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May 27, 1999

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Attn: Electric Utility Steam Generating unit Mercury Test Program

Dear Sir or Madam:

New Century Energies (NCE) hereby is transmitting two packages of information to you concerning the above referenced test program.

The first package includes three copies of the test report for source emissions testing conducted in October 1998, on Comanche Station Unit No. 2. Unit 2 is one of the two units owned and operated by NCE at the Comanche Station located in Pueblo Colorado; and was identified by the EPA as subject to the Information Collection Request (ICR) pertaining to mercury emissions. Through our consultant - Armstrong Durham Associates (ADA), we have previously obtained your concurrence that this report was suitable for submission in lieu of conducting new testing on one of the units at Comanche Station. ADA prepared the test report per your instructions to them and in compliance with the specifications of the ICR.

The second package includes three copies of a Site Specific Sampling and Analytical test plan and three copies of a Quality Assurance Plan for new source emissions testing to be conducted on NCE's Valmont Station Unit No. 5. The EPA also identified this unit as subject to a test requirement under the mercury ICR. These documents were prepared by, and Energy and Environmental Research Center (EERC), under contract to NCE, will conduct the emissions testing.

Please contact the undersigned, at 303.571.7047, with any questions concerning this matter.

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file

**Mercury Emission Test Report at  
Public Service Company of Colorado's  
Comanche Station – Unit #2**

Prepared for:

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For Submittal To:

Emission Measurement Center (MD-19)  
U.S. Environmental Protection Agency  
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## 1.0 Introduction

### 1.1 Summary of Test Program

Comanche Station Unit #2 has been chosen to provide speciated mercury emission data per the Environmental Protection Agency (EPA) Information Collection Request (ICR) under authority of Section 114 of the Clean Air Act. This information will assist the Administrator of the EPA in determining whether it is appropriate and necessary to regulate emissions of hazardous air pollutants (HAPs), specifically mercury, from the electric utility industry. Since speciated mercury testing has recently been conducted at Comanche Unit #2, Public Service Company of Colorado (PSCo) has contracted ADA Technologies to report the results of the mercury testing in the EPA requested format.

Comanche is an electric generating station that produces electricity from two coal-fired boilers. The boilers are designated Units 1 and 2. Testing at Comanche Unit #2 for mercury speciation quantification in the combustion flue gas stream was conducted on October 12-16, 1998. At that time, Unit #2 was firing its normal fuel during this October testing, Powder River Basin (PRB) coal from the South Belle Ayr Mine near Gillette, Wyoming. The unit was operating at full load, approximately 355 MWe (gross). Standard operating processes incorporate several air pollution control technologies that are used to reduce various emissions. During the combustion process which takes place in the furnace, over-fire air ports are used to reduce NOx emissions in the combustion flue gas stream. In order to minimize sulfur emissions, a low sulfur coal is burned. Additionally, each unit (Comanche Unit #1 and #2) exhausts through its own fabric filter dust collector (baghouse) to control particulate emissions.

The purpose of the October 1998 testing was to quantify mercury speciation concentrations within the flue gas stream prior to and after the particulate control device. To compile the required data, simultaneous measurements of mercury in the flue gas were conducted at the inlet and outlet of the reverse gas baghouse for Unit #2. During testing, coal and ash samples were also collected in order to perform a mercury mass balance around the unit. Coal samples were collected from the feeders of each pulverizer during the stack testing. Coal flyash was collected from the baghouse hoppers after testing. This testing was performed as part of a DOE/FETC program to investigate the use of carbonaceous sorbents for mercury removal in the combustion flue gas stream of coal-fired power plants. PSCo coordinated the emission

measurement activities at this plant. ADA Technologies and CONSOL Inc. performed the emission tests. The target emission component measured was mercury (Hg). Hg analyses were performed on the combustion flue gas stream, inlet coal samples and baghouse hopper ash.

## 1.2 Key Personnel

Testing at Comanche Unit #2 occurred October 12-16, 1998. During that time, the ADA Technology test team consisted of the following key personnel:

Gary Anderson, Senior Technician ..... 303/792-5615  
 Keith Cummer, Intern..... 303/792-5615  
 Jason Ruhl, Project Engineer..... 303/792-5615  
 Justin Smith, Associate Engineer..... 303/792-5615

The CONSOL Inc. test team consisted of the following key personnel:

Leonard L. Anthony, Technician..... 412/854-6702  
 Orville C. Bedillion Jr., Technician..... 412/854-6678  
 Matthew DeVito, Group Leader..... 412/854-6679  
 Ron Oda, Research Engineer ..... 412/854-6539

The tests were coordinated with Public Service Company of Colorado key personnel:

Mark Andarka, Comanche Plant Engineer ..... 719/549-3705  
 Mark Fox, Environmental Services Project Lead..... 303/571-7047  
 Terry Hunt, Professional Engineer ..... 303/571-7113

After testing was completed in October 1998, the mercury emission results were reported to DOE as part of the contract requirements. This report has been generated specifically to satisfy the reporting requirements of the EPA Information Collection Request. The following key person from Public Service Company of Colorado is responsible for submitting this report:

Mark Fox, Environmental Services Project Lead..... 303/571-7047

The following key personnel from ADA Technologies are involved in preparing this report:

Jim Butz, Chief Engineer..... 303/792-5615  
 Cathy Grover, Project Engineer/Principal Author ..... 303/792-5615  
 Justin Smith, Associate Engineer..... 719/549-3784

## 2.0 Plant and Sampling Location Descriptions

### 2.1 Process Description and Operation

Comanche Unit #2 is an electric generating unit with a rated gross generating capacity of 375 MW. Powder River Basin Coal, received by railroad car from the Belle Ayr Mine near Gillette, Wyoming, is used as the primary fuel source. The steam-generating unit is a Babcock and Wilcox Company design. The single reheat, suction-fired Carolina-type, radiant steam generator, is a natural circulation unit designed to fire pulverized coal in a water-cooled balanced draft furnace. The steam generator has a design pressure of 2,400 psig and is capable of producing 2,482,697 pounds of steam per hour with main steam and reheat temperature of approximately 1000°F.

Four (4) "Gravimetric" coal mills supply pulverized coal for combustion at a maximum rate of 480,000 pounds per hour. Coal is received from the coal silos, located directly above the feeders. The coal enters the top of the pulverizers and falls into the rotating grinding ring where it is crushed between the roll wheels and grinding ring. Tempered primary air from the primary air fans enters each pulverizer at the grinding zone and carries the fine pulverized coal particles to the burners. The primary air is maintained at approximately 140°F so it can dry and preheat the coal before it enters the steam generator.

The combustion of fuel and air takes place in the furnace section of the steam generator. The furnace is a water-cooled, dry bottom-type furnace measuring 43 feet wide by 45 feet deep by 162 feet high. The front and rear walls of the furnace slope down toward the center of the furnace to form the inclined sides of the bottom. Ash and/or slag from the furnace is discharged through the bottom opening into an ash hopper directly below it.

The furnace is equipped with thirty-two circular, pulverized coal burners and eight overfire air ports. Sixteen of the coal-fired burners are located on the south side of the boiler, arranged in four rows of four burners per row at elevations 4,918' 10 3/4", 4,906 10 3/4", 4,894 10 3/4", and 4,882 10 3/4". Additionally, four overfire air ports located at a platform elevation of 4,927' 6" serve the four burner quadrants. The same configuration is repeated on the north side of the boiler. Combustion air provided by forced draft fans enters the burners through the secondary air ducts. Secondary air from the windbox enters the burner through adjustable registers located around the

periphery of the burner. The burner registers impart a whirling action to the air, which combines with the pulverized coal and this mixture is ignited.

The steam generating unit is equipped with sootblowers manufactured by Diamond Power Specialty Corporation. The sootblowers are used to clean the boiler heat transfer surfaces of ash and slag with direct streams of high-pressure air in strategic areas inside the boiler. Sootblowers are also used to clean ash from the induced draft fans and air heaters

Particulate emissions are controlled with a reverse gas fabric filter dust collector. The flue gas stream leaves the steam generator, flows to an air preheater, then enters the fabric filter dust collector (FFDC) before discharging up the stack.

Figure 2-1 illustrates the basic utility operation schematic. The basic process schematic includes:

- Sub-bituminous Powder River Basin coal from the Belle Ayr Mine is received via rail car.
- The coal is stored in the coal silos before being fed to the coal mill and pulverizer.
- The pulverized coal is entrained in primary air before being fed through burners installed through the water walls which form the combustion chamber. Overfire air is used as a NO<sub>x</sub> control mechanism.
- The single reheat, suction-fired Carolina-type, radiant steam generator, is a natural circulation water-cooled balanced draft furnace designed to fire up to 480,000 pounds of pulverized coal per hour.
- The flue gas stream leaves the steam generator, flows through an air preheater, then enters the fabric filter dust collector (FFDC) before discharging out the stack.

The test program to collect mercury in flue gas data consisted of paired flue gas measurements at the reverse gas baghouse inlet and outlet. Coal and ash samples were also collected during testing for mercury analysis. Coal samples were collected at the coal feeders, prior to the coal pulverizer. The ash samples were collected from the baghouse hoppers.

