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A Study of Toxic Emissions From A Coal-Fired Gasification Plant

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Final Report, December 1995

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EXECUTIVE SUMMARY

Under the Fine Particulate Control/Air Toxics Program, the U.S. Department of Energy (DOE) has been performing comprehensive assessments of toxic substance emissions from coal-fired electric utility units. An objective of this program is to provide information to the U.S. Environmental Protection Agency (EPA) for use in evaluating hazardous air pollutant emissions as required by the Clean Air Act Amendments (CAAA) of 1990. The Electric Power Research Institute (EPRI) has also performed comprehensive assessments of emissions from many power plants and provided the information to the EPA. The DOE program was implemented in two phases. Phase 1 involved the characterization of eight utility units, with options to sample additional units in Phase 2. Radian was one of five contractors selected to perform these toxic emission assessments.

Radian's Phase 1 test site was at Southern Company Service's Plant Yates, Unit 1, which, as part of the DOE's Clean Coal Technology Program, was demonstrating the CT-121 flue gas desulfurization technology. A commercial-scale prototype integrated gasification-combined cycle (IGCC) power plant was selected by DOE for Phase 2 testing. Funding for the Phase 2 effort was provided by DOE, with assistance from EPRI and the host site, the Louisiana Gasification Technology, Inc. (LGTI) project. This document presents the results of that effort.

The Louisiana Gasification Technology Inc. (LGTI) project was selected by the U.S. Synthetic Fuels Corporation to demonstrate the Dow Syngas process. The project commenced operation in 1987. It was partially funded by a Price Guarantee Commitment between Dow and the Synfuels Corporation. The guarantee has since been assumed by the Office of Synthetic Fuels, U.S. Treasury Department. Dow has formed a subsidiary, Destec Energy, which operates and markets their gasification technology.

Radian's assessment of emissions involved the collection and analysis of samples from the major input, process, and output streams of the IGCC plant for selected substances including those identified in Title III of the CAAA. These measurements provide information on the performance of processing systems within the plant and data on the fate of trace substances.

Site Description

The LGTI plant is located within Dow Chemical's Louisiana Division complex in Plaquemine, Louisiana. The petrochemical complex located there produces chlorine and caustic. The gasifier provides both process steam and synthesis gas, which is burned in turbines to produce electricity for the complex. Subbituminous coal from the Rochelle mine in the Powder River basin is used to produce a medium-Btu synthesis gas. At design feed rates of 2,200 tons of coal per day, 30,000 MM Btu of syngas is produced. Additional steam generated in the process produces a net output equivalent to 161 MW of electric power.

Process Description

Dow's gasifier design is proprietary but can be classified as a high-temperature, entrained-flow, slagging type. Coal is fed to the gasifier as a coal-water slurry which eliminates the need for coal lock hoppers. Oxygen and steam are added in a controlled manner to maintain the reactor within the design temperature range. Slag is removed as a water slurry, while hot synthesis gas is cooled in a heat recovery train that raises process steam. A particulate scrubber removes char from the gas, which is recycled to the gasifier. Further cooling of the syngas occurs followed by processing in a Selectamine™ unit to remove H_2S. The sweet syngas that results is blended with natural gas and fired in two gas turbines to produce electricity. The acid gas from the Selectamine™ unit is processed in a Selectox™ unit, producing elemental sulfur. Sour condensate is steam stripped, and the sour off-gas and Selectox™ tail gases are incinerated. Figure ES-1 shows a simplified block diagram of the plant. It also identifies the sampling locations used during this project.

Sampling Locations/Analytes

Due to the number and type of sampling locations as well as the groups of analytes measured, it was necessary to employ a phased approach during the test program. The majority of the plant was characterized during three consecutive test periods in November 1994. In a fourth test period, conducted in May 1995, a hot-gas probe was used to gather high-temperature/pressure samples from the raw syngas. Table ES-1 lists the sampling locations (the number refers to the location on Figure ES-1), the test period, and the types of analytes measured.

Quality Assurance and Quality Control

A rigorous QA/QC program was employed to ensure that the quality of the data produced during this effort would be well defined. Three major questions were addressed during this assessment: First, was the plant operating in a normal condition? Second, was the sampling of process streams representative, and last, were the analytical results obtained correct? Each of these concerns is discussed briefly below.

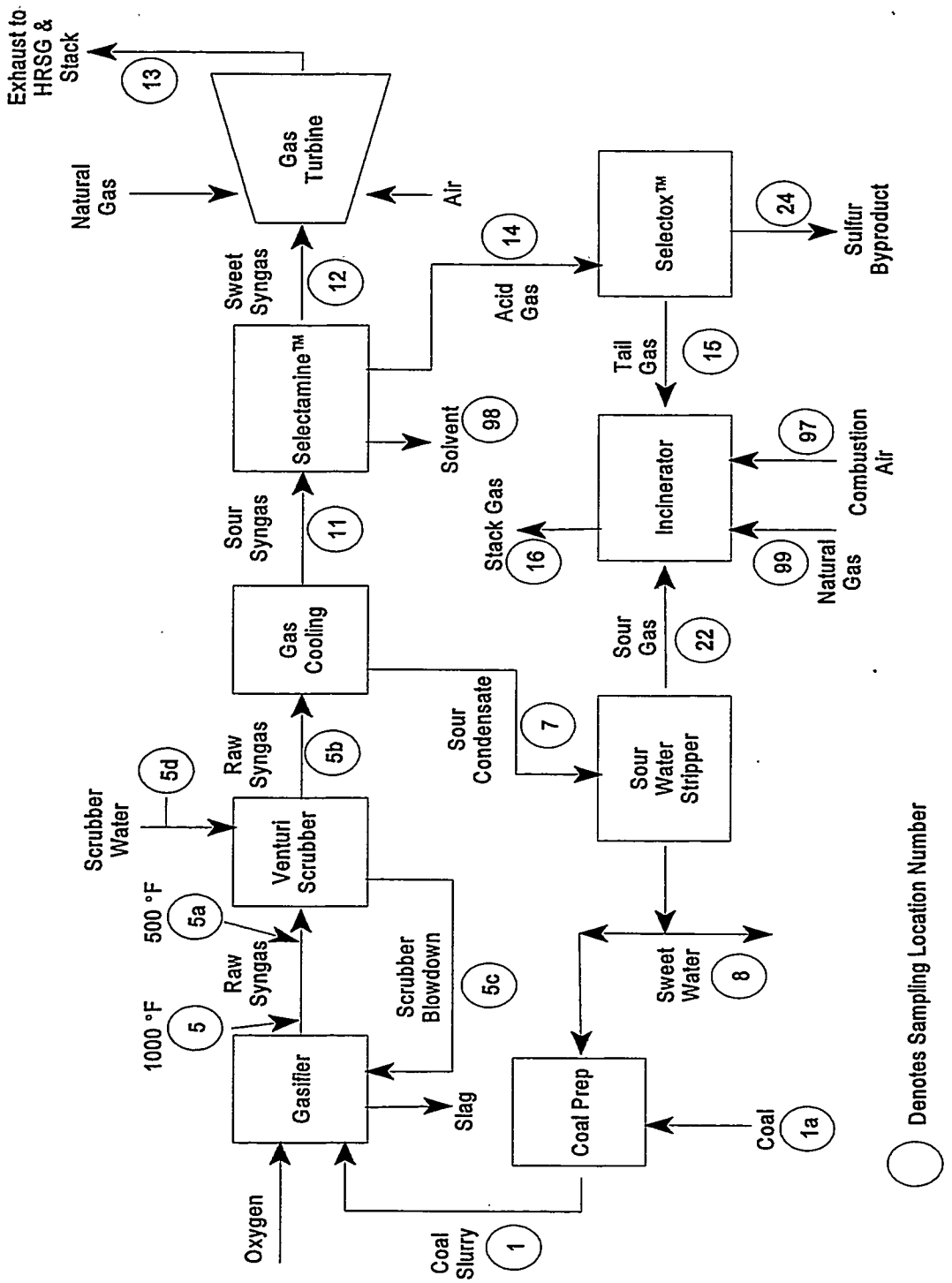

Figure ES-1
LGTI Block Flow Diagram

Table ES-1
Sampling Locations and Analytes

Location	Stream	Test Period	Analytes
1	Coal slurry	1, 2, 3	Metals, ultimate, proximate, anions
1a	Coal pile	1, 2, 3	Metals, ultimate, proximate, anions
		3	Radionuclides
4	Slag	1, 2, 3	Metals, ultimate, proximate, anions
		3	Radionuclides
5	Raw gas, 1,000°F	4	Vapor: metals, Cl, F, NH_3, HCN Particulate: metals
5a	Raw gas, 500°F	3	Metals, C_1-C_{10}, Cl, F, NH_3, HCN
5a	Raw gas, 500°F	probe shakedown test	Particulate: metals
5b	Raw gas, scrubbed	3	Metals, C_1-C_{10}, Cl, F, NH_3, HCN
5c	Scrubber blowdown (char)	3	Metals, ultimate, proximate, anions
	(filtrate)	3	Metals, ultimate, proximate, anions, ammonia, cyanide, suspended solids
5d	Scrubber water	3	Metals, ultimate, proximate, anions, ammonia, cyanide
7	Sour condensate	2	Metals, cyanide, volatile/semivolatile organics, aldehydes, anions, ammonia, phenol, sulfide, water quality
8	Sweet water	2	Metals, cyanide, volatile/semivolatile organics, aldehydes, anions, ammonia, phenol, sulfide, water quality
11	Sour syngas	1	Particulates, metals, C_1-C_{10}, volatile organics, major gases, sulfur species, semivolatile organics, aldehydes, Cl, F, NH_3, HCN
12	Sweet syngas	1	Particulates, metals, C_1-C_{10}, volatile organics, major gases, sulfur species, semivolatile organics, aldehydes, Cl, F, NH_3, HCN
13	Turbine exhaust	1	Particulates, PM-10, metals, VOST, semivolatile organics, aldehydes, Cl, F, NH_3, HCN, H_2SO_4, CEM gases
14	Acid gas	1	Metals, C_1-C_{10}, major gases, sulfur species, semivolatile organics, Cl, F, NH_3, HCN
15	Tail gas	1	Metals, C_1-C_{10}, major gases, sulfur species, semivolatile organics, NH_3, HCN
		2	C_1-C_{10}, sulfur species, semivolatile organics, NH_3, HCN, CEM gases
16	Incinerator stack	2	Particulates, PM-10, metals, VOST, sulfur species, semivolatile organics, aldehydes, Cl, F, NH_3, HCN, H_2SO_4, CEM gases

Table ES-1 (Continued)

Location	Stream	Test Period	Analytes
22	Sour gas	2	C_1-C_{10}, major gases, NH_3, HCN
24	Sulfur	1	Metals, ultimate, proximate
97	Combustion air	2	C_1-C_{10}, major gases, sulfur species, NH_3, HCN
98	Selectamine™ solvent	1	Metals, ash, volatile organics, heat stable salts
		3	Metals, ash, heat stable salts
99	Natural gas	2	Metals, C_1-C_{10}, sulfur species

Plant Operating Conditions

In general, the plant operation was very consistent, and the major monitored processes varied by less than ±10% during the test periods. On the third day of testing, November 5, the coal feed system plugged briefly, resulting in the unit going off-line for about 24 hours. Testing was resumed after another 24 hours on November 7. All other plant operational periods were normal with minimal variability.

Sample Collection

With the exceptions of the sampling locations discussed below, all other locations were sampled with minimal problems. While the collected samples are considered to be representative of normal process operation, some of the sampling methodologies used on the internal steams for trace elements are in their developmental stages and have not been validated or fully demonstrated. Therefore, the vapor phase metals results for some of the *internal* process streams are considered to be semi-quantitative.

Several of the internal gas streams contain high levels of water vapor. When the pressure and temperature is reduced during sample collection, moisture condensation occurs. Three sampling locations were heat traced to minimize condensation but another was physically impractical to heat trace. Therefore, at this one sample location, the mass of condensate collected was not equivalent to the gas volume collected, and some results for water-soluble components (i.e. ammonia) from this location have been invalidated due to their being non-representative.

One stream (the sour water stripper overhead), contained particularly high levels of NH_3 and CO_2. Upon cooling, ammonium carbonate deposits formed and plugged the sample line, preventing the collection of many of the planned samples for this stream.

In spite of the severe conditions encountered at many of the internal sampling locations, over 97% of the samples identified in the test plan were successfully obtained.

Analytical Results

At LGTI, both oxidized (containing excess oxygen) and reduced (containing hydrogen or sub-stoichiometric amounts of oxygen) streams exist in the process. Analytical QC results for the influent and effluent streams (coal, slag, sweet water, incinerator stack gas, and turbine stack gas) indicate that the analytical data are, with very few exceptions, of good quality and acceptable for use. This statement implies that the bias and precision of the results met the project data quality objectives and that minimal contamination was identified as a result of reagent, sampling, or analytical procedures. The material balances that were performed around the entire plant also support the "reasonableness" of the data obtained for the input and output streams of the plant.

A similar statement can be made for measurements of many of the internal streams. However, confidence in some of the results is not as high for the following reasons:

- No standard or validated methods are available or exist for sampling some of the substances measured.
- Some of the streams were sampled and analyzed by more than one method. When compared, the results from the different methods, in some instances, were conflicting.
- It was not possible to accurately determine particulate loading in most internal streams. This makes elemental material balances particularly difficult since trace element concentrations are typically highest in the particulate phase.
- Comparisons between different streams (e.g., by mass balance) sometimes produced illogical results.

Because of these reasons, the internal stream results, particularly for vapor-phase trace metal concentrations, should be considered only as approximations of the true concentrations.

Results

Testing at the LGTI facility has shown the following results:

- **LGTI's emissions of hazardous air pollutants were quite low.** For many substances the combined emission factors (turbine and incinerator stack) were lower than well-controlled pulverized coal steam-electric plants. QA/QC results for the emissions streams show, among other things, that ***76% of the trace element balances met the material balance objectives of 70 - 130% closure.*** Emission factors for selected HAPs are presented in Table ES-2.
- **The particulate emissions from the turbine exhaust stack were very low, measuring approximately 4 mg/Nm^3.**
- **The majority of trace and major elements present in the coal were found in the slag.**

Table ES-2
Emission Factors for Selected HAPs

	Combined Incinerator and Turbine Stack Emissions		
	Emission Rate	Emission Factor	
	lb/hr	lb/10^{12} Btu	95% CI
Particulate Loading	25	9,100	6,000
Ionic Species			
Chloride	1.7	740	180
Fluoride	0.090	38	22
Ammonia as N	1.2	440	430
Metals			
Antimony	0.011	4	4.7
Arsenic	0.0056	2.1	1.9
Barium	0.0096	3.5	1.3
Beryllium	2.5e-04	0.09	0.03
Cadmium	0.0078	2.9	3.8
Chromium	0.0073	2.7	0.63
Cobalt	0.0015	0.57	0.58
Lead	0.0077	2.9	1.5
Manganese	0.0083	3.1	6.5
Mercury	0.0046	1.7	0.43
Molybdenum	0.019	6.9	5.6
Nickel	0.011	3.9	3.6
Selenium	0.008	2.9	1.3
Aldehydes			
Acetaldehyde	0.0048	1.8	1.5
Benzaldehyde	0.0079	2.9	2.6
Formaldehyde	0.045	17	7.5
Volatile Organic Compounds			
Benzene	0.012	4.4	1.7
Carbon Disulfide	0.12	46	14
Toluene	5.3e-05	0.033	0.02
PAHs/SVOCs			
2-Methylnaphthalene	9.8e-04	0.36	0.55
Acenaphthylene	7.1e-05	0.026	0.0075
Benzo(a)anthracene	6.2e-06	0.0023	0.0002
Benzo(e)pyrene	1.5e-05	0.0056	0.0007
Benzo(g,h,i)perylene	2.6e-05	0.0096	0.0005
Naphthalene	1.1e-03	0.4	0.12
Benzoic acid	0.39	140	65

- **Some reduction in the concentration of trace substances in the syngas was measured across the Selectamine™ unit for both vapor-phase elements and organics.** Unfortunately, an operational procedure prevented an accurate assessment of the change in Selectamine™ liquid composition during the test period and trace element accumulation in the Selectamine™ solvent could not be determined.

- **The performance of the Selectox™ unit in converting H_2S to sulfur was relatively low, presumably due to the overall low sulfur level of the feed coal.** The SO_2 emissions from the gas turbine were about 0.02 lbs/MM Btu, and 0.13 lbs/MM Btu for the turbine and incinerator combined. These values were, however, well within the permitted limits for the LGTI facility.

- **Trace element mass balances around internal systems were uncertain due to the problems associated with the chemistry of sample collection and analysis of a reduced gas matrix.**

- **Although this test program was not focused on methods development, critical information was obtained regarding the characterization of trace elements in a reduced gas matrix.** The EPA Reference Method 29 (proposed), i.e., the multi-metals train, ***was ineffective in syngas (reduced gas) matrices (with the exception of mercury, discussed below)***. Although not validated, two other sampling techniques (charcoal tubes and VPAAS) for selected trace elements were implemented in parallel and these provided valuable insights into the deficiencies of EPA Method 29 for reduced gas matrices. The information obtained in this program, will provide a basis for method modifications needed for future work in characterizing IGCC systems.

- **This program resulted in a major breakthrough in the characterization of mercury in a syngas matrix.** The use of a semi-continuous mercury analyzer indicates that at least two forms of mercury are present in the synthesis gas. One of the forms is believed to be elemental mercury, the other is probably ionic. Additionally, valuable information was obtained for several absorbing/speciating solutions for the collection of mercury. The information obtained during the first three sampling periods provided the basis for modifications to the EPA Method 29 sampling train **specifically for the collection of mercury** during test Period 4. These modifications proved to be effective in the collection of mercury and will pave the way for future quantitative mercury measurements (perhaps even speciation) in syngas matrices.

- **A sampling probe was designed, fabricated, and successfully used to extract samples from a high temperature (1,000°F) and high pressure (350 psi) location.** Demonstrating the ability to collect representative samples under these conditions will allow the DOE to conduct further research in the characterization of hot gas removal systems.

Recommendations

During the four test periods at the LGTI plant and the subsequent examination and treatment of the collected data, the need for improvements, particularly in the area of syngas sampling, became apparent. These needs are expressed below as recommendations for further activities in these areas .

- **Improved and/or new methods for quantitatively collecting vapor-phase metals from syngas matrices are needed.**

  — It appears that the charcoal adsorbent used at LGTI behaved differently in the sour syngas compared to the sweet syngas. Charcoals impregnated with substances such as iodine or sulfide should be investigated for potential application in sampling syngas streams. Other adsorbents could also be investigated.

  — The existing Method 29 train was shown to be ineffective for quantitatively collecting samples of most vapor-phase metals from syngas streams (with modifications, mercury was an exception). By using other absorbing solutions, it might be possible to improve the effectiveness of this method, at least for some selected vapor-phase metals in syngas streams.

  — An on-line, vapor phase atomic absorption spectrophotometer (VPAAS) system (one of the alternate methods used for measuring trace metals in syngas) proved to be an effective method for determining the level of vapor-phase metals in the syngas streams. This method should be investigated further to add more elements to the list of analytes and to improve use of the instrumentation in the field environment. In particular, the studies for mercury speciation should be expanded to include identification of the different ionic mercury forms and potentially the development of "wet" test methods for mercury characterization in syngas matrices.

- **The hot gas probe insertion system designed and built for the LGTI testing was a complete success.** Further, the probe itself was also successfully used to collect and recover particulate samples from the hot gas stream. However, some improvements to the probe and enhancements of its capabilities should be considered.

  — The syngas stream at the LGTI plant (as well as streams at certain other syngas plants) contains a large amount of water vapor. The hot gas probe was designed for a gas stream temperature of 1,200°F. At LGTI, somewhat lower gas temperatures(< 1,000°) were encountered. As a result, during the collection of vapor-phase samples, the gas temperature dropped below the dew point and some of the water vapor in the syngas stream condensed in the probe. The probe should be modified to allow for internal heating so that condensation can be prevented.

— With minimal modifications, the probe could be used to determine particulate loadings and particle size distribution in low dust loading environments like those found at the outlet (and perhaps inlet) of a hot gas removal system.

— The probe insertion system will be effective regardless of the severity of the conditions in the gas stream being sampled. However, if the probe is to be used to sample gas streams at temperatures above 1250°F, the current materials of construction may be inappropriate. The material requirements and design changes necessary to accommodate very high temperature gas streams should be determined.

Conclusions

The data quality objectives were met and the results supported by the mass balance calculations, indicating that the characterization of the emission streams was very successful and the measured emissions from the LGTI process are very low. In comparison to the best controlled fossil steam-electric plants, the emissions are lower or equivalent for most substances. In Figure ES-2 the emission factors for those metals classified as HAPs from this project are compared to those obtained during the Phase I testing at Plant Yates (coal-fired boiler with ESP and scrubber).

[The reader should keep in mind that in spite of the information shown in Figure ES-2, the emission factors presented in this report are ***not*** *directly comparable to those of a conventional coal-fired power plant. At the LGTI facility, the syngas is co-fired with natural gas in two gas turbines, so there are two sources of Btu input, the coal and the natural gas. It is known that a fully natural gas-fired turbine can produce significant, measurable levels of HAPs. Unfortunately, at LGTI it is impossible to know how much of the emissions are attributable to the co-firing of the natural gas. As a result, the emission factors have been prepared as total mass out (turbine and incinerator) divided by total Btu content in (coal+incinerator natural gas+turbine natural gas).]*

Accurate quantification of internal process streams for vapor phase metals was hindered by the absence of suitable or fully developed sampling procedures. However, semi-quantitative measurements of vapor phase metals concentration were made, and valuable information was obtained to direct the development of new and/or modified methods for future tests.

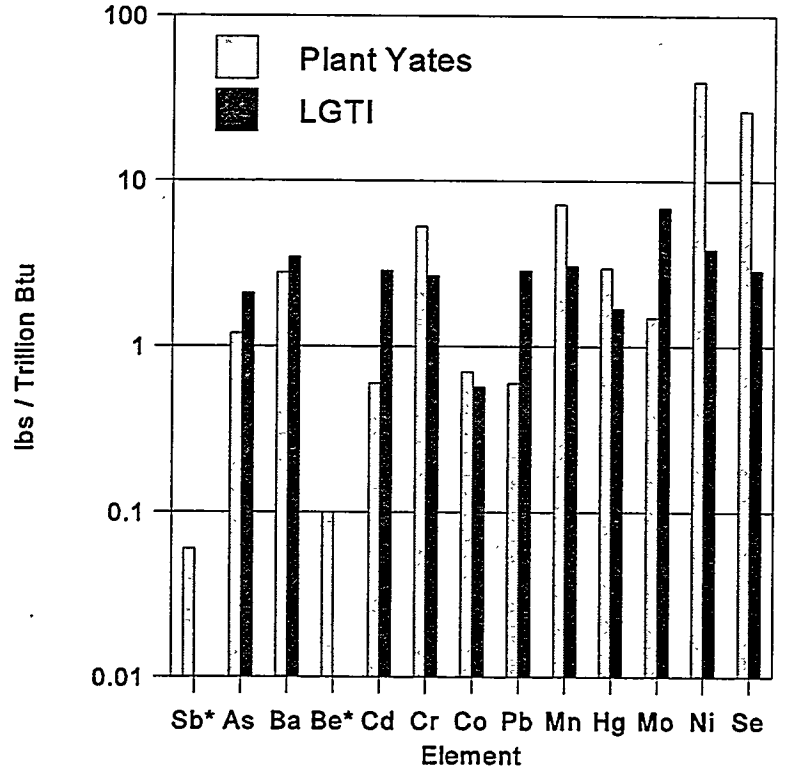

Figure ES-2
Metals Emission Factor Comparison to Plant Yates

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1

INTRODUCTION

Background

The Louisiana Gasification Technology, Inc. (LGTI) project was selected by the U.S. Synfuels Corporation to demonstrate the Dow gasification process. During the LGTI demonstration program, the environmental characteristics of some streams, particularly the discharge streams, have been regularly monitored as part of the Environmental Monitoring Program. However, with the passage of the Clean Air Act Amendments (CAAA) in 1990, it became very important to understand and to define the fate of currently unregulated hazardous air pollutants (HAPs) within and from various power plant configurations. The majority of HAPs have not yet been measured at the LGTI facility. For that reason, the U.S. Department of Energy (DOE) and the Electric Power Research Institute (EPRI) retained Radian Corporation to measure selected HAPs in the discharge streams and in most of the major internal process streams of the LGTI demonstration plant.

The HAPs test program was carried out in three consecutive test periods from October 30 through November 14, 1994, along with the collection of samples in support of the LGTI Environmental Monitoring Plan (EMP). This effort was jointly funded by the Pittsburgh Energy Technology Center (PETC), the Electric Power Research Institute (EPRI), and Destec Energy, Inc. Approximately 20 Radian personnel as well as representatives of PETC and Destec were on site during the two-week test effort. During this time, over 600 process samples were collected from approximately 20 locations throughout the gasification process.

In May 1995, during a fourth test period, samples of the hot, raw syngas under high pressure were obtained with the use of a specially designed probe.

Objectives

Specific objectives of this project were:

- To collect and subsequently analyze representative solid, liquid and gaseous samples of specified input and output streams of the Dow gasifier for selected hazardous air pollutants that are contained in Title III of the 1990 Clean Air Act Amendments, and to assess the potential level (concentration) of release (emission factors) of these pollutants to the atmosphere;

- To determine the removal efficiencies of specified pollution control subsystems for selected pollutants of the gasification plant; and

- To determine material balances for selected pollutants in specified input and output streams of the gasification plant, and mass flows for specific subsystems.

Table 1-1 lists the chemical substances analyzed during this project.

Emission factors, removal efficiencies, and other results rely on measurement data that vary and/or may be near or below the limit of detection for many of the substances of interest. This report includes uncertainty analyses and confidence intervals in order to assess the quality of the data.

Auditing

During the field sampling program conducted at the LGTI gasifier, a quality assurance (QA) audit was conducted by Radian Corporation's internal QA auditor. Radian's audit was conducted with the purpose of providing an objective, independent assessment of the sampling effort, thus ensuring that the sampling procedures, data generating, data gathering, and measurement activities produced reliable and useful results. As part of the audit, calibration documentation, quality control (QC) data documentation, data forms and notebooks, data review/validation procedures, and sample logging procedures were reviewed.

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 indicate that the samples were well characterized. An assessment of the accuracy, precision, and bias of the data, if only on a qualitative level, was considered to be an important part of the data evaluation. A full discussion of each of these components can be found in Section 5 and in Appendix A.

Project Organization.

Figure 1-1 shows the organization of this project.

Report Organization

This report presents a comprehensive assessment of the results of this test effort. Section 2 presents a summary of the sampling activities. Section 3 contains process operation information including trend plots of key operating parameters. Analytical results are presented in Section 4, and Section 5 contains an evaluation of data and quality. The results are discussed in Section 6. Sections 7 and 8 address special topics. Section 7 presents a comparison of the results from three different methods used to analyze for selected trace elements in a syngas matrix. Section 8 contains a discussion of mercury measurement methods, including speciation in a syngas matrix.

