interval overlap). Other elements appear to be enriched (arsenic, selenium, and chromium); however, the confidence intervals for these elements overlap, so definitive statements about enrichment cannot be made. It is speculated that the enrichment mechanism is carbon adsorption by the carbon in the char in the relatively cooler and *quenched* environment of the particulate scrubber.

Results for vapor-phase species were not so well defined. Trace elements were collected and measured by both charcoal adsorption and the EPA Method 29 sampling train. Results using these techniques are presented in Figure 6-2. Only those elements whose results were above the detection limit have been graphed. Wide variations are exhibited between methods as well as between the two test locations for most of the elements. As discussed before, this is most likely due to inaccuracies in the sampling methodologies used.

Figure 6-2
Comparison of Vapor-Phase Elemental Results

7

COMPARISON OF METAL MEASUREMENT TECHNIQUES

Most of the EPA standard reference methods used in HAPs test programs for the utility industry were designed for the collection of samples from an oxidized or combustion gas source. In this test program, only the incinerator and turbine exhaust stacks fall into the combustion gas category. All of the internal process streams that were tested were primarily synthesis gas streams which are a reducing gas matrix. This means that trace elements, found primarily in the oxidized form in combustion gas matrices, may also be present in reduced forms such as the hydride or carbonyl, or even in an elemental form. The use of traditional EPA sampling methods, especially Method 29, for the collection of trace elements may not be applicable for the typical internal gas streams found in a gasification process.

With IGCC processes coming into greater use and with the current emphasis on hot gas cleanup processes, accurately characterizing the trace metal content of a syngas matrix is critical. In current IGCC systems, sulfur removal is typically achieved by cooling the hot syngas (which lowers the process efficiency), then removing the H_2S in an amine based absorbing solution, during which, several trace elements are also removed from the syngas. In a hot gas cleanup system, these elements may deactivate catalysts, or may not be removed and subsequently show up as a gaseous emission. The impacts of cleanup systems on trace metals and their ultimate fate requires effective measurement techniques on the hot gas.

Radian's prior experience with EPA Method 29 indicated that it was not an efficient collection technique for most trace elements in a reducing gas matrix. Therefore, two additional techniques were also used for the characterization of trace elements in the reduced gas environment. Quartz tubes containing specially prepared charcoal were used in conjunction with Method 29 at most internal process gas locations. Additionally, a method using a specially modified atomic absorption spectrophotometer to directly measure selected trace elements was used at the sour and sweet syngas locations. The charcoal tube collection technique and the vapor-phase AAS technique are described in Appendix B, Sampling Methodologies.

The average results of the measurements made by these three techniques are compared in Table 7-1. Mercury is not included in this comparison as it is discussed separately in Section 8 of this report. From Table 7-1, zinc is the only metal that is captured in the Method 29 train at levels exceeding those measured by the other two techniques.

Some of the differences in results between the three methods are illustrated in Figure 7-1. Arsenic, chromium, nickel and selenium determined by each method are plotted for the sour

syngas stream. In general, the values determined by VPAAS are the highest, followed by charcoal, with Method 29 giving the lowest results. This information could be interpreted as:

- The VPAAS technique tends to yield the highest results. This is because the technique determines total elemental concentration, regardless of elemental species;
- Elemental adsorption onto charcoal *may* be species dependent, and could explain differences with the VPAAS technique;
- Low values obtained by the Method 29 sampling system are due to poor impinger absorption/ scrubbing efficiency for the nitric acid/peroxide absorbing solutions in a reduced gas matrix.

None of these three methods (Method 29, charcoal, or VPAAS) has been validated for this type of sample matrix; however, the wide range of resulting values from these three techniques suggests that if rigorous testing of the trace metals is required for syngas matrices, EPA Method 29 is not appropriate, and alternate techniques should be used. Ultimately method validation testing should be performed.

Table 7-1
Comparative Trace Element Analysis

	Sour Syngas, µg/M³			Sweet Syngas, µg/M³		
Element	**Charcoal**	**M-29**	**VPAAS**	**Charcoal**	**M-29**	**VPAAS**
Antimony	<1.1	<0.018		<0.039	<0.017	
Arsenic	270	0.5	870	6.0	0.42	<2,200
Barium	6.3	0.064		0.23	0.17	
Beryllium	<0.36	<0.033		<0.013	<0.03	
Boron	100	<4.1		3.2	7.1	
Cadmium	<0.85	0.27	<2.2	<0.031	0.44	9.5
Chromium	93	1.6	142	3.6	1.4	<39
Cobalt	<5.9	0.021		<0.22	0.038	
Copper	46	<0.046		1.8	3.7	
Iron	2,300	6.7	<85	85	7.8	<85
Lead	<0.85	0.75		<0.031	0.33	
Manganese	10	0.018		0.4	<0.017	
Molybdenum	45	0.16		1.6	0.13	
Nickel	17	2.3	500	0.94	1.2	19
Selenium	2.8	0.18	560	0.18	0.26	200
Vanadium	8.3	0.06		0.28	0.05	
Zinc	<3.8	8.71	<2.2	0.37	5.3	<2.2

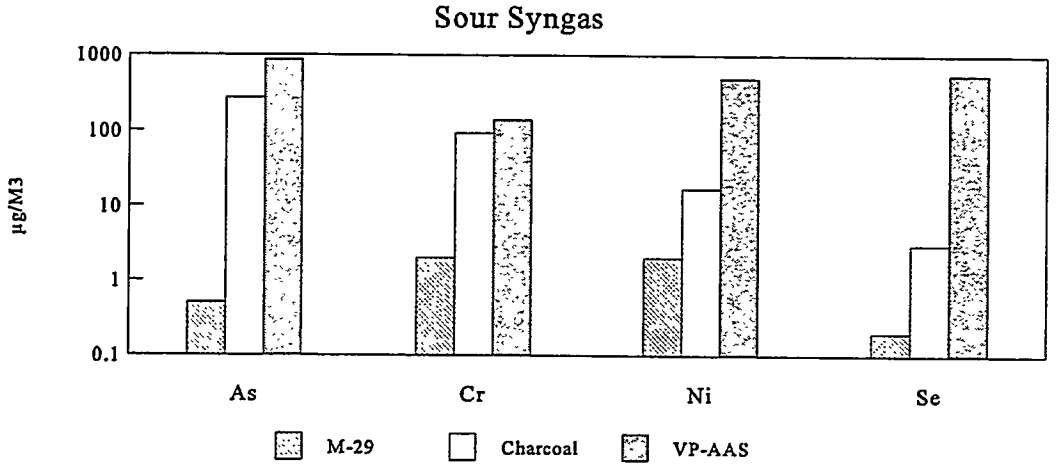

Figure 7-1
Trace Element Methods Comparison

8

MEASUREMENT OF MERCURY IN COAL SYNTHESIS GAS

Integrated gasification combined-cycle (IGCC) technologies represent an advanced power system with the potential for effective mercury emission control prior to combustion. Methods for measuring mercury (and other toxic trace elements) in syngas around existing and developing gas treatment systems are needed to determine the performance characteristics of these systems for mercury removal. However, due to the current focus on methods applicable to oxidized flue gases from conventional power systems, there is little public information available concerning mercury measurements in coal synthesis gas.

Radian Corporation has tested for HAPs at numerous commercial and pilot-scale coal gasification systems. It has been our experience that testing for mercury in the internal process gas streams presents sampling and analytical challenges not found in oxidized flue gas streams typical of conventional coal-fired boilers. Nitric acid/hydrogen peroxide and acidic potassium permanganate impingers commonly used for oxidized flue gases have been used for many years for sampling metals and mercury in syngas; however, poor collection efficiencies have prompted a search for alternative sampling techniques.

During this program, extensive research was performed to investigate alternative mercury measurement methods. Although not conclusive, the results to date are promising.

In Periods 1-3 of testing, this research focused on two gas streams—sour and sweet syngas. In Period 4, the hot, raw syngas was analyzed. Briefly, the following techniques were employed during the first three periods of testing.

- A modified Method 29 train was used (i.e., the acidic potassium permanganate impingers were not used). Prior work has demonstrated that H_2S quickly exhausts the permanganate, which allows mercury to pass through the impingers. This means that the only mercury captured was present in the nitric acid/peroxide impingers.

- A charcoal based absorbent was used to sample vapor-phase mercury.

- Various impinger solution combinations were employed with an on-line CVAAS. Both total and "speciated" mercury forms were measured with this technique.

The results of these tests, while not conclusive, demonstrated that if H_2S is removed from the gas stream, an oxidizing impinger such as potassium permanganate is capable of removing a large fraction of mercury. This observation was put in practice during Period 4 testing. In this test, the

charcoal and Method 29 train (modified by the insertion of a caustic impinger) produced equivalent results.

Discussed below is a description of each method, with an emphasis on the impinger chemistry in a reduced atmosphere. Then, total mercury and speciated results are presented.

Method Descriptions

A full description of the mercury sampling and analytical methods used for Periods 1-3 and Period 4 testing is provided below. Each method's background and applicability, sample preparation activities, analysis, individual results, and quality control activities are discussed.

EPA Method 29

EPA Draft Method 29 consists of a filter substrate, which removes particulate matter, and a series of impingers. It is often assumed that substances collected in the impinger solutions represent vapor-phase metals, however it should be noted that extremely fine particulate matter (<0.2 μm) may also contribute to the amount collected in the impingers if it passes through the filter. This method specifies the use of two impingers containing a 5% HNO_3/10% H_2O_2 solution, followed by two impingers containing a 4% $KMnO_4$/10% H_2SO_4 solution for total mercury collection. Current research is investigating the speciation capabilities of Method 29. Some studies indicate that oxidized forms of mercury preferentially report to the nitric/peroxide impingers and all forms of mercury are trapped in the permanganate impingers.[1] When this method is applied to syngas or other reducing gas matrices containing hydrogen sulfide (H_2S), the permanganate impinger solution is quickly reduced so it is ineffective at collecting mercury. The standard nitric acid solution (5% nitric/10% peroxide) has also been shown to be ineffective in syngas applications for the collection of total mercury and other trace elements.[2]

In an effort to enhance the collection efficiency of the nitric acid impingers for other vapor-phase metals in syngas, the concentrations of nitric acid and hydrogen peroxide were increased to 10% and 30%, respectively. Although the oxidation potential of this solution is enhanced, it will not effectively trap H_2S. The low pH of the impinger solution keeps the H_2S equilibrium shifted towards the gas phase. As such, H_2S will not dissociate so oxidation and removal will not occur. Therefore, permanganate impingers were not used in the sampling trains for the sweet and sour syngas samples collected during the Periods 1-3 test. The potential to collect valid elemental mercury with this configuration is minimal.

The Method 29 approach was modified for the Period 4 test on hot raw syngas by the insertion of two impingers containing a 2 N solution of sodium hydroxide (NaOH) immediately upstream of the acidic $KMnO_4$ impingers. Periods 1-3 test results using the CVAAS system (presented later in this section) indicated that a sodium hydroxide scrubbing solution was effective at removing H_2S from the sample gas and ineffective at collecting mercury.

Due to the high concentration of H_2S in the raw syngas and the limited amount of NaOH available in the scrubbing solutions, a gas sample volume of only 40 dry standard cubic feet (dscf) could be passed through the NaOH impingers before exhausting the NaOH capacity, assuming 100% collection efficiency. To avoid compromising the gas volume targeted for collecting other vapor-phase metals in the HNO_3 impingers, the NaOH and $KMnO_4$ impingers were disconnected from the sampling train after the first 30-40 dscf of sample had been collected. Sampling was continued with the remaining nitric acid impingers until approximately 100 dscf of syngas was collected. The individual gas sample volumes were considered in the calculation of vapor-phase mercury concentrations from each impinger solution during Period 4.

Sample Recovery and Analysis. The nitric acid/hydrogen peroxide impingers were recovered and rinsed into a 1000-mL plastic bottle. At the laboratory, a 50 mL aliquot was taken for mercury analysis by CVAAS (SW7470). Additional potassium permanganate was required to reduce the excess peroxide and render the sample susceptible to complete reduction by stannous chloride. Matrix-spiked and matrix-spiked duplicate samples were prepared and analyzed to assess the accuracy and precision of the digestion and CVAAS recovery and analytical system. Spike recovery from the syngas matrix for Periods 1-3 was 99% and 104 percent. Matrix spikes for raw syngas collected during Period 4 were recovered at 110% and 113 percent. These data indicate excellent analytical accuracy and precision for mercury collected in the 10% HNO_3/30% H_2O_2 solution; however, the data do not reflect the impinger solution's retention efficiency since no spikes were performed prior to sampling.

The caustic solution used for collecting H_2S (Period 4 only) was also collected and rinsed into 1000-mL plastic bottles. Visual inspection of these samples showed precipitation of ammonium carbonate/bicarbonate (a white crystalline powder) and a slight yellow color from the adsorption of sulfide. As expected, carbon dioxide and H_2S, both acid gases, react with NaOH in solution and begin neutralizing the NaOH until the drop in pH impairs ammonia collection. Once an equilibrium pH is established, the continuous addition of ammonia and CO_2 drives ammonium carbonate and/or bicarbonate salts out of solution. The removal efficiency for H_2S is, therefore, affected by the equilibrium pH which is approximately 10. In the laboratory, 50 mL aliquots were taken for mercury analysis by CVAAS (SW7470). The samples were filtered to remove most of the precipitated carbonate salts in order to avoid excessive CO_2 generation in the closed loop of the cold vapor generator. No mercury spikes were performed on this matrix; however, the samples were split and analyzed by the CVAAS system used on-site during Periods 1-3. That system used a gold plug to amalgamate and concentrate mercury vapor driven off from the sample during sample reduction with sodium borohydride, rather than recirculate the mercury vapor in a closed loop. The entire sample was well mixed and added to the gold amalgamation-CVAAS system so any mercury possibly removed by filtration of carbonate salts for the SW7470 test would be accounted for. The results for each sample were comparable by both analytical techniques indicating no detectable loss of mercury associated with the ammonium carbonate.

The acidic $KMnO_4$ impingers used for elemental mercury collection downstream of the NaOH impingers in the Period 4 test were recovered and rinsed into amber glass bottles. The impingers

were rinsed a second time with 8N hydrochloric acid (HCl). These rinses were held in separate glass bottles for transportation to the laboratory where they were combined with their respective $KMnO_4$ impinger samples before analysis. Visual inspection of the samples during recovery, and again prior to adding the HCl rinses showed a loss of the characteristic purple color of the $KMnO_4$ solution. This indicates some sample reduction occurred during sampling and after sample collection, presumably from H_2S passing through the NaOH solution. Reduction of $KMnO_4$ during sample collection could bias the results low due to a reduction in the solution's oxidation potential and collection efficiency.

Similar to the NaOH samples, the $KMnO_4$ impinger samples were split and analyzed by both the SW7470 and the gold amalgamation-CVAAS systems. The initial matrix spikes of the $KMnO_4$ samples were recovered by SW7470 at 212% and 220%; however, spike levels were well below the concentration measured in the parent sample. This spiking error accounts for the poor recovery calculated. Analytical spikes at approximately 30% of the parent sample concentration were subsequently performed with recoveries of 52% and 47 percent. These spike recoveries indicate that mercury measured in the $KMnO_4$ impingers may be biased low, however parallel results by the gold amalgamation-CVAAS system were in close agreement.

Since the analytical results from SW7470 compared favorably with the gold amalgamation-CVAAS results and were supported by a standard reference method, they were used for calculating the vapor-phase mercury concentrations in each impinger solution. The results for each impinger sample are reported in Appendix Table G-1.

Charcoal Sorbents

Some industrial processes utilize charcoal sorbents in guard beds to protect catalysts from metal poisoning. Using the same principle, charcoal has also been demonstrated as a suitable sorbent for the collection of total mercury in flue gas.[3,4] In adapting this method to syngas, Radian takes coconut-based charcoal (20-40 mesh) subjected to an aggressive cleaning procedure using concentrated nitric acid. The charcoal is soaked in nitric acid overnight at 80°F and then the hot acid is decanted off and fresh nitric acid is added. This cycle is repeated for five consecutive days before the charcoal is rinsed with ultra-pure (Milli-Q) deionized water. The rinsed charcoal is then dried overnight at 150°C before being loaded into precleaned quartz tubes. The charcoal is held in place by plugs of pre-cleaned quartz wool.

For sampling, two charcoal tubes were placed in series using Teflon® tubing and plastic connectors. A total volume of 100 L of syngas was sampled through the tubes at ambient temperature at a maximum flow rate of 1 L/min. After sample collection, the charcoal tubes were sealed with plastic caps and sent to the laboratory for analysis. The charcoal sorbent was digested with nitric acid in a closed microwave digestion vessel to minimize the potential losses of volatile elements that might occur with open-vessel digestion techniques. This digestate was analyzed for mercury by CVAAS (SW7470).

During Period 4 sampling of the hot raw syngas, the addition of a condensate trap upstream of the charcoal sorbent tubes was required to remove excessive moisture from the syngas sample. The condensate collected during the entire charcoal sampling period was retained for analysis by SW7470. Sample analysis indicated 1.3 μg of mercury was retained in the condensate which was approximately 7.5% of the total mercury collected in the three pairs of sample tubes combined. The individual sample results presented in Appendix Table G-1 include the mercury collected in the condensate which was divided proportionately by sample volume across the three sample runs.

To assess the mercury recovery from the charcoal digestion procedure, blank charcoal media was spiked before digestion with a commercially-prepared aqueous standard solution. Duplicate spikes of blank charcoal tubes prepared for Periods 1-3 at 1 μg were recovered at 52 and 62 percent. This spiking regimen was repeated for Period 4 and both spikes were recovered at 75 percent. Analytical spikes introduced in the sweet syngas sample digestates (Periods 1-3) were recovered at 77 and 81 percent. Blank media was analyzed to provide a measure of background concentrations for correction of the sample results. Three blanks were analyzed with concentrations ranging from 0.08-0.10 μg mercury per tube. Blank media analyzed for Period 4 averaged 0.003 μg mercury per tube and was insignificant compared to the sample concentrations. Based on these quality control results, Periods 1-3 mercury data from the charcoal tube sampling method may be biased low. Quality control results for Period 4 indicate better analytical performance.

Gold Amalgamation-CVAAS

Method Background. For the past several years, Radian has participated in numerous investigations studying mercury (and other HAP) emissions from coal burning utility facilities. Initial work involved the determination of total mercury emissions; but more recently, attempts have been made to determine the oxidation state of mercury in the flue gas. Research has concentrated on separating the elemental (Hg^0) and oxidized (mono- and di-valent) forms of inorganic mercury in the gas during sampling.

Radian has conducted research studying possible ways to speciate mercury in coal-combustion flue gases using both classical and novel sampling methods.[5] This has included EPA Method 29 in which the solution components have been modified in attempts to separate the oxidized and elemental forms of mercury. As indicated earlier, results have shown that oxidized forms of mercury can be effectively trapped in most aqueous solutions. The very low aqueous solubility of elemental mercury enables it to pass through nearly all solutions, with the exception of strongly oxidizing solutions such as potassium permanganate. This solubility difference has been exploited by using appropriate impinger combinations to selectively trap, and thus separate, the different forms of mercury.

The use of such separation impingers has been coupled with cold vapor atomic absorption (CVAAS) spectrophotometry to create a semicontinuous method to measure and speciate mercury from an industrial gas stream. A sample is purged through an impinger train which may

either trap various mercury species or reduce them all to the elemental form, depending upon the desired results. The mercury from the sample is then concentrated by amalgamating it on a gold surface downstream of the impinger train. The amalgamated mercury is released from the gold by thermal desorption and carried by an inert carrier gas to an atomic absorption spectrophotometer for analysis.

Advantages of this method include the low detection limits made possible by the amalgamation method (10-15 ng Hg per sample) as well as the ability to analyze mercury on a semicontinuous basis. The latter avoids the problems associated with mercury sample shelf-life since the analysis can be carried out immediately following sampling. In addition, the sampling method uses small-volume fritted impingers which enables smaller sampling volumes and shorter sampling times.

Laboratory tests have verified that the impinger-CVAAS method can effectively separate and measure elemental and oxidized forms of mercury from simulated flue gas streams.[5] In the context of oxidized flue gas, these tests have included an investigation into the effects of potential interferents such as SO_2, O_2 and HCl gases. The presence of reducing species, such as SO_2, may result in chemical reactions within the impinger solutions, resulting in the loss of captured mercury and thus, inaccurate results.[6] Solution modifications have been made to minimize the absorption of interferents such as SO_2. One example is the use of non-aqueous solutions, such as isopropyl alcohol (IPA), which can also be used in combination with mercury-binding chelants, such as diethyldithiocarbamic acid (DEDTC).

Sampling. Process gas was sampled from the LGTI sour and sweet syngas streams during Periods 1-3 and analyzed on a semicontinuous basis using a gold amalgamation-cold vapor atomic absorption (CVAAS) method. The sampling and analytical setup consisted of an impinger train, a gold amalgamation unit, CVAAS instrumentation, two gas scrubbing units and a gas volume measuring unit. A diagram of the gold amalgamation-CVAAS sampling system is shown in Figure 8-1.

Sweet and sour syngas samples were transferred from the sampling points to the impinger sampling train via Teflon® tubing. Care was taken to maintain the temperature of the sample stream to avoid water condensation within the sample line. A continuous flow was also maintained through the sample line by venting the sample back into the process stream when sampling was not actively being performed.

In general, sample gas was passed through the impinger train where either 1) all forms of mercury were reduced to the elemental form, 2) oxidized and elemental forms of mercury were selectively retained in the impinger train, or 3) oxidized forms were retained while elemental mercury was passed on to the gold amalgamation unit. Depending on the configuration, mercury was either allowed to concentrate on the gold trap during sample collection, or it was later purged with argon from the collecting impinger solutions after the introduction of a reducing agent (sodium borohydride). The sample gas was vented through additional scrubbing solutions before being metered with a dry gas meter. After the desired sample volume was delivered,

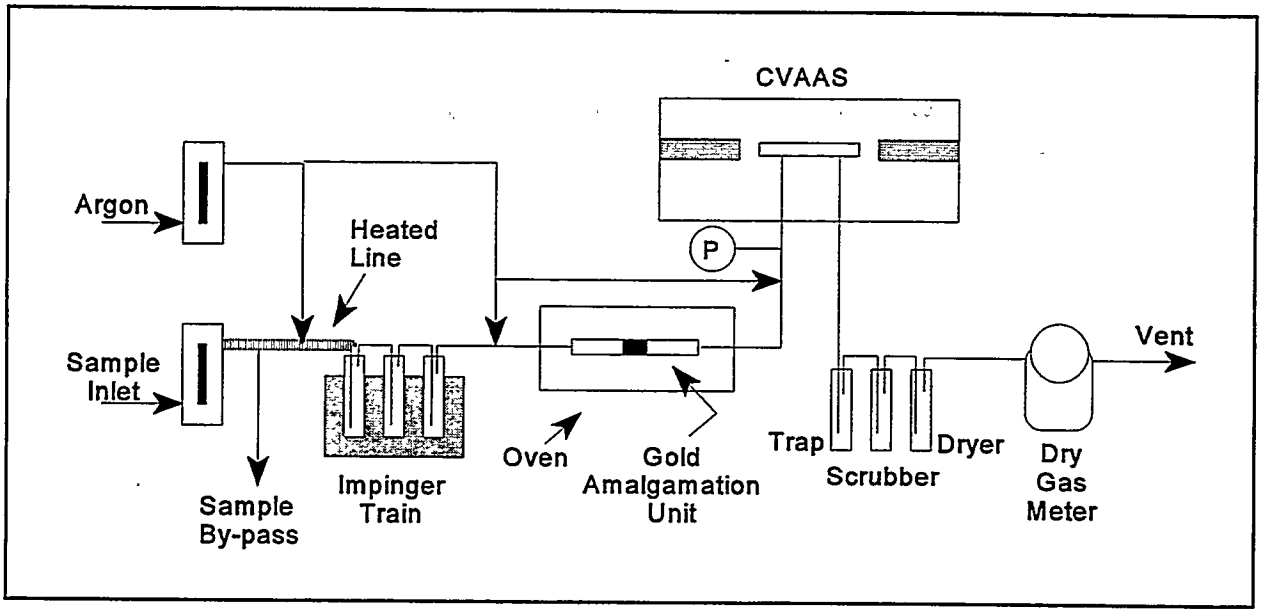

Figure 8-1
Schematic of CVAAS Sample Analysis System

enough argon was passed through the impingers to purge any remaining gaseous mercury through the system.

The various configurations of impinger solutions used to collect mercury are listed in Table 8-1. In most cases, the volume of solution in each impinger was 35 mL and the sample flow rate ranged between 0.75-1.5 L/min. As indicated in Table 8-1, the solutions in the impinger train serve a variety of functions. By varying the impinger configurations, the analyst can effectively collect or convert total mercury, or speciated mercury before the gas reaches the gold amalgamation unit where elemental mercury is trapped and concentrated.

The gold amalgamation unit consisted of a gas-permeable gold-mesh plug housed within a half-inch quartz tube. The tube was located in a temperature-controlled tubular oven capable of reaching a temperature of 950°F. During sample collection, the gold plug was maintained at ambient temperature to permit amalgamation of mercury either passing through the impinger train, or being generated from it after being retained in one or more collecting solutions. Once the sample was collected, the gold trap was heated to 750°F to thermally desorb the mercury from the gold where it passed to a flow cell held in the light path of an atomic absorption spectrophotometer (AAS).

The spectrophotometer used in this test was an ARL Model 93 AAS equipped with a 20-mL cold vapor flow cell through which the sample gas flowed for analysis. Mercury absorbance was measured at 253.3 nm and the results were recorded on an HP3390A integrator.

The gas scrubbing unit located downstream of the flow cell consists of separate impingers containing 1 N NaOH and 4% $KMnO_4$ solutions to scrub H_2S and Hg from the waste gas, respectively. A dry gas meter measured the volume of gas sampled. Calibrated flow meters were used to control the flow rates of sample and purge gases through the system.

Analysis. Total mercury concentrations were determined by flowing gas samples through a reducing impinger, such as sodium borohydride ($NaBH_4$) or stannous chloride ($SnCl_2$). These impinger train configurations are identified in Table 8-1 as Configurations No.1, 2, 2a, and No. 9, respectively. Here, all of the mercury present in the sample was reduced to the elemental form and passed directly to the gold amalgamation unit.[7]

Table 8-1
Gold Amalgamation-CVAAS Impinger Train Configurations

Configuration No.	Impinger Solutions				Function
	Impinger 1	Impinger 2	Impinger 3	Impinger 4	
1	50 mM $NaBH_4$	NA	NA	NA	Total Hg reduction to Hg^0
2	0.1 N NaOH	50 mM $NaBH_4$	NA	NA	Remove H_2S prior to total Hg reduction to Hg^0
2a	2M NaOH	50 mM $NaBH_4$	NA	NA	Remove H_2S prior to total Hg reduction to Hg^0
3	4% H_2O_2/5% HNO_3	0.1N NaOH	cold knockout	4% $KMnO_4$ /10% H_2SO_4	Collect oxidized and elemental Hg separately with H_2S removal
4	80% IPA / 0.1M DEDTC	0.1N NaOH	cold knockout	4% $KMnO_4$ /10% H_2SO_4	Collect oxidized and elemental Hg separately with H_2S removal
5	80% IPA / 0.1M DEDTC	0.1N NaOH	4% $KMnO_4$/10% H_2SO_4	NA	Collect oxidized and elemental Hg separately with H_2S removal
6	4% H_2O_2/5% HNO_3	cold knockout	4% $KMnO_4$ /10% H_2SO_4	NA	Collect oxidized and elemental Hg separately without H_2S removal
7	4% H_2O_2/5% HNO_3	50 mM $NaBH_4$	NA	NA	Collect oxidized Hg and reduce penetrated Hg
8	80% IPA / 0.1M DEDTC	50 mM $NaBH_4$	NA	NA	Collect oxidized Hg and reduce penetrated Hg
9	Sat'd $SnCl_2$ / 0.5N H_2SO_4	NA	NA	NA	Total Hg reduction to Hg^0

Collecting impingers, such as hydrogen peroxide (H_2O_2) or the combination of isopropyl alcohol (IPA) and diethyldithiocarbamic acid (DEDTC), were used in some tests to separate oxidized mercury species from the elemental form. In these tests (Configurations No. 7, 8), the amount of mercury that passed through the impinger was analyzed, as described above, as elemental mercury. The amount of mercury captured in the impinger was determined by reducing the collecting solution with 50 mM $NaBH_4$ and purging the released mercury over the cooled gold following the elemental mercury analysis. The measured amount was reported as "oxidized" mercury.

In some cases, a potassium permanganate ($KMnO_4$) solution was used to capture the elemental mercury (or other forms of mercury) that passed through the initial oxidizing impinger (Configurations No. 3,4,5,6). After the sample was obtained, the $KMnO_4$ solution was neutralized with hydroxylamine sulfate ($(NH_2OH)_2 \cdot H_2SO_4$) prior to reduction with $NaBH_4$. The sample was then purged and analyzed, as described above. Mercury measured in the $KMnO_4$ impingers was reported as elemental mercury.

Results

In addition to determining vapor-phase mercury concentrations in the sweet and sour syngas streams, the testing described in this section was conducted for the purpose of indicating differences between various collection techniques for mercury. Conventional mercury and vapor-phase metal sampling methods have proven unsatisfactory or unworkable for a reducing coal-syngas matrix. In the process of using and screening multiple measurement techniques, it is expected that a more accurate characterization of the syngas streams will be realized and potential solutions to existing sampling and analytical difficulties will be identified. Consequently, the results presented here should be considered semiquantitative in the absence of validated methods for mercury measurements in coal syngas.

Total Mercury Measurements

The three mercury sampling and analysis techniques were applied to the sour and sweet syngas during test Periods 1-3. These two syngas streams are virtually identical in composition except for the H_2S and moisture content. Hydrogen sulfide in the sour syngas measured about 900 ppmv compared to about 30 ppmv in the sweet syngas. The sweet syngas is virtually dry, while the sour syngas, although sampled downstream of a moisture knock-out, still contained a significant amount of water. Unfortunately, both H_2S and water can have an effect on some of the methods that were used. The average and 95% confidence intervals results obtained from the methods are compared in Tables 8-2 and 8-3. Note that several different impinger solutions were examined with the CVAAS technique.

Table 8-2
Mercury Measured in Sour Syngas

Method	Impingers	Hg, µg/Nm³	95% CI
Charcoal	None	11	13
CVAAS	(1) 2N $NaOH/NaBH_4$	6.1	2.1
	(2) IPA/2N $NaOH/KMnO_4$	3.2	5.2
Method 29	HNO_3/H_2O_2 only	0.8	0.9

Table 8-3
Mercury Measured in Sweet Syngas

Method	Impingers	Hg, µg/Nm³	95% CI
Charcoal	None	0.1	0.02
CVAAS	(1) 0.1N $NaOH/NaBH_4$	3.8	3.6
	(2) IPA/0.1N $NaOH/KMnO_4$	3.0	3.2
	(3) H_2O_2/0.1N $NaOH/KMnO_4$	3.6	2.3
	(4) IPA/0.1N $NaOH/NaBH_4$	3.1	1.5
Method 29	HNO_3/H_2O_2	0.2	0.2

The following observations were made during this method comparison for the sour gas:

- Although highly variable, the charcoal tube method reported the higher total mercury value. These values are the average of three daily measurements and do not coincide with the test period using the CVAAS technique.

- The presence of H_2S appears to have an effect on the impinger capture (or measurement) of total mercury. Case 2 results by CVAAS was lower than Case 1. During sample collection in Case 2, there was H_2S breakthrough from the NaOH scrubbing solution, which may have biased the results low.

- The Method 29 result, with only one type of impinger solution, indicates minimal capture.

The following observations were made regarding this data set for sweet syngas:

- Mercury collected on charcoal, with respect to the other collection/measurement methods, is significantly lower in the sweet syngas matrix. Commercial activated carbon for mercury control is usually sulfur impregnated. The absence (low level) of H_2S in this stream may be responsible for the poor capture efficiency.

- All of the impinger combinations used in the CVAAS study produced very similar results. Comparison with the charcoal or $NaBH_4$ results in Table 1 indicates 44-70% mercury removal across the Selectamine™ process.

- Other test results indicated the need for an H_2S scrubber upstream of the $KMnO_4$ or $NaBH_4$ impinger.

Period 4 testing of the hot raw syngas permitted the opportunity to test the effectiveness of an H_2S scrubbing solution (NaOH) upstream of the $KMnO_4$ impingers in the Method 29 train. The CVAAS system was not used at the raw syngas location. The results from the Method 29 train and charcoal were similar, when considering the 95% CI. Based upon the Hg value in the coal, a theoretical syngas concentration for Hg would be 58 $\mu g/m^3$, assuming all Hg in the coal was present in the syngas (an assumption that is probably valid given the volatility of Hg). The data obtained by both the charcoal (at this location) and the Method 29 (modified) compare favorably with a theoretical maximum Hg concentration. The results for each impinger solution and the total mercury captured in the Method 29 train and the results obtained from the charcoal sorbents are presented in Table 8-4.

Table 8-4
Mercury Measured in Raw Syngas

Method	Impingers	Hg, $\mu g/Nm^3$	95% CI
Charcoal	None		
Method 29	Total		
	(1) 10% HNO_3 / 30%H_2O_2		
	(2) 2 N NaOH		
	(3) 4%$KMnO_4$ / 10%H_2SO_4		

The following observations were made during this method comparison for raw syngas:

- With the addition of NaOH scrubbers in the Method 29 train, total mercury collection was significantly improved (equivalent to total mercury collection by charcoal adsorption);
- The use of NaOH upstream of the $KMnO_4$ impingers appeared effective in collecting H_2S and protecting the $KMnO_4$ solution which collected 87% of the total Hg measured;
- The presence of oxidized mercury in the raw syngas stream (collected by HNO_3 / H_2O_2 solution) is measurable and accounts for 10% of the total mercury captured; and
- A small amount of Hg was retained in the NaOH impinger solution (3% of the total Hg collected); however, no post-sampling purge was performed.

CVAAS Speciation Results

A summary of the sampling runs performed with the gold amalgamation-CVAAS system are presented in Table 8-5. A comparison of mercury results by the various impinger solutions is presented in Table 8-6.

Sweet syngas samples applied directly to $NaBH_4$ impingers indicated that mercury could be detected using a minimum sample volume of 10 liters (SG-1-11/3 and SG-2-11/3). Relatively low spike recoveries using this method indicated that some type of matrix interference may be involved, so tests were repeated using $SnCl_2$ as the reductant. Sampling with this solution resulted in precipitation within the reductant impinger. It is believed that the syngas sulfide concentration played a role in this mechanism, so $SnCl_2$ was eliminated as a reductant for the sweet syngas samples.

Additional $NaBH_4$ tests were performed in which a NaOH scrubbing solution was placed upstream of the reductant to remove H_2S (SG-1-11/6 and SG-1-11/7). This increased the mercury recovery through the $NaBH_4$ and also resulted in better peak shapes during the CVAAS analysis. This indicated that in the previous samples, H_2S may have reacted with the gold surface, interfering with the thermal desorption of mercury and may be responsible for the low spike recoveries. It was determined that some type of H_2S scrubbing solution should be included upstream of the reductant impinger.

During these tests, no mercury was captured in the NaOH solution. To test mercury retention of the NaOH solution, an oxidized mercury standard ($HgCl_2$) was spiked before sampling. No oxidized mercury was detected in the spiked NaOH impinger solution after sampling. This suggests that reactions occurred within the impinger resulting in the reduction of the mercury present.

One sour syngas test (XG-1-11/7) was performed using a 2M NaOH impinger upstream of the reductant impinger. This was done to scrub H_2S from the sour syngas prior to reaching the

Table 8-5
Individual CVAAS Mercury Results by Impinger Train

Test ID Number-Date	Impinger Config.[a]	Mercury (µg/Nm³)			Percent Oxidized	MDL[b] µg/Nm³	Comments
		Hg^0	Oxidized	Total			
Sweet Syngas (Total mercury determined by reduction through $NaBH_4$)							
SG-1-11/3	1	NA[c]	NA	1.87	--	1.07	No H_2S scrubber. Result biased low?
SG-2-11/3	1	NA	NA	<MDL	--	1.07	Sampling system leaks detected.
SG-1-11/6	2	3.82	<MDL	3.82	--	0.27	With H_2S scrubber. No Hg detected in NaOH solution.
SG-1-11/7	2	0.39	--[d]	--	--	0.27	NaOH analysis problems experienced.
Sweet Syngas (Hg^0 - $KMnO_4$, ox-Hg - HNO_3/H_2O_2)							
SGPK-1-11/6	3	--[e]	0.94	--	--	0.54	Hg^0 results out-of-range high.
SGPK-2-11/6	3	--[e]	0.73	--	--	0.54	Hg^0 results out-of-range high.
SGPK-3-11/6	3	2.21	1.37	3.58	0.34	1.07	
Sweet Syngas (Hg^0 - $NaBH_4$, ox-Hg - HNO_3/H_2O_2)							
SGP-1-11/4	7	NA[f]	0.63	--	--	0.36	
Sweet Syngas (Hg^0 - $KMnO_4$, ox-Hg - IPA/DEDTC)							
SGIPK-1-11/6	4	<MDL	<MDL	<MDL	--	0.54	?
SGPK-1-11/7	4	3.75	<MDL	3.85	--	0.20	
SGPK-2-11/7	4	3.82	0.72	4.54	0.14	0.20	
SGIP-3-11/6	5	3.61	NA[g]	--	--	0.65	Only analyzed $KMnO_4$ impinger solution.
Sweet Syngas (Hg^0 - $NaBH_4$, ox-Hg - IPA/DEDTC)							
SGIP-1-11/6	8	2.83	0.91	3.74	0.22	0.41	
SGIP-2-11/6	8	2.03	0.46	2.49	0.16	0.21	
Sour Syngas (Total mercury determined by reduction through $NaBH_4$)							
XG-1-11/8	1	--	--	5.12	--	0.51	No H_2S scrubber. Result biased low?
XG-2-11/8	1	--	--	5.63	--	0.46	
XG-1-11/7	2a	7.45	<MDL	7.45	--	0.90	With H_2S scrubber. No Hg detected in NaOH solution.

Table 8-5 (Continued)

Test ID Number-Date	Impinger Config.[a]	Mercury (µg/Nm^3)			Percent Oxidized	MDL[b] µg/Nm^3	Comments
		Hg^0	Oxidized	Total			
Sour Syngas (Hg^0 - $KMnO_4$, ox-Hg - IPA/DEDTC)							
XGIP-1-11/8	4	3.09	1.93	5.02	0.34	0.54	Hg breakthrough observed and linked to inefficient H_2S removal.
XGIPK-3	4	1.29	<MDL[h]	--	--	0.27	

[a] Impinger configurations are defined in Table 8-1.

[b] MDL = Method detection limit based on gas volume sampled.

[c] NA = Not analyzed/not applicable.

[d] Analytical problems experienced during analysis.

[e] Peak off-scale of recorder.

[f] Not analyzed for elemental Hg (instrument down).

[g] IPA/DEDTC solution not analyzed.

[h] Precipitation observed in impinger; results are suspect.