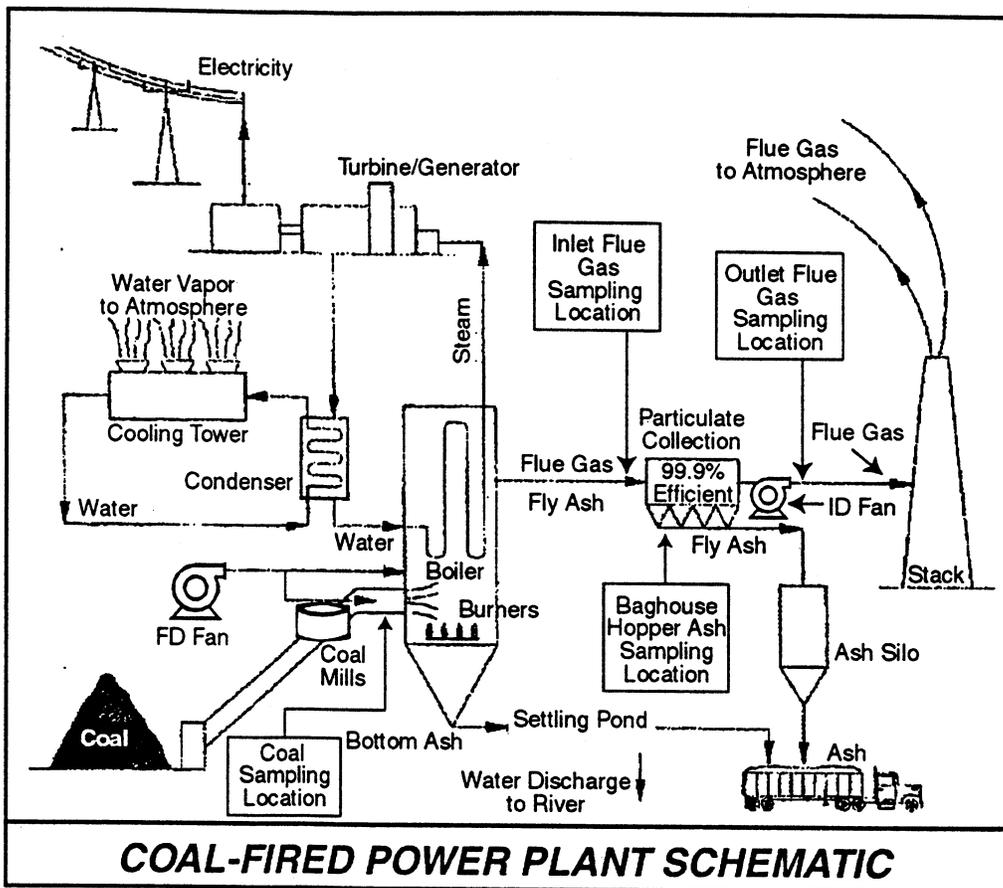


Figure 2-1. Comanche Unit #2 Process Flow Diagram.

## 2.2 Control Equipment Description

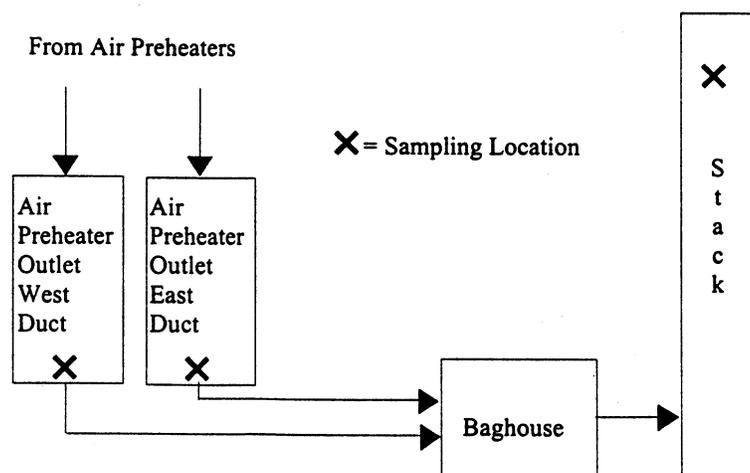
Particulate matter in the combustion flue gas stream is controlled by a Fabric Filter Dust Collector. The FFDC is configured for reverse gas cleaning. Particulate laden flue gas enters the baghouse and flows through the bags from the inside out; therefore, particulate remain and collection occurs on the inside surface of the bags. The bags are cleaned periodically by reversing the flow of air, causing the previously collected dust cake to fall from the bags into a hopper below. As dust accumulates on the fabric filter, the pressure differential across the bag increases. In order to remove collected particulates, the FFDC is cleaned by reversing the flow of air thru the bags once a pressure differential of approximately 5" is reached. Upon cleaning, the fabric filters release the collected particulate which falls into the ash hopper. The hoppers are purged when full. After passing through the reverse gas baghouse, the particulate cleaned flue gas exits through the stack. An opacity monitor is installed to continuously measure and report opacity at the stack.

NOx emissions are controlled with overfire air. The upper secondary air supply duct provides 10-20% of the combustion air through eight overfire air ports (4 overfire air ports per side) located above the upper burner row of each wall. The overfire air ports are located at a platform elevation of 4,927' 6", or approximately 8' 5 1/4" above the top row of burners.

In order to minimize sulfur oxide emissions, a low sulfur content coal is burned as the primary fuel.

## 2.3 Flue Gas Sampling Locations

Flue gas emission sampling was conducted at 1) the baghouse inlet and 2) the baghouse outlet. Figure 2-2 is a schematic of the sampling location with the direction of the flue gas indicated by arrows.



**Figure 2-2.** Flow Path Schematic and Sampling Ports at Comanche Unit #2.

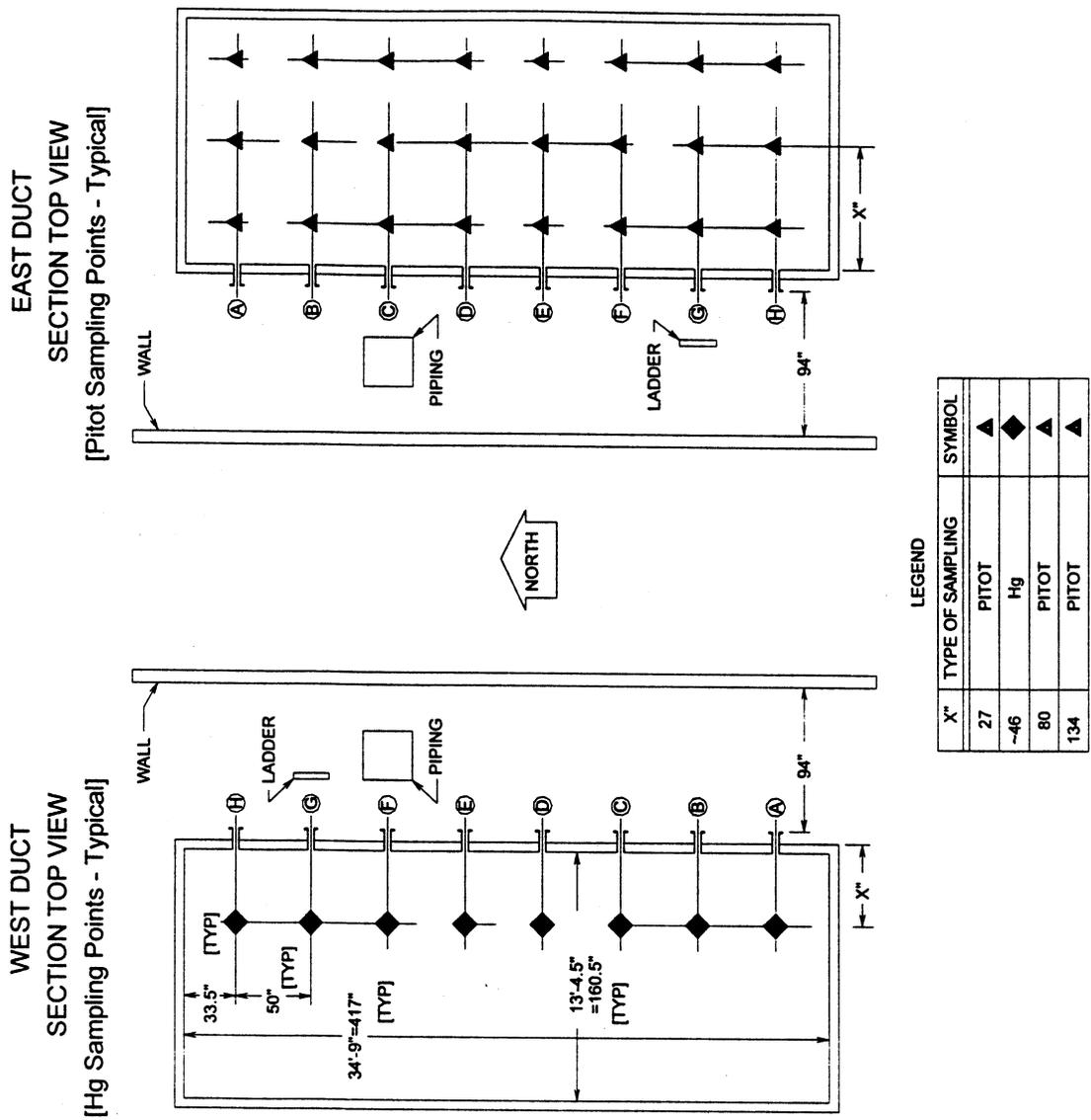
### 2.3.1 Host Baghouse Inlet (APH Outlet)

The host baghouse inlet sampling location is located at the air preheater (APH) outlet. The APH outlet is divided into east and west ducts, as illustrated in Figure 2-3. Each duct is approximately 13' 5" deep and 34' 9" wide. Six of the eight sampling ports located on each duct were accessible for testing. Each sample port was 4" in diameter and had a 24" long nipple.

The configurations of both ducts, upstream and downstream of the sampling ports, do not conform to optimum volumetric flow rate measurements as suggested by EPA Method 1. Approximately 12 - 24" upstream of the sampling location, internal structures prevented sampling. Approximately 12" downstream of the sampling location, the duct wall converges per the attached photo, Figure 2-4. Other considerations when sampling at this location include APH soot blowing cycle and the rotation of the APH, causing variable duct velocities. However, these are the only sampling ports associated with the existing available platforms. Sampling was performed at the locations illustrated in Figure 2-3 above. Therefore, the sample location was less than 1 equivalent diameter from the downstream disturbance and less than 1 equivalent diameter from the upstream disturbance. As illustrated in Figure 2-3, an 24 point pitot traverse (three sampling points per port) was used. Due to clearance

problems associated with testing at this location, an extendable S-type pitot was fabricated to reach the three sampling points per port.