Table 1-1
Target Analytes

Trace Elements			
Antimony	Boron	Copper	Molybdenum
Arsenic	Cadmium	Lead	Nickel
Barium	Chromium, total	Manganese	Selenium
Beryllium	Cobalt	Mercury	Vanadium
Radionuclides			
Anions			
Chloride (HCl)			
Fluoride (HF)			
Sulfate			
Reduced Species			
Ammonia			
Cyanide			
Hydrogen Sulfide			
Carbonyl Sulfide			
Carbon Disulfide			
Major Gases			
Carbon Dioxide			
Carbon Monoxide			
Hydrogen			
Oxygen			
Nitrogen			
Minor Gases			
Sulfur Dioxide			
Nitrogen Oxides			
C_1-C_{10} Hydrocarbons			
Aldehydes			
Acetaldehyde			
Acrolein			
Benzaldehyde			
Formaldehyde			

Table 1-1 (Continued)

Volatile Organics	
Benzene	Methyl Chloroform (1,1,1-Trichloroethane)
Bromoform	Methyl Ethyl Ketone (2-Butanone)
Carbon Disulfide	Methylene Chloride (Dichloromethane)
Carbon Tetrachloride	Propylene Dichloride (1,2-Dichloropropane)
Chlorobenzene	Styrene
Chloroform	1,1,2,2-Tetrachloroethane
1,4-Dichlorobenzene	Tetrachloroethene
cis-1,3-Dichloropropene	Toluene
trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Ethyl Benzene	Trichloroethene
Ethyl Chloride (Chloroethane)	Vinyl Acetate
Ethylene Dichloride (1,2-Dichloroethane)	Vinyl Chloride
Ethylidene Dichloride (1,1-Dichloroethane)	Vinylidene Chloride (1,1-Dichloroethene)
Methyl Bromide (Bromomethane)	m,p-Xylene
Methyl Chloride (Chloromethane)	o-Xylene

Semivolatile Organics		
Acenaphthene	Indeno(1,2,3-cd)pyrene	7,12-Dimethylbenz(a)anthracene
Acenaphthylene	Isophorone	Dimethylphenethylamine
Acetophenone	Methyl Methanesulfonate	2,4-Dimethylphenol
4-Aminobiphenyl	3-Methylchlolanthrene	Dimethylphthalate
Aniline	2-Methylnaphthalene	4,6-Dinitro-2-methylphenol
Anthracene	2-Methylphenol (o-cresol)	2,4-Dinitrophenol
Benzidine	4-Methylphenol (p-cresol)	2,4-Dinitrotoluene
Benzo(a)anthracene	N-Nitroso-di-n-butylamine	2,6-Dinitrotoluene
Benzo(a)pyrene	N-Nitrosodimethylamine	Diphenylamine
Benzo(b)fluoranthene	N-Nitrosodiphenylamine	1,2-Diphenylhydrazine
Benzo(g,h,i)perylene	N-Nitrosopropylamine	Ethyl Methanesulfonate
Benzo(k)fluoranthene	N-Nitrosopiperidine	2-Nitrophenol
Benzoic Acid	Naphthalene	4-Nitrophenol
Benzyl Alcohol	1-Naphthylamine	Pentachlorobenzene
4-Bromophenyl Phenyl Ether	2-Naphthylamine	Pentachloronitrobenzene
Butylbenzylphthalate	2-Nitroaniline	Pentachlorophenol
4-Chloro-3-Methylphenol	3-Nitroaniline	Phenacetin
p-Chloraniline	4-Nitroaniline	Phenanthrene
bis(2-Chloroethoxy)methane	Nitrobenzene	Phenol
bis(2-Chloroethyl)ether	Di-n-octylphthalate	2-Picoline
bis(2-Chloroisopropyl)ether	Dibenz(a,h)anthracene	Pronamide

Table 1-1 (Continued)

Semivolatile Organics (Continued)		
1-Chloronaphthalene	Dibenz(a,j)acridine	Pyrene
2-Chloronaphthalene	Dibenzofuran	Pyridine
2-Chlorophenol	Dibutylphthalate	1,2,4,5-Tetrachlorobenzene
4-Chlorophenyl Phenyl Ether	1,2-Dichlorobenzene	2,3,4,6-Tetrachlorophenol
Chrysene	1,3-Dichlorobenzene	1,2,24-Trichlorobenzene
bis(2-Ethylhexyl)phthalate	1,4-Dichlorobenzene	2,4,5-Trichlorophenol
Fluoranthene	3,3'-Dichlorobenzidine	2,4,6-Trichlorophenol
Fluorene	2,4-Dichlorophenol	2-Fluorobiphenyl
Hexachlorobenzene	2,6-Dichlorophenol	2-Fluorophenol
Hexachlorobutadiene	2,6-Dichlorophenol	Nitrobenzene-d5
Hexachlorocyclopentadiene	Diethylphthalate	Phenol-d5
Hexachloroethane	p-Dimethylaminoazobenzene	Terphenyl-d14
		2,4,6-Tribromophenol

Additional Elements			
Aluminum	Magnesium	Silicon	Zinc
Calcium	Phosphorus	Sodium	
Iron	Potassium	Titanium	

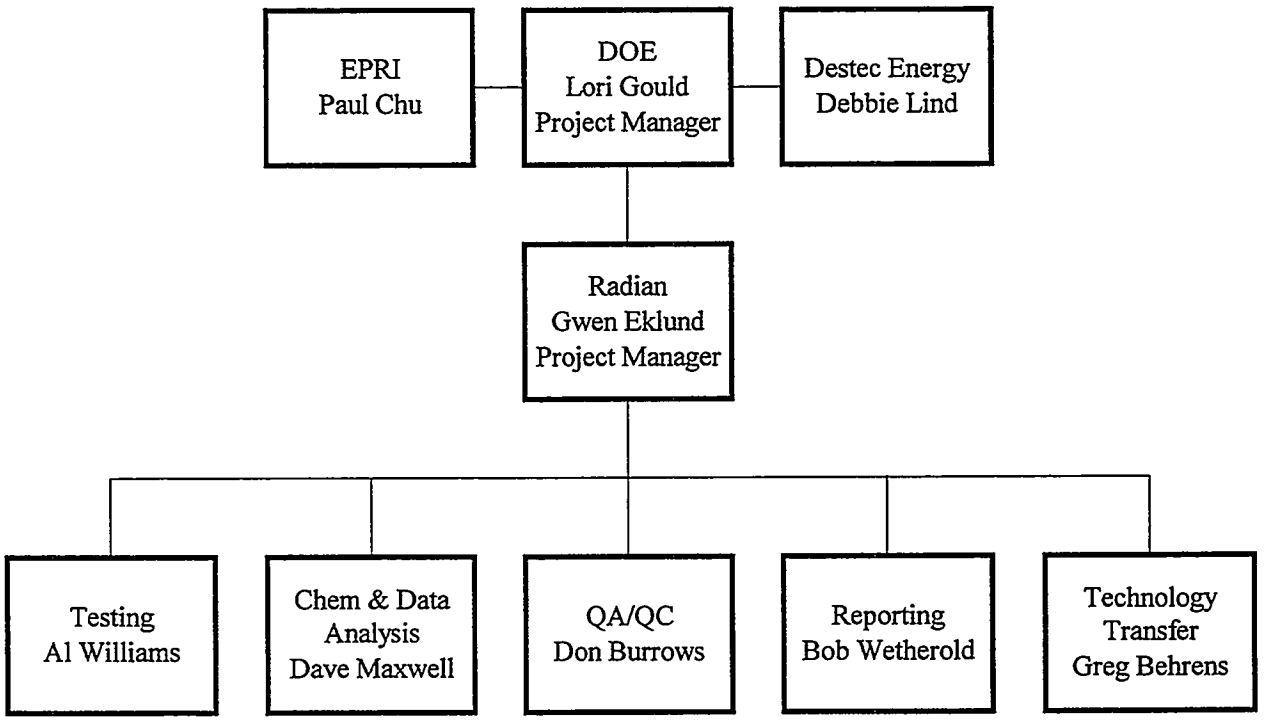

Figure 1-1
Project Team Organization

Nine appendices are also included with this report. Appendix A contains detailed information on quality control sample results and the Radian independent auditor's report. Appendix B contains a description of the sampling procedures that were used. Sample preparation and analytical techniques are presented in Appendix C. Appendix D contains example calculations for bias and precision. Appendix E contains example calculations for material balances, removal efficiency and emission factors. Appendix F contains field sampling data summary reports and the detailed analytical results are contained in Appendix G. Appendix H is a description of the hot-gas sampling probe design, and Appendix I is a glossary of terms.

2

SUMMARY OF SAMPLING ACTIVITIES

LGTI (Louisiana Gasification Technology Inc.), a subsidiary of Destec Energy Inc., operates the coal gasification plant at the Dow Louisiana Division chemical complex in Plaquemine, Louisiana. The gasification unit produces medium Btu synthesis gas (syngas) for consumption by two gas turbine power generating units at the Louisiana site.

At full capacity, the LGTI Plant produces 30,000 MM Btu of equivalent syngas per day from approximately 2,200 tons per day of western subbituminous coal from the Rochelle mine in the Powder River Basin in Wyoming. This is the equivalent of 160 MW of net power, considering both electricity and steam production.

Process Descriptions/Sample Locations

Figure 2-1 is a block flow diagram of the LGTI gasification facility at Plaquemine with the sampling points identified. The block diagram includes coal handling, gas production, particulate removal, moisture removal, acid gas cleanup, power production, wastewater stripping, acid gas treatment, sulfur production, and tail gas incineration. The following paragraphs provide a description of the process and include information on the sample collection points.

Coal Slurry

The plant receives the feedstock, Rochelle coal, by rail car. Coal is transported by conveyor (partly covered) to a coal pile. Reclaiming and transfer from the coal pile is accomplished by bulldozers that fill reclaim pit hoppers for feed to conveyors that transport coal to the precrusher. Coal samples (1a) were collected at this preparation plant. Precrushed coal is held in a feed-hopper and then transferred by weigh belt feeders to an enclosed coal slurry grinder, where recycled process water is added. Coal slurry is transported from the grinder product tank by slurry pumps to slurry storage tanks located at the gasifier area. Coal slurry (1) samples were collected from the plant's 33 and 33a feed pumps.

Gasification

The gasifier is a high-temperature, oxygen-blown, entrained-flow, slagging design. Pumps designed for handling liquid-solid suspensions at high pressure control the slurry feed rate to the gasifier. The coal slurry, which is fed to the reactor, is mixed with oxygen in the burner nozzles. The oxygen feed rate is carefully controlled to maintain the reactor temperature within a narrow range. Sulfur in the coal is converted almost totally to H_2S, and small amounts of COS, while

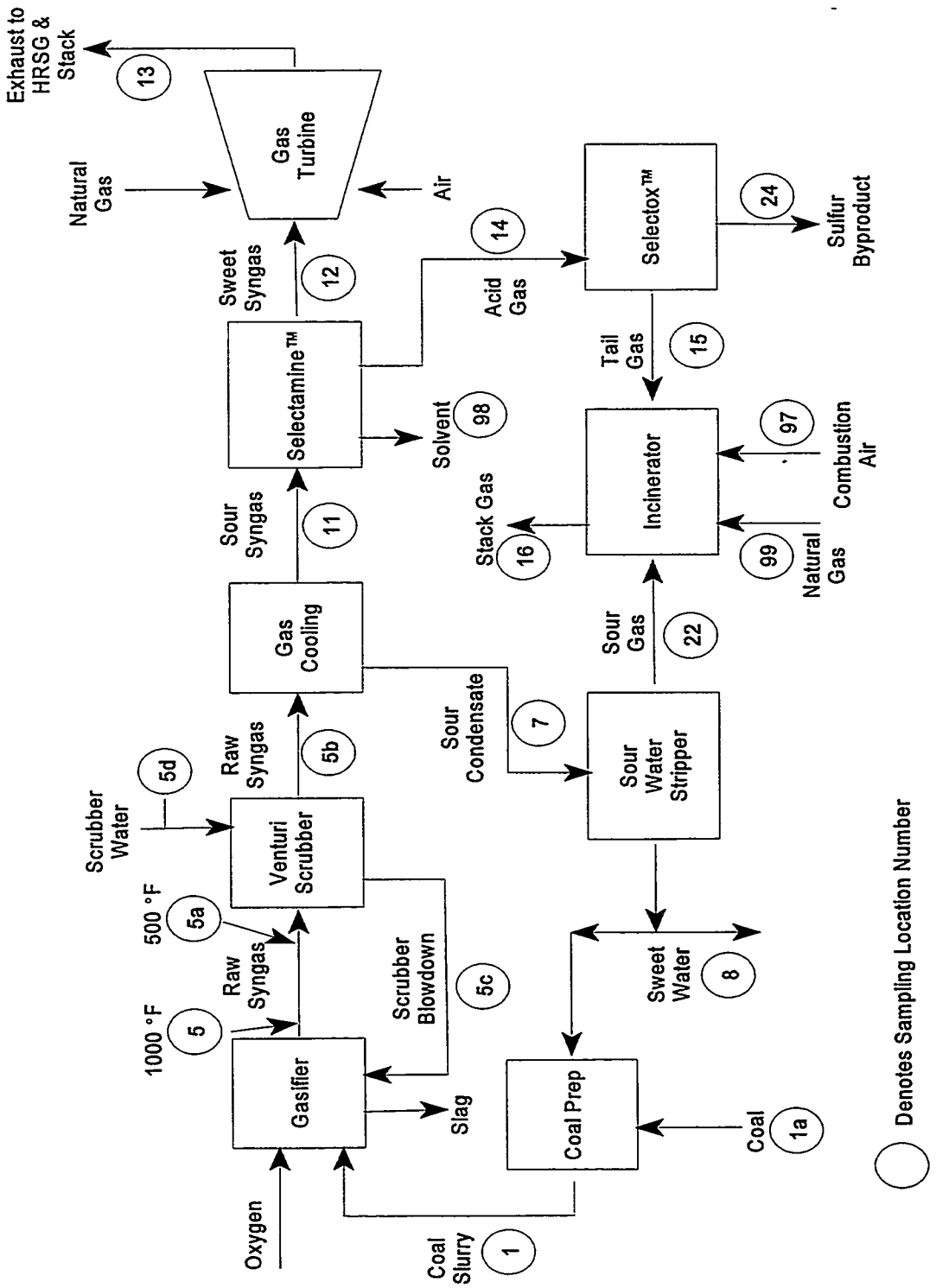

Figure 2-1
LGTI Block Flow Diagram

nitrogen is efficiently converted to NH_3, and trace amounts of cyanide and thiocyanate. Ash from the coal is fused in the combustion area of the reactor, and the molten slag is drained from the reactor bottom through an enclosed system. The molten slag is quenched and cooled by water. Slag (4) was collected from a slipstream near the slag hopper discharge.

Particulate Removal

The hot raw syngas produced in the gasifier passes through several gas cooling and cleaning systems. Before particulate removal, the gas is first cooled in a convection cooler by heat exchange with water for steam production. Hot raw syngas (Stream 5) was sampled from this location using a high-temperature, high-pressure retractable sampling probe (fourth test period). The syngas temperature at this sample point was 1,000°F, and the stream's characteristics are considered typical of the syngas likely to be encountered by hot gas cleanup systems currently being developed and evaluated.

Entrained particulate matter (char) is removed from the gas by a wet venturi scrubber system. The raw syngas was sampled at the inlet to the scrubber (Stream 5a), and the scrubbed raw syngas was collected from the scrubber outlet duct (Stream 5b). The syngas temperature at the scrubber inlet was approximately 450°F.

The scrubber water containing the removed char is recycled by injection into the gasifier with the secondary slurry feed. Samples of the clean scrubber water feeding the venturi were collected from the storage tank at the suction of the venturi scrubber pumps (Stream 5d). The char solids were recovered by filtering the recycled char water (Stream 5c) collected from a tap in the transfer line.

Gas Cooling/Moisture Removal

The particulate-free gas is cooled further to condense moisture from the gas. The cooled syngas entering the acid gas removal system (sour syngas-11) was sampled downstream of a large condensate knock-out vessel immediately upstream of the acid gas removal system. The condensate removed from the cooled gas (sour condensate-7) contains substantial amounts of soluble sulfide, ammonia, and carbon dioxide and was collected from a tap in the line transferring the sour condensate to the sour water stripper.

Acid Gas Removal

Dow's Selectamine™ acid gas removal process removes over 97% of the sulfur species from the sour syngas. The principal ingredient in the Selectamine™ solvent is methyl diethanolamine (MDEA). The acid gas is absorbed in the MDEA solution. A portion of the MDEA is removed periodically and is regenerated or replenished with fresh solution to control the buildup of contaminants in the solution. The sweetened product gas (12) is sent by pipeline to the power plant for use as gas turbine fuel. It was sampled near the LGTI control room on the main transfer pipe to Power II. The concentrated acid gas (14), which consists primarily of H_2S, CO_2 and

water, is recovered by stripping the rich MDEA solvent. The concentrated acid gas stream is sent to the sulfur recovery unit. It was sampled from a transfer line in the sulfur unit. The MDEA solvent (98) was sampled by Dow personnel from the spent solvent line before a partial regeneration step which was performed during the test period.

Sulfur Recovery/Incineration

The Selectox™ process is used to recover sulfur from the acid gas produced in the Selectamine™ unit. This process uses a fixed bed of Selectox™ catalyst to oxidize a portion of the H_2S to SO_2 prior to sulfur production, as opposed to the combustion furnace in the Claus process. A Claus reactor then catalyzes a redox reaction between the H_2S and SO_2 to produce elemental sulfur. Sulfur byproduct (24) was collected from the reservoir with metal sampling cups. The tail gas from the Selectox™ unit is fed to an incinerator to oxidize the small amount of remaining H_2S to SO_2. The tail gas (15) was sampled beneath the Selectox™ reactor. The incinerator stack gas (16) is exhausted to the atmosphere. Ports in the stack were used for this sample. Combustion air (97) from tank vents and natural gas (99) for the incinerator were sampled from available ports and taps.

Power Production

Clean syngas from the acid gas removal unit is co-fired with natural gas in two gas turbines at Dow's Power II facility. The two Westinghouse WD501-D5 gas turbines, can burn either natural gas or a blend of syngas and natural gas, and each can produce up to 105 MW of electrical power. In addition, a waste heat boiler recovers much of the energy in the turbine exhaust and produces steam for the Dow Chemical facility. Each turbine exhaust is routed through a heat recovery boiler and emitted to the atmosphere from the turbine exhaust stacks. During the testing, the Btu content of the fuel was approximately 63% syngas and 37% natural gas for the turbine that was tested.

Process Wastewater

The sour water condensed from the product gas as it cools is directed to the wastewater treatment system which includes filtration and stripping. Stripped sour water (sweet water) from the treatment system is recycled to the coal preparation area. Excess sweet water (8) is discharged through a permitted outfall. The sour gas (22) stripped from the condensate is routed to the tail gas incinerator. It was sampled at the fan deck.

Samples Collected

Tables 2-1 through 2-7 define the samples targeted for collection. The samples that *were not* collected, primarily from the sour gas location, have been shaded. The problems associated with sampling (or collecting samples) at this and other locations are discussed below. Overall sample

Table 2-1
Gas Stream Sampling Matrix—Period 1[a]

Parameter	Sour Syngas, 11	Sweet Syngas, 12	Acid Gas, 14	Tail Gas, 15	Turbine Stack, 13	Media Blank	PE Audit
Particulate Loading		3			3		
PM-10					3		
Metals, M-29	3	3+FB	3	3	3+FB	1	
Metals, Direct AAS	1	2					
Metals, Charcoal Tubes	3	3+FB	3				
C_1 - C_{10} by GC-FID	3	3	3	3			
Volatile Organics by VOST					3x(3+FB)	1	
Volatile Organics by TO-14	3	3			3	1	
Major Gases by GC-TCD	3	3	3	3			
H_2S, COS, CS_2, DMS, DMDS, methyl mercaptan by GC-FPD	3	3	3	3			
H_2S by M-11		3					
Semivolatile Organics	3	3	3	3	3+FB	1	
Aldehydes	3	3			3+FB	1	
Chloride, Fluoride	3	3	3		3+FB	1	
Ammonia	3	3	3	3	3+FB	1	1
Cyanide	3	3	3	3	3+FB	1	1
SO_2/H_2SO_4 by M-8					3	1	1
SO_2, NO_x, CO,O_2, CO_2 by CEM					3		

[a] Shaded areas indicate samples not collected.

PE = Performance evaluation audit sample.

FB = Field blank.

Table 2-2
Solid and Liquid Stream Sampling Matrix—Period 1[a]

Parameter	Raw Coal, 1a Field Samples	Coal Slurry, 1 Field Samples	Slag, 4 Field Samples	MDEA, 98 Field Samples	Sulfur Byproduct, 24 Field Samples
Metals	3	3 x 2	3	1	3 (2)
Ultimate, Proximate	3	3 x 2	3		3 (2)
Chloride, Fluoride	3	3 x 2	3		
Ash				1	
Volatile Organics				1	
Heat stable salts				1	

[a] Shaded areas indicate samples not collected; actual number in parentheses.

Table 2-3
Gas Stream Sampling Matrix—Period 2[a]

Parameter	Tail Gas, 15	Natural Gas, 99	Sour Gas, 22	Combustion Air, 97	Incinerator Stack, 16	Media Blank	PE Audit
Particulate Loading					3	1	
PM-10					3	1	
Metals, M29[b]		3	3		3+FB	1	1
C_1 - C_{10}	3	3	3	3	3		
Volatile Organics by VOST					3x(3+FB)	1	
Volatile Organics by TO-14					3	1	
Total Chromatographical Organics					3		
Major Gases by GC-TCD			3	3			
H_2S, COS, CS_2, DMS, DMDS, methyl mercaptan by GC-FPD	3	3		3	3		
H_2S by M-11					3		
Semivolatiles	3		3		3+FB	1	
Aldehydes					3+FB	1	
Chloride, Fluoride					3	1	
Ammonia	3		3+FB	3	3		
Cyanide	3		3+FB	3	3		
SO_2/H_2SO_4, M-8					3+FB		
NO_x, M-7D					3		1
SO_2,NO_x,CO, O_2, CO_2 by CEM	3 (1)				3 (2)		

[a] Shaded areas indicate samples not collected; actual number in parentheses.

[b] Sb, As, Ba, Be, B, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Se, Pb, V, and Zn. Alkali metals: Ca, Mg, Na and K. Major metals: Al, Fe, and Ti.

FB = Field blank.

Table 2-4
Solid and Liquid Stream Sampling Matrix—Period 2

Parameter	Raw Coal, 1a	Coal Slurry, 1	Slag, 4		Sour Condensate, 7		Sweet Water, 8		
	Field Samples	Field Samples	Field Samples	Duplicate Sample	Field Samples	Duplicate Sample	Field Samples	Duplicate Sample	PE Audit[a]
Metals[b]	3	3 x 2[c]	3	1	3	1	3	1	1
Ultimate, Proximate[d]	3	3 x 2	3	1					
Chloride, Fluoride	3	3 x 2	3	1					
Cyanide[e]					3	1	3	1	1
Volatile Organics					3	1	3	1	
Semivolatile Organics					3	1	3	1	
Aldehydes					3	1	3	1	
Anions[f]					3	1	3	1	1
Ammonia, Phenol, COD					3	1	3	1	1
Sulfide					3	1	3	1	
Water Quality[g]					3	1	3	1	

[a] Performance evaluation (PE) audit sample.

[b] Coal slurry collected from two locations, 33 and 33a: 3 (samples) x 2 (streams).

[c] Target trace metals: Sb, As, Ba, Be, B, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Se, Pb, V, and Zn. Alkali metals: Ca, Mg, Na and K. Major metals: Al, Fe, Si, and Ti.

[d] Moisture, ash, higher heating value (HHV), C, H, O, N, S, volatile and fixed carbon. Slag samples not analyzed for HHV or volatile and fixed carbon.

[e] Water streams analyzed for total and free cyanide and thiocyanate.

[f] Anions: Cl^-, F^-, $SO_4^{=}$, PO_4^{-3}, and formate.

[g] Water Quality: pH, conductivity, and total suspended solids (TSS).

Table 2-5
Gas Stream Sampling Matrix—Period 3

Parameter	Raw Gas (500°F), 5a	Scrubbed Raw Gas, 5b	Media Blank	PE Audit
Particulate Loading		3		
Metals, Method 29[b]	3[c]+FB[d]	3	1	1
Metals, Direct AAS[e]				
Metals, Charcoal Tubes[f]	3	3	3	
C_1 - C_{10}[g]	3	3		
Ammonia	3	3+FB		
Cyanide	3	3+FB		
Chloride, Fluoride	3	[c] 3+FB	1	1

[a] Performance evaluation (PE) audit sample.

[b] Target trace metals: Sb, As, Ba, Be, B, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Se, Pb, V, and Zn. Alkali metals: Ca, Mg, Na and K. Major metals: Al, Fe, and Ti. Silicon determined in the raw gas (5a) particulate phase only.

[c] Includes particulate- and vapor-phase analysis.

[d] FB = Field blank.

[e] Direct atomic absorption spectrophotometry (AAS) for As, Cd, Cu, Fe, Pb, Hg, Ni, Se, Zn.

[f] Sb, As, Cd, Fe, Pb, Hg, Ni, Zn.

[g] C_1 - C_6 hydrocarbons, benzene, toluene, and xylene.

Table 2-6
Solid and Liquid Stream Sampling Matrix—Period 3

Parameter	Raw Coal, 1a	Coal Slurry, 1			Slag, 4		Rcy. Char Solids, 5c		Rcy. Char Filtrate		Scrubber Inlet Water	MDEA, 98
	Field Samples	Field Samples	Dup	PE Audit	Field Samples	PE Audit	Field Samples	Dup	Field Samples	Dup	Field Samples	Field Samples
Metals	3	3 x 2	1	1	3	1	3	1	3	1	3	1
Ultimate/Proximate	3	3 x 2	1	1	3	1	3	1	3	1	3	
Radionuclides	3				3							
Chloride/Fluoride	3	3 x 2	1	1	3	1	3	1	3	1	3	
Ammonia									3	1	3	
Cyanide									3	1	3	
Ash												1
Heat Stable Salts												1
Total Suspended Solids									3	1		

Dup = Duplicate sample.

PE = Performance evaluation audit sample.

Table 2-7
Gas Stream Sampling Matrix, Period 4

Parameter	(Shakedown) Raw Gas Particulate, 5a	Raw Gas, 1,000°F, 5
Metals, Method 29[a]	2[b]	3
Charcoal Tubes[c]		3
Ammonia		3
Cyanide		3
Chloride, fluoride		3

[a] Target trace metals: Sb, As, Ba, Be, B, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Se, Pb, V, and Zn. Alkali metals: Ca, Mg, Na, and K. Major metals: Al, Fe, Si, and Ti.

[b] Particulate only.

[c] Sb, As, Cd, Fe, Pb, Hg, Ni, Zn.

capture for the testing effort exceeded 97 percent. Because of the number of sampling locations, the effort was conducted in four periods. Period 1 targeted the sulfur removal and turbines, Period 2 focused on the sour water and incinerator, and Period 3 addressed gasifier streams. Period 4, conducted separately in May 1995, sampled the hot raw syngas (Location 5). Prior to that, *shakedown* testing to assess the operability and functionality of the hot gas probe was completed in April, 1995. Shakedown testing was done at low (500°F) temperature conditions. The sampling matrix is presented in Table 2-7. All targeted samples were collected and no problems were encountered during the hot gas sampling phase of the program. The temperature at the hot gas location was over 900°F. The sampling system that was used was designed for a maximum temperature of 1,250°F.

Problems Encountered

Particulate-Free Gas (5b)

The sample port at the outlet of the venturi scrubber required the use of a long run (>20 feet) of stainless steel tubing to reach the sampling station. The hot gas extracted from the process line cooled rapidly in the tubing. Consequently, the condensed moisture in the gas line could not be sampled or collected representatively. To minimize the effects from the condensed moisture, the first sample tap was run continually and the condensed moisture was collected in a knockout. All samples were collected from taps downstream of the first tap in an attempt to collect only vapor-phase concentrations. (Multiple taps were used on sample lines to provide samples for the various measurement methods.)

Because some of the vapor-phase species are soluble in water, these gas samples may be biased low. Fractions of the "condensed" water in the knockout were collected and analyzed for specific volatile species (such as ammonia, cyanide and metals for example). "Vapor-phase" data for ammonia and cyanide were adjusted to account for the water fractions.

Sour Syngas (11)

Similar difficulties were encountered at the sour syngas location. Even though the sample port was downstream of a knock-out vessel, an excessive amount of water was collected from the first sample tap relative to the others. The high moisture content precluded the measurement of particulate loading as the filter substrates became wet, and the filtration capacity was compromised. Samples collected at this location, prior to the discovery of the moisture problem, were not analyzed and sample collection was repeated with a condensate knock-out system upstream of the sample header.

Sour Gas (22)

High concentrations of ammonia and carbon dioxide in the sour gas resulted in the crystallization of ammonium carbonate in the sample lines. The ammonium carbonate formed plugged each of the sampling systems used preventing most samples from being obtained. The only samples collected were for cyanide and ammonia. This was possible only because the sample volume required for these species is small (one cubic foot).

Plant Operational Problems

On the third day of testing (Period 1), Saturday, November 5, plant operations were suspended due to equipment plugging in the coal feed system. The plant was off-line for approximately 24 hours and was given another 24 hours to restabilize. Testing resumed on Monday, November 7.

Sampling Equipment Problems

On the same day the plant was taken off line, the compressor in a refrigerator, used to keep samples for semivolatile organic analyses cool, malfunctioned and heated the interior and freezer compartments. This refrigerator contained samples from the first two days of testing as well as blank or empty XAD resin cartridges for future test periods. The possibility of either loss of sample or contamination of the blank media for those yet to be used was considered. Many of the internal process steams were known to contain measurable quantities of semivolatile organics and the emission gas steams (turbine and incinerator stacks) were thought to contain extremely low concentrations of semivolatile organics (these steams were being analyzed by HRGC/MS). Following discussions with PETC, EPRI, and Destec, a decision was reached to rerun all semivolatile tests and to replace all the blank XAD resin that was subjected to the temperature excursion in the refrigerator. New resins were obtained and all affected tests were repeated.