Table 8-6
Comparison of Mercury Results from Different Impingers

Sample Stream	Mercury Species	Impinger Solution	n [a]	Mercury, (µg/Nm³)		
				Average	S.D.	C.V. [b]
Sweet Syngas	Total	$NaBH_4$	4	2.98	0.96	32
		$KMnO_4$	3	3.99	0.49	12
		All	7	3.41	0.91	27
	Elemental	$NaBH_4$	4	2.27	1.45	64
		$KMnO_4$	4	3.35	0.76	23
		All	8	2.81	1.22	43
	Oxidized	H_2O_2	4	0.91	0.33	36
		IPA	3	0.70	0.23	33
		All	7	0.82	0.30	36
Sour Syngas	Total	$NaBH_4$	3	6.07	1.23	20
		$KMnO_4$	1	5.02	--	--
		All	4	5.81	1.13	19
	Elemental	$KMnO_4$	2	2.19	1.27	58
	Oxidized	IPA	1	1.93	--	--

[a] Number of data points included.

[b] C.V. = Coefficient of variation = [(standard deviation/average) x 100].

$NaBH_4$ solution. Analysis showed that the mercury captured in the NaOH was below the method detection limit. Results from the $NaBH_4$ impinger (XG-1-11/7) showed a slightly higher total mercury result (7.45 µg/Nm³) than that measured by the other sour syngas tests performed without the NaOH scrubber (XG-1-11/8 and XG-2-11/8). The higher values consistently obtained from the sampling trains equipped with NaOH scrubbers suggests that this impinger combination warrants further investigation in future testing.

Solutions of 4% H_2O_2 /5% HNO_3 were used in some tests to test the collection of oxidized mercury. Initial sample and spike recovery results showed that the recovery of mercury from this impinger was very low. Mercury recovery was improved by increasing the pH of the impinger solution from approximately 0.5 to 7 prior to reduction with $NaBH_4$. A mercury spike recovery

of 72% was achieved using this method, indicating a possible low bias for oxidized mercury recovery from this impinger solution. Mercury recoveries from peroxide solutions in the absence of the sample matrix were found to be acceptable (>80%).

A solution of 80% IPA with 0.1 M diethyldithiocarbamic acid (DEDTC) was also used as a collecting impinger solution for oxidized mercury. DEDTC has been shown in laboratory tests to be an effective complexing agent of mercury. Matrix spike recoveries for IPA/DEDTC impingers were found to be better (86%) than with peroxide. It is interesting, however, that the amount of captured mercury from the sweet syngas was generally lower in the IPA solutions than with H_2O_2. No sampling problems were observed with the IPA/DEDTC impingers, although they had to be kept well-chilled during sampling to prevent excessive IPA vaporization. In most IPA tests, a blank impinger was placed downstream of the former to act as a cold knockout trap.

Potassium permanganate impingers were used to verify the total mercury concentrations measured with $NaBH_4$ impingers. As indicated earlier, $KMnO_4$ impingers could not be used in EPA Method 29 sampling trains since they were readily reduced by the H_2S in the sample. However, the lower flow rates and volumes used with this testing along with the ability to scrub out a majority of the H_2S prior to the $KMnO_4$ solution enabled its use. Analysis of the $KMnO_4$ impingers generally produced higher elemental mercury concentrations than the $NaBH_4$ in the sweet syngas (Table 8-6). This may indicate that the latter was sensitive to some matrix effects. Total mercury results from $KMnO_4$ and $NaBH_4$ impingers were in closer agreement for the sour syngas, although only one $KMnO_4$ sample was analyzed and it reported lower results than the average result obtained by the $NaBH_4$ solution. Some breakthrough (approximately 11%) of elemental mercury was observed with the $KMnO_4$ impinger while sampling the sour syngas; no breakthrough was detected while sampling the sweet syngas. This indicates that some H_2S from the sour syngas was not efficiently removed by the single NaOH impinger. H_2S breakthrough would have reacted with the $KMnO_4$, reducing its oxidative potential and collection efficiency for mercury. A single NaOH impinger may not be sufficient to effectively scrub H_2S from gas streams with higher H_2S concentrations. The ability to detect this type of sampling problem is a good example of an advantage of this type of sampling and analysis technique.

Calibration and Quality Control

The CVAAS was calibrated using mercury vapor standards injected into the inert carrier gas upstream of the amalgamation unit. Standards were obtained by pulling vapor from the headspace of a sealed mercury reservoir held in a controlled temperature water bath. The concentration was calculated using the mercury vapor pressure as a function of the temperature. Analysis of the standards was carried out similarly to the samples by first amalgamating the mercury vapor onto the gold trap, and analyzing the mercury which is thermally desorbed from the trap under an argon carrier gas. A calibration curve was generated by varying the volume of the injected Hg^0 standard. A linear regression of the calibration curve was made from which the results of the process samples were compared.

Quality control samples were analyzed to verify the mercury calibration curve and recovery from the impinger solutions, as well as ensure proper control and operation of the CVAAS instrument. Control samples included both liquid and vapor standards. Liquid standards were obtained from a prepared solution of mercuric chloride ($HgCl_2$) and a commercially purchased AAS standard solution ($HgNO_3$). The calibration curve was checked periodically by either injecting a vapor standard directly to the gold amalgamation unit, or by reducing a liquid standard solution and purging the released mercury to the gold. Results were consistently within 10% of the calibration standard value.

Process gas streams contain matrix species which can react with mercury trapped in an impinger solution potentially influencing results. It was therefore necessary to verify the recovery of mercury from each impinger solution used. Sodium borohydride and potassium permanganate impingers were spiked with vapor (elemental) mercury standards while IPA, sodium hydroxide and hydrogen peroxide impingers were spiked with aqueous, oxidized mercury standards. Recoveries within 20% of the theoretical value were considered acceptable. Table 8-7 presents the spike recovery results.

Vapor standards were injected over the heated gold, in duplicate, after each sample analysis, as a sensitivity check of the CVAAS. The response and sensitivity of the CVAAS have been found to fluctuate throughout a given day, and such drifts can be compensated for by analyzing vapor standards following each sample. The instrument response was normalized for the measured amount of mercury in the vapor standard and the sample results were adjusted accordingly.

Mercury contamination present in many solutions can be substantial relative to the concentrations measured from gas samples. All impinger solutions were therefore analyzed for their mercury content by reducing them with $NaBH_4$ (similar to the sample analyses) and purged with argon over cooled gold. The results of the solution blank analyses, normalized for impinger volume, are given in Table 8-8. The initial $NaBH_4$ solution contained a relatively high mercury contamination whereas the subsequent preparations did not contain detectable amounts. The IPA solutions also contained relatively high blank values. This has been shown in laboratory studies to be related to mercury contamination of the DEDTC chelant.

The results of the sample analyses were normalized for the instrument sensitivity check and compared to the calibration curve from which the mass of mercury in the sample was calculated. The mercury concentration in the impinger (blank) solution was then subtracted from this total leaving the mercury content of the sample. The concentration of mercury in the gas stream was determined by normalizing for total sample volume. In reduction experiments, in which only one reducing impinger was used, the results were given as total mercury. In multiple impinger experiments, the amount as elemental mercury was added to the captured oxidized mercury observed to calculate the total mercury of the sample. The method detection limit (MDL) was determined for each sample by normalizing the instrumental detection limit for total sample volume; the former was calculated to be 10 ng for this testing.

Table 8-7
Results of QC Recoveries of Mercury Spikes

Test No.	QC Test	Impinger Type	Percent Hg Recovery
QC-1-11/3	Liquid Spike	4% H_2O_2	112
SGP-1-11/4	Liquid Spike	4% H_2O_2	72
QC-1-11/4	Vapor Standard	None	112
BIP-2-11/6	Liquid Spike	IPA/DEDTC	86
SGPK-2c-11/6	Vapor Spike	4% $KMnO_4$	72
QC-1-11/6	Vapor Standard	None	80
SG-2a-11/7	Vapor Spike	50 mM $NaBH_4$	124
SG-2b-11/7	Liquid Spike	0.1 N NaOH	0
QC-1-11/7	Liquid Spike	4% H_2O_2	107
XGIPK-2a-11/8	Liquid Spike	IPA/DEDTC	99
XGIPK-2b-11/8	Vapor Spike	$KMnO_4$	109

Table 8-8
Mercury Concentrations in Blank Impinger Solutions

Date	Impinger Solution	Mercury (ng/mL)
03-Nov-94	50 mM $NaBH_4$(1)	2.73
04-Nov-94	4% H_2O_2(1)	0.46
06-Nov-94	IPA/DEDTC* (1)	1.42
	4% H_2O_2(2)	0.50
	0.1N NaOH (1)	0.39
	4% $KMnO_4$	1.03
	50 mM $NaBH_4$	0
07-Nov-94	0.1N NaOH (2)	0
	IPA/DEDTC* (2)	3.70
	4% H_2O_2 (3)	0.50
	2M NaOH	0.29
	50 mM $NaBH_4$	0
08-Nov-94	IPA/DEDTC*	2.41

Conclusions and Recommendations

The results of this testing indicated that both elemental and oxidized mercury were present in the syngas at parts per trillion (pptv) levels. The result of all reductant and multiple-impinger tests using the gold-CVAAS system was a "total" mercury concentration in the sweet syngas of 3.41 $\mu g/Nm^3$ (381 pptv). This was lower than the average value determined for the sour syngas at 5.81 $\mu g/Nm^3$ (649 pptv). It should be noted that fewer sour syngas tests were performed during this testing. Although these results must be viewed as semiquantitative, the data indicate a higher mercury concentration in the sour syngas than in the sweet syngas.

Measurement results for total mercury in sour syngas using charcoal sorbents were consistent with that of the gold amalgamation-CVAAS system; however, mercury values of sweet syngas measured by charcoal sorbents were significantly lower than the gold-CVAAS results. Since the sweet syngas has significantly reduced levels of H_2S and H_2O, their presence in the raw and sour syngas may introduce a desired matrix effect that improves mercury collection on charcoal. The effect of H_2S on charcoal may be related to previous studies demonstrating the effectiveness of sulfur-impregnated charcoal sorbents for mercury collection.[8] For comparison, H_2S concentrations in raw syngas are similar to those found in sour syngas. However, the total mercury in the hot raw syngas was measured at 64 $\mu g/Nm^3$ by charcoal. The average total mercury result for the sour syngas stream was 11 $\mu g/Nm^3$. Further investigations into the collection efficiency of coconut-based charcoal in syngas at various H_2S and H_2O levels and with various mercury species is warranted. The gold amalgamation-CVAAS system makes this testing possible by measuring the effluent gases from various collection systems.

The data showed relatively large variability. This was attributed to the low levels of mercury present in the syngas and the presence of matrix interferences. The variation did not appear to be related to specific impinger trains or solutions, but rather was observed with all combinations tested. The addition of a NaOH scrubber was necessary to protect reductant impingers as well as the gold surface from the syngas matrix. Reactions within the nitric/peroxide impingers also made it necessary to adjust the solution pH prior to the reduction step in order to obtain acceptable mercury recoveries using the gold-CVAAS system.

The ratio of oxidized mercury to elemental mercury varied from test to test with the calculated percentage of oxidized mercury ranging from 16 to 38% of the total mercury measured in the sweet syngas. Oxidized mercury in sour syngas was measured at 2.2 $\mu g/Nm^3$, approximately 38% of the average total mercury measured. Analysis of the raw syngas by Method 29 (adapted with NaOH impingers) indicates oxidized mercury present at approximately 10% of the total mercury collected. The average results of peroxide impinger tests using the gold-CVAAS indicated an oxidized mercury concentration in the sweet syngas of 0.9 $\mu g/Nm^3$, while IPA tests indicate 0.7 $\mu g/Nm^3$. These two impinger solution results (H_2O_2 and IPA) differed by 27% and the coefficients of variation were 36% and 33%, respectively.

Analysis of sodium borohydride impingers measured an elemental mercury concentration of 2.3 $\mu g/Nm^3$ in the sweet syngas, whereas mercury collected with the $KMnO_4$ impingers measured 3.3 $\mu g/Nm^3$. This represents a relative percent difference of 39% between the two methods.

Overall, favorable results were not found in this study for using charcoal sorbents to sample sweet syngas, although improved collection may be related to syngas streams with higher H_2S concentrations.

The best measurement technique identified during this methods comparison is the gold amalgamation-CVAAS technique. The gold-CVAAS system appears to provide complete mercury collection, assuming the quantitative retention of mercury on the gold mesh. By incorporating various impinger solutions designed to remove matrix interferents, this technique offered the most consistent results for the two primary gas streams analyzed.

The initial results by Method 29 (modified to include NaOH impingers) also indicate a promising alternative for total mercury collection, if not for its potential to separate elemental and oxidized mercury species. A more comprehensive testing of the Method 29 approach (with NaOH impingers) is needed to qualify the approach as an alternative to the gold-CVAAS system.

Recommendations for Further Testing

Direct CVAAS analyses performed on-site identified promising alternatives or modifications to the Method 29 approach which were investigated during Period 4 testing of the hot raw syngas stream. Future efforts at quantifying mercury in syngas should consider incorporating the following recommendations and assessing the results through a comprehensive quality control program.

- An H_2S scrubbing impinger (NaOH, etc.) is required upstream of any reductant impingers to eliminate matrix effects with both the impinger solution and the gold amalgam trap. Sodium hydroxide appeared to work well for this purpose without absorbing significant quantities of mercury. Particular attention to H_2S collection efficiency is necessary to prevent passing H_2S to the permanganate impinger solution.

- The Method 29 HNO_3/H_2O_2 impinger solution demonstrated the ability to absorb mercury, presumably in the oxidized form, from the syngas. The results of this testing indicate that the addition of H_2S scrubbing impingers, upstream of the $KMnO_4$ impinger, may be a suitable modification to this method to enable the collection of total mercury.

- Testing of charcoal sorbents in conjunction with the gold amalgamation-CVAAS system could 1) determine breakthrough potential of charcoal, and 2) indicate if charcoal adsorption of mercury is species dependent, or quantitative for all forms of mercury.

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APPENDIX A: QUALITY CONTROL

Table A-1
Analytical Results for Audit Samples

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
Multi-Metals Train								
Radian	Filter-Q-2608	Filter w/NBS 1633a Fly Ash	Aluminum	µg/g	132,000	143,000	91.7	75-125
			Antimony	µg/g	ND	6.8	NC	75-125
			Arsenic	µg/g	168	145	115	75-125
			Barium	µg/g	1,441	1,500	96.4	75-125
			Beryllium	µg/g	11.5	12	96.4	75-125
			Cadmium	µg/g	ND	1.00	NC	75-125
			Calcium	µg/g	10,500	11,100	94.4	75-125
			Chromium	µg/g	163	196	83.1	75-125
			Cobalt	µg/g	43.6	46	94.9	75-125
			Copper	µg/g	80.7	118	68.6	75-125
			Iron	µg/g	87,400	94,000	92.9	75-125
			Lead	µg/g	36.9	72.4	51.0	75-125
			Magnesium	µg/g	4,090	4,550	90.1	75-125
			Manganese	µg/g	175	179	98.2	75-125
			Mercury	µg/g	0.13	0.16	80.0	75-125
			Molybdenum	µg/g	14.1	29	48.3	75-125
			Nickel	µg/g	103	127	81.2	75-125
			Potassium	µg/g	1,900	NS	NC	75-125
			Selenium	µg/g	10.6	10.3	103	75-125
			Silicon	µg/g	17,500	18,800	93.2	75-125
			Sodium	µg/g	84.2	1,700	4.95	75-125
			Titanium	µg/g	7,580	8,000	94.8	75-125
			Vanadium	µg/g	273	297	92.1	75-125
			Zinc	µg/g	35.5	220	16.1	75-125
Radian	LGTI-TS-104	HNO_3 Impingers by ICP-AES and AAS	Aluminum	mg/L	0.0332	0.04	83.0	75-125
			Antimony	mg/L	0.00303	0.02	15.2	75-125
			Arsenic	mg/L	0.0374	0.04	93.5	75-125
			Barium	mg/L	0.0717	0.08	89.6	75-125
			Beryllium	mg/L	0.0356	0.04	89.0	75-125
			Boron	mg/L	0.00889	NS	NC	75-125
			Cadmium	mg/L	0.0412	0.04	103	75-125
			Calcium	mg/L	0.132	NS	NC	75-125
			Chromium	mg/L	0.00995	NS	NC	75-125
			Cobalt	mg/L	ND	NS	NC	75-125
			Copper	mg/L	0.0202	0.02	101	75-125
			Iron	mg/L	0.0284	NS	NC	75-125
			Lead	mg/L	ND	NS	NC	75-125
			Magnesium	mg/L	0.331	NS	NC	75-125
			Manganese	mg/L	0.0269	0.02	135	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
Radian	LGTI-TS-104	HNO_3 Impingers by ICP-AES and AAS (Cont'd)	Mercury	mg/L	NA	NS	NC	75-125
			Molybdenum	mg/L	0.00843	0.02	42.2	75-125
			Nickel	mg/L	0.0122	NS	NC	75-125
			Potassium	mg/L	0.799	NS	NC	75-125
			Selenium	mg/L	ND	NS	NC	75-125
			Silicon	mg/L	0.319	NS	NC	75-125
			Sodium	mg/L	0.224	NS	NC	75-125
			Titanium	mg/L	0.000930	NS	NC	75-125
			Vanadium	mg/L	0.00238	NS	NC	75-125
			Zinc	mg/L	0.0175	NS	NC	75-125
Harvard ICP/MS	LGTI-TS-104	HNO_3 Impingers	Antimony	mg/L	0.0177	0.02	88.3	75-125
			Arsenic	mg/L	0.0390	0.04	97.5	75-125
			Barium	mg/L	0.0791	0.08	98.9	75-125
			Beryllium	mg/L	0.0392	0.04	98.0	75-125
			Cadmium	mg/L	0.0353	0.04	88.2	75-125
			Chromium	mg/L	ND	NS	NC	75-125
			Cobalt	mg/L	0.00001	NS	NC	75-125
			Copper	mg/L	0.0239	0.02	119	75-125
			Lead	mg/L	0.0007	NS	NC	75-125
			Manganese	mg/L	0.0267	0.02	134	75-125
			Mercury	mg/L	0.0010	NS	NC	75-125
			Molybdenum	mg/L	0.011	0.02	55.5	75-125
			Nickel	mg/L	ND	NS	NC	75-125
			Selenium	mg/L	ND	NS	NC	75-125
			Vanadium	mg/L	0.00006	NS	NC	75-125
Radian	LGTI-TS-105	$KMnO_4$ Impingers	Mercury	mg/L	0.0176	0.6	2.93	75-125
Charcoal Tube								
Radian	LGTI-CMS-001 LGTI-CMS-002	Charcoal Tube	Aluminum	µg	4529/4659	5,000	90.6/93.2	75-125
			Antimony	µg	10.1/14.2	100	10.1/14.2	75-125
			Arsenic	µg	18.5/18.3	20.0	92.5/91.5	75-125
			Barium	µg	89.1/92.2	100	89.1/92.2	75-125
			Beryllium	µg	87.6/87.3	100	87.6/87.3	75-125
			Boron	µg	69.3/77.5	100	69.3/77.5	75-125
			Cadmium	µg	4.26/4.23	4.0	107/106	75-125
			Calcium	µg	4539/4549	5,000	90.8/91.0	75-125
			Chromium	µg	82.2/77.6	100	82.2/77.6	75-125
			Cobalt	µg	86.7/84.8	100	86.7/84.8	75-125
			Copper	µg	83.8/86.1	100	83.8/86.1	75-125
			Iron	µg	4157/4157	5,000	83.1/83.1	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
Radian	LGTI-CMS-001 LGTI-CMS-002	Charcoal Tube (Cont'd)	Lead	µg	21.3/21.1	20.0	107/106	75-125
			Magnesium	µg	4538/4608	5,000	90.8/92.2	75-125
			Manganese	µg	87.5/87.5	100	87.5/87.5	75-125
			Mercury	µg	0.52/0.62	1.00	52.4/61.6	75-125
			Molybdenum	µg	42.0/45.6	100	42.0/45.6	75-125
			Nickel	µg	89.7/86.6	100	89.7/86.6	75-125
			Potassium	µg	4028/4048	5,000	80.6/81.0	75-125
			Selenium	µg	4.76/4.63	5.0	95.2/92.6	75-125
			Silicon	µg	169/275	500	33.8/55.0	75-125
			Sodium	µg	4,607/4,797	5,000	92.1/95.9	75-125
			Titanium	µg	80.9/84.5	100	80.9/84.5	75-125
			Vanadium	µg	85.7/86.6	100	85.7/86.6	75-125
			Zinc	µg	86.7/82.5	100	86.7/82.5	75-125
Method 8 Train								
Radian	LGTI-TS-102	IPA Impingers	Sulfate	mg/L	5.22	3.86	135	80-120
	LGTI-TS-103	H_2O_2 Impingers	Sulfate	mg/L	10.2	12.1	84.3	80-120
Anions/Ammonia/Cyanide Train								
Radian	LGTI-AG-025	1% H_2SO_4 Impingers	Ammonia	mg/L	0.954	1.01	94.5	80-120
			Chloride	mg/L	ND	NS	NC	80-120
			Fluoride	mg/L	0.019	NS	NC	80-120
	LGTI-AG-026	ZnOAc Impingers	Cyanide	mg/L	3.74	3.07	122	75-125
	LGTI-TS-106	0.1N H_2SO_4 Impingers	Chloride	mg/L	4.06	0.20	2,030	80-120
			Fluoride	mg/L	0.299	0.32	93.4	80-120
Method 7E Train								
Radian	LGTI-INC-115	$KMnO_4$/NaOH Impingers	Nitrate	mg/L	60.0	66.6	90.2	80-120
Aqueous								
Radian	LGTI-MW-201	Aqueous	Aluminum	mg/L	0.0586	0.08	73.3	75-125
			Antimony	mg/L	0.0151	0.04	37.8	75-125
			Arsenic	mg/L	0.074	0.08	92.5	75-125
			Barium	mg/L	0.148	0.16	92.5	75-125
			Beryllium	mg/L	0.077	0.08	96.3	75-125
			Boron	mg/L	0.00876	NS	NC	75-125
			Cadmium	mg/L	0.0813	0.08	102	75-125
			Calcium	mg/L	0.0400	NS	NC	75-125
			Chromium	mg/L	ND	NS	NC	75-125
			Cobalt	mg/L	ND	NS	NC	75-125
			Copper	mg/L	0.0404	0.04	101	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
Radian	LGTI-MW-201	Aqueous (Cont'd)	Iron	mg/L	0.00465	NS	NC	75-125
			Lead	mg/L	0.000535	NS	NC	75-125
			Magnesium	mg/L	0.00594	NS	NC	75-125
			Manganese	mg/L	0.0345	0.04	86.3	75-125
			Molybdenum	mg/L	0.0181	0.04	45.3	75-125
			Nickel	mg/L	ND	NS	NC	75-125
			Potassium	mg/L	0.476	NS	NC	75-125
			Selenium	mg/L	ND	NS	NC	75-125
			Silicon	mg/L	0.0469	NS	NC	75-125
			Sodium	mg/L	0.0384	NS	NC	75-125
			Titanium	mg/L	0.00276	NS	NC	75-125
			Vanadium	mg/L	0.00110	NS	NC	75-125
			Zinc	mg/L	ND	NS	NC	75-125
			Ammonia	mg/L	771	884	87.2	80-120
			Chloride	mg/L	195	202.8	96.2	80-120
			Fluoride	mg/L	30.5	28.6	107	80-120
			Formate	mg/L	898	897.6	100	80-120
			Phosphate as P	mg/L	0.993	0.752	132	80-120
			Sulfate	mg/L	18.8	24.1	78.0	80-120
			COD	mg/L	2867	2,898	98.9	75-125
			Phenol	mg/L	1,180	1172	101	75-125
			Thiocyanate	mg/L	5.00	5.00	100	80-120
			Total Cyanide	mg/L	8.41	10.12	83.1	75-125
			Free Cyanide	mg/L	5.3	5.12	104	75-125
Slag								
CT & E	LGTI-SLG-401 (Not a SRM)	Slag	Aluminum	µg/g	59,300	50,000	119	75-125
			Antimony	µg/g	5	<9.0	NA	75-152
			Arsenic	µg/g	4	18	22.2	75-125
			Barium	µg/g	1,520	2,167	70.1	75-125
			Beryllium	µg/g	<1.0	0.46	NA	75-125
			Boron	µg/g	430	160	269	75-125
			Cadmium	µg/g	<1	<0.44	NA	75-125
			Calcium	µg/g	102,400	87,667	117	75-125
			Chromium	µg/g	650	957	67.9	75-125
			Cobalt	µg/g	20	15	133	75-125
			Copper	µg/g	670	897	74.7	75-125
			Iron	µg/g	86,300	74,667	116	75-125
			Lead	µg/g	32	28	114	75-125
			Magnesium	µg/g	14,000	12,000	117	75-125
			Manganese	µg/g	700	643	109	75-125
			Mercury	µg/g	0.16	0.11	145	75-125
			Molybdenum	µg/g	<10	49	NA	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
CT & E	LGTI-SLG-401 (Not a SRM)	Slag (Cont'd)	Nickel	µg/g	180	405	44.4	75-125
			Potassium	µg/g	5,600	8,467	66.1	75-125
			Selenium	µg/g	8	<0.77	NA	75-125
			Silicon	µg/g	213,100	76,333	279	75-125
			Sodium	µg/g	19,400	24,000	80.8	75-125
			Titanium	µg/g	6,300	6,400	98.4	75-125
			Vanadium	µg/g	170	114	149	75-125
			Zinc	µg/g	560	3,067	18.3	75-125
			Phosphorus Pentoxide	% in Ash	6.13	8.7	70.5	75-125
			Carbon	Wt. %	1.79	2.22	80.6	80-120
			Hydrogen	Wt. %	0.05	0.03	167	80-120
			Nitrogen	Wt. %	<0.01	0.13	NA	80-120
			Sulfur	Wt. %	0.12	0.11	109	80-120
			Ash	Wt. %	97.89	98	99.9	80-120
Radian	LGTI-SLG-401 (Not a SRM)	Slag	Chloride	µg/g	89.6/82.5	325	27.6/25.4	80-120
			Fluoride	µg/g	146/37.5	81	180/46.3	80-120
Harvard ICP/MS	LGTI-SLG-401 (Not a SRM)	Slag	Antimony	µg/g	4.21	<9.0	NC	75-125
			Arsenic	µg/g	5.65	18	31.4	75-125
			Beryllium	µg/g	2.08	0.46	452	75-125
			Cadmium	µg/g	3.90	<0.44	NC	75-125
			Chromium	µg/g	566	957	59.1	75-125
			Cobalt	µg/g	23.8	15	159	75-125
			Copper	µg/g	647	897	72.1	75-125
			Lead	µg/g	20.36	28	72.7	75-125
			Manganese	µg/g	537	643	83.5	75-125
			Mercury	µg/g	3.75	0.11	3,409	75-125
			Molybdenum	µg/g	61.2	49	125	75-125
			Nickel	µg/g	747	405	184	75-125
			Selenium	µg/g	22.73	<0.77	NC	75-125
			Vanadium	µg/g	157	114	138	75-125
Coals								
CT & E	LGTI-SLY33-401 (Round Robin D)	Coal	Antimony	µg/g	<1	0.47	NC	75-125
			Arsenic	µg/g	<1	1.24	NC	75-125
			Barium	µg/g	220	370	59.5	75-125
			Beryllium	µg/g	<0.4	0.42	NC	75-125
			Boron	µg/g	130	83.4	156	75-125
			Cadmium	µg/g	<0.2	0.058	NC	75-125
			Chromium	µg/g	5.00	4.40	114	75-125
			Cobalt	µg/g	1	0.86	116	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
CT & E	LGTI-SLY33-401 (Round Robin)	Coal (Cont'd)	Copper	μg/g	11	9.52	116	75-125
			Lead	μg/g	30	5.22	575	75-125
			Manganese	μg/g	220	145	152	75-125
			Mercury	μg/g	0.06	0.084	71.4	75-125
			Molybdenum	μg/g	<4	7.93	NC	75-125
			Nickel	μg/g	4	5.09	78.6	75-125
			Selenium	μg/g	2	0.84	238	75-125
			Vanadium	μg/g	15	9.36	160	75-125
			Alumina	% in Ash	19.16	16.48	116	75-125
			Silica	% in Ash	43.56	42.12	103	75-125
			Titania	% in Ash	0.86	0.88	97.7	75-125
			Calcium Oxide	% in Ash	11.77	7.79	151	75-125
			Ferric Oxide	% in Ash	6.64	6.07	109	75-125
			Magnesium Oxide	% in Ash	3.85	2.55	151	75-125
			Phosphorus Pentoxide	% in Ash	0.39	0.37	105	75-125
			Potassium Oxide	% in Ash	0.52	0.51	102	75-125
			Sodium Oxide	% in Ash	0.35	0.29	121	75-125
			Sulfur Trioxide	% in Ash	11.58	11.41	101	75-125
			Carbon	Wt. %	68.02	67.6	101	80-120
			Hydrogen	Wt. %	4.49	4.80	93.5	80-120
			Nitrogen	Wt. %	1.00	1.01	99.0	80-120
			Sulfur	Wt. %	0.97	1.15	84.4	80-120
			Chlorine	μg/g	<100	3.0	NC	80-120
			Ash	Wt. %	11.64	11.7	99.5	80-120
			HHV	Btu/lb	11,671	11,350	103	80-120
Radian	LGTI-SLY33-401 (Round Robin D)	Coal	Chloride	mg/kg	23.5	300	7.8	80-120
			Fluoride	mg/kg	32.4	44.3	73.1	80-120
Harvard ICP/MS	LGTI-SLY33-401 (Round Robin D)	Coal	Antimony	μg/g	0.42/0.39	0.47	89.4/83.7	75-125
			Arsenic	μg/g	1.90/1.72	1.24	153/138	75-125
			Beryllium	μg/g	0.37/0.42	0.42	88.1/99.9	75-125
			Cadmium	μg/g	0.12/0.07	0.058	207/126	75-125
			Chromium	μg/g	4.91/4.68	4.4	112/107	75-125
			Cobalt	μg/g	1.03/0.93	0.86	120/108	75-125
			Copper	μg/g	8.00/8.55	9.52	84.0/89.8	75-125
			Lead	μg/g	1.52/1.58	5.22	29.1/30.3	75-125
			Manganese	μg/g	84.5/80.3	145	58.3/55.4	75-125
			Mercury	μg/g	0.12/0.04	0.084	143/53.4	75-125
			Molybdenum	μg/g	6.72/6.88	7.93	84.7/86.8	75-125
			Nickel	μg/g	35.7/33.1	5.09	702/650	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
Harvard ICP/MS	LGTI-SLY33-401 (Round Robin D)	Coal (Cont'd)	Selenium	µg/g	4.10/2.20	0.84	488/262	75-125
			Vanadium	µg/g	8.11/7.92	9.36	86.6/84.7	75-125
CT & E	LGTI-SLY33a-401 (AR 2780)	Coal SRM	Alumina	% in Ash	11.83	14.4	82.2	75-125
			Silica	% in Ash	28.09	33.71	83.3	75-125
			Titania	% in Ash	0.51	0.73	69.9	75-125
			Calcium Oxide	% in Ash	0.57	0.54	106	75-125
			Ferric Oxide	% in Ash	38.85	46.35	83.8	75-125
			Magnesium Oxide	% in Ash	0.335	0.53	63.2	75-125
			Manganese Oxide	% in Ash	0.26	0.29	89.7	75-125
			Phosphorus Pentoxide	% in ash	ND	0.11	NC	75-125
			Potassium Oxide	% in Ash	1.22	1.43	85.3	75-125
			Sodium Oxide	% in Ash	0.27	0.17	159	75-125
			Strontium Oxide	% in Ash	ND	0.01	NC	75-125
			Sulfur Trioxide	% in Ash	0.66	0.53	125	75-125
			Carbon	Wt. %	72.01	70.87	102	80-120
			Hydrogen	Wt. %	4.77	5.05	94.5	80-120
			Nitrogen	Wt. %	1.46	1.58	92.4	80-120
			Sulfur	Wt. %	3.61	3.58	101	80-120
			Chlorine	µg/g	<100	0.00	NC	80-120
			Ash	Wt. %	9.90	8.23	120	80-120
			HHV	Btu/lb	12,713	12,748	99.7	80-120
			Volatile Matter	Wt. %	39.3	39.05	101	80-120
			Fixed Carbon	Wt. %	52.45	52.72	99.5	80-120
Radian	LGTI-SLY33a-401 (AR 2780)	Coal SRM	Chloride	mg/kg	1,050	1,260	83.3	80-120
			Fluoride	mg/kg	9.37	40	23.4	80-120
CT & E	LGTI-RC-401 (AR 1801)	Coal SRM	Antimony	µg/g	<1	0.9	NC	75-125
			Arsenic	µg/g	4	6.1	65.6	75-125
			Beryllium	µg/g	3.5	3.3	106	75-125
			Boron	µg/g	110	118	93.2	75-125
			Cadmium	µg/g	<0.2	<0.2	NC	75-125
			Chromium	µg/g	17	20	85.0	75-125
			Cobalt	µg/g	11	11	100	75-125
			Copper	µg/g	19	18	106	75-125
			Lead	µg/g	56	8	700	75-125
			Manganese	µg/g	15	14	107	75-125
			Mercury	µg/g	0.08	0.04	200	75-125
			Molybdenum	µg/g	<3	2	NC	75-125

Table A-1 (Continued)

Lab	Sample ID	Matrix	Analyte	Units	Lab Result	Reference Value	Percent Recovery	Audit Objective
CT & E	LGTI-RC-401 (AR 1801)	Coal SRM (Cont'd)	Nickel	µg/g	70	58	121	75-125
			Selenium	µg/g	3	2.6	115	75-125
			Vanadium	µg/g	25	28	89.3	75-125
			Zinc	µg/g	130	109	119	75-125
			Fluorine	µg/g	65	68	95.6	80-120
Harvard ICP/MS	LGTI-RC-401 (AR 1801)	Coal SRM	Antimony	µg/g	0.94/1.24	0.9	104/138	75-125
			Arsenic	µg/g	3.77/5.54	6.1	61.8/90.8	75-125
			Beryllium	µg/g	3.31/3.94	3.3	100/119	75-125
			Cadmium	µg/g	0.30/0.38	<0.2	NC/NC	75-125
			Chromium	µg/g	13.7/18.0	20	68.6/89.9	75-125
			Cobalt	µg/g	10.8/13.9	11	98.3/126	75-125
			Copper	µg/g	15.6/13.1	18	86.8/72.9	75-125
			Lead	µg/g	2.52/2.63	8	31.5/32.8	75-125
			Manganese	µg/g	14.6/19.7	14	105/141	75-125
			Mercury	µg/g	0.04/0.05	0.04	100/117	75-125
			Molybdenum	µg/g	0.68/0.87	2	34.0/43.4	75-125
			Nickel	µg/g	73.6/99.7	58	127/172	75-125
			Selenium	µg/g	1.39/2.73	2.6	53.5/105	75-125
			Vanadium	µg/g	19.0/24.5	28	68.0/87.6	75-125

NC = Not calculated.
ND = Not detected.
NS = Not spiked.
NA = Not applicable.
SRM = Standard Reference Material.

