

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

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

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

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

$$\theta_i = \frac{\partial r}{\partial pi} \quad (4)$$

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

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

Equation 5 was applied to the calculations in this report. The perturbation selected for each parameter was the larger of the normalized standard deviation, $S_{\bar{p}i}$, or the bias, β_{pi}.

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

$$S_{\bar{p}i} = \frac{S_{pi}}{\sqrt{N}} \quad (6)$$

The degrees of freedom for each parameter is found from

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

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

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

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

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

- 5% bias on coal flow rates.

- No bias in gas flow rates.
- No bias in analytical results if the result is greater than the reporting limit. One-half of the reporting limit is used for both the parameter value and its bias in calculations if the result is below the reporting limit.

Assignment of the flow rate bias values is based on 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 detection limit may be especially large. The uncertainty values calculated for this report are, therefore, subject to these limitations.

The calculations assume that the population distribution of each measurement is normal 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.

Improvements in bias estimates will be made as more data are collected and the QA/QC database is expanded. Spike and standard recoveries can be used to estimate analytical bias. Also, as the analytical methods improve, accuracy will improve, resulting in the true bias of the analytical results being closer to the zero bias now assigned. Accounting for long-term system variability will require repeated sampling trips to the same location.

Appendix F: Quality Assurance/Quality Control

The objective of quality assurance and quality control (QA/QC) efforts associated with the Site 22 study is to ensure that all data collected are of known and sufficient quality to characterize the various process streams. This section addresses the QA/QC associated with the chemical analyses of gas, solid, and aqueous samples from the program.

The tables in this section include QA/QC data for all of the analytes that were measured as part of the multi-element analytical methods which were applied in this study. However, only the QA/QC information associated with the target analytes listed in Section 1 will be discussed.

Summary of Data Quality and QA/QC Approach

Quality assurance and quality control procedures used for this program are consistent with those described in the Site 22 Sampling and Analytical Plan and the Laboratory Quality Assurance Program Plan for Radian's Austin Laboratories. The following key types of QA/QC provide the primary basis for quantitatively evaluating data quality:

- Laboratory and field blank samples;
- Laboratory quality control check samples;
- Laboratory spiked samples;
- Duplicate samples;

- Duplicate analyses; and
- Performance evaluation audit samples.

Quality assurance/quality control data associated with the sampling and analytical procedures for this study indicate that, with a few exceptions discussed in Section 4, the data quality was acceptable for the associated samples and analyses . There were QC indicators that were outside nominal laboratory objectives, but these are not intended as validation criteria: rather, they are meant to indicate where potential problems might exist, and thereby prompt further scrutiny by the data users. Reanalysis and alternate approaches were followed as necessary to obtain acceptable data. Ultimately, the data may be considered valid and usable for the project needs. Quality control data are summarized in Tables F-1 through F-5. These include results for metals that, while they are not specifically of interest to the program, they may be considered in evaluating the quality of the primary elements of interest.

Blank sample results are summarized in Table F-1. Table F-2 presents a summary of laboratory control sample results. Matrix spiked sample results are summarized in Table F-3. Duplicate analysis results are listed in Table F-4. Table F-5 is a summary of audit sample results. Surrogate recoveries for PAH and PCDD/PCDF analyses are collected in Table F-6.

Table F-1
Summary of Blank Sample Results

Analyte	No. of Blanks Analyzed	No. of Detects [a]	Range of Compounds Detected	Detection Limit
Polynuclear Aromatic Hydrocarbons				
Laboratory (Method) Blanks (XAD)				
Acenaphthalene	4	1	4.4 ng	NS
Acenaphthene	4	1	9.2 ng	NS
Fluorene	4	1	15.7 ng	NS
Phenanthrene	4	1	44.5 ng	NS
Anthracene	4	1	4.4 ng	NS
Fluoranthene	4	1	11.3 ng	NS
Pyrene	4	1	6.8 ng	NS
Benzo(a)anthracene	4	1	0.4 ng	NS
Chrysene	4	1	1.1 ng	NS
5-methyl chrysene	4	0	ND	0.16 ng
Benzo(b,j&k)fluoranthenes	4	1	1.8 ng	NS
Benzo(a)pyrene	4	0	ND	4.5 ng
Dibenz(a,h)acridine	4	1	5.9 ng	NS
Dibenz(a,i)acridine	4	1	9.3 ng	NS
Indeno(1,2,3-cd)pyrene	4	1	3.2 ng	NS
Dibenz(a,h)anthracene	4	1	3.0 ng	NS
7H-dibenzo(c,g)carbazole	4	1	6.9 ng	NS
Benzo(g,h,i)perylene	4	1	10.8 ng	NS
Dibenzo(a,e)pyrene	4	1	2.7 ng	3.4 ng
Dibenzo(a,i)pyrene	4	1	2.2 ng	2.4 ng
Dibenzo(a,h)pyrene	4	1	2.6 ng	3.3 ng
Trip Blanks - PAHs				
Acenaphthalene	1	1	2.3 ng	NS
Acenaphthene	1	1	7.7 ng	NS
Fluorene	1	1	14.9 ng	NS
Phenanthrene	1	1	72.2 ng	NS
Anthracene	1	1	5.9 ng	NS
Fluoranthene	1	1	20.6 ng	NS
Pyrene	1	1	15.6 ng	NS
Benzo(a)anthracene	1	0	ND	1.7 ng
Chrysene	1	1	1.5 ng	NS
5-methyl chrysene	1	0	ND	1.4 ng
Benzo(b,j&k)fluoranthenes	1	1	2.5 ng	NS
Benzo(a)pyrene	1	1	1.8 ng	NS
Dibenz(a,h)acridine	1	1	2.6 ng	NS
Dibenz(a,i)acridine	1	1	6.5 ng	NS
Indeno(1,2,3-cd)pyrene	1	1	1.9 ng	NS

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Dibenz(a,h)anthracene	1	1	1.2 ng	NS
7H-dibenzo(c,g)carbazole	1	0	ND	8.7 ng
Benzo(g,h,i)perylene	1	1	12.9 ng	NS
Dibenzo(a,e)pyrene	1	0	ND	2.9 ng
Dibenzo(a,i)pyrene	1	0	ND	3.7 ng
Dibenzo(a,h)pyrene	1	0	ND	3.2 ng
Field Blanks - PAHs				
Acenaphthalene	1	1	1.8 ng	NS
Acenaphthene	1	1	6.2 ng	NS
Fluorene	1	1	8.3 ng	NS
Phenanthrene	1	1	27.7 ng	NS
Anthracene	1	1	3.5 ng	NS
Fluoranthene	1	1	10.7 ng	NS
Pyrene	1	1	5.7 ng	NS
Benzo(a)anthracene	1	1	1.0 ng	NS
Chrysene	1	1	1.5 ng	NS
5-methyl chrysene	1	0	ND	1.3 ng
Benzo(b,j&k)fluoranthenes	1	1	3.4 ng	NS
Benzo(a)pyrene	1	1	1.4 ng	NS
Dibenz(a,h)acridine	1	1	3.0 ng	NS
Dibenz(a,i)acridine	1	1	5.4 ng	NS
Indeno(1,2,3-cd)pyrene	1	1	1.9 ng	NS
Dibenz(a,h)anthracene	1	0	ND	1.7 ng
7H-dibenzo(c,g)carbazole	1	0	ND	8.2 ng
Benzo(g,h,i)perylene	1	1	6.9 ng	NS
Dibenzo(a,e)pyrene	1	0	ND	1.4 ng
Dibenzo(a,i)pyrene	1	0	ND	2.8 ng
Dibenzo(a,h)pyrene	1	0	ND	4.1 ng
Method Blank - PCDD/PCDF				
2,3,7,8-TCDF	1	0	ND	0.0059 ng
2,3,7,8-TCDD	1	0	ND	0.0150 ng
1,2,3,7,8-PeCDF	1	0	ND	0.0045 ng
2,3,4,7,8-PeCDF	1	0	ND	0.0059 ng
1,2,3,7,8-PeCDD	1	0	ND	0.0057 ng
1,2,3,4,7,8-HxCDF	1	0	ND	0.0100 ng
1,2,3,6,7,8-HxCDF	1	0	ND	0.0067 ng
2,3,4,6,7,8-HxCDF	1	0	ND	0.0042 ng
1,2,3,7,8,9-HxCDF	1	0	ND	0.0051 ng
1,2,3,4,7,8-HxCDD	1	0	ND	0.0098 ng

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
1,2,3,6,7,8-HxCDD	1	0	ND	0.0082 ng
1,2,3,7,8,9-HxCDD	1	0	ND	0.0100 ng
1,2,3,4,6,7,8-HpCDF	1	1	0.0085	–
1,2,3,4,7,8,9-HpCDF	1	0	ND	0.0110 ng
1,2,3,4,6,7,8-HpCDD	1	1	0.0170	–
OCDF	1	1	0.0140	–
OCDD	1	1	0.1200	–
Trip Blank - PCDD/PCDF				
2,3,7,8-TCDF	1	0	ND	0.0064 ng
2,3,7,8-TCDD	1	0	ND	0.0130 ng
1,2,3,7,8-PeCDF	1	0	ND	0.0095 ng
2,3,4,7,8-PeCDF	1	0	ND	0.0086 ng
1,2,3,7,8-PeCDD	1	0	ND	0.0094 ng
1,2,3,4,7,8-HxCDF	1	0	ND	0.0049 ng
1,2,3,6,7,8-HxCDF	1	0	ND	0.0061 ng
2,3,4,6,7,8-HxCDF	1	1	0.0069 ng	–
1,2,3,7,8,9-HxCDF	1	0	ND	0.0081 ng
1,2,3,4,7,8-HxCDD	1	0	ND	0.0110 ng
1,2,3,6,7,8-HxCDD	1	0	ND	0.0110 ng
1,2,3,7,8,9-HxCDD	1	0	ND	0.0082 ng
1,2,3,4,6,7,8-HpCDF	1	1	0.0180 ng	–
1,2,3,4,7,8,9-HpCDF	1	0	ND	0.0099 ng
1,2,3,4,6,7,8-HpCDD	1	0	ND	–
OCDF	1	0	ND	–
OCDD	1	0	ND	–
Field Blank - PCDD/PCDF				
2,3,7,8-TCDF	1	0	ND	0.0064 ng
2,3,7,8-TCDD	1	0	ND	0.0200 ng
1,2,3,7,8-PeCDF	1	0	ND	0.0140 ng
2,3,4,7,8-PeCDF	1	0	ND	0.0060 ng
1,2,3,7,8-PeCDD	1	0	ND	0.0086 ng
1,2,3,4,7,8-HxCDF	1	0	ND	0.0150 ng
1,2,3,6,7,8-HxCDF	1	0	ND	0.0360 ng
2,3,4,6,7,8-HxCDF	1	0	ND	0.0080 ng
1,2,3,7,8,9-HxCDF	1	0	ND	0.0100 ng
1,2,3,4,7,8-HxCDD	1	0	ND	0.0088 ng
1,2,3,6,7,8-HxCDD	1	0	ND	0.0160 ng
1,2,3,7,8,9-HxCDD	1	0	ND	0.0110 ng
1,2,3,4,6,7,8-HpCDF	1	0	ND	0.0170 ng
1,2,3,4,7,8,9-HpCDF	1	0	ND	0.0130 ng
1,2,3,4,6,7,8-HpCDD	1	1	0.018 ng	–

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
OCDF	1	0	ND	0.0320 ng
OCDD	1	1	0.120	–
Laboratory (Method) Blanks - Ashes				
ICP-AES Metals				
Aluminum	2	2	47.4-64.3 mg/kg	135 mg/kg
Antimony	2	1	1.90 mg/kg	75.5 mg/kg
Arsenic	2	2	12.7-12.9 mg/kg	52.0 mg/kg
Barium	2	1	0.350 mg/kg	2.15 mg/kg
Beryllium	2	1	0.390 mg/kg	0.610 mg/kg
Boron	NA	NA	NA	15.7 mg/kg
Cadmium	2	2	0.570 - 1.81 mg/kg	3.21 mg/kg
Calcium	2	2	15.2-25.5 mg/kg	225 mg/kg
Chromium	2	2	3.83-4.01 mg/kg	10.5 mg/kg
Cobalt	2	2	6.40-7.53 mg/kg	14.0 mg/kg
Copper	2	1	2.14 mg/kg	10.5 mg/kg
Iron	2	2	6.96-9.76 mg/kg	309 mg/kg
Lead	2	1	11.1 mg/kg	67.3 mg/kg
Magnesium	2	1	8.43 mg/kg	92.1 mg/kg
Manganese	2	1	0.800 mg/kg	2.72 mg/kg
Molybdenum	2	1	3.74 mg/kg	14.6 mg/kg
Nickel	2	1	1.03 mg/kg	24.4 mg/kg
Potassium	2	0	ND	1640 mg/kg
Selenium	2	0	ND	155 mg/kg
Silicon	2	2	6720-6870 mg/kg	132 mg/kg
Silver	2	0	ND	11.2 mg/kg
Sodium	2	2	324-337 mg/kg	61.2 mg/kg
Strontium	2	1	0.390 mg/kg	1.38 mg/kg
Thallium	2	2	3.68-72.8 mg/kg	104 mg/kg
Titanium	2	0	ND	4.20 mg/kg
Vanadium	2	2	4.64-6.02 mg/kg	15.5 mg/kg
Zinc	2	1	0.870 mg/kg	2.73 mg/kg
Laboratory (Method) Blanks - Ashes				
GFAAS and CVAAS Metals				
Arsenic	1	1	0.05 mg/kg	0.0933 mg/kg
Cadmium	1	1	0.240 mg/kg	0.423 mg/kg
Lead	1	0	ND	0.11 mg/kg
Mercury	2	1	0.0025 mg/kg	0.012 mg/kg
Nickel	1	0	ND	0.117 mg/kg
Selenium	1	0	ND	1.16 mg/kg
Laboratory (Method) Blanks - Impingers				
ICP-AES Metals				
Aluminum	1	0	ND	0.0284 mg/L
Antimony	1	0	ND	0.0241 mg/L
Arsenic	1	1	0.00139 mg/L	0.0225 mg/L