3

PROCESS OPERATION

The plant operation was stable and within the specified or target process operating limits during the monitoring period, except for one brief shutdown that occurred on November 5. Following the shutdown, the plant was restarted on November 6 and reached stable and normal operating conditions within a few hours.

To ensure that samples were taken under typical and representative operating conditions, key process data and information were manually acquired and logged on a regular basis. These data were selected to allow monitoring of all the major systems within the gasification unit during testing. At the completion of testing, detailed (five-minute averages) process data were obtained from the LGTI data acquisition system in the form of computer printouts.

The values of several key operating parameters obtained from the data acquisition system are summarized in Table 3-1. Average values of the selected parameters are presented for the overall testing period as well as the three primary test periods. The data represent the daily periods (0700 through 1800 hours) during which most of the testing was conducted. Also shown in the table are the maximum, minimum, and standard deviation for each set of data. Several subsets of these data are shown graphically in Figures 3-1 through 3-7. Each of the figures shows key parameters associated with a specific process unit. In a few cases, parameter data are shown in more than one figure to illustrate the stability of the particular process unit during the testing periods. The data in each figure represent hourly values during the hours of 0700 through 1800 for November 3-4 and November 6-13. The consistent and stable operation of the plant can be seen from the graphs, as well as from the relatively low standard deviations of the data sets presented in Table 3-1.

Process operation was not directly monitored during Period 4, however, an LGTI operator was stationed at the sampling location and was in direct contact with the control room at all times. No upsets which would affect the gasification system were noted during the Period 4 testing.

Table 3-1
Summary of Key Process Parameters

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
Slag Production, dry ton/day (LGTI)				
Coal Feed Rate, dry ton/day (LGTI)				
Coal Feed Rate (Radian calc), ton/day (dry)				
Average Calculated Rate				
Maximum Calculated Rate				
Minimum Calculated Rate				
Sample Standard Deviation				
Primary Slurry, % solids				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Primary Slurry Rate 1, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Primary Slurry Rate 2, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Second Stage Slurry, % solids				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Second Stage Slurry Rate, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Average				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
HR120B Average Temperature, °F				
Maximum				
Minimum				
Sample Standard Deviation				
Sour Syngas Flow From E-163, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Scrubber Gas Inlet Temperature, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Scrubber Gas Inlet Pressure, psig				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Water Flow to Venturi Scrubber, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Water Flow to Tangential Nozzles, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Demineralized Water Flow to Scrubber				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
Water Flow to MX-125, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
C-165 Blowdown to C-160, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Scrubber Outlet Gas Temperature, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Scrubber Outlet Gas Pressure, psig				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Sweet Syngas Flow Rate. lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Acid Gas Flow Rate, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
Combustion Air Rate to F-251, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
F-251 (Incinerator) Exit Temperature, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Incinerator Stack Gas Flow Rate, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Total SO_2 Flow Rate to Incinerator Stack, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Stripper Overhead Temperature, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Sweet Water to Ditch, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
C-170 (Absorber) Overhead Temperature. °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
C-180 (Stripper) Temperature above Tray 17, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Lean MDEA Rate to C-170, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Tail Gas Flow Rate, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
K-250 Vent Gas Flow Rate, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Methane Flow Rate to F-251 (Incinerator), lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
Steam Drum Blowdown to C-270, pph				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
D-251 Blowdown to C-270, pph				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
C-180 Water Purge to C-270, gpm				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Steam Flow to Stripper Reboiler, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Syngas Flow to GT-400, M lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Fuel Gas (Natural Gas) to GT-400, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Table 3-1 (Continued)

	Test Period Average	Period 1 11/4-11/7	Period 2 11/8-11/11	Period 3 11/11-11/13
Syngas to GT-400, % of total fuel				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
Power Produced by GT-400, MW				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
GT-400 Stack C Temperature, °F				
Average				
Maximum				
Minimum				
Sample Standard Deviation				
SO_2 Emitted from GT-400, lb/hr				
Average				
Maximum				
Minimum				
Sample Standard Deviation				

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Figure 3-1
Process Parameters for Gasification System

Figure 3-2
Process Parameters for Power II

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Figure 3-3
Selectamine™ Process Parameters

Figure 3-4
Selectox™ Process Parameters

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Figure 3-5
Incinerator Process Parameters

Figure 3-6
Incinerator Process Parameters

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Figure 3-7
Sour Water Stripper Process Parameters

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4

ANALYTICAL RESULTS

The analytical results for all four test periods of the test program are presented in this section. The results have been organized by stream matrix (gaseous, solid, or liquid) and are reported as averages with a 95% confidence interval. *[Since EPA Method 29 was determined to be ineffective in the collection of trace elements in reduced gas matrices, results from Method 29 are not reported in this section, when data from alternative trace element techniques are available.]* The results reported for the organic compounds have been limited to only those compounds which were detected. Complete details of all results including individual test runs may be found in Appendix G.

Results for the analyses performed by on-line VPAAS are contained in Section 7, mercury is discussed further in Section 8.

Continuous emission monitors were run at both the turbine exhaust stack and the incinerator stack. The results for the CEMs are presented graphically in Figures 4-1 and 4-2.

The incinerator was monitored during test Period 2 for three days, November 9-11, 1994. On November 11, a leak was discovered in the sample delivery system. Efforts to detect the source of the leak were unsuccessful and, since the system was off and on all during the day, no useful data was obtained for November 11. Upon reduction of all the CEM data, it was obvious, based upon oxygen content, that the data from November 10 was also obtained with a leak in the sampling system. The bias produced by the leak appeared to be consistent throughout the day, therefore, the data from November 10 could be adjusted to account for the dilution due to ambient air. The adjusted data is presented in all appropriate tables and figures.

The analytical data have been summarized and a consistent "cell labeling" convention has been used in the tables as follows:

- ND or "<" = Not detected;
- NA = Not analyzed;
- NC = Not calculated (such as the 95% CI for ND values);
- NS = Not able to obtain a sample; and
- IS = Invalid sample (due to sampling or analytical bias).

All analyses performed on solids are reported on a dry basis.

The analytical data for all streams are presented in Tables 4-1 through 4-13 and are organized as follows:

Table	Title	Process Streams
4-1	CEM Data Summary	Turbine and Incinerator Stacks
4-2	Turbine Stack Emissions	Turbine Stack
4-3	Incinerator Stack Emissions	Incinerator Stack
4-4	Synthesis Gas Streams	Raw, Sweet, and Sour Syngas
4-5	Internal Process Streams	Sour, Acid, and Tail Gas
4-6	Incinerator Fuel Gases	Natural Gas and Combustion Air
4-7	Hot Raw Syngas Streams	Syngas at 1000°F, Syngas at 500°F
4-8	Solid Feed Streams	Raw Coal, Primary and Secondary Slurry Feed
4-9	Recycled Char Streams	Recycled Char, Dry Char (1000°F), Dry Char (500°F)
4-10	Solid Effluent Streams	Slag, Sulfur
4-11	Sour Water Stripper, Aqueous Streams	Sour Condensate, Sweet Water
4-12	Recycled Char Water	Scrubber Inlet Water, Recycle Char Filtrate
4-13	Selectamine™ Solvent	Selectamine™ Solvent

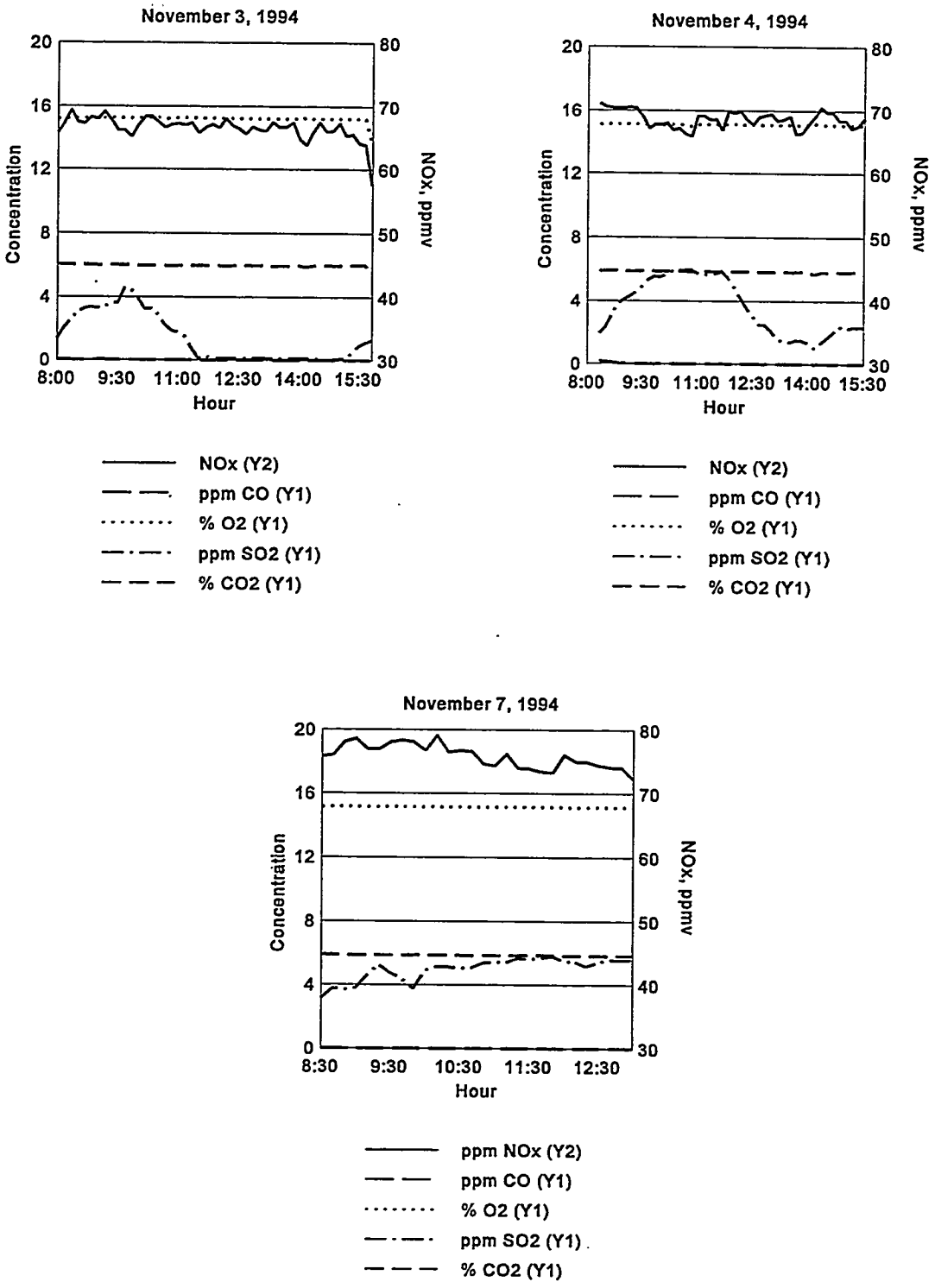

Figure 4-1
CEM Results, Turbine Exhaust Stack

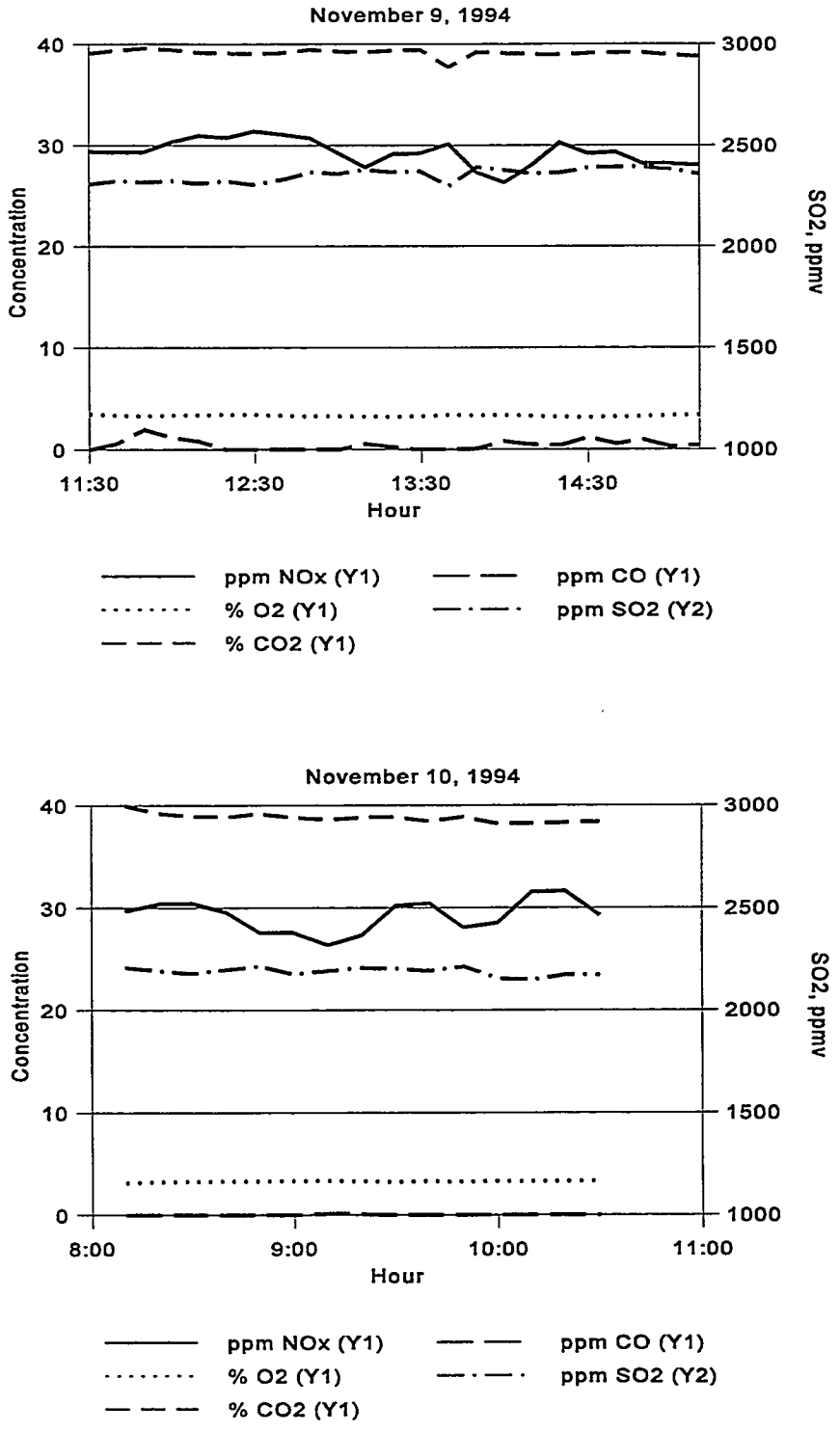

Figure 4-2
CEM Results, Incinerator Stack

Table 4-1
CEM Data Summary

	Oxygen, %	Carbon Dioxide, %	Carbon Monoxide, ppmv	Sulfur Dioxide, ppmv	Nitrogen Oxides, ppmv
Turbine Stack (13) - Reporting Periods 11/3, 11/4, 11/7					
Average	15.2	5.9	<1	3.1	70.5
Maximum	16.3	6.1	NC	6.2	80.6
Minimum	14.5	4.8	NC	<1	54.6
Standard Deviation	0.07	0.08	NC	1.9	4.9
Incinerator Stack (16) - Reporting Period 11/9					
Average	3.5	38.5	0.9	2340	28
Maximum	4.3	40.2	2.9	2480	33.4
Minimum	3.1	34.1	0	2240	20.1
Standard Deviation	0.3	0.9	0.7	50	1.8

Table 4-2
Turbine Stack Emissions

Analyte	Particulate Phase		Vapor Phase		Total[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Particulate Loading, mg/Nm³	3.86	3.5	NC	NC	NC	NC
Ionic Species, µg/Nm³						
Chloride	68	200	350	270	420	110
Fluoride	2.4	3.7	19	18	22	13
Sulfate	1,100	680	23,000	7,500	24,000	7,500
Ammonia as N	NA	NC	190	250	NC	NC
Cyanide	NA	NC	< 3.2	NC	NC	NC
Metals, µg/Nm³						
Aluminum	34	18	< 15	NC	34	18
Antimony	< 2.2	NC	< 0.022	NC	< 2.2	NC
Arsenic	1.1	1.6	0.084	0.089	1.2	1.1
Barium	1.6	1.3	0.36	0.62	2	0.75
Beryllium	< 0.012	NC	< 0.04	NC	< 0.012	NC
Boron	NA	NC	< 4.9	NC	NC	NC
Cadmium	0.62	0.035	0.99	3.1	1.6	2.2
Calcium	82	200	34	27	120	150
Chromium	1	0.61	0.47	0.57	1.5	0.38
Cobalt	0.29	0.48	0.028	0.026	0.32	0.34
Copper	7.5	15	0.77	3.2	8.3	11
Iron	74	230	8	5.6	82	160
Lead	0.99	1.5	0.6	0.92	1.6	0.90
Magnesium	10	12	< 13	NC	10	12
Manganese	0.45	1.3	1.2	5.2	1.7	3.8
Mercury, total	0.01	0.01	0.7	0.19	0.71	0.26
Molybdenum	3.8	3.3	< 0.037	NC	3.8	3.3
Nickel	0.98	0.53	1.2	2.9	2.2	2.1
Phosphorus	130	5.2	< 31	NC	130	5.2
Potassium	81	250	< 230	NC	81	250

Table 4-2 (Continued)

Analyte	Particulate Phase		Vapor Phase		Total[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Selenium	0.44	1.2	1.2	1.1	1.7	0.76
Silicon	NA	NC	40	20	NC	NC
Sodium	140	82	64	64	210	48
Titanium	2.9	6.3	0.48	0.73	3.3	4.5
Vanadium	0.37	0.19	0.1	0.21	0.47	0.13
Zinc	13	13	14	26	28	15
Aldehydes, µg/Nm³						
Acetaldehyde	NA	NC	0.99	0.86	NC	NC
Benzaldehyde	NA	NC	1.7	1.5	NC	NC
Formaldehyde	NA	NC	9.4	4.4	NC	NC
Volatile Organic Compounds, µg/Nm³						
Benzene	NA	NC	2.5	1.0	NC	NC
Carbon Disulfide	NA	NC	2.8	8.4	NC	NC
Methylene Chloride	NA	NC	55	81	NC	NC
Trichlorofluoromethane	NA	NC	26	45	NC	NC
PAHs/SVOCs, ng/Nm³						
Benzoic acid[b]	< 1,300	NC	80,000	38,000	80,000	38,000
bis(2-Ethylhexyl)phthalate[c]	4,200	18,000	< 800	NC	4,200	18,000
Di-n-butylphthalate[c]	< 400	NC	100,000	350,000	100,000	350,000

[a] Total concentration calculated only when results for both particulate and vapor phases were analyzed.

[b] Probable artifact of XAD resin.

[c] Most likely sample contamination.

Table 4-3
Incinerator Stack Emissions

Analyte	Particulate Phase		Vapor Phase		Total[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Particulate Loading, mg/Nm³	141	26	NC	NC	NC	NC
Ionic Species, µg/Nm³						
Chloride	< 150	NC	< 2,100	NC	< 2,100	NC
Fluoride	< 1.7	NC	26	22	26	22
Sulfate	140,000	19,000	11,000,000	1,700,000	12,000,000	1,700,000
Ammonia as N	NA	NC	750	1,400	NC	NC
Cyanide	NA	NC	5	7.9	NC	NC
Metals, µg/Nm³						
Aluminum	52	0.9	< 18	NC	52	0.9
Antimony	< 2.1	NC	< 0.028	NC	< 2.1	NC
Arsenic	0.18	0.41	0.51	1.2	0.69	0.90
Barium	1.7	0.98	0.11	0.21	1.8	0.71
Beryllium	< 0.012	NC	< 0.051	NC	< 0.012	NC
Boron	NA	NC	14	17	NC	NC
Cadmium	0.44	0.18	1.5	4.1	2	2.9
Calcium	42	4.1	42	48	85	34
Chromium	2.8	1.2	0.84	1.1	3.6	0.75
Cobalt	0.33	0.31	0.048	0.08	0.38	0.22
Copper	1.9	1	0.65	2.6	2.6	1.5
Iron	190	130	16	9.8	200	89
Lead	0.27	0.47	1.9	2.9	2.2	2.1
Magnesium	7.3	1.1	< 17	NC	7.3	1.1
Manganese	1.4	0.65	8.2	30	9.6	22
Mercury, total	0.015	0.018	28	2.4	28	3.7
Molybdenum	5.1	0.5	0.048	0.064	5.2	0.36
Nickel	3.1	3.1	2.1	1.4	5.2	1.8
Phosphorus	180	12	< 760	NC	180	12
Potassium	< 16	NC	< 290	NC	< 16	NC

Table 4-3 (Continued)

Analyte	Particulate Phase		Vapor Phase		Total[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Selenium	< 0.029	NC	< 0.21	NC	< 0.21	NC
Silicon	NA	NC	57	17	NC	NC
Sodium	130	150	86	120	210	87
Titanium	0.9	0.46	< 0.55	NC	0.9	0.46
Vanadium	0.55	0.12	0.74	1.9	1.3	1.3
Zinc	9.5	7.4	16	28	26	20
Aldehydes, µg/Nm³						
Acetaldehyde	NA	NC	0.65	0.95	NC	NC
Acrolein	NA	NC	< 0.59	NC	NC	NC
Benzaldehyde	NA	NC	< 0.59	NC	NC	NC
Formaldehyde	NA	NC	0.78	0.29	NC	NC
Volatile Organic Compounds, µg/Nm³						
Benzene	NA	NC	2.5	3	NC	NC
Bromomethane	NA	NC	3.8	2.3	NC	NC
Carbon Disulfide	NA	NC	5.2	1.6	NC	NC
Toluene	NA	NC	0.91	1	NC	NC
Trichlorofluoromethane	NA	NC	0.5	0.45	NC	NC
PAHs/SVOCs, ng/Nm³						
Di-n-butylphthalate[b]	4,100	18,000	31,000	29,000	35,000	18,000
Benzoic acid[c]	< 2,000	NC	81,000	20,000	81,000	20,000
bis(2-Ethylhexyl)phthalate[b]	< 600	NC	5,300	23,000	5,300	23,000

[a] Total concentration calculated only when results for both particulate and vapor phases were analyzed.

[b] Likely due to sample contamination.

[c] Probable artifact of XAD resin.

Table 4-4
Synthesis Gas Streams

Analyte	Raw Syngas (5b)		Sour Syngas (11)		Sweet Syngas (12)	
	Average	95% CI	Average	95% CI	Average	95% CI
Particulate Loading, mg/Nm³					0.0038	0.0098
Ionic Species, µg/Nm³						
Ammonia as N			3,400	2,700	310	270
Chloride			< 2,100	NC	< 2,400	NC
Cyanide			5,600	12,000	110	130
Fluoride			15	6.5	21	3.6
Metals-Vapor Phase (Charcoal), µg/Nm³						
Antimony			< 1.1	NC	< 0.039	NC
Arsenic			270	270	6	2.1
Barium			6.3	2.3	0.23	0.14
Beryllium			< 0.36	NC	< 0.013	NC
Boron			100	15	3.2	0.23
Cadmium			< 0.85	NC	< 0.031	NC
Chromium			93	14	3.6	1.7
Cobalt			< 5.9	NC	< 0.22	NC
Copper			46	10	1.8	0.18
Iron			2,300	190	85	12
Lead			< 0.85	NC	< 0.031	NC
Manganese			10	4.2	0.4	0.57
Mercury			11	13	0.099	0.024
Molybdenum			45	20	1.6	0.74
Nickel			17	24	0.94	1.5
Selenium			2.8	5.9	0.18	0.15
Vanadium			8.3	2.8	0.28	0.31
Zinc			< 3.8	NC	0.37	0.32

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Table 4-4 (Continued)

Analyte	Raw Syngas (5b)		Sour Syngas (11)		Sweet Syngas (12)	
	Average	95% CI	Average	95% CI	Average	95% CI
Aldehydes, µg/Nm³						
Acetaldehyde			9.2	0.99	140	41
Acrolein			< 0.6	NC	< 1.3	NC
Benzaldehyde			0.72	1.1	< 1.3	NC
Formaldehyde			1.6	1.6	2.3	2.3
PAHs/SVOCs-Vapor Phase, µg/Nm³						
2-Methylnaphthalene			60	21	8.9	3
Acenaphthene			110	19	< 2.8	NC
Acenaphthylene			260	19	8.4	2.6
Anthracene			8.5	0.06	< 3.5	NC
Dibenzofuran			22	7	< 2.5	NC
Fluoranthene			8	NC	< 1.2	NC
Fluorene			28	97	< 2	NC
Naphthalene			6,900	1,300	960	88
Pentachlorophenol			17	58	< 1.3	NC
Phenanthrene			55	87	< 2.6	NC
Phenol			7.6	35	< 3.7	NC
Pyrene			10	9.6	< 1.7	NC

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Table 4-4 (Continued)

Analyte	Raw Syngas (5b)		Sour Syngas (11)		Sweet Syngas (12)	
	Average	95% CI	Average	95% CI	Average	95% CI
Gas Composition						
Hydrogen (mol%)			32	4	32	8
Nitrogen (mol%)			2	2	2	0.5
Oxygen/Argon (mol%)			< 1	NC	< 1	NC
Carbon Dioxide (mol%)			31	2	30	5
Carbon Monoxide (mol%)			33	2	33	3
Methane (mol%)			2	0.1	2	0.1
C2 (ppmv)			7.8	0.1	9.2	1.2
C3 (ppmv)			0.2	0.3	0.2	0.2
C4 (ppmv)			< 0.1	NC	< 0 .1	NC
C5 (ppmv)			0.1	0.1	0.1	0.1
C6 (ppmv)			250	16	240	23
C7 (ppmv)			1	0.5	2.9	3.1
C8 (ppmv)			< 0.1	NC	< 0.1	NC
H_2S (ppmv)			920	70	27	10
COS (ppmv)			29	4	26	2
CS_2 (ppmv)			< 1	NC	< 1	NC

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Table 4-5
Internal Process Streams

Analyte	Sour Gas (22)		Acid Gas (14)		Tail Gas (15)	
	Average	95% CI	Average	95% CI	Average	95% CI
Ionic Species, µg/Nm³						
Ammonia as N	34%	11%	19,000	12,000	100,000	130,000
Chloride	NS	NC	< 5,600	NC	NA	NC
Cyanide	190,000	110,000	1,400	150	89,000	22,000
Fluoride	NS	NC	42	18	NA	NC
Metals-Vapor Phase (Charcoal), µg/Nm³						
Antimony	NS	NC	< 2.1	NC	NA	NC
Arsenic	NS	NC	4.9	7.3	NA	NC
Barium	NS	NC	12	5.4	NA	NC
Beryllium	NS	NC	< 0.69	NC	NA	NC
Boron	NS	NC	180	56	NA	NC
Cadmium	NS	NC	< 1.6	NC	NA	NC
Chromium	NS	NC	210	78	NA	NC
Cobalt	NS	NC	< 11	NC	NA	NC
Copper	NS	NC	58	20	NA	NC
Iron	NS	NC	4,000	1,600	NA	NC
Lead	NS	NC	9.6	30	NA	NC
Manganese	NS	NC	11	27	NA	NC
Mercury	NS	NC	4	1.1	NA	NC
Molybdenum	NS	NC	72	13	NA	NC
Nickel	NS	NC	25	29	NA	NC
Selenium	NS	NC	< 1.7	NC	NA	NC
Vanadium	NS	NC	9.9	8.2	NA	NC
Zinc	NS	NC	< 7.3	NC	NA	NC
Metals-Vapor Phase (M-29), µg/Nm³						
Aluminum	NS	NC	< 13	NC	< 120	NC
Antimony	NS	NC	0.062	0.043	0.072	0.23
Arsenic	NS	NC	2.7	4.9	0.4	1.2

Table 4-5 (Continued)