Table A-2
QC Blanks

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Laboratory Method Blank - Filter & PNR				
ICP-AES Metals				
Aluminum	1	1	3.89 µg	2.76 µg
Antimony	1	1	8.90 µg	5.86 µg
Barium	1	1	0.148 µg	0.0697 µg
Beryllium	1	0	ND	0.0329 µg
Calcium	1	1	5.30 µg	1.37 µg
Chromium	1	1	0.787 µg	0.197 µg
Cobalt	1	1	1.01 µg	0.538 µg
Copper	1	1	0.154 µg	0.502 µg
Iron	1	1	2.50 µg	0.509 µg
Magnesium	1	1	5.19 µg	9.63 µg
Manganese	1	0	ND	0.492 µg
Molybdenum	1	1	0.404 µg	0.384 µg
Nickel	1	1	0.669 µg	1.14 µg
Phosphorus	1	1	160 µg	10.9 µg
Potassium	1	1	17.0 µg	44.1 µg
Sodium	1	1	116 µg	3.05 µg
Titanium	1	0	ND	0.716 µg
Vanadium	1	1	0.763 µg	0.292 µg
Zinc	1	0	ND	0.347 µg
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.0946 µg
Cadmium	1	0	ND	0.0238 µg
Lead	1	1	0.0800 µg	0.0800 µg
Mercury	1	0	ND	0.000033 µg
Selenium	1	0	ND	0.0802 µg
Reagent Blank - Filter & PNR				
ICP-AES Metals				
Aluminum	2	2	55.7-76.5 µg	2.76 µg
Antimony	2	0	ND	5.86 µg
Barium	2	2	2.08-3.72 µg	0.0697 µg
Beryllium	2	0	ND	0.0329 µg
Calcium	2	2	52.5-57.6 µg	1.37 µg
Chromium	2	2	1.56-1.67 µg	0.197 µg
Cobalt	2	2	0.673-1.35 µg	0.538 µg
Copper	2	2	0.772-1.00 µg	0.502 µg
Iron	2	2	13.6-20.4 µg	0.509 µg
Magnesium	2	2	10.7-11.4 µg	9.63 µg
Manganese	2	2	0.134-0.266 µg	0.492 µg
Molybdenum	2	2	8.36-11.8 µg	0.384 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Nickel	2	2	2.39-3.54 µg	1.14 µg
Phosphorus	2	2	326-360 µg	10.9 µg
Potassium	2	2	41.7-53.9 µg	44.1 µg
Sodium	2	2	108-188 µg	3.05 µg
Titanium	2	2	0.558-1.02 µg	0.716 µg
Vanadium	2	2	0.733-0.877 µg	0.292 µg
Zinc	2	2	2.37-2.40 µg	0.347 µg
GFAAS and CVAAS Metals				
Arsenic	2	1	0.748 µg	0.0946 µg
Cadmium	2	1	0.870 µg	0.0238 µg
Lead	2	2	0.250-0.256 µg	0.0800 µg
Mercury	2	2	0.0150-0.0160 µg	0.0033 µg
Selenium	2	0	ND	0.0802 µg
Field Blank - Filter & PNR				
ICP-AES Metals				
Aluminum	2	2	67.2-117 µg	2.76 µg
Antimony	2	1	4.31 µg	5.86 µg
Barium	2	2	2.43-3.77 µg	0.0697 µg
Beryllium	2	0	ND	0.0329 µg
Calcium	2	2	76.1-104 µg	1.37 µg
Chromium	2	2	1.97-4.20 µg	0.197 µg
Cobalt	2	2	0.331-0.840 µg	0.538 µg
Copper	2	2	2.94-5.41 µg	0.502 µg
Iron	2	2	39.4-63.5 µg	0.509 µg
Magnesium	2	2	15.8-20.0 µg	9.63 µg
Manganese	2	2	0.662-0.803 µg	0.492 µg
Molybdenum	2	2	8.28-13.7 µg	0.384 µg
Nickel	2	2	4.87-6.69 µg	1.14 µg
Phosphorus	2	2	333-359 µg	10.9 µg
Potassium	2	2	26.0-66.5 µg	44.1 µg
Sodium	2	2	230-334 µg	3.05 µg
Titanium	2	2	1.11-2.13 µg	0.716 µg
Vanadium	2	2	0.908-1.07 µg	0.292 µg
Zinc	2	2	14.3-42.2 µg	0.347 µg
GFAAS and CVAAS Metals				
Arsenic	2	1	0.410 µg	0.0946 µg
Cadmium	2	2	0.806-2.61 µg	0.0238 µg
Lead	2	2	0.877-1.79 µg	0.0800 µg
Mercury	2	2	0.0200-0.0230 µg	0.0033 µg
Selenium	2	0	ND	0.0802 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Laboratory Method Blank - HNO_3 /H_2O_2 Impingers				
ICP-AES Metals				
Aluminum	3	0	ND	0.0523 mg/L
Antimony	3	2	0.0301-0.0321 mg/L	0.0760 mg/L
Barium	3	1	0.00049 mg/L	0.00086 mg/L
Beryllium	3	0	ND	0.00051 mg/L
Boron	3	1	0.00888 mg/L	0.0176 mg/L
Calcium	3	3	0.0346-0.0573 mg/L	0.0175 mg/L
Chromium	3	1	0.00301 mg/L	0.00524 mg/L
Cobalt	3	0	ND	0.00407 mg/L
Copper	3	2	0.00154-0.0295 mg/L	0.00916 mg/L
Iron	3	3	0.00318-0.0490 mg/L	0.00452 mg/L
Magnesium	3	2	0.0136-0.0353 mg/L	0.0479 mg/L
Manganese	3	2	0.00130-0.00515 mg/L	0.00155 mg/L
Molybdenum	3	2	0.00124-0.00210 mg/L	0.00739 mg/L
Nickel	3	2	0.00563-0.0123 mg/L	0.0141 mg/L
Phosphorus	3	2	0.0483-0.116 mg/L	0.109 mg/L
Potassium	3	3	0.222-0.324 mg/L	0.822 mg/L
Silicon	3	2	0.0131-0.0332 mg/L	0.0318 mg/L
Sodium	3	3	0.0396-0.0465 mg/L	0.0401 mg/L
Titanium	3	2	0.00184-0.00275 mg/L	0.00159 mg/L
Vanadium	3	2	0.00015-0.00214 mg/L	0.00454 mg/L
Zinc	3	0	ND	0.00402 mg/L
GFAAS and CVAAS Metals				
Arsenic	2	0	ND	0.000647 mg/L
Cadmium	2	1	0.000320 mg/L	0.000270 mg/L
Lead	2	1	0.000230 mg/L	0.00205 mg/L
Mercury	3	1	0.000200 mg/L	0.000165 mg/L
Selenium	2	0	ND	0.00177 mg/L
Reagent Blank - HNO_3 /H_2O_2 Impingers				
ICP-AES Metals				
Aluminum	1	0	ND	0.0523 mg/L
Antimony	1	0	ND	0.0760 mg/L
Barium	1	1	0.00149 mg/L	0.00086 mg/L
Beryllium	1	0	ND	0.00051 mg/L
Boron	1	0	ND	0.0176 mg/L
Calcium	1	1	0.112 mg/L	0.0175 mg/L
Chromium	1	1	0.00451 mg/L	0.00524 mg/L
Cobalt	1	0	ND	0.00407 mg/L
Copper	1	1	0.00155 mg/L	0.00916 mg/L
Iron	1	1	0.0261 mg/L	0.00452 mg/L
Magnesium	1	1	0.0336 mg/L	0.0479 mg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Manganese	1	1	0.00129 mg/L	0.00155 mg/L
Molybdenum	1	1	0.00211 mg/L	0.00739 mg/L
Nickel	1	0	ND	0.0141 mg/L
Phosphorus	1	1	0.0482 mg/L	0.109 mg/L
Potassium	1	1	0.347 mg/L	0.822 mg/L
Silicon	1	1	0.270 mg/L	0.0318 mg/L
Sodium	1	1	0.203 mg/L	0.0401 mg/L
Titanium	1	1	0.000930 mg/L	0.00159 mg/L
Vanadium	1	0	ND	0.00454 mg/L
Zinc	1	1	0.0176 mg/L	0.00402 mg/L
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.000647 mg/L
Cadmium	1	0	ND	0.000270 mg/L
Lead	1	0	ND	0.000996 mg/L
Mercury	1	1	0.00080 mg/L	0.000165 mg/L
Selenium	1	0	ND	0.000592 mg/L
ICP-MS Metals				
Antimony	2	2	0.02-0.07 µg/L	0.077 µg/L
Arsenic	2	2	0.21-0.22 µg/L	0.138 µg/L
Barium	2	2	0.20-0.26 µg/L	NA
Beryllium	2	2	0.16-0.28 µg/L	0.142 µg/L
Cadmium	2	2	0.15-0.24 µg/L	0.160 µg/L
Chromium	2	2	6.19-14.76 µg/L	0.109 µg/L
Cobalt	2	2	0.10-0.17 µg/L	0.051 µg/L
Copper	2	1	8.97 µg/L	0.199 µg/L
Lead	2	2	0.31-0.38 µg/L	0.097 µg/L
Manganese	2	0	ND	0.080 µg/L
Mercury	2	0	ND	0.302 µg/L
Molybdenum	2	2	0.72-1.58 µg/L	0.134 µg/L
Nickel	2	2	2.85-8.54 µg/L	0.114 µg/L
Selenium	2	0	ND	0.591 µg/L
Vanadium	2	2	0.56-0.62 µg/L	0.068 µg/L
Field Blank - HNO_3/H_2O_2 Impingers				
ICP-AES Metals				
Aluminum	4	3	0.00734-0.0664 mg/L	0.0523 mg/L
Antimony	4	1	0.00312 mg/L	0.0760 mg/L
Barium	4	4	0.00049-0.0483 mg/L	0.00086 mg/L
Beryllium	4	0	ND	0.00051 mg/L
Boron	4	3	0.00006-0.0178 mg/L	0.0176 mg/L
Calcium	4	4	0.0919-0.277 mg/L	0.0175 mg/L
Chromium	4	4	0.00765-0.0130 mg/L	0.00524 mg/L
Cobalt	4	0	ND	0.00407 mg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Copper	4	4	0.00155-0.0194 mg/L	0.00916 mg/L
Iron	4	4	0.0254-0.5140 mg/L	0.00452 mg/L
Magnesium	4	4	0.0101-0.0430 mg/L	0.0479 mg/L
Manganese	4	4	0.00001-0.0499 mg/L	0.00155 mg/L
Molybdenum	4	4	0.00036-0.00728 mg/L	0.00739 mg/L
Nickel	4	2	0.00845-0.0141 mg/L	0.0141 mg/L
Phosphorus	4	4	0.0269-0.0723 mg/L	0.109 mg/L
Potassium	4	4	0.405-0.694 mg/L	0.822 mg/L
Silicon	4	4	0.0741-0.222 mg/L	0.0318 mg/L
Sodium	4	4	0.106-0.232 mg/L	0.0401 mg/L
Titanium	4	4	0.00092-0.00276 mg/L	0.00159 mg/L
Vanadium	4	2	0.00431-0.00477 mg/L	0.00454 mg/L
Zinc	4	4	0.0176-0.0450 mg/L	0.00402 mg/L
GFAAS and CVAAS Metals				
Arsenic	4	0	ND	0.000647 mg/L
Cadmium	4	4	0.00001-0.00157 mg/L	0.000270 mg/L
Lead	4	1	0.00072 mg/L	0.000996 mg/L
Mercury	4	4	0.00080-0.00465 mg/L	0.000165 mg/L
Selenium	4	1	0.00243 mg/L	0.000592 mg/L
ICP-MS Metals				
Antimony	3	3	0.04-0.07 µg/L	0.077 µg/L
Arsenic	3	3	0.21-0.31 µg/L	0.138 µg/L
Barium	3	3	0.34-0.47 µg/L	NA
Beryllium	3	3	0.16-0.35 µg/L	0.142 µg/L
Cadmium	3	3	0.68-1.42 µg/L	0.160 µg/L
Chromium	3	3	5.64-11.21 µg/L	0.109 µg/L
Cobalt	3	3	0.10-0.14 µg/L	0.051 µg/L
Copper	3	2	17.82-19.67 µg/L	0.199 µg/L
Lead	3	3	0.88-1.18 µg/L	0.097 µg/L
Manganese	3	0	ND	0.080 µg/L
Mercury	3	1	2.19 µg/L	0.302 µg/L
Molybdenum	3	3	0.47-1.29 µg/L	0.134 µg/L
Nickel	3	3	2.89-6.50 µg/L	0.114 µg/L
Selenium	3	2	0.04-2.15 µg/L	0.591 µg/L
Vanadium	3	3	0.53-0.65 µg/L	0.068 µg/L
Laboratory Method Blank - $KMnO_4$ Impingers				
Mercury by CVAAS	1	0	ND	0.000033 mg/L
Reagent Blank - $KMnO_4$ Impingers				
Mercury by CVAAS	1	1	0.00015 mg/L	0.000033 mg/L
Field Blank - $KMnO_4$ Impingers				
Mercury by CVAAS	1	1	0.00001 mg/L	0.000033 mg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Laboratory Method Blank - Charcoal Tube				
ICP-AES Metals				
Aluminum	1	1	0.403 µg	2.76 µg
Antimony	1	1	3.84 µg	5.86 µg
Barium	1	1	0.103 µg	0.0697 µg
Beryllium	1	0	ND	0.0329 µg
Boron	1	1	2.71 µg	0.938 µg
Calcium	1	1	4.52 µg	1.37 µg
Chromium	1	1	0.0120 µg	0.197 µg
Cobalt	1	0	ND	0.538 µg
Copper	1	1	0.0790 µg	0.502 µg
Iron	1	1	0.459 µg	0.509 µg
Magnesium	1	1	2.68 µg	9.63 µg
Manganese	1	0	ND	0.492 µg
Molybdenum	1	1	0.161 µg	0.384 µg
Nickel	1	0	ND	1.14 µg
Phosphorus	1	0	ND	10.9 µg
Potassium	1	1	7.16 µg	44.1 µg
Silicon	1	1	18.6 µg	29.3 µg
Sodium	1	1	30.2 µg	3.05 µg
Titanium	1	0	ND	0.716 µg
Vanadium	1	0	ND	0.292 µg
Zinc	1	1	0.0200 µg	0.347 µg
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.118 µg
Cadmium	1	0	ND	0.0783 µg
Lead	1	0	ND	0.0776 µg
Mercury	3	0	ND	0.000033 µg
Selenium	1	0	ND	0.0802 µg
Reagent Blank - Charcoal Tube				
ICP-AES Metals				
Aluminum	2	2	17.8-42.8 µg	2.76 µg
Antimony	2	2	5.48-5.80 µg	5.86 µg
Barium	2	2	0.412-0.670 µg	0.0697 µg
Beryllium	2	0	ND	0.0329 µg
Boron	2	2	8.25-9.15 µg	0.938 µg
Calcium	2	2	16.2-22.6 µg	1.37 µg
Chromium	2	2	8.19-10.5 µg	0.197 µg
Cobalt	2	1	0.317 µg	0.538 µg
Copper	2	2	2.55-3.11 µg	0.502 µg
Iron	2	2	173-201 µg	0.509 µg
Magnesium	2	2	8.64-12.7 µg	9.63 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Manganese	2	2	0.479-1.53 µg	0.492 µg
Molybdenum	2	2	2.80-2.84 µg	0.384 µg
Nickel	2	2	0.380-1.52 µg	1.14 µg
Phosphorus	2	2	1.69-13.4 µg	10.9 µg
Potassium	2	2	326-370	44.1 µg
Silicon	2	2	79.4-115 µg	29.3 µg
Sodium	2	2	16.4-23.8 µg	3.05 µg
Titanium	2	2	3.30-4.53 µg	0.716 µg
Vanadium	2	2	0.565-0.776 µg	0.292 µg
Zinc	2	0	ND	0.347 µg
GFAAS and CVAAS Metals				
Arsenic	2	0	ND	0.118 µg
Cadmium	2	0	ND	0.0783 µg
Lead	2	0	ND	0.0776 µg
Mercury	2	2	0.169-0.181 µg	0.0033 µg
Selenium	2	0	ND	0.0802 µg
Mercury				
Field Blank - Charcoal Tube				
ICP-AES Metals				
Aluminum	1	1	33.7 µg	2.76 µg
Antimony	1	1	0.0410 µg	5.86 µg
Barium	1	1	0.463 µg	0.0697 µg
Beryllium	1	0	ND	0.0329 µg
Boron	1	1	9.16 µg	0.938 µg
Calcium	1	1	24.4 µg	1.37 µg
Chromium	1	1	7.80 µg	0.197 µg
Cobalt	1	1	0.652 µg	0.538 µg
Copper	1	1	4.14 µg	0.502 µg
Iron	1	1	204 µg	0.509 µg
Magnesium	1	1	14.4 µg	9.63 µg
Manganese	1	1	0.625 µg	0.492 µg
Molybdenum	1	1	4.25 µg	0.384 µg
Nickel	1	1	1.24 µg	1.14 µg
Phosphorus	1	1	11.0 µg	10.9 µg
Potassium	1	1	361 µg	44.1 µg
Silicon	1	1	126 µg	29.3 µg
Sodium	1	1	58.2 µg	3.05 µg
Titanium	1	1	2.93 µg	0.716 µg
Vanadium	1	1	1.05 µg	0.292 µg
Zinc	1	1	1.14 µg	0.347 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.118 µg
Cadmium	1	1	0.0020 µg	0.0783 µg
Lead	1	0	ND	0.0776 µg
Mercury	1	1	0.196 µg	0.0033 µg
Selenium	1	0	ND	0.0802 µg
Mercury				
Laboratory Method Blank - Aqueous Samples				
ICP-AES Metals				
Aluminum	2	0	ND	0.0523 mg/L
Antimony	2	1	0.0321 mg/L	0.0760 mg/L
Barium	2	2	0.00049 mg/L	0.00086 mg/L
Beryllium	2	0	ND	0.00051 mg/L
Boron	2	0	ND	0.0176 mg/L
Calcium	2	2	0.0205-0.0384 mg/L	0.0175 mg/L
Chromium	2	1	0.00308 mg/L	0.00524 mg/L
Cobalt	2	0	ND	0.00407 mg/L
Copper	2	2	0.00154-0.00233 mg/L	0.00916 mg/L
Iron	2	2	0.00637-0.00965 mg/L	0.00452 mg/L
Magnesium	2	2	0.0227-0.0353 mg/L	0.0479 mg/L
Manganese	2	2	0.00128-0.00515 mg/L	0.00155 mg/L
Molybdenum	2	0	ND	0.00739 mg/L
Nickel	2	1	0.0123 mg/L	0.0141 mg/L
Phosphorus	2	1	0.116 mg/L	0.109 mg/L
Potassium	2	2	0.222-0.717 mg/L	0.822 mg/L
Silicon	2	2	0.0275-0.0332 mg/L	0.0318 mg/L
Sodium	2	2	0.0156-0.0465 mg/L	0.0401 mg/L
Titanium	2	1	0.00275 mg/L	0.00159 mg/L
Vanadium	2	1	0.00015 mg/L	0.00454 mg/L
Zinc	2	1	0.00003 mg/L	0.00402 mg/L
GFAAS and CVAAS Metals				
Arsenic	1	0	ND	0.000647 mg/L
Cadmium	1	0	ND	0.00027 mg/L
Lead	3	1	0.00029 mg/L	0.0022 mg/L
Mercury	1	0	ND	0.000033 mg/L
Selenium	1	0	ND	0.000592 mg/L
Laboratory Method Blank - Solid Samples				
ICP-MS Metals				
Antimony	3	3	0.10-0.33 µg/L	
Arsenic	3	2	0.02-0.08 µg/L	
Barium	3	3	0.65-2.23 µg/L	
Beryllium	3	0	ND	

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Cadmium	3	2	0.04-0.09 µg/L	
Chromium	3	3	2.19-2.70 µg/L	
Cobalt	3	2	0.01-0.34 µg/L	
Copper	3	1	0.09 µg/L	
Lead	3	3	0.19-0.51 µg/L	
Manganese	3	1	0.03 µg/L	
Mercury	3	2	0.21-0.27 µg/L	
Molybdenum	3	3	0.08-0.29 µg/L	
Nickel	3	0	ND	
Selenium	3	0	ND	
Vanadium	3	0	ND	
Laboratory Method Blank - Filter & PNR				
Anions				
Chloride (EPA 300)	1	0	ND	2.00 µg
Fluoride (EPA 340.2)	1	1	1.70 µg	2.35 µg
Sulfate (EPA 300)	1	1	102 µg	6.0 µg
Reagent Blank - Filter & PNR				
Anions				
Chloride (EPA 300)	2	2	10.3-13.2 µg	0.0225 mg/L
Fluoride (EPA 340.2)	2	2	1.69-1.89 µg	0.00551 mg/L
Sulfate (EPA 300)	2	2	116-1640 µg	0.0471 mg/L
Field Blank - Filter & PNR				
Anions				
Chloride (EPA 300)	1	1	24.8 µg	2.00 µg
Fluoride (EPA 340.2)	1	1	3.80 µg	2.35 µg
Sulfate (EPA 300)	1	1	607 µg	6.00 µg
Laboratory Method Blank - H_2SO_4 Impingers				
Anions				
Chloride (EPA 300)	4	0	ND	0.0281 mg/L
Fluoride (EPA 340.2)	2	2	0.0178-0.0193 mg/L	0.00551 mg/L
Ammonia in Stack Gas				
Ammonia (EPA 350.2)	3	2	0.0296-0.0371 mg/L	0.0156 mg/L
Reagent Blank -H_2SO_4 Impingers				
Anions				
Chloride (EPA 300)	1	0	ND	2.81 mg/L
Fluoride (EPA 340.2)	1	1	0.0318 mg/L	0.00551 mg/L
Ammonia in Stack Gas				
Ammonia (EPA 350.2)	1	1	0.348 mg/L	0.0624 mg/L
Field Blank - H_2SO_4 Impingers				
Anions				
Chloride (EPA 300)	2	0	ND	2.81 mg/L
Fluoride (EPA 340.2)	2	2	0.0245-0.0256 mg/L	0.00551 mg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Ammonia in Stack Gas				
Ammonia (EPA 350.2)	3	3	0.446-1.17 mg/L	0.0624 mg/L
Laboratory Method Blank - Cyanide in Stack Gas				
Cyanide (SW 9012)	5	5	0.0017-0.0202 mg/L	0.00942 mg/L
Reagent Blank - Cyanide in Stack Gas				
Cyanide (SW 9012)	1	1	0.0006 mg/L	0.00942 mg/L
Field Blanks - Cyanide in Stack Gas				
Cyanide (SW 9012)	3	3	0.0006-0.274 mg/L	0.00942 mg/L
Laboratory Method Blank - $KMnO_4$/NaOH Impingers				
Nitrate (Method 7E)	1	0	ND	0.0280 mg/L
Reagent Blank - $KMnO_4$/NaOH Impingers				
Nitrate (Method 7d)	1	0	ND	0.0280 mg/L
Reagent Blank - IPA Impingers				
Sulfate (EPA 300)	1	1	3.15 mg/L	0.0471 mg/L
Field Blank - IPA Impingers				
Sulfate (EPA 300)	1	1	13.9 mg/L	0.0471 mg/L
Laboratory Method Blank - H_2O_2 Impingers				
Sulfate (EPA 300)	2	0	ND	0.0471 mg/L
Reagent Blank - H_2O_2 Impingers				
Sulfate (EPA 300)	1	1	1.99 mg/L	0.0471 mg/L
Field Blank - H_2O_2 Impingers				
Sulfate (EPA 300)	1	1	15.3 mg/L	0.0471 mg/L
Laboratory Method Blank - Aqueous Samples				
Anions				
Chloride (EPA 300)	1	0	ND	0.0281 mg/L
Fluoride (EPA 340.2)	1	1	0.0181 mg/L	0.00551 mg/L
Formate (IC)	1	0	ND	0.25 mg/L
Sulfate (EPA 300)	2	0	ND	0.0471 mg/L
Phosphate as Total Phosphorus (EPA 365.2)	1	0	ND	0.00692 mg/L
Thiocyanate	4	0	0.0259-0.2342 mg/L	
Ammonia in Aqueous				
Ammonia (EPA 350.2)	3	3	0.0063-0.0678 mg/L	0.0156 mg/L
Cyanide in Aqueous				
Cyanide (SW 9012)	5	5	0.0000104-0.0050 mg/L	0.00942 mg/L
Total Phenolics in Aqueous				
Total Phenolics (EPA 420.2)	2	2	0.00168-0.00307 mg/L	0.0108 mg/L
Laboratory Method Blank - Solid Samples				
Anions				
Chloride (Potentiometric)	1	0	ND	1.33 mg/kg
Chloride (EPA 300)	3	0	ND	0.0200 mg/kg
Fluoride (EPA 340.2)	1	2	12.1-13.7 mg/kg	11.8 mg/kg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Fluoride (EPA 300)	4	0	ND	0.0490 mg/kg
Laboratory Method Blank - Aldehydes in DNPH Impingers				
Formaldehyde	3	0	ND	0.50 µg
Acetaldehyde	3	0	ND	0.50 µg
Acrolein	3	0	ND	0.50 µg
Benzaldehyde	3	0	ND	0.50 µg
Reagent Blanks - Aldehydes in DNPH Impingers				
Formaldehyde	6	3	1.8-5.5 µg	0.50 µg
Acetaldehyde	6	1	0.66 µg	0.50 µg
Acrolein	6	0	ND	0.50 µg
Benzaldehyde	6	0	ND	0.50 µg
Field Blanks - Aldehydes in DNPH Impingers				
Formaldehyde	2	2	2.6-2.7 µg	0.50 µg
Acetaldehyde	2	1	0.52 µg	0.50 µg
Acrolein	2	0	ND	0.50 µg
Benzaldehyde	2	0	ND	0.50 µg
Laboratory Method Blank - Aldehydes in Aqueous Samples				
Formaldehyde	2	0	ND	0.010 µg/ml
Acetaldehyde	2	0	ND	0.010 µg/ml
Acrolein	2	0	ND	0.010 µg/ml
Benzaldehyde	2	0	ND	0.010 µg/ml
Laboratory Method Blank - VOST				
Volatile Organic Compounds				
Chloromethane	3	2	10-30 ng	10 ng
Vinyl Chloride	3	0	ND	10 ng
Bromomethane	3	1	10 ng	10 ng
Chloroethane	3	0	ND	10 ng
Trichlorofluoromethane	3	0	ND	10 ng
1,1-Dichloroethene	3	0	ND	10 ng
Carbon Disulfide	3	0	ND	10 ng
Acetone	3	0	ND	50 ng
Methylene Chloride	3	0	ND	10 ng
trans-1,2-Dichloroethene	3	0	ND	10 ng
1,1-Dichloroethane	3	0	ND	10 ng
Vinyl Acetate	3	0	ND	50 ng
2-Butanone	3	0	ND	50 ng
Chloroform	3	0	ND	10 ng
1,1,1-Trichloroethane	3	0	ND	10 ng
Carbon Tetrachloride	3	0	ND	10 ng
Benzene	3	0	ND	10 ng
1,2-Dichloroethane	3	0	ND	10 ng
Trichloroethene	3	0	ND	10 ng

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
1,2-Dichloropropane	3	0	ND	10 ng
Bromodichloromethane	3	0	ND	10 ng
trans-1,3-Dichloropropene	3	0	ND	10 ng
4-methyl-2-Pentanone	3	0	ND	50 ng
Toluene	3	0	ND	10 ng
cis-1,3-Dichloropropene	3	0	ND	10 ng
1,1,2-Trichloroethane	3	0	ND	10 ng
Tetrachloroethene	3	0	ND	10 ng
2-Hexanone	3	0	ND	50 ng
Dibromochloromethane	3	0	ND	10 ng
Chlorobenzene	3	0	ND	10 ng
Ethyl Benzene	3	0	ND	10 ng
m,p-Xylene	3	0	ND	10 ng
o-Xylene	3	0	ND	10 ng
Styrene	3	0	ND	10 ng
Bromoform	3	0	ND	10 ng
1,1,2,2-Tetrachloroethane	3	0	ND	10 ng
1,3-Dichlorobenzene	3	0	ND	10 ng
1,4-Dichlorobenzene	3	0	ND	10 ng
1,2-Dichlorobenzene	3	0	ND	10 ng
Trip Blank - VOST				
Volatile Organic Compounds				
Chloromethane	1	0	ND	10 ng
Vinyl Chloride	1	0	ND	10 ng
Bromomethane	1	0	ND	10 ng
Chloroethane	1	0	ND	10 ng
Trichlorofluoromethane	1	0	ND	10 ng
1,1-Dichloroethene	1	0	ND	10 ng
Carbon Disulfide	1	0	ND	10 ng
Acetone	1	0	ND	50 ng
Methylene Chloride	1	0	ND	10 ng
trans-1,2-Dichloroethene	1	0	ND	10 ng
1,1-Dichloroethane	1	0	ND	10 ng
Vinyl Acetate	1	0	ND	50 ng
2-Butanone	1	0	ND	50 ng
Chloroform	1	0	ND	10 ng
1,1,1-Trichloroethane	1	0	ND	10 ng
Carbon Tetrachloride	1	0	ND	10 ng
Benzene	1	0	ND	10 ng
1,2-Dichloroethane	1	0	ND	10 ng
Trichloroethene	1	0	ND	10 ng
1,2-Dichloropropane	1	0	ND	10 ng

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
trans-1,3-Dichloropropene	5	0	ND	10 ng
4-methyl-2-Pentanone	5	0	ND	50 ng
Toluene	5	0	ND	10 ng
cis-1,3-Dichloropropene	5	0	ND	10 ng
1,1,2-Trichloroethane	5	0	ND	10 ng
Tetrachloroethene	5	0	ND	10 ng
2-Hexanone	5	0	ND	50 ng
Dibromochloromethane	5	0	ND	10 ng
Chlorobenzene	5	0	ND	10 ng
Ethyl Benzene	5	0	ND	10 ng
m,p-Xylene	5	0	ND	10 ng
o-Xylene	5	0	ND	10 ng
Styrene	5	0	ND	10 ng
Bromoform	5	0	ND	10 ng
1,1,2,2-Tetrachloroethane	5	0	ND	10 ng
1,3-Dichlorobenzene	5	0	ND	10 ng
1,4-Dichlorobenzene	5	0	ND	10 ng
1,2-Dichlorobenzene	5	0	ND	10 ng
Trip Blank - Volatile Organic Compounds in Stack Gas - Canisters				
C-4 VOC	2	0	ND	
C-5 VOC	2	1	0.0002 ppmv	
Benzene	2	2	0.0002 ppmv	
Toluene	2	0	ND	
Ethyl Benzene	2	0	ND	
m,p-Xylene	2	0	ND	
o-Xylene	2	0	ND	
Isopropylbenzene	2	0	ND	
N-Propylbenzene	2	0	ND	
M-Ethyltoluene	2	0	ND	
P-Ethyltoluene	2	0	ND	
O-Ethyltoluene	2	0	ND	
1,3,5-Trimethylbenzene	2	0	ND	
1,2,4-Trimethylbenzene + T-Butylbenzene	2	0	ND	
Isobutylbenzene	2	0	ND	
1,2,3-Trimethylbenzene	2	0	ND	
P-Isopropyltoluene	2	0	ND	
M-Diethylbenzene	2	0	ND	
N-Butylbenzene	2	0	ND	
P-Diethylbenzene	2	0	ND	
Naphthalene	2	0	ND	

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Bromodichloromethane	1	0	ND	10 ng
trans-1,3-Dichloropropene	1	0	ND	10 ng
4-methyl-2-Pentanone	1	0	ND	50 ng
Toluene	1	0	ND	10 ng
cis-1,3-Dichloropropene	1	0	ND	10 ng
1,1,2-Trichloroethane	1	0	ND	10 ng
Tetrachloroethene	1	0	ND	10 ng
2-Hexanone	1	0	ND	50 ng
Dibromochloromethane	1	0	ND	10 ng
Chlorobenzene	1	0	ND	10 ng
Ethyl Benzene	1	0	ND	10 ng
m,p-Xylene	1	0	ND	10 ng
o-Xylene	1	0	ND	.10 ng
Styrene	1	0	ND	10 ng
Bromoform	1	0	ND	10 ng
1,1,2,2-Tetrachloroethane	1	0	ND	10 ng
1,3-Dichlorobenzene	1	0	ND	10 ng
1,4-Dichlorobenzene	1	0	ND	10 ng
1,2-Dichlorobenzene	1	0	ND	10 ng
Field Blank - VOST				
Volatile Organic Compounds				
Chloromethane	5	0	ND	10 ng
Vinyl Chloride	5	0	ND	10 ng
Bromomethane	5	0	ND	10 ng
Chloroethane	5	0	ND	10 ng
Trichlorofluoromethane	5	1	31 ng	10 ng
1,1-Dichloroethene	5	0	ND	10 ng
Carbon Disulfide	5	1	10 ng	10 ng
Acetone	5	0	ND	50 ng
Methylene Chloride	5	4	36-450 ng	10 ng
trans-1,2-Dichloroethene	5	0	ND	10 ng
1,1-Dichloroethane	5	0	ND	10 ng
Vinyl Acetate	5	0	ND	50 ng
2-Butanone	5	0	ND	50 ng
Chloroform	5	0	ND	10 ng
1,1,1-Trichloroethane	5	0	ND	10 ng
Carbon Tetrachloride	5	0	ND	10 ng
Benzene	5	0	ND	10 ng
1,2-Dichloroethane	5	0	ND	10 ng
Trichloroethene	5	0	ND	10 ng
1,2-Dichloropropane	5	0	ND	10 ng
Bromodichloromethane	5	0	ND	10 ng