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Barium	1	0	ND	0.0005 mg/L
Beryllium	1	0	ND	0.0005 mg/L
Boron	1	1	0.0128 mg/L	0.0150 mg/L
Cadmium	1	1	0.0007 mg/L	0.00172 mg/L
Calcium	1	1	0.00836 mg/L	0.148 mg/L
Chromium	1	1	0.00250 mg/L	0.00249 mg/L
Cobalt	1	0	ND	0.00340 mg/L
Copper	1	1	0.00764 mg/L	0.00381 mg/L
Iron	1	1	0.00366 mg/L	0.00596 mg/L
Lead	1	0	ND	0.0270 mg/L
Magnesium	1	1	0.00386 mg/L	0.0228 mg/L
Manganese	1	1	0.00046 mg/L	0.000395 mg/L
Molybdenum	1	0	ND	0.00463 mg/L
Nickel	1	0	ND	0.00986 mg/L
Potassium	1	1	0.351 mg/L	0.370 mg/L
Selenium	1	1	0.002 mg/L	0.0417 mg/L
Silicon	1	1	0.0608 mg/L	0.0273 mg/L
Silver	1	1	0.00188 mg/L	0.00492 mg/L
Sodium	1	1	0.0162 mg/L	0.0397 mg/L
Strontium	1	0	ND	0.000166 mg/L
Thallium	1	0	ND	0.0172 mg/L
Titanium	1	1	0.0002 mg/L	0.00102 mg/L
Vanadium	1	1	0.00290 mg/L	0.00236 mg/L
Zinc	1	0	ND	0.00153 mg/L
Laboratory (Method) Blanks - Impingers				
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.00657 mg/L
Cadmium	1	1	0.00013 mg/L	0.00031 mg/L
Lead	1	0	ND	0.00105 mg/L
Mercury	2	0	ND	0.000048 µg/L
Nickel	1	1	0.0001 µg/L	0.00182 mg/L
Selenium	1	0	ND	0.00084 mg/L
Laboratory (Method) Blank - Filter + Probe and Nozzle Rinse				
Anions				
Chloride (BIF)	1	0	ND	0.02 mg/L
Fluoride (EPA 340.2)	1	1	0.0202 mg/L	0.0235 mg/L
Sulfate (EPA 300)	1	1	0.412 mg/L	0.06 mg/L
Laboratory (Method) Blank - Ashes				
Anions				
Chloride (SM 407C)	1	0	ND	100 mg/kg
Fluoride (EPA 340.2)	1	1	0.470 mg/kg	0.470 mg/kg
Sulfur (LECO)	1	0	ND	0.005%
Lab (Method) Blank - Impingers				
Chloride (EPA 300.0)	1	0	ND	0.02 mg/L
Fluoride (EPA 340.2)	1	1	0.0202 mg/L	0.0235 mg/L
Sulfate (EPA 300.0)	1	1	0.412 mg/L	0.06 mg/L

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Laboratory (Method) Blanks - Filter + Probe and Nozzle Rinse				
ICP-AES Metals				
Aluminum	2	1	ND-1.37 μg	13.5 μg
Antimony	2	0	ND	7.55 μg
Arsenic	2	0	ND	5.2 μg
Barium	2	0	ND	2.15 μg
Beryllium	2	0	ND	0.061 μg
Boron	NA	NA	NA	1.57 μg
Cadmium	2	0	ND	0.321 μg
Calcium	2	0	ND	22.5 μg
Chromium	2	1	ND-0.615 μg	1.05 μg
Cobalt	2	2	0.249-0.336 μg	1.40 μg
Copper	2	2	0.224-0.315 μg	1.05 μg
Iron	2	1	ND-0.513 μg	30.9 μg
Lead	2	1	ND-0.820 μg	6.73 μg
Magnesium	2	0	ND	9.21 μg
Manganese	2	1	ND-0.0290 μg	0.272 μg
Molybdenum	2	0	ND	1.46 μg
Nickel	2	2	0.616-2.76 μg	2.44 μg
Potassium	2	1	ND-1.36 μg	164 μg
Selenium	2	1	ND-3.11 μg	15.5 μg
Silicon	2	2	111-126 μg	13.2 μg
Silver	2	0	ND	1.12 μg
Sodium	2	0	ND-0.731 μg	6.12 μg
Strontium	2	1	ND-0.210 μg	0.138 μg
Thallium	2	2	0.965-2.70 μg	10 μg
Titanium	1	1	0.147 μg	0.420 μg
Vanadium	2	2	0.3160-0.439 μg	1.55 μg
Zinc	1	0	ND	0.273 μg
Laboratory (Method) Blanks - Filters + Probe and Nozzle Rinse				
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.0933 μg
Cadmium	1	0	ND	0.1 μg
Lead	1	1	0.01 μg	0.11 μg
Mercury	1	1	0.014 μg	0.0096 μg
Nickel	1	0	ND	0.117 μg
Selenium	1	0	ND	0.116 μg
Field Blanks - $HCO_3/CO_3/H_2O_2$ Impingers				
Anions				
Chloride	1	1	53.8 μg	0.02 μg
Fluoride	1	1	12.0 μg	5.76 μg
Sulfate	1	1	189 μg	0.06 μg

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Field Blanks - Probe and Nozzle Rinse + Filter				
Anions				
Chloride (EPA 300)	1	1	100 μg	0.02 μg
Fluoride (EPA 340.2)	1	1	14.1 mg/L	6.11 μg
Sulfate (EPA 300)	1	1	522 μg	0.06 μg
Field Blanks - Stack Gas - H_2O_2/HNO_3 Impinger Solutions				
ICP-AES Metals				
Aluminum	1	1	0.0374 mg/L	0.0284 mg/L
Antimony	1	1	0.0260 mg/L	0.0241 mg/L
Arsenic	1	0	ND	0.0255 mg/L
Barium	1	1	0.00179 mg/L	0.000530 mg/L
Beryllium	1	0	ND	0.000554 mg/L
Boron	1	1	0.0210 mg/L	0.0150 mg/L
Cadmium	1	1	0.000280 mg/L	0.00172 mg/L
Calcium	1	1	0.152 mg/L	0.148 mg/L
Chromium	1	1	0.00177 mg/L	0.00249 mg/L
Cobalt	1	1	0.00350 mg/L	0.00340 mg/L
Copper	1	1	0.0136 mg/L	0.00381 mg/L
Iron	1	1	0.0507 mg/L	0.00596 mg/L
Lead	1	0	ND	0.0270 mg/L
Magnesium	1	1	0.00903 mg/L	0.0228 mg/L
Manganese	1	1	0.00444 mg/L	0.000395 mg/L
Molybdenum	1	0	ND	0.00463 mg/L
Nickel	1	0	ND	0.00986 mg/L
Potassium	1	0	ND	0.370 mg/L
Selenium	1	1	0.00116 mg/L	0.0417 mg/L
Silicon	1	1	0.392 mg/L	0.0273 mg/L
Silver	1	1	0.00438 mg/L	0.00492 mg/L
Sodium	1	1	0.426 mg/L	0.0397 mg/L
Strontium	1	1	0.00089 mg/L	0.000166 mg/L
Thallium	1	0	ND	0.0172 mg/L
Titanium	1	1	0.00222 mg/L	0.00102 mg/L
Vanadium	1	0	ND	0.00236 mg/L
Zinc	1	1	0.0184 mg/L	0.00153 mg/L
Field Blanks - Stack Gas - $H_2O_2HNO_3$ Impinger Solutions				
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.000657 mg/L
Cadmium	1	1	0.000140 mg/L	0.000310 mg/L
Lead	1	1	0.000680 mg/L	0.00105 mg/L
Mercury	1	1	0.00310 mg/L	0.00690 mg/L
Nickel	1	1	0.00100 mg/L	0.00182 mg/L
Selenium	1	0	ND	0.000843 mg/L
Field Blanks - Stack Gas - H_2SO_4/$KMnO_4$ Impinger Solutions				
Mercury	1	0	ND	0.000240 μg/L

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Trip Blank - Method 5 Filter				
ICP Metals				
Aluminum	3	3	73.5-81.2 μg	13.5 μg
Antimony	3	0	ND	7.55 μg
Arsenic	3	0	NA	30.2 μg
Barium	3	3	3.16-3.46 μg	0.215 μg
Beryllium	3	0	ND	0.0610 μg
Cadmium	3	2	0.178-0.340 μg	0.321 μg
Calcium	3	3	41.5-44.3 μg	22.5 μg
Chromium	3	2	0.690-0.0730 μg	1.05 μg
Cobalt	3	1	0.248 μg	1.40 μg
Copper	3	3	0.473-0.500 μg	1.05 μg
Iron	3	3	0.184-1.92 μg	30.9 μg
Lead	3	3	1.27-4.88 μg	6.73 μg
Magnesium	3	3	5.54-7.30 μg	9.21 μg
Manganese	3	3	0.170-0.272 μg	0.272 μg
Molybdenum	3	3	11.3-12.6 μg	1.46 μg
Nickel	3	1	0.0750	2.44 μg
Potassium	3	1	41.7 μg	164 μg
Selenium	3	2	0.610-2.15 μg	15.5 μg
Silicon	3	3	168000-175000 μg	52.8 μg
Silver	3	0	ND	1.12 μg
Sodium	3	3	74.0-90.1 μg	6.12 μg
Strontium	3	3	0.379-0.443 μg	0.138 μg
Thallium	3	0	ND	10.4 μg
Titanium	3	1	1.70 μg	41.6 μg
Vanadium	3	3	0.432-0.997 μg	1.55 μg
Zinc	3	1	0.177 μg	0.273 μg
Trip Blank - Method 5 Filter				
GFAAS and CVAAS Metals				
Arsenic	3	0	ND	0.0933 μg
Cadmium	3	0	ND	0.1 μg
Lead	3	3	0.100-0.120 μg	0.110 μg
Mercury	3	3	0.0260-0.0480 μg	0.00960 μg
Nickel	3	3	0.300-0.530 μg	0.117 μg
Selenium	3	0	ND	0.116 μg
Field Blank - Stack Gas - PNR + Filter				
ICP-AES Metals				
Aluminum	1	1	166 μg	13.5 μg
Antimony	1	0	ND	7.55 μg
Arsenic	1	0	ND	20.8 μg
Barium	1	1	6.04 μg	0.215 zμg
Beryllium	1	0	ND	0.0610 μg

Table F-1 Continued

Analyte	No. of Blanks Analyzed	No. of Detects	Range of Compounds Detected	Detection Limit
Cadmium	1	1	0.548 μg	0.321 μg
Calcium	1	1	212 μg	22.5 μg
Chromium	1	1	1.44 μg	1.05 μg
Cobalt	1	1	0.754 μg	1.40 μg
Copper	1	1	1.57 μg	1.05 μg
Iron	1	1	46.3 μg	30.9 μg
Magnesium	1	1	31.4 μg	9.21 μg
Manganese	1	1	0.769 μg	0.272 μg
Molybdenum	1	1	13.3 μg	1.46 μg
Nickel	1	1	4.24 μg	2.44 μg
Potassium	1	1	67.0 μg	164 μg
Selenium	1	1	0.866 μg	15.5 μg
Silicon	1	1	2220 μg	52.8 μg
Silver	1	1	3.86 μg	4.48 μg
Sodium	1	1	270 μg	24.5 μg
Strontium	1	1	1.90 μg	0.138 μg
Thallium	1	0	ND	10.4 μg
Titanium	1	1	5.80 μg	1.68 μg
Vanadium	1	1	0.724 μg	1.55 μg
Zinc	1	1	10.1 μg	0.273 μg
Field Blank - Stack Gas - Filter				
GFAAS and CVAAS Metals				
Arsenic	1	1	0.250 μg	0.0933 μg
Cadmium	1	1	0.599 μg	0.1 μg
Lead	1	1	0.370 μg	0.110 μg
Mercury	1	1	0.0240 μg	0.00960 μg
Nickel	1	1	4.09 μg	0.117 μg
Selenium	1	0	ND	0.116 μg

[a] Reporting limits for aqueous and impinger samples are generally the method reporting limits which may be greater than or equal to the instrument specific (laboratory derived) method detection limits. Reporting limits for solid samples and probe/nozzle rinses plus filter samples are the sample or matrix reporting limits which are calculated by multiplying the method reporting limits for a particular analyte by the dilution factor for that sample.