Clearance problems at this test location coupled with the complexity of the Ontario-Hydro (O-H) mercury flue gas sampling train dictated single point sampling at each test port. As illustrated in Figure 2-3, the single sampling point per port was located at about one-third of the duct width into the duct. Mercury measurements were conducted both isokinetically and non-isokinetically at this sampling location. Concurrent with flue gas sampling, coal and ash samples were collected in order to perform a mercury mass balance. The effect of using a single mercury flue gas testing point per port is shown to be minimal when reviewing the Hg mass balance calculations. The mass balance shows overall mercury convergence within 20%. The results of the mass balance are discussed in Section 3.3.



**Figure 2-3.** Comanche Unit #2 Baghouse Inlet (APH Outlet) Sampling Location.

*Note: While only the East Duct pitot sampling is shown in the East Duct sketch, this figure is also representative of pitot sampling conducted on the West Duct. Additionally, while only the West Duct Hg sampling is shown in the West Duct sketch, the West Duct figure is representative of Hg sampling conducted on the East Duct.*

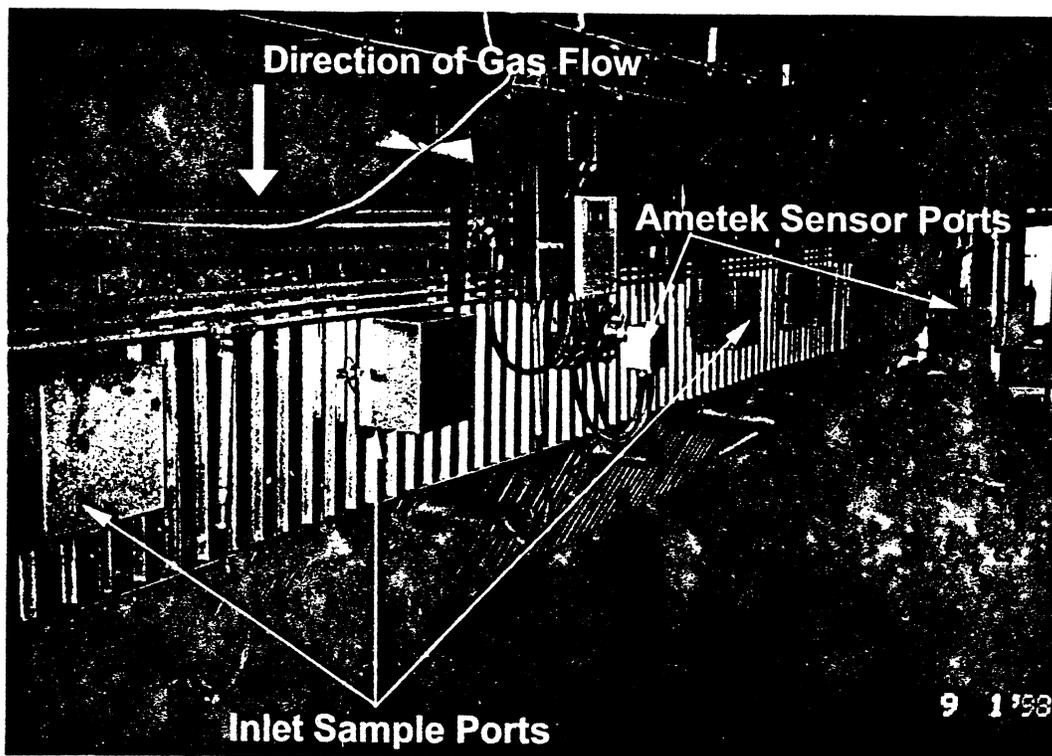


Figure 2-4. Comanche Unit #2 Baghouse Inlet Ports (Air Preheater Outlet).

### 2.3.2 Host Baghouse Outlet (Stack)

The host baghouse outlet sampling location was located at the 375' level of the stack. The 375' level sampling platform was accessible by elevator and there was adequate space for sampling equipment. Figure 2-5 illustrates the stack sampling location. At the 375' level, the inside diameter of the stack is 23' 2 3/4". Downstream of the sample location, the flue gas enters the stack breeching at an elevation of 72' 0". The stack exit is at an elevation of 500'. Therefore, the sample location was approximately 13 diameters from the downstream disturbance and 5 diameters from the upstream disturbance which complies with EPA Method 1. As illustrated in Figure 2-5, a 12 point pitot traverse (three sampling points per port) was used. There are four sampling ports spaced at 90° intervals around the circumference of the stack. The sample port is 4" in diameter and has a nipple length of 24". The configuration of the stack and subsequent sampling conforms to optimum volumetric flow rate measurements as suggested by EPA Method 1.

Downstream of the baghouse, it was anticipated that there would be very little particulate at this sampling location and the mercury would be thoroughly mixed and in the vapor phase. Therefore Hg measurements were conducted isokinetically from a single point using the Ontario-Hydro sampling method. The effect of a single mercury test point on the stack is shown to be minimal when reviewing the mass balance calculations. The mass balance shows overall mercury convergence within 20%. The results of the mass balance are discussed in Section 3.3.

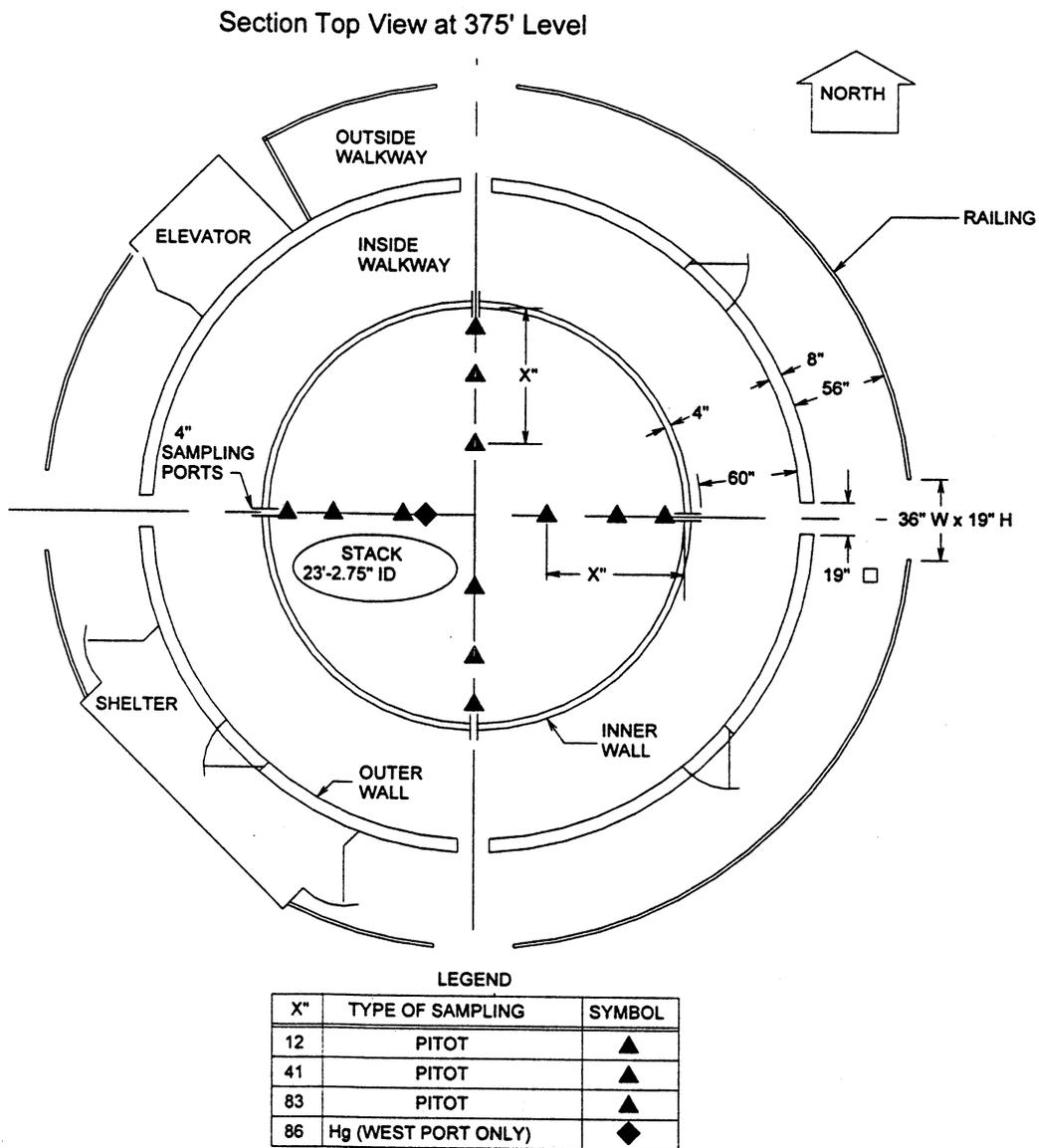


Figure 2-5. Comanche Unit #2 Baghouse Outlet (Stack) Sampling Location.

## 2.4 Process Sampling Locations

### 2.4.1 Coal Sample

Coal samples were taken approximately once per hour during the Ontario-Hydro testing from all four of the mills to ensure that the composite was representative of the coal burned during Ontario-Hydro sampling. The coal sample was extracted directly ahead of the feeder providing coal to the pulverizers. Each feeder is equipped with a

sample port and ball valve located above the feeder. Approximately 500 ml (volume basis) of coal was removed per hour from each sample location throughout the course of the test day and collected in a common 5-gallon bucket. At the end of each test day, these coal samples were thoroughly mixed into one bucket to obtain a composite coal sample. The container was properly labeled with the date and a sample identification number. It should be noted here that this daily coal sample was collected upstream of the mill and was an unpulverized coal sample.

#### **2.4.2 Ash Samples**

At the beginning of each test day, ADA Technologies requested that the baghouse be cleaned and the ash pulled from the hoppers just prior to the start of testing. Once the baghouse was cleaned, the ash pull system was stopped while mercury flue gas testing was conducted. The cleaning procedure functioned normally which allowed for ash accumulation in the hoppers. At the end of each test day, ash samples were collected from each of the 24 baghouse hoppers at the Comanche Unit #2 baghouse as shown per Figure 2-6. An existing port on each hopper was used to extract the ash. Each hopper baghouse sample was stored in a separate container and labeled with the corresponding hopper number, date, and time. (These samples were not combined to form a composite sample.) The containers consisted of double bagged ziploc baggies. All the baggies were placed in a common 5-gallon bucket and covered with aluminum foil for storage.