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
4-Aminobiphenyl	4	0	ND	1.07-1.82 µg
Aniline	4	0	ND	0.809-0.951 µg
Anthracene	4	0	ND	0.510-0.539 µg
Benz(a)anthracene	4	0	ND	0.348-0.401 µg
Benz(a)pyrene	4	0	ND	0.540-0.625 µg
Benzidine	4	0	ND	20.0 µg
Benzo(b)fluoranthene	4	0	ND	0.983-0.959 µg
Benzo(g,h,i)perylene	4	0	ND	0.435-0.554 µg
Benzo(k)fluoranthene	4	0	ND	0.660-0.820 µg
Benzoic Acid	4	0	ND	2.99-6.03 µg
Benzyl Alcohol	4	0	ND	0.461-1.44 µg
4-Bromophenylphenylether	4	0	ND	0.543-0.610 µg
Butylbenzylphthalate	4	0	ND	0.528-0.751 µg
4-Chloro-3-methylphenol	4	0	ND	0.335-0.458 µg
p-Chloroaniline	4	0	ND	0.595-0.980 µg
bis(2-Chloroethoxy)methane	4	0	ND	0.276-0.334 µg
bis(2-Chloroethyl)ether	4	0	ND	0.397-0.466 µg
bis(2-Chloroisopropyl)ether	4	0	ND	0.434-0.571 µg
2-Chloronaphthalene	4	0	ND	0.625-0.899 µg
2-Chlorophenol	4	0	ND	0.328-0.452 µg
4-Chlorophenylphenylether	4	0	ND	0.732-0.755 µg
Chrysene	4	0	ND	0.274-0.585 µg
Di-n-butylphthalate	4	0	ND	0.310-0.438 µg
Di-n-octylphthalate	4	0	ND	0.442-0.582 µg
Dibenz(a,h)anthracene	4	0	ND	0.486-0.683 µg
Dibenzofuran	4	0	ND	0.360-0.391 µg
1,2-Dichlorobenzene	4	0	ND	0.306-0.641 µg
1,3-Dichlorobenzene	4	0	ND	0.286-0.712 µg
1,4-Dichlorobenzene	4	0	ND	0.485-0.707 µg
3,3-Dichlorobenzidine	4	0	ND	1.05-1.09 µg
2,4-Dichlorophenol	4	0	ND	0.314-0.581 µg
Diethylphthalate	4	0	ND	0.315-0.370 µg
P-Dimethylaminoazobenzene	4	0	ND	2.00-2.25 µg
2,4-Dimethylphenol	4	0	ND	1.26-1.27 µg
Dimethylphthalate	4	0	ND	0.323-0.471 µg
4,6-Dinitro-2-methylphenol	4	0	ND	0.731-0.758 µg
2,4-Dinitrophenol	4	0	ND	2.59-2.67 µg
2,4-Dinitrotoluene	4	0	ND	0.457-0.769 µg
2,6-Dinitrotoluene	4	0	ND	0.387-1.05 µg
Diphenylamine/N-NitrosoDPA	4	0	ND	0.744-0.764 µg
bis(2-Ethylhexyl)phthalate	4	1	1.84 µg	1.64-1.77 µg
Fluoranthene	4	0	ND	0.169-0.425 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Laboratory Method Blank - Volatile Organic Compounds in Aqueous Samples				
Chloromethane	2	0	ND	0.519 µg/L
Vinyl Chloride	2	0	ND	0.685 µg/L
Bromomethane	2	0	ND	0.539 µg/L
Chloroethane	2	0	ND	0.772 µg/L
1,1-Dichloroethene	2	0	ND	0.344 µg/L
Carbon Disulfide	2	0	ND	0.491 µg/L
Acetone	2	2	3.97-4.41 µg/L	2.87 µg/L
Methylene Chloride	2	2	2.05-5.70 µg/L	3.03 µg/L
trans-1,2-Dichloroethene	2	0	ND	0.541 µg/L
1,1-Dichloroethane	2	0	ND	0.590 µg/l
Vinyl Acetate	2	0	ND	0.638 µg/L
2-Butanone	2	0	ND	1.60 µg/L
Chloroform	2	0	ND	0.533 µg/L
1,1,1-Trichloroethane	2	0	ND	0.870 µg/L
Carbon Tetrachloride	2	0	ND	0.796 µg/L
Benzene	2	0	ND	0.462 µg/L
1,2-Dichloroethane	2	0	ND	0.822 µg/L
Trichloroethene	2	0	ND	0.455 µg/L
1,2-Dichloropropane	2	0	ND	0.161 µg/L
Bromodichloromethane	2	0	ND	0.370 µg/L
trans-1,3-Dichloropropene	2	0	ND	0.416 µg/L
4-methyl-2-Pentanone	2	0	ND	0.493 µg/L
Toluene	2	0	ND	0.409 µg/L
cis-1,3-Dichloropropene	2	0	ND	0.414 µg/L
1,1,2-Trichloroethane	2	0	ND	0.268 µg/L
Tetrachloroethene	2	0	ND	0.541 µg/L
2-Hexanone	2	0	ND	0.713 µg/L
Dibromochloromethane	2	0	ND	0.246 µg/L
Chlorobenzene	2	0	ND	0.319 µg/l
Ethyl Benzene	2	0	ND	0.588 µg/L
m,p-Xylene	2	0	ND	0.509 µg/L
o-Xylene	2	0	ND	0.402 µg/L
Styrene	2	0	ND	0.432 µg/L
Bromoform	2	0	ND	0.563 µg/L
1,1,2,2-Tetrachloroethane	2	0	ND	0.627 µg/L
1,4-Dichlorobenzene	2	0	ND	0.558 µg/L
Laboratory Method Blank - XAD & Condensate				
Semivolatile Organic Compounds by SW8270				
Acenaphthene	4	0	ND	0.411-0.482 µg
Acenaphthylene	4	0	ND	0.658-0.761 µg
Acetophenone	4	0	ND	0.338-0.355 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Fluorene	4	0	ND	0.284-0.345 µg
Hexachlorobenzene	4	0	ND	0.474-0.528 µg
Hexachlorobutadiene	4	0	ND	0.242-0.657 µg
Hexachlorocyclopentadiene	4	0	ND	1.64 µg
Hexachloroethane	4	0	ND	0.246-1.00 µg
Indeno(1,2,3)pyrene	4	0	ND	0.481-519 µg
Isophorone	4	0	ND	0.302-0.369 µg
2-Methylnaphthalene	4	0	ND	0.537-0.605 µg
4-Methylphenol/3-Methylphenol	4	0	ND	1.04-1.24 µg
2-Methylphenol	4	0	ND	0.790-1.15 µg
N-Nitrosodipropylamine	4	0	ND	0.569-0.785 µg
N-Nitrosodimethylamine	4	0	ND	0.417-0.783 µg
Naphthalene	4	2	0.923-1.15 µg	0.627-0.668 µg
2-Nitroaniline	4	0	ND	0.422-0.799 µg
3-Nitroaniline	4	0	ND	0.322-0.507 µg
4-Nitroaniline	4	0	ND	0.510-0.599 µg
Nitrobenzene	4	0	ND	0.325-0.389 µg
2-Nitrophenol	4	0	ND	0.425-0.502 µg
4-Nitrophenol	4	0	ND	0.455-0.631 µg
Pentachloronitrobenzene	4	0	ND	1.11-2.34 µg
Pentachlorophenol	4	0	ND	0.181-0.192 µg
Phenanthrene	4	0	ND	0.374-0.554 µg
Phenol	4	0	ND	0.535-1.01 µg
Pyrene	4	0	ND	0.244-0.462 µg
1,2,4-Trichlorobenzene	4	0	ND	0.192-0.218 µg
2,4,5-Trichlorophenol	4	0	ND	0.410-0.586 µg
2,4,6-Trichlorophenol	4	0	ND	0.377-0.485 µg
Trip Blank - XAD & Condensate				
Semivolatile Organic Compounds by SW8270				
Acenaphthene	2	0	ND	0.482 µg
Acenaphthylene	2	0	ND	0.658 µg
Acetophenone	2	1	0.549 µg	0.355 µg
4-Aminobiphenyl	2	0	ND	1.07 µg
Aniline	2	0	ND	0.951 µg
Anthracene	2	0	ND	0.539 µg
Benz(a)anthracene	2	0	ND	0.348 µg
Benz(a)pyrene	2	0	ND	0.540 µg
Benzidine	2	0	ND	20.0 µg
Benzo(b)fluoranthene	2	0	ND	0.959 µg
Benzo(g,h,i)perylene	2	0	ND	0.554 µg
Benzo(k)fluoranthene	2	0	ND	0.820 µg
Benzoic Acid	2	1	2.92 µg	2.99 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Benzyl Alcohol	2	0	ND	1.44 µg
4-Bromophenylphenylether	2	0	ND	0.610 µg
Butylbenzylphthalate	2	0	ND	0.751 µg
4-Chloro-3-methylphenol	2	0	ND	0.458 µg
p-Chloroaniline	2	0	ND	0.980 µg
bis(2-Chloroethoxy)methane	2	0	ND	0.334 µg
bis(2-Chloroethyl)ether	2	0	ND	0.466 µg
bis(2-Chloroisopropyl)ether	2	0	ND	0.571 µg
2-Chloronaphthalene	2	0	ND	0.899 µg
2-Chlorophenol	2	0	ND	0.328 µg
4-Chlorophenylphenylether	2	0	ND	0.732 µg
Chrysene	2	0	ND	0.585 µg
Di-n-butylphthalate	2	0	ND	0.310 µg
Di-n-octylphthalate	2	0	ND	0.582 µg
Dibenz(a,h)anthracene	2	0	ND	0.683 µg
Dibenzofuran	2	0	ND	0.391 µg
1,2-Dichlorobenzene	2	0	ND	0.641 µg
1,3-Dichlorobenzene	2	0	ND	0.712 µg
1,4-Dichlorobenzene	2	0	ND	0.707 µg
3,3-Dichlorobenzidine	2	0	ND	1.09 µg
2,4-Dichlorophenol	2	0	ND	0.581 µg
Diethylphthalate	2	0	ND	0.370 µg
P-Dimethylaminoazobenzene	2	0	ND	2.00 µg
2,4-Dimethylphenol	2	0	ND	1.27 µg
Dimethylphthalate	2	0	ND	0.471 µg
4,6-Dinitro-2-methylphenol	2	0	ND	0.731 µg
2,4-Dinitrophenol	2	0	ND	2.59 µg
2,4-Dinitrotoluene	2	0	ND	0.769 µg
2,6-Dinitrotoluene	2	0	ND	1.05 µg
Diphenylamine/N-NitrosoDPA	2	0	ND	0.764 µg
bis(2-Ethylhexyl)phthalate	2	0	ND	1.64 µg
Fluoranthene	2	0	ND	0.425 µg
Fluorene	2	0	ND	0.345 µg
Hexachlorobenzene	2	0	ND	0.528 µg
Hexachlorobutadiene	2	0	ND	0.657 µg
Hexachlorocyclopentadiene	2	0	ND	1.64 µg
Hexachloroethane	2	0	ND	1.00 µg
Indeno(1,2,3)pyrene	2	0	ND	0.481 µg
Isophorone	2	0	ND	0.302 µg
2-Methylnaphthalene	2	0	ND	0.605 µg
4-Methylphenol/3-Methylphenol	2	0	ND	1.24 µg
2-Methylphenol	2	0	ND	0.790 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
N-Nitrosodipropylamine	2	0	ND	0.785 µg
N-Nitrosodimethylamine	2	0	ND	0.783 µg
Naphthalene	2	2	2.02-2.40 µg	0.668 µg
2-Nitroaniline	2	0	ND	0.799 µg
3-Nitroaniline	2	0	ND	0.322 µg
4-Nitroaniline	2	0	ND	0.599 µg
Nitrobenzene	2	0	ND	0.389 µg
2-Nitrophenol	2	0	ND	0.425 µg
4-Nitrophenol	2	0	ND	0.631 µg
Pentachloronitrobenzene	2	0	ND	2.34 µg
Pentachlorophenol	2	0	ND	0.192 µg
Phenanthrene	2	0	ND	0.554 µg
Phenol	2	0	ND	1.01 µg
Pyrene	2	0	ND	0.462 µg
1,2,4-Trichlorobenzene	2	0	ND	0.192 µg
2,4,5-Trichlorophenol	2	0	ND	0.586 µg
2,4,6-Trichlorophenol	2	0	ND	0.485 µg
Laboratory Method Blank - Filter & PNR				
Semivolatile Organic Compounds by S8270				
Acenaphthene	1	0	ND	0.37 µg
Acenaphthylene	1	0	ND	0.22 µg
Acetophenone	1	0	ND	0.48 µg
4-Aminobiphenyl	1	0	ND	0.38 µg
Aniline	1	0	ND	0.36 µg
Anthracene	1	0	ND	0.29 µg
Benzo(a)anthracene	1	0	ND	0.44 µg
Benzo(a)pyrene	1	0	ND	0.51 µg
Benzidine	1	0	ND	0.65 µg
Benzo(b)fluoranthene	1	0	ND	0.47 µg
Benzo(g,h,i)perylene	1	0	ND	0.55 µg
Benzo(k)fluoranthene	1	0	ND	0.49 µg
Benzoic Acid	1	0	ND	1.28 µg
Benzyl Alcohol	1	1	0.82 µg	4.05 µg
4-Bromophenylphenylether	1	0	ND	1.40 µg
Butylbenzylphthalate	1	0	ND	0.53 µg
4-Chloro-3-methylphenol	1	0	ND	0.72 µg
p-Chloroaniline	1	0	ND	0.49 µg
bis(2-Chloroethoxy)methane	1	0	ND	0.54 µg
bis(2-Chloroethyl)ether	1	0	ND	0.63 µg
bis(2-Chloroisopropyl)ether	1	0	ND	0.98 µg
2-Chloronaphthalene	1	0	ND	0.34 µg
2-Chlorophenol	1	0	ND	0.56 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
4-Chlorophenylphenylether	1	0	ND	0.83 µg
Chrysene	1	0	ND	0.48 µg
Di-n-butylphthalate	1	1	3.19 µg	--
Di-n-octylphthalate	1	0	ND	0.27 µg
Dibenz(a,h)anthracene	1	0	ND	0.63 µg
Dibenzofuran	1	0	ND	0.25 µg
1,2-Dichlorobenzene	1	0	ND	0.57 µg
1,3-Dichlorobenzene	1	0	ND	0.53 µg
1,4-Dichlorobenzene	1	0	ND	0.53 µg
3,3-Dichlorobenzidine	1	0	ND	1.08 µg
2,4-Dichlorophenol	1	0	ND	0.71 µg
Diethylphthalate	1	1	4.70 µg	1.04 µg
P-Dimethylaminoazobenzene	1	0	ND	0.93 µg
2,4-Dimethylphenol	1	0	ND	0.65 µg
Dimethylphthalate	1	0	ND	0.32 µg
4,6-Dinitro-2-methylphenol	1	0	ND	2.23 µg
2,4-Dinitrophenol	1	0	ND	2.85 µg
2,4-Dinitrotoluene	1	0	ND	0.96 µg
2,6-Dinitrotoluene	1	0	ND	1.21 µg
bis(2-Ethylhexyl)phthalate	1	1	0.40 µg	--
Fluoranthene	1	0	ND	0.30 µg
Fluorene	1	0	ND	0.35 µg
Hexachlorobenzene	1	0	ND	1.04 µg
Hexachlorobutadiene	1	0	ND	1.31 µg
Hexachlorocyclopentadiene	1	0	ND	1.11 µg
Hexachloroethane	1	0	ND	1.08 µg
Indeno(1,2,3)pyrene	1	0	ND	0.50 µg
Isophorone	1	0	ND	0.35 µg
2-Methylnaphthalene	1	0	ND	0.34 µg
4-Methylphenol/3-Methylphenol	1	0	ND	0.61 µg
2-Methylphenol	1	0	ND	0.64 µg
N-Nitrosodipropylamine	1	0	ND	1.11 µg
N-Nitrosodimethylamine	1	0	ND	1.85 µg
Naphthalene	1	0	ND	0.22 µg
2-Nitroaniline	1	0	ND	1.29 µg
3-Nitroaniline	1	0	ND	1.17 µg
4-Nitroaniline	1	0	ND	1.31 µg
Nitrobenzene	1	0	ND	0.62 µg
2-Nitrophenol	1	0	ND	1.04 µg
4-Nitrophenol	1	0	ND	2.30 µg
Pentachloronitrobenzene	1	0	ND	2.94 µg
Pentachlorophenol	1	0	ND	1.75 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Phenanthrene	1	0	ND	0.28 µg
Phenol	1	0	ND	0.46 µg
Pyrene	1	0	ND	0.32 µg
1,2,4-Trichlorobenzene	1	0	ND	0.67 µg
2,4,5-Trichlorophenol	1	0	ND	1.13 µg
2,4,6-Trichlorophenol	1	0	ND	1.19 µg
PAHs by CARB 429				
Naphthalene	1	1	330 ng	--
2-Methylnaphthalene	1	1	189 ng	--
Acenaphthene	1	1	10.3 ng	--
2-Chloronaphthalene	1	0	ND	0.2 ng
Acenaphthylene	1	1	6.0 ng	--
Fluorene	1	1	30.1 ng	--
Phenanthrene	1	1	43.0 ng	--
Anthracene	1	1	3.3 ng	--
Fluoranthene	1	1	11.8 ng	--
Pyrene	1	1	15.9 ng	--
Benzo(a)anthracene	1	1	0.86 ng	--
Chrysene	1	1	1.8 ng	--
Perylene	1	1	0.26 ng	--
Benzo(b)fluoranthene	1	1	1.6 ng	--
Benzo(k)fluoranthene	1	1	0.50 ng	--
Benzo(a)pyrene	1	1	1.7 ng	--
Benzo(e)pyrene	1	1	5.8 ng	--
Benzo(g,h,i)perylene	1	1	9.9 ng	--
Indeno(1,2,3-cd)pyrene	1	1	1.3 ng	--
Dibenz(a,h)anthracene	1	0	ND	0.1 ng
Trip Blank - Filter & PNR				
Semivoltile Organic Compounds by SW8270				
Acenaphthene	2	0	ND	0.39-1.34 µg
Acenaphthylene	2	0	ND	0.23-0.75 µg
Acetophenone	2	1	0.15 µg	2.27 µg
4-Aminobiphenyl	2	0	ND	0.42-1.10 µg
Aniline	2	0	ND	0.38-1.89 µg
Anthracene	2	0	ND	0.31-0.78 µg
Benz(a)anthracene	2	0	ND	0.49-1.00 µg
Benz(a)pyrene	2	0	ND	0.57-1.48 µg
Benzidine	2	0	ND	0.73-1.55 µg
Benzo(b)fluoranthene	2	0	ND	0.52-1.21 µg
Benzo(g,h,i)perylene	2	0	ND	0.62-1.81 µg
Benzo(k)fluoranthene	2	0	ND	0.55-1.47 µg
Benzoic Acid	2	0	ND	1.30-5.56 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Benzyl Alcohol	2	1	0.80 µg	4.01 µg
4-Bromophenylphenylether	2	0	ND	1.53-3.28 µg
Butylbenzylphthalate	2	0	ND	0.59-1.63 µg
4-Chloro-3-methylphenol	2	0	ND	0.73-2.80 µg
p-Chloroaniline	2	0	ND	0.50-2.17 µg
bis(2-Chloroethoxy)methane	2	0	ND	0.55-2.61 µg
bis(2-Chloroethyl)ether	2	0	ND	0.67-3.78 µg
bis(2-Chloroisopropyl)ether	2	0	ND	1.05-3.01 µg
2-Chloronaphthalene	2	0	ND	0.37-1.11 µg
2-Chlorophenol	2	0	ND	0.60-2.73 µg
4-Chlorophenylphenylether	2	0	ND	0.88-1.95 µg
Chrysene	2	0	ND	0.54-1.16 µg
Di-n-butylphthalate	2	2	2.81-3.81 µg	--
Di-n-octylphthalate	2	0	ND	0.31-0.94 µg
Dibenz(a,h)anthracene	2	0	ND	0.70-1.95 µg
Dibenzofuran	2	0	ND	0.26-0.75 µg
1,2-Dichlorobenzene	2	0	ND	0.61-2.25 µg
1,3-Dichlorobenzene	2	0	ND	0.57-2.30 µg
1,4-Dichlorobenzene	2	0	ND	0.57-2.10 µg
3,3-Dichlorobenzidine	2	0	ND	1.21-2.61 µg
2,4-Dichlorophenol	2	0	ND	0.73-2.61 µg
Diethylphthalate	2	1	0.52 µg	0.87 µg
P-Dimethylaminoazobenzene	2	0	ND	1.04-3.18 µg
2,4-Dimethylphenol	2	0	ND	0.67-2.69 µg
Dimethylphthalate	2	0	ND	0.34-0.98 µg
4,6-Dinitro-2-methylphenol	2	0	ND	2.43-4.99 µg
2,4-Dinitrophenol	2	0	ND	3.03-7.97 µg
2,4-Dinitrotoluene	2	0	ND	1.02-2.73 µg
2,6-Dinitrotoluene	2	0	ND	1.28-3.97 µg
bis(2-Ethylhexyl)phthalate	2	1	0.68 µg	1.37 µg
Fluoranthene	2	0	ND	0.32-0.74 µg
Fluorene	2	0	ND	0.37-1.04 µg
Hexachlorobenzene	2	0	ND	1.13-2.40 µg
Hexachlorobutadiene	2	0	ND	1.34-3.18 µg
Hexachlorocyclopentadiene	2	0	ND	1.18-3.26 µg
Hexachloroethane	2	0	ND	1.16-3.99 µg
Indeno(1,2,3)pyrene	2	0	ND	0.56-1.49 µg
Isophorone	2	0	ND	0.36-1.45 µg
2-Methylnaphthalene	2	0	ND	0.35-1.39 µg
4-Methylphenol/3-Methylphenol	2	0	ND	0.65-2.98 µg
2-Methylphenol	2	0	ND	0.69-3.33 µg
N-Nitrosodipropylamine	2	0	ND	1.19-4.40 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
N-Nitrosodimethylamine	2	0	ND	1.99-7.71 µg
Naphthalene	2	0	ND	0.22-0.94 µg
2-Nitroaniline	2	0	ND	1.37-3.52 µg
3-Nitroaniline	2	0	ND	1.24-4.01 µg
4-Nitroaniline	2	0	ND	1.39-3.88 µg
Nitrobenzene	2	0	ND	0.63-2.53 µg
2-Nitrophenol	2	0	ND	1.06-4.08 µg
4-Nitrophenol	2	0	ND	2.45-4.57 µg
Pentachloronitrobenzene	2	0	ND	3.20-6.43 µg
Pentachlorophenol	2	0	ND	1.90-4.99 µg
Phenanthrene	2	0	ND	0.31-0.76 µg
Phenol	2	1	0.52 µg	2.60 µg
Pyrene	2	0	ND	0.36-0.68 µg
1,2,4-Trichlorobenzene	2	0	ND	0.68-2.13 µg
2,4,5-Trichlorophenol	2	0	ND	1.20-2.55 µg
2,4,6-Trichlorophenol	2	0	ND	1.27-2.69 µg
PAHs by CARB 429				
Naphthalene	2	2	180-218 ng	--
2-Methylnaphthalene	2	2	123-157 ng	--
Acenaphthene	2	2	8.5-27.4 ng	--
2-Chloronaphthalene	2	0	ND	0.05-0.13 ng
Acenaphthalene	2	2	3.8-4.3 ng	--
Fluorene	2	2	30.0-50.6ng	--
Phenanthrene	2	2	69.4-98.5 ng	--
Anthracene	2	2	3.2-4.1 ng	--
Fluoranthene	2	2	12.8-16.7 ng	--
Pyrene	2	2	13.3-14.3 ng	--
Benzo(a)anthracene	2	2	0.76-1.2 ng	--
Chrysene	2	2	2.4-6.3 ng	--
Perylene	2	1	0.18 ng	0.3 ng
Benzo(b)fluoranthene	2	2	1.6-2.9 ng	--
Benzo(k)fluoranthene	2	2	0.44-0.79 ng	--
Benzo(a)pyrene	2	2	0.75-1.1 ng	--
Benzo(e)pyrene	2	2	2.7-3.2 ng	--
Benzo(g,h,i)perylene	2	2	3.6-5.7 ng	--
Indeno(1,2,3-cd)pyrene	2	2	1.4-1.6 ng	--
Dibenz(a,h)anthracene	2	2	0.36 ng	0.3 ng
Field Blank - Filter & PNR				
Semivolatile Compounds by SW8270				
Acenaphthene	2	0	ND	0.43-1.34 µg
Acenaphthylene	2	0	ND	0.25-0.75 µg
Acetophenone	2	0	ND	0.54-2.16 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
4-Aminobiphenyl	2	0	ND	0.46-1.07 µg
Aniline	2	0	ND	0.40-1.80 µg
Anthracene	2	0	ND	0.35-0.77 µg
Benzo(a)anthracene	2	0	ND	0.60-1.00 µg
Benz(a)pyrene	2	0	ND	0.67-1.51 µg
Benzidine	2	0	ND	0.88-1.57 µg
Benzo(b)fluoranthene	2	0	ND	0.61-1.23 µg
Benzo(g,h,i)perylene	2	0	ND	0.72-1.85 µg
Benzo(k)fluoranthene	2	0	ND	0.65-1.50 µg
Benzoic Acid	2	0	ND	1.37-5.48 µg
Benzyl Alcohol	2	1	0.61 µg	3.82 µg
4-Bromophenylphenylether	2	0	ND	1.70-3.22 µg
Butylbenzylphthalate	2	0	ND	0.72-1.65 µg
4-Chloro-3-methylphenol	2	0	ND	0.77-2.76 µg
p-Chloroaniline	2	0	ND	0.52-2.14 µg
bis(2-Chloroethoxy)methane	2	0	ND	0.57-2.58 µg
bis(2-Chloroethyl)ether	2	0	ND	0.71-3.60 µg
bis(2-Chloroisopropyl)ether	2	0	ND	1.10-2.87 µg
2-Chloronaphthalene	2	0	ND	0.40-1.11 µg
2-Chlorophenol	2	0	ND	0.63-2.60 µg
4-Chlorophenylphenylether	2	0	ND	0.96-1.95 µg
Chrysene	2	0	ND	0.65-1.17 µg
Di-n-butylphthalate	2	2	3.64-7.53 µg	--
Di-n-octylphthalate	2	0	ND	0.36-0.96 µg
Dibenz(a,h)anthracene	2	0	ND	0.82-1.99 µg
Dibenzofuran	2	0	ND	0.29-0.75 µg
1,2-Dichlorobenzene	2	0	ND	0.63-2.15 µg
1,3-Dichlorobenzene	2	0	ND	0.60-2.19 µg
1,4-Dichlorobenzene	2	1	0.97 µg	2.00 µg
3,3-Dichlorobenzidine	2	0	ND	1.47-2.63 µg
2,4-Dichlorophenol	2	0	ND	0.76-2.58 µg
Diethylphthalate	2	1	0.57 µg	0.87 µg
P-Dimethylaminoazobenzene	2	0	ND	1.26-3.20 µg
2,4-Dimethylphenol	2	0	ND	0.70-2.65 µg
Dimethylphthalate	2	0	ND	0.37-0.98 µg
4,6-Dinitro-2-methylphenol	2	0	ND	2.71-4.89 µg
2,4-Dinitrophenol	2	0	ND	3.30-7.97 µg
2,4-Dinitrotoluene	2	0	ND	1.11-2.72 µg
2,6-Dinitrotoluene	2	0	ND	1.40-3.97 µg
bis(2-Ethylhexyl)phthalate	2	2	4.04-4.12 µg	--
Fluoranthene	2	0	ND	0.36-0.73 µg
Fluorene	2	0	ND	0.40-1.04 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Hexachlorobenzene	2	0	ND	1.26-2.35 µg
Hexachlorobutadiene	2	0	ND	1.41-3.14 µg
Hexachlorocyclopentadiene	2	0	ND	1.28-3.26 µg
Hexachloroethane	2	0	ND	1.21-3.80 µg
Indeno(1,2,3-cd)pyrene	2	0	ND	0.66-1.52 µg
Isophorone	2	0	ND	0.38-1.43 µg
2-Methylnaphthalene	2	0	ND	0.36-1.37 µg
4-Methylphenol/3-Methylphenol	2	0	ND	0.68-2.85 µg
2-Methylphenol	2	0	ND	0.72-3.18 µg
N-Nitrosodinpropylamine	2	0	ND	1.25-4.20 µg
N-Nitrosodimethylamine	2	0	ND	2.08-7.36 µg
Naphthalene	2	0	ND	0.23-0.93 µg
2-Nitroaniline	2	0	ND	1.49-3.52 µg
3-Nitroaniline	2	0	ND	1.35-4.01 µg
4-Nitroaniline	2	0	ND	1.51-3.88 µg
Nitrobenzene	2	0	ND	0.67-2.49 µg
2-Nitrophenol	2	0	ND	1.12-4.03 µg
4-Nitrophenol	2	0	ND	2.67-4.56 µg
Pentachloronitrobenzene	2	0	ND	3.56-6.31 µg
Pentachlorophenol	2	0	ND	2.12-4.89 µg
Phenanthrene	2	0	ND	0.34-0.74 µg
Phenol	2	1	0.46 µg	2.48 µg
Pyrene	2	0	ND	0.43-0.68 µg
1,2,4-Trichlorobenzene	2	0	ND	0.71-2.10 µg
2,4,5-Trichlorophenol	2	0	ND	1.31-2.55 µg
2,4,6-Trichlorophenol	2	0	ND	1.38-2.69 µg
PAHs by CARB 429				
Naphthalene	2	2	129-221 ng	--
2-Methylnaphthalene	2	2	105-149 ng	--
Acenaphthene	2	2	26.7-29.2 ng	--
2-chloronaphthalene	2	1	0.11 ng	0.2 ng
Acenaphthalene	2	2	3.7-4.7 ng	--
Fluorene	2	2	35.1-46.7 ng	--
Phenanthrene	2	2	93.7-99.6 ng	--
Anthracene	2	2	2.9-3.4 ng	--
Fluoranthene	2	2	16.0-18.9 ng	--
Pyrene	2	2	14.8-15.8 ng	--
Benzo(a)anthracene	2	2	0.70-0.78 ng	--
Chrysene	2	2	1.6-1.9 ng	--
Perylene	2	2	0.27-0.34 ng	--
Benzo(b)fluoranthene	2	2	1.7-1.8 ng	--
Benzo(k)fluoranthene	2	2	0.37-0.52 ng	--

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Benzo(a)pyrene	2	2	0.72-0.90 ng	--
Benzo(e)pyrene	2	2	2.7-3.3 ng	--
Benzo(g,h,i)perylene	2	2	5.4-5.8 ng	--
Indeno(1,2,3-cd)pyrene	2	2	1.4-1.5 ng	--
Dibenz(a,h)anthracene	2	0	ND	0.1 ng
Laboratory Method Blank - XAD & Condensate				
Semivolatile Organic Compounds by SW8270				
Acenaphthene	1	0	ND	1.59 µg
Acenaphthylene	1	0	ND	0.86 µg
Acetophenone	1	0	ND	2.20 µg
4-Aminobiphenyl	1	0	ND	1.27 µg
Aniline	1	0	ND	1.90 µg
Anthracene	1	0	ND	0.90 µg
Benz(a)anthracene	1	0	ND	1.37 µg
Benz(a)pyrene	1	0	ND	1.74 µg
Benzidine	1	0	ND	2.21 µg
Benzo(b)fluoranthene	1	0	ND	1.53 µg
Benzo(g,h,i)perylene	1	0	ND	2.02 µg
Benzo(k)fluoranthene	1	0	ND	1.65 µg
Benzoic Acid	1	1	34.9µg	--
Benzyl Alcohol	1	0	ND	4.05 µg
4-Bromophenylphenylether	1	0	ND	3.90 µg
Butylbenzylphthalate	1	0	ND	2.15 µg
4-Chloro-3-methylphenol	1	0	ND	3.06 µg
p-Chloroaniline	1	0	ND	2.34 µg
bis(2-Chloroethoxy)methane	1	0	ND	2.89 µg
bis(2-Chloroethyl)ether	1	0	ND	3.89 µg
bis(2-Chloroisopropyl)ether	1	0	ND	3.20 µg
2-Chloronaphthalene	1	0	ND	1.28 µg
2-Chlorophenol	1	0	ND	2.82 µg
4-Chlorophenylphenylether	1	0	ND	2.36 µg
Chrysene	1	1	0.97 µg	--
Di-n-butylphthalate	1	1	5.83 µg	--
Di-n-octylphthalate	1	0	ND	1.07 µg
Dibenz(a,h)anthracene	1	0	ND	2.33 µg
Dibenzofuran	1	0	ND	0.87 µg
1,2-Dichlorobenzene	1	0	ND	2.41 µg
1,3-Dichlorobenzene	1	0	ND	2.32 µg
1,4-Dichlorobenzene	1	0	ND	2.26 µg
3,3-Dichlorobenzidine	1	0	ND	3.63 µg
2,4-Dichlorophenol	1	0	ND	2.93 µg
Diethylphthalate	1	0	ND	1.04 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
P-Dimethylaminoazobenzene	1	0	ND	4.43 µg
2,4-Dimethylphenol	1	0	ND	2.91 µg
Dimethylphthalate	1	0	ND	1.17 µg
4,6-Dinitro-2-methylphenol	1	0	ND	5.54 µg
2,4-Dinitrophenol	1	0	ND	8.24 µg
2,4-Dinitrotoluene	1	0	ND	3.39 µg
2,6-Dinitrotoluene	1	0	ND	4.57 µg
bis(2-Ethylhexyl)phthalate	1	1	1.93 µg	--
Fluoranthene	1	0	ND	0.88 µg
Fluorene	1	0	ND	1.23 µg
Hexachlorobenzene	1	0	ND	2.89 µg
Hexachlorobutadiene	1	0	ND	3.67 µg
Hexachlorocyclopentadiene	1	0	ND	3.67 µg
Hexachloroethane	1	0	ND	4.08 µg
Indeno(1,2,3)pyrene	1	0	ND	1.75 µg
Isophorone	1	0	ND	1.68 µg
2-Methylnaphthalene	1	0	ND	1.51 µg
4-Methylphenol/3-Methylphenol	1	0	ND	3.06 µg
2-Methylphenol	1	0	ND	3.20 µg
N-Nitrosodipropylamine	1	0	ND	4.64 µg
N-Nitrosodimethylamine	1	0	ND	8.27 µg
Naphthalene	1	0	ND	1.04 µg
2-Nitroaniline	1	0	ND	4.13 µg
3-Nitroaniline	1	0	ND	4.73 µg
4-Nitroaniline	1	0	ND	4.89 µg
Nitrobenzene	1	0	ND	2.77 µg
2-Nitrophenol	1	0	ND	4.47 µg
4-Nitrophenol	1	0	ND	5.73 µg
Pentachloronitrobenzene	1	0	ND	7.45 µg
Pentachlorophenol	1	0	ND	4.76 µg
Phenanthrene	1	0	ND	0.87 µg
Phenol	1	0	ND	2.86 µg
Pyrene	1	0	ND	0.91 µg
1,2,4-Trichlorobenzene	1	0	ND	2.44 µg
2,4,5-Trichlorophenol	1	0	ND	3.02 µg
2,4,6-Trichlorophenol	1	0	ND	3.12 µg
PAHs by CARB 429				
Naphthalene	1	1	76.6 ng	--
2-Methylnaphthalene	1	1	164 ng	--
Acenaphthene	1	1	32.4 ng	--
2-Chloronaphthalene	1	0	ND	0.2 ng
Acenaphthylene	1	1	10.5 ng	--

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Fluorene	1	1	39.6 ng	--
Phenanthrene	1	1	150 ng	--
Anthracene	1	1	5.0 ng	--
Fluoranthene	1	1	25.4 ng	--
Pyrene	1	1	26.0 ng	--
Benzo(a)anthracene	1	1	1.5 ng	--
Chrysene	1	1	2.7 ng	--
Perylene	1	0	ND	0.2 ng
Benzo(b)fluoranthene	1	1	2.6 ng	--
Benzo(k)fluoranthene	1	1	0.61 ng	--
Benzo(a)pyrene	1	1	1.5 ng	--
Benzo(e)pyrene	1	1	3.6 ng	--
Benzo(g,h,i)perylene	1	1	9.7 ng	--
Indeno(1,2,3-cd)pyrene	1	1	2.9 ng	--
Dibenz(a,h)anthracene	1	0	ND	0.3 ng
Trip Blank - XAD & Condensate				
Semivolatile Organic Compounds by SW8270				
Acenaphthene	2	0	ND	1.56-1.70 µg
Acenaphthylene	2	0	ND	0.84-0.92 µg
Acetophenone	2	1	0.62 µg	2.38 µg
4-Aminobiphenyl	2	0	ND	1.30-1.43 µg
Aniline	2	0	ND	1.95-2.06 µg
Anthracene	2	0	ND	0.92-1.01 µg
Benz(a)anthracene	2	0	ND	1.18-1.38 µg
Benz(a)pyrene	2	0	ND	1.64-1.77 µg
Benzidine	2	0	ND	1.91-2.23 µg
Benzo(b)fluoranthene	2	0	ND	1.45-1.56 µg
Benzo(g,h,i)perylene	2	0	ND	1.91-2.05 µg
Benzo(k)fluoranthene	2	0	ND	1.56-1.68 µg
Benzoic Acid	2	2	26.55-47.52 µg	--
Benzyl Alcohol	2	0	ND	4.15-4.38 µg
4-Bromophenylphenylether	2	0	ND	3.98-4.39 µg
Butylbenzylphthalate	2	0	ND	1.86-2.17 µg
4-Chloro-3-methylphenol	2	0	ND	2.98-3.29 µg
p-Chloroaniline	2	0	ND	2.28-2.52 µg
bis(2-Chloroethoxy)methane	2	0	ND	2.82-3.11 µg
bis(2-Chloroethyl)ether	2	0	ND	3.99-4.21 µg
bis(2-Chloroisopropyl)ether	2	0	ND	3.27-3.46 µg
2-Chloronaphthalene	2	0	ND	1.26-1.37 µg
2-Chlorophenol	2	0	ND	2.89-3.05 µg
4-Chlorophenylphenylether	2	0	ND	2.31-2.52 µg
Chrysene	2	0	ND	1.34-1.57 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Di-n-butylphthalate	2	2	65.25-85.44 µg	--
Di-n-octylphthalate	2	0	ND	1.01-1.09 µg
Dibenz(a,h)anthracene	2	0	ND	2.20-2.37 µg
Dibenzofuran	2	0	ND	0.86-0.94 µg
1,2-Dichlorobenzene	2	0	ND	2.47-2.61 µg
1,3-Dichlorobenzene	2	0	ND	2.37-2.50 µg
1,4-Dichlorobenzene	2	0	ND	2.31-2.44 µg
3,3-Dichlorobenzidine	2	0	ND	3.13-3.66 µg
2,4-Dichlorophenol	2	0	ND	2.86-3.16 µg
Diethylphthalate	2	2	1.55 µg	1.12 µg
P-Dimethylaminoazobenzene	2	0	ND	3.82-4.47 µg
2,4-Dimethylphenol	2	0	ND	2.84-3.13 µg
Dimethylphthalate	2	0	ND	1.14-1.25 µg
4,6-Dinitro-2-methylphenol	2	0	ND	5.65-6.24 µg
2,4-Dinitrophenol	2	0	ND	8.09-8.82 µg
2,4-Dinitrotoluene	2	0	ND	3.33-3.63 µg
2,6-Dinitrotoluene	2	0	ND	4.48-4.89 µg
bis(2-Ethylhexyl)phthalate	2	2	3.68-5.41 µg	--
Fluoranthene	2	0	ND	0.90-0.99 µg
Fluorene	2	0	ND	1.21-1.32 µg
Hexachlorobenzene	2	0	ND	2.94-3.25 µg
Hexachlorobutadiene	2	0	ND	3.58-3.94 µg
Hexachlorocyclopentadiene	2	0	ND	3.60-3.92 µg
Hexachloroethane	2	0	ND	4.18-4.41 µg
Indeno(1,2,3)pyrene	2	0	ND	1.65-1.78 µg
Isophorone	2	0	ND	1.64-1.81 µg
2-Methylnaphthalene	2	1	0.21 µg	1.62 µg
4-Methylphenol/3-Methylphenol	2	0	ND	3.14-3.31 µg
2-Methylphenol	2	0	ND	3.28-3.46 µg
N-Nitrosodipropylamine	2	0	ND	4.75-5.02 µg
N-Nitrosodimethylamine	2	0	ND	8.47-8.94 µg
Naphthalene	2	1	0.99 µg	1.11 µg
2-Nitroaniline	2	0	ND	4.06-4.42 µg
3-Nitroaniline	2	0	ND	4.64-5.06 µg
4-Nitroaniline	2	0	ND	4.80-5.24 µg
Nitrobenzene	2	0	ND	2.70-2.98 µg
2-Nitrophenol	2	0	ND	4.36-4.81 µg
4-Nitrophenol	2	0	ND	5.63-6.14 µg
Pentachloronitrobenzene	2	0	ND	7.59-8.39 µg
Pentachlorophenol	2	0	ND	4.85-5.36 µg
Phenanthrene	2	0	ND	0.89-0.98 µg
Phenol	2	1	1.07 µg	3.09 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Pyrene	2	0	ND	0.78-0.92 µg
1,2,4-Trichlorobenzene	2	0	ND	2.38-2.63 µg
2,4,5-Trichlorophenol	2	0	ND	2.96-3.23 µg
2,4,6-Trichlorophenol	2	0	ND	3.06-3.33 µg
PAHs by CARB 429				
Naphthalene	2	2	418-684 ng	--
2-Methylnaphthalene	2	2	144-225 ng	--
Acenaphthene	2	2	23.5-34.3 ng	--
2-Chloronaphthalene	2	0	ND	0.16-0.3 ng
Acenaphthalene	2	2	12.9-22.0 ng	--
Fluorene	2	2	29.5-53.5 ng	--
Phenanthrene	2	2	108-187 ng	--
Anthracene	2	2	4.4-6.6 ng	--
Fluoranthene	2	2	22.6-40.2 ng	--
Pyrene	2	2	18.0-32.8 ng	--
Benzo(a)anthracene	2	2	2.0-2.6 ng	--
Chrysene	2	2	3.8-4.8 ng	--
Perylene	2	2	0.43-0.72 ng	--
Benzo(b)fluoranthene	2	2	3.4-4.8 ng	--
Benzo(k)fluoranthene	2	2	0.99-2.3 ng	--
Benzo(a)pyrene	2	2	1.2-1.9 ng	--
Benzo(e)pyrene	2	2	3.1-3.5 ng	--
Benzo(g,h,i)perylene	2	2	4.6-6.8 ng	--
Indeno(2,2,3-cd)pyrene	2	2	1.9-2.5 ng	--
Dibenz(a,h)anthracene	2	0	ND	0.2-0.3 ng
Field Blank - XAD & Condensate				
Semivolatile Organic Compounds by SW8270				
Acenaphthene	2	0	ND	1.73-1.79 µg
Acenaphthylene	2	0	ND	0.93-0.96 µg
Acetophenone	2	0	ND	2.45-2.51 µg
4-Aminobiphenyl	2	0	ND	1.43-1.44 µg
Aniline	2	0	ND	2.12-2.17 µg
Anthracene	2	0	ND	1.01-1.02 µg
Benz(a)anthracene	2	0	ND	1.35-1.41 µg
Benz(a)pyrene	2	0	ND	1.72-1.83 µg
Benzidine	2	0	ND	2.18-2.27 µg
Benzo(b)fluoranthene	2	0	ND	1.52-1.61 µg
Benzo(g,h,i)perylene	2	0	ND	2.00-2.13
Benzo(k)fluoranthene	2	0	ND	1.64-1.74 µg
Benzoic Acid	2	2	20.3-21.4 µg	--
Benzyl Alcohol	2	1	0.76 µg	4.52 µg
4-Bromophenylphenylether	2	0	ND	4.39-4.44 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Butylbenzylphthalate	2	0	ND	2.13-2.22 µg
4-Chloro-3-methylphenol	2	0	ND	3.25-3.36 µg
p-Chloroaniline	2	0	ND	2.48-2.57 µg
bis(2-Chloroethoxy)methane	2	0	ND	3.07-3.18 µg
bis(2-Chloroethyl)ether	2	0	ND	4.34-4.44 µg
bis(2-Chloroisopropyl)ether	2	0	ND	3.57-3.65 µg
2-Chloronaphthalene	2	0	ND	1.40-1.44 µg
2-Chlorophenol	2	0	ND	3.15-3.22 µg
4-Chlorophenylphenylether	2	0	ND	2.57-2.66 µg
Chrysene	2	0	ND	1.54-1.60 µg
Di-n-butylphthalate	2	2	25.2-26.0 µg	--
Di-n-octylphthalate	2	0	ND	1.06-1.13 µg
Dibenz(a,h)anthracene	2	0	ND	2.31-2.45 µg
Dibenzofuran	2	0	ND	0.95-0.98 µg
1,2-Dichlorobenzene	2	0	ND	2.69-2.75 µg
1,3-Dichlorobenzene	2	0	ND	2.58-2.64 µg
1,4-Dichlorobenzene	2	0	ND	2.52-2.58 µg
3,3-Dichlorobenzidine	2	0	ND	3.58-3.74 µg
2,4-Dichlorophenol	2	0	ND	3.12-3.22 µg
Diethylphthalate	2	1	1.03 µg	1.14 µg
P-Dimethylaminoazobenzene	2	0	ND	4.37-4.56 µg
2,4-Dimethylphenol	2	0	ND	3.09-3.20 µg
Dimethylphthalate	2	0	ND	1.27-1.31 µg
4,6-Dinitro-2-methylphenol	2	0	ND	6.24-6.31 µg
2,4-Dinitrophenol	2	0	ND	9.00-9.28 µg
2,4-Dinitrotoluene	2	0	ND	3.70-3.82 µg
2,6-Dinitrotoluene	2	0	ND	4.99-5.14 µg
bis(2-Ethylhexyl)phthalate	2	2	3.88-4.19 µg	--
Fluoranthene	2	0	ND	0.99-1.00 µg
Fluorene	2	0	ND	1.35-1.39 µg
Hexachlorobenzene	2	0	ND	3.25-3.28 µg
Hexachlorobutadiene	2	0	ND	3.89-4.03 µg
Hexachlorocyclopentadiene	2	0	ND	4.00-4.13 µg
Hexachloroethane	2	0	ND	4.55-4.66 µg
Indeno(1,2,3)pyrene	2	0	ND	1.73-1.84 µg
Isophorone	2	0	ND	1.78-1.84 µg
2-Methylnaphthalene	2	0	ND	1.60-1.66 µg
4-Methylphenol/3-Methylphenol	2	0	ND	3.42-3.49 µg
2-Methylphenol	2	0	ND	3.57-3.65 µg
N-Nitrosodipropylamine	2	0	ND	5.18-5.30 µg
N-Nitrosodimethylamine	2	0	ND	9.23-9.44 µg
Naphthalene	2	1	0.86 µg	1.10 µg