Table F-2
Summary of Quality Control Check Sample Results for Site 22

Analyte	No. of LCS/ LCSD Pairs	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
Metals by ICP-AES - Impingers						
Aluminum	1	95	1.0	0	0	80-120
Antimony	1	100	0.0	0	0	80-120
Arsenic	1	102	2.0	0	0	80-120
Barium	1	98	1.0	0	0	80-120
Beryllium	1	102	1.0	0	0	80-120
Boron	1	103	0.0	0	0	80-120
Cadmium	1	96	4.0	0	0	80-120
Calcium	1	100	1.0	0	0	80-120
Chromium	1	98	1.0	0	0	80-120
Cobalt	1	96	1.0	0	0	80-120
Copper	1	98	1.0	0	0	80-120
Iron	1	99	0	0	0	80-120
Lead	2	96	0	0	0	80-120
Magnesium	1	96	1.0	0	0	80-120
Manganese	1	96	1.0	0	0	80-120
Molybdenum	1	96	1.0	0	0	80-120
Nickel	1	99	0.0	0	0	80-120
Potassium	1	98	3.0	0	0	80-120
Selenium	1	96	3.0	0	0	80-120
Silicon	1	100	1.0	0	0	80-120
Silver	1	96	1.0	0	0	80-120
Sodium	1	90	1.0	0	0	80-120
Strontium	1	98	1.0	0	0	80-120
Thallium	1	98	1.0	0	0	80-120
Titanium	1	98	1.0	0	0	80-120
Vanadium	1	100	2.0	0	0	80-120
Zinc	1	96	1.0	0	0	80-120

Table F-2 (Continued)

Analyte	No. of LCS/ LCSD Pairs	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
GFAAS and CVAAS Metals - Impingers						
Arsenic	1	102	6.0	0	0	75-125
Cadmium	1	106	4.0	0	0	75-125
Lead	1	102	2.0	0	0	75-125
Mercury	2	100	2.0	0	0	80-120
Nickel	1	99	1.0	0	0	75-125
Selenium	1	80	7.0	1	0	75-125
Anions (Spikes into Reagent Waters) - Impingers						
Chloride	1	100	0	0	0	83-112
Fluoride	1	94	4	0	0	90-110
Sulfate	1	98	0	0	0	85-107
Metals by ICP-AES (Spikes into Reagent Water) - Ashes						
Aluminum	2	93	0.6	0	0	80-120
Antimony	2	79	4.3	3	0	80-120
Arsenic	2	92	14	1	0	80-120
Barium	2	98	0	0	0	80-120
Beryllium	2	88	0	0	0	80-120
Cadmium	2	81	6.6	1	0	80-120
Calcium	2	95	0	0	0	80-120
Chromium	2	92	1.1	0	0	80-120
Cobalt	2	91	1.1	0	0	80-120
Copper	2	95	1.1	0	0	80-120
Iron	2	90	0	0	0	80-120
Lead	2	88	3.4	0	0	80-120
Magnesium	2	93	0.6	0	0	80-120
Manganese	2	92	1.1	0	0	80-120
Molybdenum	2	89	2.8	0	0	80-120

Table F-2 (Continued)

Analyte	No. of LCS/ LCSD Pairs	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
Nickel	2	90	1.6	0	0	80-120
Potassium	2	94	0.6	0	0	80-120
Selenium	2	104	73	1	2	80-120
Silicon	2	97	0.5	0	0	80-120
Silver	2	33	4.6	4	0	80-120
Sodium	2	92	0	0	0	80-120
Strontium	2	95	0	0	0	80-120
Thallium	2	90	3.4	0	0	80-120
Titanium	2	92	0.6	0	0	80-120
Vanadium	2	93	0.6	0	0	80-120
Zinc	2	88	0	0	0	80-120
GFAAS and CVAAS Metals (Spikes into Reagent Water) - Ashes						
Arsenic	1	121	1.0	0	0	75-125
Cadmium	1	113	1.0	0	0	75-125
Lead	1	103	2.0	0	0	75-125
Mercury	2	101	0.0	0	0	75-125
Nickel	1	106	2.8	0	0	75-125
Selenium	1	96	6.3	0	0	75-125
Anions (Spikes into Reagent Water) - Ashes						
Chloride	1	99	0	0	0	80-120
Fluoride	1	99	13	0	0	80-120
Metals by ICP-AES (Spikes into Reagent Water) - Filters						
Aluminum	2	91	2.0	0	0	80-120
Arsenic	2	107	6.0	0	0	80-120
Barium	2	96	2.0	0	0	80-120
Beryllium	2	89	2.0	0	0	80-120

Table F-2 (Continued)

Analyte	No. of LCS/ LCSD Pairs	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
Cadmium	2	85	3.0	0	0	80-120
Calcium	2	94	2.0	0	0	80-120
Chromium	2	92	2.0	0	0	80-120
Cobalt	2	91	2.0	0	0	80-120
Copper	2	94	2.0	0	0	80-120
Iron	2	90	2.0	0	0	80-120
Lead	2	90	8.0	0	0	80-120
Magnesium	2	93	2.0	0	0	80-120
Manganese	2	92	2.0	0	0	80-120
Molybdenum	2	84	2.0	0	0	80-120
Nickel	2	92	4.0	0	0	80-120
Potassium	2	94	2.0	0	0	80-120
Selenium	2	1137	756	2	2	80-120
Silicon	2	95	3.0	0	0	80-120
Silver	2	21	106	4	0	80-120
Sodium	2	92	2.0	0	0	80-120
Strontium	2	96	2.0	0	0	80-120
Thallium	2	83	1.0	0	0	80-120
Titanium	2	90	2.0	0	0	80-120
Vanadium	2	93	2.0	0	0	80-120
Zinc	2	69	1.0	2	0	80-120
Metals by GFAA or CVAA - Filters						
Antimony	1	106	3	0	0	75-125
Cadmium	1	104	1	0	0	75-125
Lead	1	93	1	0	0	80-120
Mercury	2	110	1.0	0	0	75-125
Nickel	1	106	0	0	0	75-125
Selenium	1	78	1	0	0	75-125

Table F-2 (Continued)

Analyte	No. of LCS/ LCSD Pairs	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
Anions - Filters						
Chloride	2	97	2	0	0	83-112
Fluoride	1	94	4	0	0	90-110
Sulfate	2	97	0.5	0	0	85-107
Metals by ICP-AES - NIST 1633a Fly Ash Standard						
Aluminum	4	91	1.1	0	0	75-125
Barium	4	84	1.5	0	0	75-125
Beryllium	4	94	25	1	0	75-125
Calcium	4	98	0.5	0	0	75-125
Chromium	4	97	34	0	0	75-125
Cobalt	4	95	15	0	0	75-125
Copper	4	98	4.5	0	0	75-125
Iron	4	93	1.6	0	0	75-125
Magnesium	4	91	3.3	0	0	75-125
Manganese	4	91	0.3	0	0	75-125
Nickel	4	71	12	2	0	75-125
Potassium	4	90	2.8	0	0	75-125
Silicon	4	100	2.6	0	0	75-125
Sodium	4	125	36	0	0	75-125
Strontium	4	92	1.1	0	0	75-125
Titanium	3	99	1.3	0	0	75-125
Vanadium	4	96	1.3	0	0	75-125
Zinc	3	88	5.5	0	0	75-125
GFAAS and CVAAS Metals - NIST 1633a Fly Ash Standard						
Arsenic	2	120	1.0	0	0	75-125
Cadmium	2	79	44	0	0	75-125

Table F-2 (Continued)

Analyte	No. of LCS	Mean % Rec.	Mean RPD Std. Dev.	No. Below Limits	Above Limits	QC Limits
Lead	2	79	2.6	0	0	75-125
Nickel	2	109	1.4	0	0	75-125
Selenium	26	145	15.8	0	2	75-125
CVAAS Metals - ERA 212 Mercury Standard						
Mercury	2	101	2.0	0	0	75-125

Table F-3
Summary of Spiked Samples Results

Analyte	No. of Spikes	Mean % Recovery	RPD	No. Below Limits	No. Above Limits	QC Limits
Polynuclear Aromatic Hydrocarbons						
Spiked Sample Results (XAD Resin)						
Acenaphthalene	1	102	NA	0	0	50-150
Acenaphthene	1	110	NA	0	0	50-150
Fluorene	1	106	NA	0	0	50-150
Phenanthrene	1	128	NA	0	0	50-150
Anthracene	1	113	NA	0	0	50-150
Fluoranthene	1	104	NA	0	0	50-150
Pyrene	1	98	NA	0	0	50-150
Benzo(a)anthracene	1	99	NA	0	0	50-150
Chrysene	1	101	NA	0	0	50-150
5-methyl chrysene	1	107	NA	0	0	50-150
Benzo(b,j&k)fluoranthenes	1	102	NA	0	0	50-150
Benzo(a)pyrene	1	100	NA	0	0	50-150
Dibenz(a,h)acridine	1	117	NA	0	0	50-150
Benz(a,i)acridine	1	133	NA	0	0	50-150
Indeno(1,2,3-cd)pyrene	1	110	NA	0	0	50-150
Dibenz(a,h)anthracene	1	100	NA	0	0	50-150
7H-dibenzo(c,g)carbazole	1	143	NA	0	0	50-150
Benzo(g,h,i)perylene	1	91	NA	0	0	50-150
Dibenzo(a,e)pyrene	1	98	NA	0	0	50-150
Dibenzo(a,i)pyrene	1	103	NA	0	0	50-150
Dibenzo(a,h)pyrene	1	98	NA	0	0	50-150
PCDD/PCDF Analysis						
2,3,7,8-TCDF	1	126	NA	0	0	70-130
2,3,7,8-TCDD	1	123	NA	0	0	70-130
1,2,3,7,8-PeCDF	1	119	NA	0	0	70-130
2,3,4,7,8-PeCDF	1	136	NA	0	0	70-130
1,2,3,7,8-PeCDD	1	116	NA	0	0	70-130
1,2,3,4,7,8-HxCDF	1	100	NA	0	0	70-130
1,2,3,6,7,8-HxCDF	1	120	NA	0	0	70-130
2,3,4,6,7,8-HxCDF	1	106	NA	0	0	70-130
1,2,3,7,8,9-HxCDF	1	103	NA	0	0	70-130
1,2,3,4,7,8-HxCDD	1	126	NA	0	0	70-130
1,2,3,6,7,8-HxCDD	1	112	NA	0	0	70-130
1,2,3,7,8,9-HxCDD	1	125	NA	0	0	70-130

Table F-3 Continued

Analyte	No. of Spikes	Mean% Recovery	RPD	No. Below Limits	No. Above Limits	QC Limits
1,2,3,4,6,7,8-HpCDF	1	117	NA	0	0	70-130
1,2,3,4,7,8,9-HpCDF	1	128	NA	0	0	70-130
1,2,3,4,6,7,8-HpCDD	1	110	NA	0	0	70-130
OCDF	1	111	NA	0	0	70-130
OCDD	1	106	NA	0	0	70-130
Ashes - GFAAS and CVAAS Metals						
Arsenic	1	12.6	0.80	0	1	75-125
Cadmium	1	114	0.88	0	0	75-125
Lead	1	106	2.8	0	0	75-125
Mercury	2	109	5.0	0	0	75-125
Nickel	1	106	0.94	0	0	75-125
Selenium	1	96	6.2	0	0	75-125
Ashes - ICP-AES Metals						
Aluminum	8	113	38	0	2	75-125
Antimony	8	81	9.1	1	0	75-125
Arsenic	8	85	38	1	3	75-125
Barium	8	65	113	3	0	75-125
Beryllium	8	89	2.0	0	0	75-125
Cadmium	8	94	21	4	2	75-125
Calcium	8	114	39	0	2	75-125
Chromium	8	91	3.3	0	0	75-125
Cobalt	8	92	1.6	0	0	75-125
Copper	8	95	15	0	0	75-125
Iron	8	91	7.6	0	0	75-125
Lead	8	104	2.8	2	0	75-125
Magnesium	8	116	36	0	2	75-125
Manganese	8	90	3.1	0	0	75-125
Molybdenum	8	91	4.8	0	0	75-125
Nickel	8	89	9.1	0	0	75-125
Potassium	8	96	3.2	0	0	75-125
Selenium	8	3.1	NC	6	1	75-125
Silver	8	22	28	8	0	75-125
Sodium	8	115	37	1	2	75-125
Strontium	8	78	29	4	0	75-125
Thallium	8	79	30	4	0	75-125
Titanium	6	86	5.9	0	0	75-125

Table F-3 Continued

Analyte	No. of Spikes	Mean % Recovery	RPD	No. Below Limits	No. Above Limits	QC Limits
Vanadium	8	91	3.3	0	0	75-125
Zinc	8	90	2.5	0	0	75-125
GFAAS and CVAAS Metals Impinger Solutions						
Arsenic	1	101	6.0	0	0	75-125
Cadmium	1	100	3.0	0	0	75-125
Lead	1	87	6.0	0	0	75-125
Mercury	2	95	2.0	0	0	75-125
Selenium	1	0	NC	2	0	75-125
ICP-AES Metals Impinger Solutions						
Aluminum	1	93	1.0	0	0	75-125
Antimony	1	96	0	0	0	75-125
Arsenic	1	99	2.0	0	0	75-125
Barium	1	95	4.0	0	0	75-125
Beryllium	1	101	1.0	0	0	75-125
Cadmium	2	94	1.0	0	0	75-125
Calcium	1	99	1.0	0	0	75-125
Chromium	1	97	1.0	0	0	75-125
Cobalt	1	95	1.0	0	0	75-125
Copper	1	97	1.0	0	0	75-125
Iron	1	98	1.0	0	0	75-125
Lead	1	93	4.0	0	0	75-125
Magnesium	1	93	1.0	0	0	75-125
Manganese	1	95	1.0	0	0	75-125
Molybdenum	1	94	2.0	0	0	75-125
Nickel	1	97	2.0	0	0	75-125
Potassium	1	96	1.0	0	0	75-125
Selenium	1	105	4.0	0	0	75-125
Silicon	1	95	3.0	0	0	75-125
Silver	1	94	1.0	0	0	75-125
Sodium	1	87	1.0	0	0	75-125
Strontium	1	97	1.0	0	0	75-125
Thallium	1	95	0	0	0	75-125
Titanium	1	97	1.0	0	0	75-125
Vanadium	1	98	1.0	0	0	75-125
Zinc	1	93	1.0	0	0	75-125