A "boiler bottom" ash sample was obtained once per day. This sample was marked with an "A" or "B" to indicate boiler location, date, and time. "A" denotes the East side of the boiler and "B" denotes the West side of the boiler. Economizer ash samples were taken once per day, and labeled A & B using the same methodology mentioned above.

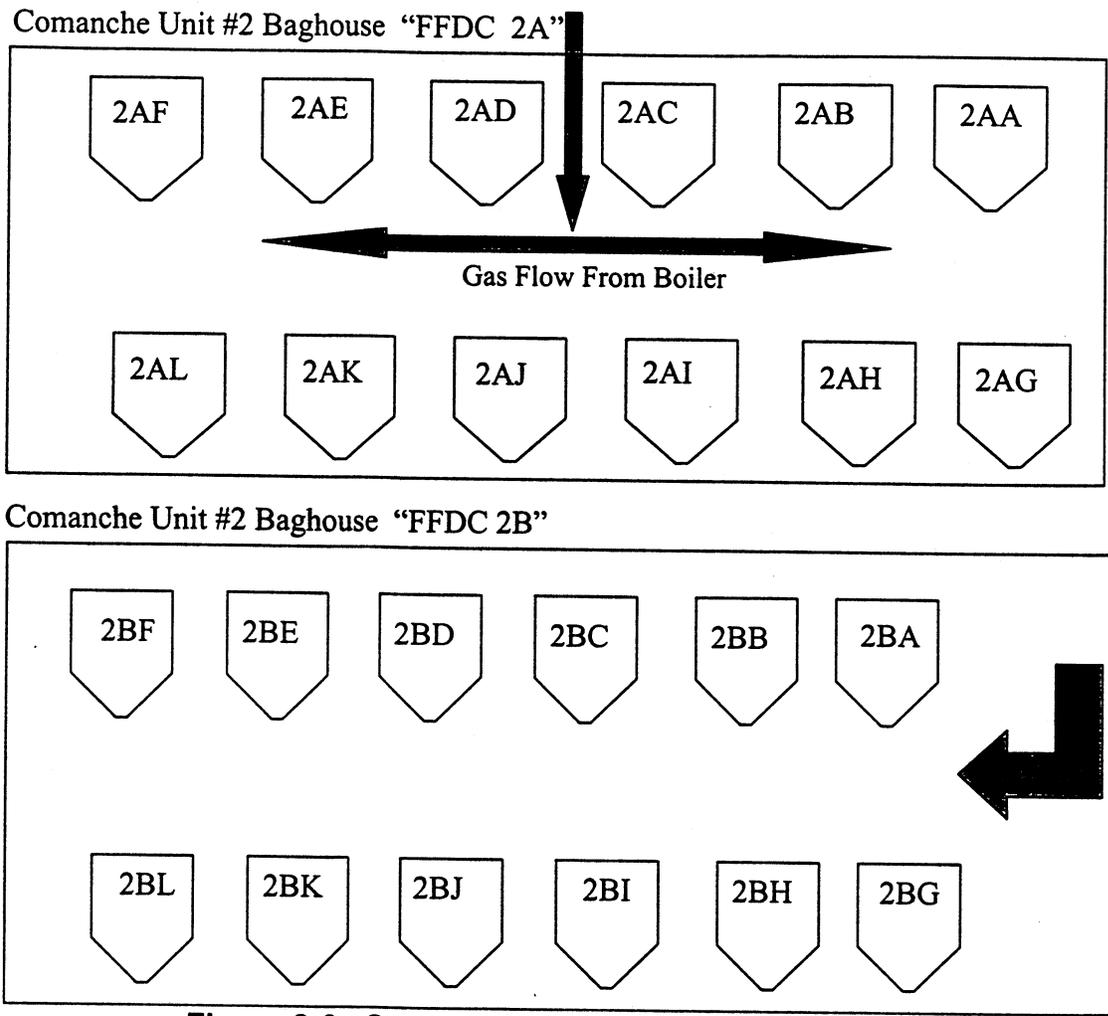


Figure 2-6. Comanche Unit #2 Baghouse Arrangement.

### 3.0 Summary and Discussion of Test Results

#### 3.1 Objectives and Test Matrix

The purpose of the test program at Comanche Unit #2 was to quantify and partition flue gas mercury upstream and downstream of the plant particulate control device. As part of a DOE/FETC contract, ADA is investigating the use of carbon based sorbents to remove mercury from coal fired flue gas streams. Full-scale mercury data was gathered to establish a mercury baseline. The specific objectives were:

- Measure flue gas mercury using isokinetic sampling.
- Measure flue gas mercury using "low-ash" (non-isokinetic) sampling methodology.
- Quantify flue gas vapor-phase mercury (Hg) at the particulate control device inlet. Compare standard testing methodology to "non-isokinetic" testing at the particulate control device inlet.
- Quantify flue gas particulate mercury (Hg) at the particulate control device inlet.
- Quantify flue gas vapor-phase mercury (Hg) at the particulate control device exit.
- Collect coal samples during O-H testing in order to perform a mercury mass balance.
- Collect ash samples during O-H testing in order to perform a mercury mass balance.
- Obtain unit process operation data during testing to confirm steady-state operation.
- Perform mercury mass balance based upon analyses of collected test samples.

CONSOL R&D performed the sampling program which was limited to volumetric flow rate measurements and flue gas Hg measurements using the Ontario-Hydro sampling method. Flue gas Hg measurements at Comanche Unit #2 consisted of a total of 10 tests over three consecutive days of sampling. ADA Technologies collected the coal and ash samples and unit process operation data as well as performed the mercury mass balance computations.

The first day and a half consisted of ten Ontario-Hydro Hg flue gas tests across the Comanche Unit #2 baghouse. This is illustrated in Table 3-1. A total of six of these tests were performed on the baghouse inlet, three isokinetically and three non-isokinetically ("low-ash"). The non-isokinetic tests were performed with an oversized sampling nozzle directed opposite to the flow to reduce the particulate catch and its potential influence upon the Hg speciation. Each isokinetic test on the baghouse inlet was conducted simultaneously with a non-isokinetic host baghouse inlet test and a baghouse outlet test for a total of nine tests. The tenth test was a repeat test on the baghouse outlet. Testing at the APH outlet on October 15, 1998 was performed to compare to pilot data. These pilot results are presented in the DOE/FETC contract reporting, however the APH outlet data will be shown and discussed in this report.

**Table 3-1. Flue Gas Test Matrix, Comanche Unit #2.**

Location	Date		
	10/13/98	10/14/98-am	10/15/98-am *1
Host Baghouse			
Inlet (APH Outlet), Isokinetic	9:30-12:10	8:30-10:45	12:00-14:03
	14:20-17:00	N/A *1	15:15-17:20
Inlet (APH Outlet), Non -Isokinetic	9:30-12:20	8:30-10:48	N/A
	14:23-16:58	N/A *1	
Outlet (Stack)	9:35-11:55	8:23-10:00	Pilot
	14:30-16:50	10:50 - 12:25 *1	

*Note \*1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to obtain data to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.*

Volumetric flow rate measurements were taken prior to each Hg measurement test, except for those taken at the baghouse inlet. Preliminary pitot surveys and the one volumetric flow rate measurement that was taken at the baghouse inlet indicated that the flow was badly skewed. This is a result of the close proximity to upstream and downstream flow disturbances. Therefore, an accurate determination of volumetric flow rate at this location was not possible.

Coal samples were collected roughly on an hourly basis during O-H testing from each of the four coal mills throughout the course of testing. At the end of each test day, all individual coal samples were combined to obtain a composite coal sample. Additionally, at the end of the test program, all daily collected coal composite samples

were combined to form 1 overall composite coal sample. This combined composite sample was pulverized and blended in order to obtain a single sample for coal mercury analysis. Table 3-2 presents the sampling matrix. The original data log can be found in appendix A.

**Table 3-2. Coal Sample Matrix, Comanche Unit #2.**

Location	Date		
	10/13/98	14/14/98	10/15/98 *1
Unpulverized Coal to Mill Feeder			
Pulverizer #1	9:48	9:15	12:25
	11:40	10:36	14:45
	14:30	12:23	17:43
Pulverizer #2	9:48	9:15	12:25
	11:40	10:36	14:45
	14:30	12:23	17:43
Pulverizer #3	9:48	9:15	12:25
	11:40	10:36	14:45
	14:30	12:23	17:43
Pulverizer #4	9:48	9:15	12:25
	11:40	10:36	14:45
	14:30	12:23	17:43
		15:10	

*Note \*1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to obtain data to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.*

*Note \*2: The above samples collected were combined to form one composite sample, including samples from 10/15/98.*

Ash samples were collected at the end of the testing day. A sample was taken from each of the 24 hoppers that are at the Comanche Unit #2 baghouse. The baghouse samples were kept separate from each other and labeled with the corresponding hopper number, date and time. Economizer ash samples and "wet bottom" ash sample were also obtained at the end of each test day. Table 3-3 presents the sampling matrix summary. A detailed sample log can be found in Appendix B.

**Table 3-3. Ash Sample Matrix, Comanche Unit #2.**

Location	Date		
	10/13/98	14/14/98	10/15/98 *1
Ash Location			
Ash/Hopper 2AA-2AL, 2BA-BL	1 each compartment	1 each compartment	1 each compartment
Economizer Ash East	2	1	1
Economizer Ash West	2	1	1
Boiler Bottom Ash East	1	1	1
Boiler Bottom Ash West	1	1	1

*Note \*1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.*

**3.2 Field Changes and Problems**

**3.2.1 Additional O-H Samples**

During stack testing on October 14<sup>th</sup>, 1998, it was believed that the O-H impinger system had “backflushed” (one of the impinger solutions backfilled into the previous impinger solutions). Therefore, an additional outlet stack sample was performed. However, laboratory analysis results for the “backflushed” impinger and the repeated impinger were similar. No other field problems were encountered during testing.