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
2-Nitroaniline	2	0	ND	4.51-4.65 µg
3-Nitroaniline	2	0	ND	5.17-5.33 µg
4-Nitroaniline	2	0	ND	5.34-5.51 µg
Nitrobenzene	2	0	ND	2.94-3.04 µg
2-Nitrophenol	2	0	ND	4.75-4.91 µg
4-Nitrophenol	2	0	ND	6.26-6.46 µg
Pentachloronitrobenzene	2	0	ND	8.39-8.47 µg
Pentachlorophenol	2	0	ND	5.36-5.41 µg
Phenanthrene	2	0	ND	0.98-0.99 µg
Phenol	2	1	0.89 µg	3.26 µg
Pyrene	2	0	ND	0.90-0.93 µg
1,2,4-Trichlorobenzene	2	0	ND	2.59-2.68 µg
2,4,5-Trichlorophenol	2	0	ND	3.30-3.40 µg
2,4,6-Trichlorophenol	2	0	ND	3.40-3.51 µg
PAHs by CARB 429				
Naphthalene	2	2	382-599 ng	--
2-Methylnaphthalene	2	2	140-234 ng	--
Acenaphthene	2	2	20.7-36.4 ng	--
2-Chloronaphthalene	2	1	0.09 ng	0.3 ng
Acenaphthalene	2	2	12.8-19.0 ng	--
Fluorene	2	2	34.6-49.3 ng	--
Phenanthrene	2	2	105-160 ng	--
Anthracene	2	2	4.3-6.0 ng	--
Fluoranthene	2	2	26.9-38.4 ng	--
Pyrene	2	2	17.8-30.9 ng	--
Benzo(a)anthracene	2	2	1.8-2.4 ng	--
Chrysene	2	2	3.1-4.0 ng	--
Perylene	2	1	0.65 ng	0.3 ng
Benzo(b)fluoranthene	2	2	3.8-5.6 ng	--
Benzo(k)fluoranthene	2	2	1.0-1.6 ng	--
Benzo(a)pyrene	2	2	1.5-1.7 ng	--
Benzo(e)pyrene	2	2	3.6-4.2 ng	--
Benzo(g,h,i)perylene	2	2	5.9-9.1 ng	--
Indeno(1,2,3-cd)pyrene	2	2	2.0-3.8 ng	--
Dibenz(a,h)anthracene	2	1	0.49 ng	0.4 ng
Laboratory Method Blank - Semivolatile Compounds in Aqueous Samples				
Acenaphthene	2	0	ND	0.604-0.669 µg/L
Acenaphthylene	2	0	ND	0.456-0.616 µg/L
Acetophenone	2	0	ND	0.539-0.594 µg/L
4-Aminobiphenyl	2	0	ND	3.81-4.09 µg/L
Aniline	2	0	ND	0.682-1.02 µg/L
Anthracene	2	0	ND	0.460-0.664 µg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Benz(a)anthracene	2	0	ND	0.511-0.728 µg/L
Benz(a)pyrene	2	0	ND	0.661-0.682 µg/L
Benzidine	2	0	ND	20.0 µg/L
Benzo(b)fluoranthene	2	0	ND	0.649-0.768 µg/L
Benzo(g,h,i)perylene	2	0	ND	0.684-0.702 µg/L
Benzo(k)fluoranthene	2	0	ND	0.945-1.11 µg/L
Benzoic Acid	2	0	ND	3.11-6.03 µg/L
Benzyl Alcohol	2	0	ND	0.428-0.698 µg/L
4-Bromophenylphenylether	2	0	ND	0.288-0.752 µg/L
Butylbenzylphthalate	2	0	ND	0.474-0.896 µg/L
4-Chloro-3-methylphenol	2	0	ND	0.380-0.625 µg/L
p-Chloroaniline	2	0	ND	0.898-1.01 µg/L
bis(2-Chloroethoxy)methane	2	0	ND	0.546-0.673 µg/L
bis(2-Chloroethyl)ether	2	0	ND	0.595-0.670 µg/L
bis(2-Chloroisopropyl)ether	2	0	ND	0.555-1.11 µg/L
2-Chloronaphthalene	2	0	ND	0.797-0.962 µg/L
2-Chlorophenol	2	0	ND	0.537-0.637 µg/L
4-Chlorophenylphenylether	2	0	ND	0.451-0.898 µg/L
Chrysene	2	0	ND	0.618-0.737 µg/L
Di-n-butylphthalate	2	0	ND	0.343-0.475 µg/L
Di-n-octylphthalate	2	0	ND	0.646-0.673 µg/L
Dibenz(a,h)anthracene	2	0	ND	0.729-0.810 µg/L
Dibenzofuran	2	0	ND	0.535-0.608 µg/L
1,2-Dichlorobenzene	2	0	ND	0.604-0.704 µg/L
1,3-Dichlorobenzene	2	0	ND	0.405-0.760 µg/L
1,4-Dichlorobenzene	2	0	ND	1.04-1.59 µg/L
3,3-Dichlorobenzidine	2	0	ND	0.716-3.70 µg/L
2,4-Dichlorophenol	2	0	ND	0.404-0.701 µg/L
Diethylphthalate	2	0	ND	0.297-0.649 µg/L
P-Dimethylaminoazobenzene	2	0	ND	0.485-0.754 µg/L
2,4-Dimethylphenol	2	0	ND	0.65-0.658 µg/L
Dimethylphthalate	2	0	ND	0.405-0.444 µg/L
4,6-Dinitro-2-methylphenol	2	0	ND	0.457-2.89 µg/L
2,4-Dinitrophenol	2	0	ND	1.21-1.91 µg/L
2,4-Dinitrotoluene	2	0	ND	0.317-0.777 µg/L
2,6-Dinitrotoluene	2	0	ND	0.618-0.752 µg/L
Diphenylamine/N-NitrosoDPA	2	0	ND	0.649-0.658 µg/L
bis(2-Ethylhexyl)phthalate	2	0	ND	0.840-0.963 µg/L
Fluoranthene	2	0	ND	0.672-0.686 µg/L
Fluorene	2	0	ND	0.635-0.710 µg/L
Hexachlorobenzene	2	0	ND	0.537-1.51 µg/L
Hexachlorobutadiene	2	0	ND	0.714-0.983 µg/L

Table A-2 (Continued)

Analyte	Number of Blanks Analyzed	Number of Detects	Range of Compounds Detected	Detection Limit
Hexachlorocyclopentadiene	2	0	ND	0.850-1.98 µg/L
Hexachloroethane	2	0	ND	1.79-5.56 µg/L
Indeno(1,2,3)pyrene	2	0	ND	0.534-0.763 µg/L
Isophorone	2	0	ND	0.340-0.548 µg/L
2-Methylnaphthalene	2	0	ND	0.811-1.17 µg/L
4-Methylphenol/3-Methylphenol	2	0	ND	0.442-0.859 µg/L
2-Methylphenol	2	0	ND	0.477-0.575 µg/L
N-Nitrosodipropylamine	2	0	ND	0.567-0.804 µg/L
N-Nitrosodimethylamine	2	0	ND	0.506-0.832 µg/L
Naphthalene	2	1	1.78 µg/L	0.719-0.828 µg/L
2-Nitroaniline	2	0	ND	0.515-0.748 µg/L
3-Nitroaniline	2	0	ND	0.511-0.894 µg/L
4-Nitroaniline	2	0	ND	0.575-0.621 µg/L
Nitrobenzene	2	0	ND	0.544-0.841 µg/L
2-Nitrophenol	2	0	ND	0.773-1.08 µg/L
4-Nitrophenol	2	0	ND	0.761-1.15 µg/L
Pentachloronitrobenzene	2	0	ND	1.32-1.78 µg/L
Pentachlorophenol	2	0	ND	0.486-0.648 µg/L
Phenanthrene	2	0	ND	0.617-0.634 µg/L
Phenol	2	0	ND	0.429-0.707 µg/l
Pyrene	2	0	ND	0.798-0.814 µg/L
1,2,4-Trichlorobenzene	2	0	ND	0.498-0.645 µg/L
2,4,5-Trichlorophenol	2	0	ND	0.323-0.476 µg/L
2,4,6-Trichlorophenol	2	0	ND	0.385-0.450 µg/L

Table A-3
Matrix Spike/Duplicate Sample Results

Measurement Parameter	How Measured	Objectives		Measurement 1			Measurement 2		
		Precision	Bias	% Recovery		Precision	% Recovery		Precision
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)	Sample	Duplicate	(% RPD)
Metals in Gas Particulate Phase - ICP-AES									
Filter & PNR: Incinerator Stack	Precision - Matrix-spiked Duplicate								
	Accuracy - Matrix-spiked Sample								
Aluminum		20	75-125	90	89	1.1			
Antimony		20	75-125	80	76	5.1			
Barium		20	75-125	98	96	2.1			
Beryllium		20	75-125	97	96	1.0			
Calcium		20	75-125	93	93	0			
Chromium		20	75-125	92	92	0			
Cobalt		20	75-125	89	88	1.1			
Copper		20	75-125	94	93	1.1			
Iron		20	75-125	93	93	0			
Magnesium		20	75-125	86	85	1.2			
Manganese		20	75-125	91	91	0			
Molybdenum		20	75-125	93	92	1.1			
Nickel		20	75-125	95	89	6.5			
Phosphorus		20	75-125	97	90	7.5			
Potassium		20	75-125	91	89	2.2			
Sodium		20	75-125	92	90	2.2			
Titanium		20	75-125	93	93	0			
Vanadium		20	75-125	93	91	2.2			
Zinc		20	75-125	85	84	1.2			
Filter & PNR: Turbine Stack	Precision - Matrix-spiked Duplicate								
	Accuracy - Matrix-spiked Sample								
Aluminum		20	75-125	91	91	0			
Antimony		20	75-125	88	94	6.6			
Barium		20	75-125	98	98	0			
Beryllium		20	75-125	93	93	0			
Calcium		20	75-125	95	95	0			

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Chromium		20	75-125	94	94	0					
Cobalt		20	75-125	91	89	2.2					
Copper		20	75-125	95	95	0					
Iron		20	75-125	94	94	0					
Magnesium		20	75-125	88	88	0					
Manganese		20	75-125	93	93	0					
Molybdenum		20	75-125	92	92	0					
Nickel		20	75-125	93	91	2.2					
Phosphorus		20	75-125	95	98	3.1					
Potassium		20	75-125	91	93	2.2					
Sodium		20	75-125	94	95	1.1					
Titanium		20	75-125	93	94	1.1					
Vanadium		20	75-125	94	94	0					
Zinc		20	75-125	88	87	1.1					
Metals in Gas Particulate Phase - GFAAS and CVAAS											
Filter & PNR: Incinerator Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Arsenic		20	75-125	100	97	3.0					
Cadmium		20	75-125	74 Q	85	14					
Lead		20	75-125	59 Q	62 Q	5.0					
Mercury (CVAAS)		20	75-125	112	111	0					
Selenium		20	75-125	18 Q	18 Q	0					
Filter & PNR: Turbine Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Arsenic		20	75-125	104	104	0					
Cadmium		20	75-125	86	87	1.2					
Lead		20	75-125	81	78	3.8					
Mercury (CVAAS)		20	75-125	110	111	0.9					
Selenium		20	75-125	83	85	2.4					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Metals in Gas Vapor Phase - ICP/MS											
HNO_3/H_2O_2 Impingers: Turbine Stack	Precision - NA										
	Accuracy - Matrix-spiked Sample										
Antimony		20	75-125	92							
Arsenic		20	75-125	105							
Barium		20	75-125	110							
Beryllium		20	75-125	112							
Cadmium		20	75-125	94							
Chromium		20	75-125	94							
Cobalt		20	75-125	87							
Copper		20	75-125	99							
Lead		20	75-125	107							
Manganese		20	75-125	95							
Mercury		20	75-125	56	Q						
Molybdenum		20	75-125	95							
Nickel		20	75-125	100							
Selenium		20	75-125	102							
Vanadium		20	75-125	101							
10% HNO_3/30% H_2O_2 Impingers: Raw Syngas	Precision - NA										
	Accuracy - Matrix-spiked Sample										
Antimony		20	75-125	92							
Arsenic		20	75-125	107							
Barium		20	75-125	98							
Beryllium		20	75-125	110							
Cadmium		20	75-125	95							
Chromium		20	75-125	99							
Cobalt		20	75-125	94							
Copper		20	75-125	101							
Lead		20	75-125	108							
Manganese		20	75-125	102							

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Mercury		20	75-125	149 Q							
Molybdenum		20	75-125	91							
Nickel		20	75-125	96							
Selenium		20	75-125	124							
Vanadium		20	75-125	129 Q							
10% HNO_3/30% H_2O_2 Impingers: Tail Gas	Precision - NA										
	Accuracy - Matrix-spiked Sample										
Antimony		20	75-125	79							
Arsenic		20	75-125	92							
Barium		20	75-125	95							
Beryllium		20	75-125	89							
Cadmium		20	75-125	87							
Chromium		20	75-125	59 Q							
Cobalt		20	75-125	73 Q							
Copper		20	75-125	77							
Lead		20	75-125	96							
Manganese		20	75-125	75							
Mercury		20	75-125	162 Q							
Molybdenum		20	75-125	81							
Nickel		20	75-125	70 Q							
Selenium		20	75-125	90							
Vanadium		20	75-125	81							
Metals in Gas Vapor Phase - ICP-AES											
HNO_3/H_2O_2 Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Aluminum		20	75-125	91	90	1.1					
Antimony		20	75-125	86	83	3.6					
Barium		20	75-125	91	91	0					
Beryllium		20	75-125	93	92	1.1					
Boron		20	74-125	96	95	1.0					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1			Measurement 2		
		Precision	Bias	% Recovery		Precision	% Recovery		Precision
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)	Sample	Duplicate	(% RPD)
Calcium		20	75-125	93	92	1.1			
Chromium		20	75-125	91	90	1.1			
Cobalt		20	75-125	88	88	0			
Copper		20	75-125	91	90	1.1			
Iron		20	75-125	93	91	2.2			
Magnesium		20	75-125	90	90	0			
Manganese		20	75-125	90	89	1.1			
Molybdenum		20	75-125	91	90	1.1			
Nickel		20	75-125	92	89	3.3			
Phosphorus		20	75-125	89	90	1.1			
Potassium		20	75-125	93	92	1.1			
Silicon		20	75-125	100	95	5.1			
Sodium		20	75-125	90	89	1.1			
Titanium		20	75-125	90	89	1.1			
Vanadium		20	75-125	92	90	2.2			
Zinc		20	75-125	90	89	1.1			
10% HNO_3/30% H_2O_2 Impingers: Raw Syngas	Precision - Matrix-spiked Duplicate								
	Accuracy - Matrix-spiked Sample								
Aluminum		20	75-125	91	89	2.2			
Antimony		20	75-125	85	92	7.9			
Barium		20	75-125	92	90	2.2			
Beryllium		20	75-125	94	92	2.2			
Boron		20	75-125	95	92	3.2			
Calcium		20	75-125	93	91	2.2			
Chromium		20	75-125	91	90	1.1			
Cobalt		20	75-125	89	89	0			
Copper		20	75-125	91	90	1.1			
Iron		20	75-125	92	90	2.2			
Magnesium		20	75-125	92	89	3.3			
Manganese		20	75-125	91	90	1.1			

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Molybdenum		20	75-125	92	91	1.1					
Nickel		20	75-125	91	90	1.1					
Phosphorus		20	75-125	91	87	4.5					
Potassium		20	75-125	92	90	2.2					
Silicon		20	75-125	97	96	1.0					
Sodium		20	75-125	89	86	3.4					
Titanium		20	75-125	90	89	1.1					
Vanadium		20	75-125	91	90	1.1					
Zinc		20	75-125	89	89	0					
10% HNO_3/30% H_2O_2 Impingers:	Precision - Matrix-spiked Duplicate										
Tail Gas	Accuracy - Matrix-spiked Sample										
Aluminum		20	75-125	91	92	1.1		88	87	1.1	
Antimony		20	75-125	98	94	4.2		86	87	1.2	
Barium		20	75-125	60 Q	55 Q	8.7		64 Q	58 Q	9.8	
Beryllium		20	75-125	101	102	0.98		95	95	0	
Boron		20	75-125	93	91	2.2		89	92	3.3	
Calcium		20	75-125	95	96	1		90	90	0	
Chromium		20	75-125	91	91	0		89	89	0	
Cobalt		20	75-125	91	91	0		87	88	1.1	
Copper		20	75-125	90	91	1.1		90	88	2.2	
Iron		20	75-125	94	94	0		90	89	1.1	
Magnesium		20	75-125	90	90	0		86	86	0	
Manganese		20	75-125	91	92	1.1		89	88	1.1	
Molybdenum		20	75-125	92	91	1.1		90	90	0	
Nickel		20	75-125	89	95	6.5		90	91	1.1	
Phosphorus		20	75-125	95	101	6.1		95	96	1.0	
Potassium		20	75-125	92	93	1.1		87	86	1.2	
Silicon		20	75-125	94	94	0		92	91	1.1	
Sodium		20	75-125	90	91	1.1		85	86	1.2	
Titanium		20	75-125	93	93	0		89	88	1.1	

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1						Measurement 2					
		Precision	Bias	% Recovery			Precision			% Recovery			Precision		
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)		Sample		Duplicate		(% RPD)	
Vanadium		20	75-125	92		92		0		90		89		1.1	
Zinc		20	75-125	87		84		3.5		87		86		1.2	
Metals in Gas Vapor Phase - GFAAS and CVAAS															
HNO_3/H_2O_2 Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Arsenic		20	75-125	95		97		2.1							
Cadmium		20	75-125	113		118		4.3							
Lead		20	75-125	89		91		2.2							
Mercury (CVAAS)		20	75-125	93		93		0							
Selenium		20	75-125	90		91		1.1							
10% HNO_3/30% H_2O_2 Impingers: Raw Syngas	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Arsenic		20	75-125	93		95		2.1							
Cadmium		20	75-125	117		119		1.7							
Lead		20	75-125	100		105		4.9							
Mercury (CVAAS)		20	75-125	99		104		4.9							
Selenium		20	75-125	102		101		1.0							
10% HNO_3/30% H_2O_2 Impingers: Tail Gas	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Arsenic		20	75-125	93		93		0							
Cadmium		20	75-125	129	Q	124		4.0		127	Q	134	Q	5.4	
Lead		20	75-125	101		100		1.0							
Mercury (CVAAS)		20	75-125	108		108		0							
Selenium		20	75-125	8.9	Q	24	Q	92	Q	8.5	Q	10	Q	16	Q
$KMnO_4$ Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Mercury (CVAAS)		20	75-125	63	Q	80		24	Q	74	Q	86		15	

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Vapor Phase Metals on Charcoal - ICP-AES											
Charcoal Tubes: Sweet Syngas	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Aluminum		20	75-125	94	102	8.2		96	98	2.1	
Antimony		20	75-125	89	90	1.1		86	89	3.4	
Barium		20	75-125	84	77	8.7		80	83	3.7	
Beryllium		20	75-125	81	75	7.7		77	80	3.8	
Boron		20	75-125	84	74 Q	13		75	83	10	
Calcium		20	75-125	97	105	7.9		99	100	1.0	
Chromium		20	75-125	80	75	6.4		77	80	3.8	
Cobalt		20	75-125	79	72 Q	9.3		75	77	2.6	
Copper		20	75-125	81	74 Q	9		77	80	3.8	
Iron		20	75-125	95	102	7.1		97	98	1.0	
Magnesium		20	75-125	94	102	8.2		96	98	2.1	
Manganese		20	75-125	81	75	7.7		77	80	3.8	
Molybdenum		20	75-125	80	76	5.1		76	80	5.1	
Nickel		20	75-125	78	74 Q	5.3		70 Q	76	8.2	
Phosphorus		20	75-125	77	74 Q	4.0		73 Q	77	5.3	
Potassium		20	75-125	94	102	8.2		96	98	2.1	
Silicon		20	75-125	83	77	7.5		81	83	2.4	
Sodium		20	75-125	96	103	7.0		97	99	2.0	
Titanium		20	75-125	82	75	8.9		78	81	3.8	
Vanadium		20	75-125	82	76	7.6		78	82	5.0	
Zinc		20	75-125	75	68 Q	9.8		71 Q	73 Q	2.8	
Charcoal Tubes: Media Spike	Precision - Spiked Media Blank Duplicate										
	Accuracy - Spiked Media Blank Sample										
Aluminum		20	75-125	91	93	2.2					
Antimony		20	75-125	10 Q	14 Q	33.3	Q				
Barium		20	75-125	89	92	3.3					
Beryllium		20	75-125	88	87	1.1					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1					Measurement 2				
		Precision	Bias	% Recovery				Precision	% Recovery				Precision
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)	Sample		Duplicate		(% RPD)
Boron		20	75-125	69		78		12.2					
Calcium		20	75-125	91		91		0.0					
Chromium		20	75-125	82		78		5.0					
Cobalt		20	75-125	87		85		2.3					
Copper		20	75-125	84		86		2.4					
Iron		20	75-125	83		83		0.0					
Magnesium		20	75-125	91		92		1.1					
Manganese		20	75-125	88		88		0.0					
Molybdenum		20	75-125	42	Q	46	Q	9.1					
Nickel		20	75-125	90		87		3.4					
Potassium		20	75-125	81		81		0.0					
Silicon		20	75-125	34	Q	55		47.2	Q				
Sodium		20	75-125	92		96		4.3					
Titanium		20	75-125	81		85		4.8					
Vanadium		20	75-125	86		87		1.2					
Zinc		20	75-125	87		83		4.7					
Vapor Phase Metals on Charcoal - GFAAS and CVAAS													
Charcoal Tubes: Sweet Syngas	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Arsenic		20	75-125	102		105		2.9					
Cadmium		20	75-125	93		96		3.2					
Lead		20	75-125	103		104		0.97					
Mercury (CVAAS)		20	75-125	77		81		5.1					
Selenium		20	75-125	96		99		3.1					
Charcoal Tubes: Media Spike	Precision - Spiked Media Blank Duplicate												
	Accuracy - Spiked Media Blank Sample												
Arsenic		20	75-125	93		92		1.1					
Cadmium		20	75-125	107		106		0.9					
Lead		20	75-125	107		106		0.9					
Mercury (CVAAS)		20	75-125	52	Q	62	Q	17.5					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1						Measurement 2					
		Precision	Bias	% Recovery				Precision		% Recovery				Precision	
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)		Sample		Duplicate		(% RPD)	
Selenium		20	75-125	95		93		2.1							
Metals in Process Solids - ICP/MS															
Matrix: Raw Coal	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Antimony		20	75-125	75		99		25.9	Q						
Arsenic		20	75-125	92		130	Q	30.1	Q						
Beryllium		20	75-125	86		117		29.4	Q						
Cadmium		20	75-125	73	Q	99		29.8	Q						
Chromium		20	75-125	50	Q	92		27.4	Q						
Cobalt		20	75-125	86		122		27.4	Q						
Copper		20	75-125	25	Q	92		29.0	Q						
Lead		20	75-125	31	Q	68	Q	49.4	Q						
Manganese		20	75-125	24	Q	84		30.8	Q						
Mercury		20	75-125	6	Q	55	Q	100.0	Q						
Molybdenum		20	75-125	84		117		30.1	Q						
Nickel		20	75-125	71	Q	155	Q	25.7	Q						
Selenium		20	75-125	-64	Q	-21	Q	34.6	Q						
Vanadium		20	75-125	17	Q	86		27.3	Q						
Matrix: Slurry 32	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Antimony		20	75-125	96		84		13.1							
Arsenic		20	75-125	124		109		11.7							
Beryllium		20	75-125	115		100		13.7							
Cadmium		20	75-125	96		87		9.3							
Chromium		20	75-125	95		75		13.0							
Cobalt		20	75-125	121		100		15.0							
Copper		20	75-125	83		46	Q	14.5							
Lead		20	75-125	41	Q	27	Q	25.9	Q						
Manganese		20	75-125	90		54	Q	15.3							
Mercury		20	75-125	22	Q	10	Q	73.1	Q						

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1					Measurement 2				
		Precision	Bias	% Recovery				Precision	% Recovery				Precision
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)	Sample		Duplicate		(% RPD)
Molybdenum		20	75-125	114		99		12.8					
Nickel		20	75-125	83		25	Q	15.5					
Selenium		20	75-125	120		97		16.3					
Vanadium		20	75-125	81		43	Q	14.3					
Matrix: Slurry 33	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Antimony		20	75-125	98		97		0.7					
Arsenic		20	75-125	132	Q	131	Q	0.5					
Beryllium		20	75-125	116		115		0.7					
Cadmium		20	75-125	103		108		4.3					
Chromium		20	75-125	111		113		1.6					
Cobalt		20	75-125	130	Q	129	Q	0.6					
Copper		20	75-125	115		113		0.8					
Lead		20	75-125	78		69	Q	6.6					
Manganese		20	75-125	114		116		0.7					
Mercury		20	75-125	55	Q	49	Q	10.9					
Molybdenum		20	75-125	119		119		0.1					
Nickel		20	75-125	134	Q	134	Q	0.0					
Selenium		20	75-125	113		129	Q	9.6					
Vanadium		20	75-125	115		118		0.9					
Matrix: Recycled Char Solids	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Antimony		20	75-125	103		94		6.9					
Arsenic		20	75-125	131	Q	137	Q	1.9					
Beryllium		20	75-125	126	Q	122		2.6					
Cadmium		20	75-125	102		92		2.2					
Chromium		20	75-125	106		109		1.3					
Cobalt		20	75-125	129	Q	129	Q	0.1					
Copper		20	75-125	109		101		1.8					
Lead		20	75-125	49	Q	63	Q	1.5					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1						Measurement 2					
		Precision	Bias	% Recovery				Precision		% Recovery				Precision	
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)		Sample		Duplicate		(% RPD)	
Manganese		20	75-125	101		98		1.3							
Mercury		20	75-125	45	Q	69	Q	40.8	Q						
Molybdenum		20	75-125	119		117		1.5							
Nickel		20	75-125	120		119		0.3							
Selenium		20	75-125	88		111		3.6							
Vanadium		20	75-125	102		100		0.7							
Matrix: Slag	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Antimony		20	75-125	90		96		6.7							
Arsenic		20	75-125	113		123		7.7							
Beryllium		20	75-125	115		115		0.1							
Cadmium		20	75-125	94		99		5.2							
Chromium		20	75-125	82		93		6.2							
Cobalt		20	75-125	107		112		3.4							
Copper		20	75-125	62	Q	74	Q	4.7							
Lead		20	75-125	74	Q	69	Q	6.7							
Manganese		20	75-125	67	Q	81		5.8							
Mercury		20	75-125	52	Q	52	Q	1.7							
Molybdenum		20	75-125	107		110		2.6							
Nickel		20	75-125	52	Q	79		6.4							
Selenium		20	75-125	96		106		7.9							
Vanadium		20	75-125	63	Q	80		5.8							
Metals in Process Solids - ICP-AES															
Matrix: Coal	Precision - NA														
Antimony	Accuracy - SRM 1633-b	20	75-125	120											
Barium	1633-b	20	75-125	98											
Beryllium	GBW 07103	20	75-125	96											
Boron	SARM 20	20	75-125	110											
Chromium	1633-b	20	75-125	101											
Cobalt	1633-b	20	75-125	93											

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Copper	1633-b	20	75-125	103							
Molybdenum	GBW 07103	20	75-125	NC							
Manganese	1633-b	20	75-125	98							
Nickel	1633-b	20	75-125	109							
Vanadium	1633-b	20	75-125	99							
Zinc	1633-b	20	75-125	104							
Matrix: Slurry 33	Precision - Analytical Duplicate										
	Accuracy - NA										
Antimony		20	75-125	<1	<1	0.0					
Barium		20	75-125	460	490	6.3					
Beryllium		20	75-125	0.2	0.2	0.0					
Boron		20	75-125	390	380	2.6					
Boron		20	75-125	130	130	0.0					
Chromium		20	75-125	5	5	0.0					
Copper		20	75-125	13	14	7.4					
Cobalt		20	75-125	1	1	0.0					
Manganese		20	75-125	9	9	0.0					
Molybdenum		20	75-125	<2	<2	0.0					
Nickel		20	75-125	2	3	40.0	Q				
Vanadium		20	75-125	14	15	6.9					
Zinc		20	75-125	35	37	5.6					
Matrix: Slag	Precision - Analytical Duplicate										
	Accuracy - NA										
Antimony		20	75-125	<2	<2	0.0					
Barium		20	75-125	5500	5570	1.3					
Beryllium		20	75-125	1.9	1.9	0.0					
Chromium		20	75-125	64	66	3.1					
Copper		20	75-125	140	140	0.0					
Cobalt		20	75-125	32	27	16.9					
Manganese		20	75-125	110	110	0.0					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Molybdenum		20	75-125	<10	<10	0.0					
Nickel		20	75-125	38	34	11.1					
Vanadium		20	75-125	170	170	0.0					
Zinc		20	75-125	54	53	1.9					
Matrix: Sulfur	Precision - Analytical Duplicate										
	Accuracy - NA										
Aluminum		20	75-125	28	27	3.6					
Antimony		20	75-125	<3	<3	0.0					
Barium		20	75-125	<2	<2	0.0					
Beryllium		20	75-125	<2	<2	0.0					
Chromium		20	75-125	<2	<2	0.0					
Copper		20	75-125	<2	<2	0.0					
Cobalt		20	75-125	<4	<4	0.0					
Iron		20	75-125	7	5	33.3	Q				
Manganese		20	75-125	<2	<2	0.0					
Molybdenum		20	75-125	<20	<20	0.0					
Nickel		20	75-125	<4	<4	0.0					
Vanadium		20	75-125	<2	<2	0.0					
Zinc		20	75-125	<2	<2	0.0					
Metals in Process Solids - GFAAS and CVAAS											
Metals in Coal - GFAAS and CVAAS	Precision - NA										
Arsenic	Accuracy - SRM 1632-b	20	75-125	123							
Cadmium	1633-b	20	75-125	100							
Lead	1633-b	20	75-125	117							
Mercury	SARM 20	20	75-125	100							
Selenium	1632-b	20	75-125	65	Q						

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Matrix: Slurry 33	Precision - Analytical Duplicate										
	Accuracy - NA										
Arsenic		20	75-125	<1	<1	0					
Cadmium		20	75-125	2.9	2.8	3.5					
Lead		20	75-125	5	5	0					
Mercury (CVAAS)		20	75-125	<0.02	<0.02	0					
Selenium		20	75-125	3	3	0					
Matrix: Slag	Precision - Analytical Duplicate										
	Accuracy - NA										
Arsenic		20	75-125	3	4	28.6	Q				
Cadmium		20	75-125	<1	<1	0					
Lead		20	75-125	3	3	0					
Matrix: Sulfur	Precision - Analytical Duplicate										
	Accuracy - NA										
Arsenic		20	75-125	<3	<3	0					
Cadmium		20	75-125	<2	<2	0					
Lead		20	75-125	<3	<3	0					
Selenium		20	75-125	10	10	0					
Metals in Process Solids - XRF											
Metals in Coal -XRF	Precision - NA										
	Accuracy - SRM (NBS 2689)										
Silicon		20	75-125	100							
Aluminum		20	75-125	154	Q						
Titanium		20	75-125	101							
Iron		20	75-125	100							
Calcium		20	75-125	1,234	Q						
Magnesium		20	75-125	115							
Potassium		20	75-125	103							
Sodium		20	75-125	114							
Phosphorus		20	75-125	100							

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Strontium		20	75-125	133 Q							
Metals in Coal - XRF	Precision - NA										
	Accuracy - SRM (NBS 2690)										
Silicon		20	75-125	100							
Aluminum		20	75-125	99							
Titanium		20	75-125	93							
Iron		20	75-125	104							
Calcium		20	75-125	97							
Magnesium		20	75-125	97							
Potassium		20	75-125	100							
Sodium		20	75-125	114							
Sulfur		20	75-125	136 Q							
Phosphorus		20	75-125	102							
Strontium		20	75-125	83							
Manganese		20	75-125	50 Q							
Metals in Coal - XRF	Precision - NA										
	Accuracy - SRM (NBS 2691)										
Silicon		20	75-125	99							
Aluminum		20	75-125	100							
Titanium		20	75-125	101							
Iron		20	75-125	102							
Calcium		20	75-125	101							
Magnesium		20	75-125	99							
Potassium		20	75-125	110							
Sodium		20	75-125	95							
Sulfur		20	75-125	87							
Phosphorus		20	75-125	109							
Strontium		20	75-125	93							
Manganese		20	75-125	40 Q							

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1			Measurement 2			
		Precision	Bias	% Recovery		Precision	% Recovery			Precision
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)	Sample		Duplicate	(% RPD)
Matrix: Recycled Char Solids	Precision - Sample Duplicate									
	Accuracy - NA									
Silicon		20	75-125	8.51	8.48	0.4				
Aluminum		20	75-125	5.45	5.43	0.4				
Titanium		20	75-125	0.50	0.50	0.0				
Iron		20	75-125	2.44	2.40	1.7				
Calcium		20	75-125	10.05	9.94	1.1				
Magnesium		20	75-125	2.06	2.02	2.0				
Potassium		20	75-125	0.23	0.22	4.4				
Sodium		20	75-125	1.10	1.09	0.9				
Phosphorus		20	75-125	0.35	0.34	2.9				
Matrix: Slag	Precision - Sample Duplicate									
	Accuracy - NA									
Silicon		20	75-125	21.31	21.09	1.0				
Aluminum		20	75-125	5.93	5.97	0.7				
Titanium		20	75-125	0.63	0.64	1.6				
Iron		20	75-125	8.63	8.59	0.5				
Calcium		20	75-125	10.24	10.5	2.5				
Magnesium		20	75-125	1.40	1.50	6.9				
Potassium		20	75-125	0.56	0.56	0.0				
Sodium		20	75-125	1.94	1.97	1.5				
Phosphorus		20	75-125	2.67	2.55	4.6				
Metals in Aqueous Samples - ICP-AES										
Matrix: Sweet Water	Precision - Matrix-spiked Duplicate									
	Accuracy - Matrix-spiked Sample									
Aluminum		20	75-125	95	97	2.1	93		94	1.1
Antimony		20	75-125	108	110	1.8	100		95	5.1
Barium		20	75-125	91	97	6.4	95		96	1.0
Beryllium		20	75-125	102	104	1.9	98		98	0
Boron		20	75-125	97	102	5.0	3.0	Q	100	188 Q

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Calcium		20	75-125	95	98	3.1		98	98	0	
Chromium		20	75-125	94	95	1.1		94	93	1.1	
Cobalt		20	75-125	94	94	0		93	92	1.1	
Copper		20	75-125	92	96	4.3		91	91	0	
Iron		20	75-125	95	97	2.1		96	96	0	
Magnesium		20	75-125	94	98	4.2		94	94	0	
Manganese		20	75-125	93	95	2.1		94	94	0	
Molybdenum		20	75-125	97	98	1.0		0.12	Q 95	199	Q
Nickel		20	75-125	95	94	1.1		91	91	0	
Phosphorus		20	75-125	89	95	6.5		96	94	2.1	
Potassium		20	75-125	96	98	2.1		93	95	2.1	
Silicon		20	75-125	88	102	15		6.6	Q 109	177	Q
Sodium		20	75-125	91	96	5.4		93	95	2.1	
Titanium		20	75-125	94	96	2.1		0.84	Q 95	200	Q
Vanadium		20	75-125	95	96	1.0		94	94	0	
Zinc		20	75-125	93	96	3.2		95	95	0	
Matrix: Sour Condensate	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Aluminum		20	75-125	92	94	2.2					
Antimony		20	75-125	96	94	2.1					
Barium		20	75-125	91	94	3.2					
Beryllium		20	75-125	97	98	1.0					
Boron		20	75-125	100	96	4.1					
Calcium		20	75-125	95	97	2.1					
Chromium		20	75-125	94	94	0					
Cobalt		20	75-125	93	93	0					
Copper		20	75-125	93	93	0					
Iron		20	75-125	94	95	1.1					
Magnesium		20	75-125	91	94	3.2					
Manganese		20	75-125	94	94	0					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Molybdenum		20	75-125	93	93	0					
Nickel		20	75-125	93	94	1.1					
Phosphorus		20	75-125	87	92	5.6					
Potassium		20	75-125	92	95	3.2					
Silicon		20	75-125	95	100	5.1					
Sodium		20	75-125	91	93	2.2					
Titanium		20	75-125	93	94	1.1					
Vanadium		20	75-125	93	94	1.1					
Zinc		20	75-125	95	94	1.1					
Matrix: Recycle Char Filtrate	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Aluminum		20	75-125	95	96	1.0		88	91	3.4	
Antimony		20	75-125	100	97	3.0		91	95	4.3	
Barium		20	75-125	93	95	2.1		88	90	2.2	
Beryllium		20	75-125	102	103	1.0		95	96	1.0	
Boron		20	75-125	60 Q	79	27	Q	21 Q	65 Q	102	Q
Calcium		20	75-125	92	99	7.3		84	90	6.9	
Chromium		20	75-125	92	94	2.2		88	89	1.1	
Cobalt		20	75-125	91	93	2.2		86	88	2.3	
Copper		20	75-125	92	95	3.2		89	91	2.2	
Iron		20	75-125	94	100	6.2		89	95	6.5	
Magnesium		20	75-125	86	91	5.6		75	87	15	
Manganese		20	75-125	91	93	2.2		88	90	2.2	
Molybdenum		20	75-125	93	97	4.2		87	88	1.1	
Nickel		20	75-125	92	95	3.2		88	90	2.2	
Phosphorus		20	75-125	94	96	2.1		87	91	4.5	
Potassium		20	75-125	96	101	5.1		85	93	9.0	
Silicon		20	75-125	104	102	1.9		98	99	1.0	
Sodium		20	75-125	77	52 Q	39	Q	52 Q	59 Q	13	
Titanium		20	75-125	94	95	1.1		89	90	1.1	

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1					Measurement 2				
		Precision	Bias	% Recovery			Precision		% Recovery			Precision	
		(% RPD)	(% Rec)	Sample	Duplicate		(% RPD)		Sample	Duplicate		(% RPD)	
Vanadium		20	75-125	92	94		2.2		88	90		2.2	
Zinc		20	75-125	91	96		5.4		87	92		5.6	
Metals in Aqueous Samples - GFAAS and CVAAS													
Matrix: Sweet Water	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Arsenic		20	75-125	100	100		0		100	99		1.0	
Cadmium		20	75-125	116	115		0.87		94	85		5.0	
Lead		20	75-125	NA	174	Q	NC		93	56	Q	50	Q
Mercury (CVAAS)		20	75-125	114	112		1.8						
Selenium		20	75-125	108	111		2.7						
Matrix: Sour Condensate	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Arsenic		20	75-125	100	105		4.9		111				
Cadmium		20	75-125	105	100		4.9						
Lead		20	75-125	123	109		12		108				
Mercury (CVAAS)		20	75-125	119	118		0.84						
Selenium		20	75-125	78	84		7.4						
Matrix: Recycle Char Filtrate	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Arsenic		20	75-125	110	110		0		118	109		7.9	
Cadmium		20	75-125	114	117		2.6						
Lead		20	75-125	74	57	Q	26	Q	71	63	Q	12	
Mercury (CVAAS)		20	75-125	69	74	Q	7.0						
Selenium		20	75-125	110	100		9.5						
Ionic Species in Gas Particulate Phase													
Filter/PNR: Incinerator Stack	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Chloride		20	80-120	96	94		2.1						
Fluoride		20	80-120	97	100		3.0						
Sulfate		20	80-120	95	97		2.1						

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1						Measurement 2					
		Precision	Bias	% Recovery				Precision		% Recovery				Precision	
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)		Sample		Duplicate		(% RPD)	
Filter/PNR: Turbine Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	91		90		1.1							
Fluoride		20	80-120	89		89		0							
Sulfate		20	80-120	94		94		0							
Ionic Species in Gas Vapor Phase															
0.1 N H_2SO_4 Impingers: Incinerator Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	129	Q	130	Q	0.77		124	Q	123	Q	0.81	
Fluoride		20	80-120	97		99		2.0							
Ammonia		20	80-120	106		88		19							
0.1 N H_2SO_4 Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	109		105		3.7							
Fluoride		20	80-120	99		104		4.9							
Ammonia		20	80-120	87		90		3.4							
1% H_2SO_4 Impingers: Raw Syngas	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	78	Q	81		3.8		76	Q	82		7.6	
Fluoride		20	80-120	98		102		4.0							
Ammonia		20	80-120	94		89		5.5							
1% H_2SO_4 Impingers: Acid Gas	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	98		94		4.2							
M-8 IPA Impingers: Incinerator Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Sulfate		20	80-120	84		63	Q	29	Q	41	Q	41	Q	0	
M-8 IPA Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Sulfate		20	80-120	29	Q	33	Q	13		22	Q	19	Q	15	

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
M-8 H_2O_2 Impingers: Incinerator Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Sulfate		20	80-120	103	100	3.0					
M-8 H_2O_2 Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Sulfate		20	80-120	106	106	0					
M-7E $KMnO_4$/NaOH Impingers: Incinerator Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Nitrate		20	80-120	86	110	24	Q				
ZnOAc Impingers: Turbine Stack	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Cyanide		20	75-125	99	100	1.0					
ZnOAc Impingers: Comb. Air	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Cyanide		20	75-125	101	91	10		104	97	7.0	
Ionic Species in Process Solids											
Matrix: Slurry 33	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Chloride (D4208/IC)		20	80-120	101	99	2.0		101	103	2.0	
Fluoride (D4208/IC)		20	80-120	104	107	2.8		103	107	3.8	
Matrix: Slurry 33	Precision - Analytical Duplicate										
	Accuracy - None										
Chloride (D4208)		20	80-120	<100	<100	0.0					
Fluoride (D3761)		20	80-120	450	460	2.2					
Matrix: Coal	Precision - NA										
Chloride (D4208)	Accuracy - SRM 1632-b	20	80-120	93							
Fluoride (D3761)	ECR 40	20	80-120	110							