Table F-3 Continued

Analyte	No. of Spikes	Mean % Recovery	RPD	No. Below Limits	No. Above Limits	QC Limits
GFAAS and CVAAS Metals - Filters						
Arsenic	1	85	2.4	1	0	85-115
Cadmium	1	86	2.3	0	0	85-115
Lead	1	72	0.0	2	0	75-125
Mercury	1	113	1.0	0	0	80-120
Nickel	1	95	2.1	0	0	85-115
Selenium	1	100	1.0	0	0	75-125
Filters - ICP-AES Metals						
Aluminum	2	93	1.0	0	0	85-115
Antimony	2	93	9.4	2	1	85-115
Arsenic	2	93	13	0	0	85-115
Barium	2	94	2.2	0	0	85-115
Beryllium	2	93	1.6	0	0	85-115
Cadmium	2	65	64	3	0	85-115
Calcium	2	98	1.0	0	0	85-115
Chromium	2	94	2.2	0	0	85-115
Cobalt	2	91	1.6	0	0	85-115
Copper	2	96	3.6	0	0	85-115
Iron	2	92	2.2	0	0	85-115
Lead	2	104	11	0	1	85-115
Magnesium	2	94	1.6	0	0	85-115
Manganese	2	94	2.7	0	0	85-115
Molybdenum	2	93	4.6	1	0	85-115
Nickel	2	92	1.7	1	0	85-115
Potassium	2	94	2.1	0	0	85-115
Selenium	2	232	58	1	3	85-115
Silicon	1	98	1.0	2	0	85-115
Silver	2	68	2.2	4	0	85-115
Sodium	2	93	2.7	0	0	85-115
Strontium	2	96	3.7	0	0	85-115
Thallium	2	67	3.7	4	0	85-115
Vanadium	2	94	1.6	0	0	85-115
Zinc	1	85	2.4	1	0	85-115
Ashes - Anions						
Chloride	1	84	8.3	2	0	80-120
Fluoride	2	86	18	2	0	80-120

Table F-3 Continued

Analyte	No. of Spikes	Mean % Recovery	RPD	No. Below Limits	No. Above Limits	QC Limits
Impingers - Anions						
Chloride	1	82	3.0	0	0	80-120
Fluoride	2	103	4.0	0	0	80-120
Sulfate	1	97	12	0	0	80-120
Filters - Anions						
Chloride	1	104	9.0	0	0	80-120
Fluoride	2	103	4.0	0	0	80-120
Sulfate	1	100	2.0	0	0	80-120

Table F-4
Summary of Duplicate Sample Results - Site 22

Analyte	Number of Pairs	Mean	RPD %
Coal - Ultimate/Proximate (%, dry)			
Ash	3	6.87	1.4
Volatile Matter	3	32.64	0.37
Fixed Carbon	3	33.07	0.53
Sulfur	3	0.39	2.8
Carbon	3	69.54	0.37
Hydrogen	3	4.89	2.2
Nitrogen	3	1.02	2.0
Heating Value (Btu)	3	11982	0.32
Coal- NAA Metals			
Aluminum	3	4306	4.0
Antimony	3	0.11	4.6
Arsenic	3	0.67	0.86
Barium	3	303	2.4
Bromine	3	0.61	2.8
Cadmium	3	<2.5	NC
Calcium	3	6114	2.9
Cerium	3	5.436	4.8
Cesium	3	0.1011	9.6
Chlorine	2	43.840	7.2
Chromium	3	3.4	1.7
Cobalt	3	1.11	0.84
Copper	2	12.3	18
Europium	3	0.1080	3.7
Hafnium	3	0.4791	3.4
Iodine	3	1.926	12.5
Iron	3	1874	1.2
Lanthanum	3	2.492	1.2
Lutetium	3	0.035	4.7
Magnesium	3	453.06	7.3
Manganese	3	4.1	5.4
Mercury	3	0.110	31.8

Table F-4 (Continued)

Analyte	Number of Pairs	Mean	RPD %
Molybdenum	3	1.708	5.4
Neodymium	3	3.172	13.2
Nickel	3	<3.1	14
Potassium	3	1019	7
Rubidium	3	1.844	4.82
Samarium	3	0.4919	1.41
Scandium	3	1.206	0.77
Selenium	3	0.62	15
Silver	3	<0.26	NC
Sodium	3	460.46	0.97
Strontium	3	118.30	0.21
Tantalum	3	0.1099	7.9
Terbium	3	0.0747	1.7
Thorium	3	1.21	0.99
Tin	3	<10.0	NC
Titanium	3	397.2	3.8
Tungsten	3	0.172	1.9
Uranium	3	0.427	1.6
Vanadium	3	9.944	4.3
Zinc	3	6.920	9.40
Zirconium	3	20.32	6.2
Ash - GFAAS and CVAAS Metals (μg/g), WAL			
Arsenic	3	21.6	0.5
Cadmium	3	NC	NC
Lead	3	NC	NC
Mercury	3	NC	NC

Table F-4 (Continued)

Analyte	Number of Pairs	Mean	RPD %
Coal - ICP-AES, GFAAS, CVAAS, and XRF Metals (μg/g), WAL			
Arsenic	3	$\leq$0.1	NC
Beryllium	3	0.4	17
Cadmium	3	$\leq$2	NC
Chromium	3	$\leq$5	NC
Copper	3	15	8.5
Lead	3	19	17
Manganese	3	26	1.3
Mercury	3	0.11	12
Nickel	3	$\leq$5	NC
Phosphorous	3	287	2.3
Selenium	3	$\leq$1	NC
Titanium	3	795	3.8

NC = Not able to calculate.

Table F-5
Summary of Audit Sample Results - Site 22

Analyte	Certified Value[a] µg/g	Analyzed Result µg/g	Rec. %
NIST 1632b Coal - INAA Metals			
Aluminum	29500.0	29731.0	101
Antimony	0.600	0.6148	102
Arsenic	9.3	9.5	102
Barium	120.0	128.5	107
Bromine	41.0	42.7	104
Cadmium	0.17	<2.0	NC
Calcium	2410.0	2376.4	98.6
Cerium	29.0	29.8	103
Cesium	2.3	2.2	95.7
Chlorine	756.0	777.1	103
Chromium	34.3	34.5	101
Cobalt	6.7	6.9	103
Copper	16.5	<25	NC
Europium	0.520	0.520	100
Hafnium	1.62	1.71	106
Iodine	1.80	2.05	114
Iron	11,100	11,245	101
Lanthanum	15.0	15.4	103
Lutetium	0.170	0.162	95.3
Magnesium	1150	1115	97.0
Manganese	28.0	27.9	99.6
Mercury	0.130	<0.25	NC
Molybdenum	3.85	4.17	108
Neodynmium	12.0	10.4	86.7
Nickel	19.4	19.1	98.5
Potassium	4110	3868	94.1
Rubidium	30.0	30.4	101
Samarium	2.4	2.5	104
Scandium	6.3	6.4	102
Selenium	2.6	2.7	104
Silver	0.3	<0.4	NC

Table F-5 (Continued)

Analyte	Certified Value[a] µg/g	Analyzed Result µg/g	Rec. %
Sodium	828.0	802.5	96.9
Strontium	85.0	83.8	98.6
Tantalum	0.420	0.402	95.7
Terbium	0.311	0.307	98.7
Thorium	4.5	4.6	102
Tin	4.0	<10.0	NC
Titanium	1630	1580	96.9
Tungsten	0.880	0.615	69.9
Uranium	1.28	1.31	102
Vanadium	44.0	42.6	96.8
Ytterbium	1.08	1.07	99.1
Zinc	28.0	27.6	98.6
Zirconium	53.0	50.3	94.9
SARM 20 Coal-INAA			
Aluminum	59644	58203	97.6
Antimony	0.4	0.4224	106
Arsenic	4.7	4.378	93.1
Barium	372	417.02	112
Bromine	2	4.0870	204
Calcium	13365	18,299	137
Cerium	87	87.802	101
Cesium	2	2.6274	131
Chromium	67	75.566	113
Cobalt	8.3	8.6386	104
Copper	18	<25	NC
Europium	1	1.2916	129
Hafnium	4.8	5.240	109
Iron	8183	8307.1	102
Lanthanum	43	47.387	110
Magnesium	2593	2580.7	99.5
Manganese	80	77.42	96.8
Mercury	0.25	0.287	115
Nickel	25	23.950	95.8

Table F-5 (Continued)

Analyte	Certified Value* µg/g	Analyzed Result µg/g	Rec. %
Rubidium	10	11.262	113
Samarium	6.3	6.199	98.4
Selenium	0.8	0.957	120
Sodium	2003	2143.8	107
Strontium	330	304.71	92.3
Tantalum	1.2	1.3401	112
Terbium	0.9	1.1736	130
Thorium	18	18.365	102
Tungsten	3	1.777	59.2
Uranium	4	4.3669	109
Vanadium	47	46.396	98.7
Ytterbium	20	3.478	174
Zinc	17	<25	NC
Zirconium	180	188.97	105
SARM 20 Coal-GFAAS, CVAA, XRF, ICP (WAL Analyses)			
Arsenic (GFAAS)	4.7	2.5	53
Beryllium (ICP)	2.5	2.3	92
Cadmium (ICP)	-	3	NC
Chromium (ICP)	67	65	97
Copper (XRF)	18	24	133
Lead (XRF)	26	29	111
Manganese (XRF)	80	69	86
Mercury (DGA/CVAAS)	0.25	0.23	92
Nickel (XRF)	25	26	104
Phosphorous (XRF)	611	420	69
Selenium (XRF)	0.8	≤1	NC
Titanium (XRF)	3777	3410	90
Sarm 20 Coal-ICP-AES, GFAAS, CVAAS (CT&E Analyses)			
Arsenic (GFAAS)	4.7	2.0	43
Beryllium (ICP)	2.5	2.6	104
Cadmium (GFAAS)	–	<0.1	NC

Table F-5 (Continued)

Analyte	Certified Value[a] µg/g	Analyzed Result µg/g	Rec. %
Chromium (ICP)	67	66	99
Copper (ICP)	18	16	89
Lead (GFAAS)	26	22	85
Manganese (ICP)	80	81	101
Mercury (DGA/CVAAS)	0.25	0.29	116
Nickel (ICP)	25	24	96
Phosphorus (ICP)	611	670	110
Selenium (GFAAS)	0.8	<1	NC
Titanium (ICP)	3,777	3,600	95
NIST 1633a Coal Fly Ash- ICP-AES Metals			
Aluminum (%)	14.3	13.4	93.7
Barium (%)	(0.15)	0.13	86.7
Beryllium	(12)	11.2	93.3
Calcium (%)	1.11	1.11	100
Chromium	196	194	98.9
Cobalt	(46)	47	102
Copper	118	118	100
Iron (%)	9.4	8.86	94.2
Magnesium (%)	0.455	0.429	94.2
Manganese	179	163	91.1
Nickel	127	96.0	75.6
Potassium (%)	1.88	1.68	89.4
Silicon (%)	22.8	22.5	98.7
Sodium (%)	0.17	0.172	101
Strontium	830	781	94.1
Titanium (%)	(0.8)	0.79	98.8
Vanadium	297	285	96.0
Zinc	220	204	92.7

Table F-5 (Continued)

Analyte	Certified Value* µg/g	Analyzed Result µg/g	Rec. %
NIST 1633a Coal Fly Ash - GFAAS, and CVAAS Metals			
Arsenic	145	196	135
Cadmium	1.00	1.08	108
Lead	72.4	58.1	80
Nickel	127	143	113
Selenium	10.3	15.0	146
ERA 216 - Mercury			
Mercury	2.36	2.24	95

*Results in parentheses are not certified.

NC = Not able to calculate.

Table F-6
Summary of Surrogate Recoveries for Semivolatile and PCDD/PCDF Analyses

Surrogate Compound	No. of Analyses	Mean % Recovery	% CV[a]	No. Below Limits	No. Above Limits	QC Limits
PAH Analyses by HRGCMS (XAD)						
Biphenyl-d10	5	85	14	0	0	50-150
Hexachlorobenzene	5	67	24	1	0	50-150
Perylene-d12	5	99	37	1	0	50-150
PCDD/PCDF Analyses by HRGCMS (XAD)						
2,3,7,8-TCDD-$^{37}Cl_4$	5	91	4.7	0	70-130	NS
2,3,4,8-PeCDF-^{13}C	5	82	6.2	0	70-130	NS
1,2,3,4,7,8-HxCDF-^{13}C	5	103	21	0	70-130	NS
1,2,3,4,7,8-HxCDD-^{13}C	5	95	0.84	0	70-130	NS
1,2,3,4,7,8,9-HpCDF-^{13}C	5	75	4.4	0	70-130	NS

[a] % CV = Percent coefficient of variation = (standard deviation/mean) x 100.