Analyte	Sour Gas (22)		Acid Gas (14)		Tail Gas (15)	
	Average	95% CI	Average	95% CI	Average	95% CI
Metals-Vapor Phase (M-29), µg/Nm³ (Continued)						
Barium	NS	NC	0.47	0.5	0.69	1.9
Beryllium	NS	NC	< 0.034	NC	< 0.32	NC
Boron	NS	NC	5.8	9	< 40	NC
Cadmium	NS	NC	0.41	0.26	1.7	5.6
Calcium	NS	NC	95	110	220	580
Chromium	NS	NC	65	110	27	110
Cobalt	NS	NC	1.2	1.8	6.5	25
Copper	NS	NC	15	21	3.8	9
Iron	NS	NC	140	190	34	91
Lead	NS	NC	0.66	0.71	7.8	25
Magnesium	NS	NC	21	30	< 110	NC
Manganese	NS	NC	19	45	0.76	1.9
Mercury	NS	NC	0.99	1.2	11	31
Molybdenum	NS	NC	4.6	7.2	0.61	1.7
Nickel	NS	NC	230	340	33	60
Phosphorus	NS	NC	88	120	< 5,000	NC
Potassium	NS	NC	< 200	NC	< 1,900	NC
Selenium	NS	NC	3.7	9.7	< 1.3	NC
Silicon	NS	NC	74	36	170	480
Sodium	NS	NC	78	110	270	740
Titanium	NS	NC	0.82	0.91	< 3.6	NC
Vanadium	NS	NC	0.94	2.5	0.42	1.2
Zinc	NS	NC	14	34	50	120
PAHs/SVOCs-Vapor Phase, µg/Nm³						
Acenaphthene	NS	NC	1,700	440	44	140
Acenaphthylene	NS	NC	3,100	800	< 27	NC
Anthracene	NS	NC	31	18	< 18	NC

Table 4-5 (Continued)

Analyte	Sour Gas (22)		Acid Gas (14)		Tail Gas (15)	
	Average	95% CI	Average	95% CI	Average	95% CI
PAHs/SVOCs-Vapor Phase, µg/Nm³ (Continued)						
Dibenzofuran	NS	NC	270	82	160	440
Fluorene	NS	NC	660	12	11	81
Naphthalene	NS	NC	110,000	28,000	89,000	38,000
Phenanthrene	NS	NC	240	290	< 150	NC
Gas Composition						
Hydrogen (mol%)	NA	NC	< 1	NC	NA	NC
Nitrogen (mol%)	NA	NC	< 1	NC	3	0.3
Oxygen/Argon (mol%)	NA	NC	< 1	NC	< 1	NC
Carbon Dioxide (mol%)	NA	NC	98	NC	97	0.3
Carbon Monoxide (mol%)	NA	NC	< 1	NC	< 0 .1	NC
Methane (ppmv)	700	50	420	44	390	40
C2 (ppmv)	5.4	0.5	2.3	0.6	1.3	0.5
C3 (ppmv)	170	40	8.8	2.4	6.5	4.7
C4 (ppmv)	7	4	< 0.1	NC	< 0.1	NC
C5 (ppmv)	< 0.1	NC	< 0.1	NC	< 0.1	NC
C6 (ppmv)	440	20	350	24	330	30
C7 (ppmv)	2	3	2.1	1.5	2.7	2
C8 (ppmv)	6	7	< 0.1	NC	2	4
H_2S (ppmv)	1.3%	NC	1.5%	0.2	3,000	300
COS (ppmv)	< 1	NC	170	NC	85	55
CS_2 (ppmv)	< 1	NC	< 1	NC	42	28

Table 4-6
Incinerator Fuel Gases

Analyte	Natural Gas (99)		Combustion Air (97)	
	Average	95% CI	Average	95% CI
Ionic Species, µg/Nm³				
Ammonia as N	NA	NC	460,000	55,000
Cyanide	NA	NC	10,000	2,400
Metals-Vapor Phase (M-29), µg/Nm³				
Aluminum	< 12	NC	NA	NC
Antimony	< 0.018	NC	NA	NC
Arsenic	0.068	0.031	NA	NC
Barium	0.04	0.043	NA	NC
Beryllium	< 0.029	NC	NA	NC
Boron	< 4.1	NC	NA	NC
Cadmium	0.37	0.26	NA	NC
Calcium	43	19	NA	NC
Chromium	1.5	0.22	NA	NC
Cobalt	0.54	2.2	NA	NC
Copper	< 0.047	NC	NA	NC
Iron	6	3.5	NA	NC
Lead	3.6	12	NA	NC
Magnesium	< 11	NC	NA	NC
Manganese	0.049	0.17	NA	NC
Mercury	0.35	0.16	NA	NC
Molybdenum	0.14	0.055	NA	NC
Nickel	0.74	1.6	NA	NC
Phosphorus	< 26	NC	NA	NC
Potassium	< 190	NC	NA	NC
Selenium	< 0.14	NC	NA	NC
Silicon	24	10	NA	NC
Sodium	28	12	NA	NC

Table 4-6 (Continued)

Analyte	Natural Gas (99)		Combustion Air (97)	
	Average	95% CI	Average	95% CI
Metals-Vapor Phase (M-29), µg/Nm³ (Continued)				
Titanium	< 0.33	NC	NA	NC
Vanadium	0.032	0.037	NA	NC
Zinc	8.3	2.3	NA	NC
Gas Composition				
Hydrogen (mol%)	<1	NC	NA	NC
Nitrogen (mol%)	< 1	NC	NA	NC
Oxygen/Argon (mol%)	< 1	NC	NA	NC
Carbon Dioxide (mol%)	< 1	NC	NA	NC
Carbon Monoxide (mol%)	< 1	NC	NA	NC
Methane (ppmv)	99%	NC	650	120
C2 (ppmv)	4,500	90	5	3
C3 (ppmv)	1,400	140	0.9	0.3
C4 (ppmv)	390	50	0.2	0.2
C5 (ppmv)	140	20	< 0.1	NC
C6 (ppmv)	53	12	7.2	0.3
C7 (ppmv)	110	20	1	1
C8 (ppmv)	10	1	0.6	0.05
H_2S (ppmv)	< 1	NC	< 1	NC
COS (ppmv)	< 1	NC	< 1	NC
CS_2 (ppmv)	< 1	NC	< 1	NC

Table 4-7
Hot Raw Syngas Streams

Analyte	Raw Syngas @ 1000°F (5)[a]		Raw Syngas @ 500°F (5a)	
	Average	95% CI	Average	95% CI
Ionic Species, µg/Nm ^{3}				
Ammonia as N				
Chloride				
Cyanide				
Fluoride				
Metals-Vapor Phase (Charcoal), µg/Nm ^{3}				
Antimony				
Arsenic				
Barium				
Beryllium				
Boron				
Cadmium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Manganese				
Mercury				
Molybdenum				
Nickel				
Selenium				
Vanadium				
Zinc				

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Table 4-7 (Continued)

Analyte	Raw Syngas @ 1000°F (5)[a]		Raw Syngas @ 500°F (5a)	
	Average	95% CI	Average	95% CI
Gas Composition				
Hydrogen (mol%)				
Nitrogen (mol%)				
Oxygen/Argon (mol%)				
Carbon Dioxide (mol%)				
Carbon Monoxide (mol%)				
Methane (mol%)				
C2 (ppmv)				
C3 (ppmv)				
C4 (ppmv)				
C5 (ppmv)				
C6 (ppmv)				
C7 (ppmv)				
C8 (ppmv)				
H_2S (ppmv)				
COS (ppmv)				
CS_2 (ppmv)				

[a] Samples collected with high temperature/pressure sampling probe.

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Table 4-8
Solid Feed Streams

Analyte	Raw Coal (1a)		Primary Slurry Feed (32)		Secondary Slurry Feed (33)	
	Average	95% CI	Average	95% CI	Average	95% CI
Ultimate/Proximate Parameters						
Moisture, total (Wt.%)	29	0.24	45	0.30	48	0.44
% Solids in Slurry (Wt.%)	NA	NC	55	0.30	52	0.44
Ash (Wt. %)	6.7	0.30	6.4	0.08	7.7	0.15
Carbon (Wt. %)	70	0.37	69	0.43	69	0.31
Hydrogen (Wt. %)	4.6	0.08	4.8	0.09	4.7	0.07
Nitrogen (Wt. %)	0.99	0.02	1	0.02	1.1	0.06
Sulfur (Wt. %)	0.28	0.01	0.28	0.01	0.28	0.01
Oxygen (by difference) (Wt. %)	17	0.39	19	0.34	17	0.31
Volatile Matter (Wt. %)	46	0.34	46	0.29	45	0.48
Fixed Carbon (Wt. %)	47	0.43	48	0.32	47	0.52
Higher Heating Value (Btu/lb)	12,000	68	12,000	39	12,000	46
Chloride (µg/g)	39	5.7	43	7.0	56	9.2
Fluoride (µg/g)	76	27	45	5.0	260	45
Metals, µg/g						
Aluminum	6,300	400	5,800	140	7,200	220
Antimony	0.12	0.023	0.01	0.031	0.43	0.047
Arsenic	0.98	0.066	0.74	0.13	2.2	0.32
Barium	370	97	390	8.6	500	27
Beryllium	0.27	0.029	0.21	0.04	0.31	0.039
Boron	32	0.95	26	2.4	35	0.77
Cadmium	0.1	0.035	0.074	0.016	1.8	0.87
Calcium	11,000	440	10,000	170	13,000	280
Chromium	4.7	2.1	3.3	1.4	5.2	1.9
Cobalt	1.9	0.11	1.5	0.27	2.2	0.15
Copper	11	0.79	9.6	1.5	15	1.6
Iron	2,400	98	2,300	77	2,900	84
Lead	1.3	0.20	0.85	0.16	8.3	2.2

Table 4-8 (Continued)

Analyte	Raw Coal (1a)		Primary Slurry Feed (32)		Secondary Slurry Feed (33)	
	Average	95% CI	Average	95% CI	Average	95% CI
Metals, µg/g (Continued)						
Magnesium	2,200	93	2,100	41	2,700	78
Manganese	9.9	0.62	8	1.3	11.0	0.80
Mercury	0.11	0.013	0.11	0.028	0.087	0.0086
Molybdenum	0.55	0.054	0.49	0.091	0.73	0.073
Nickel	1.6	0.56	1.8	0.51	1.1	0.37
Phosphorus	300	50	290	26	310	26
Potassium	210	26	200	50	210	26
Selenium	3.4	2.6	1.4	0.54	5	1.3
Silicon	11,000	790	9,900	230	12,000	340
Sodium	1,000	34	1,000	500	1,300	50
Strontium	200	50	200	50	200	50
Titanium	580	75	540	56	690	26
Vanadium	13	0.87	10	1.4	14	0.91
Zinc	7.9	0.81	8.6	2.5	57	25
Radionuclides, pCi/g						
Actinium-228 @ 338 KeV	0.047	0.29	NA	NC	NA	NC
Actinium-228 @ 911 KeV	0.12	0.45	NA	NC	NA	NC
Actinium-228 @ 968 KeV	0.14	0.85	NA	NC	NA	NC
Bismuth-212 @ 727 KeV	-0.27	1.5	NA	NC	NA	NC
Bismuth-214 @ 1120 KeV	0.31	0.11	NA	NC	NA	NC
Bismuth-214 @ 1764 KeV	0.32	0.61	NA	NC	NA	NC
Bismuth-214 @ 609 KeV	0.16	0.19	NA	NC	NA	NC
Lead-210 @ 46 KeV	0.4	1.7	NA	NC	NA	NC
Lead-212 @ 238 KeV	0.13	0.029	NA	NC	NA	NC
Lead-214 @ 295 KeV	0.21	0.11	NA	NC	NA	NC
Lead-214 @ 351 KeV	0.15	0.066	NA	NC	NA	NC
Potassium-40 @ 1460 KeV	-0.038	0.12	NA	NC	NA	NC

Table 4-8 (Continued)

Analyte	Raw Coal (1a)		Primary Slurry Feed (32)		Secondary Slurry Feed (33)	
	Average	95% CI	Average	95% CI	Average	95% CI
Radium-226 @ 226 KeV	0.057	0.038	NA	NC	NA	NC
Thallium-208 @ 583 KeV	0.037	0.076	NA	NC	NA	NC
Thallium-208 @ 860 KeV	-0.23	0.95	NA	NC	NA	NC
Thorium-234 @ 63 KeV	0.13	0.29	NA	NC	NA	NC
Thorium-234 @ 92 KeV	0.047	0.38	NA	NC	NA	NC
Uranium-235 @ 143 KeV	-0.023	0.17	NA	NC	NA	NC

Table 4-9
Recycled Char Streams

Analyte	Recycled Char (5c)		Dry Char (1000°F)[a]		Dry Char (500°F)[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Ultimate/Proximate Parameters						
Ash (Wt. %)						
Carbon (Wt. %)						
Hydrogen (Wt. %)						
Nitrogen (Wt. %)						
Sulfur (Wt. %)						
Oxygen (by difference) (Wt. %)						
Volatile Matter (Wt. %)						
Fixed Carbon (Wt. %)						
Higher Heating Value (Btu/lb)						
Chloride (µg/g)						
Fluoride (µg/g)						
Metals, µg/g						
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Boron						
Cadmium						
Calcium						
Chromium						
Cobalt						

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Table 4-9 (Continued)

Analyte	Recycled Char (5c)		Dry Char (1000°F)[a]		Dry Char (500°F)[a]	
	Average	95% CI	Average	95% CI	Average	95% CI
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Molybdenum						
Nickel						
Phosphorus						
Potassium						
Selenium						
Silicon						
Sodium						
Strontium						
Titanium						
Vanadium						
Zinc						

[a] Dry char samples filtered from raw syngas with hot gas sampling probe, only two samples collected at 500 degree location.

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Table 4-10
Solid Effluent Streams

Analyte	Slag (4)		Sulfur (24)	
	Average	95% CI	Average	95% CI
Ultimate/Proximate Parameters				
Ash (Wt. %)	89.8	5.1	NA	NC
Carbon (Wt. %)	9.5	5.2	NA	NC
Hydrogen (Wt. %)	0.15	0.07	NA	NC
Nitrogen (Wt. %)	0.04	0.05	NA	NC
Sulfur (Wt. %)	0.03	0.02	98.7	7.0
Oxygen (by difference) (Wt. %)	0.27	0.19	NA	NC
Volatile Matter (Wt. %)	NA	NC	NA	NC
Fixed Carbon (Wt. %)	NA	NC	NA	NC
Higher Heating Value (Btu/lb)	NA	NC	NA	NC
Chloride (µg/g)	84	56	NA	NC
Fluoride (µg/g)	200	50	NA	NC
Metals, µg/g				
Aluminum	91,000	5,300	16	150
Antimony	1.07	0.25	<3	NC
Arsenic	6	0.96	<3	NC
Barium	5,900	390	<2	NC
Beryllium	3.4	0.37	<2	NC
Boron	350	30	<10	NC
Cadmium	0.20	0.11	<2	NC
Calcium	160,000	9,800	20	95
Chromium	76	8.3	4	38
Cobalt	26	2.8	<4	NC
Copper	150	11	<2	NC
Iron	37,000	2,200	9	38
Lead	3	1.1	<3	NC

Table 4-10 (Continued)

Analyte	Slag (4)		Sulfur (24)	
	Average	95% CI	Average	95% CI
Magnesium	33,000	2,200	4	25
Manganese	130	12	<2	NC
Mercury	0.020	0.006	0.095	0.19
Molybdenum	7.6	0.52	<20	NC
Nickel	38	4.1	<4	NC
Phosphorus	4,100	210	NA	NC
Potassium	2,700	250	<20	NC
Selenium	14	5.7	24	180
Silicon	160,000	9,500	<20	NC
Sodium	16,000	1,100	<20	NC
Strontium	2,300	910	NA	NC
Titanium	8,100	500	2	13
Vanadium	170	13	<2	NC
Zinc	47	5.6	15	170
Radionuclides, pCi/g				
Actinium-228 @ 338 KeV	2.3	0.9	NA	NC
Actinium-228 @ 911 KeV	2.5	1.1	NA	NC
Actinium-228 @ 968 KeV	2.5	1.1	NA	NC
Bismuth-212 @ 727 KeV	2.8	1.1	NA	NC
Bismuth-214 @ 1120 KeV	2.7	0.9	NA	NC
Bismuth-214 @ 1764 KeV	2.6	1.4	NA	NC
Bismuth-214 @ 609 KeV	2.7	1.2	NA	NC
Lead-210 @ 46 KeV	0.33	1.4	NA	NC
Lead-212 @ 238 KeV	2.3	0.87	NA	NC
Lead-214 @ 295 KeV	2.8	1.2	NA	NC
Lead-214 @ 351 KeV	2.8	1.2	NA	NC
Potassium-40 @ 1460 KeV	2	1.8	NA	NC
Radium-226 @ 226 KeV	3.5	1.3	NA	NC

Table 4-10 (Continued)

Analyte	Slag (4)		Sulfur (24)	
	Average	95% CI	Average	95% CI
Thallium-208 @ 583 KeV	0.82	0.34	NA	NC
Thallium-208 @ 860 KeV	1.1	0.52	NA	NC
Thorium-234 @ 63 KeV	1.9	2.3	NA	NC
Thorium-234 @ 92 KeV	1.4	0.76	NA	NC
Uranium-235 @ 143 KeV	0	0.56	NA	NC

Table 4-11
Sour Water Stripper Aqueous Streams

Analyte	Sour Condensate (7)		Sweet Water (8)	
	Average	95% CI	Average	95% CI
Water Quality Parameters				
pH			8.75	0.22
Specific conductance (µmhos)			71.8	18
Total Suspended Solids (mg/L)			1.9	3.4
Chemical Oxygen Demand (mg/L)			53	3.9
Total phenolics (mg/L)			0.55	0.1
Ionic Species, mg/L				
Ammonia as N			7.3	3.6
Chloride			0.88	0.15
Cyanide, amenable			0.035	0.1
Cyanide, total			1.5	1.2
Fluoride			1.8	0.7
Formate			3.2	0.41
Phosphate, total (as P)			0.26	0.1
Sulfate			< 0.047	NC
Thiocyanate			0.82	0.72
Metals, mg/L				
Aluminum			0.5	0.14
Antimony			< 0.076	NC
Arsenic			0.0038	0.0024
Barium			0.53	0.07
Beryllium			0.0006	0.0013
Boron			0.039	0.051
Cadmium			0.005	0.0024
Calcium			2.6	0.11
Chromium			0.0087	0.003
Cobalt			< 0 .004	NC
Copper			0.015	0.0044
Iron			1.2	0.14
Lead			0.33	0.25
Magnesium			1	0.058

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Table 4-11 (Continued)

Analyte	Sour Condensate (7)		Sweet Water (8)	
	Average	95% CI	Average	95% CI
Manganese			0.0024	0.0034
Mercury			< 0.000033	NC
Molybdenum			0.011	0.0051
Nickel			0.022	0.042
Phosphorus			0.24	0.07
Potassium			0.9	1.1
Selenium			0.032	0.02
Silicon			7.23	0.35
Sodium			3.79	0.038
Titanium			0.02	0.011
Vanadium			< 0.0045	NC
Zinc			0.25	0.13
Aldehydes, mg/L				
Acetaldehyde			< 0.01	NC
Acrolein			< 0.01	NC
Benzaldehyde			< 0.01	NC
Formaldehyde			< 0.01	NC
Volatile Organic Compounds, µg/L				
1,4-Bromofluorobenzene			46	1.2
Acetone			5.2	3.2
Benzene			< 0.46	NC
Methylene chloride			< 3	NC
Semivolatile Organic Compounds, µg/L				
2,4,6-Tribromophenol			160	32
2-Fluorobiphenyl			61	15
2-Fluorophenol			160	33
4-Methylphenol/3-Methylphenol			0.49	1.1
Benzoic acid			9.2	26
Fluoranthene			2.6	1.1
Phenol			400	84
Pyrene			11	5.6

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Table 4-12
Recycled Char Water

Analyte	Scrubber Inlet Water		Recycle Char Filtrate	
	Average	95% CI	Average	95% CI
Water Quality Parameters				
Total Suspended Solids (mg/L)				
Ionic Species (mg/L)				
Ammonia as N				
Chloride				
Cyanide, amenable				
Cyanide, total				
Fluoride				
Thiocyanate				
Metals, mg/L				
Aluminum				
Antimony				
Arsenic				
Barium				
Beryllium				
Boron				
Cadmium				
Calcium				
Chromium				
Cobalt				
Copper				
Iron				
Lead				
Magnesium				

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Table 4-12 (Continued)

Analyte	Scrubber Inlet Water		Recycle Char Filtrate	
	Average	95% CI	Average	95% CI
Manganese				
Mercury				
Molybdenum				
Nickel				
Phosphorus				
Potassium				
Selenium				
Silicon				
Sodium				
Titanium				
Vanadium				
Zinc				

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Table 4-13
Selectamine™ Solvent

Analyte	Units	Pre-Test Sample Average	Post-Test Sample Average[a]
Ash	Wt.%		
Density	g/cc		
Heat Stable Salts	Wt.%		
Total Suspended Solids	mg/L		

[a] The solvent was regenerated during the test program.

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DATA EVALUATION AND QUALITY CONTROL

The measurement data obtained from this project were subjected to numerous quality control (QC) checks designed to measure the completeness, representativeness, and comparability of the results. The results of these QC checks were evaluated and compared to the data quality objectives (DQOs) established at the onset of this project for precision, accuracy, and representativeness of individual sample measurements. Quality control checks that fail to meet the DQOs do not necessarily render the data unacceptable; however, they may affect the representativeness and comparability of the results reported. It is not intended that DQOs be used as acceptance criteria, but rather as empirical estimates of the precision and accuracy expected from existing reference measurement methods considered acceptable for the task of providing meaningful results. This section presents the precision and accuracy actually obtained, with the DQOs serving as benchmarks for comparison.

A comprehensive listing of all individual quality control sample results (and data quality objectives) are presented in Appendix A. Results for blank samples, matrix-spike and surrogate-spike recoveries, and performance evaluation standards are presented. In reference to these results, this section:

- Provides a performance assessment of the analytical systems for each matrix and analyte and the rationale for reporting data from specific sampling and analytical methods;
- Discusses sample representativeness and identifies any potentially affected results;
- Describes the conventions for handling analytical data; and
- Presents the results of material balances which are used to assess the overall representativeness of the data.

Quality Control Results

Insuring the quality of the analytical data is key to the design of a quality control system. Numerous quality control checks were used throughout sample preparation and analysis to indicate the accuracy and precision of the handling procedures and analytical instrumentation. Table 5-1 defines the various QC measures performed.

Quality control measurements internal to the laboratory were made to confirm the accurate operation of the analytical instrumentation and to verify the absence of any significant source of contamination. Specifically, laboratory control standards (LCS) were used to confirm that the

Table 5-1
Quality Control Checks

QC Check	Description	Purpose
Laboratory Check Standard/ Laboratory Check Standard Duplicate (LCS/LCSD)	Standard samples prepared in a "clean" matrix from sources other than the calibration solutions.	Results are used to measure the accuracy and precision of the analytical system after calibration and at regular intervals during analysis of samples. LCS/LCSD results must meet control limits before analysis of samples can proceed.
Performance Evaluation Audit Sample (PE Sample)	Standard Reference Material (SRM), well-characterized reference sample, or standard sample prepared in a similar matrix to that of the samples. Submitted to the lab double-blind (i.e. unknown to the lab in concentration and not identified as a QC sample).	Enables an independent assessment of the laboratory's ability to provide accurate results in a matrix similar to that of the samples.
Matrix-Spiked Sample/ Matrix-Spiked Duplicate (MS/MSD)	Real samples spiked with the analyte(s) of interest, usually before sample preparation.	Spike recovery between MS/MSD samples indicates accuracy and precision of the preparation and analytical method for the spiked sample matrix. Poor recovery may indicate matrix effects, loss during sample prep, or disparity between the spiked and parent sample concentration.
Analytical Spiked Sample/ Analytical Spiked Duplicate (AS/ASD)	Real samples spiked with the analyte(s) of interest after sample preparation, immediately before analysis.	Similar to MS/MSD, but spike recovery between AS/ASD samples only indicates accuracy and precision of the analytical method for the spiked sample matrix. Poor recovery may indicate matrix effects or disparity between the spiked and parent sample concentration.
Surrogate Spiked Samples	Isotopically-labeled or substituted compounds similar to the compound(s) of interest which are added to the sample matrix when matrix spiking is not feasible.	Results indicate accuracy of the analytical system for a limited number of analytes which are related to the surrogate compounds.
Method Blanks	Sample of all reagents used in sample preparation and carried through all sample preparation steps.	Method blanks are used to indicate potential contamination from sample preparation and/or reagent impurities.
Media Blanks	Samples of prepared, unused collection media (i.e. filters, impinger solutions, sorbents, etc.)	Analysis is used to indicate the background levels of analytes present in the media used for sample collection.
Field Blanks	Samples of collection media that have been prepared and recovered like actual samples, but with no sample contact.	Analysis indicates the levels of analytes present from media background and that potentially introduced from sample handling.
Duplicate Samples	Samples from the same source collected simultaneously.	Results indicate total variability (precision) between samples due to sample collection, handling, preparation, and analysis.
Analytical Duplicates	Duplicate analysis of the same sample.	Indicates analytical precision only.

analytical instrumentation was operating within specified control parameters. Method blanks were used to indicate background levels of analytes in the materials used in the preparation of samples. Failure to meet the method criteria for LCS recovery and method blank levels would result in reanalysis, repreparation of samples, or both until the system was found to be in control. During this program, there were no LCS results reported outside of method-specified control range indicating that all analytical systems were performing within method-specified limits for accuracy and precision.

Matrix-spiked and matrix-spiked duplicate (MS/MSD) samples were analyzed to determine accuracy and precision of the analytical techniques. Wherever practical, matrix-spiked samples were prepared for each matrix to provide an indication of an analytical technique's accuracy. For analyses where the entire sample was required, analytical spikes (spikes added to the final prepared sample) were substituted for matrix spikes. In addition to matrix-spiked samples, or for analytes where matrix-spiking was not practical, surrogate-spiked samples, standard reference materials, and/or performance evaluation audit samples were analyzed to provide an additional measurement of accuracy. In addition to MS/MSD samples, duplicate analyses were used to measure analytical precision.

This section continues with a discussion on the significance of blank sample results and on the accuracy and precision of each method used to measure each group of target analytes (i.e., metals, anions, volatile organics, etc.) relative to each sample matrix. The sample preparation and analytical techniques summarized in Tables 5-2, 5-3, and 5-4 were assessed for data quality of the gas, solid, and aqueous sample matrices, respectively.

Metals—Solid Streams and Gas Particulate Samples

The analytical techniques used for metals analysis of coal, slurry, slag, char, sulfur, and filtered gas particulate samples include inductively coupled plasma emission spectroscopy (ICP-AES), atomic absorption spectrophotometry [i.e. graphite furnace (GFAAS) and cold vapor (CVAAS)], and x-ray fluorescence spectrophotometry (XRF). Due to the low concentrations of many trace elements found in these matrices, the more sensitive inductively coupled plasma mass spectrometry (ICP/MS) technique was used for all samples except sulfur and gas particulate filters. All of these analytical techniques require a sample digestion or fusion step to prepare the solid sample for analysis, so similar QC samples were prepared for these matrices.

With the exception of the solid digestate analyses for nickel by ICP/MS, analysis results for method blank samples showed no evidence of significant contamination from either laboratory or field handling, respectively. Nickel contamination can be explained since the skimmer cones used to sample the plasma in an ICP/MS are nickel-plated to withstand the high temperatures of the plasma. High nickel concentrations in the blanks (and high matrix spike recoveries) suggest some corrosion of the cones by the sample matrix was taking place, therefore the nickel values measured by ICP-AES were used.

Table 5-2
Summary of Analytical Methods for Gas Samples

Sample Matrix	Sampling Train	Analyte	Sample Preparation and Analytical Method
Filter and probe rinse solids [a]	EPA Method 29 (draft)	Al, Sb, Ba, Be, Ca, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, P, K, Si, Na, Ti, V, & Zn.	Mixed-acid microwave digestion/ ICP-AES (SW6010)
		As, Cd, Pb, & Se.	Mixed-acid microwave digestion/ GFAAS (SW7060,7131,7421,7740)
		Hg	Mixed-acid microwave digestion/ CVAAS (SW7470)
HNO_3/H_2O_2 impinger solutions	EPA Method 29 (draft)	Al, Sb, Ba, Be, B, Ca, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, P, K, Si, Na, Ti, V, & Zn.	Digestion (SW3005)/ICP-AES (SW6010)
		As, Cd, Pb, & Se.	Digestion (SW3020)/GFAAS (SW7060,7131,7421,7740)
		Hg	Peroxide reduction/CVAAS (SW7470)
		Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, & V.	ICP/MS (SW6020)
4% $KMnO_4$/10% H_2SO_4 impinger solution [a]	EPA Method 29 (draft)	Hg	CVAAS (SW7470)
Charcoal Sorbent [b]	Radian	Al, Sb, Ba, Be, Ca, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, P, K, Si, Na, Ti, V, & Zn.	Nitric acid microwave digestion/ ICP-AES (SW6010)
		As, Cd, Pb, & Se.	Nitric acid microwave digestion/ GFAAS (SW7060,7131,7421,7740)
		Hg	Nitric acid microwave digestion/ CVAAS (SW7470)
H_2SO_4 impinger solutions	EPA Method 26 (modified)	Ammonia	Colorimetric (EPA Method 350.2, 350.1)
		Chloride	Ion Chromatography (EPA Method 26)
		Fluoride	Specific Ion Electrode (EPA Method 340.2)
2% $Zn(C_2H_3O_2)_2$ impinger solution	Texas Air Control Board	Total Cyanide	Colorimetric (EPA Method 335.2)

Table 5-2 (Continued)

Sample Matrix	Sampling Train	Analyte	Sample Preparation and Analytical Method
Filter and FH rinse [a]	EPA Method 0010	SVOCs/PAHs	GC/MS (SW8270) [a] HRGC/MS (CARB 429) [a]
XAD, condensate, and BH rinse		SVOCs/PAHs	GC/MS (SW8270) HRGC/MS (CARB 429) [a]
2,4-DNPH impinger solution	EPA Method 0011	Aldehydes	HPLC (EPA Method 0011)
VOST [a]	EPA Method 0030	Volatile organic compounds	GC/MS (SW8240)
4% $KMnO_4$/10% NaOH impinger solution [a]	EPA Method 7D	NO_x	Ion Chromatography

[a] Turbine stack and incinerator flue gas samples only.