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1						Measurement 2					
		Precision	Bias	% Recovery				Precision		% Recovery				Precision	
		(% RPD)	(% Rec)	Sample		Duplicate		(% RPD)		Sample		Duplicate		(% RPD)	
Matrix: Recycled Char Solids	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride (D4208/IC)		20	80-120	92		98		6.3							
Fluoride (D4208/IC)		20	80-120	1.9	Q	-319	Q	202	Q	36	Q	-264	Q	263	Q
Matrix: Recycled Char Solids	Precision - Analytical Duplicate														
	Accuracy - None														
Chloride (D4208)		20	80-120	<100		<100		0.0							
Fluoride (D3761)		20	80-120	4200		4200		0.0							
Matrix: Slag	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride (SIE)		20	80-120	110		103		6.6							
Chloride (D4208/IC)		20	80-120	83		74	Q	11.5		83		76	Q	8.8	
Fluoride (SIE)		20	80-120	31	Q	69	Q	76	Q	27	Q	57	Q	71	Q
Fluoride (D4208/IC)		20	80-120	90		76		16.9		86		77		11.0	
Anions in Aqueous Samples															
Matrix: Sweet Water	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	95		89		6.5							
Fluoride		20	80-120	111		111		0							
Phosphate (as Total P)		20	75-125	97		101		4.0							
Sulfate		20	80-120	88		88		0							
Ammonia		20	80-120	100		112		11		104		114		9.2	
Cyanide		20	75-125	87		89		2.3							
Thiocyanate		20	80-120	128	Q	158	Q	20.7	Q	128	Q	109		16.7	
Matrix: Sour Condensate	Precision - Matrix-spiked Duplicate														
	Accuracy - Matrix-spiked Sample														
Chloride		20	80-120	20	Q	21	Q	4.9		21	Q	20	Q	4.9	
Fluoride		20	80-120	108		110		1.8							
Phosphate (as Total P)		20	75-125	89		96		7.6							
Sulfate		20	80-120	88		89		1.1							

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1					Measurement 2				
		Precision	Bias	% Recovery			Precision		% Recovery			Precision	
		(% RPD)	(% Rec)	Sample	Duplicate		(% RPD)		Sample	Duplicate		(% RPD)	
Cyanide		20	75-125	96	96		0.0						
Matrix: Recycle Char Filtrate	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Chloride		20	80-120	87	89		2.3						
Fluoride		20	80-120	110	109		0.91						
Ammonia		20	80-120	99	100		1.0		98	100		2.0	
Cyanide		20	75-125	83	207	Q	86	Q	102	237	Q	80	Q
Water Quality Parameters													
Matrix: Sweet Water	Precision - Matrix-spiked Duplicate												
	Accuracy - Matrix-spiked Sample												
Total Phenolics		20	75-125	104	104		0.0						
Chemical Oxygen Demand		20	75-125	82	76		7.6						
Matrix: DI Water													
Chemical Oxygen Demand		20	75-125	85	80		6.1						
Aldehydes in Gas Vapor Phase													
Trip Spike	Precision - NA												
	Accuracy - Trip Spike												
Formaldehyde		50	50-150	77	95				95				
Acetaldehyde		50	70-130	79	92				95				
Field Spike	Precision - NA												
	Accuracy - Field Spike												
Formaldehyde		50	50-150	108					106				
Lab Spike	Precision - NA												
	Accuracy - Lab Spike												
Formaldehyde		50	50-150	92	97				100				
Acetaldehyde		50	70-130	104	84				88				
Acrolein		50	70-130	105	64	Q			NS				

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1			Measurement 2		
		Precision	Bias	% Recovery		Precision	% Recovery		Precision
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)	Sample	Duplicate	(% RPD)
Aldehydes in Aqueous Samples									
Lab Spike	Precision - NA								
	Accuracy - Lab Spike								
Formaldehyde		50	50-150	91			95		
Acetaldehyde		50	70-130	103			103		
Acrolein		50	70-130	105			104		
Matrix: Sweet Water	Precision - NA								
	Accuracy - Matrix Spike								
Formaldehyde		50	50-150	77			64		
Acetaldehyde		50	50-150	107			60		
Acrolein		50	50-150	110			70		
Volatile Organic Compounds in Gas Vapor Phase									
VOST Method Spike	Precision - NA								
	Accuracy - Method Spike								
Chloromethane		NA	D-273	106					
Vinyl Chloride		NA	D-251	115					
Bromomethane		NA	D-242	92					
Chloroethane		NA	NA	102					
Trichlorofluoromethane		NA	NA	66					
1,1-Dichloroethene		NA	D-234	110					
Carbon Disulfide		NA	NA	114					
Acetone		NA	NA	101					
Methylene Chloride		NA	D-221	112					
trans-1,2-Dichloroethene		NA	54-156	107					
1,1-Dichloroethane		NA	59-155	122					
Vinyl Acetate		NA	NA	140					
2-Butanone		NA	NA	43					
Chloroform		NA	51-138	118					
1,1,1-Trichloroethane		NA	52-162	122					
Carbon Tetrachloride		NA	70-140	127					

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Benzene		NA	37-151	106							
1,2-Dichloroethane		NA	49-155	106							
Trichloroethene		NA	71-157	86							
1,2-Dichloropropane		NA	D-210	106							
Bromodichloromethane		NA	35-155	101							
trans-1,3-Dichloropropene		NA	17-183	127							
4-Methyl-2-Pentanone		NA	NA	86							
Toluene		NA	47-150	110							
cis-1,3-Dichloropropene		NA	D-227	110							
1,1,2-Trichloroethane		NA	52-150	107							
Tetrachloroethene		NA	64-148	105							
2-Hexanone		NA	NA	56							
Dibromochloromethane		NA	53-149	116							
Chlorobenzene		NA	37-160	104							
Ethyl Benzene		NA	37-162	107							
m,p-Xylene		NA	NA	108							
o-Xylene		NA	NA	106							
Styrene		NA	NA	107							
Bromoform		NA	45-169	114							
1,1,2,2-Tetrachloroethane		NA	46-157	139							
1,3-Dichlorobenzene		NA	NA	103							
1,4-Dichlorobenzene		NA	NA	103							
1,2-Dichlorobenzene		NA	NA	100							
Volatile Organic Compounds in Aqueous Samples											
Matrix: Sour Condensate	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Benzene		50	37-151	36	Q	52		36			
Chloroethene		50	37-160	100		97		3.0			
1,1-Dichloroethene		50	D-234	87		81		7.1			
Toluene		50	47-150	101		98		3.0			

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1			Measurement 2		
		Precision	Bias	% Recovery		Precision	% Recovery		Precision
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)	Sample	Duplicate	(% RPD)
Trichloroethene		50	71-157	100	95	5.1			
Semivolatile Organic Compounds - Gas Vapor Phase									
XAD Resin Media Spike	Precision - Spiked Media Blank Duplicate								
	Accuracy - Spiked Media Blank Sample								
Acenaphthene		50	47-145	89	93	4.4			
4-Chloro-3-methylphenol		70	22-147	83	87	4.7			
2-Chlorophenol		60	23-134	84	89	5.8			
1,4-Dichlorobenzene		50	20-124	89	93	4.4			
2,4-Dinitrotoluene		50	39-139	96	101	5.1			
N-nitrosodipropylamine		130	D-230	105	109	3.7			
4-Nitrophenol		80	D-132	78	83	6.2			
Pentachlorophenol		90	14-176	46	53	14			
Phenol		50	5-112	80	85	6.1			
Pyrene		50	52-115	83	90	8.1			
1,2,4-Trichlorobenzene		50	44-142	94	99	5.2			
XAD Resin Media Spike	Precision - Spiked Media Blank Duplicate								
	Accuracy - Spiked Media Blank Sample								
Acenaphthene		50	47-145	87	88	1.1			
4-Chloro-3-methylphenol		70	22-147	85	86	1.2			
2-Chlorophenol		60	23-134	84	87	3.5			
1,4-Dichlorobenzene		50	20-124	90	92	2.2			
2,4-Dinitrotoluene		50	39-139	93	94	1.1			
N-nitrosodipropylamine		130	D-230	100	101	1.0			
4-Nitrophenol		80	D-132	88	90	2.2			
Pentachlorophenol		90	14-176	57	52	9.2			
Phenol		50	5-112	83	85	2.4			
Pyrene		50	52-115	80	83	3.7			
1,2,4-Trichlorobenzene		50	44-142	95	96	1.0			

Table A-3 (Continued)

Measurement Parameter	How Measured	Objectives		Measurement 1				Measurement 2			
		Precision	Bias	% Recovery		Precision		% Recovery		Precision	
		(% RPD)	(% Rec)	Sample	Duplicate	(% RPD)		Sample	Duplicate	(% RPD)	
Semivolatile Organic Compounds - Aqueous Samples											
Matrix: Sweet Water	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Acenaphthene		50	47-145	82	90	9.3					
4-Chloro-3-methylphenol		70	22-147	87	95	8.8					
2-Chlorophenol		60	23-134	76	85	11					
1,4-Dichlorobenzene		50	20-124	69	70	1.4					
2,4-Dinitrotoluene		50	39-139	76	86	12					
N-nitrosodipropylamine		130	D-230	114	128	12					
4-Nitrophenol		80	D-132	98	111	12					
Pentachlorophenol		90	14-176	45	65	36					
Phenol		50	5-112	46	83	57	Q				
Pyrene		50	52-115	72	87	19					
1,2,4-Trichlorobenzene		50	44-142	78	80	2.5					
Matrix: Sour Condensate	Precision - Matrix-spiked Duplicate										
	Accuracy - Matrix-spiked Sample										
Acenaphthene		50	47-145	85	89	4.6					
4-Chloro-3-methylphenol		70	22-147	89	91	2.2					
2-Chlorophenol		60	23-134	79	80	1.3					
1,4-Dichlorobenzene		50	20-124	66	73	10					
2,4-Dinitrotoluene		50	39-139	76	77	1.3					
N-nitrosodipropylamine		130	D-230	95	99	4.1					
4-Nitrophenol		80	D-132	105	115	9.1					
Pentachlorophenol		90	14-176	29	47	47					
Phenol		50	5-112	73	85	15					
Pyrene		50	52-115	67	70	4.4					
1,2,4-Trichlorobenzene		50	44-142	74	79	6.5					

Table A-4
Surrogate Spike Recovery Results

Measurement Parameter	Surrogate Spike Recovery (%)			Number Outside Objective
	Objective	Range of Recovery	Number Analyzed	
Volatile Organic Compounds - Vapor Phase				
VOST Tubes - Turbine Stack				
1,2-Dichloroethane-d4	51-145	126-139	9	0
Toluene d-8	77-122	96-121	9	0
4-Bromofluorobenzene	60-128	74-95	9	0
VOST Tubes - Incinerator Stack				
1,2-Dichloroethane-d4	51-145	19-149	10	3
Toluene d-8	77-122	96-528	10	4
4-Bromofluorobenzene	60-128	72-173	10	1
VOST Tubes - Turbine Field Blanks				
1,2-Dichloroethane-d4	51-145	120-121	2	0
Toluene d-8	77-122	99-109	2	0
4-Bromofluorobenzene	60-128	89	2	0
VOST Tubes - Incinerator Field Blanks				
1,2-Dichloroethane-d4	51-145	123-126	3	0
Toluene d-8	77-122	110-120	3	0
4-Bromofluorobenzene	60-128	89-91	3	0
VOST Tubes - Trip Blank				
1,2-Dichloroethane-d4	51-145	128	1	0
Toluene d-8	77-122	92	1	0
4-Bromofluorobenzene	60-128	88	1	0
Volatile Organic Compounds - Aqueous Samples				
Matrix: Sweet Water				
1,2-Dichloroethane-d4	76-114	85-87	4	0
Toluene d-8	88-110	101-103	4	0
1,4-Bromofluorobenzene	86-115	90-93	4	0
Matrix: Sour Condensate				
1,2-Dichloroethane-d4	76-114	85-87	6	0
Toluene d-8	88-110	100-104	6	0
1,4-Bromofluorobenzene	86-115	91-94	6	0
Semivolatile Organic Compounds (SW 8270) - Particulate Phase				
MM-5 Sampling Train (Front Half) - Turbine Stack Samples				
Phenol-d5	50-150	44-65	3	1
Nitrobenzene-d5	50-150	56-77	3	0
1,3,5-Trichlorobenzene-d3	50-150	53-71	3	0
1,4-Dibromobenzene-d4	50-150	54-72	3	0
2-Fluorobiphenyl	50-150	46-63	3	1
2,4,6-Tribromophenol	50-150	69-96	3	0

Table A-4 (Continued)

Measurement Parameter	Surrogate Spike Recovery (%)			Number Outside Objective
	Objective	Range of Recovery	Number Analyzed	
MM-5 Sampling Train (Front Half) - Incinerator Stack Samples				
Phenol-d5	50-150	49-52	3	1
Nitrobenzene-d5	50-150	70-73	3	0
1,3,5-Trichlorobenzene-d3	50-150	67-68	3	0
1,4-Dibromobenzene-d4	50-150	62-66	3	0
2-Fluorobiphenyl	50-150	54-58	3	0
2,4,6-Tribromophenol	50-150	79-80	3	0
MM-5 Sampling Train (Front Half) - Turbine Field Blanks				
Phenol-d5	50-150	69	1	0
Nitrobenzene-d5	50-150	78	1	0
1,3,5-Trichlorobenzene-d3	50-150	69	1	0
1,4-Dibromobenzene-d4	50-150	69	1	0
2-Fluorobiphenyl	50-150	60	1	0
2,4,6-Tribromophenol	50-150	66	1	0
MM-5 Sampling Train (Front Half) - Incinerator Field Blanks				
Phenol-d5	50-150	53	1	0
Nitrobenzene-d5	50-150	73	1	0
1,3,5-Trichlorobenzene-d3	50-150	68	1	0
1,4-Dibromobenzene-d4	50-150	68	1	0
2-Fluorobiphenyl	50-150	54	1	0
2,4,6-Tribromophenol	50-150	74	1	0
MM-5 Sampling Train (Front Half) - Trip Blanks				
Phenol-d5	50-150	56-77	2	0
Nitrobenzene-d5	50-150	72-86	2	0
1,3,5-Trichlorobenzene-d3	50-150	71-78	2	0
1,4-Dibromobenzene-d4	50-150	76-78	2	0
2-Fluorobiphenyl	50-150	51-69	2	0
2,4,6-Tribromophenol	50-150	68-71	2	0
Polycyclic Aromatic Hydrocarbons (PAHs by CARB 429) - Vapor Phase				
MM-5 Sampling Train (Back Half) - Turbine Stack Samples				
d10-Fluorene	50-150	65-86	4	0
d14-Terphenyl	50-150	125-150	4	0
MM-5 Sampling Train (Back Half) - Incinerator Stack Samples				
d10-Fluorene	50-150	66-69	3	0
d14-Terphenyl	50-150	112-151	3	1
MM-5 Sampling Train (Back Half) - Turbine Field Blank				
d10-Fluorene	50-150	71	1	0
d14-Terphenyl	50-150	260	1	1

Table A-4 (Continued)

Measurement Parameter	Surrogate Spike Recovery (%)			Number Outside Objective
	Objective	Range of Recovery	Number Analyzed	
MM-5 Sampling Train (Back Half) - Incinerator Field Blank				
d10-Fluorene	50-150	67	1	0
d14-Terphenyl	50-150	113	1	0
MM-5 Sampling Train (Back Half) - Trip Blanks				
d10-Fluorene	50-150	69-99	2	0
d14-Terphenyl	50-150	144-229	2	1
Semivolatile Organic Compounds (SW 8270) - Vapor Phase				
MM-5 Sampling Train (Back Half) - Turbine Stack Samples				
Phenol-d5	50-150	42-59	6	3
Nitrobenzene-d5	50-150	47-73	6	1
1,3,5-Trichlorobenzene-d3	50-150	41-64	6	1
1,4-Dibromobenzene-d4	50-150	52-78	6	0
2-Fluorobiphenyl	50-150	44-71	6	1
2,4,6-Tribromophenol	50-150	85-116	6	0
MM-5 Sampling Train (Back Half) - Incinerator Stack Samples				
Phenol-d5	50-150	52-63	3	0
Nitrobenzene-d5	50-150	65-71	3	0
1,3,5-Trichlorobenzene-d3	50-150	55-59	3	0
1,4-Dibromobenzene-d4	50-150	56-67	3	0
2-Fluorobiphenyl	50-150	59-66	3	0
2,4,6-Tribromophenol	50-150	101-119	3	0
MM-5 Sampling Train (Back Half) - Turbine Field Blank				
Phenol-d5	50-150	66	1	0
Nitrobenzene-d5	50-150	80	1	0
1,3,5-Trichlorobenzene-d3	50-150	66	1	0
1,4-Dibromobenzene-d4	50-150	82	1	0
2-Fluorobiphenyl	50-150	69	1	0
2,4,6-Tribromophenol	50-150	68	1	0
MM-5 Sampling Train (Back Half) - Incinerator Field Blank				
Phenol-d5	50-150	48	1	1
Nitrobenzene-d5	50-150	73	1	0
1,3,5-Trichlorobenzene-d3	50-150	58	1	0
1,4-Dibromobenzene-d4	50-150	58	1	0
2-Fluorobiphenyl	50-150	54	1	0
2,4,6-Tribromophenol	50-150	91	1	0

Table A-4 (Continued)

Measurement Parameter	Surrogate Spike Recovery (%)			Number Outside Objective
	Objective	Range of Recovery	Number Analyzed	
MM-5 Sampling Train (Back Half) - Trip Blanks				
Phenol-d5	50-150	50-53	2	0
Nitrobenzene-d5	50-150	64-69	2	0
1,3,5-Trichlorobenzene-d3	50-150	56-60	2	0
1,4-Dibromobenzene-d4	50-150	64	2	0
2-Fluorobiphenyl	50-150	48-56	2	1
2,4,6-Tribromophenol	50-150	89-93	2	0
Semivolatile Organic Compounds (SW 8270) - Internal Process Gas Streams				
MM-5 Sampling Train (Back Half) - Sweet Syngas				
2-Fluorobiphenyl	30-115	88-97	5	0
2-Fluorophenol	25-121	71-98	5	0
Nitrobenzene-d5	23-120	60-92	5	0
Phenol-d5	24-113	85-102	5	0
Terphenyl-d14	18-137	80-100	5	0
2,4,6-Tribromophenol	19-122	56-90	5	0
MM-5 Sampling Train (Back Half) - Sour Syngas				
2-Fluorobiphenyl	30-115	92-97	3	0
2-Fluorophenol	25-121	74-87	3	0
Nitrobenzene-d5	23-120	67-72	3	0
Phenol-d5	24-113	89-107	3	0
Terphenyl-d14	18-137	79	3	0
2,4,6-Tribromophenol	19-122	58-106	3	0
MM-5 Sampling Train (Back Half) - Tail Gas				
2-Fluorobiphenyl	30-115	88	1	0
2-Fluorophenol	25-121	63	1	0
Nitrobenzene-d5	23-120	NC	1	--
Phenol-d5	24-113	83	1	0
Terphenyl-d14	18-137	NC	1	--
2,4,6-Tribromophenol	19-122	73	1	0
MM-5 Sampling Train (Back Half) - Acid Gas				
2-Fluorobiphenyl	30-115	88-98	3	0
2-Fluorophenol	25-121	67-77	3	0
Nitrobenzene-d5	23-120	NC	3	--
Phenol-d5	24-113	81-96	3	0
Terphenyl-d14	18-137	65-70	3	0
2,4,6-Tribromophenol	19-122	60-73	3	0

Table A-4 (Continued)

Measurement Parameter	Surrogate Spike Recovery (%)			Number Outside Objective
	Objective	Range of Recovery	Number Analyzed	
Semivolatile Organic Compounds - Aqueous Samples				
Matrix: Sweet Water				
2-Fluorobiphenyl	43-116	48-72	8	0
2-Fluorophenol	21-100	65-86	8	0
Nitrobenzene-d5	35-114	60-93	8	0
Phenol-d5	10-94	68-96	8	1
Terphenyl-d14	33-141	72-104	8	0
2,4,6-Tribromophenol	10-123	67-87	8	0
Matrix: Sour Condensate				
2-Fluorobiphenyl	43-116	50-77	8	0
2-Fluorophenol	21-100	61-88	8	0
Nitrobenzene-d5	35-114	57-94	8	0
Phenol-d5	10-94	65-93	8	0
Terphenyl-d14	33-141	68-93	8	0
2,4,6-Tribromophenol	10-123	58-75	8	0

APPENDIX B: SAMPLING METHODS

Radian used established sampling methods, where possible, to collect representative samples from the various sampling locations within the LGTI and Power II plant sites.

For the gaseous emission sources (incinerator and turbine exhaust stacks), the sampling methods that were used are standard methods with known performance characteristics, specific for the collection of representative samples from these stream matrices. These standard methods, summarized in Table B-1, provide for data comparisons with industry standards and are comparable to those methods used in the EPRI-sponsored Field Chemical Emissions Monitoring (FCEM) programs. All of the internal process gas streams were sampled using techniques that, although they have not been validated for syngas matrices, are generally considered appropriate for representative sample collection. Some slight modifications to the methods were required, however. These method modifications are summarized in Table B-2. The sampling methods used during the toxics characteristics testing (Periods 1-3) are described first. Test procedures used during the testing of the hot syngas are described separately, as some minor changes were made to some of the methods, based upon the first set of results.

Gas Streams

Sampling methods are described by type in the following sections. The standard approach is described first. These were applied to the emission sources. Following the standard method descriptions, any deviations from the standard approach which were required to adapt the procedure to a syngas matrix are described.

Particulate Loading

EPA Method 5. EPA Reference Method 5 was performed to determine particulate emission loading. This method provides for isokinetic extraction of particulate matter on a glass fiber filter. However, since particulate loading was performed in conjunction with the determination of particulate and vapor-phase metals, quartz fiber filters were used in place of glass. The particulate mass, which includes all material that condenses at or above the filtration temperature, was determined gravimetrically, after the removal of uncombined water.

Table B-1
Summary of Standard Sampling Methods

Stream Type	Parameter	Frequency	Sampling Method
Solids	All	Three grab samples to form daily composite.	EPA Method S007 (trowel/ scoop). EPA Method S004 (slurry, slag).
Liquids	All inorganic & SVOC.	Three grab samples to form daily composite. VOC, aldehydes once per day.	EPA Method S004 (tap)
Turbine and Incinerator Stack Gases	Volatile organics	3 pairs of VOST traps over 2-hour time period	VOST (SW-846 Method 0030)
	Semivolatile organics—PAHs	Isokinetic, integrated 4- to 6-hour sample.	Modified Method 5 (SW-846) Method 0010, CARB 429
	Vapor-phase inorganic species, Cl^-, F^-, NH_3, HCN	Integrated sample over 1- to 2-hour time period	Absorption into various impinger solutions.
	Aldehydes	Integrated 1-2 hour sample.	Method 0011, absorption into DNPH solution
	Trace elements (metals)	Isokinetic, integrated 4- to 6-hour sample.	Method 29 multi-metals sampling train
	Reduced sulfur species	Integrated sample into Tedlar® bag	On-site analysis using GC-FPD (modified Method 16)
	SO_2, NO_x, CO	Continuous emission monitors	EPA Methods 6C, 7E, and 10.
	Particulate	Isokinetic, integrated 4-6 hour sample.	EPA Method 5
	PM10	Single point, semi-isokinetic, sample over appropriate time period	EPA Method 201A

Table B-2
Method Modifications

Parameter	Standard Method	Modification	Streams Affected
Metals	Method 29	Steel instead of glass lined components; Isokinetics are approximated; Fixed point sampling, no traversing; Increase nitric acid and peroxide content to 10 and 30%, respectively; and Filter housing not maintained at 250°F.	5a, 5b, 12, 14, 15, 22, Nat'l Gas
Metals	Method 29	Vapor phase only.	14, 15, 22, Nat'l Gas
Metals	Method 29	All listed above and no permanganate impinger solution.	5, 5a, 5b, 11, 14, 15, 22
Cyanide	ZnOAc absorbing solution	H_2S removal prior to absorbing solution via Pb salt solution (PbOAc).	All internal

The sampling was conducted at equal time intervals along selected traverse points as determined by EPA Reference Method 1.

Sample recovery includes the particulate that has been deposited inside the sample nozzle, heated probe liner, and filter holder (designated as the front half probe and nozzle rinse, PNR), as well as the particulate collected on the filter substrate.

PM-10, EPA Method 201A. EPA Method 201A was used to perform an in-stack measurement of particulate matter equal to or less than an aerodynamic diameter of nominally 10 μm. A gas sample was extracted at a constant rate through an in-stack sizing device. Variation from isokinetic sampling conditions were maintained within well defined limits. Particulate mass was determined gravimetrically after removal of uncombined water. A stainless steel impactor served as the sizing device, and a backup glass fiber filter was used to capture the fine particulate.

Particulate Loading Modifications. Particulate loading was attempted on the raw syngas stream in and out of the particulate removal system and on the sweet syngas. Due to high moisture content of the raw syngas in and out of the venturi scrubber, particulate loading samples could not be collected from these locations. The flow rate was monitored at the sweet syngas location with a differential pressure gauge across a flow orifice. The filter housing was constructed of stainless steel specifically designed for high pressure applications. Gas volumes were calculated by measuring gas flow and sampling duration. Also, there was no probe and nozzle rinse associated with this process system since the probe is not removable. No significant impact on the determination of particulate loading is expected from these modifications.

Particulate- and Vapor-Phase Metals

Sampling for particulate and vapor-phase metals was performed in conjunction with Method 5 using the procedures detailed in EPA Draft Method 29. Method 29 is similar to Method 5 with a few sample train modifications. Modifications to Method 5 include replacing the stainless steel nozzle and probe liner with glass components. The particulate material was collected on quartz fiber substrates, replacing the standard glass fiber filters normally used with Method 5. Vapor-phase metals are collected in a series of impinger solutions. The first two impingers contain a dilute nitric acid and hydrogen peroxide solution. The third impinger is empty. The next two impingers contain acidic potassium permanganate solution for elemental mercury collection. These impingers were followed by one dry impinger, and an impinger filled with silica gel. A minimum of 100 dry standard cubic feet of gas was collected isokinetically. A description of the sample train configuration and recovery procedures is presented in Table B-3.

Method 29 Modifications

The EPA Method 29 sampling train was designed for oxidizing atmospheres. In reducing atmospheres such as those found in gasification systems, the oxidizing potential of the absorbing solutions is rapidly consumed, leading to greatly reduced collection efficiency. In an effort to

Table B-3
Description and Recovery of Method 29 (Multi-Metals) Sampling Train

Component	Contents	Recovery [a]	Container
Probe Nozzle Rinse and front half of filter holder. rinse [b]	NA	Rinse probe, nozzle, and front half of filter holder with acetone into sample container.	500 mL amber glass bottle
Probe Nozzle Rinse and front half of filter holder rinse [b]	NA	Rinse probe, nozzle, and front half of filter holder with 0.1N HNO_3 into sample container.	500 mL amber glass bottle
Filter	Tared quartz filter	Place filter in sample container.	Plastic petri dish
Transfer Line Rinse	NA	Rinse transfer line with 0.1N HNO_3 into sample container.	Combine transfer line rinse and impingers 1 and 2 in a 1000 mL amber glass bottle
Impinger #1	5% nitric acid in 10% hydrogen peroxide (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with 0.1N HNO_3 into sample container.	
Impinger #2	5% nitric acid in 10% hydrogen peroxide (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with 0.1N HNO_3 into sample container.	
Impinger #3	Dry	Recover condensate, then rinse impinger and connecting glassware with fresh $KMnO_4$ solution into sample container.	Combine impingers 3,4, and 5 in a 1000 mL amber glass bottle.
Impinger #4	4% potassium permanganate in 10% sulfuric acid (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with fresh $KMnO_4$ solution into sample container.	
Impinger #5	4% potassium permanganate in 10% sulfuric acid (200 mL)	Recover impinger solution, then rinse impinger with fresh $KMnO_4$ solution into sample container.	
Impinger #4 - Second Rinse	NA	Rinse impinger with 8N HCl into sample container. Not to exceed 25 mL HCl.	250 mL amber glass bottle
Impinger #5 - Second Rinse	NA	Rinse impinger with 8N HCl into sample container. Not to exceed 25 mL HCl.	
Impinger #6	Silica Gel (300 g)	Replace when exhausted.	None

NA = Not applicable.

[a] All impingers will be weighed prior to recovery to determine gas sample moisture content by EPA Reference Method 4.

[b] Turbine exhaust and incinerator stack only.

offset the effects of the reducing gas matrix on the absorbing solutions, a modification was applied to all internal process gas streams. The hydrogen peroxide concentration in impingers one and two was increased from 10 to 30 percent. The permanganate impingers for the collection of elemental mercury were not used in those gas streams with high concentrations of H_2S as the permanganate solution would be rapidly consumed. Elemental mercury was determined at these locations by charcoal adsorption or on-line AAS techniques described below.

On-line AAS. Selected vapor-phase metals were determined directly using an atomic absorption spectrophotometer (AAS). The AAS was modified to accept syngas for a portion of the fuel gas going to the nebulizer mixing chamber and flame. In the flame, vapor-phase trace elements are atomized and absorb light energy from an element-specific light source just like aqueous samples in conventional AAS. The sample gas, fuel gas and air supplies are regulated and monitored to determine the syngas component going to the flame, and ultimately the elemental concentration in the gas sample stream. Absorbance and concentration are related by Beer's law and gas concentrations are determined by comparison with standard curves generated from aqueous standards. The following metals, were determined using the on-line AAS:

- Arsenic;
- Cadmium;
- Nickel;
- Mercury;
- Chromium;
- Lead;
- Selenium; and
- Zinc.

A schematic of the AAS system is shown in Figure B-1.

Charcoal Adsorption

Many gas-phase trace elements in gasification systems can be adsorbed onto charcoal. A slipstream of the particulate-free gas was drawn through specially prepared charcoal tubes at rates not exceeding 1 liter/minute. Previous studies for EPRI have shown charcoal to be a very effective sorbent for some metals, especially iron and nickel (which may be present as the carbonyl) and arsenic. Arsenic (in the form of arsine, AsH_3) is readily adsorbed on charcoal, but it is unknown whether other forms of arsenic are as effectively collected. Target species for

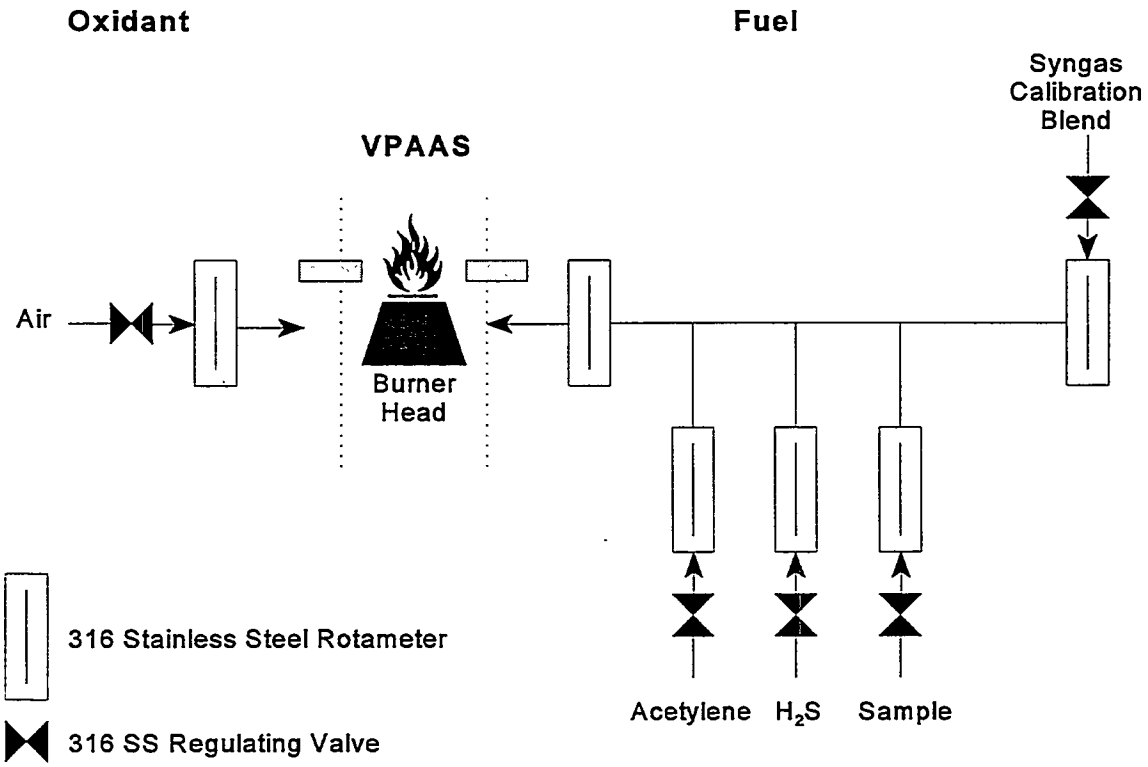

Figure B-1
Schematic of Online AAS

quantification using the charcoal tubes were antimony, arsenic, iron, lead, mercury, nickel, and zinc.

Coconut-based charcoal was aggressively cleaned using concentrated nitric acid followed by an ultra-pure deionized water rinse then dried over night. The charcoal was then loaded into precleaned quartz tubes. Two charcoal tubes were placed in series and exposed to a minimum volume of 50 liters of gas (100 liters typical). Following collection, the charcoal tubes were sealed with plastic caps and sent to the laboratory for analysis.

Sulfur Dioxide/Sulfuric Acid

Sulfur dioxide and sulfuric acid mist were determined on the two stacks using EPA Method 8. This method is identical to EPA Method 5 with the exception of the impinger contents. The first impinger contains an 80% isopropanol (IPA) mixture and the second and third impingers contain a 6% hydrogen peroxide solution. Sulfuric acid mist is collected in the IPA solution, sulfur dioxide in the peroxide solution.

Ammonia/Hydrogen Cyanide/Anions

The sample collection train for the determination of ammonia, hydrogen cyanide, and anions (acid gas) was similar to the EPA Method 5 train, except isokinetic sampling was not required. For the collection of ammonia and anions, dilute sulfuric acid was placed in the first two impingers of the condenser assembly. For the collection of cyanide, a dilute zinc acetate solution was placed in the fourth and fifth impingers of the condenser assembly. Sample volume for each run was typically 30 to 40 dry standard cubic feet, depending upon the location. A description of the sample train configuration and recovery procedures is presented in Table B-4.

Modifications

For internal gas streams, the sulfuric acid absorbing solution was increased to 5 percent. Also, for gas streams containing H_2S, an impinger(s) containing PbOAc (pH<4), was placed in front of the cyanide absorbing solution for H_2S removal.

Volatile Organics

The volatile organic compounds were collected using a volatile organic sampling train (VOST). The VOST is described in Method 0030 in SW-846, *Test Methods for Evaluating Solid Waste*, Third Edition, November 1986. In the VOST, volatile organics are removed from the sample gas by sorbent traps maintained at 20°C. The first sorbent trap contains Tenax resin and the second trap contains Tenax followed by petroleum-based charcoal. To increase the collection efficiency, the sample gas is cooled and dried by passing it through a water-cooled condenser prior to its contact with the sorbent trap. A dry gas meter is used to measure the volume of gas passed through the pair of traps. Sample volumes of 20 liters were collected on three separate pairs of traps at 0.5 liter per minute sampling rate.

Table B-4
Description and Recovery of Ammonia, Anions, and Hydrogen Cyanide Sampling Train

Component	Solution	Recovery[a]	Container
Transfer Line Rinse	NA	Rinse transfer line with 0.1N sulfuric acid into sample container.	1000 mL Nalgene bottle
Impinger #1 (NH_3)	0.1N H_2SO_4 (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with deionized water into sample container.	
Impinger #2 (NH_3)	0.1N H_2SO_4 (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with deionized water into sample container.	
Impinger #3	Dry	Recover condensate, then rinse impinger and connecting glassware with deionized water into sample container.	
Impinger #4 (CN)	2% w/v ZnOAc (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with deionized water into sample container.	1000 mL Nalgene bottle Cool to 4°C
Impinger #5 (CN)	2% w/v ZnOAc (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with deionized water into sample container.	
Impinger #6	Silica Gel (300 g)	Not recovered.	None

NA = Not applicable.

[a] All impingers were weighed prior to analysis.

Leak checks are performed before and after collection on each pair of resin traps. After the post-collection leak check was completed, the traps were sealed with their end caps and returned to their respective glass containers for storage and transport. During storage and transportation, the traps were kept cool (4°C).

Aldehydes

Aldehydes were collected using an impinger train containing 2,4-dinitrophenylhydrazine (DNPH) as described in EPA Method 0011. The sampling system was cleaned prior to shipment according to the protocol method and transported to the site. After sampling, the impinger solutions were combined into one sample along with the methylene chloride glassware rinse. The solutions were sealed in amber glass containers with Teflon closures and stored at 4°C. A description of the sample train configuration and recovery procedures is presented in Table B-5.

Semivolatile Organic Compounds and Selected Polycyclic Aromatic Hydrocarbons

Semivolatile organics (SVOCs) and polycyclic aromatic hydrocarbons (PAHs) were collected using a Modified Method 5 (MM5) sampling train. The probe washes, filter catches, XAD sorbent traps, and aqueous condensates were extracted and analyzed by a combination of analytical protocols, SW-846 Method 8270 and CARB Method 429 (PAHs).

The MM5 protocol is outlined as Method 0010 in SW-846, "Test Methods for Evaluating Solid Waste," Third Edition, November 1986. The sampling system consists of a heated probe, heated filter, sorbent module, and pumping and metering unit. A gooseneck nozzle of an appropriate diameter to allow isokinetic sample collection is attached to the probe. S-type pitot tube differential pressure is monitored to determine the isokinetic sampling rate.

Sampling of the stack gases was conducted in accordance with the published MM5 protocol. This involved collecting the samples isokinetically across two cross-sectional diameters of the stack. The sampling rate for each train was between 0.5 and 1.0 dscfm. A minimum of 100 dscf was collected by each train over a minimum sampling period of two hours.

Sampling train preparation and sample recovery were performed in a controlled environment to reduce the possibility of sample contamination. Prior to assembly, each component of the sampling train was thoroughly rinsed with methylene chloride. The XAD sorbent traps were prepared by the CARB 429 protocol and spiked with isotopically labeled surrogate PAHs. These traps are kept refrigerated after spiking to preserve the spike integrity.