Appendix G: Process Data

Run 1

July 13, 1993

FCEM Site 22

Process Data - 7/13/93

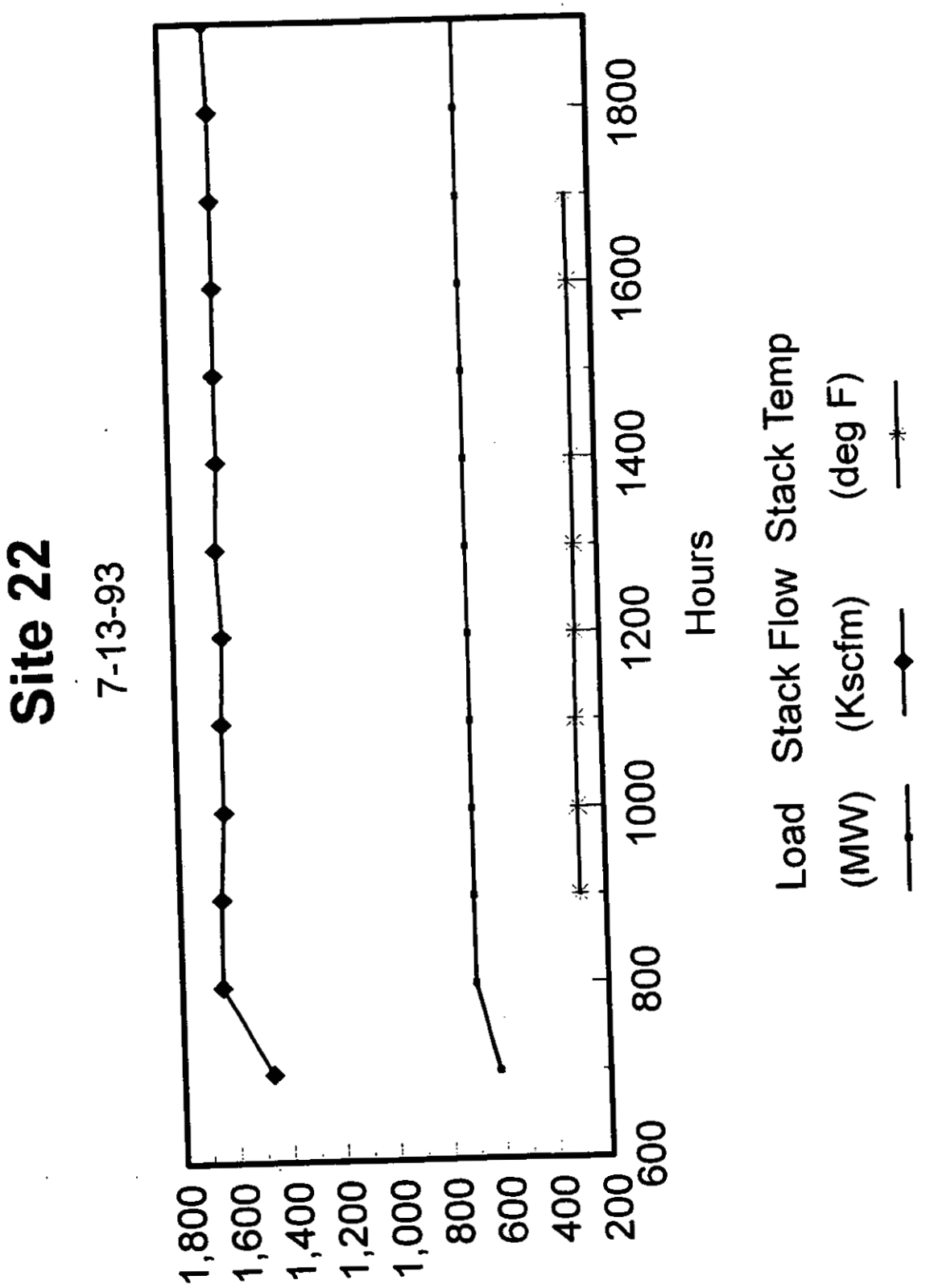
HRS	LOAD (MW)	CO2 (%)	OPAC (%)	FLOW (Kscfm)	SO2 (ppmv)	NOx (ppmv)	Economizer Outlet O2 (N) (%)	O2 (S) (%)	O2 - Av (%)
0700	618	9.89	5	1468	294	178	-	-	-
0800	702	14.7	5.5	1651	286	172	2.2	2.7	2.45
0900	702	10.8	5.9	1646	282	174	2.5	2.7	2.6
1000	701	10.9	6	1629	284	174	2.4	2.7	2.55
1100	700	11	6	1630	271	168	2.4	2.9	2.65
1200	699	11.2	6	1620	263	155	2.2	2.7	2.45
1300	700	11.1	6	1635	267	158	2.1	2.8	2.45
1400	698	11	6	1623	305	148	2	2.8	2.4
1500	697	11	6.1	1626	302	148	2	2.7	2.35
1600	701	11	6	1622	289	149	2	2.8	2.4
1700	701	11.2	6.1	1623	277	151	2.2	2.7	2.45
1800	701	11	6.1	1623	278	155	2.2	2.7	2.45
1900	701	11.2	6.2	1636	268	169	2.4	2.6	2.5
AVG	700.3	11.3	6.0	1630.3	281.0	160.1	2.2	2.7	2.5
STD DEV	1.54	1.06	0.17	9.93	13.25	10.55	0.17	0.08	0.09
CV	0.002	0.094	0.029	0.006	0.047	0.066	0.079	0.028	0.035

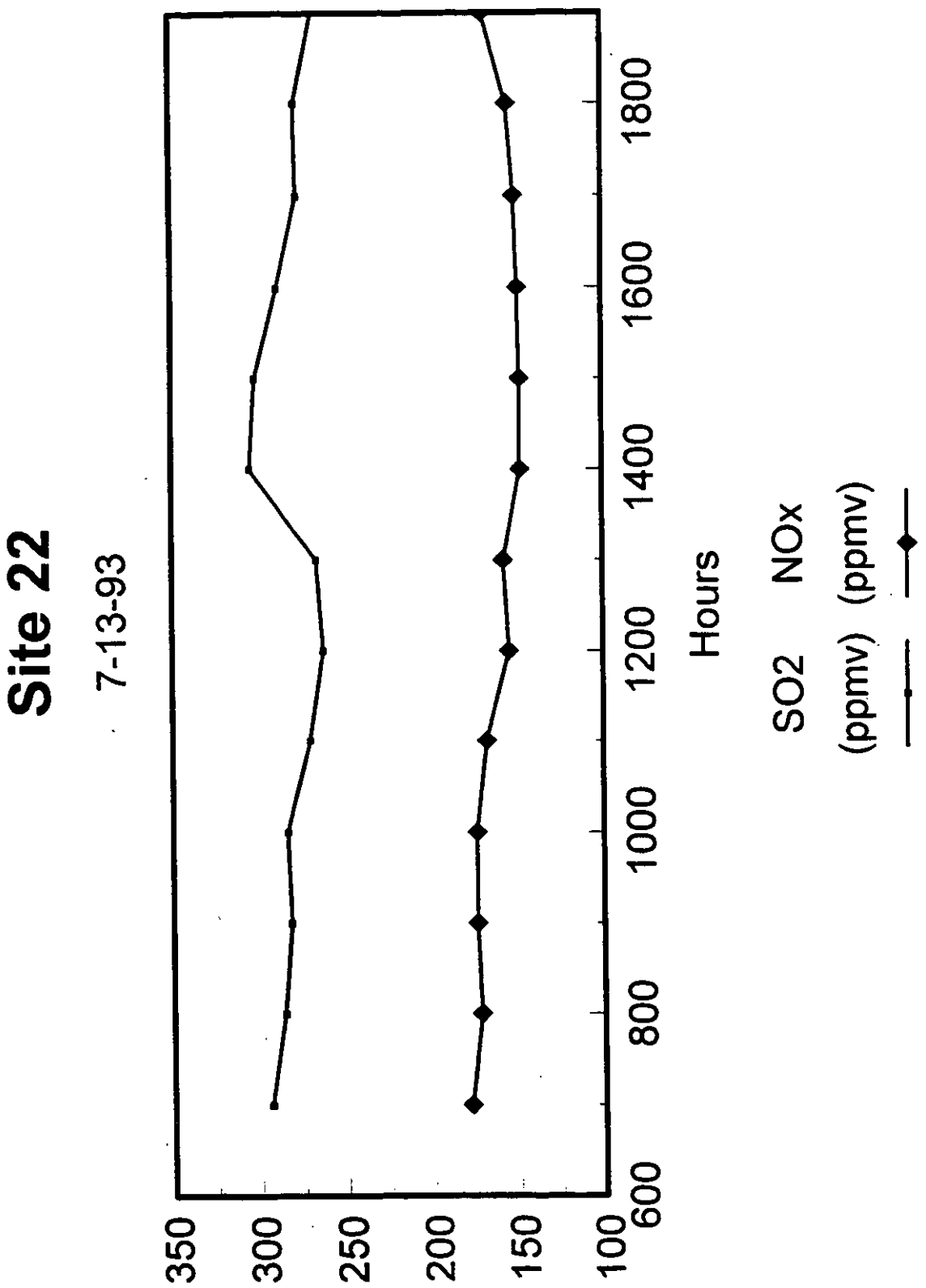
Note: Averages do not include data from 0700