[b] Internal process streams only.

Table 5-3
Summary of Analytical Methods for Solid Streams

Matrix	Sample Handling	Analyte	Sample Preparation and Analytical Method
Coal/Char/Slag	Composite samples are air-dried and ground to pass a 60-mesh sieve.	Carbon, Hydrogen, Nitrogen	ASTM D5373
		Sulfur	ASTM D4239
		Ash	ASTM D3174
		Volatile Matter [a]	ASTM D3175
		Fixed Carbon [a]	ASTM D3172
		HHV [a]	ASTM D2015
		Chlorine (as Cl^-)	ASTM D4208 (adapted for IC analysis) [a] HNO_3 acid leach/potentiometry [b]
		Fluorine (as F^-)	ASTM D3761/SIE [a] NaOH fusion/SIE [b] ASTM D4208 (adapted for IC analysis) [a]
		Major ash minerals: Al, Ca, Fe, Mg, P, K, Si, Na, & Ti.	ASTM D4326 (XRF)
		Ba, Ni, & Zn.	ASTM D3683/ICP-AES
		Sb, As, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Se, & V.	ASTM D3683, mixed-acid microwave digestion (HF, HCl, HNO_3), and EPA SW3020. Analysis by ICP/MS.
		Boron	Na_2CO_3 fusion/ICP-AES
		Mercury	Double gold amalgamation/CVAAS
Sulfur	Grab samples were ground and mixed.	Sulfur	ASTM D4239
		Ash	ASTM D3174
		Sb, Ba, Be, Cr, Co, Cu, Mn, Mo, Ni, & V.	ASTM D3683/ICP-AES
		As, Cd, Pb, & Se	ASTM D3683/GFAAS
		Boron	Na_2CO_3 fusion/ICP-AES
		Mercury	Double gold amalgamation/CVAAS

[a] Coal and char samples only.

[b] Slag samples only.

Table 5-4
Summary of Analytical Methods for Liquid Process Streams

Process Stream	Sample Handling	Analyte	Sample Preparation and Analytical Method
Sour Condensate, Sweet Water, Scrubber Inlet Water, and Recycle Char Filtrate	On-site analysis.	pH	EPA 150.1
		Specific conductance	EPA 120.1
		Total Suspended Solids (TSS)	EPA 160.2
		Sulfide	Orion SIE
	Composite samples cooled to 4° C. Filtered prior to analysis.	Fluoride	EPA 340.2
		Chloride, Sulfate	EPA 300.0
		Formate	EPA 300.0 (modified)
		Phosphate	EPA 365.1
	Grab samples treated with $PbCO_3$, filtered, and pH adjusted >12 with CaO.	Total Cyanide	EPA 335.2
		Free Cyanide	EPA 335.1
		Thiocyanate	SM 412K
	Composite samples treated with H_2SO_4 to pH<2.	Ammonia	EPA 350.2, 350.1
		COD	EPA 410.1
		Phenol	EPA 420.1
	Composite samples treated with HNO_3 to pH<2.	Al, Sb, Ba, Be, B, Ca, Cr, Co, Cu, Fe, Mg, Mn, Mo, Ni, P, K, Si, Na, Ti, V, & Zn.	SW3005/SW6010
		As, Cd, Pb, & Se	SW3020/SW7060, 7131, 7421, 7740
		Mercury	SW7470
	Grab samples cooled to 4° C.	Semivolatile Organic Compounds	SW8270
		Aldehydes	SW8315 (proposed)
		Volatile Organic Compounds	SW8240
Selectamine™ Solvent	Grab samples stored in amber glass bottles at room temperature.	Heat Stable Salts	Union Carbide - Titration with NaOH
		Ash	ASTM D3174
		Total Suspended Solids (TSS)	EPA 160.2

Any metal concentrations in gas-filter media blank samples would be significant considering the small amount of particulate material collected for analysis. For these samples, multiple blanks were analyzed and the results averaged. This provided a value for correcting the sample results for background concentrations associated with the collection media. This correction was necessary to eliminate the reporting of extremely high or extremely low concentrations that could not be statistically differentiated from background concentrations in the filter media. A blank filter containing approximately 0.1 gram of a standard fly ash (NBS 1633a Ref. Filter Q-2608) was analyzed as a performance evaluation sample. The results reported in Appendix Table A-1 reflect background corrected results. Recovery of the ash standard was within 75-125% for all target metals except copper (68.6%), lead (51.0%), and molybdenum (48.3%).

Matrix spiked sample results for metals indicate digestion and analytical performance within specified recovery objectives with few consistent exceptions. In cases where two or more analytical techniques were performed (e.g., ICP-AES and ICP/MS), the technique offering the best QC sample performance and/or best detection limit was selected for the reported results. Table 5-5 identifies those metals analyzed by selected analytical techniques that did not meet the DQOs in at least 2 MS samples for the respective sample streams. These results may indicate matrix effects or uncertainty in the data for a specific process stream. The material balances presented at the end of this section can be used to assess the overall believability of the data and the effect of a potential analytical bias indicated by poor spike recovery results.

From the data presented in Tables 5-5 and Appendix A-3, the following key observations can be made:

- Lead results may be biased low in coal, slurry, char, slag, and incinerator stack gas particulate samples based on consistently low spike recovery and standard sample results;

- Copper results for coal and slag samples may be biased slightly low, although excellent spike recovery results were obtained for char, secondary slurry feed, and emitted particulate matter;

- Arsenic and cobalt results by ICP/MS in coal, slurry, and char may be biased slightly high based on spike recovery results; and

- Manganese, selenium, and vanadium results are mixed and variable, based upon the MS/MSD and PE results.

Performance evaluation standards (coal SRMs) and reference samples (well characterized slag and coal samples) were also analyzed to provide an indication of analytical accuracy for major ash minerals by XRF. These results, presented in Appendix Table A-1, do not indicate any consistent analytical bias for any of the target metals. However, individual results outside the DQOs were reported for calcium, titanium, magnesium, sodium, potassium, silicon, and phosphorus.

Table 5-5
Metal Spike Recoveries Outside DQOs—Solid Streams

Matrix/Sample Stream	Metal	Analysis Method	MS/MSD Recovery	Additional Information
Raw Coal/Slurry/Char	Arsenic	ICP/MS	130%, 132%, 131%, 131%, 137%	PE Standard: 61.8%, 90.8%
	Cobalt	ICP/MS	130%, 129%, 129%, 129%	PE Standard: 98.3%, 126%
	Copper	ICP/MS	25%, 46%	PE Standard: 86.8%, 72.9%
	Lead	ICP/MS	31%, 68%, 41%, 27%, 69%, 49%, 63%	PE Standard: 31.5%, 32.8%
	Manganese	ICP/MS	24%, 54%	PE Standard: 105%, 141%
	Selenium	ICP/MS	-64%, -21%, 129%	PE Standard: 53.5%, 105%
	Vanadium	ICP/MS	17%, 43%	PE Standard: 68.0%, 87.6%
Slag	Copper	ICP/MS	62%, 74%	PE Reference Sample: 72.1%
	Lead	ICP/MS	74%, 69%	PE Reference Sample: 72.7%
Gas-Particulate Phase, Incinerator Stack Only	Lead	GFAAS	59%, 62%	PE Sample: 51.0%
	Selenium	GFAAS	18%, 18%	PE Sample: 103%

Metals-Aqueous Streams, Impinger Solutions, and Charcoal Sorbent Extracts

Metal concentrations in media blank and method blank samples were considered insignificant for all aqueous/impinger/extract samples with the exception of those collected on charcoal sorbents (vapor-phase metals) and impinger samples analyzed by ICP/MS. For these two sample sets, at least three blank samples were analyzed and the blank results were averaged to provide a representative value for correcting the sample results for background concentrations associated with the collection media. Similar to the situation with filtered gas-particulate samples, this correction was necessary to eliminate the reporting of extremely high or extremely low concentrations that could not be statistically differentiated from background concentrations in the collection media.

Matrix-spiked impinger samples (HNO_3/H_2O_2) analyzed for metals by ICP/MS were prepared at 10-20 parts per billion (ppb); concentrations much closer to the natural concentrations found in these samples and below a reasonable spiking level for ICP-AES. Spike recoveries for all of the ICP/MS elements (see Appendix Table C-1) were between 87%-124% at low ppb levels for the turbine/incinerator stack matrix and syngas matrix. Low spike recoveries were obtained for chromium (59%), cobalt (73%), and nickel (70%) in the matrix-spiked tail gas sample, one of the more complex gas matrices; however due to the low concentrations present in the gas streams, these potential biases are not considered significant. No digestion step was applied to the HNO_3/H_2O_2 samples analyzed by ICP/MS which should be advantageous in preventing losses from sample handling and volatilization from the pre-digestion step prescribed in EPA Method 29 for ICP-AES and GFAAS analyses.

Highly variable spike recovery results were obtained for mercury by ICP/MS. This is consistent with previous air-toxics assessment projects where attempts to qualify mercury by this technique have met with limited success. For this reason, CVAAS results are reported for mercury in impinger solutions. Mercury spikes were easily recovered by CVAAS from the HNO_3/H_2O_2 solutions (93%-108%). Potassium permanganate impinger solutions spiked with mercury were recovered by CVAAS with less precision (63%, 80%, 74%, and 86%).

The remaining metals not analyzed by ICP/MS were analyzed by ICP-AES. No spike recoveries outside the DQOs were obtained except for barium in the spiked tail gas (55%-64%). Gas impinger samples that contain high levels of sulfur dioxide may potentially yield a low analytical bias for barium.

Samples from each process water matrix, sweet water, sour condensate, and recycled char filtrate, were spiked prior to digestion and analyzed for metals by ICP-AES, GFAAS, and CVAAS. Spike recoveries for all metals met the DQOs in each matrix except for boron (sweet water and recycle char filtrate), lead (sweet water and recycle char filtrate), sodium (recycle char filtrate), and mercury (recycle char filtrate) which exhibited low spike recovery results. The frequency of poor spike recoveries for lead and mercury was greatest in the recycle char filtrate samples, indicating a possible matrix effect.

Analytical spikes (rather than matrix spikes) were performed on the charcoal sorbent digestates since the entire sample was digested for analysis. A blank set of charcoal tubes was spiked before digestion to check retention and recovery through the digestion process. All analytical spikes were recovered within the DQOs except for boron (74%), cobalt (72%), copper (74%), nickel (74%,70%), and zinc (68%, 71%, 73%). Matrix-spiked blank tubes demonstrated recovery for all elements except antimony (10%,14%), molybdenum (42%,46%), and mercury (52%,62%). Results for these elements by charcoal adsorption may be biased low if this is an accurate representation of digestion recovery.

Aqueous and impinger solution audit samples were also prepared for a limited set of target analytes (Al, Sb, Ba, Be, Cd, Cu, Mn, and Mo) as an additional indication of analytical accuracy. By the analytical techniques selected, audit sample recovery was within the specified DQOs for

these elements spiked in the HNO_3/H_2O_2 impinger audit sample except manganese (134%). Recovery objectives for the aqueous audit sample were met for all of the selected elements except aluminum (73.2%), antimony (37.8%), and molybdenum (45.3%). Results for these elements in their respective matrices may be biased accordingly.

In summary, significant analytical bias may be indicated by poor matrix spike recovery and audit sample results for the metals in the matrices identified below:

- Chromium and barium in the tail gas appear to be biased low (this result may be present in acid gas samples as well);
- Lead, antimony, and molybdenum concentrations measured in process water streams may be biased low; and
- Incomplete recovery of antimony, molybdenum, and mercury from the charcoal sorbent samples (vapor-phase metals by charcoal) may be indicated by low recovery results for pre-digestion spikes onto blank charcoal.

Anions—Solid Streams and Gas Particulate Samples

Chloride and fluoride were measured in coal, slurry, char, and sulfur matrices prepared by combustion in an oxygen combustion bomb and analyzed by ion chromatography (IC) or specific ion electrode (SIE-sulfur only). Slag samples, since they are not combustible, were leached with a dilute nitric acid solution for chloride analysis and were fused with sodium hydroxide (NaOH) and dissolved for fluoride analysis. Gas particulate samples were leached with a carbonate/ bicarbonate solution for subsequent analysis by IC (Cl^- and $SO_4^=$) and SIE (F^-).

Method blanks prepared for chloride and fluoride by oxygen bomb/IC were free of significant detectable concentrations. However, method blank samples prepared for fluoride by NaOH fusion exhibited detectable concentrations of fluoride that were approximately equal to 10% of the actual slag sample results. No blank corrections were made to the reported slag fluoride results.

A comparison of method blank, reagent blank, and field blank samples analyzed for gas particulate-phase anions indicated varying levels of contamination either associated with the filter and/or filter leaching media or potentially introduced during field sample handling. The chloride results for the turbine stack media and field blanks account for approximately 10-25% of the average chloride concentration reported. Fluoride was detected near the detection limit in all blank samples at approximately the same concentrations found in the turbine and incinerator stack samples. No blank corrections were made, so the reported emissions for chloride and fluoride in gas particulate matter may be overestimated. Sulfate concentrations in the field blank filter samples collected at the incinerator stack and turbine stack indicate varying levels of sample media contamination. Relative to the high concentrations of sulfate detected in the incinerator stack gas particulate samples, these blank results are not significant. However, the

sulfate results for the gas particulate emissions from the turbine stack were lower and may therefore be overestimated if the blank concentrations are representative of the sampling media.

Matrix spikes and audit samples were analyzed to measure the accuracy of the preparation and analysis methods. Recovery of filtered gas particulate matrix spikes were 89%-100% for chloride, fluoride, and sulfate. Coal and char samples prepared by oxygen bomb and analyzed by IC were spiked with chloride and fluoride. Recovery of chloride met the DQOs for all coal matrices and char. Fluoride spikes in char were outside the DQOs, however the spiked concentration was extremely low relative to the high sample concentration. Low fluoride spike recovery was also experienced for slag samples prepared by NaOH fusion. A sodium fluoride salt solution is spiked into the NaOH solution used to fuse the slag in a muffle furnace. Poor fluoride spike recovery may indicate incomplete retention of fluoride during the fusion step, or a matrix effect related to slag metals that interfere with the performance of the fluoride specific electrode.

The coal audit SRM (AR 2780) analyzed for chloride and fluoride reported chloride at 83.3%, and fluoride at 23.4% of the reference concentration value. Chloride and fluoride results for a coal round robin sample reported chloride and fluoride at 7.8% and 73.1%, respectively. The characterization of the round robin sample was highly variable, indicating the uncertainty associated with low level chloride and fluoride measurements in coal.

Significant quality control results affecting the analysis of anionic species in the solid sample matrices are summarized for the following sample streams:

- Chloride, fluoride, and sulfate were detected at various concentrations in blank samples of the gas particulate filters; emissions data for particulate-phase anions may be overestimated;
- Fluoride results for slag may be biased low based on poor matrix-spike recovery results; and
- Chloride and fluoride concentrations in coal and slurry are low and prone to analytical uncertainty and imprecision as indicated by spike and audit sample results.

Anions—Aqueous Streams and Impinger Solutions

Anionic species in this section include chloride, fluoride, formate (aqueous stream samples only), nitrate (Gas impinger Method 7d - NO_x), and sulfate. Only fluoride and sulfate were detected in method blanks, reagent blanks, and field blanks. Fluoride was detected between 0.0178 mg/L in the method blanks to 0.0318 mg/L in the H_2SO_4 impinger solutions used to collect HF and HCl from the gas streams. These levels are not significant with respect to the measured sample concentrations. Sulfate detected in the reagent and field blanks collected for the Method 8 sampling train indicate minor sulfate contamination in both field blanks (IPA and H_2O_2 impinger solutions). The blank concentrations were not significant relative to the H_2SO_4 and SO_2 concentrations measured in the turbine and incinerator stack gas samples.

Thiocyanate matrix spikes in one sweet water sample were recovered at 128% and 158% indicating a potentially high analytical bias.

The audit samples for ammonia (prepared in H_2SO_4 impinger solution) and cyanide (prepared in zinc acetate solution) were recovered at 94.5% and 122%, respectively. The aqueous ammonia audit sample was recovered at 87.2 percent. Cyanide audit samples were prepared with both free and complexed forms of cyanide in order to assess the laboratory's ability to differentiate both total and free cyanide as required for the process water samples. Sodium cyanide salts were used as the free cyanide source, and iron ferricyanide was used as the complexed cyanide source. Total cyanide (free + complexed) was recovered at 83.1 percent. Free cyanide alone was recovered at 104 percent. Thiocyanate, also spiked in the audit standard, was 100% recovered.

The analytical techniques for ammonia and cyanide are very sensitive, and based on the spike recovery and audit sample results, fairly accurate. The cyanide analysis methods for total and free cyanide adequately demonstrated the ability to differentiate the multiple forms of cyanide in the process water samples. No outstanding analytical problems were noted except for the possible high bias indicated for thiocyanate in process water stream samples.

Ultimate/Proximate Analysis—Solid Streams

Three reference materials were analyzed to check the methods' accuracy for the ultimate/ proximate analysis parameters: carbon, hydrogen, nitrogen, sulfur, ash, volatile matter, fixed carbon, and higher heating value. A standard reference coal (Alpha Resources AR 2780) and a well-characterized "round robin" coal sample (Powder River Basin subbituminous) were submitted as blind audit coal-matrix samples. A well-characterized sample of gasifier slag was also submitted as an audit sample for the slag matrix. Only the SRM audit sample has certified reference values for measuring the accuracy of the ultimate/proximate analyses. These audit samples were selected since their matrix-analyte concentrations were similar to the samples being analyzed.

All ultimate/proximate results for the AR 2780 SRM sample met the data quality objectives (80%-120% recovery of the reference value). The results for the round robin coal sample also met the data quality objectives. Slag audit sample results for ash (99.9%), sulfur (109%), and carbon (80.6%) met the data quality objectives while hydrogen (167%) and nitrogen (not detected) did not. The slag audit sample was 98% ash—hydrogen and nitrogen concentrations were very low and close to the practical quantitation limits of the methods used.

Water Quality Parameters—Aqueous Streams

The water quality parameters determined in the process water streams were pH, conductivity, total suspended solids (TSS), chemical oxygen demand (COD), and total phenolics. Conductivity, pH, and TSS were determined on site and with the exception of pH and conductivity meter calibrations to verify proper operation, no additional quality control checks were performed.

All matrix-spiked aqueous samples indicate accurate analytical results for fluoride and sulfate in all process water streams. Fluoride and sulfate spike recoveries were between 88%-110% in all three process water matrices (sweet water, sour condensate, recycle char/scrubber water). Chloride spikes were recovered within the DQO for sweet water and recycled char water (87%-95%), but were only 20% from the sour condensate. Duplicate results indicate a matrix effect is likely, so the chloride results for the sour condensate may be biased low.

Results of the aqueous audit sample analysis demonstrated good analytical accuracy for chloride (96.2%), fluoride (106.6%), formate (100%), and sulfate (78%). Sulfate recovery from the Method 8 impinger solutions was mixed at 135% in the IPA solution and 84.3% in the H_2O_2 solution. Chloride and fluoride spiked in the H_2SO_4 impinger solution as an audit sample were recovered at 2,030% and 93.4%, respectively. The high sulfate concentration in the impinger solution presented an analytical problem with the IC system specified by the boiler and industrial furnace (BIF) regulations promulgated in 40 CFR Part 266, Appendix IX. The variability in the results is likely a result of the sample dilutions required to overcome analytical difficulties. These dilutions also increased the detection limits for chloride which produced numerous undetected results.

In summary, the analytical uncertainty associated with the anionic components in the following streams may be considered significant:

- Chloride results for the sour condensate may be biased low;
- Chloride matrix spikes and audit sample results indicate analytical imprecision for vapor-phase chloride (HCl) results in the H_2SO_4 impingers; and
- Dilutions of the H_2SO_4 impinger solutions effectively raised the limit of detection for chloride above the concentration in many of the gas stream samples.

Ammonia and Cyanide—Aqueous Streams and Impinger Solutions

Detectable concentrations of ammonia were measured in the aqueous sample method blanks and in all H_2SO_4 impinger solution blanks. Cyanide was also detected in numerous blanks, however in most cases, the detected concentration was below the reported method detection limit. Relative to the sample concentrations of ammonia and cyanide, none of the blank concentrations were significant.

Matrix spiked samples and audit samples submitted for ammonia and cyanide analysis were all recovered within the DQOs except for a single MSD sample for cyanide. Ammonia matrix spikes were recovered from gas impinger solutions between 87% and 106% and from aqueous samples between 99% and 112 percent. Cyanide spikes were recovered from zinc acetate impinger solutions between 91% and 104 percent. Aqueous sample cyanide spikes were recovered between 83% and 102% with one matrix spike of a recycle char filtrate sample reported at 207 percent. The duplicate matrix spike in this sample was recovered at 83 percent.

Matrix spikes were added to sweet water samples for phenolics, and COD. In addition, an aqueous audit sample was prepared with both phenol and potassium acid phthalate (KHP) as a COD/phenol standard.

Total phenolics were recovered from the sweet water MS/MSD pair at 104% in both samples. Total phenolics in the audit standard were recovered at 101 percent. COD measurements of KHP spikes in sweet water demonstrated 76%-85% recovery with COD in the audit sample recovered at 98.9% of the theoretical value. These results are all within the data quality objectives and indicate accurate and precise results for phenol and COD in the process water.

PAHs/SVOCs—Gas Samples

Stack gas samples and internal process gas samples were analyzed by EPA Method 8270. The stack gas samples were also analyzed by CARB Method 429 for added analytical sensitivity. Filtered gas-particulate samples collected at the turbine and incinerator stacks were also analyzed by Method 8270 and CARB 429 to provide particulate- and vapor-phase data separately. Method blanks, media blanks, and field blanks were analyzed to provide a complete characterization of the sampling media and reagents.

As many as six semivolatile organic compounds on the target analyte list were detected in the blank front-half samples (gas-particulate phase) analyzed by Method 8270 for the turbine and incinerator stacks. Only di-n-butylphthalate and bis-(2-ethylhexyl)phthalate were detected in both blanks and samples. Concentrations detected in both field and media blanks were significant with respect to the sample concentrations and may account for all or part of the sample concentration reported.

Blank samples of XAD-2 resin and rinse reagents (back-half samples) analyzed by Method 8270 contained measurable quantities of nine different semivolatile organic compounds on the target analyte list. Benzoic acid, di-n-butylphthalate, and bis-(2-ethylhexyl)phthalate were measured in all blank samples and all stack gas samples. Phthalates are present in both blank and gas sample fractions at comparable levels with the exception of a few individual sample results. The reported concentrations of these phthalate compounds in the vapor-phase samples can be attributed to the background concentrations associated with the sampling media and sample handling procedures.

Blank samples of the particulate- and vapor-phase sample collection media analyzed by high resolution gas chromatography/mass spectrometry (HRGC/MS) indicated measurable quantities of nearly all of the CARB 429 analytes. Many of the compounds were detected at levels comparable to the concentrations detected in the stack samples, so results were reported with the percentage of the value attributed to the blank.

Internal process gas streams were sampled for vapor-phase semivolatile organics only. Four method blanks and two media blanks were analyzed by Method 8270 along with all of the gas samples. Naphthalene was detected in both media blanks and one-half of the method blanks.

This was the only compound detected with any regularity in these samples although single sample results for acetophenone, benzoic acid, and bis-(2-ethylhexyl)phthalate were also measured. Naphthalene concentrations measured in the blank samples were not significant with respect to the natural sample concentrations.

Surrogate spike recoveries indicate analytical accuracy for semivolatile organic compounds. Appendix Table A-4 presents the surrogate spike recoveries for all of the particulate and vapor-phase gas samples analyzed by Method 8270 and CARB 429. The surrogate compounds and the individual recovery objectives are presented in Table 5-6.

Table 5-6
Surrogate Compounds for Method 8270 and CARB 429

Sample Sources	Analytical Method	Surrogate Compounds	Recovery Objective
Emissions Sources	EPA Method 8270	1,4-Dibromobenzene-d4	50-150%
		2-Fluorobiphenyl	30-115%
		Nitrobenzene-d5	23-120%
		Phenol-d5	24-113%
		2,4,6-Tribromophenol	19-122%
		1,3,5-Trichlorobenzene-d3	50-150%
	CARB 429	Fluorene-d10	50-150%
		Terphenyl-d14	50-150%
Internal Process Gas Streams	EPA Method 8270	2-Fluorobiphenyl	30-115%
		2-Fluorophenol	25-121%
		Nitrobenzene-d5	23-120%
		Phenol-d5	24-113%
		Terphenyl-d14	18-137%
		2,4,6-Tribromophenol	19-122%

Nearly all surrogate spikes were recovered within the method-specified recovery objectives indicating acceptable method performance. Instances where surrogate-spike recovery objectives were not met are sporadic and do not indicate any definitive analytical bias. However, some analytical difficulties were encountered with sour syngas and tail gas samples. Dilution of the sample extracts was required to minimize the matrix effects produced by high concentrations of naphthalene (and possibly other semivolatile organic compounds) which overloaded the detector.

Some samples were diluted to the point where many compounds detected in an initial sample injection went undetected in the diluted samples. These dilutions were considered when calculating the reported sample concentrations and detection limits. The handling of this data is discussed later in this section.

The following observations are summarized for SVOCs and PAHs in the gas samples:

- Phthalate-compounds detected in the SVOC stack gas samples can be attributed in part or in total to the presence of these compounds in the blank media, or introduced through sample handling and analysis;

- High resolution GC/MS provides adequate sensitivity to lower detection limits nearly 3-5 orders of magnitude (compared to GC/MS) and consequently, a complete characterization of the blank media is required to provide a statistical means of differentiating background media concentrations and actual sample results;

- Surrogate spike recoveries indicate acceptable method performance for related compounds; and

- High naphthalene concentrations in the sour syngas and tail gas matrices required that samples be diluted for analysis thereby compromising the detection limits for other compounds.

PAHs/SVOCs—Aqueous Streams

Semivolatile organic compounds were measured in sweet water and sour condensate by EPA Method 8270. Two method blanks were prepared to assess the contamination potential of the sample extraction and handling procedures. Naphthalene was the only target compound detected in the method blanks.

Surrogate spikes were added to each water sample to assess analytical accuracy. All surrogates in all samples were recovered within the method-specified recovery objectives with the exception of one phenol-d5 sample spike. No other analytical problems were noted. These results indicate acceptable method performance for the analysis of semivolatile organics in the process water samples.

Aldehydes—Impinger Solutions and Aqueous Streams

The 2,4-dinitrophenylhydrazine (DNPH) impinger solution used to collect aldehydes and ketones is very susceptible to outside sources of contamination. Consequently, numerous laboratory method blanks, media trip blanks, and impinger field blanks were collected for each batch of impinger solutions and samples shipped to the laboratory to assess the potential for sample contamination from various stages of sample handling.

None of the four target aldehyde compounds were found in any of the laboratory blanks for analytical batches of gas impinger solutions or aqueous samples. No field blanks were collected representing aqueous sample collection and handling, however a total of six DNPH media blanks were submitted; at least one for each shipment of impinger samples and each reagent batch. In three of the six DNPH media blank samples, formaldehyde was detected. In one of the six, acetaldehyde was detected, although only slightly above the detection limit. Formaldehyde and acetaldehyde were also detected at comparable levels in the field blank samples. Trip blank samples sent from the laboratory and returned to the laboratory unopened showed no signs of contamination during shipment. Since the trip blank samples were unopened in the field, these results indicate that some formaldehyde and acetaldehyde contamination of the samples may have occurred during sample handling in the field environment. Since the LGTI gasifier is located at a large chemical manufacturing complex, this possibility seems likely.