**3.3 Presentation of Results**

As noted above in Section 3.0, the purpose of the test program at Comanche Unit #2 was to quantify flue gas mercury at a full-scale facility. In this section, a summary of the results for each specific objective is presented.

**3.3.1 Flue Gas Particulate Emissions (Isokinetic Sampling)**

A listing of some of the principal sampling parameters at the inlet and outlet of Comanche Unit #2 baghouse is shown in Table 3-4. A more complete listing of the sampling parameters, as well as the field data sheets, can be found in the Appendix C.

**Table 3-4. Comanche Unit #2 Baghouse Particulate Sampling Results.**

Location	Unit 2 In	Unit 2 Out	Unit 2 In	Unit 2 Out	Unit 2 In	Unit 2 Out
Isokinetic Configuration?	Yes	Yes	Yes	Yes	Yes	Yes
Date	10/13/98	10/13/98	10/13/98	10/13/98	10/14/98	10/14/98
Test	1	1	2	2	3	3
Start Time	0930	0935	1420	1430	0830	0823
End Time	1210	1155	1700	1650	1045	1000
Sampling Time	96	140	96	130	96	85
Barometric Pressure [ " Hg ]	25.66	25.66	25.55	25.55	25.40	25.40
Static Pressure [ " H <sub>2</sub> O ]	-13.8	-0.84	-13.8	-0.83	-14.4	-0.83
O <sub>2</sub> [ % ]	5.4	4.5	6.5	4.5	5.2	4.5
CO <sub>2</sub> [ % ]	14.7	15.5	13.7	15.5	14.9	15.4
Duct Temp [F]	289	301	302	316	287	297
Gas Velocity [ ft/sec ]	---	62.71	---	64.34	---	64.81
Gas Flow Rate [ ACFM ]	---	1,404,000	---	1,431,000	---	1,455,000
Gas Flow Rate [ DSCFM ]	---	743,000	---	737,700	---	759,700
Particulate Conc [ gr/dscf ]	2.56	<0.001	2.09	<0.001	2.27	<0.001
Particulate Flow Rate [ lb/hr ]	16,100	<2	13,200	<2	14,600	<2
% Isokinetic	106	105	101	106	104	103

These data show the consistent operation of Comanche Unit #2 baghouse over the three test periods. The average gas flow rate measured at the baghouse outlet or stack was 746,800 DSCFM. The average gas flow rate on the baghouse inlet was not measured because of the skewed flow conditions. The average particulate emissions rate at the Comanche Unit #2 baghouse inlet was 14,600 lb/hr. The baghouse was effective at reducing the average particulate emission rate below our detection limit of 0.001 gr/dscf (2 lb/hr). All the Hg flue gas tests were within the isokinetic range of 101 - 106%.

### 3.3.2 Flue Gas Particulate Emissions (Non-Isokinetic)

Table 3-5 compares the results of baghouse inlet sampling using both an isokinetic and a non-isokinetic sampling configuration. As previously discussed, the objective of the non-isokinetic sampling was to reduce the particulate loading to the quartz filter and the potential for interaction between the fly ash and the gas phase Hg. The O-H sample train with minimum ash collection consisted of a large nozzle pointed away from the flue gas flow to reduce the ash quantity collected on the filter. This yields ash (although not as an isokinetic sample), oxidized-vapor and elemental-vapor mercury values. The non-isokinetic samples were extracted simultaneously with the isokinetic samples.

**Table 3-5.** Comanche Unit #2 Baghouse Inlet Particulate: Isokinetic vs. Non-Isokinetic

Location	Unit 2 In					
Isokinetic Configuration?	Yes	No	Yes	No	Yes	No
Date	10/13/98	10/13/98	10/13/98	10/13/98	10/14/98	10/14/98
Test	1	1	2	2	3	3
Start Time	0930	0930	1420	1423	0830	0830
End Time	1210	1220	1700	1658	1045	1048
Sampling Time	96	96	96	96	96	96
Nozzle Diameter [ in ]	0.252	0.500	0.252	0.500	0.252	0.500
Nozzle Area [ in <sup>2</sup> ]	0.0499	0.196	0.0499	0.196	0.0499	0.196
Barometric Pressure [ " Hg ]	25.66	25.66	25.55	25.55	25.40	25.40
Static Pressure [ " H <sub>2</sub> O ]	-13.8	-13.8	-13.8	-13.8	-14.4	-14.4
O <sub>2</sub> [ % ]	5.4	5.6	6.5	5.7	5.2	5.5
CO <sub>2</sub> [ % ]	14.7	14.5	13.7	14.4	14.9	14.5
Duct Temp [ F ]	289	287	302	299	287	285
Particulate Conc [ gr/dscf ]	2.56	0.227	2.09	0.251	2.27	0.255
Particulate Flow Rate [ lb/hr ]	16,100	1431	13,200	1580	14,600	1633
% Isokinetic	106	---	101	---	104	---

The average particulate concentration in the isokinetic sample is 2.31 [gr/dscf]. The average particulate concentration in the non-isokinetic sample is 0.2443 [gr/dscf].

These results show that the non-isokinetic sampling configuration resulted in an average 89% reduction in the collected particulate mass.

### 3.3.3 Inlet Flue Gas Mercury (Hg) Concentrations

A total of nine flue gas Hg tests were conducted at Comanche Unit #2 baghouse inlet. Three of these measurements were completed using the "oversized, reverse nozzle" non-isokinetic configuration and with the other six completed using the isokinetic sampling configuration. Table 3-6 summarizes the Hg measurements at the baghouse inlet as measured. Table 3-7 summarizes the Hg measurements at the baghouse inlet corrected to 3% O<sub>2</sub>. Hg<sup>0</sup> represents elemental mercury. Hg ++ represents oxidized mercury.

**Table 3-6.** Inlet Flue Gas Hg Speciation (as measured) at the Baghouse Inlet.

Date	Test	Isokinetic	Hg Concentration [ $\mu\text{g}/\text{dm}^3$ ]			
			Particulate	Hg++	Hg <sup>0</sup>	Hg (total)
10/13/98	1	Yes	1.57	3.41	4.95	9.93
10/13/98	2	Yes	2.09	1.17	4.65	7.91
10/14/98	3	Yes	4.63	1.12	3.22	8.97
Avg $\pm$ Standard Deviation (1-3)			2.76 $\pm$ 1.64	1.90 $\pm$ 1.31	4.27 $\pm$ 0.92	8.94 $\pm$ 1.01
Avg / Hgtot [ % ]			31%	21%	48%	100%
10/14/98	4	Yes	3.75	2.02	7.19	12.96
10/15/98	5	Yes	2.23	2.17	4.85	9.25
10/15/98	6	Yes	3.72	1.78	4.45	9.95
Avg $\pm$ Standard Deviation (1-6)			3.00 $\pm$ 1.20	1.95 $\pm$ 0.83	4.89 $\pm$ 1.29	9.83 $\pm$ 1.71
Avg / Hgtot [ % ]			30%	20%	50%	100%
10/13/98	1	No	1.43	2.39	7.63	11.45
10/13/98	2	No	1.48	1.63	7.09	10.20
10/14/98	3	No	1.88	1.80	4.07	7.75
Avg $\pm$ Standard Deviation (1-3)			1.60 $\pm$ 0.25	1.94 $\pm$ 0.40	6.26 $\pm$ 1.92	9.80 $\pm$ 1.88
Avg / Hgtot [ % ]			16%	20%	64%	100%

Note 1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.

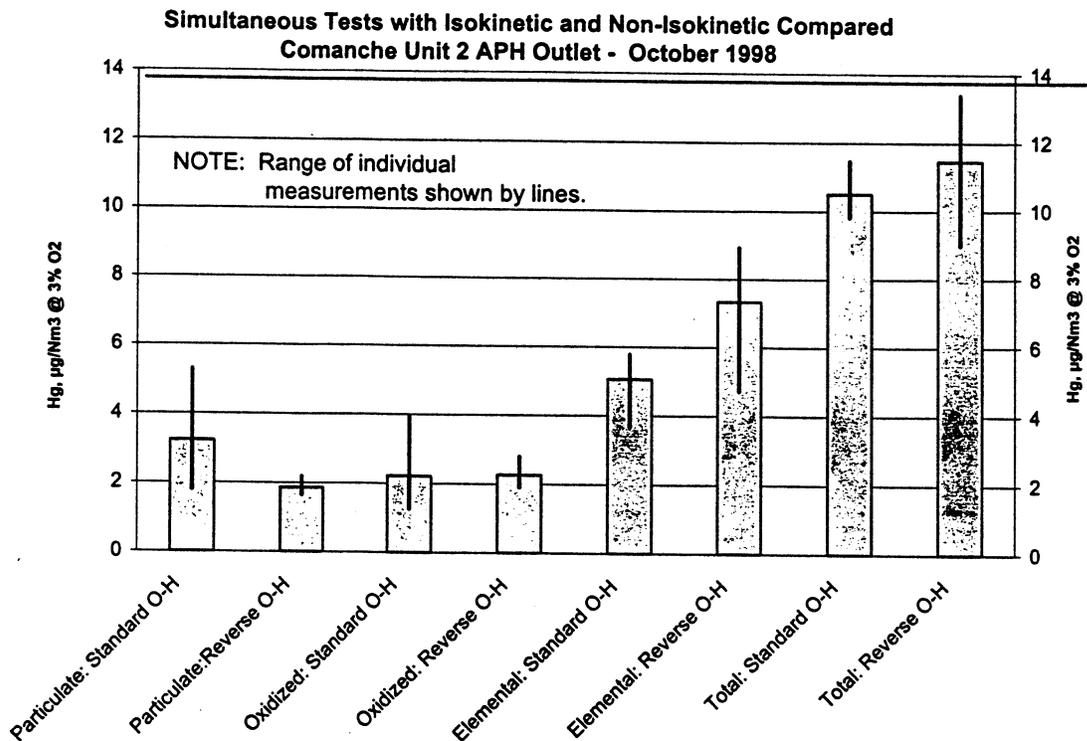
**Table 3-7. Flue Gas Hg Speciation (corrected to 3% O<sub>2</sub>) at the Baghouse Inlet.**