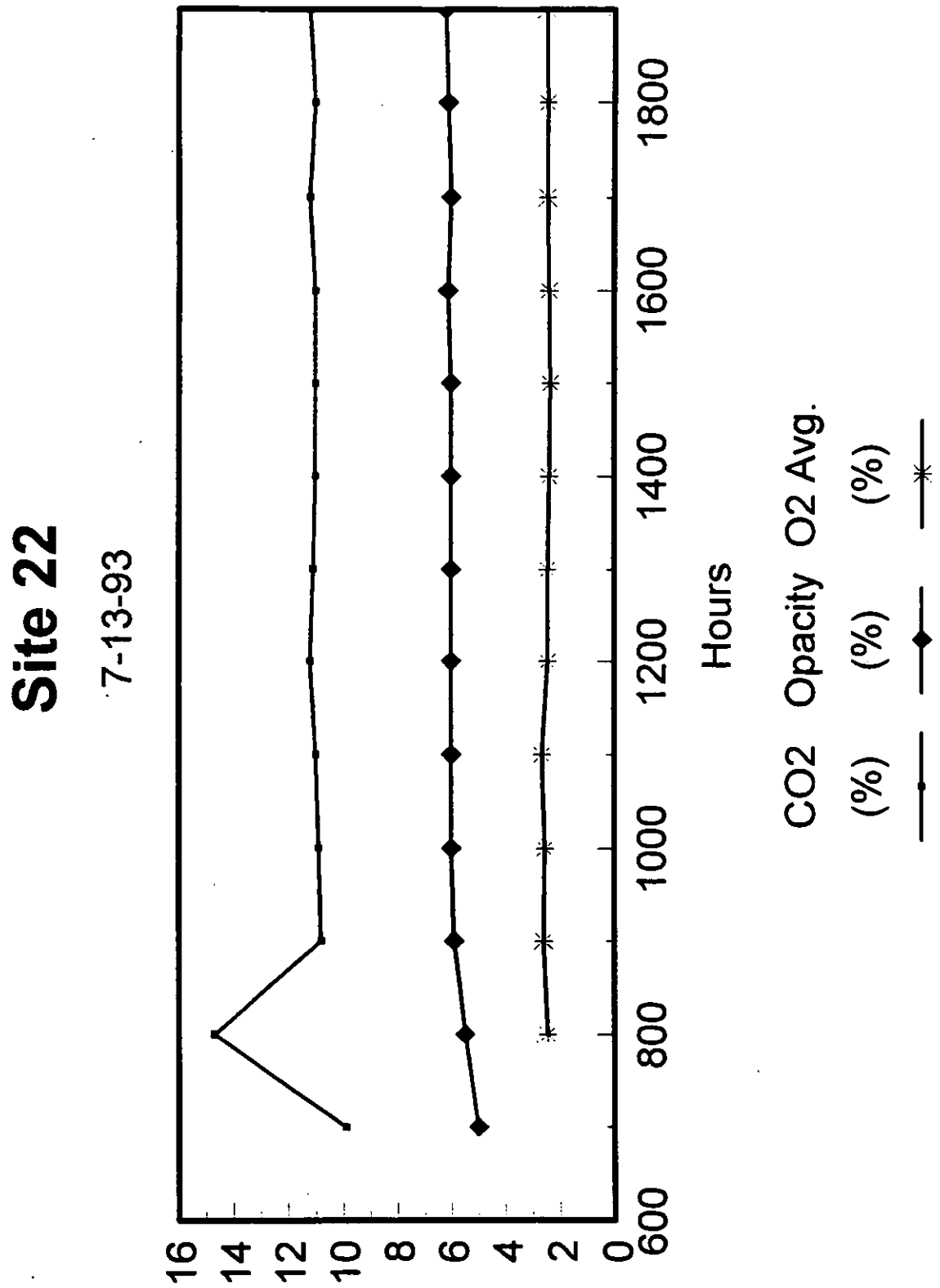
FCEM Site 22

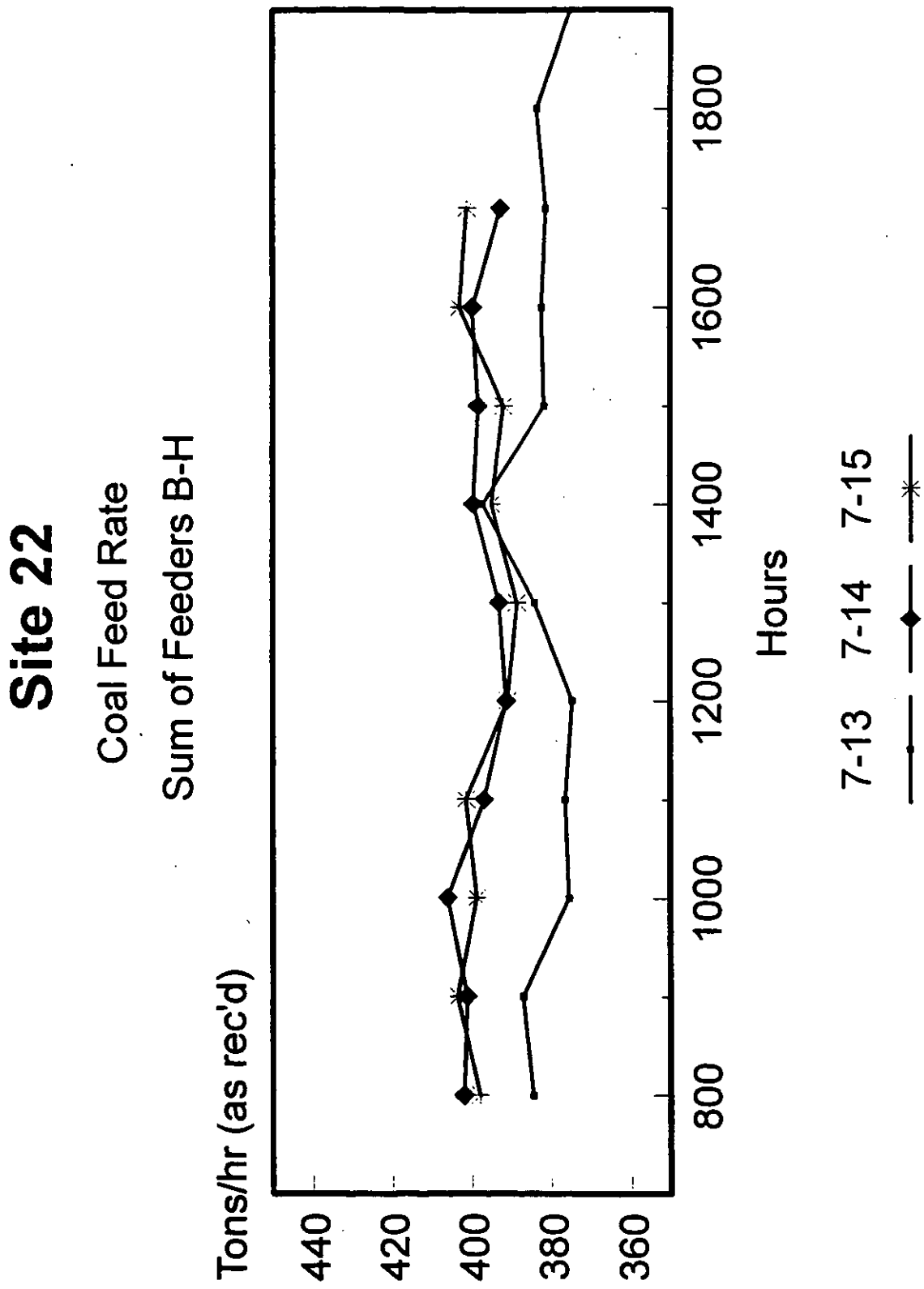
Process Data - 7/13/93 **COAL FEED RATE (tons/hr, as rec'd)**

HRS	Feeder B	Feeder C	Feeder D	Feeder E	Feeder F	Feeder G	Feeder H	Sum (B-H)
0800	58.3	56.7	57	50.8	56.2	48	57.5	384.5
0900	57.2	56.5	57.7	50.3	57.4	49.5	58.5	387.1
1000	55.2	55.3	55.8	48.4	55.2	49.3	56.2	375.4
1100	54.7	55.5	56.4	49.2	55.2	49.4	56	376.4
1200	55.2	55.2	55.7	48.8	55.8	47.4	56.5	374.6
1300	56.1	57.1	57.6	50.1	57.1	48.5	57.6	384.1
1400	56.9	57.8	59.2	52.3	59.9	51.9	59.3	397.3
1500	57.3	57.2	57.9	49.1	56.2	47.8	56.2	381.7
1600	56.1	56.5	57.2	49.7	55.8	48.7	58.3	382.3
1700	55	55.6	56.7	49.3	57	50.5	57.1	381.2
1800	57.2	56.7	58.2	50.3	57	47.4	56.7	383.5
1900	55.7	55.1	56.4	48.8	54.6	48.2	56.3	375.1
AVG	56.2	56.3	57.2	49.8	56.5	48.9	57.2	381.9
STD DEV	1.13	0.90	1.03	1.08	1.39	1.33	1.07	6.40
CV	0.020	0.016	0.018	0.022	0.025	0.027	0.019	0.017









Run 2

July 14, 1993

FCEM Site 22

Process Data - 7/14/93

HRS	LOAD (MW)	CO2 (%)	OPAC (%)	FLOW (Kscfm)	SO2 (ppmv)	NOx (ppmv)	Economizer Outlet O2 (N) (%)	O2 (S) (%)	O2 - Av (%)
0700	698	11.2	6	1667	325	141	2.3	2.6	2.45
0800	705	10.7	6.1	1642	329	164	2.6	2.9	2.75
0900	703	10.8	6	1650	326	167	2.6	2.8	2.7
1000	702	10.9	6	1651	335	168	2.6	2.6	2.6
1100	703	10.9	6	1666	340	156	2.5	2.8	2.65
1200	705	11	6	1646	330	163	2.6	2.7	2.65
1300	703	10.8	6	1652	334	168	2.7	2.8	2.75
1400	704	11	6.7	1640	324	169	2.7	2.7	2.7
1500	703	11	6.7	1652	308	179	2.6	2.8	2.7
1600	702	10.8	6.6	1643	320	169	2.4	2.8	2.6
1700	701	11	6.5	1636	331	154	2.2	2.7	2.45
AVG	703.1	10.9	6.3	1647.8	327.7	165.7	2.6	2.8	2.7
STD DEV	1.29	0.11	0.32	8.44	8.98	7.09	0.15	0.08	0.09
CV	0.002	0.010	0.051	0.005	0.027	0.043	0.059	0.031	0.034

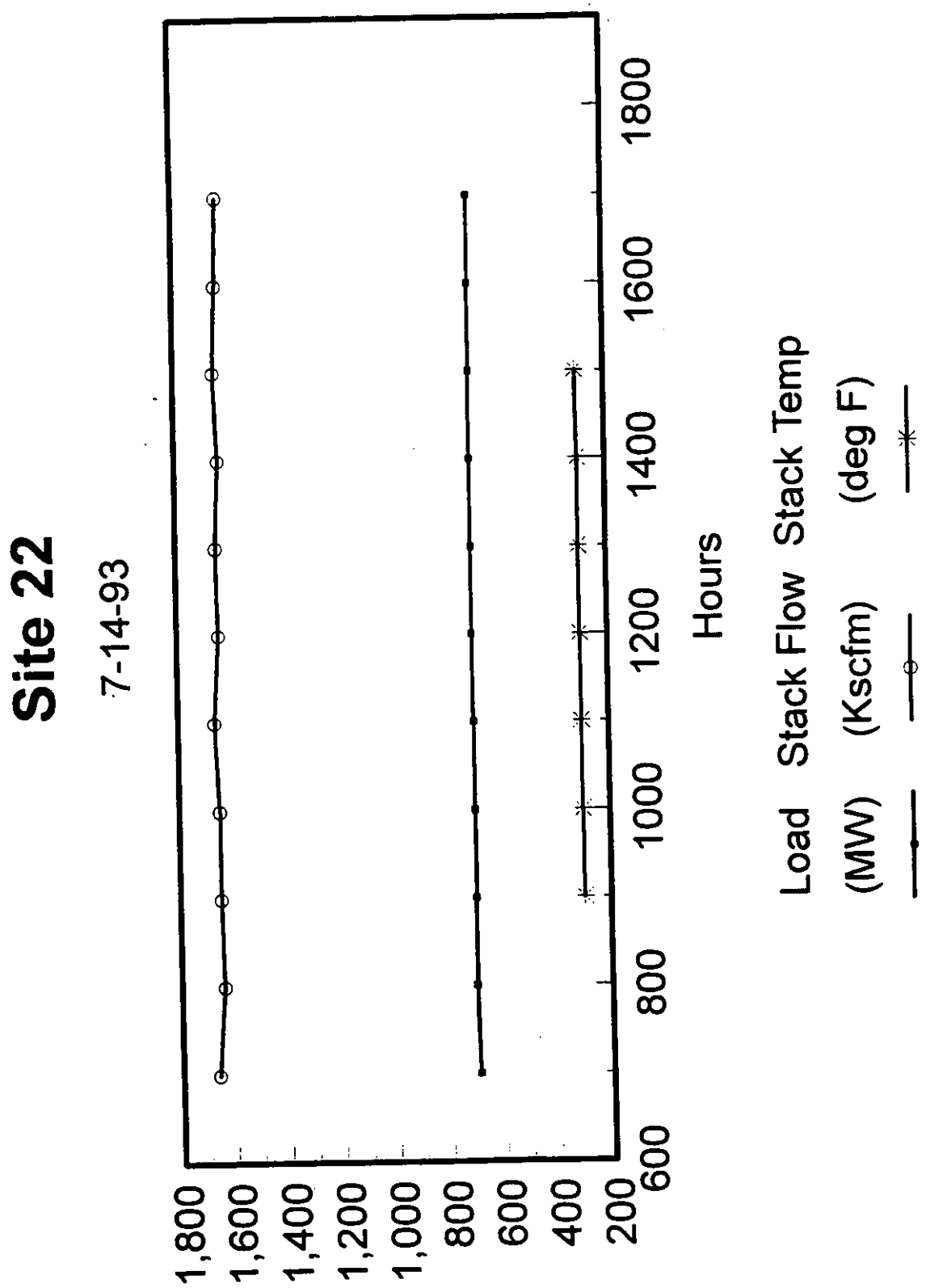
Note: Averages do not include data from 0700