Sample results for formaldehyde in the following process streams may be affected (biased high): sour syngas (11), sweet syngas (12), incinerator stack gas (16), and to a lesser extent, turbine stack gas (13). Acetaldehyde was also detected in samples from each of these gas streams. This was not a factor in the sour and sweet syngas samples where acetaldehyde concentrations were high; however, it was significant with respect to the incinerator and turbine stack gas samples where blank levels accounted for approximately 50% to over 100% of the amount measured.

Analytical accuracy for DNPH impinger samples was measured by spike recovery from laboratory prepared spikes, trip spikes (to measure effectiveness of the preservative between the laboratory and field location), and field spikes. Formaldehyde and acetaldehyde spikes were recovered from all samples (where spiking was performed) within the method-specified recovery objectives. Acrolein was spiked in one sample and was recovered at 64%, below the 70% recovery objective. For process water samples, the lab spike and aqueous sample matrix spike were recovered within the method-specified recovery objectives for formaldehyde, acetaldehyde, and acrolein. Benzaldehyde was not spiked.

The following can be summarized from the data quality measurements for aldehydes:

- Formaldehyde and acetaldehyde were detected in the blank DNPH impinger solutions collected in the field indicating possible sample contamination from ambient sources which may bias results high (samples were not blank corrected); and

- Spike recoveries for formaldehyde and acetaldehyde indicate acceptable and accurate method performance for both aqueous samples and DNPH impinger samples.

Volatile Organics—Gas Samples (VOST) and Aqueous Streams

Volatile organic compounds were collected from the incinerator and turbine stacks using the volatile organic sampling train (VOST), while aqueous samples were collected in vials with no headspace. Due to the presence of volatile organic compounds in the field and laboratory environment, each VOST sample run included a field blank sample collected at the gas sampling

location. Laboratory method blanks, and trip blanks (media blanks) were also analyzed to assess potential contamination sources. Aqueous field blank samples were not collected.

Chloromethane and bromomethane were the only compounds detected in the VOST laboratory method blanks. Analysis of field blank samples indicated the occasional presence of trichlorofluoromethane and carbon disulfide, but a more frequent and significant presence of dichloromethane (methylene chloride) which is commonly used at the stack locations for recovery of the semivolatile organics sampling train. Although efforts were made to complete SVOC sampling before running the VOST, the reported concentrations of methylene chloride in the incinerator and turbine stack samples is likely due to the presence of methylene chloride contamination from field handling.

Similarly, low concentrations of two common field and lab reagents (acetone and methylene chloride) were detected in both laboratory method blanks for aqueous samples. Both of these compounds were measured in the sour condensate and sweet water samples. Again steps were taken to isolate these samples from any direct exposure by maintaining a separate handling area apart from the sampling train recovery and chemical storage areas of the field laboratory. Based on the sample concentrations however, the consistent presence of these two compounds in the sour condensate stream cannot be ruled out as a matter of sample contamination.

To assess desorption efficiency and analytical accuracy VOST sorbents (Tenax resin and charcoal) were spiked with surrogate compounds and thermally desorbed as a single unit. Aqueous samples collected in vials with no headspace were purged entirely making surrogate spiking the best way to assess analytical recovery and accuracy. The volatile organic surrogates for both VOST and aqueous sample analysis were 1,2-dichloroethane-d4, toluene-d8, and 4-bromofluorobenzene. These surrogate spikes were recovered in all VOST and aqueous samples with the exception of some selected VOST samples collected from the incinerator stack. The high levels of SO_2 present in the incinerator stack gas may be responsible for providing a matrix effect, nonetheless, most of the results appear within the method-specified surrogate recovery objectives. Acceptable analytical performance for volatile organic compounds is indicated.

Sample Collection

Although the analytical quality control measures discussed indicate that the methods used for sample analysis were good, they say nothing about the quality of the samples collected. Numerous factors may affect representative sample collection for HAPs, especially with unconventional process systems. In fact, the sampling location itself or the nature of the process stream can present problems too difficult to overcome without costly modifications. This section discusses noteworthy observations made during process sampling that might affect a sample's representativeness.

Table 5-7 summarizes the process streams and test parameters that presented special circumstances potentially affecting representative sample collection. Of these, the moisture content of the syngas upstream of the Selectamine™ sulfur removal system was the most problematic.

Table 5-7
Sampling Issues

Sample Point	Sampling Concern	Potentially Affected Analytes	Action Taken and Possible Affect on Data Quality
Sour Syngas (11)	Excessive condensate present in main sample line, believed to be wall creep from process duct.		Sample lines arranged vertically to avoid collection of condensate in individual sample lines. Assuming gas-liquid equilibrium exists, there should be no significant bias to the samples.
Scrubbed Raw Syngas (5b)	Sample moisture condensing in long sample line before distribution from the sample header.	NH_3, HCN	Condensate knock-out impingers were used to collect the gas condensate for analysis. Impinger train results were corrected based on an aliquot of condensate representative of the theoretical gas moisture content.
Raw Syngas (5a)	Sample moisture building up in the sample line produced sporadic "burps" of condensate into the impinger trains.	NH_3 and others—high variability between sample runs indicates high degree of uncertainty associated with these results.	Condensate flow was inconsistent and not controlled or collected separately. Samples may be biased low since uncollected condensate was not accounted for.
Hot Raw Syngas (5) (High-temp Sampling Probe)	Sample moisture condensing within the sheathed part of the probe.		The sample line downstream of the probe sheath was heated and the gas flow through the main sample line was increased in an effort to maintain the gas temperature above the dew point. This appeared successful since the condensate collected in the impinger trains reflected the theoretical moisture content of the gas.
Slag	Slag fines not effectively retained by sample collection system.	Trace elements, carbon	Slag fines are usually higher in carbon and enriched with trace metals. Slag results may be biased low with respect to these components if the percentage of fines lost through the sample screen is significant.
Selectamine™ Solvent	Samples were collected before and after a regeneration period.	Metals, heat stable salts	Accumulation of salts, metals, and solids throughout the test period could not be determined.

Table 5-7 (Continued)

Sample Point	Sampling Concern	Potentially Affected Analytes	Action Taken and Possible Affect on Data Quality
Sour Gas (22)	Gas composition (primarily NH_3 and CO_2)	Metals, semivolatile organics	The chemistry of the sampling trains and sour gas components results in the precipitation of $(NH_4)_2CO_3$ or NH_4HCO_3 salts which effectively, and rapidly plug up the sampling train. Samples for metals and semivolatile organics could not be collected.
Internal process gas streams	Apparent low collection efficiency of the multi-metals sampling train (Method 29) .	Vapor-phase metals	All results reported for vapor-phase metals were much lower than results obtained by charcoal or direct AAS analysis. The use of Method 29 for quantifying vapor-phase metals in reducing gas matrices is not recommended.
Sweet and Sour Syngas	Selected results for vapor-phase metals collected by charcoal appear biased low when compared to direct AAS analysis.	Vapor-phase metals (arsenic).	Charcoal may be species-selective in its collection of vapor-phase metals from syngas.

Assuming saturation, the water vapor concentration in the hot raw syngas is about 35% by volume. As the syngas sample is extracted from the process duct it rapidly cools and water vapor is condensed in the sample line. Condensed or entrained moisture may also creep along the inside of the duct and find its way into the sample line from surface-mounted taps. The collection of a non-representative volume of water vapor in an impinger train will skew the results high for any water-soluble species of interest, particularly ammonia, hydrogen cyanide, acid gases, and possibly some metals.

Condensation and/or water entrainment upstream of the individual impinger train sampling lines was observed at the sour syngas (11), scrubbed raw syngas (5b), raw syngas (5a), and hot raw syngas (5) sampling points. Although excess condensate was present in the main sample line and could absorb any of the water soluble species of interest, it was assumed that a gas-liquid equilibrium had been established and that any gas collected in the absence of entrained water was representative of the gas stream, plus whatever representative portion of collected condensate could be attributed to each individual gas sample.

Special condensate knock-out and collection impingers were placed upstream of the sampling impingers at the scrubbed raw syngas sampling point at the outlet of the venturi scrubber. The condensate collected here is believed to represent gas moisture condensing in the line since the sample tap was taken directly from the top of the process duct where water could not easily be entrained. The collected condensate was analyzed for ammonia, cyanide, and metals. Ammonia and cyanide concentrations in the condensate were significant so the mass of analyte collected in the sample train impingers was supplemented by the mass of analyte contained in a representative aliquot of condensate. A representative aliquot of condensate was determined as follows:

1) The theoretical, saturated moisture content of the syngas at process conditions as calculated;

2) The gas moisture content, as determined from the sampling train, was calculated;

3) The difference in these two numbers represented the amount of condensate that *would have been collected* in the sampling train under ideal conditions; and

4) Assuming that any species that were found in the condensate were in equilibrium with the gas phase, the quantity of analyte represented by the condensate volume determined in Step 3 was added to the sample.

For instance, assume the theoretical moisture in Step 1 was 35 percent, and the measured moisture in Step 2 was 10 percent. What volume of moisture is represented by the 15% difference in these two numbers? Let's assume the volume is 15 mL of condensate. Then assume the analysis of the condensate was 45 μg/mL NH_3 (45 μg/mL * 15 mL = 675 μg NH_3). This 675 μg NH_3 would then be added to the total number of μg of NH_3 determined in the sample and the gas-phase concentration of ammonia was reported as the *[total measured + condensate amount]/ sample gas volume.*

Ideally, short heated sample lines would prevent condensation of water vapor, but this was not physically possible at all sampling locations. In future sampling efforts, extra attention should be given to hot gas locations. If physical limitations will bias representative sample collection, then the sampling scope should be adjusted accordingly or the resulting data evaluated with respect to the potential sample collection induced bias.

Concerns over the collection of slag samples, Selectamine™ solvent samples, and sour gas samples is explained in Table 5-7. Additional gas sampling issues include an assessment of collection efficiency for vapor-phase metals by charcoal and Method 29 in comparison to the total characterization provided by direct AAS analysis of the gas. This is discussed in more detail in Sections 7 and 8.

Data Handling Conventions

Over 25,000 individual pieces of sampling and analytical data were processed to determine the process stream sample concentrations. The concentration results for each sample are presented

in Appendix Tables G-1 and G-2. The individual sample results in Table G-1 were averaged to provide the stream concentration results summarized in Section 4. This section describes the protocols used for handling data, including "not detected" results, to determine average (mean) concentration results and confidence intervals. Example calculations for determining standard deviations and confidence intervals can be found in Appendix D.

Several conventions have been developed for treating the test data and developing average concentrations of substances in the various streams. In general, there are three cases that affect the calculation of average concentrations:

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

- For results that included values both above and below the detection limit, one-half the detection limit was used to calculate the mean. For example:

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

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

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

- When all analytical results for a given variable are below the detection limit, the mean is reported as "< X," where the X is the largest detection limit. The bias estimate (used where calculating confidence intervals for other parameters) is one-half of the detection level, and no confidence interval is reported.

The same convention using one-half the detection limit was also used to determine the standard deviation and 95% confidence intervals, where appropriate. All calculations were performed with unrounded numbers and the results were rounded to two or three significant figures for presentation in the tables; therefore, slight differences in calculated means and confidence intervals are attributable to round-off errors.

In a few isolated cases, samples were diluted prior to analysis in order to avoid overloading the analytical detection system. The increase in the detection limit resulting from these dilutions was taken into consideration when reporting the average results where the DL is higher than the concentrations detected at lower levels in other samples from the same test period. The analytical results for semivolatile organic compounds in sour syngas and tail gas, and chloride in turbine stack gas fit this category. The high detection limits reported for one of the three runs

were not used in the calculation of the average unless the analyte was detected in the diluted sample. The omitted results are identified in Appendix Table G-1.

In addition to reporting average concentrations, total gas emissions were measured independently in two phases, vapor and particulate. To determine the total concentration for gas streams within a run, both the solid- and vapor-phase contributions were considered; however, the absence of some detectable concentrations in either (or both) phase(s) required that conventions be developed for dealing with these data. These conventions are summarized below for the following three cases:

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

Case 2: The concentrations in both the solid and vapor phases are below 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 inorganic constituents of interest other than HF, HCl, NH_3, HCN, and mercury, the flue gas stream data from previous studies of coal-fired power plants have shown that most of the material is present in the solid phase, and that only a small fraction is generally found in the vapor phase. The opposite is generally true for organic species. 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 average total chromium concentration in the turbine stack gas is calculated as follows:

Chromium in the particulate phase = 1.02 $\mu g/Nm^3$

Chromium in the vapor phase = 0.474 $\mu g/Nm^3$

Total chromium in the turbine stack gas = 1.49 $\mu g/Nm^3$

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

For example, the total beryllium concentration in the incinerator stack gas is calculated as follows:

Beryllium in the solid phase = < 0.012 $\mu g/Nm^3$

Beryllium in the vapor phase = < 0.051 $\mu g/Nm^3$

Total beryllium in the ESP inlet gas = < 0.012 μg/Nm^3

An example for HCl in incinerator stack gas illustrates the exception that is applied to species predominantly associated with the vapor phase:

Chloride in the solid phase = < 150 μg/Nm^3

Chloride in the vapor phase = < 2,100 μg/Nm^3

Total chloride in the incinerator stack gas = < 2,100 μg/Nm^3

- For Case 3, multiple conventions have been established, depending on the group of substances being considered.

For metals train results, if the substance is not detected in the solid phase and detected in the vapor phase at levels below the detection limit of the solid phase, the total concentration is reported as the detection limit of the solid phase and the total is flagged to note that the substance was detected at lower levels in the vapor phase. For the turbine and incinerator stack multimetals trains, this scenario did not occur.

For metals train results, if the substance is not detected in the vapor phase and detected in the solid phase, the vapor-phase component is considered to be equal to zero.

For example, the total magnesium concentration in the turbine stack gas is calculated as follows:

Magnesium in the solid phase = 9.97 μg/Nm^3

Magnesium in the vapor phase = < 13 μg/Nm^3

Total magnesium in the turbine stack gas = 9.97 μg/Nm^3

For semivolatile organic compounds and PAHs, the opposite is assumed. If the substance is not detected in the solid phase and detected in the vapor phase, the reported total is the concentration in the vapor phase.

For example, the average total 2-chloronaphthalene concentration in the incinerator stack gas is calculated as follows:

2-chloronaphthalene in the solid phase = < 0.077 ng/Nm^3

2-chloronaphthalene in the vapor phase = 0.0976 ng/Nm^3

Total 2-chloronaphthalene in the incinerator stack gas = 0.0976 ng/Nm^3

Material Balances

The results of material balance closures are presented in the following sections. Example calculations are presented in Appendix E.

Process flow rates used to develop mass balances are summarized in Table 5-8. Mass balances were performed around the Selectamine™ and Selectox™ systems, the sour water stripper, incinerator, gas turbine, and the total plant. In addition, balances were performed around the combined Selectamine™ and gas turbine systems. Figure 5-1 depicts the mass balance boundaries for each of the individual systems. Steady-state process operation was assumed for all process systems, except the Selectamine™ unit. Due to the solvent holdup volume, significant accumulations of a substance could occur in the Selectamine™ process unit. Over a long period of steady operation, the accumulation in the Selectamine™ system could be measurable. However, the Selectamine™ was regenerated during the test period which made it impossible to quantify accumulation.

A general mass balance equation which applies to any system is:

$$\begin{bmatrix}\text{Accumulation of}\\ \text{Mass in System}\end{bmatrix} = \begin{bmatrix}\text{Mass into}\\ \text{System}\end{bmatrix} - \begin{bmatrix}\text{Mass out}\\ \text{of System}\end{bmatrix} + \begin{bmatrix}\text{Mass Generated}\\ \text{in System}\end{bmatrix} \qquad \text{(eq. 5-1)}$$

The following general equation was used to calculate mass balance closures.

$$\text{Mass Balance Closure (\%)} = 100 * \left[\frac{\text{Total Mass Out}}{(\text{Mass In} - \text{Mass Accumulated})}\right] \qquad \text{(eq. 5-2)}$$

For all systems other than the Selectamine™ unit, the accumulation term should be negligible and was assumed to be zero. Development of specific mass balance equations is presented in Appendix E.

The mass balance closure for each element met the project objective if it was between 70 and 130 percent. Poor closures and high uncertainties have their root cause in sampling, analytical, or process problems. Since an analysis of the process showed that operations were steady and representative of normal operation, problems with mass balance closures for some substances may reflect problems with analytical or sampling techniques. For all the internal process streams, sampling methodologies for trace metals are most likely the reasons for poor closure. As stated before, validated methods for trace element characterization of a syngas matrix have not been developed.

Table 5-8
Process Flow Rates During LGTI Testing

	Period 1 (11/4-11/7)	Period 2 (11/8-11/11)	Period 3 (11/11-11/13)	Total (11/4-11/13)	Std. Dev.
Coal Feed Rate (t/d, dry)					
Slag Production (t/d, dry)					
Primary Slurry Rate (gpm)					
Primary Slurry (% solids)					
Second Stage Slurry Rate (gpm)					
Second Stage Slurry (% solids)					
Sour Syngas Flow (lb/hr, wet)					
Sour Syngas Flow (lb/hr, dry)					
Demineralized Water to Venturi Scrubber (gpm)					
Sweet Syngas Flow (lb/hr, dry)					
Acid Gas Flow (lb/hr, dry)					
Tail Gas Flow Rate (lb/hr, dry)					
Vent Gas Flow to Incinerator (lb/hr)					
Methane Fuel Rate to Incinerator (lb/hr)					
Sweet Water to Ditch (gpm)					
Steam Drum blowdown to SWS (lb/hr)					
D-251 Blowdown to SWS (lb/hr)					
C-180 Blowdown to SWS (gpm)					
Sour Water to Stripper (gpm)					
C-180 Blowdown to SWS (lb/hr)					
Incinerator					
Incinerator Stack Gas Flow Rate (Nm^3/hr)	NA	19,400	NA	19,400	620
Particulate Concentration (mg/Nm^3)	NA	141	NA	141	10
Particulate Emission Rate (lb/hr)	NA	6.03	NA	6.03	0.38

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Table 5-8 (Continued)

	Period 1 (11/4-11/7)	Period 2 (11/8-11/11)	Period 3 (11/11-11/13)	Total (11/4-11/13)	Std. Dev.
Power II					
Syngas to GT-400 (lb/hr)					
Fuel Gas (Methane) to GT-400 (lb/hr)					
GT-400 Fuel (%Syngas)					
Syngas to GT-300 (lb/hr)					
GT-400 Stack Gas Rate (Nm^3/hr)				$1.1x10^6$	
Particulate Concentration (mg/Nm^3)	3.86	NA	NA	3.86	1.41
GT-400 Particulate Emission Rate (lb/hr)	9.3	NA	NA	9.3	3.3

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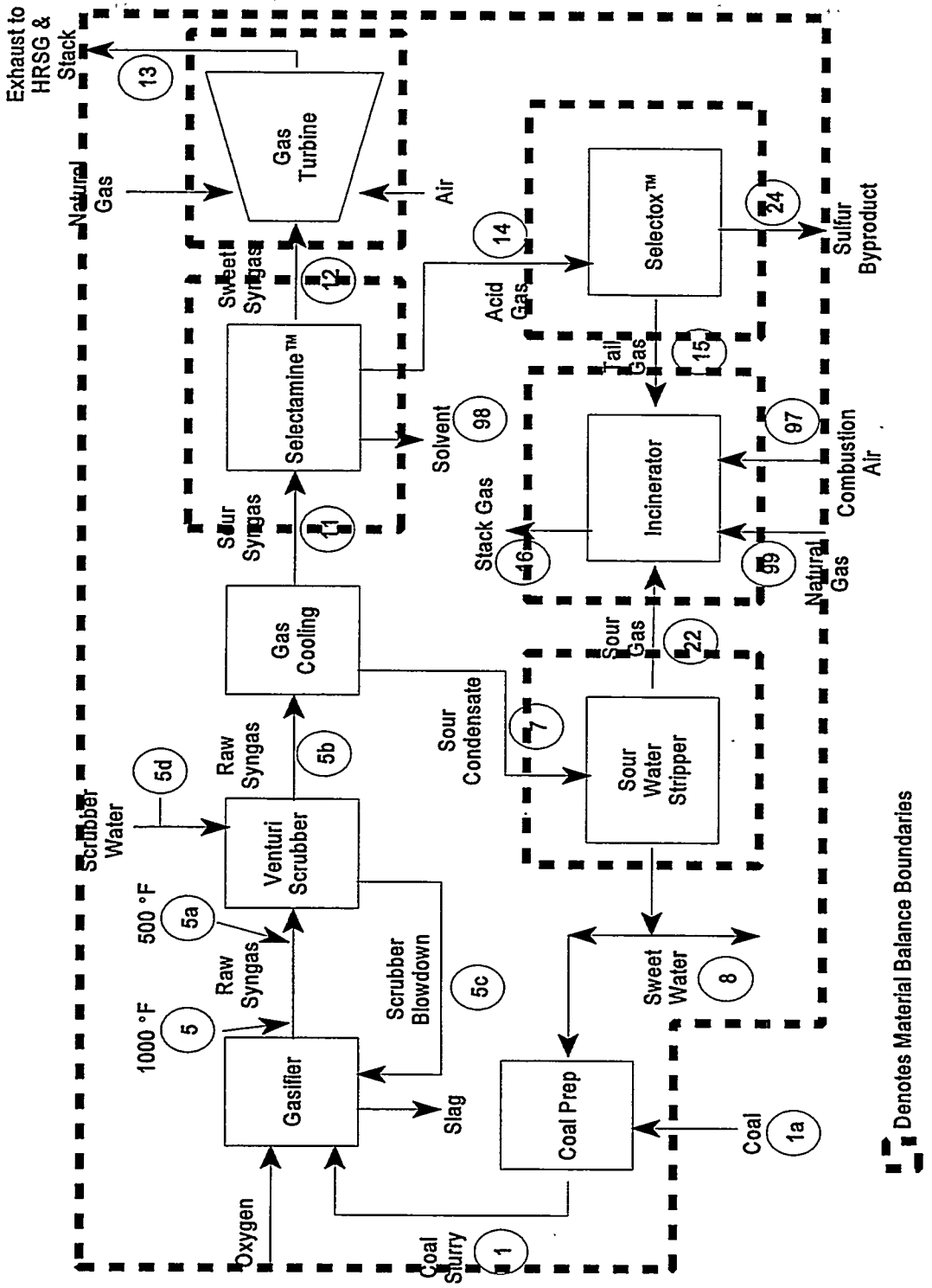

Figure 5-1
LGTI Block Flow Diagram with Material Balance Boundaries

Concerns with mass balance closures fall into three categories:

- Mass balance closure is outside target range of 70-130 percent.
- High uncertainty—uncertainty in closure exceeds ±50 percent.
- Clear bias—closure ± uncertainty does not encompass 100% closure.

For the overall plant closure, 76% of the mass balances performed fell within the target range. This compares to 59% for the total plant results from Radian's Phase I DOE project conducted at Plant Yates[1]. This overall high degree of closure is excellent, given the complexity of an IGCC process, when compared to that of a conventional coal-fired power plant. Also, the gas phase methods (EPA Method 29) used for the gaseous effluent streams (turbine and incinerator stacks) were developed specifically for these types of process streams, and historically produce very reliable data.

The percentage of mass balance closures that are within the target range for the individual subsystems were much lower. There are several factors that contribute to the lower percentages of elements that met the mass closure targets for the internal process streams. The sampling method issue was discussed previously. Two other factors contributing to poor mass balance closures were the inability to sample the sour water stripper off gas due to the high ammonium carbonate content of the stream, and to quantitate any accumulation of substances in the Selectamine™ system. The original intent was to analyze the Selectamine™ solvent at the beginning and again at the end of the test period. However, the Selectamine™ solvent system was treated or "regenerated" during the test period, and it was impossible to quantify the effects of this treatment on the accumulation of trace metals in the Selectamine™ system.

Additional factors that may cause inaccuracies in closing material balances around several of the units were the relatively low levels of most of the inorganic substances in the vapor phase of the gas streams flowing to and from the subsystems. Many of the measured vapor-phase compositions approach the analytical detection limits, and even when concentrations are above the detection limits, only small quantities of substances are being measured. Material balances were also hampered by the lack of particulate loading and composition data for most streams. Particulate loadings and compositions could only be measured in the incinerator and gas turbine stacks. Particulate loading was measured in the sweet syngas stream, but the loading was quite low and the amount of particulate collected was insufficient to characterize.

Table 5-9 presents material balance closures for the various sub-systems and target species. Again, none of these methods have been validated in the reducing atmospheres that exist in most of the internal process streams in gasification systems.

In developing the results summarized in Table 5-9, balances were not generally calculated (NC) if a substance was not detected in one of the major streams around the system. However, if the

Table 5-9
Material Balance Closures

Substance	Selectamine™ System		Selectox™ System		Sour Water Stripper		Incinerator		Gas Turbine		Combined Selectamine™ System and Gas Turbine		Overall Balance	
	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure, %	95% CI	Closure %	95% CI
Ionic Species														
Chloride	NC	NC	NA	NC	230	214	NA	NC	280	NC	NA	NC	57	15
Fluoride	146	46	NA	NC	87	28	NA	NC	1,600	NC	NA	NC	22	6
Reduced Sulfur Species														
H_2S	73	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC
COS	108	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC
CS_2	NC	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC	NA	NC
Metals (Vapor-Phase AAS)														
Arsenic	NC	NC	NA	NC	NA	NC	NA	NC	NC	NC	NC	NC	NA	NC
Cadmium	NC	NC	NA	NC	NA	NC	NA	NC	160	NC	NC	NC	NA	NC
Chromium	NC	NC	NA	NC	NA	NC	NA	NC	NC	NC	16	NC	NA	NC
Lead	NC	NC	NA	NC	NA	NC	NA	NC	NC	NC	NC	NC	NA	NC
Mercury	63	NC	NA	NC	NA	NC	NA	NC	175	NC	107	NC	NA	NC
Nickel	4	NC	NA	NC	NA	NC	NA	NC	107	NC	5	NC	NA	NC
Selenium	34	NC	NA	NC	NA	NC	NA	NC	8	NC	3	NC	NA	NC
Zinc	NC	NC	NA	NC	NA	NC	NA	NC	NC	NC	NC	NC	NA	NC

Table 5-9 (Continued)

Substance	Selectamine™ System		Selectox™ System		Sour Water Stripper		Incinerator		Gas Turbine		Combined Selectamine™ System and Gas Turbine		Overall Balance	
	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure %	95% CI	Closure, %	95% CI	Closure %	95% CI
Metals														
Aluminum	NC	NC	NC	NC	57	23	NC	NC	NC	NC	NC	NC	107	9
Antimony	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC
Arsenic	2	1	NC	NC	58	81	NC	NC	190	310	NC	NC	50	6
Barium	12	3	NC	NC	73	13	960	1,200	800	6,000	280	170	119	27
Beryllium	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	95	12
Boron	11	2	NC	NC	120	1,600	120	130	NC	NC	NC	NC	82	6
Cadmium	NC	NC	NC	NC	76	99	400	540	NC	NC	NC	NC	74	67
Calcium	NC	NC	NC	NC	47	8	140	160	NC	NC	NC	NC	111	8
Chromium	13	3	51	350	NC	NC	48	73	380	210	24	6	120	39
Cobalt	NC	NC	NC	NC	NC	NC	21	29	NC	NC	NC	NC	101	10
Copper	9	2	NC	NC	81	110	240	260	4,400	11,000	170	400	99	9
Iron	11	3	5	18	73	42	2,100	2,500	900	3,400	39	120	113	8
Lead	NC	NC	NC	NC	92	120	96	110	NC	NC	NC	NC	55	5
Magnesium	NC	NC	NC	NC	52	6	NC	NC	3,800	15,000	NC	NC	109	8
Manganese	9	10	NC	NC	78	93	4,500	8,700	NC	NC	150	630	99	9
Mercury	2	2	330	770	NC	NC	900	1,200	5,000	2,000	58	73	25	7
Molybdenum	10	3	NC	NC	NC	NC	3,000	3,900	2,300	3,200	84	110	132	19
Nickel	12	9	NC	NC	NC	NC	57	56	2,000	2,900	120	160	188	53
Phosphorus	NC	NC	NC	NC	40	10	NC	NC	NC	NC	NC	NC	103	16
Potassium	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	98	13
Selenium	NC	NC	NC	NC	68	99	NC	NC	8,100	8,500	NC	NC	34	16
Silicon	NC	NC	NC	NC	92	7	120	150	NC	NC	NC	NC	108	10
Sodium	NC	NC	NC	NC	150	7	280	340	NC	NC	NC	NC	117	8
Titanium	NC	NC	NC	NC	54	46	NC	NC	NC	NC	NC	NC	85	14
Vanadium	8	4	400	410	NC	NC	1,100	1,200	1,600	2,000	56	26	103	6
Zinc	NC	NC	NC	NC	78	94	180	190	22,000	9,100	NC	NC	102	8

effect of a particular "not detected" value was negligible in calculating the balances, the balances are shown.