Date	Test	Isokinetic	Hg Concentration [ $\mu\text{g}/\text{m}^3$ @ 3% O <sub>2</sub> ]			
			Particulate	Hg <sup>++</sup>	Hg <sup>0</sup>	Hg (total)
10/13/98	1	Yes	1.81	3.94	5.72	11.47
10/13/98	2	Yes	2.60	1.45	5.78	9.83
10/14/98	3	Yes	5.28	1.28	3.67	10.23
Avg $\pm$ Standard Deviation (1-3)			3.23 $\pm$ 1.82	2.22 $\pm$ 1.49	5.06 $\pm$ 1.20	10.51 $\pm$ 0.86
Avg / Hgtot [ % ]			31%	21%	48%	100%
10/14/98	4	Yes	4.22	2.27	8.09	14.59
10/15/98	5	Yes	2.51	2.44	5.46	10.41
10/15/98	6	Yes	4.19	2.00	5.01	11.20
Avg $\pm$ Standard Deviation (1-6)			3.44 $\pm$ 1.33	2.23 $\pm$ 0.95	5.62 $\pm$ 1.44	11.29 $\pm$ 1.73
Avg / Hgtot [ % ]			30%	20%	50%	100%
10/13/98	1	No	1.67	2.80	8.93	13.40
10/13/98	2	No	1.74	1.92	8.35	12.01
10/14/98	3	No	2.19	2.09	4.73	9.01
Avg $\pm$ Standard Deviation			1.87 $\pm$ 0.28	2.27 $\pm$ 0.47	7.34 $\pm$ 2.28	11.47 $\pm$ 2.24
Avg / Hgtot [ % ]			16%	20%	64%	100%

Note \*1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to obtain data to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.

The as-measured isokinetic Hg concentration (gas and solid phase) at the baghouse inlet for test points 1-3 averaged 10.51  $\mu\text{g}/\text{m}^3$  @ 3% O<sub>2</sub> (range 9.83 – 11.47  $\mu\text{g}/\text{m}^3$  @ 3% O<sub>2</sub>). Approximately 31% of the total mercury was in the particulate phase, 21% of the mercury was in the oxidized vapor-phase, and 48% of the mercury was in the elemental vapor-phase. However, the isokinetic vs. non-isokinetic measurements showed a decrease of particulate Hg from 31% to 16% of the total Hg present in the samples. In addition, there was a corresponding increase in the elemental Hg from 48% to 64%. As previously discussed, the non-isokinetic vs. isokinetic particulate sample results indicated an ~89% reduction in particulate capture. These data suggest that particulate matter (fly ash) is absorbing gas phase Hg. A closer look at these data show that the oxidized Hg concentration was unchanged which indicates that the fly ash

is removing the elemental Hg fraction. Figure 3-7 depicts a comparison of the results of the three test runs with isokinetic and non-isokinetic ("low-ash") sampling procedures.



**Figure 3-7.** O-H Tests 1-3 at Comanche Unit #2 Baghouse Inlet (APH Exit), October 1998.

In summary, the conclusions from the flue gas isokinetic and non-isokinetic O-H testing are:

- The split for speciation in Comanche Unit #2's flue gas is approximately: 16% particulate mercury, 20% oxidized mercury, and 64% elemental mercury (from "non-isokinetic" test results).
- The particulate appears to preferentially adsorb elemental, vapor-phase mercury under the tested conditions.
- Either the particulate fines are much more efficient at mercury adsorption during the sampling process than the total particulate, or most of the mercury present on the fines is already there prior to sampling.
- Total mercury measurements for standard isokinetic and reverse-nozzle sampling agree either way, and are mostly unaffected by the speciation splits.

### 3.3.4 Inlet Flue Gas Mercury Particulate (Hg) Concentrations

The isokinetic sampling at this location was conducted using a glass cyclone assembly upstream of the quartz filter. Using this configuration resulted in three discrete particulate catches; (1) sample nozzle and probe, (2) cyclone, and (3) quartz filter. For the first test, all of the solids fractions were combined in one particulate composite. However, for the remaining five test, these samples were individually recovered and analyzed. Table 3-8 lists the mass, % mass, and Hg concentration of these samples.

**Table 3-8.** Hg Particulate Components at the Comanche Unit #2 Baghouse Inlet.

Date	Test	Sample Train Component	Particulate Sample		Hg		
			Mass [g]	% Mass	Conc [mg/g]	Mass [mg]	% Mass
10/13/98	2	Probe	0.7673	12	0.16	0.12	4
		Cyclone	4.5935	72	0.11	0.51	20
		Filter	0.9856	16	1.98	1.95	76
10/14/98	3	Probe	1.4972	21	0.27	0.40	7
		Cyclone	4.7564	65	0.14	0.67	11
		Filter	1.0265	14	4.85	4.98	82
10/14/98	4	Probe	0.7000	13	0.24	0.17	4
		Cyclone	3.9326	71	0.15	0.59	13
		Filter	0.8864	16	4.37	3.87	83
10/15/98	5	Probe	1.0225	18	0.14	0.14	5
		Cyclone	3.6484	66	0.11	0.40	14
		Filter	0.8902	16	2.64	2.35	81
10/15/98	6	Probe	0.4593	8	0.22	0.10	2
		Cyclone	4.6570	76	0.12	0.56	12
		Filter	0.9973	16	4.08	4.07	86
Average (Test Pts. 2-6)		Probe		14			4
		Cyclone		70			14
		Filter		16			82

Note \*1: Testing at Comanche Unit #2 APH outlet continued after the paired O-H testing at the particulate control device inlet/outlet was completed in order to obtain data to compare to pilot flue gas mercury measurements. These pilot results are presented in the DOE/FETC contract reporting.

The average mass collection breakdown for test points 1-6 is 14% by the probe, 70% by the cyclone, and 16% by the quartz filter. The filter solids, although representing just 16% of the total collected solids, contains 82% of the particulate Hg. These data show that the extended surface area of the smallest particles and the intimate gas-to-particle contact of the solids on the filter are the most likely mechanisms leading to Hg absorbence on the particulate solids. The adsorption of flue gas Hg on the particulate filter can result in misleading speciation data. More work is needed to understand this mechanism.

### 3.3.5 Baghouse Outlet (Stack) Flue Gas Hg Concentrations

A total of four Ontario-Hydro Hg measurements were completed at the Comanche Unit #2 baghouse outlet (stack) location. These four samples consisted of replicate sampling on October 13 and 14, 1998. The as measured results are summarized in Table 3-9. Table 3-10 summarizes these results corrected to 3% O<sub>2</sub>.

**Table 3-9.** Flue Gas Hg Speciation (as measured) at the Comanche Unit #2 Baghouse Outlet (Stack).

Date	Test	Isokinetic?	Hg Concentration [ $\mu\text{g}/\text{m}^3$ ]			
			Particulate	Hg <sup>++</sup>	Hg <sup>0</sup>	Hg (total)
10/13/98	1	Yes	0.00	3.05	0.25	3.30
10/13/98	2	Yes	0.00	3.66	0.60	4.26
10/14/98	3	Yes	0.00	2.93	0.30	3.23
10/14/98	3A	Yes	0.00	2.92	0.60	3.52
Average $\pm$ Standard Deviation			0.00	3.14 $\pm$ 0.35	0.44 $\pm$ 0.19	3.58 $\pm$ 0.47
Average / Hgtot [ % ]			0%	88%	12%	100%

**Table 3-10. Flue Gas Hg Speciation (3% O<sub>2</sub>) at the Comanche Unit #2 Baghouse Outlet (Stack).**

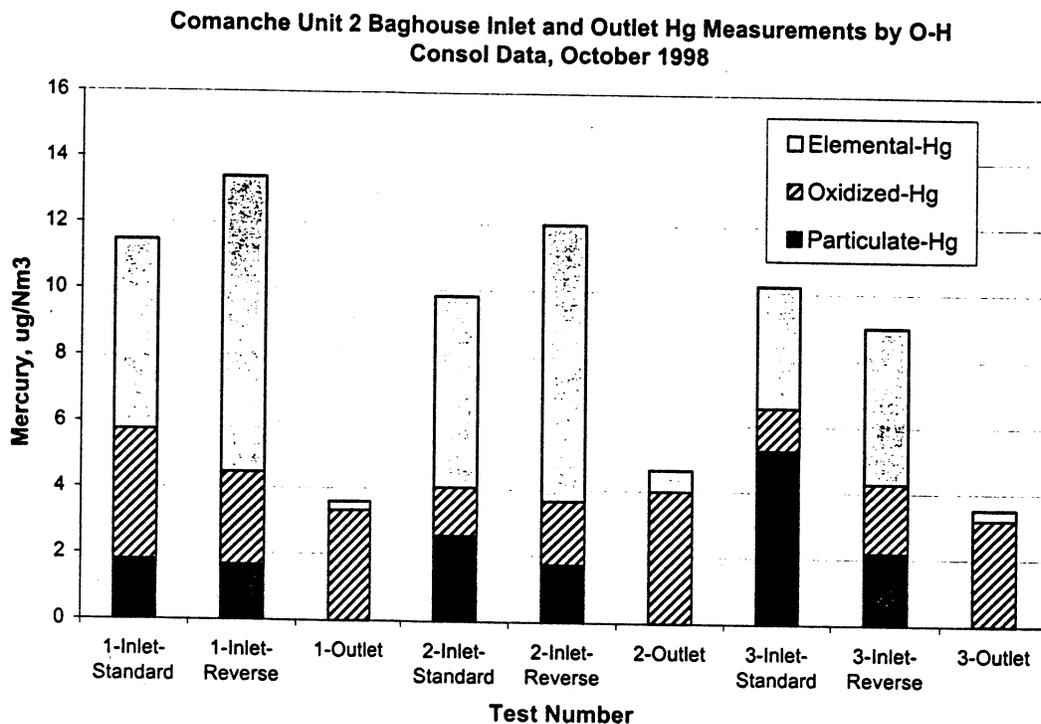
Date	Test	Isokinetic?	Hg Concentration [ $\mu\text{g}/\text{m}^3$ @ 3% O <sub>2</sub> ]			
			Particulate	Hg <sup>++</sup>	Hg <sup>0</sup>	Hg (total)
10/13/98	1	Yes	0.00	3.33	0.27	3.60
10/13/98	2	Yes	0.00	3.99	0.65	4.65
10/14/98	3	Yes	0.00	3.20	0.33	3.53
10/14/98	3A	Yes	0.00	3.23	0.66	3.89
Average $\pm$ Standard Deviation			0.00	3.44 $\pm$ 0.37	0.48 $\pm$ 0.21	3.92 $\pm$ 0.51
Average / Hgtot [%]			0%	88%	12%	100%