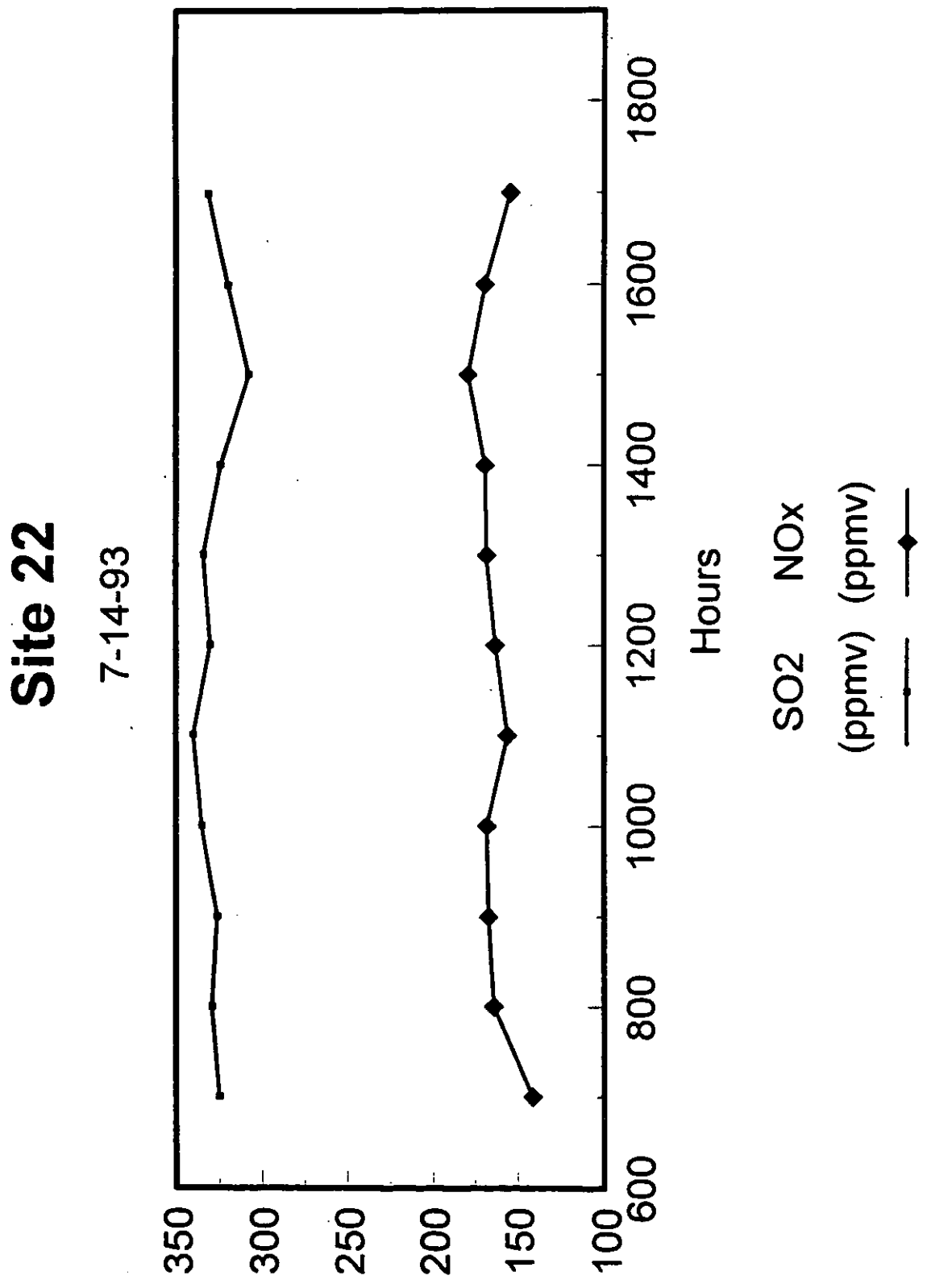
FCEM Site 22

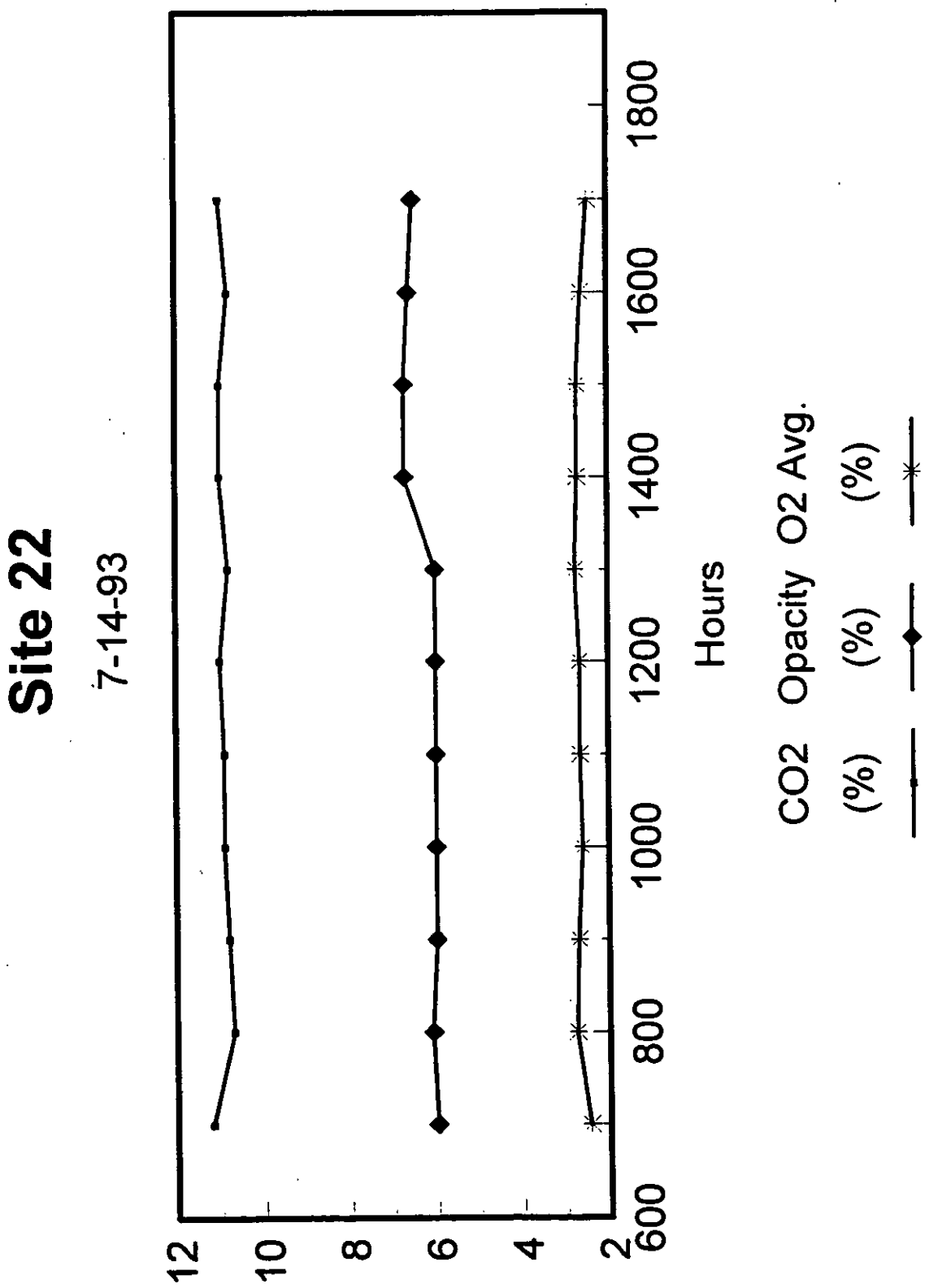
Process Data - 7/14/93 **COAL FEED RATE (tons/hr, as rec'd)**

HRS	Feeder B	Feeder C	Feeder D	Feeder E	Feeder F	Feeder G	Feeder H	Sum (B-H)
0700	56.6	57.2	58.5	56.8	58.1	53.8	58.5	399.5
0800	57.5	58.3	57.5	57.8	57.4	54.6	58.9	402.0
0900	58.2	57.3	58.4	57	57.4	53.5	59.4	401.2
1000	58.5	58.4	58.8	58.5	58.3	54.7	58.8	406.0
1100	57.5	57.7	58.7	57.6	56.3	51.7	57.4	396.9
1200	56	55.4	56.7	56.2	55.5	54.5	57	391.3
1300	57.6	57.6	58	55.7	56.6	51.3	56.3	393.1
1400	56.7	56.8	57.7	57.4	57.6	56.3	57.1	399.6
1500	57.6	57.7	58.5	57.7	56.3	54	56.6	398.4
1600	57.4	57.9	57.6	55.4	56.8	56.6	58.1	399.8
1700	55.2	55.3	57	56.2	57.1	55.4	56.6	392.8
AVG	57.2	57.2	57.9	57.0	56.9	54.3	57.6	398.1
STD DEV	1.00	1.10	0.72	1.02	0.80	1.74	1.10	4.63
CV	0.017	0.019	0.012	0.018	0.014	0.032	0.019	0.012

Note: Averages do not include data from 0700







Run 3

July 15, 1993

FCEM Site 22

Process Data - 7/15/93

HRS	LOAD (MW)	CO2 (%)	OPAC (%)	FLOW (Kscfm)	SO2 (ppmv)	NOx (ppmv)	Economizer Outlet O2 (N) (%)	O2 (S) (%)	O2 - Av (%)
0700	627	16.3	5	1470	325	107	1.2	2.4	1.8
0800	699	14.9	5	1591	315	131	2.1	2.8	2.45
0900	705	10.8	6	1650	322	162	2.4	2.6	2.5
1000	703	10.9	6	1667	321	166	2.6	2.9	2.75
1100	702	10.9	6	1658	320	167	2.7	2.8	2.75
1200	702	10.8	6	1642	333	162	2.5	2.9	2.7
1300	701	10.9	6	1627	298	164	2.4	2.8	2.6
1400	701	10.9	6.3	1642	311	160	2.4	2.7	2.55
1500	701	10.7	6.4	1636	322	165	2.4	2.8	2.6
1600	700	10.8	6.3	1635	314	160	2.4	2.9	2.65
1700	701	10.8	6.2	1647	333	154	2.3	2.8	2.55
AVG	701.5	11.2	6.0	1639.5	318.9	159.1	2.4	2.8	2.6
STD DEV	1.65	1.29	0.39	20.60	10.33	10.56	0.16	0.09	0.10
CV	0.002	0.115	0.065	0.013	0.032	0.066	0.067	0.034	0.039

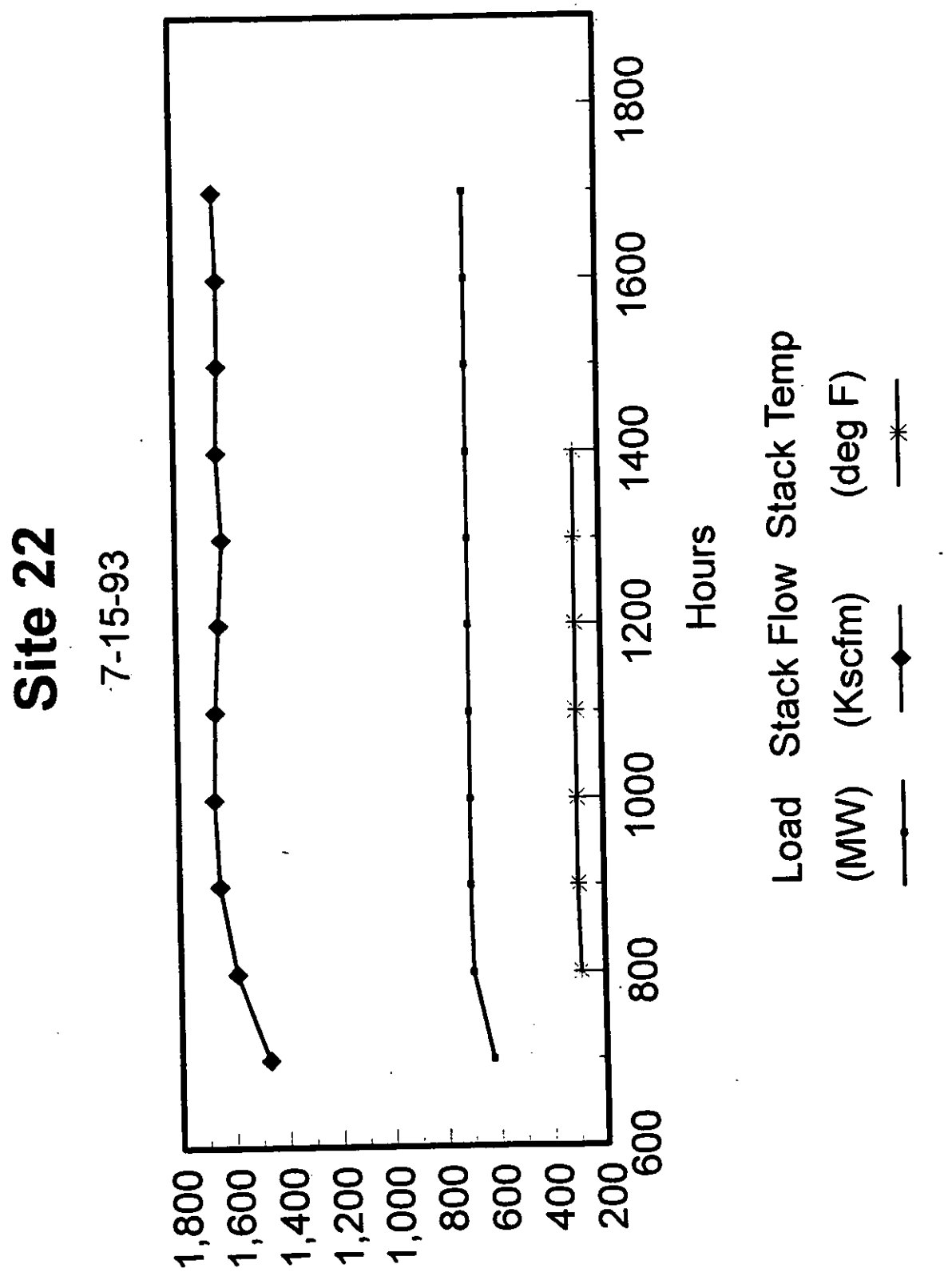
Note: Averages do not include data from 0700