Overall Plant Material Balances

Validated sampling methods only apply to the balances around the entire plant. The inlet streams are the coal, makeup water to the scrubber, natural gas, oxygen, and ambient combustion air. Outlet streams include the slag, sweet water (from the sour water stripper) leaving the plant, sulfur byproduct, incinerator exhaust, and turbine exhaust. The levels of the metals and other inorganic substances in the makeup water, oxygen, and combustion air were assumed to be negligibly small (they were not sampled), and these streams were not included in the overall balances.

There are six elements that did not meet the closure criteria of 70-130 percent:

≤ 70% Closure	≥ 130% Closure
Arsenic	Molybdenum
Mercury	Nickel
Lead	
Selenium	

The particular substances for which balance closures were below 70% were not unexpected. Measurements using the vapor-phase AAS suggest that some of the more volatile substances such as arsenic, mercury, and selenium may be partially removed in the Selectamine™ system.

One would also expect that lead and possibly nickel may form insoluble sulfides and be removed in the Selectamine™ system also. However, lead was generally not detected in either the sour or sweet syngas, so possible lead removal in the Selectamine™ unit can be neither confirmed nor refuted.

Selectamine™ System

The inlet stream to this unit is the sour syngas stream, and the outlet streams are the sweet syngas and the acid gas streams. Carbon adsorption and Method 29 techniques were used to sample all three streams, so complete mass balance closures could be determined independently from the results of each of the sampling methods.

The balances achieved with carbon adsorption appear to be biased low by about an order of magnitude. This large bias could be caused be one or more factors. The most obvious possible causes of such biased balances are 1) accumulation of the vapor-phase metals in the Selectamine™ solvent, 2) measured concentrations that are biased high in the sour syngas and/or biased low in the acid gas or sweet gas streams , and 3) inaccurate stream flow rates. The latter potential cause of bias seems very unlikely given the maturity of the plant and the consistency of

the flow rates from day to day. In addition, the balances for fluoride and the reduced sulfur species appear to be quite reasonable, indicating accurate flow rates.

If the acid gas compositions were biased low, then the Selectox™ balances would tend to be biased high, since the acid gas is the inlet stream to the Selectox™ unit. Although only a few component balances could be determined about the Selectox™ unit, the results indicate the possibility of balances being biased somewhat high, but not of the magnitude found in the Selectamine™ balances. This indicates that the acid gas composition is probably not biased high to a great degree.

If the sweet syngas compositions were biased low, the gas turbine balances would be biased high, since the sweet syngas is an inlet stream to the turbine system. This is the case, as seen in Table 5-9, the closures around the turbine are high by one to two orders of magnitude. To further explore this issue, a balance was made around the combined Selectamine™ -gas turbine system. The inlet streams for this system are sour syngas and natural gas, while the outlets are the acid gas and turbine stack streams. The sweet syngas is not included in this balance. The results of those balances are also provided in Table 5-9. The balance closures are quite reasonable for most of the metals. Thus, it appears that the sweet syngas vapor-phase compositions measured with charcoal are all consistently low by substantial amounts.

One major difference between the sweet syngas composition and most of the other internal gas streams is the sulfide content. The reduced sulfur species are present at significant levels in the acid gas and sour syngas streams, while the concentrations in the sweet syngas are much lower. It is known that sulfided charcoal adsorbs mercury more effectively than untreated charcoal. Thus the presence of sulfides in the gas stream may enhance the effectiveness of adsorption of vapor-phase metals, while the absence of these sulfides may result in low adsorption efficiencies.

[Note: The balance closures for vapor-phase metals as determined from Method 29 measurements appear to be more reasonable than those found with the charcoal method. However, these results may be only fortuitous and not truly accurate (i.e., the measured concentrations in the sour and sweet syngas and in the acid gas may all be consistently low). This possibility is reinforced by the high closures found around the Selectox™ unit and gas turbine when using the Method 29 concentrations for the syngas and acid gas streams. Even the combined Selectamine™-turbine system closures are very high as determined by Method 29 results, indicating the probability of a significantly low bias in the sour syngas concentrations.]

Balances for mercury, zinc, and selenium were also calculated using the on-line AAS analyses. However, only the sweet and sour syngas streams were analyzed by this method, so the charcoal method results were used for the acid gas composition in performing the material balances. The closures around the Selectamine™ system for mercury and selenium using the AAS results were significantly better than those using only the charcoal results, although still appearing to be biased low. However, the closure for nickel was very low due to the high nickel concentration found by AAS in the sour syngas relative to the sweet syngas and gas turbine exhaust. The balances around the combined Selectamine™-turbine system were low for chromium, nickel, and

selenium, but the mercury balance closure was 107 percent. The low closures would indicate that the measured concentrations of these metals in the sour syngas were high, that the levels in the acid gas and/or turbine exhaust were low, and/or that there was some accumulation of these substances in the Selectamine™ system.

Selectox™ System

The inlet stream to the Selectox™ system is the acid gas, and the outlet streams are the tail gas and the sulfur byproduct. Material balances around the Selectox™ system could only be performed for a few of the metals. Most of the metals were present in the sulfur byproduct at levels that were below the detection limits. However, at these detection limits, the amounts of many of the metals that might be present in the sulfur were very significant relative to the amounts in both the acid gas and tail gas streams. Thus, reliable balances could not be performed for most metals.

Balances were calculated using a combination of charcoal method (for the acid gas stream) and Method 29 results (for the tail gas stream). The Method 29 should be applicable to the tail gas, since the gas consists almost exclusively of CO_2. Balance calculations produced mixed results. The combined charcoal-Method 29 could only be used to calculate balances for four metals (chromium, iron, mercury, and vanadium). The closures ranged from 5% for iron to 397% for vanadium. There did not appear to be a consistent bias in the closures.

Sour Water Stripper

Incoming streams to the sour water stripper include sour condensate, steam drum blowdown, D-251 blowdown, and C-180 water purge. Outlet streams are sour gas and sweet water. The sour condensate rate is not measured, so the rate was estimated by subtracting the measured rates of the several clean water blowdown streams from the sweet water rate.

The sour gas rate and vapor-phase metals content of the sour gas could not be measured because of the sampling problems mentioned earlier. As a result, the balance was made on the assumption that the incoming stream was the sour condensate (estimated by difference) and the outgoing stream was the sweet water. The sour gas was not included in the balance. The balance results are varied, with closures for 9 of 17 metals falling within the target range of 70-130 percent. However, it appears that the closures may be biased a little low, with only two of the 17 closures being above 100 percent. The average closure is 75 percent. The low bias may indicate that the estimated rate for the sour condensate is somewhat low.

Incinerator Exhaust

There are five gas streams that feed into the incinerator. These streams are the tail gas from the Selectox™ process, the sour gas from the sour water stripper, vent gas stream, combustion air, and natural gas. The metals content was not determined for the vent stream and for the sour gas

stream. For material balance purposes, the input streams were effectively the tail gas and natural gas. The outlet stream was the incinerator exhaust.

The material balance closures varied widely, ranging from 21% (cobalt) to 4500% (manganese). Most of the closures were high; only 4 of the 17 substance closures were below 100 percent. Four metals (iron, manganese, molybdenum, and vanadium) had closures that were above 1000 percent. The measurement of substance levels in the stack gas should be the most accurate of the three gas streams, since standard sampling methods are used and both vapor and particulate phases are measured in this stream. On the other hand, there are reasons to suspect the measured levels of some substances in the tail gas. One potential source of inaccuracy in the tail gas might stem from the possible presence of particulate in the tail gas. Particulate was not measured in the tail gas, but may have been present from the acid gas entering the Selectox™ or from the catalyst in the Selectox™ unit. It is interesting to note that the closures for iron, molybdenum, and vanadium were also among the highest around the gas turbine (molybdenum closures were not calculated around the turbine).

Gas Turbine Exhaust

Four gas streams are associated with this system. The inlet streams are the sweet syngas, combustion air, and natural gas streams, while the turbine exhaust is the only outlet stream. As with incinerator exhaust sampling, established methods were used to sample the turbine stack, so the stack testing results are expected to provide the most accurate and reliable concentrations of the three streams included in this balance.

The material balance closures for the metals are included in Table 5-9. Two sets of closures were determined using the results of two sampling methods (i.e., charcoal and on-line AAS) to measure the vapor-phase metals content in the sweet syngas. The material balance closures are high for all cases, but particularly when using the sweet syngas concentrations determined with either the charcoal or Method 29 techniques. As addressed previously in the discussion of material balances around the Selectamine™ system, the concentrations of the vapor- phase metals in the sweet syngas are thought to be significantly low. Inaccurately low concentrations in the sweet syngas would produce the high closures found in the material balances around the gas turbine.

In addition to the possible sources of inaccuracies described in the discussion of Selectamine™ material balances, another source of error could be present. The particulate-, as well as the vapor-phase metals, was collected during the sampling of the sweet syngas stream. The particulate loading was found to be quite low, but measurable. However, the amount of particulate collected was too small to analyze for the metals content. Thus, the total metals concentrations reported for the sweet syngas are low because the particulate contributions are not included. However, the stack gas composition does include the particulate matter concentrations. Thus, the closures around the turbine are inherently high. However, with the available data, it is not possible to assess the magnitude of the impact of any particulate in the sweet syngas to the material balance closures.

References

1. *A Study of Toxic Emissions from a Coal-Fired Power Plant Utilizing an ESP While Demonstrating the ICCT CT-121 FGD Project.* Prepared for U.S. Department of Energy, Contract No. DE-AC22-93PC93253, Radian Corporation, Austin, TX (June 1994).

6

DISCUSSION OF RESULTS

The major results of the testing at LGTI are summarized and briefly discussed in this section. The objectives of the LGTI testing included 1) the development of emission estimates and emission factors, and 2) the estimation of substance removals across some of the process units within the plant. Emission factors and substance removals are presented in this section of the report. Additionally, the fates and distributions of some selected inorganic and organic substances in the process are discussed.

Sampling of the hot synthesis gas was conducted in the spring of 1995. Although some results of this testing have already been described in other sections of this document, the hot gas and solids compositions are summarized and discussed in this section.

Fates and Distributions of Metals

The distribution of metals, chloride, fluoride, and sulfur are summarized in Tables 6-1, 6-2, and 6-3. These inorganic substances cannot be destroyed in the LGTI process, so they must exit the plant in one of the emission or discharge streams. In developing these distributions, it was assumed that there was no accumulation of these substances within the plant. As discussed in Section 5, there may indeed be some accumulation of some of the metals in the Selectamine™ system may occur; however, this could not be definitively determined.

The material balance envelope was drawn around the total plant, as shown in Figure 5-1. The slurry preparation, char dewatering, and slurry charge system were encompassed by the boundaries of the envelope, so the streams associated with these systems were not included as inlet or outlet streams in the balances or as destinations for substances of interest. In addition to the incoming coal and natural gas streams, there are other streams that come in to the plant which were not considered in the mass balances or definition of fates. These streams include the oxygen fed to the gasifier, the demineralized water sent to the scrubber, and the ambient combustion air used in both the incinerator and gas turbine. None of these streams are thought to contain significant amounts of the substances of interest. The outlet streams consist of the gasifier slag, sulfur byproduct, sweet water (from the sour water stripper), incinerator exhaust gas, and the turbine exhaust gas. Flow rates of the process streams were steady throughout the testing, and there was no evidence of significant bias in any of the reported flow rates.

Many of the metals were not detected in several of the various feed and discharge streams. In these cases, the analytical detection limits were used as an estimate of the substance

Table 6-1
Elemental Flows Around LGTI Process

Analyte	Overall Flows and Closure[a]							
	Coal Feed Flow, lb/hr	NG Flow, lb/hr	Slag Flow, lb/hr	Sulfur Flow, lb/hr	Sweet Water Flow, lb/hr	Incin Flow, lb/hr	GT Flow, lb/hr	Material Balance Closures, Out/In, %
Chloride	5.3		0.83		0.075	0.09	2	57
Fluoride	10		2.0		0.16	0.0012	0.1	22
Sulfur	380		3.0	240		38	170	119
Aluminum	850	2.1e-05	900	3.8e-03	4.2e-02	3.0e-03	2.0e-01	107
Antimony	0.02	3.1e-08	0.011	7.2e-04	6.5e-03	9.0e-05	1.1e-02	213
Arsenic	0.13	1.2e-07	0.059	7.2e-04	3.2e-04	2.9e-05	5.6e-03	50
Barium	49	6.8e-08	59	4.8e-04	4.5e-02	7.8e-05	9.5e-03	119
Beryllium	0.04	5.0e-08	0.034	4.8e-04	5.0e-05	2.7e-06	2.0e-04	95
Boron	4.3	7.1e-06	3.5	2.4e-03	3.3e-03	5.8e-04	2.4e-02	82
Cadmium	0.01	6.3e-07	0.002	4.8e-04	4.2e-04	8.3e-05	7.7e-03	74
Calcium	1,400	7.4e-05	1,600	4.7e-03	2.2e-01	3.6e-03	5.6e-01	111
Chromium	0.64	2.6e-06	0.76	9.6e-04	7.4e-04	1.6e-04	7.1e-03	120
Cobalt	0.26	9.4e-07	0.26	9.6e-04	3.5e-04	1.6e-05	1.5e-03	101
Copper	1.6	8.1e-08	1.5	4.8e-04	1.2e-03	1.1e-04	4.0e-02	99
Iron	330	1.0e-05	370	2.2e-03	1.0e-01	8.6e-03	3.9e-01	113
Lead	0.18	6.2e-06	0.03	7.2e-04	2.8e-02	9.3e-05	7.6e-03	55
Magnesium	300	1.9e-05	330	9.6e-04	8.5e-02	1.0e-03	7.9e-02	109
Manganese	1.3	8.3e-08	1.3	4.8e-04	2.0e-04	4.1e-04	8.0e-03	99
Mercury	0.01	6.0e-07	2.0e-04	2.3e-05		1.2e-03	3.4e-03	25
Molybdenum	0.07	2.4e-07	0.075	4.8e-03	9.7e-04	2.2e-04	1.8e-02	132
Nickel	0.21	1.3e-06	0.38	9.6e-04	1.8e-03	2.2e-04	1.0e-02	188
Phosphorus	40	4.5e-05	41	0.0e+00	2.1e-02	7.3e-03	7.0e-01	103
Potassium	28	3.3e-04	27	4.8e-03	7.7e-02	1.3e-02	9.4e-01	98
Selenium	0.45	2.4e-07	0.13	5.8e-03	2.7e-03	9.8e-06	8.0e-03	56
Silicon	1,500	4.1e-05	1,600	4.8e-03	6.1e-01	2.4e-03	1.9e-01	108
Sodium	140	4.8e-05	160	4.8e-03	3.2e-01	9.0e-03	1.0e+00	117
Titanium	78	5.7e-07	80	4.8e-04	1.7e-03	6.2e-05	1.6e-02	103
Vanadium	1.7	5.6e-08	1.7	4.8e-04	3.8e-04	5.5e-05	2.3e-03	102
Zinc	1.1	1.4e-05	0.47	3.5e-03	2.1e-02	1.1e-03	1.3e-01	60

[a] Shaded entries indicate that the substance was not detected at that location. Detection levels were used in computing flows and closures.

Table 6-2
Material Distributions as a Function of Material Input

Analyte	Material Flows. % of Input[a]						
	Coal Feed Flow, %	NG Flow, %	Slag Flow, %	Sulfur Flow, %	Sweet Water Flow, %	Incin. Flow, %	GT Flow, %
Chloride	100	0	16	0	1.4	1.7	38
Fluoride	100	0	23	0	1.5	<0.1	1
Sulfur	100	0	0.79	63	<0.1	10	46
Aluminum	100	<0.1	110	<0.1	<0.1	<0.1	<0.1
Antimony	100	<0.1	65	4.4	40	0.55	63
Arsenic	100	<0.1	45	0.55	0.24	<0.1	4.3
Barium	100	<0.1	120	<0.1	<0.1	<0.1	<0.1
Beryllium	100	<0.1	93	1.3	0.13	<0.1	0.68
Boron	100	<0.1	81	<0.1	<0.1	<0.1	0.55
Cadmium	100	<0.1	15	3.5	3.1	0.61	56
Calcium	100	<0.1	110	<0.1	<0.1	<0.1	<0.1
Chromium	100	<0.1	120	0.15	0.12	<0.1	1.1
Cobalt	100	<0.1	100	0.37	0.14	<0.1	0.6
Copper	100	<0.1	97	<0.1	<0.1	<0.1	2.5
Iron	100	<0.1	110	<0.1	<0.1	<0.1	0.12
Lead	100	<0.1	16	0.4	15	<0.1	4.2
Magnesium	100	<0.1	110	<0.1	<0.1	<0.1	<0.1
Manganese	100	<0.1	99	<0.1	<0.1	<0.1	0.6
Mercury	100	<0.1	1.4	0.16	<0.1	8.2	23
Molybdenum	100	<0.1	100	6.5	1.3	0.3	25
Nickel	100	<0.1	180	0.46	0.88	0.11	4.9
Phosphorus	100	<0.1	100	<0.1	<0.1	<0.1	1.7
Potassium	100	<0.1	94	<0.1	0.27	<0.1	3.3
Selenium	100	<0.1	49	2.1	0.6	<0.1	1.8
Silicon	100	<0.1	110	<0.1	<0.1	<0.1	<0.1
Sodium	100	<0.1	120	<0.1	0.23	<0.1	0.73
Titanium	100	<0.1	100	<0.1	<0.1	<0.1	<0.1
Vanadium	100	<0.1	100	<0.1	<0.1	<0.1	0.13
Zinc	100	<0.1	44	0.33	2	0.1	12

[a] Shaded entries indicate the substance was not detected at that location. Detection levels were used to compute material distributions.

Table 6-3
Material Distributions as a Function of Material Output

Analyte	Material Flows. % of Output[a]						
	Coal Feed Flow, %	NG Flow, %	Slag Flow, %	Sulfur Flow, %	Sweet Water Flow, %	Incin. Flow, %	GT Flow, %
Chloride	180	<0.1	28	<0.1	2.5	3	67
Fluoride	450	<0.1	89	<0.1	6.8	<0.1	4.5
Sulfur	84	<0.1	0.7	53	<0.1	8.4	38
Aluminum	94	<0.1	100	<0.1	<0.1	<0.1	<0.1
Antimony	57	<0.1	37	2.5	23	0.3	37
Arsenic	200	<0.1	90	1.1	0.5	<0.1	8.5
Barium	84	<0.1	100	<0.1	0.1	<0.1	<0.1
Beryllium	110	<0.1	98	1.4	0.1	<0.1	0.7
Boron	120	<0.1	99	0.1	0.1	<0.1	0.7
Cadmium	130	<0.1	19	4.5	3.9	0.8	72
Calcium	90	<0.1	100	<0.1	<0.1	<0.1	<0.1
Chromium	83	<0.1	99	0.1	0.1	<0.1	0.9
Cobalt	99	<0.1	99	0.4	0.1	<0.1	0.6
Copper	100	<0.1	97	<0.1	0.1	<0.1	2.5
Iron	88	<0.1	100	<0.1	<0.1	<0.1	0.1
Lead	270	<0.1	45	1.1	42	0.1	12
Magnesium	91	<0.1	100	<0.1	<0.1	<0.1	<0.1
Manganese	100	<0.1	99	<0.1	<0.1	<0.1	0.6
Mercury	300	<0.1	4.1	0.5	0.1	25	71
Molybdenum	74	<0.1	76	4.8	1	0.2	19
Nickel	53	<0.1	97	0.2	0.5	0.1	2.6
Phosphorus	98	<0.1	98	<0.1	0.1	<0.1	1.7
Potassium	100	<0.1	96	<0.1	0.3	<0.1	3.4
Selenium	300	<0.1	89	3.8	1.8	<0.1	5.3
Silicon	93	<0.1	100	<0.1	<0.1	<0.1	<0.1
Sodium	86	<0.1	99	<0.1	0.2	<0.1	0.6
Titanium	97	<0.1	100	<0.1	<0.1	<0.1	<0.1
Vanadium	98	<0.1	100	<0.1	<0.1	<0. 1	0.1
Zinc	170	<0.1	75	<0.1	3.3	0.2	21

[a] Shaded entries indicate the substance was not detected at that location. Detection levels were used to compute material distributions.

concentrations. In cases where this was done, the corresponding entry in Tables 6-1 through 6-2 was shaded to indicate that the value was calculated using the detection limit. Where calculated flows represent less than 0.1% of the total flow they are shown as such.

In Table 6-1, the mass flows of the individual inorganic substances in each stream are summarized. The mass balance closures around the entire plant are also shown for each of the substances. The two dominant streams are the coal fed to the unit and the slag leaving the gasifier. The natural gas contributes only negligible quantities of inorganic substances to the overall balance. Essentially all of these substances entering the system come in with the coal, and most of the inorganic substances leaving the plant are concentrated in the slag.

The distribution of the inorganic substances among the inlet and discharge streams are expressed as percentages of the incoming material (Table 6-2) and the discharged material (Table 6-3). In Table 6-2, the amount of each chemical substance in the discharge streams is expressed as a percent of the amount in the coal, so the sum of the percentages in the outlet streams can sum to more than 100 percent. In Table 6-3, on the other hand, the amount of each of the substances in each of the discharge streams is expressed as a percentage of the total amount of the substance in all of the discharge streams. Therefore, the percentages in the discharge stream sum to 100 percent.

The majority (19 of 29) substance material balances around the entire plant close within the targeted range of 70-130 percent. A few of the substances under discussion are either not concentrated almost exclusively in the slag, are present at significant levels in streams other than the slag, or manifest poor closures. These substances are considered further in the following discussion.

Antimony

The apparent closure for the antimony material balance is over 200%, but the distribution of this element is very uncertain, and the closure cannot be given a great deal of credence. Only a relatively small amount (equivalent to 0.02 lb/hr) of antimony is present in the coal. As shown in Table 6-3, the amounts of antimony in the sweet water and the turbine stack appear to be about the same as those in the slag. The problem may lie in the fact that these latter amounts were calculated using the detection limits as concentrations, since antimony was not detected in the sweet water nor in the gas turbine exhaust. Actual levels could be substantially lower, which would improve the balance closures.

The QA/QC results indicate the possibility of antimony concentrations being biased low in the sweet water stream and in the vapor-phase metals as measured with charcoal. Directionally, however, these biases would tend to show lower recoveries than were actually achieved.

Nickel

The mass balance closure for nickel is also high at nearly 200 percent. Almost all of the nickel, about 96% of the output, is concentrated in the slag. Based on the closure, it appears that either the coal or the slag analyses are inaccurate. The reported nickel concentrations in the coal range from 1 to 3 mg/kg and are reported to only one significant figure. The confidence interval about the average concentration of 1.6 mg/kg encompasses 3 mg/kg. The average concentration of nickel in the slag, on the other hand, is 38 mg/kg with a much narrower confidence interval. It would appear, then, that the nickel concentration in the coal may be biased low.

The possibility of contamination of gas and particulate samples from stainless steel sampling equipment can also exist for some sample sets. However, most of the nickel in the discharge streams is found in the slag. These samples should not be subject to contamination.

Chromium

The closure for chromium is 120 percent. Chromium is often a component of refractory material, and small amounts may shed or be eroded from the refractory and be incorporated into the slag. In this way, chromium may be "generated" in the plant and closures above 100% found. If this gradual shedding/erosion of the refractory occurs at the LGTI plant, it may explain the difficulty in obtaining an accurate chromium material balance. The reported chromium distributions, however, may be correct in spite of the high material balance. Chromium is also a constituent of stainless steel and could be present as a contamination in some samples. However, almost all of the chrome is discharged from the plant in the slag, so contaminant is not a likely cause of the somewhat high material balance closure.

Arsenic

Only about 50% of the arsenic found in the coal is accounted for in the discharge streams of the plant. About 90% of the 50% found is contained in the slag. There is no apparent reason for the discrepancy in the material balance closure. The arsenic analyses in both the coal and in the slag appear to be reasonably consistent. It is possible that some arsenic accumulates in the Selectamine™ system. The results of the QA/QC program have also raised the possibility that the measured levels of arsenic in the coal may be biased slightly high.

Lead

About one-half of the lead found in the coal is unaccounted for in the plant discharges. Of the 50% accounted for, about a third is included with the slag, and about two-thirds in the sweet water stream. Only a relatively small fraction leaves in the turbine stack exhaust. While the concentration of lead in the coal is low, the analytical results are quite consistent, and the variability is low. The concentrations of lead in the sweet water were variable, but this variability was not excessive. The cause of the poor overall balance is not known.

Mercury

Determining the fate of mercury in any coal combustion system is almost always a difficult task. The LGTI plant was no exception. Only about 25% of the mercury reported in the coal was accounted for in exit streams. Of that 25% identified, about 80% was in the turbine exhaust, and another 15% was discharged in the incinerator stack gas. The average mercury concentration in the coal was about 0.11 μg/g, and the concentrations were quite consistent for all nine coal samples.

The mercury levels in the turbine and incinerator stack gases were about the same concentration range, 0.010-0.015 $\mu g/Nm^3$. The 95% confidence interval is about 0.01 $\mu g/Nm^3$ for the turbine exhaust analysis and about 0.02 $\mu g/Nm^3$ for the incinerator stack gas. Even at the upper limits of the confidence interval, however, only an additional 25% of the incoming mercury would be identified. QA/QC results indicated the possibility of incomplete recovery of mercury from charcoal sorbents, potentially biasing low the measured mercury levels in some internal gas streams. However, the overall balance would not be affected by these internal balances. Thus, the fate of mercury in the LGTI plant was not accurately defined in this testing program.

Selenium

The material balance closure for selenium is low, with only about one-half of the amount in the coal being accounted for in the discharge streams. Most of the selenium that was found in the discharge streams is included in the slag. Selenium is another metal that is often difficult to analyze accurately in many streams and matrices. The measured levels in the coal are reasonably consistent, with the exception of one high value, which was deemed an outlier and excluded when determining the average concentration of selenium in the coal. The variability among the remaining analyses was not unreasonably high and would not account for the large discrepancy in the material balance. The slag analyses were variable, and one very low concentration was excluded as an outlier. While the variabilities of the selenium analyses in the coal and slag were significant, they would not alone be responsible for the poor material balance.

Cadmium

The mass balance closure for cadmium was 74%, within the targeted range. Cadmium is present in the coal closure around the turbine system (Table 5-9) was 159% using the on-line AA cadmium analysis for the sweet syngas composition. This result provides some confirmation of the relatively high fraction of cadmium found in the turbine exhaust. The analyses of cadmium in the particulate phase of the turbine exhaust gas were quite consistent, and the concentration in the particulate was of roughly the same magnitude (on a $\mu g/Nm^3$ basis) as that of the gas-phase levels. The variability of the gas-phase analyses was considerably greater than that of the particulate phase, however, with one of three concentrations being almost an order of magnitude higher than the other two. Even accounting for this uncertainty, the cadmium emitted in the turbine exhaust represents a significant fraction of the cadmium present in the coal being gasified.

Molybdenum

The material balance closure for this metal was 132%, slightly outside the acceptable range. About 22% of the molybdenum found in the coal was measured in the turbine exhaust. Molybdenum was also found in the sulfur byproduct and in the sweet water. According to the QA/QC results, molybdenum levels in the sweet water and other process water streams may be biased low. If this is the case, the calculated closure might be even higher than 132 percent. The material balance around the combined Selectamine™-gas turbine system closed within 84%, using the charcoal samples for the sour syngas and acid gas streams. The analyses of molybdenum in the coal was quite consistent. The molybdenum in the turbine exhaust was found only in the particulate phase: the metal was not detected in the vapor phase. There was a significant level of uncertainty in the particulate analyses. The average concentration was 3.8 $\mu g/Nm^3$, while the 95% confidence interval about the mean concentration was 3.3 $\mu g/Nm^3$.