The total Hg concentration averaged  $3.92 \mu\text{g}/\text{m}^3$  @ 3% O<sub>2</sub> with a standard deviation of 0.51 (percent relative standard deviation, PRSD, of 13%). All of the Hg at Comanche Unit #2 baghouse outlet was in the gas phase with 88% present as Hg<sup>++</sup> and 12% present as Hg<sup>0</sup>. On a total Hg basis, the Comanche Unit #2 baghouse is removing 63% of the Hg. The elemental Hg fraction is the species that is being removed. These data also suggest that the fly ash may be altering the flue gas Hg speciation. The baghouse inlet showed an oxidized Hg concentration of  $\sim 2.2 \mu\text{g}/\text{m}^3$  while the outlet showed an oxidized Hg concentration of  $\sim 3.4 \mu\text{g}/\text{m}^3$ .

Figure 3-8 illustrates the stack "outlet" tests 1-3 with the corresponding baghouse "inlet" tests 1-3. Both isokinetic and non-isokinetic baghouse inlet tests are shown. The temperature at the stack location was approximately 300°F, which was fairly uniform across the stack. The temperature at the APH outlet location averaged 300°F. Conclusions from the removal tests across the Comanche Unit #2 baghouse are:

- 63% of the total mercury was removed across the baghouse. This is significantly more mercury than is in the particulate phase at the inlet to the baghouse (31%). Therefore adsorption of the vapor-phase mercury by the ash is taking place across the baghouse.
- On average more of the mercury was in the oxidized form at the stack than at the inlet to the baghouse:  $3.4 \mu\text{g}/\text{Nm}^3$  at the stack, as compared with  $2.2 \mu\text{g}/\text{Nm}^3$  at the baghouse inlet. There could be oxidation of the mercury from the elemental to oxidized phase through the baghouse, or it could be that the APH outlet values for oxidized mercury are measured low because of the presence of particulate. If the

latter is true, adsorption of the oxidized mercury onto the ash in the sample train is implied, and is not dependent on the quantity of particulate in the range tested. Tests run with minimal particulate collection showed similar oxidized mercury results to those tests run isokinetically. At this location, mercury that is in the elemental form is removed efficiently by the baghouse, and some may be oxidizing in the baghouse.



**Figure 3-8. O-H Tests 1-3 at Comanche Unit #2 Baghouse Inlet and Outlet, October 1998.**

### 3.3.6 Coal Samples

Simultaneous with flue gas mercury testing, coal samples were collected for later mercury analysis. Unpulverized coal was collected at various time intervals. After testing, all collected coal samples were combined to form a composite sample. An airtight lid was placed on the container to protect the unpulverized composite coal sample. The unpulverized sample was then sent to Hazen Laboratories for pulverization as well as ultimate, proximate, Btu and Cl analysis. These results are presented in Table 3-11. Upon receipt of the pulverized coal samples, four aliquots were prepared by cone and quartering and sent to Frontier GeoSciences for mercury

analysis. These results are presented in Table 3-12. All mercury mass balance calculations presented in Section 3.3.8 are based on the ultimate coal analysis by Hazen Laboratories. The reported air dry loss was 10.49%.

**Table 3-11. As Received Coal Analyses by Hazen Laboratories.**

HHV, Btu/lb	Moisture	Carbon	Hydrogen	Nitrogen	Sulfur	Ash	O <sub>2</sub> (by diff.)	Cl
8,669	27.1	50.48	2.61	0.73	0.31	4.70	14.07	ND<0.01

Note: Chlorine was measured and was found to be below the level of detection.

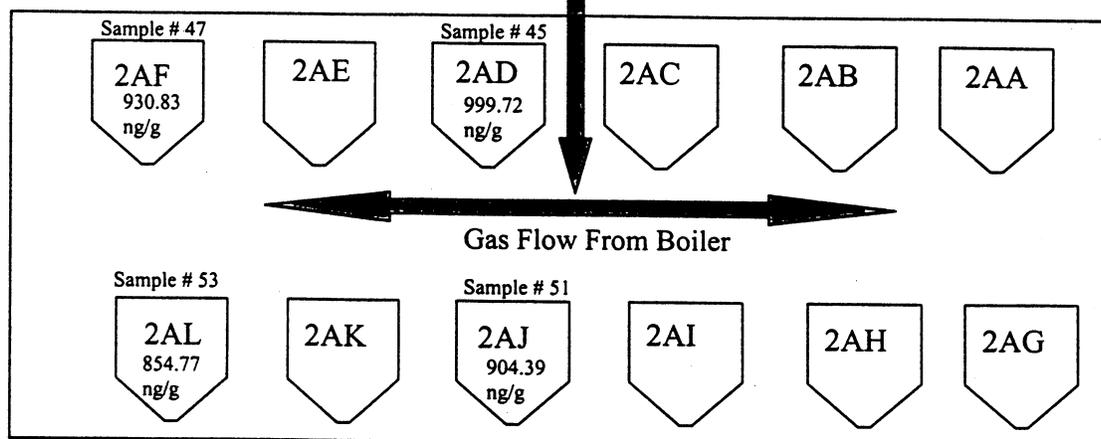
**Table 3-12. Coal Mercury Analysis by Frontier GeoSciences.**

Sample ID	As Reported Hg, ng/g
C1	68.56
C1-Dup	70.23
C4	62.86
C8	57.31
Average	64.74

**3.3.7 Ash Samples**

Several hopper ash samples collected from the Comanche Unit #2 baghouse on October 13, 1998 were randomly selected for Hg analysis. Frontier GeoSciences performed the Hg analysis of the ash samples. The results of the ash analysis are shown in Figure 3-13. The ash mercury values have quite a bit of variability even within one baghouse, as was seen when several samples from different locations in the Comanche Unit #2 baghouse were analyzed for Hg. The average of these samples is 978 ng Hg/g ash. The range is 855 to 1,160 ng/g. This represents %RSD of 10.8%.

Comanche Unit 2 Baghouse "FFDC 2A"



Comanche Unit 2 Baghouse "FFDC 2B"

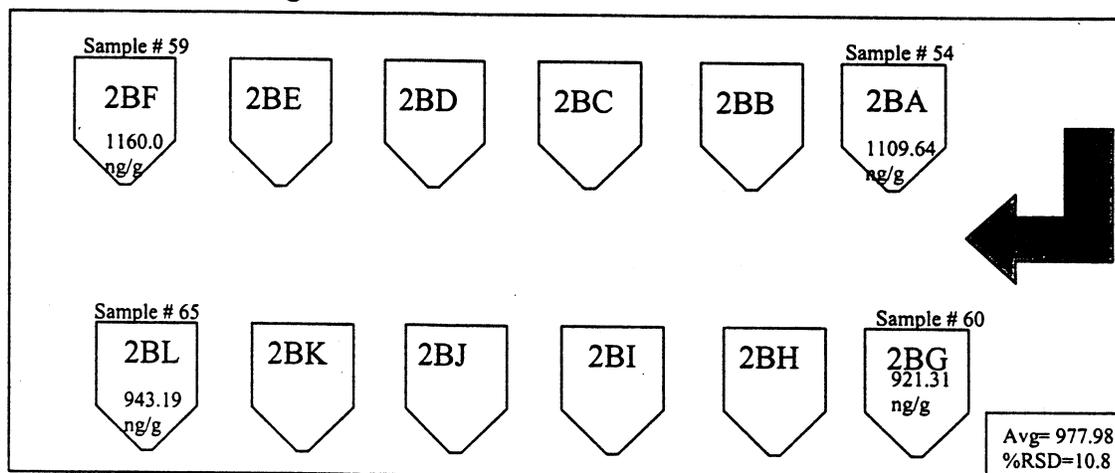


Figure 3-9. Comanche Unit #2 Baghouse Hopper Ash Hg Analysis.

The results of analyses of wet bottom ash and economizer ash samples yielded undetectable or negligible levels of mercury. Only one of the four samples analyzed yielded a mercury result over the detection limit, and the value was 1.4 ng Hg/g wet bottom ash. Compared to the hopper ash value of 978 ng/g ash, this value is negligible and has not been included in the mass balance calculations.

### 3.3.8 Unit Process Operation Data

Process operation data was monitored from the control room to record any process upsets during testing. Comanche Unit #2 operated at approximately 350 MW gross during testing. Unit operation remained in the steady state condition throughout testing. All control room data sheets can be found in Appendix E.