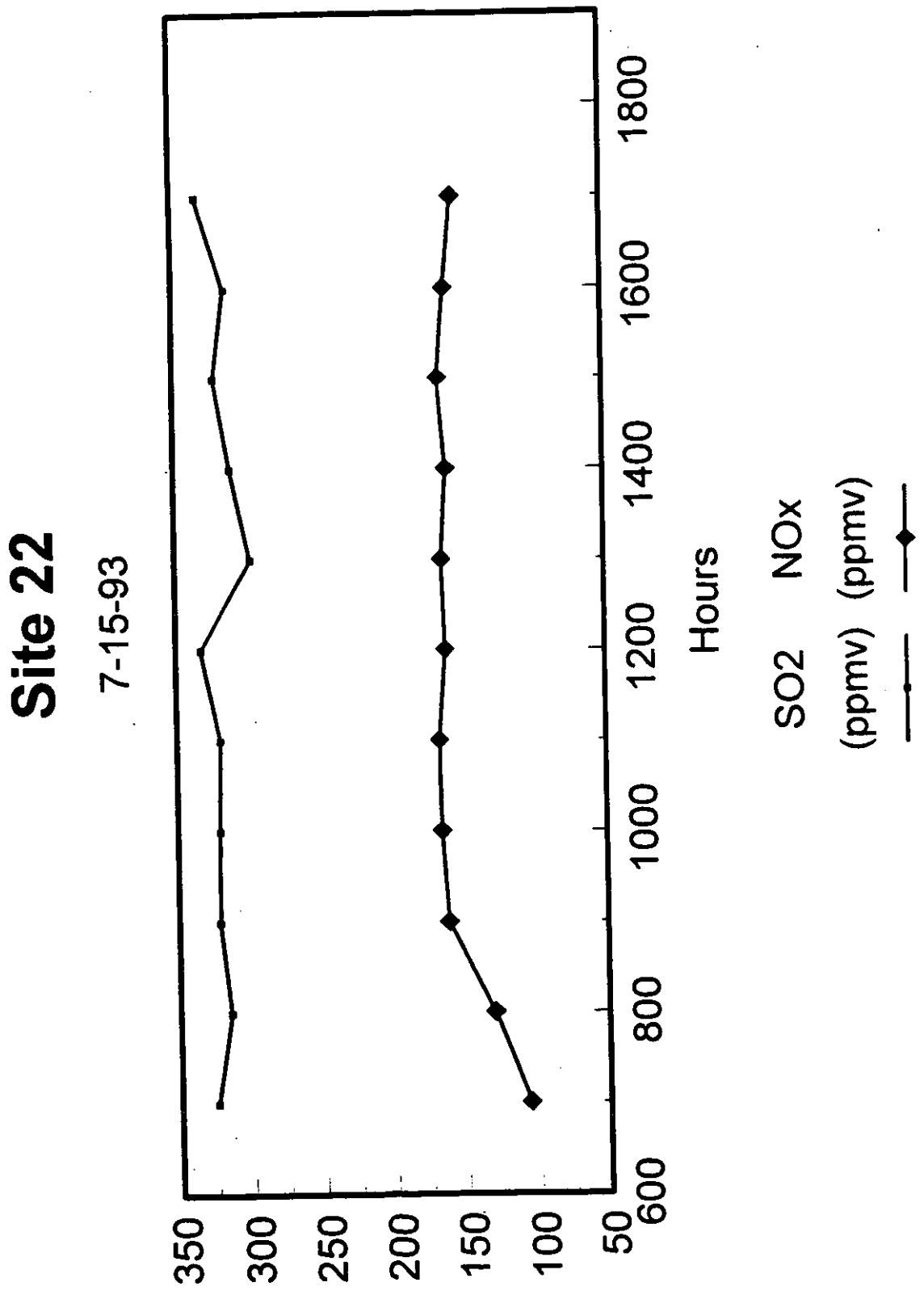
FCEM Site 22

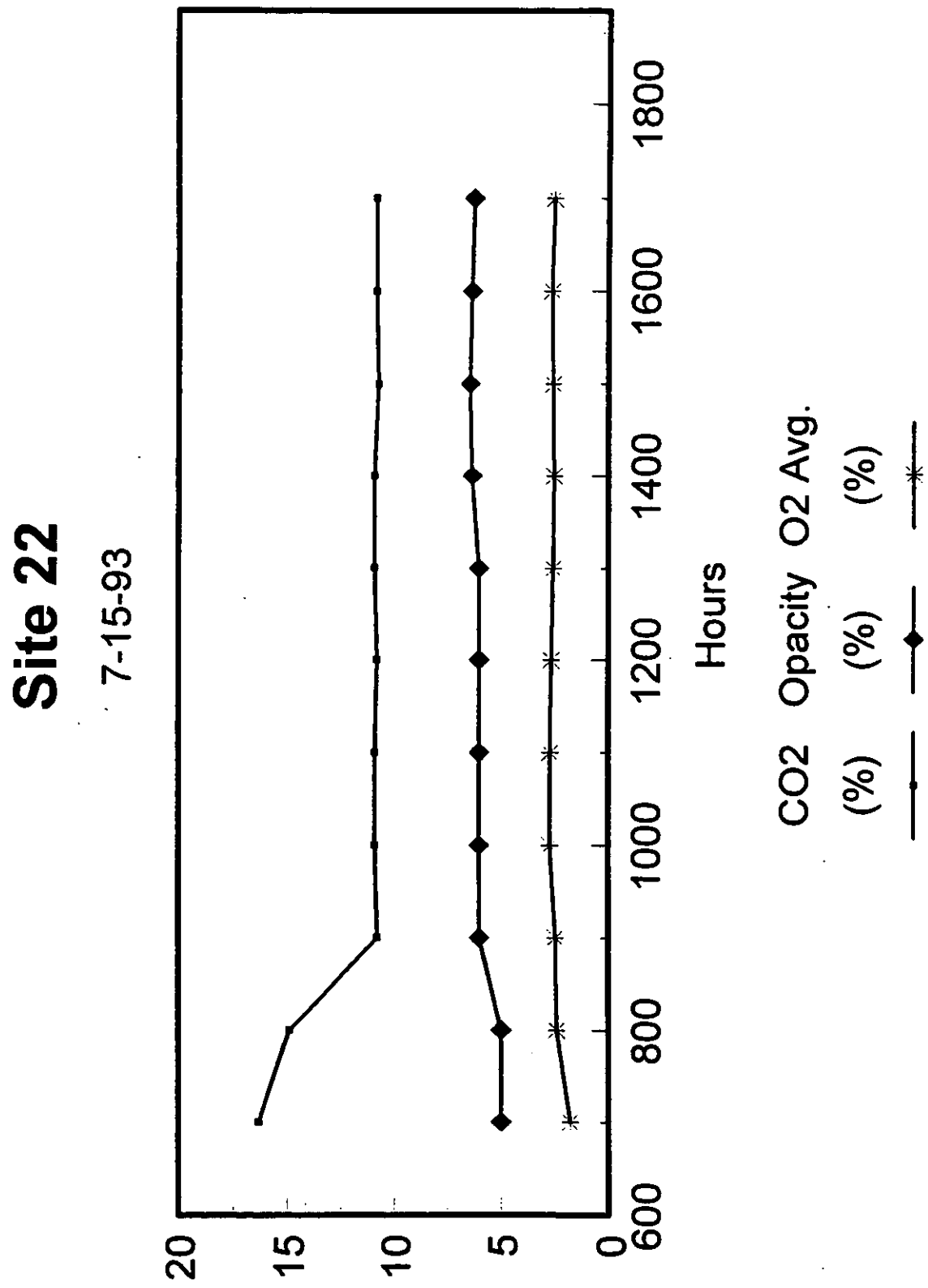
Process Data - 7/15/93 **COAL FEED RATE (tons/hr, as rec'd)**

HRS	Feeder B	Feeder C	Feeder D	Feeder E	Feeder F	Feeder G	Feeder H	Sum (B-H)
0700	56.9	57.2	59	51.5	57.1	49.4	58.7	389.8
0800	57.3	57.8	59.7	51.6	59.2	52.2	60.2	398.0
0900	58.9	60.1	61.3	53	59.3	50.7	60.2	403.5
1000	58.4	58.4	59.1	51.1	58.7	52.1	61	398.8
1100	58.3	58.9	59.9	52.7	59.7	51.8	60.2	401.5
1200	57.7	57.1	58.1	50.9	57.8	50.3	58.1	390.0
1300	57.5	57.3	57.8	50.3	57.8	50.2	57.7	388.6
1400	57.3	58.2	59.9	51.8	58.2	51.6	58	395.0
1500	57	56.6	58.6	51.5	59.5	50.4	58.4	392.0
1600	59.4	60.3	61	52.5	59.5	51	59.3	403.0
1700	59.7	59.3	60.1	51.5	59.1	52.3	59.2	401.2
AVG	58.2	58.4	59.6	51.7	58.9	51.3	59.2	397.2
STD DEV	0.94	1.25	1.15	0.84	0.71	0.83	1.14	5.46
CV	0.016	0.021	0.019	0.016	0.012	0.016	0.019	0.014

Note: Averages do not include data from 0700







Appendix H: Frontier Geosciences' Mercury Data

FRONTIER GEOSCIENCES
ENVIRONMENTAL RESEARCH CORPORATION
414 Pontius North • Seattle, WA 98109
(206) 622-6960 • fax: (206) 622-6870

August 4, 1993
Eric Prestbo Ph.D.

Report on the Analysis of Mercury Species in Combustion Flue Gas at FCEM Site 22 Using a Solid Sorbent Method and CVAFS

Analytical

The samples arrived intact at Frontier Geosciences on July 17, 1993. They were stored in our laboratory in a closed container until analysis. The solid sorbent traps were analyzed between August 2 and 3, 1993. The analytical procedures used for analysis are included as an attachment. It should be noted that we are now using 10 mL of a 7:3 mixture of HNO_3:H_2SO_4 and 0.05 N BrCl for the digestion and dilution of the iodated carbon traps. The higher concentration of acid and BrCl for the iodated carbon traps has been shown to extend the storage time of the digest.

The laboratory analysis was normal; no analytical difficulties were encountered. The results are documented below in Tables 1-4. The low values found for Hg(II) and methyl Hg are very near the blank values. Normally we expect to observe between 40 and 90% of the total Hg as oxidized Hg. The high percentage Hg^0 is unusual when compared to other sites. As discussed with Larry Rohlach, there was nothing done in the field or unusual flue gas conditions which would explain the results.

The results documented below (Tables 1-4) are reported as blank corrected nanograms/trap. Several duplicates and triplicates were run, providing a measure of the laboratory analytical precision (Tables 2-4). The field blank values are well within the range we have observed in past analyses. Based on 3 sigma of the field blanks detection limits were calculated using a nominal volume of 70 liters (Table 1). Because of the low values found on the KCl/soda lime traps breakthrough was impossible to estimate (Table 1). Example calculations of Hg in ng/trap are provided in Tables 2-4.

The raw data will be kept in our files for a period of 3 years and is available upon request. Please don't hesitate to call if you have questions or would like to discuss the results.

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November 3, 1993
Eric M. Prestbo

Douglas A. Orr
Radian Corporation
P.O. Box 201088
Austin, TX 78720-1088

Dear Doug,

Enclosed are the revised data tables for FCEM Site 22, sampled in July 1993. At your request the data has now been calculated on a $\mu g/Nm^3$ at 0° C based on the the field data sheets.

Call if you have any questions

Sincerely,

Eric M. Prestbo

Site 22 FCEM Results of Hg Speciation In Combustion Flue Gas

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Table 1: Sum of Species

Radian Sample ID	Frontier Sample ID	Stream/Run	Elemental Hg ug/Nm^3	Hg(II) Hg ug/Nm^3	Methyl Hg ug/Nm^3	Total Hg ug/Nm^3
FC22-SGOOHGht-FB	445	Stack/0	^	^	^	^
FC22-SGO1HGht	448	Stack/1	7.32	<0.009		7.32
FC22-SGO2HGht	440	Stack/2	9.24	0.039		9.28
FC22-SGO3HGht	447	Stack/3	7.49	<0.009		7.49

Detection limit (3X Sigma Field Blank/0.090 cubic meters)		Mean Blank ng/trap	Std Dev ng/trap
Elemental ug/m^3	0.048	2.92	1.44
Hg (II) ug/m^3	0.009	2.60	0.28
Methyl ug/m^3	0.004	0.78	0.11

Conservative Estimate of Hg Species Recovery Based on Breakthrough	
Elemental	100%
Hg (II)	^
Methyl Hg	^

Site 22 FCEM Results of Hg Speciation in Combustion Flue G

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Table 2: Elemental Hg

Radian Sample ID	Frontier Sample ID	Stream/ Run	A Trap ng/trap	Std. Dev. A Trap ng/trap (n)	B Trap ng/trap	Std. Dev. B Trap ng/trap (n)	Collected Volume (Liters)	Elemtental [Hg] ug/Nm^3
FC22-SGOOHGht-FB	445	Stack/0	4.4		1.2	0.84 (2)	0	
FC22-SGO1HGht	448	Stack/1	691		2.9		94.01	7.32
FC22-SGO2HGht	440	Stack/2	903		4.3		97.42	9.24
FC22-SGO3HGht	447	Stack/3	697	36.8 (2)	1.8		92.65	7.49

Detection limit (3 X sigma field blank/0.090 cubic meters)			Mean Blank*	Std Dev
Elemental ug/m^3	0.048		ng/trap	ng/trap
			2.92	1.44

Laboratory Spike Recovery - 1000 picograms (pg)		
pg expect	pg recovered	%Recovery
1002	1011	100.9

* Grand average of field blank and B Trap values (n=5) — ug/Nm^3 @ 0 degrees C and 1 Atm

Example Calculation for Field Blank Correct Elemental Hg ug/m^3

(A Trap-Mean Blank)/Collected Volume

Site 22 FCEM Results of Hg Speciation In Combustion Flue Gas

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Table 3: Ionic Mercury

Radian Sample ID	Frontier Sample ID	Stream/Run	A Trap ng/trap	Std. Dev. A Trap ng/trap (n)	B Trap ng/trap	Std. Dev. B Trap ng/trap (n)	Collected Volume (Liters)	Ionic [Hg] ug/Nm^3
FC22-SGOOHGht-FB	445	Stack/0	2.8	0.71 (2)	2.4		0	
FC22-SGO1HGht	448	Stack/1	1.08	0.096 (4)	2.6		94.01	<0.009
FC22-SGO2HGht	440	Stack/2	2.7		6.3		97.42	0.039
FC22-SGO3HGht	447	Stack/3	1.2	0.28 (2)	1.6		92.65	<0.009

Detection limit (3 X sigma field blank/0.090 cubic meters)			Mean Feild Blk	Std Dev
Ionic Hg ug/m^3	0.009		ng/trap	ng/trap
			2.6	0.28

Laboratory Spike Recovery - 1000 picograms (pg)		
pg expect	pg recovered	%Recovery
1027	992	96.6

ug/Nm^3 @ 0 degrees C and 1 Atm

*Example Calculation For Field Blank Correct Hg(II) ug/m^3

[(TrapA+TrapB-(2*FB)]/Sample Volume

FB=Mean Field Blank

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August 17, 1993

To: J.B. Owens

From: Nicolas Bloom

Re: Hg in coal (site #22) by perchloric acid digestion + CVAFS

sample ID	blank corrected Hg concentration, ng/g (as supplied)		
	run #1	run #2	mean
FC22-C001GCXX	56	54	55
FC22-C002GCXX	74	62	68
FC22-C003GCXX	60	64 65	62
blanks	11	11	11
NBS-1630	111 116	—	114
(certified)	—	—	127 ± 13

note: individual runs are separate digestions, while duplicates within cells are analytical reps.

Mercury Speciation
Sample Volumes

Run Number	Volume (Liters at 68° F)
1	103.8
2	108.0
3	102.3