After sample collection, the ends of the sampling train were once again sealed with solvent-rinsed foil and returned to the clean-up area for sample recovery. The filter was recovered and placed in a methylene chloride-rinsed glass petri dish. Aqueous condensate collected in the first two impingers and in the sorbent trap were transferred to methylene chloride-rinsed amber glass

Table B-5
Description and Recovery of Aldehydes Sampling Train

Component	Solution	Recovery	Container
Transfer Line Rinse	NA	Rinse transfer line with methylene chloride into sample container.	1000 mL amber glass bottle Cool to 4°C
Impinger #1	DNPH Solution (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with methylene chloride into sample container.	
Impinger #2	DNPH Solution (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with methylene chloride into sample container.	
Impinger #3	Dry	Recover condensate into sample container.	
Impinger #4	Silica Gel (300 g)	Not recovered.	None

NA = Not applicable.

bottles with Teflon®-lined screw cap closures. All components of the sampling train, from the nozzle through the sorbent module, including the probe, filter glassware, and impinger glassware were rinsed thoroughly with methylene chloride. The probe was cleaned using a nylon brush followed by rinsing with methylene chloride. The probe rinse and glassware rinses were combined with the recovered condensate sample. The XAD-2 resin cartridges were sealed and transferred to the laboratory. A description of the sample train configuration and recovery procedures is presented in Table B-6. Samples from the two emission stacks were analyzed according to the CARB Method 429, a high resolution GC/MS technique for selected PAHs.

Modifications. All internal stream samples were vapor phase only and were analyzed by standard GC/MS per Method 8270.

Majors, Reduced Sulfur, Hydrocarbons

Grab samples were collected for the characterization of major gases, reduced sulfur species and for C_1 - C_{10} hydrocarbons. Samples were collected into Tedlar® bags.

Table B-6
Description and Recovery of Modified Method 5 (Semivolatile and PAHs) Sampling Train

Component	Solution	Recovery	Container
Probe Nozzle Rinse [a]	NA	Rinse probe, nozzle, and front half of filter holder with methylene chloride into sample container.	500 mL amber glass bottle Cool to 4°C
Filter [a]	Pre-treated Quartz Filter	Place filter in sample container.	Glass petri dish Cool to 4°C
XAD Cartridge	XAD-2 Resin	Seal resin cartridge.	Wrap in aluminum foil. Cool to 4°C
Transfer Line Rinse	NA	Rinse transfer line with methylene chloride into sample container.	1000 mL amber glass bottle Cool to 4°C
Condenser	NA	Rinse condenser with methylene chloride into sample container.	
Impinger #1	Dry	Recover condensate, then rinse impinger and connecting glassware with methylene chloride into sample container.	
Impinger #2	Ultrapure Water (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with methylene chloride into sample container.	
Impinger #3	Ultrapure Water (200 mL)	Recover impinger solution, then rinse impinger and connecting glassware with methylene chloride into sample container.	
Impinger #4	Silica Gel (300 g)	Not recovered.	None

NA = Not applicable.

[a]Turbine exhaust and incinerator stack only.

Carbon Monoxide, Sulfur Dioxide, Nitrogen Oxides

Continuous emission monitors (CEMs) were used to determine the gas concentration of these species at the turbine and incinerator stacks. These instruments were operated according to the protocols of EPA Methods 10, 6C, and 7E, respectively.

Solid Sampling Procedures

Solid stream samples (raw coal, coal slurry, and slag) were collected using grab sampling techniques. Samples were collected three times per day and composited daily throughout all test periods. Daily composite samples of raw coal, coal slurry, and slag were mixed well and split to

produce a 1 kilogram (minimum) sample which was placed in a plastic container and sealed for transportation to the laboratory. Coal slurry samples were collected from two locations, the primary and secondary stage feed lines. Slag samples were collected via the slag sample collection system currently in use by LGTI. This system diverts a slip-stream of the slag (that is continually being discharged from the reactor) through a strainer/filter. The collected slag was allowed to cool (without water washing) prior to sample recovery.

Liquid Sampling Procedures

Sour and sweet water samples were collected by grab-tap techniques. Samples were collected three times per day during Period 3 and combined to form a single daily composite. The grab samples are composited directly into appropriate containers and preserved as soon as possible after collection. In some cases the sample was added directly to bottles containing the preservative in order to reduce the loss of the more volatile species (e.g., NH_3, CN^-).

Process water samples collected for the analysis of volatile organic compounds and aldehydes present the only exception to the collection procedures described above. Due to the volatility of these analytes, these samples were collected once daily directly into amber glass containers without filtration. All samples for organic compounds were chilled to 4°C following collection.

Hot Gas Sampling System

Sample collection with the hot gas sampling system was very similar to those methods used for all syngas matrices. The following insertion of the hot gas probe, the hot gas was extracted at a rate of approximately 4 acfm. Vapor-phase samples were collected from heated sample taps as slip streams to the primary gas flow. Some minor sampling method modifications to those described for Periods 1-3 were made for the Period 4 (hot gas) testing, specifically for the multi-metals (M-29) sampling train and for the anions sampling train. Following are specifics regarding changes to the test methodologies.

Potassium permanganate impingers were added to the M-29 sampling train per the standard setup, except two impingers containing sodium hydroxide (2N) were placed in front of the potassium permanganate impingers to remove H_2S. While not entirely effective at removing all H_2S from the syngas, the caustic impingers did remove enough H_2S so that elemental mercury could be collected in the permanganate solution. The caustic scrubber as well as the permanganate impingers were removed from the M-29 sampling train after the collection of 30-40 cubic feet of gas had been sampled to minimize the degradation effect of the H_2S.

The scrubbing solution for anions was also changed for hot gas testing. Sulfuric acid (1%) was used during Periods 1-3. However, the high sulfate content interfered with the chloride analysis and the necessary sample dilution increased the detection limits. For the hot gas test phase, the absorbing solution was changed to a 0.3 mM sodium bicarbonate/2.4 mM sodium carbonate solution. This is basically the ion chromatograph eluent solution used in EPA Method 300. Use

of this absorbing solution produced a transparent background for the analysis of chloride and fluoride.

Collection methods for metals by charcoal, ammonia, and cyanide were not changed from the methodologies used in Periods 1-3.

APPENDIX C: SAMPLE PREPARATION AND ANALYSIS

Like the selection of sampling methods, the analytical methods applied to process samples from coal gasification and other partial oxidation combustion systems require special attention to matrix effects and interferences from reduced species. Many process samples contain reactive substances (e.g., hydrogen sulfide) that undergo or create chemical changes before the sample is analyzed.

Before some samples can be analyzed, sample preparation is necessary to produce a suitable matrix for analysis. Once prepared, most of the chemical and instrumental methods for analysis are common to samples collected from the gas, solid, and liquid streams. This section describes the sample preparation and analysis scheme that correlates with the sample components listed for each gas sampling train and process sample described in Appendix B. This section also describes specialized analytical techniques, identifies subcontract laboratories and their roles, and presents the analytical method references. Samples collected in fulfillment of the EMP were analyzed by methods specified in the EMP. Complete lists of the target metals and ionic species, organic compounds, and radionuclide analytes for the HAPs measurement program are presented in Tables C-1 through C-3.

Gas Samples

Gas samples were collected from two distinctly different sources, internal process streams (reducing environment) and emissions sources (oxidized streams). Correspondingly, the discussion of sample preparation and analysis is presented in two parts. First, all sample preparation and analytical techniques common to both gas sources are presented. Second, the special preparation methods and analytical techniques required for the internal process streams are described.

In Table C-4, the methods that will be used in the analyses of EMP samples are summarized and compared to the analytical methods selected for the HAPs samples. The analytical methods are the same for many parameters. Where the methods differ, samples will be analyzed by both the EMP and HAPs protocols.

Table C-1
Analyte List for Inorganic Parameters

Trace Elements	Major Elements
Antimony [a]	Aluminum
Arsenic [a]	Calcium
Barium [a]	Iron
Beryllium [a]	Magnesium
Boron[b]	Potassium
Cadmium [a]	Silicon [b]
Chromium, total [a]	Sodium
Cobalt [a]	Titanium
Copper [a]	**Ultimate/Proximate Parameters**
Lead [a]	Carbon
Manganese [a]	Hydrogen
Mercury	Nitrogen
Molybdenum [a]	Sulfur
Nickel [a]	Ash
Selenium [a]	Volatile Matter
Vanadium [a]	Fixed Carbon
Zinc	Higher Heating Value (HHV)
Ionic Species	
Chloride (Cl^-)	
Fluoride (F^-)	
Phosphate (as Total P)	
Sulfate (SO_4^{-2})	
Ammonia	
Cyanide	
Sulfide	
Formate	
Thiocyanate	

[a] These elements analyzed by ICP-MS in the gas impinger samples.

[b]Silicon and boron not determined in gas particulate samples.

Table C-2
Analyte List for Organic Parameters

Volatile Organics [a] (Method 8240)	Semivolatile Organics (Method 8270/CARB 429 [b])	
Benzene	Acenaphthene [b]	2,4-Dimethylphenol
Bromoform	Acenaphthylene [b]	Dimethylphthalate
Carbon Disulfide	Acetophenone	4,6-Dinitro-2-methylphenol
Carbon Tetrachloride	4-Aminobiphenyl	2,4-Dinitrophenol
Chlorobenzene	Aniline	2,4-Dinitrotoluene
Chloroform	Anthracene [b]	2,6-Dinitrotoluene
1,4-Dichlorobenzene	Benzidine	bis(2-Ethylhexyl)phthalate
cis-1,3-Dichloropropene	Benzo(a)anthracene [b]	Fluoranthene [b]
trans-1,3-Dichloropropene	Benzo(a)pyrene [b]	Fluorene [b]
Ethylbenzene	Benzo(b)fluoranthene [b]	Hexachlorobenzene
Ethylchloride (Chloroethane)	Benzo(g,h,i)perylene [b]	Hexachlorobutadiene
Ethylene Dichloride (1,2-Dichloroethane)	Benzo(k)fluoranthene [b]	Hexachlorocyclopentadiene
Ethylidene Dichloride (1,1-Dichloroethane)	Benzoic Acid	Hexachloroethane
Methyl Bromide (Bromomethane)	Benzyl Alcohol	Indeno(1,2,3-cd)pyrene [b]
Methyl Chloride (Chloromethane)	4-Bromophenyl Phenyl Ether	Isophorone
Methyl Chloroform (1,1,1-Trichloroethane)	Butylbenzylphthalate	2-Methylnaphthalene [b]
Methyl Ethyl Ketone (2-Butanone)	4-Chloro-3-Methylphenol	2-Methylphenol (o-cresol)
Methylene Chloride (Dichloromethane)	p-Chloroaniline	4-Methylphenol (p-cresol)
Propylene Dichloride (1,2-Dichloropropane)	bis(2-Chloroethoxy)methane	N-Nitrosodimethylamine
Styrene	bis(2-Chloroethyl)ether	N-Nitrosodiphenylamine
1,1,2,2-Tetrachloroethane	bis(2-Chloroisopropyl)ether	N-Nitrosopropylamine
Tetrachloroethene	2-Chloronaphthalene [b]	Naphthalene [b]
Toluene	2-Chlorophenol	2-Nitroaniline

Table C-2 (Continued)

Volatile Organics [a] (Method 8240)	Semivolatile Organics (Method 8270/CARB 429 [b])	
1,1,2-Trichloroethane	4-Chlorophenyl Phenyl Ether	3-Nitroaniline
Trichloroethene	Chrysene [b]	4-Nitroaniline
Vinyl Acetate	Di-n-octylphthalate	Nitrobenzene
Vinyl Chloride	Dibenz(a,h)anthracene [b]	2-Nitrophenol
Vinylidene Chloride (1,1-Dichloroethene)	Dibenzofuran	4-Nitrophenol
m,p-Xylene	Dibutylphthalate	Pentachloronitrobenzene
o-Xylene	1,2-Dichlorobenzene	Pentachlorophenol
	1,3-Dichlorobenzene	Phenanthrene [b]
	1,4-Dichlorobenzene	Phenol
	3,3-Dichlorobenzidine	Pyrene [b]
	2,4-Dichlorophenol	1,2,4-Trichlorobenzene
	Diethylphthalate	2,4,5-Trichlorophenol
	p-Dimethylaminoazobenzene	2,4,6-Trichlorophenol

[a]These are the volatile organic compounds detected by VOST (Method 8240) that are listed in the Clean Air Act list of hazardous air pollutants.

[b]These semivolatile organic compounds analyzed in the gas samples by CARB Method 429 using high resolution GC/MS.

Table C-3
List of Radionuclides

Gamma Emitters	Nominal Detection Limits[a]
Actinium-228 @ 338 KeV	0.7
Actinium-228 @ 911 KeV	0.5
Actinium-228 @ 968 KeV	0.8
Bismuth-212 @ 727 KeV	1.7
Bismuth-214 @ 609.4 KeV	0.3
Bismuth-214 @ 1120.4 KeV	1.3
Bismuth-214 @ 1764.7 KeV	1.1
Lead-210 @ 46 KeV	2.8
Lead-212 @ 238 KeV	0.1
Lead-214 @ 295.2 KeV	0.4
Lead-214 @ 352.0 KeV	0.3
Potassium-40 @ 1460 KeV	2.0
Radium-226 @ 186.0 KeV	2.1
Thallium-208 @ 583 KeV	0.1
Thallium-208 @ 860 KeV	1.2
Thorium-234 @ 63.3 KeV	2.7
Thorium-234 @ 92.6 KeV	0.9
Uranium-235 @ 143 KeV	0.5

[a]Based on a four-hour count of a 100 g sample, dry basis.

Table C-4
Comparison of EMP and LGTI HAPs Analytical Methods

Stream	Parameter	Analytical Methods	
		EMP	LGTI HAPs
Sour Syngas	C_1 - C_{10}	GC	GC-FID
	Major Gases	GC	GC-TCD
Sweet Syngas	Particulate Loading	Gravimetric	Gravimetric
	H_2S	Titration	GC-FPD
	Major Gases	GC	GC-TCD
Turbine Exhaust	Particulate Loading	Gravimetric	Gravimetric
	SO_3/H_2SO_4	Ion Chromatography	Ion Chromatography
	PM-10	Gravimetric	Gravimetric
	Major Gases (CO_2, O_2, N_2)	Orsat	CEM
Incinerator Exhaust	Particulate Loading	Gravimetric	Gravimetric
	PM-10	Gravimetric	Gravimetric
	Volatile Organics	Cryogenic Focus GC-FID/PID/HECD	GC/MS
	Semivolatile Organics	EPA Method 8270 (GC-MS)	EPA Method 8270 (GC-MS) and CARB Method 429
	Total Chromatographical Organics	Sonication, GC-FID	NS[a]
	SO_3/H_2SO_4	Ion Chromatography	Ion Chromatography
	NO_x	Ion Chromatography	CEM

[a]NS = Not sampled as part of the LGTI HAPs program.

Particulate- and Vapor-Phase Metals

The sample fractions generated by the multi-metals sampling train and an overview of the sample handling process are shown in Figures C-1, C-2, and C-3. These particulate and vapor-phase sample fractions are prepared and analyzed separately for the elements listed in Table C-1.

Particulate-Phase Metals. All filter samples are desiccated and weighed to a constant weight (defined as successive weight determinations within 0.5 mg at 6-hour intervals). For all internal gas stream samples, there are no probe and nozzle rinses. For samples collected at the turbine exhaust stack and incinerator stack, the acetone probe and nozzle rinse (PNR) is evaporated, desiccated, and also weighed to a constant weight. The nitric acid PNR is added to the solids recovered from the acetone PNR, and the volume is reduced to 10 mL by evaporation on a hot plate. This volume is quantitatively transferred, along with the filter, to a microwave-digestion vessel. The total particulate sample from the collected gas is microwave digested[1] with a mixture of hydrofluoric, hydrochloric, and nitric acids. The digestate is then analyzed for metals (except boron) by a combination of techniques including inductively coupled plasma atomic emission spectroscopy (ICP-AES)[2] and graphite furnace atomic absorption spectroscopy (GFAAS).[3,4,5,6] Mercury is determined from an aliquot of the microwave digestate by cold vapor atomic absorption spectroscopy (CVAAS).[7]

Boric acid is added to the digestate to solubilize metal fluorides that precipitate during the digestion. This addition of boric acid makes the analysis of boron in these samples impractical; however, boron was determined in the collected ash samples from the raw gas and char streams where sufficient sample material permits a separate preparation procedure for boron analysis.

Vapor-Phase Metals. The two HNO_3/H_2O_2 impinger samples were combined, digested,[8,9] and analyzed for metals by ICP-AES and GFAAS. An undigested aliquot is taken for ICP/MS[10] analysis. Another aliquot is removed for mercury analysis and the excess peroxide in the sample matrix eliminated by the addition of solid $KMnO_4$ until a pale pink color persists. The sample is digested in $KMnO_4$/ H_2SO_4 solution and analyzed for mercury by CVAAS.

The contents of the third impinger, the two $KMnO_4/H_2SO_4$ impingers, and the hydrogen chloride (HCl) impinger rinse are combined and an aliquot is digested in $KMnO_4$/ H_2SO_4 solution and analyzed for mercury by CVAAS.

The direct analysis of vapor-phase metals by Radian's vapor-phase trace element atomic absorption spectrophotometer (VPTE-AAS) and the analysis of the charcoal sorbent for metals are described in special techniques later in this section.

Anions

A description of the sampling train and sample fraction recovery for the Method 5 anions sampling train is presented in Table B-4. The sample fractions generated by the anions/acid gas sampling train and an overview of the sample handling process are shown in Figures C-4 and

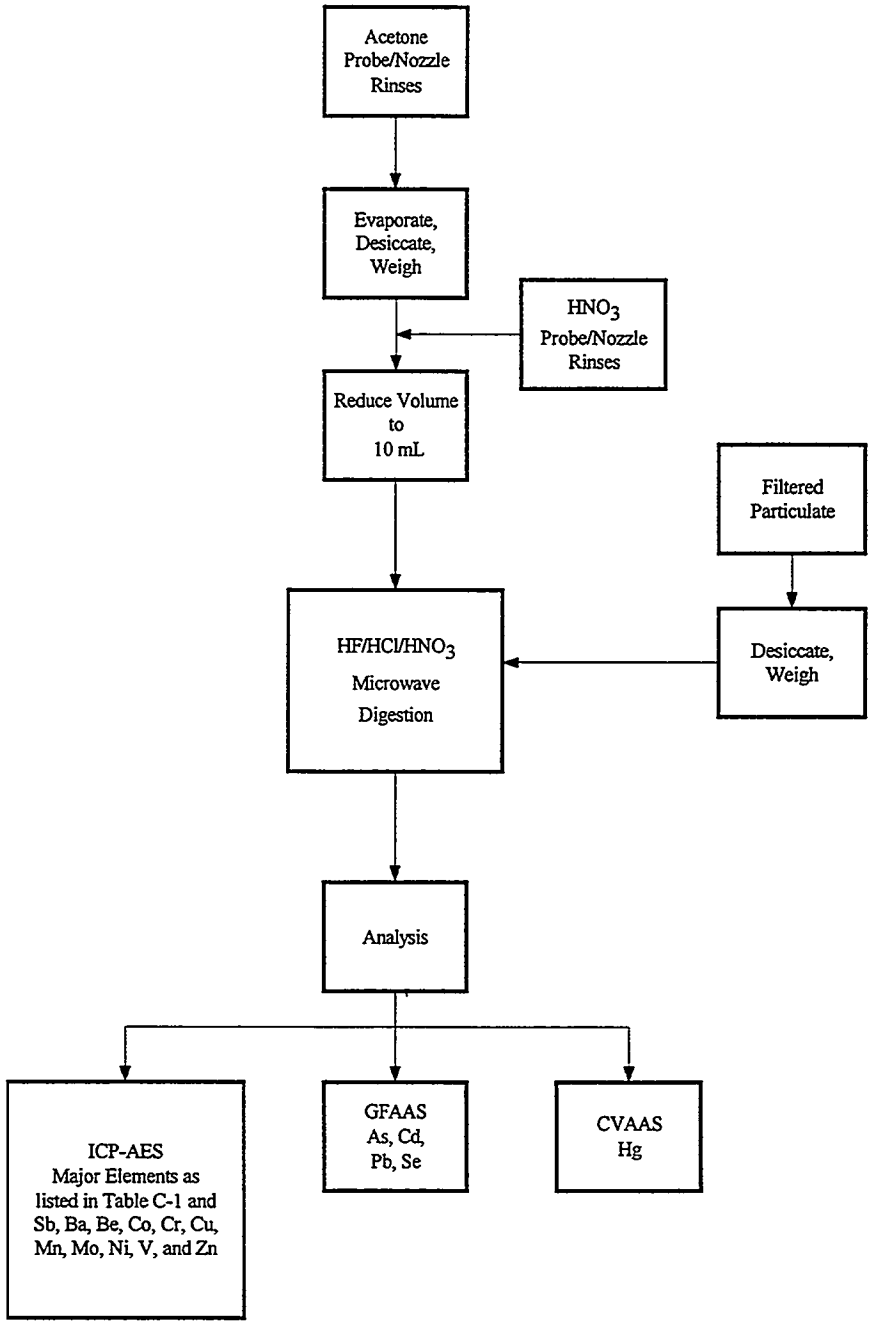

Figure C-1
Gas Particulate Sample Preparation and Analytical Plan for Metals

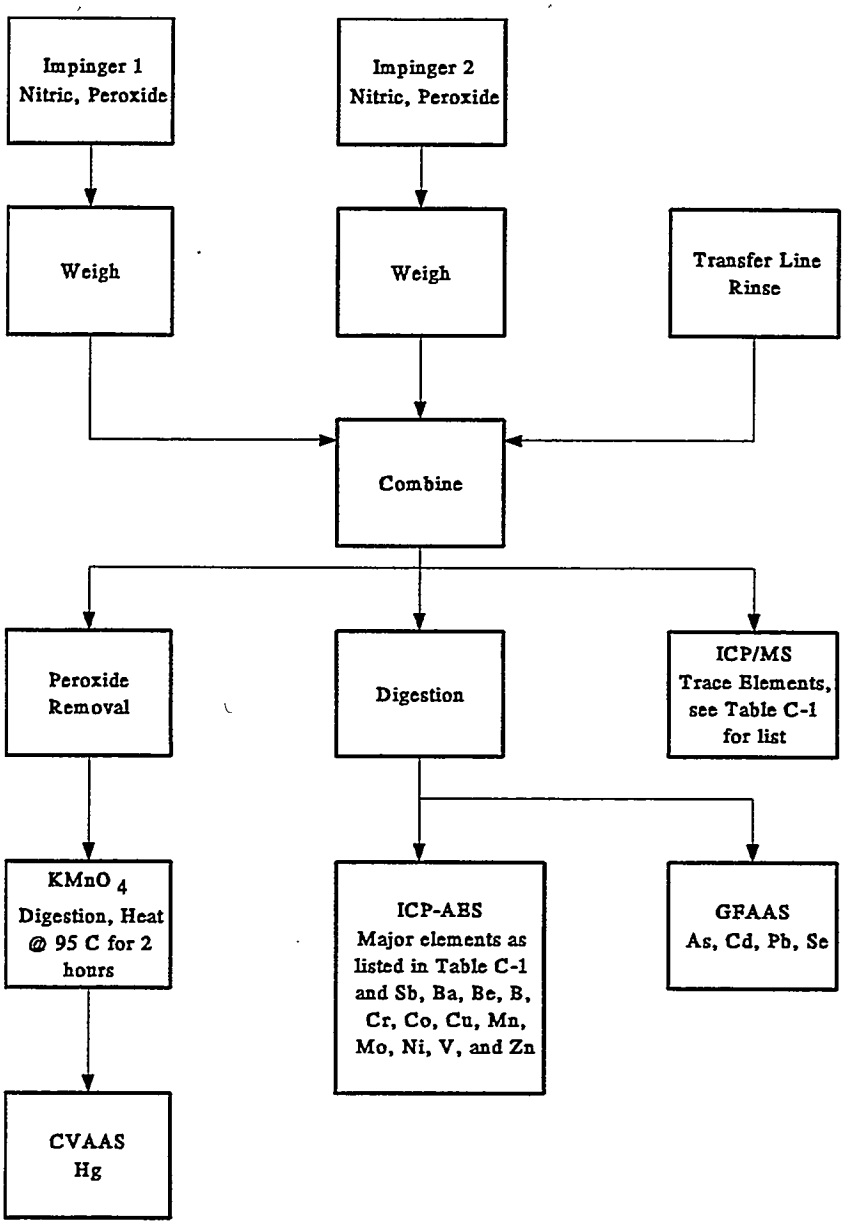

Figure C-2
Gas Impinger Sample Preparation and Analytical Plan for Metals

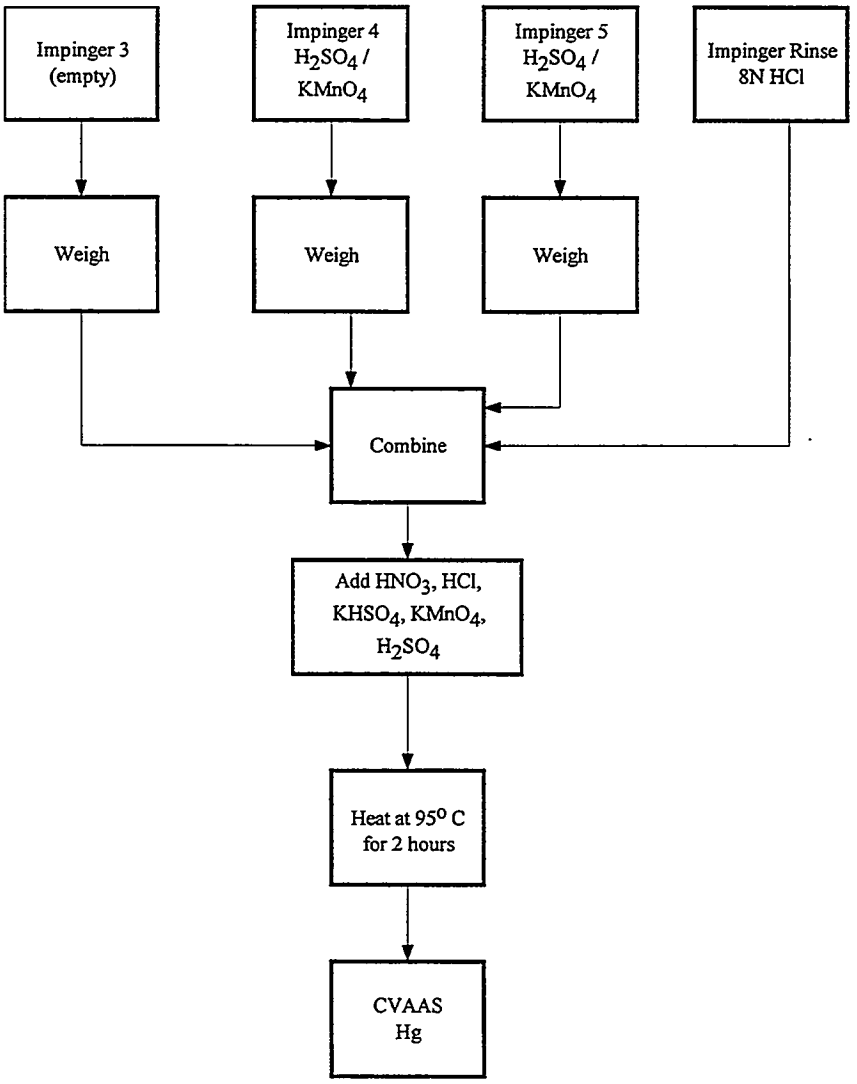

Figure C-3
Gas Impinger Sample Preparation and Analytical Plan for Mercury

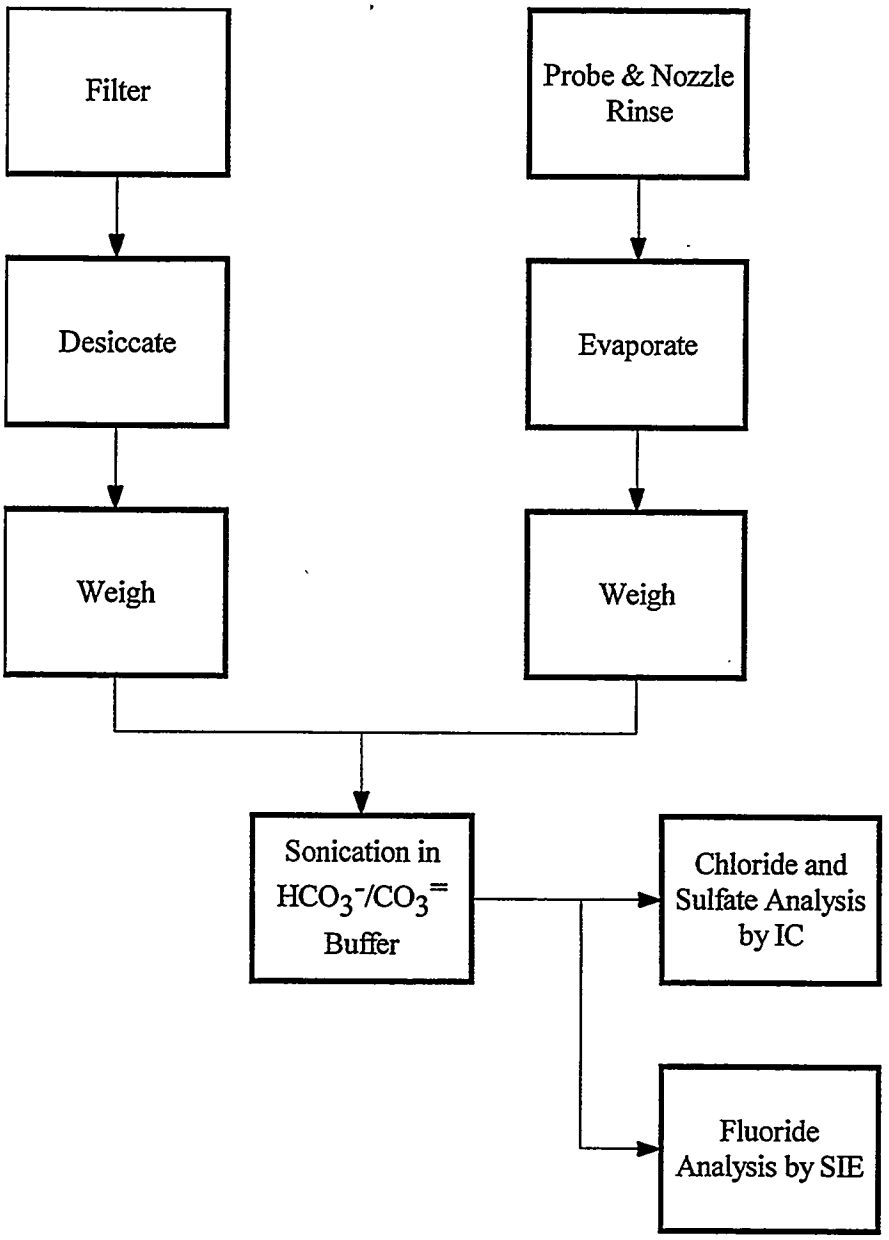

Figure C-4
Gas Particulate Sample Preparation and Analytical Plan for Anions

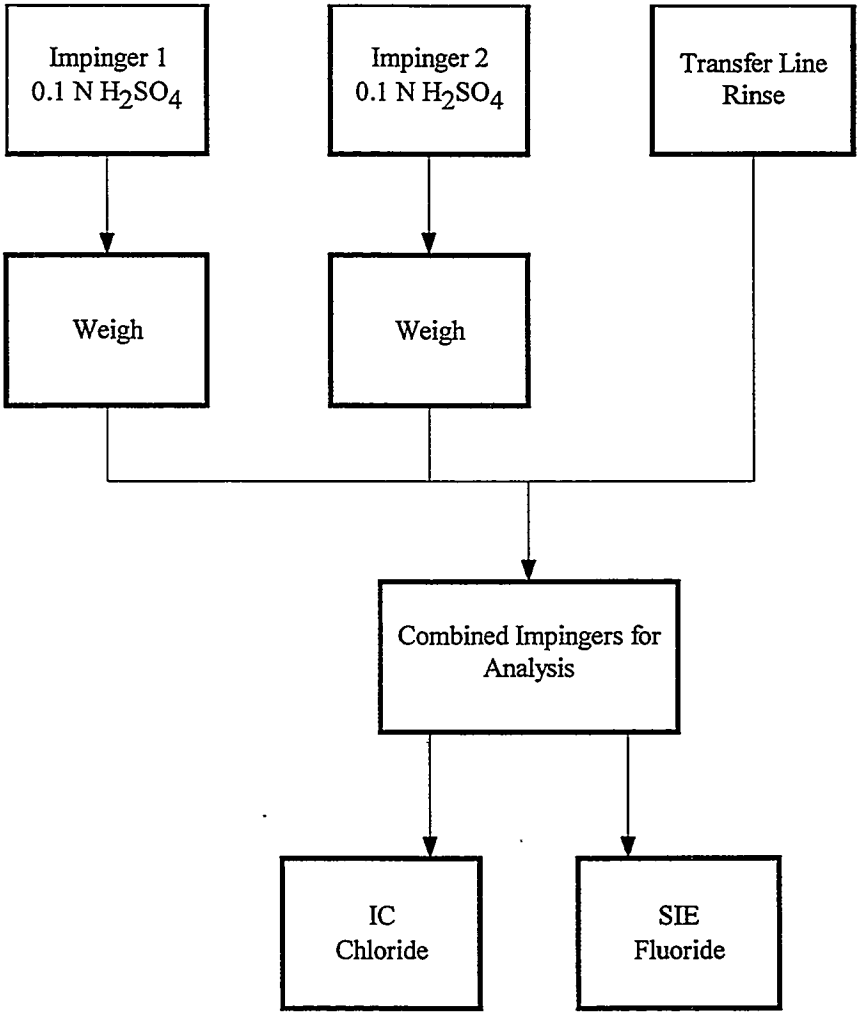

Figure C-5
Gas Impinger Sample Preparation and Analytical Plan for Anions

C-5. The particulate and vapor phases are prepared and analyzed separately for chloride and fluoride.

Particulate-Phase Anions. All filters are desiccated and weighed prior to extraction. For samples collected with probe and nozzle rinses (turbine exhaust stack and incinerator stack), the PNR sample is evaporated, desiccated, and weighed before being combined with the filter sample for extraction. The particulate matter is then sonicated with 100 mL of fresh carbonate/bicarbonate solution and analyzed for chloride and sulfate by ion chromatography (IC)[11] and for fluoride by specific ion electrode (SIE).[12]

Vapor-Phase Anions. The impinger solutions received from the test site are sent directly to the analytical laboratory for chloride analysis by IC, and fluoride analysis by SIE. Sulfate analysis of the IPA and peroxide impingers collected at the emission sources by EPA Method 8 were analyzed by IC.

Ammonia/Hydrogen Cyanide

A description of the sampling train and sample fraction recovery for the combined ammonia/ hydrogen cyanide trains is presented in Table B-4. This combined sampling train is applicable only to the two emissions gas streams. Ammonia and cyanide collection trains are operated independently for most internal gas streams. For all gas stream samples, the sample fractions generated by the ammonia sampling train are sent directly to the laboratory for analysis as shown in Figure C-6. The sulfuric acid impinger solutions (0.1N and 1% H_2SO_4) are prepared for analysis by distillation according to EPA Method 350.2,[13] and the recovered distillates are analyzed by EPA 350.1,[14] an automated colorimetric method. All cyanide impinger samples (2% zinc acetate) are digested and analyzed according to EPA Method 9012.[15] A description of the cyanide collection and analysis method for internal gas streams is presented in the special techniques section.

Aldehydes

A description of the sampling train and sample fraction recovery for the aldehydes sampling train is presented in Table B-5. The sample fractions generated by the aldehydes sampling train and an overview of the sample handling process are shown in Figure C-7. The aqueous and methylene chloride layers of the sample are separated, and the aqueous fraction is then extracted with fresh methylene chloride. The methylene chloride portion of the sample and the aqueous extract are then combined. For process stream samples where low levels of aldehydes are expected, an aliquot of this extract may be concentrated during a solvent exchange procedure into acetonitrile. The resulting extract is then analyzed by high performance liquid chromatography (HPLC) for acetaldehyde, benzaldehyde, formaldehyde, and acrolein according to EPA Method 0011A.[16] Air Toxics, Ltd. was subcontracted to perform these analyses.

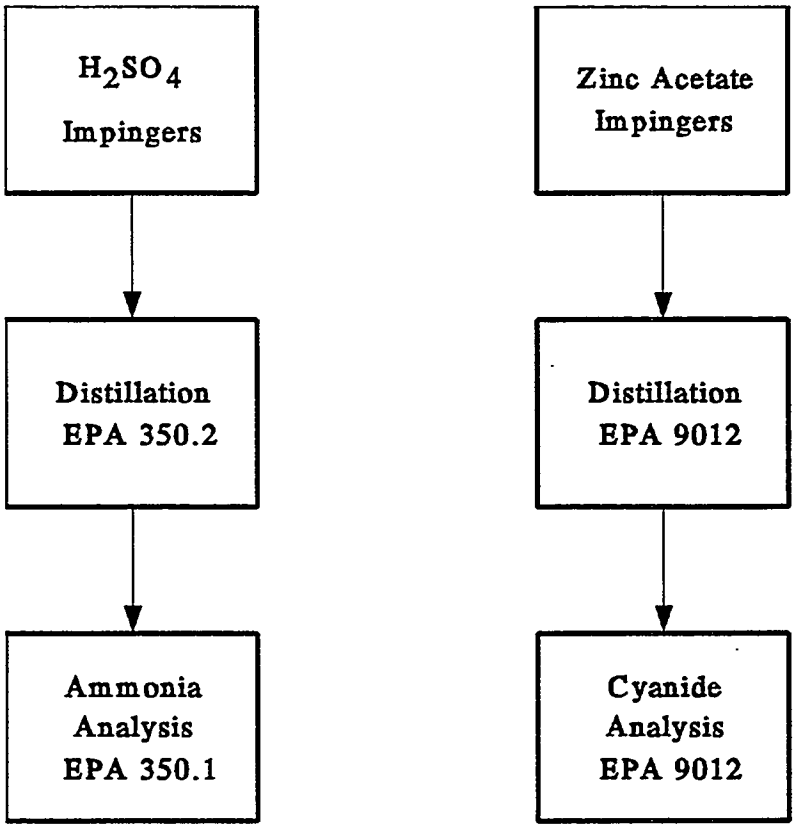

Figure C-6
Gas Impinger Sample Preparation and Analytical Plan for Ammonia and Cyanide

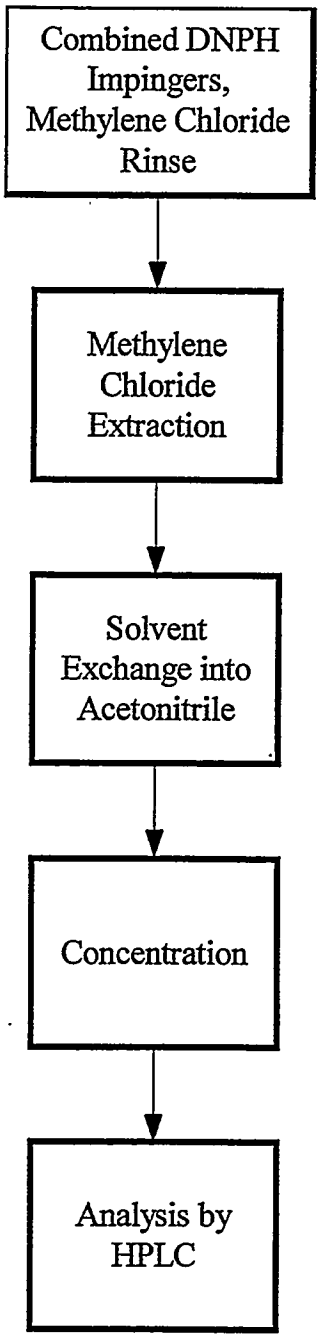

Figure C-7
Gas Impinger Sample Preparation and Analytical Plan for Aldehydes

Volatile Organics

The sample fractions generated by the VOST and an overview of the sample handling process are shown in Figure C-8. Volatile organic compound analysis is performed on the Tenax and Tenax/charcoal cartridges returned directly from the test site to the analytical laboratory. The contents of the Tenax and Tenax/charcoal cartridges are spiked with internal standards and surrogates and then thermally desorbed according to EPA Method 5040[17] and directly analyzed for the compounds listed in Table C-2 by GC/MS according to EPA Method 8240.[18] Air Toxics, Ltd. was subcontracted to perform VOST analyses.