Zinc

Only 60% of the zinc found in the coal was accounted for in the outlet streams. About 20% of the zinc leaving the LGTI plant appeared to exit in the turbine exhaust. A smaller amount, equivalent to about 5% of the zinc in the coal, was found in the sweet water. The concentrations of zinc measured in both the coal and slag samples were consistent, with relatively low variabilities. The zinc in the turbine exhaust was distributed equally between the particulate and vapor phases. The variabilities of both the vapor-phase and particulate-phase analyses were significant, with the 95% confidence intervals being about one- to two- times as great as the average measured concentrations. The internal zinc balances were almost universally poor, so no confirming information about the accuracy of the discharge stream measurements was available from that avenue. Thus, there is a considerable amount of uncertainty about the fate of zinc in the LGTI process.

Sulfur

The sulfur balance closure around the plant was 118%, well within the desired range. However, given the amount of sulfur present in the system, a somewhat better closure might have been expected. The sulfur content of the coal was low, but the analyses were very consistent. About 95% of the sulfur in the raw syngas was removed in the Selectamine™ unit. Overall, about half of the sulfur present in the coal (and in the acid gas stream from the Selectamine™ unit) was recovered as sulfur byproduct. The remainder left the system in the incinerator and turbine exhausts. The measured levels of H_2SO_4 and SO_2 in the turbine stack are more variable than those in the incinerator stack gas, where about 45% of the incoming sulfur leaves the plant. However, the variabilities are relatively small compared to the levels of sulfur compounds found in the stack gases. The rather low total sulfur removal is probably a result of using a very low-sulfur coal in the LGTI plant. The Selectox™ unit functions best with streams containing high levels of sulfides. The levels of sulfides in the acid gas are quite low at LGTI, so the Selectox™ unit is not particularly efficient in recovering the sulfur from the acid gas. In addition, there is no tail gas treatment system to reduce sulfur levels in the tail gas from the Selectox™ unit.

Chloride

The chloride material balance closure was about 54 percent. This represents the upper boundary of the actual closure, since chloride was not detected in the incinerator and turbine exhausts, and the detection limits were used to estimate amounts in these streams. Internal mass balance closures around the sour water stripper and the gas turbine were also poor, in the range of 200-300 percent. The average measured chloride level in the coal was 39 μg/g, with a standard deviation of 7.4 μg/g. Most of the chloride entering the plant in the coal would be expected to leave the system in the incinerator or turbine exhausts or in the stripped (sweet) water, but the measurements do not support this. Some chloride may also be fused into the slag matrix, and the slag analyzer may produce levels that are biased low. Therefore, a significant fraction of the chloride is unaccounted for in the plant.

Fluoride

The overall plant fluoride balance was poor, with a closure of only 28 percent. The average fluoride level of 66 μg/g in the coal was higher than the chloride content. However, these concentrations are still quite low and are subject to analytical uncertainty and imprecision, as indicated by QA/QC results. The standard deviation of the analysis was 16 μg/g, relatively high but not enough to significantly impact the material balance. Most of the fluoride found in the discharge streams was contained in the slag, with a much smaller amount exiting in the sweet water. The fluoride analyses of the slag were consistent, with a low level of variability. Less than 1% of the fluoride in the coal was found in the incinerator and turbine exhaust streams. The mode by which a substantial amount of fluoride leaves the plant is unknown, although its absence in the gas streams may indicate that the slag analysis was biased low and/or the coal analysis was biased high.

Distribution of Organic Compounds in the LGTI Process

Several of the internal streams as well as the gas and aqueous streams discharged from the plant were sampled for selected organic compounds. The organic compounds for which testing was conducted included aldehydes, volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), and semivolatile organic compounds (SVOCs). The distribution and fates of these substances within the LGTI plant are summarized and discussed below.

Aldehydes

Three aldehydes, acetaldehyde, benzaldehyde, and formaldehyde were found at low levels in several streams within the plant. Table 6-4 is a summary of the mass flows of the individual organic compounds, including the aldehydes, found in the process and discharge streams. Acetaldehyde and formaldehyde were found in most of the gas streams tested for aldehydes. The levels of the aldehydes found in the sweet syngas exiting the Selectamine™ system were higher than those found in the sour syngas entering the Selectamine™ system. It seems unlikely that

Table 6-4
Distribution of Organic Compounds in the LGTI Process

Compound	Raw Syngas (5b) Mass Flow, lb/hr	Sour Syngas Mass Flow, lb/hr	Sweet Syngas Mass Flow, lb/hr	Acid Gas Mass Flow, lb/hr	Tail Gas Mass Flow, lb/hr	Incinerator Exhaust Mass Flow, lb/hr	Turbine Exhaust Mass Flow, lb/hr	Sour Condensate Mass Flow, lb/hr	Sweet Water Mass Flow, lb/hr
Aldehydes									
Acetaldehyde		0.0026	0.039			3.0e-05	0.0026	1.8e-06	
Acrolein									
Benzaldehyde		2.1e-04					0.0043		
Formaldehyde		4.6e-04	6.3e-04			3.6e-05	0.024		
Volatile Organics									
C2 (ppmv)	4.5	2.9	3.3	0.04	0.02				
C3 (ppmv)	0.11	0.11	0.11	0.21	0.16				
C4 (ppmv)									
C5 (ppmv)	0.092	0.092	0.088						
Benzene						1.2e-04	0.0063		
C6 (ppmv)	260	250	230	15	15				
Toluene						4.3e-05			
C7 (ppmv)	8.3	14	3.3	0.11	0.14				
C8 (ppmv)					1.2				
PAHs/SVOCs									
2-Methylnaphthalene		0.017	0.0024			8.5e-07			
Acenaphthene		0.033		0.021	5.5e-04				
Acenaphthylene		0.075	0.0023	0.038		2.7e-07			
Anthracene		0.0024		0.0004					

Table 6-4 (Continued)

Compound	Raw Syngas (5b) Mass Flow, lb/hr	Sour Syngas Mass Flow, lb/hr	Sweet Syngas Mass Flow, lb/hr	Acid Gas Mass Flow, lb/hr	Tail Gas Mass Flow, lb/hr	Incinerator Exhaust Mass Flow, lb/hr	Turbine Exhaust Mass Flow, lb/hr	Sour Condensate Mass Flow, lb/hr	Sweet Water Mass Flow, lb/hr
Benzo(a)anthracene						3.5e-08			
Benzo(e)pyrene						3.4e-08			
Benzo(g,h,i)perylene							2.8e-06		
Benzoic acid						0.0034	0.33		0.0018
Dibenzofuran		0.0064		0.0033	0.0021				
Fluoranthene		0.0023						9.3e-04	5.0e-04
Fluorene				0.0081	1.4e-04				
Naphthalene		2	0.26	1.4	1.1	1.0e-06			
Pentachlorophenol									
Phenanthrene		0.016		0.0029					
Phenol		0.0022						0.084	0.075
Pyrene		0.0029						0.004	0.0021

aldehydes are formed in and/or removed from the Selectamine™ process, so the apparent increase in the mass of these substances is probably due to sampling and/or analytical difficulties.

The amount of acetaldehyde in the sweet syngas stream is reduced by about an order of magnitude as it is combusted in the gas turbine. However, the amount of formaldehyde in the turbine exhaust is about 40 times the mass entering in the sweet syngas. Formaldehyde can be formed as a trace product of combustion in the turbine, especially when natural gas is being fired in the turbine.

The distribution of the aldehydes in the acid gas, Selectox™, and tail gas systems was not measured. A very small amount of acetaldehyde was detected in the sour condensate stream, but the sweet water was not tested for aldehydes, so the fate of the acetaldehyde could not be determined. If it was stripped from the sour condensate in the sour water stripper, it would have been sent to the incinerator in the sour gas stream. Small amounts of acetaldehyde and formaldehyde were detected in the incinerator exhaust stream.

Volatile Organic Compounds

VOCs were measured by two different methods. In one method, samples of gas were collected in Tedlar® bags and analyzed on site using gas chromatography. This method provides results where compounds are grouped essentially by boiling point ranges and reported as fractions designated by the numbers of carbon atoms in the molecules (i.e., C_2 compounds, C_3 compounds, etc.). The other measurement method was the VOST method where VOCs are collected on an adsorbent and later eluted into a GC/MS to identify and quantify individual compounds (i.e., benzene, toluene, etc.).

Only small amounts of the C_2 - C_5 compounds were found in the syngas streams, with the maximum being 4.5 lb/hr in the raw syngas. The amount of C_6 compounds, however, in the raw syngas was 260 lb/hr. Most of the C_6 material is comprised of benzene produced by pyrolysis or reaction during the gasification process. There is little difference in the VOC content of the raw and sour syngas, so it does not appear that this material was condensed during scrubbing and cooling.

The level of the C_2,-C_5 VOC fraction in the sweet syngas is similar to that of the sour syngas, so there appears to be little if any removal of this fraction in the Selectamine™ process. However, the amount of C_6 VOC in the syngas stream decreased by about 20 lb/hr across the Selectamine™ absorber. Any VOC absorbed in the absorber will be stripped from the solvent in the stripper, and will appear in the acid gas stream. In the case of the C_6 fraction, about 15 lb/hr was measured in the acid gas stream. The presence of a trace amount of benzene in the incinerator exhaust gas provides additional evidence of the presence of some benzene in the acid gas stream.

The amount of the C_7 fraction decreases from about 14 lb/hr in the sour syngas to about 3 lb/hr in the sweet syngas. It is feasible that some components of this heavier fraction may have been absorbed in the Selectamine™ solvent. However, a corresponding increase in the C_7 content of the acid gas was not measured. Since the accumulation of approximately 10 lb/hr of the C_7 VOCs in the Selectamine™ system is implausible, a measurement error in either the sour syngas, sweet syngas, or acid gas streams is suspected. A significant amount of the C_7 fraction was measured in the raw syngas stream, adding credence to the levels measured in the sour syngas. The amount of C_7 found in the tail gas is similar to the level in the acid gas, giving some rough confirmation of the reported C_7 levels in the acid gas . It seems most likely, then, that the C_7 measured in the sweet syngas was low.

A small reduction in the amounts of most of the VOC across the Selectox™ system were noted. This is reasonable, since about a third of the acid gas is sent through the catalytic oxidation reactor of the Selectox™ unit. Some of the VOCs can be destroyed in this reactor.

Both the turbine and incinerator appear to be very efficient in destroying VOCs. Only very small residuals of benzene and toluene were found in the incinerator exhaust. A small amount of benzene was also detected in the turbine exhaust, but toluene was not found.

Polycyclic Aromatic Hydrocarbons/Semivolatile Organic Compounds

Several PAHs and SVOCs were detected in some of the plant streams. Naphthalene was found in several of the internal process streams at levels that were significantly higher than any other of the detected PAHs/SVOCs. These compounds generally appear to behave in similar fashions in the plant. Since naphthalene levels are highest and are most easily followed through the plant, this compound will be used an indicator for all of the PAHs and SVOCs in describing the distribution of these compounds in the process streams.

The amount of naphthalene in the syngas stream decreased significantly across the Selectamine™ system. The amount removed in the Selectamine™ absorber is apparently recovered in the stripper and appears in the acid gas stream. All of the acid gas stream is sent through the oxidation reactor of the Selectox™ unit. It might be expected that much of the naphthalene going to the oxidation reactor would be destroyed, while the amount of naphthalene in the unoxidized portion of the acid gas stream would remain virtually unchanged. Unreacted naphthalene would exit in the tail gas. It appears that this occurred, since the amount of naphthalene decreased by about 18% across the Selectox™ unit.

The naphthalene in the tail gas was destroyed in the incinerator; only a very small amount remained in the incinerator exhaust stream. The naphthalene that remained in the sweet syngas was destroyed in the turbine. The other detected PAHs and SVOCs were also destroyed in the incinerator or turbine, with only very small residual amounts left in the exhaust streams. It should be noted, however, that several of the compounds detected in the incinerator and/or turbine stack gas samples were also detected in the associated blank samples. These compounds

were considered to be present only when the differences between the sample and blank concentrations were statistically significant.

There were a few compounds which appeared to be present in suspiciously high quantities compared to most of the other PAHs/SVOCs. These included benzoic acid, bis(2-ethylhexyl) phthalate, and di-n-butylphthalate. The phthalate esters are often found in samples collected for organic compound determination. Potential sources of these plasticizers can include plastic bottles, bags, tubing, etc. all of which are present in the field testing environment. The phthalate levels were not included in Table 6-4.

While benzoic acid was not detected in any of the internal process streams, this compound can be produced from both naphthalene and toluene. Naphthalene, found in the sweet syngas, can be oxidized to phthalic acid anhydride and then decarboxylated to benzoic acid. These reactions can occur with significant yields at temperatures as low as 300°C, so it is not unreasonable to assume that some benzoic acid could be produced by this path during the combustion process. Another general preparation method for carboxylic acids is the oxidation of carbon side chains.on ring compounds. Thus, benzoic acid might be produced by the oxidation of toluene, which is also found in the sweet syngas. This oxidation reaction can be catalyzed by vanadium, which was detected at low levels in the turbine exhaust stream. Benzoic acid may also be potentially produced as a degradation product of some XAD sorbent constituents (XAD sorbent is used as a sorbent in the SVOC sampling).

Small quantities of phenol, pyrene, and fluoranthene were found in the sour condensate. It appears that some fractions of these compounds were removed during the sour water stripping, because the amounts present in the sweet water are reduced compared to levels in the sour water. The stripped fractions would have gone to the incinerator as constituents of the sour gas stream.

Subsystem Removal Efficiencies

The removal efficiencies for some substances across plant subsystems were estimated from the results of the testing. The estimated removal efficiencies for selected substances across some systems are summarized in Tables 6-5 and 6-6. These efficiencies are calculated for each individual system, and are not expressed as cumulative removals for multiple systems. Removals could only be estimated for a limited combination of substances and subsystems. Meaningful inlet and outlet compositions needed to develop removal efficiencies were often unavailable.

Since particulate loadings could not be determined in any of the internal process streams, particulate removal efficiencies could not be estimated. As previously discussed, the vapor-phase metals compositions measured in the sweet syngas are highly questionable, so vapor-phase metals removal efficiencies were not developed for the Selectamine™ system.

Those removal efficiencies that could be estimated are discussed below.

Table 6-5
Removal of Vapor-Phase Trace Elements/Metals Across Scrubber

Charcoal	Metals by Charcoal Adsorption	
	Removal, %	95% CI
Antimony	NC	NC
Arsenic	-22	240
Barium	63	66
Beryllium	NC	NC
Boron	77	73
Cadmium	>77	125[a]
Chromium	-2	99
Cobalt	>16	20[c]
Copper	19	52
Iron	72	87
Lead	65	135
Manganese	72	130
Mercury	NC	NC
Molybdenum	13	28
Nickel	>12	312[a]
Selenium	NC	NC
Vanadium	1	84
Zinc	92	94

NC = Not calculated. One or more measurements needed to calculate removal is not available.

[a] Detection levels were used for outlet concentrations in calculating removals and uncertainties.

Table 6-6
Removal of Selected Compounds Across Systems in the LGTI Plant[a]

Compound	Scrubber	Syngas Cooling	Selectamine™	Selectox™	Incinerator	Turbine	Sour Water Stripper
Ionic Species							
Fluoride							
Cyanide							
Ammonia							
Reduced Sulfur Species							
H_2S							
COS							
Volatile Organics							
C2 (ppmv)							
C3 (ppmv)							
C5 (ppmv)							
Benzene							
C6 (ppmv)							
Toluene							
C7 (ppmv)							
PAHs/SVOCs							
2-Methylnaphthalene							
Acenaphthene							
Acenaphthylene							
Dibenzofuran							
Fluoranthene							
Fluorene							
Naphthalene							
Phenol							
Pyrene							

[a] All values as percent removal.

[b] Removals were estimated using stack gas concentrations that were not blank corrected. Thus, they represent the minimum estimated removals; actual removals were higher, but they cannot be quantitated.

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Scrubber System

Removal efficiencies for vapor-phase metals and a few VOCs could be estimated The vapor-phase metals were measured at the inlet and outlet to the scrubber using both charcoal adsorption and Method 29. Removal efficiencies were calculated from the results obtained with charcoal and are presented in Table 6-5. The inlet and outlet streams of the scrubber are saturated with water, and sampling vapor-phase metals was difficult at both of these locations. As a probable result of the sampling problems, it is not surprising that the estimated removals are quite variable, and their accuracy is very uncertain. Their is no particular pattern of removals that could be discerned from the results.

In a few cases, the metals were detected in the inlet stream but not in the outlet stream. The removal efficiencies were then estimated as being greater than the efficiencies calculated using the detection limit for the outlet stream. Obviously, removal efficiencies could not be calculated when the inlet concentration was not known, and these cases are designated as "NC" (not calculated). The mercury removal efficiency was not calculated because the inlet concentration was in obvious error (much more mercury was found in the inlet stream than was contained in the coal).

VOCs were measured around the scrubber using the on-site GC. C_2, C_5, C_6, and C_7 were detected in the inlet and outlet gas streams. The removal results are mixed, with the C_2 indicating a material gain across the scrubber, and the other three fractions exhibiting an apparent positive removal. Thus, some of the VOC material may have been condensed or absorbed in the scrubber water. However, with the exception of the C_6 fraction, all of the concentrations were quite low, and the apparent removals may have been artifacts. The C_6 concentration was higher than those of the other three fractions by as much as four orders of magnitude. But, as indicated from the estimated removal efficiency, the inlet and outlet concentrations were almost the same.

Syngas Cooling

After passing through the scrubber, the sour syngas stream is cooled to below 200°F before entering the Selectamine™ system. During this cooling process, water is condensed, and some of the gas constituents are also condensed or absorbed in the sour condensate. As shown in Table 6-6, fluoride, cyanide, and ammonia levels in the syngas were reduced significantly. Hydrogen sulfide and carbonyl sulfide concentrations in the sour syngas stream remained essentially constant during the syngas cooling.

Selectamine™ System

The removals of several groups of compounds from the syngas as it passed through the Selectamine™ absorber are presented in Table 6-6. The Selectamine™ system is designed to remove sulfur compounds, primarily H_2S, and it removes about 97% of this compound. Also removed are cyanide, ammonia, and the heavier PAHs. The VOCs are not removed in the Selectamine™ process, with the exception of the C_7 fraction. This latter fraction is present at

very low levels in both the sour and sweet syngas, so the calculated removal is questionable. However, if any of the VOCs were going to be absorbed in the Selectamine™ system, the heavier constituents, like the C_{7s}, would be the most susceptible.

In the Selectamine™ stripper, the absorbed compounds are stripped from the Selectamine™ solvent and all of them, including the C_7 VOC fraction, are found in the acid gas stream which is sent to the Selectox™ unit.

Selectox™ Unit

In the Selectox™ unit, one-third of the acid gas is sent through an oxidation reactor to oxidize the H_2S. The hydrocarbons present in this stream can also be oxidized. As shown in Table 6-6, a partial removal of several of the VOC fractions and PAHs apparently takes place. The C_7 VOC shows a negative removal, but as previously discussed, the levels of this fraction are very low in both the acid gas and tail gas streams (i.e., 2.1 $\mu g/Nm^3$ inlet and 2.7 $\mu g/Nm^3$ outlet). On the other hand, the levels of the PAHs in the acid gas are significant, so the high removals of acenaphthene and fluorene appear to be real.

Incinerator

The compounds present in the tail gas from the Selectox™ unit are oxidized and destroyed in the incinerator. Other streams sent to the incinerator include the sour gas from the sour water stripper and the vent gas (from air pulled across tanks in the process). The VOC and PAH/SVOC content of these streams are probably small (although fluoranthene, phenol, and pyrene were stripped from the sour condensate in the sour water stripper), and removals were estimated using only the acid gas composition as being representative of the inlet concentrations.

Although the removal of only five compounds could be estimated (input and output concentrations were available for only these compounds), their behavior can be viewed as indicators for other substances. Benzene and toluene were not speciated in the acid gas, but the C_6 and C_7 VOC fractions were predominantly benzene and toluene, respectively. They were used to estimate the removal of benzene and toluene.

As shown in Table 6-6, the incinerator is very effective in removing/destroying the organic compounds in the acid gas. Even those compounds like benzene and naphthalene, that are more resistant to oxidation, are very effectively destroyed in the incinerator.

Gas Turbine

Of the PAHs/VOCs detected in the exhaust from the gas turbine, only three were quantified in the sweet syngas, so removals could only be estimated for these three compounds (2-methylnaphthalene, acenaphthylene, and naphthalene). However, all three of these compounds detected in the turbine stack samples were also found in the associated blank samples. The differences between the sample and blank concentrations were not statistically significant for any of the

compounds. The (blank) uncorrected levels of the three compounds in the stack gas were used to provide a quantitative estimate of the minimum removals across the turbine. These estimated minimum removals are reported in Table 6-6. Actual removals are higher but could not be quantified.

Sour Water Stripper

The sour water stripper is designed to strip sulfur and nitrogen compounds from the sour condensate. The composition of the sour condensate was compared to the composition of the sweet water to estimate removals. The sour gas rate and composition could not be accurately measured, so the calculated removals could not be confirmed based on the gas characteristics.

As shown in Table 6-6, the stripper is effective in removing ammonia and cyanide from the sour condensate. Very little of the chloride or fluoride in the condensate was removed by stripping, so these removals were not included in the table.

The heavier organics were also apparently stripped to some degree. About half of the fluorene and pyrene present in the condensate was removed. Only 11% of the phenol was removed, but this compound is difficult to strip from water. The stripped organics would be constituents in the sour gas sent to the incinerator.

Emission Factors

Emission factors for those constituents that were detected in the stack gases of the incinerator and turbine are presented in Table 6-7. Factors were developed for each of the exhaust streams. The sums of the factors for each of the two stack sources are the emission factors for the entire plant.

The emission factors are expressed as pounds per 10^{12} Btu of heat input to the entire plant. The average coal flow rate and coal heat content, as well as the natural gas feeding the incinerator and turbine stack for the test period, were used as a basis for calculating the factors.

The calculation of the emission factors for the incinerator was straightforward, but the determination of turbine emission factors was a little more complex. The composition of the gas was measured in one GT-400 turbine exhaust stack. It was assumed that this represented one-sixth of the total turbine exhaust, since there were six stacks (three associated with the GT-400 turbine and three with the GT-300 turbine).

Emission factors for di-n-butylphthalate, bis(2-ethylhexyl)phthalate, and halogenated hydrocarbons were not included, because the presence of these substances in test samples is almost certainly due to contamination from either field or laboratory operations.

Table 6-7
Emission Factors

	Incinerator			Turbine			Combined Stack Emissions		
	Emission Rate	Emission Factor		Emission Rate	Emission Factor		Emission Rate	Emission Factor	
	lb/hr	lb/10^{12} Btu	95% CI	lb/hr	lb/10^{12} Btu	95% CI	lb/hr	lb/10^{12} Btu	95% CI
Particulate Loading	6.0	3,750	600	19	6,900	200	25	9,100	6,000
Ionic Species									
Chloride	ND	NC	NC	2	1,100	185	2	740	180
Fluoride	ND	NC	NC	0.1	56	22	0.1	38	22
Sulfate	490	306,000	36,000	115	62,000	13,000	610	230,000	20,000
Ammonia as N	NA	NC	NC	1.2	650	430	1.2	440	430
Metals									
Aluminum	0.0022	1.4	0.05	0.2	74	31	0.2	75	31
Antimony	9.0e-05	0.054	0.07	0.01	3.9	5	0.011	4	4.7
Arsenic	2.9e-05	0.018	0.02	0.0056	3	2	0.0056	2.1	1.9
Barium	7.8e-05	0.049	0.02	0.0094	5.1	1.3	0.0096	3.5	1.3
Beryllium	2.7e-06	0.0018	0.0004	2.5e-04	0.92	0.03	2.5e-04	0.09	0.03
Boron	5.8e-04	0.36	0.45	0.024	8.7	10	0.024	8.9	10
Cadmium	8.3e-05	0.052	0.077	0.0077	4.2	4	0.0078	2.9	3.8
Calcium	0.0036	2.2	0.88	0.56	300	260	0.56	210	260
Chromium	1.6e-04	0.096	0.02	0.0071	3.8	0.64	0.0073	2.7	0.63
Cobalt	1.6e-05	0.010	0.01	0.0015	0.81	0.58	0.0015	0.57	0.58
Copper	1.1e-05	0.068	0.04	0.04	21	19	0.04	15	19
Iron	0.0086	5.4	2.3	0.39	210	280	0.4	150	270
Lead	9.3e-05	0.058	0.06	0.0076	4.0	1.5	0.0077	2.9	1.5
Magnesium	0.001	0.63	0.24	0.079	29	21	0.08	30	21
Manganese	4.1e-04	0.25	0.58	0.008	4.3	6.6	0.0083	3.1	6.5
Mercury	1.2e-04	0.74	0.08	0.0034	1.8	0.44	0.0046	1.7	0.43
Molybdenum	2.2e-04	0.14	0.01	0.018	9.9	5.7	0.019	6.9	5.6
Nickel	2.2e-04	0.14	0.05	0.01	5.6	3.6	0.011	3.9	3.6
Phosphorus	7.5	4.7	0.26	0.0062	3.4	8.1	0.014	5.2	8.1
Potassium	0.013	8.1	0.53	0.94	350	430	0.95	350	430
Selenium	9.6e-06	0.0061	0.01	0.008	4.3	1.3	0.008	2.9	1.3
Silicon	0.0024	1.5	0.43	0.19	100	34	0.19	72	34
Sodium	0.0090	5.6	2.3	1	540	80	1	370	79
Titanium	3.8e-05	0.024	0.012	0.016	8.7	8	0.016	5.9	8
Vanadium	5.5e-05	0.034	0.03	0.0022	1.2	0.22	0.0023	0.86	0.22
Zinc	0.0011	0.68	0.53	0.13	72	26	0.13	50	26

Table 6-7 (Continued)

	Incinerator			Turbine			Combined Stack Emissions		
	Emission Rate	Emission Factor		Emission Rate	Emission Factor		Emission Rate	Emission Factor	
	lb/hr	lb/10^{12} Btu	95% CI	lb/hr	lb/10^{12} Btu	95% CI	lb/hr	lb/10^{12} Btu	95% CI
Aldehydes									
Acetaldehyde	2.8e-05	0.017	0.025	0.0047	1.8	1	0.0048	1.8	1.5
Benzaldehyde	ND	NC	NC	0.0079	2.9	3	0.0079	2.9	2.6
Formaldehyde	3.3e-05	0.021	0.008	0.045	17	8	0.045	17	7.5
Volatile Organic Compounds									
Benzene	5.4e-05	0.034	0.08	0.012	4.4	2	0.012	4.4	1.7
Carbon Disulfide	0.0002	0.14	0.04	0.12	45	14	0.12	46	14
Toluene	5.3e-05	0.033	0.027	ND	NC	NC	5.3e-05	0.033	0.02
PAHs/SVOCs									
2-Methylnaphthalene	5.6e-06	2.8e-04	3.4e-04	9.8e-04	0.36	0.55	9.8e-04	0.36	0.55
Acenaphthylene	8.8e-07	6.6e-05	1.6e-04	7.0e-05	0.026	0.0076	7.1e-05	0.026	0.0075
Benzo(a)anthracene	5.2e-08	8.0e-06	8.0e-06	6.2e-06	0.0023	0.0002	6.2e-06	0.0023	0.0002
Benzo(e)pyrene	1.5e-07	1.5e-05	1.7e-05	1.5e-05	0.0055	0.0007	1.5e-05	0.0056	0.0007
Benzo(g,h,i)perylene	2.1e-07	3.1e-05	2.6e-05	2.6e-05	0.0095	0.0005	2.6e-05	0.0096	0.0005
Naphthalene	7.8e-06	9.6e-03	9.6e-03	1.1e-03	0.4	0.12	1.1e-03	0.4	0.12
Benzoic acid	0.0034	0.50	0.49	0.38	140	65	0.39	140	65

[a] ND = Substance not detected in this stream.

[b] NA = Not available. Concentrations of this substance were not measured.

Hot Synthesis Gas Composition

The hot gas was characterized for both vapor-phase species and particulate char at two locations upstream of the particulate scrubber. A preliminary shakedown test was done at a process temperature of 500°F, and the follow-up "hot gas test" was done at a stream temperature of approximately 000°F. Results are discussed in the following paragraphs.

Compositions of the char collected from the syngas at both 900°F and 500°F as well as from the recycle char stream are compared in Figure 6-1. The results are presented in order of increasing concentration. Although the char collected from the syngas was from two different sampling periods, the results are very similar for all of the metals listed. With the exception of phosphorus, the compositions of all three char streams are very similar for the major elements. The recycled char appears to be somewhat enriched in some of the volatile elements. Cadmium and mercury are statistically enriched (the amount of enrichment exceeds the 95% confidence

Figure 6-1
Comparison of Recycled and Gaseous Char

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