Field blanks are performed during each run at each location. This is to account for the high probability of contamination from methylene chloride and acetone which are commonly used in the recovery of other sampling trains. The internal process streams were analyzed directly for benzene, toluene, and xylene by gas chromatography with a flame ionization detector (GC-FID). This procedure is explained further in the special techniques section.

Semivolatile Compounds and Polycyclic Aromatic Hydrocarbons (PAHs)

A description of the sampling train and sample fraction recovery for the MM5 sampling train is presented in Table B-6. The sample fractions generated by the MM5 sampling train and an overview of the sample handling process are shown in Figure C-9. The particulate-phase and vapor-phase sample fractions are analyzed separately for the semivolatile organic compounds and PAHs presented in Table C-2. The sample extracts are split to provide analysis of the particulate-phase and vapor-phase samples by SW-8270.[19] The turbine stack and incinerator stack gas samples will be analyzed by both SW-8270 and CARB Method 429[20] protocols.

The particulate phase consists of the particulate filter and front half acetone/methylene chloride probe and nozzle rinses (where applicable). The vapor phase consists of the back half acetone/ methylene chloride rinse, the XAD resin, and the impinger condensate. The acetone/methylene chloride PNR fraction, the filter, and the XAD fractions are soxhlet-extracted with methylene chloride. The impinger condensate fraction is liquid-liquid extracted with methylene chloride. The XAD extract and the impinger condensate extract are then combined, concentrated to 1 mL and analyzed by gas chromatography/mass spectrometry (GC/MS) according to EPA Method 8270, and by high resolution GC/MS according to CARB Method 429. Triangle Laboratories, Inc. was subcontracted to perform these analyses.

Solid Streams

There are six solid process streams identified for sampling and analysis: coal, slurry (two streams), recycled scrubber solids (char), slag, and sulfur byproduct. The sample preparation and analytical approach for each of these streams is presented in this section.

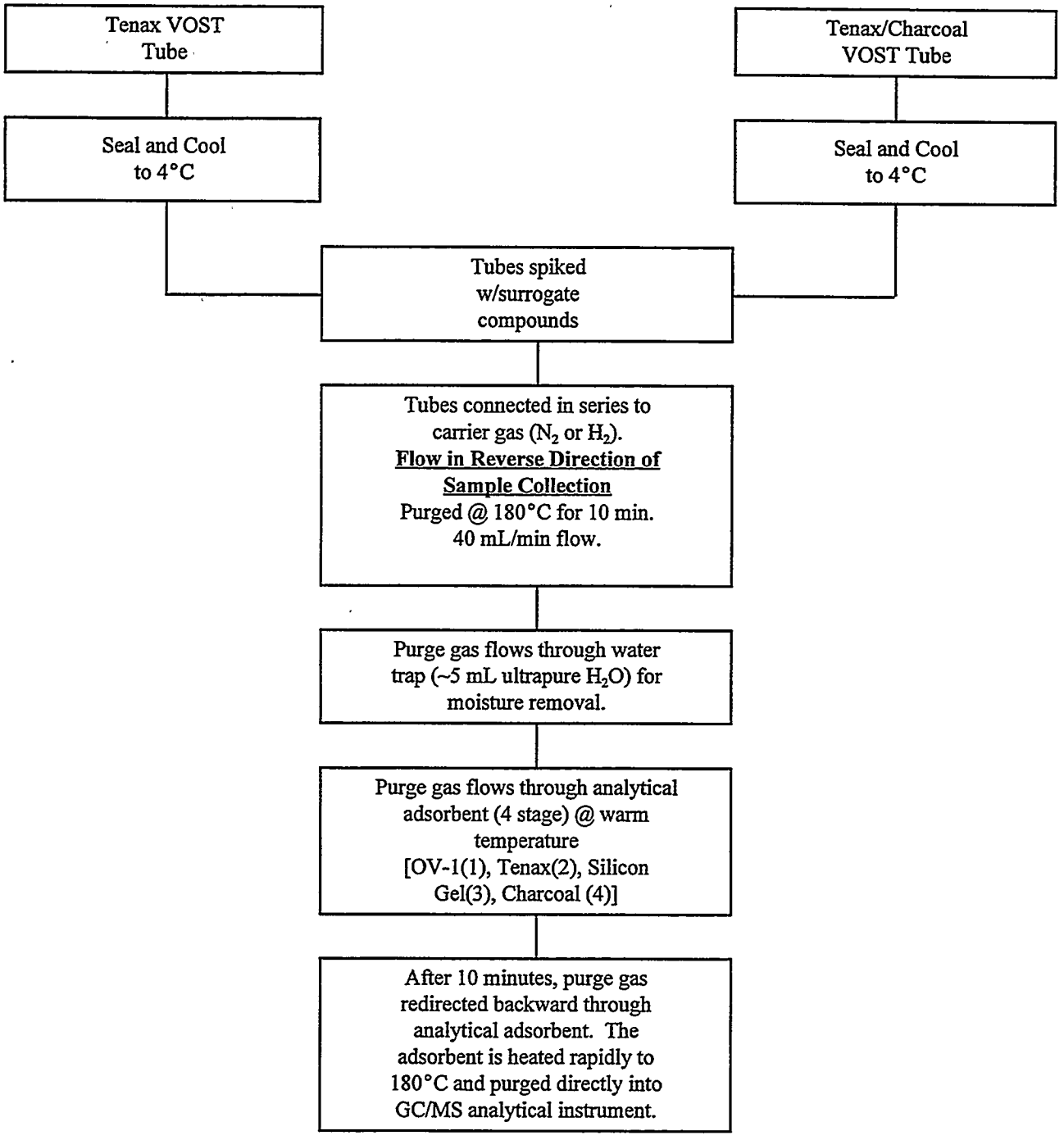

Figure C-8
VOST Sorbent Sample Preparation and Analytical Plan for Volatile Organic Compounds

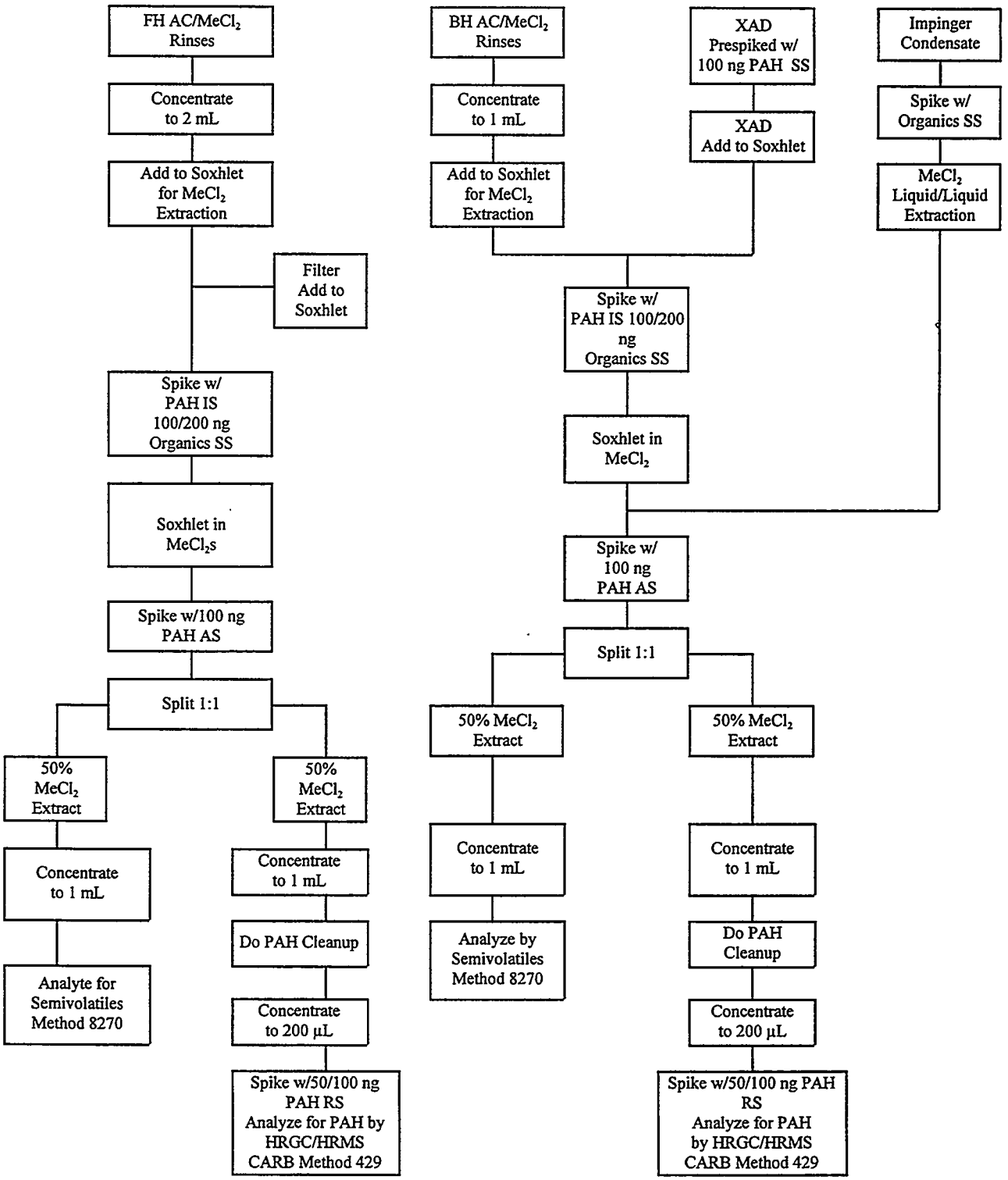

Figure C-9
Gas Sample Preparation and Analytical Plan for Semivolatile Organic Compounds and PAHs

Coal, Slurry, and Char

Each coal/slurry composite is thoroughly mixed and subsampled for drying. Weights are obtained on one subsample of the coal/slurry before and after drying at 104°C to determine the weight percent solids. A second subsample is air dried, ground to -60 mesh, and sealed in plastic bags for the analyses shown in Figure C-10. All results are reported on a dry coal basis. Char samples were also prepared and analyzed according to the plan shown in Figure C-10 and results were reported on a dry basis. Commercial Testing and Engineering Company was subcontracted to perform these analyses.

Metals. Coal and char samples were prepared for metals analysis by a variety of techniques. Samples were prepared according to ASTM D3683[21] for trace and minor elements. This method requires ashing and digesting the sample with mixed acids. Boron analysis was performed on the coal and char samples by ICP-AES after fusion with sodium carbonate. Mercury was determined by combusting a sample and trapping the mercury vapors using a double gold amalgamation technique. The amalgamated mercury is thermally desorbed and analyzed by cold vapor atomic absorption spectrophotometry (DGA-CVAA).[22] Major ash minerals were determined by X-ray fluorescence (XRF) according to ASTM Method D4326.[23]

Due to the low levels of trace elements expected in this coal matrix, an additional method was used to determine selected trace elements in the coal and char samples. A modification of ASTM D3683 was performed to provide a nitric acid matrix suitable for ICP/MS analysis. In place of the open vessel digestion specified in ASTM D3683, a mixed acid microwave digestion was performed on the ashed sample in a closed vessel to prevent loss of volatile elements. The digestate was brought to near dryness on a hot plate at low temperature. The residue was then re-digested with nitric acid to provide the sample for ICP/MS analysis. This preparation procedure eliminates the high chloride and fluoride concentrations in the analytical matrix and reduces mass spectral interferences.

Anions. Chlorine and fluorine in the dried coal slurry and char were determined by ASTM D4208[24] and D3761,[25] respectively. Prepared samples were combusted in a closed oxygen combustion bomb containing a dilute basic solution. The bomb washings were analyzed by SIE and/or IC.

Ultimate, Proximate, and Higher Heating Value. In conjunction with the other analyses, higher heating value (HHV), proximate (intrinsic moisture, volatile and fixed carbon, and ash), and ultimate (percent carbon, hydrogen, nitrogen, sulfur, oxygen, and ash) analyses were performed according to the ASTM procedures[26,27,28] listed in Table C-5.

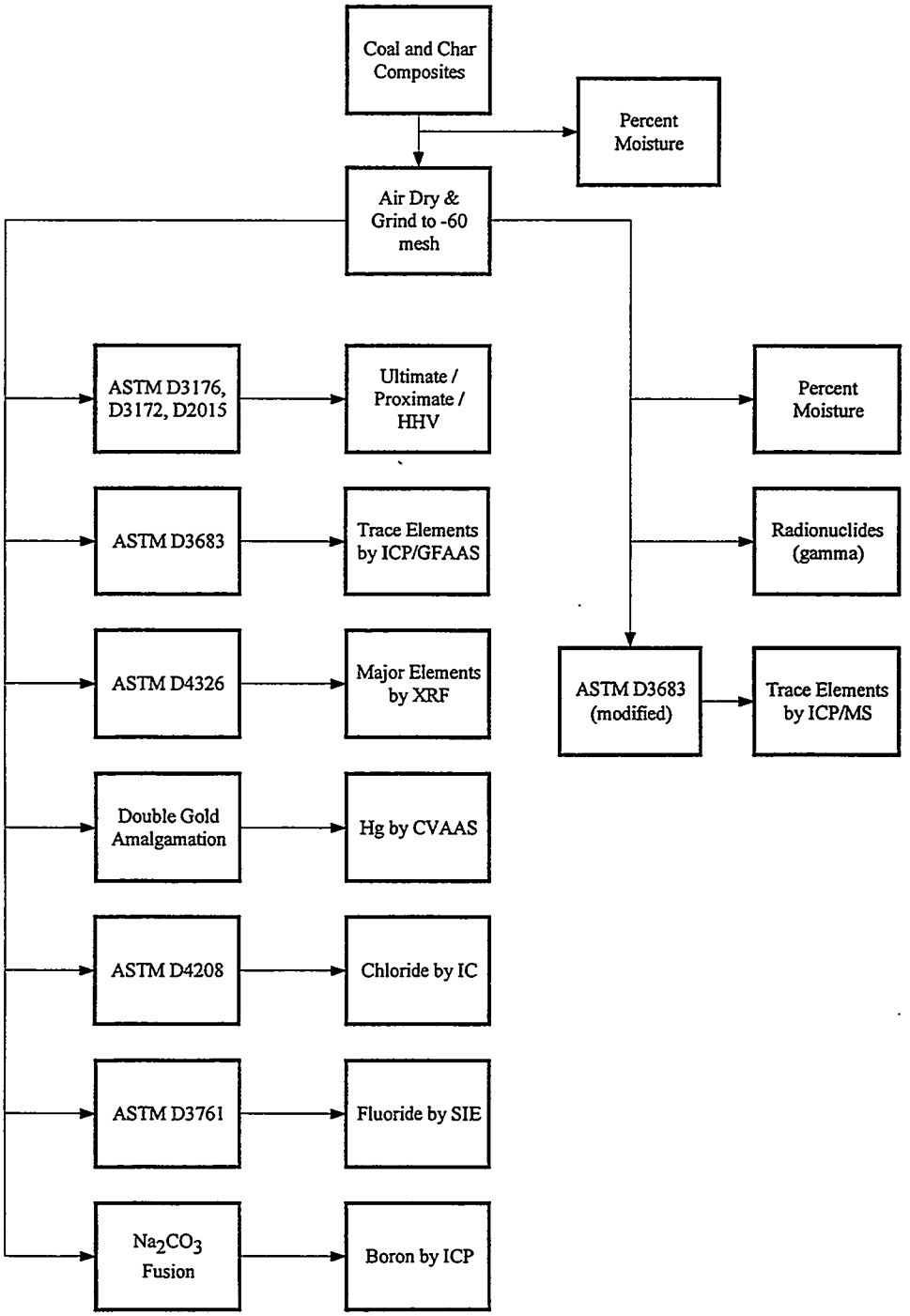

Figure C-10
Coal and Char Sample Preparation and Analytical Plan

Table C-5
Summary of Ultimate/Proximate Analytical Methods

Analyte	Analytical Method
Moisture, total	ASTM D3302
Ash	ASTM D3174
Carbon, Hydrogen, Nitrogen	ASTM D5373
Sulfur	ASTM D4239
Volatile Matter	ASTM D3175
Fixed Carbon	ASTM D3172
Heating Value	ASTM D2015

ASTM = American Society for Testing and Materials, *Annual Book of ASTM Standards*, Vol. 05.05.

Radionuclides. Coal samples were analyzed by EPA Method 901.1.[29] This method uses gamma emitting spectrometry to measure radioactivity through gamma decay.

Slag

Figure C-11 presents the sampling handling and preparation procedures for slag sample analysis. Commercial Testing and Engineering Company was subcontracted to perform these analyses.

Metals. Slag samples were air dried and ground to pass a 60-mesh sieve prior to taking aliquots for analysis. Sample preparation and analysis for metals followed the same procedures described for coal and char samples in the preceding section.

Anions. Separate preparatory techniques were necessary for the analysis of fluoride, chloride, and sulfur in slag. All sample aliquots were taken from the ground, air-dried material prepared for trace element analysis. Fluoride sample aliquots were prepared by fusion of the slag with sodium hydroxide (McQuaker-Gurney).[30] The fusion melt is dissolved in hydrochloric acid and analyzed potentiometrically by fluoride-specific ion electrode. Slag samples for chloride analysis were prepared by mild digestion in nitric acid. The digestate was analyzed potentiometrically by chloride-specific ion electrode.

Ultimate Analysis. An ultimate analysis (percent carbon, hydrogen, nitrogen, sulfur, oxygen, and ash) was performed on slag samples according to the ASTM procedures listed in Table C-5.

Radionuclides. Slag samples are analyzed by EPA Method 901.1. This method uses gamma emitting spectrometry to measure radioactivity through gamma decay.

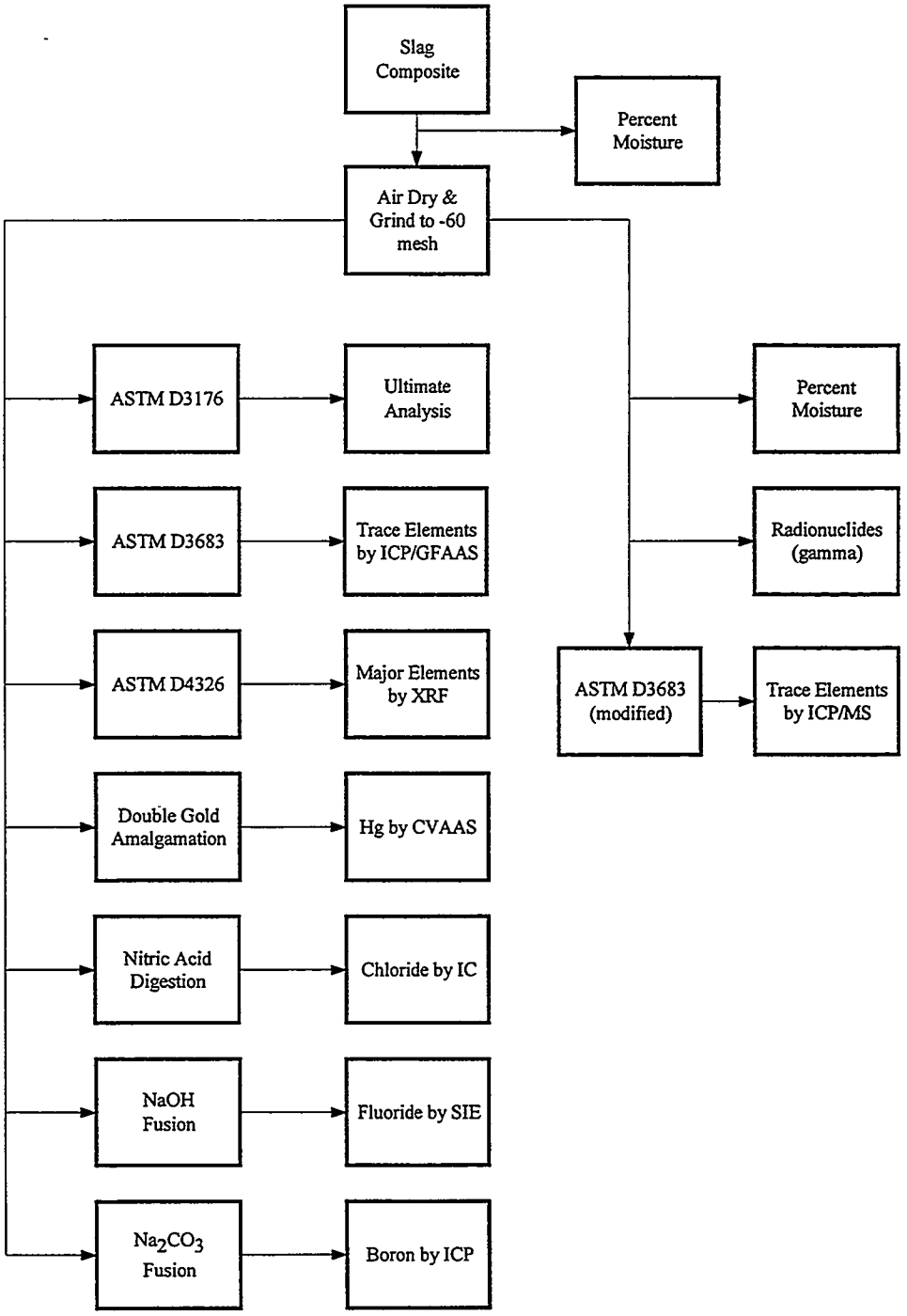

Figure C-11
Slag Sample Preparation and Analytical Plan

Sulfur Byproduct

The sulfur sample is analyzed for metals, ash, and percent sulfur as shown in Figure C-12.

Process Waters

Plant process waters sampled for analysis include the sour condensate, sweet water, and scrubber inlet and blowdown streams. The daily composite samples were preserved on site in accordance with the EPA protocols listed in Table C-6. Figure C-13 illustrates the process water sample preparation and analytical procedures.

Metals

The unfiltered water samples were prepared for total metal analysis according to EPA Methods 3005 and 3020. The samples were vigorously digested in concentrated nitric acid to dissolve any suspended material that may be present. The digestates were diluted to a known volume and analyzed by ICP-AES and GFAAS. Mercury was determined by EPA Method 7470.

Anions

Samples for the analysis of anions (chloride, fluoride, sulfate, phosphate, and formate) were filtered before analysis. Chloride, sulfate, and formate were determined by IC according to EPA Method 300.0. Fluoride was determined potentiometrically by fluoride SIE. Phosphate was determined spectrophotometrically as a measure of total phosphorus after the sample was digested according to EPA Method 365.1.[31]

Ammonia, Phenol, and Chemical Oxygen Demand (COD)

Process water samples collected for ammonia, phenol, and COD were split for each analysis. Ammonia fractions were prepared for analysis by distillation according to EPA Method 350.2, and the recovered distillates analyzed by EPA 350.1, an automated colorimetric method.

Aliquots for phenol analysis were prepared and analyzed by EPA Method 420.1,[32] and COD analysis performed by EPA Method 410.1.[33]

Cyanide and Thiocyanate

Total cyanide, free (amenable) cyanide, and thiocyanate process water samples were prepared and analyzed by EPA Methods 335.2,[34] 335.1,[35] and Standard Method 412K,[36] respectively.

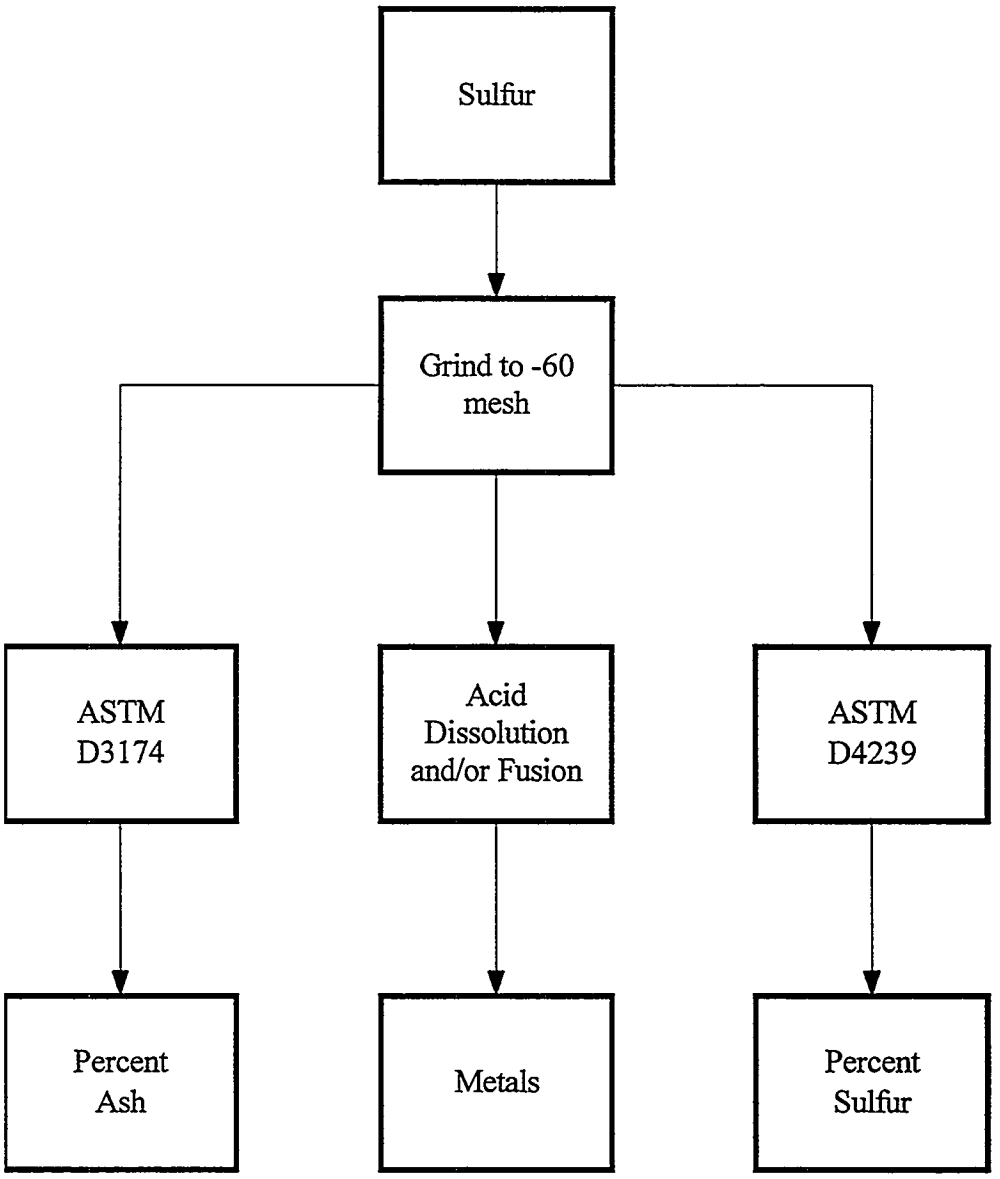

Figure C-12
Sulfur Sample Preparation and Analytical Plan

Table C-6
Aqueous Sample Preservation Requirements and Hold Times

Container[a]	Parameter	Preservation Technique	Holding Time[b]
1,000 p	pH	Cool 4°C	Immediately
	Conductivity		28
	TSS		7
500 p	Chloride	Cool 4°C	28
	Fluoride		28
	Formate		14
	Phosphate		28
	Sulfate		28
125 p	Sulfide	50 mL SAOB, Cool 4°C	7
1,000 p	Total Cyanide	$PbCO_3$, filter, pH>12 with lime	14
1,000 p	Free Cyanide	As above	14
250 p	Thiocyanate	As above	28
1,000 g	COD	pH <2 with H_2SO_4, Cool 4°C	28
	Phenol		28
	Ammonia		28
500 p	Metals, Total	pH <2 with HNO_3	180[c]
1,000 g	SVOCs[d]	Cool 4°C	7/40[e]
4x40 g[f]	VOA, purgeable	Cool 4°C	7 w/o HCl

[a]Container size provides adequate sample for all analysis listed in the group. Number specifics volume in mL, while letter specifics polypropylene (p) or glass (g) container.

[b]Holding times in days from SW-846, 1986.

[c]Holding time is 28 days for mercury, and 180 days for all other metals.

[d]See Table C-2 for SVOCs.

[e]Holding time for sample before/after extraction.

[f]Four, 40 mL vials.

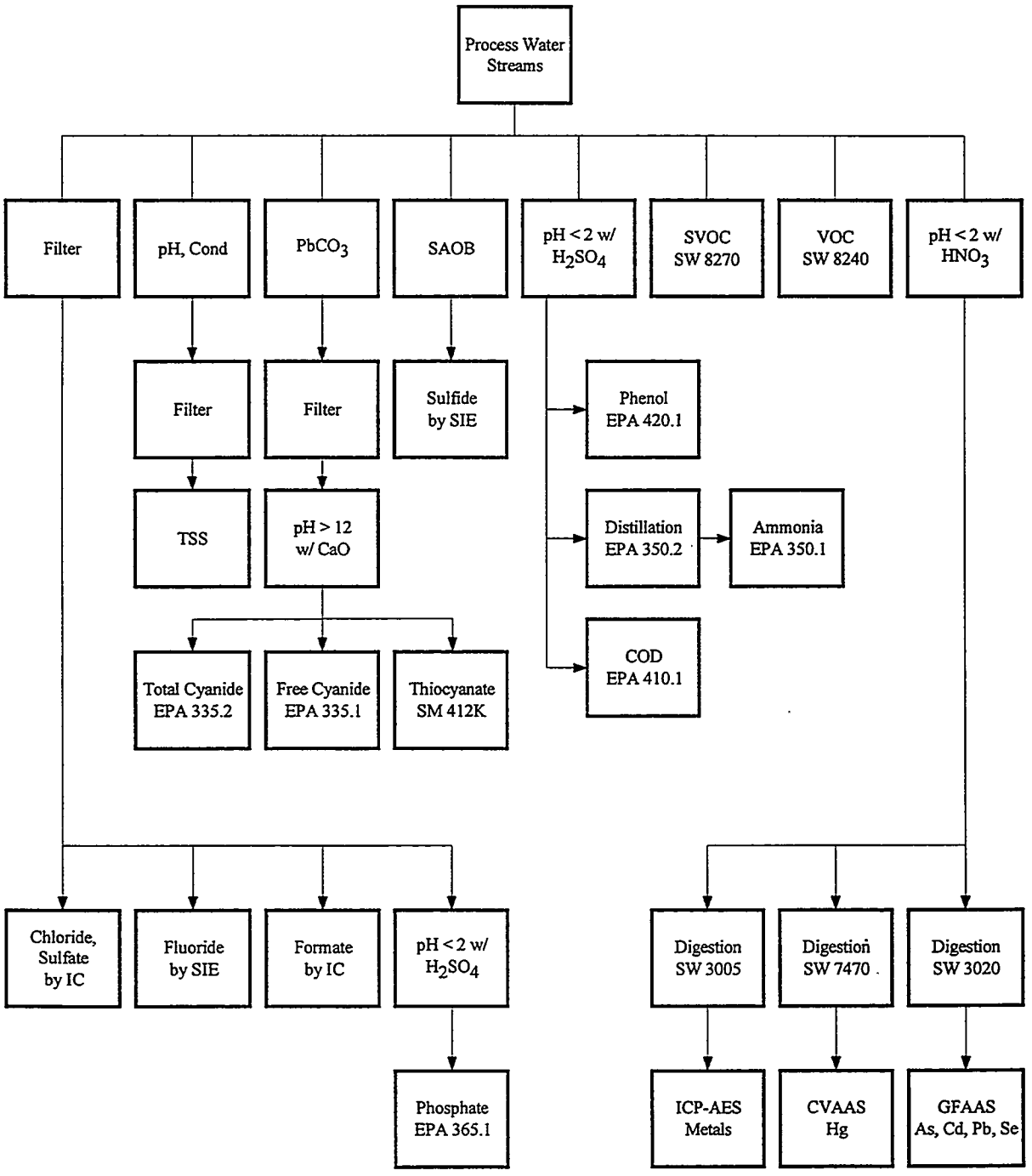

Figure C-13
Process Water Sample Preparation and Analytical Plan

Sulfide

Sulfide samples were analyzed on-site to prevent sample degradation from sulfide oxidation. Samples were preserved with sulfide anti-oxidant buffer (SAOB) and titrated with a standard lead perchlorate solution to a potentiometric endpoint using a sulfide-specific electrode.[37]

Water Quality Parameters (pH/Conductivity/Total Suspended Solids)

Process water samples were analyzed on-site for hydrogen ion concentration (pH)[38] and specific conductivity[39] as a measure of total dissolved solids. Total suspended solids (TSS) were measured by filtration and gravimetric determination of the filtered solids mass.[40]

Volatile Organic Compounds

Purgeable volatile organic compounds were determined in process water samples by Method SW-8240, purge and trap GC-MS. The samples were not preserved with hydrochloric acid to a pH<2 because the predominance of carbonates, typically found in these process waters, generates carbon dioxide bubbles in the vial. These bubbles may cause overpressurization of the sample vial or VOC losses in the resulting head space. The holding time for unpreserved samples is one week.

Aldehydes

Process water samples were prepared for analysis of acetaldehyde, benzaldehyde, formaldehyde, and acrolein by proposed Method SW-8315.[41] In this procedure, aldehydes present in the water samples are derivatized with 2,4-dinitrophenyl hydrazine (DNPH) and extracted in methylene chloride. The extracts are concentrated during solvent exchange into acetonitrile before HPLC analysis. Air Toxics, Ltd. was subcontracted to perform this analysis.

Semivolatile Organic Compounds

Liquid samples for semivolatile organic compound analyses are serially extracted using a separatory funnel with methylene chloride by EPA Method 3510.[42] The extracts are then analyzed by gas chromatography/mass spectrometry according to EPA Method 8270.

Selectamine™ Solvent

The Selectamine™ solvent samples were evaluated for ash, solids, and heat stable salts as an indication of substance accumulation that may impact material balance closures around the sulfur removal process.

Special Techniques

Some of the techniques recommended for sample analysis have not been promulgated as standard analytical protocols, although they are based on established principles of quantitative analysis. This section identifies and describes the alternative techniques and nonstandard methods recommended for selected process sample analysis.

Vapor-Phase Trace Elements by Atomic Absorption Spectrophotometry

Radian Corporation has successfully developed and demonstrated an on-line analysis technique for vapor-phase trace elements using an atomic absorption spectrophotometer (AAS). The AAS is modified to accept a syngas sample stream as part of the fuel supply going to the nebulizer mixing chamber and flame. In the flame, vapor-phase trace elements are atomized and absorb light energy from an element-specific light source just like aqueous samples in conventional AAS. The sample gas, fuel gas, and air supplies are regulated and monitored to determine the syngas component going to the flame, and ultimately the elemental concentration in the gas sample stream. Absorbance and concentration are related by Beer's law and gas concentrations are determined by comparison with standard curves generated from aqueous standards.

The following trace elements were identified for analysis by this technique:

- Arsenic;
- Cadmium;
- Nickel;
- Chromium;
- Lead;
- Selenium; and
- Zinc.

The only exception to the flame AAS analysis is mercury. Mercury was analyzed by adapting the AAS with a flow cell designed for cold vapor analysis. Mercury analysis was discussed in detail in Section 8 of this report.

Vapor-Phase Trace Elements by Charcoal Adsorption

Charcoal sorbents have been used in a number of industrial processes as guard beds to protect catalysts from metal poisoning. The same principle has been applied successfully by Radian Corporation to collect and measure selected vapor-phase trace elements in syngas samples. The

charcoal sorbent is rigorously cleaned with concentrated nitric acid before being rinsed, dried, and loaded into quartz sampling tubes. After sampling, the charcoal sorbent is recovered and digested with nitric acid in a closed vessel for ICP-AES, GFAAS,[43] and CVAAS analysis. Table C-7 lists the trace elements targeted for analysis, the analytical techniques applicable, and their respective detection limits.

Gas Chromatography

Table C-8 summarizes the on-site gas chromatographic analyses performed on selected sample streams. On-site analysis normally provides more accurate and representative process data by minimizing sample degradation and reaction time.

Analytical Subcontractors

Analytical subcontractors were selected to perform a number of specialized analytical techniques. Table C-9 summarizes the role of each subcontract laboratory and identifies the primary contact at each laboratory.

Table C-7
Trace Element Analysis of Charcoal Sorbents

Element	Analysis Method	Detection Limit ($\mu g/Nm^3$)[a]
Antimony	GFAA	1.0
Arsenic	GFAA	1.0
Barium	ICP-AES	0.6
Beryllium	ICP-AES	0.6
Cadmium	GFAA	3.0
Chromium	ICP-AES	3.0
Cobalt	ICP-AES	5.0
Copper	ICP-AES	3.0
Iron	ICP-AES	300
Lead	GFAA	0.8
Manganese	ICP-AES	0.1
Mercury	CVAA	0.05
Molybdenum	ICP-AES	3.0
Nickel	ICP-AES	11
Selenium	GFAA	1.0
Vanadium	ICP-AES	4.0
Zinc	ICP-AES	3.0

[a]DL based on 100 liter (0.1 Nm^3) gas sample.

Table C-8
Gas Chromatography Analysis Summary

Gas Components	Gas Streams	Units	Column	Detector
Major Gases (H_2, CO, CO_2, N_2, Ar)	All	mol. %	Carboxen-1000	TCD
Hydrocarbons (C_1 - C_8)	Internal only	ppmv	SP-1000	FID
Reduced Sulfur Species (H_2S, COS, CS_2)	Internal only	ppmv	Supelpak-S	FPD
Benzene, Toluene, Xylene	Internal only	ppmv	SP-1000	FID

Table C-9
Subcontract Analytical Laboratories

Laboratory	Shipping Address	Primary Contact	Analytical Service
Harvard University Department of Earth and Planetary Sciences	20 Oxford Street Cambridge, MA 02138	Dr. Ron Pflaum Phone: 617/496-8021 Fax: 617/495-8839	ICP/MS Analysis
Triangle Laboratories	801-10 Capitola Drive Durham, NC 27713	Dr. Hani Karam Phone: 919/544-5835 Fax: 919/544-5491	HRGC/MS
Air Toxics, Ltd.	180 Blue Ravine Road Suite B Folsom, CA 95630	Alexis Meredith Phone: 916/985-1000 Fax: 916/985-1020	GC/MS, HPLC
Commercial Testing & Engineering Company	4665 Paris Street Suite B-200 Denver, CO 80239	Byron Caton Phone: 303/373-4772 Fax: 303/373-4791	Solids Analysis
Accu-Labs Research, Inc.	4663 Table Mountain Dr. Golden, CO 80403	Bud Summers Phone: 303/277-9514 Fax: 303/277-9512	Radiochemical Analysis