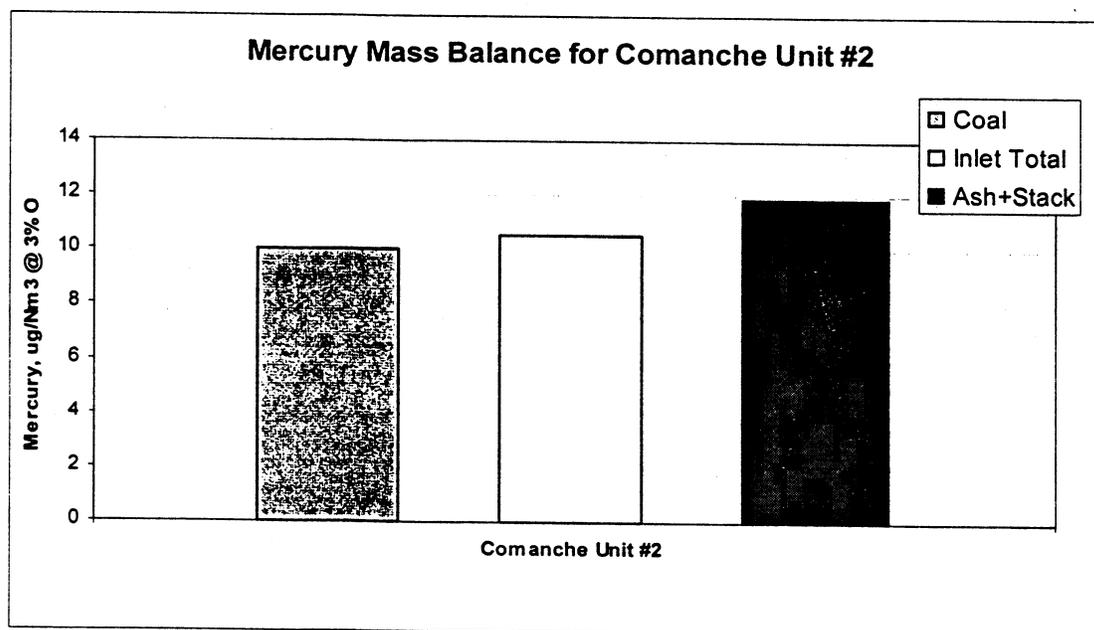
### 3.3.9 Mass Balance

A mass balance was performed on Comanche Unit #2 to evaluate mass closure between the coal Hg analysis, combustion flue gas inlet (O-H APH outlet) Hg analysis and combustion flue gas outlet (O-H stack) mercury analysis + hopper ash Hg analysis. Table 3-14 summarizes the results mentioned within the previous sections, 3.3.1 – 3.3.7. These values have been corrected to 3% O<sub>2</sub> and converted to a Hg value representative at  $\mu\text{g}/\text{Nm}^3$ . (The conversion of Hg units to  $\mu\text{g}/\text{Nm}^3$  is discussed in Section 4.1)

**Table 3-13. Comanche Unit #2 Average Hg by Location.**

Location	Sample Name	Avg Hg, $\mu\text{g}/\text{Nm}^3$	Hg Total per Location, $\mu\text{g}/\text{Nm}^3$
Coal Inlet	Coal	10.01	10.01
Particulate Control Device Inlet	Flue Gas Inlet	10.51	10.51
Particulate Control Device Outlet	Flue Gas Outlet	3.92	11.85
	Hopper Ash	7.93	

These values are illustrated in Figure 3-9. While the results show some variability, the mass balance converges <20%.



**Figure 3-10. Mercury Mass Balance at Comanche Unit #2, October 1998.**

## 4.0 Sampling and Analytical Procedures

### 4.1 Test Methods

In this section, the sampling methods will be discussed along with the analysis details.

#### 4.1.1 Flue Gas Hg Measurements

Mercury flue gas measurements were made at the inlet and outlet of the particulate control device per the March 2, 1999 draft "Standard Test Method for Elemental, Oxidized, Particle-bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro method)" with minor modifications. The deviations from the above listed method are:

Exception 1: A stainless steel nozzle was used rather than a glass nozzle.

Explanation 1: The CONSOL test team opted to use stainless steel nozzles instead of glass nozzles. At both the baghouse inlet and stack sampling location, the probe assembly had to be inserted through a four inch diameter pipe 24 inches in length. Clearance problems at each location required careful and precise insertion and removal technique. The use of stainless steel nozzles eliminated the potential for breakage and sample delays. No significant Hg was found in the nozzle rinses. Because all of the ductwork of the gas handling system is constructed of steel, it is unlikely that the use of stainless steel nozzles had any impact on the reported Hg measurements. Additionally, according to Keith Curtis (developer of O-H), stainless steel is acceptable.

Exception 2: At the inlet duct location, only a partial traverse could be achieved.

Explanation 2: Access was limited by obstructions inside and outside of the duct. The traverse was completed to the extent possible. The CONSOL test team conducted the sampling through as many of the inlet ports as possible using a consistent, single point depth in each of these ports. In this manner we have minimized any problems associated with horizontal stratification of the particulate and flue gas Hg. Per EPA Method 1, this is acceptable if the available location is not accessible. Additionally, inlet total mercury matched well with coal mercury, leading us to believe our sampling protocol was adequate.

Exception 3: The stack sample was a single point, not a traverse.

Explanation 3: Assumptions stated by the EPA note that Hg in flue gas is primarily in the gaseous phase and that stratification is not expected. Additionally, as noted previously in this report, the collection efficiency of the Comanche Unit #2 baghouse captured 99% of the particulate in the flue gas stream. Therefore, any remaining particulate-bound Hg would represent only <1% of total.

Exception 4: Separate coal samples were not taken for each of the three test runs. The coal samples were not weighed at the time of collection. The coal sample was not classified into 3 groups by top size with samples recovered from each group size according to ASTM 2234.

Explanation 4: The coal is from a single source and samples were taken regularly throughout testing. The samples were combined, pulverized and triplicate analyses made. The coal to inlet mercury mass balance matched very well.

Exception 5: During the Comanche testing, only 2 KMnO<sub>4</sub> impingers were used instead of the 3 specified in the March 2 O-H draft procedure.

Explanation 5: CONSOL has performed numerous O-H testing and found that no mercury was ever detected in the 3<sup>rd</sup> KMnO<sub>4</sub> impinger. Additionally, we have performed a mass balance and the mass balance demonstrates a Hg closure within 20%.

In summary, flue gas Hg concentrations were determined at all locations using the Ontario-Hydro Hg speciation train. The train schematic is shown in Figure 4-11. This figure includes the optional cyclone assembly installed between the probe exit and the filter inlet. The cyclone is used when high particulate concentrations are encountered and serves to reduce the particulate loading on the filter.

In the Ontario Hydro method, gas is isokinetically extracted from the flue gas stream. The gas is pulled through a heated glass-lined probe, optional cyclone, and a quartz filter. All of these components collect particulate matter (front-half) with the quartz filter serving as an absolute collection medium. Total particulate mass loading is calculated from the collected solids in the probe, optional cyclone, and filter. Probe and filter temperatures are maintained at a minimum of 258°F ±20°F. Where particle interference is suspected, the probe and filter temperature are maintained as close as practical to the flue gas temperature. The flue gas exits the filter and is pulled through a series of chilled impingers. The first three impingers are filled with 100 mL of a 1M potassium chloride (KCl) solution. The purpose of these impingers is to capture all oxidized forms of Hg in the flue gas (Hg<sup>++</sup>). The next impinger is filled with 100 mL of 5% nitric acid (v/v) and 10% H<sub>2</sub>O<sub>2</sub> (v/v) solution. The purpose of this impinger is to remove

SO<sub>2</sub> from the flue gas to preserve the oxidizing strength of the permanganate impingers. Hg collected in this impinger is assumed to be the elemental form (Hg<sup>0</sup>). The next two impingers are filled with 100 mL of a 4% (w/v) acidic potassium permanganate (KMnO<sub>4</sub>) solution and collect elemental mercury. The gas exits the impinger train through a silica gel-filled impinger which serves to remove the moisture from the flue gas. This train configuration results in the species collection Hg sequence as shown in Table 4-15:

**Table 4-14. Hg Speciation by Train Component.**

Train Component	Species Measured
Probe & Nozzle Rinse	Particulate Hg
Cyclone (Optional)	Particulate Hg
Quartz Filter	Particulate Hg
KCl Impingers	Hg <sup>++</sup>
HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> Impinger	Hg <sup>0</sup>
KMnO <sub>4</sub> Impingers	Hg <sup>0</sup>
HCl Rinse of KmnO <sub>4</sub> Impingers	Hg <sup>0</sup>

Past investigations have established that fly ash from coal combustion processes can absorb flue gas Hg. In the Ontario-Hydro train, the particulate matter is removed from the flue gas by filtration. The quartz filter used in the train is a point where intimate gas-to-solid contact constantly takes place throughout the entire duration of the sampling (96 to 140 min). Two techniques were utilized to reduce the potential for Hg absorption on the filter. At the Comanche Unit #2 baghouse inlet location, a glass cyclone was installed between the probe exit and the filter inlet to collect the majority of the particulate matter. In addition, a series of non-isokinetic measurements were made at the baghouse inlet location, using an oversized sampling nozzle pointing opposite to the gas flow. In this configuration, the particles in the flue gas must make a 180° turn to enter the sampling nozzle. In addition, employing an oversized sampling nozzle resulted in a ~25% reduction in the intake flue gas sampling velocity at the tip of the sampling nozzle. As a result, due to particle inertia, the majority of larger particulate follows the flue gas air stream and is not aspirated in the sample nozzle.

The amount of mercury collected in the impinger solutions was determined as outlined in EPA Method 29 and the Ontario-Hydro Draft Method. An aliquot of the impinger solution is acidified and the mercury is determined using cold vapor-atomic absorption spectroscopy. The atomic absorption spectrometer is calibrated with commercial mercury standard. The calibration is verified using NIST Standard 1641C. The calibration is reassessed periodically by analyzing a quality control standard. The instrument is recalibrated as required. Each sample matrix is analyzed as a set and an individual calibration curve is used for each set. Selected samples are spiked with 5 ng/mL (ppb) of mercury and reanalyzed. Spike recovery must be within  $\pm 20\%$  or the sample is diluted and reanalyzed. Selected samples are analyzed in duplicate. The duplicates must be within  $\pm 20\%$  or the analyses are repeated.

In cases where sufficient solids are collected, analysis is conducted on a sample aliquot. In cases where the particulate catch is low (primarily the Comanche Unit #2 baghouse outlet filters) the entire sample including the filter is used. The samples are digested with aqua-regia in pressure vessels prior to analysis by CVAA.

Figure 4-12 illustrates the sample recovery method.

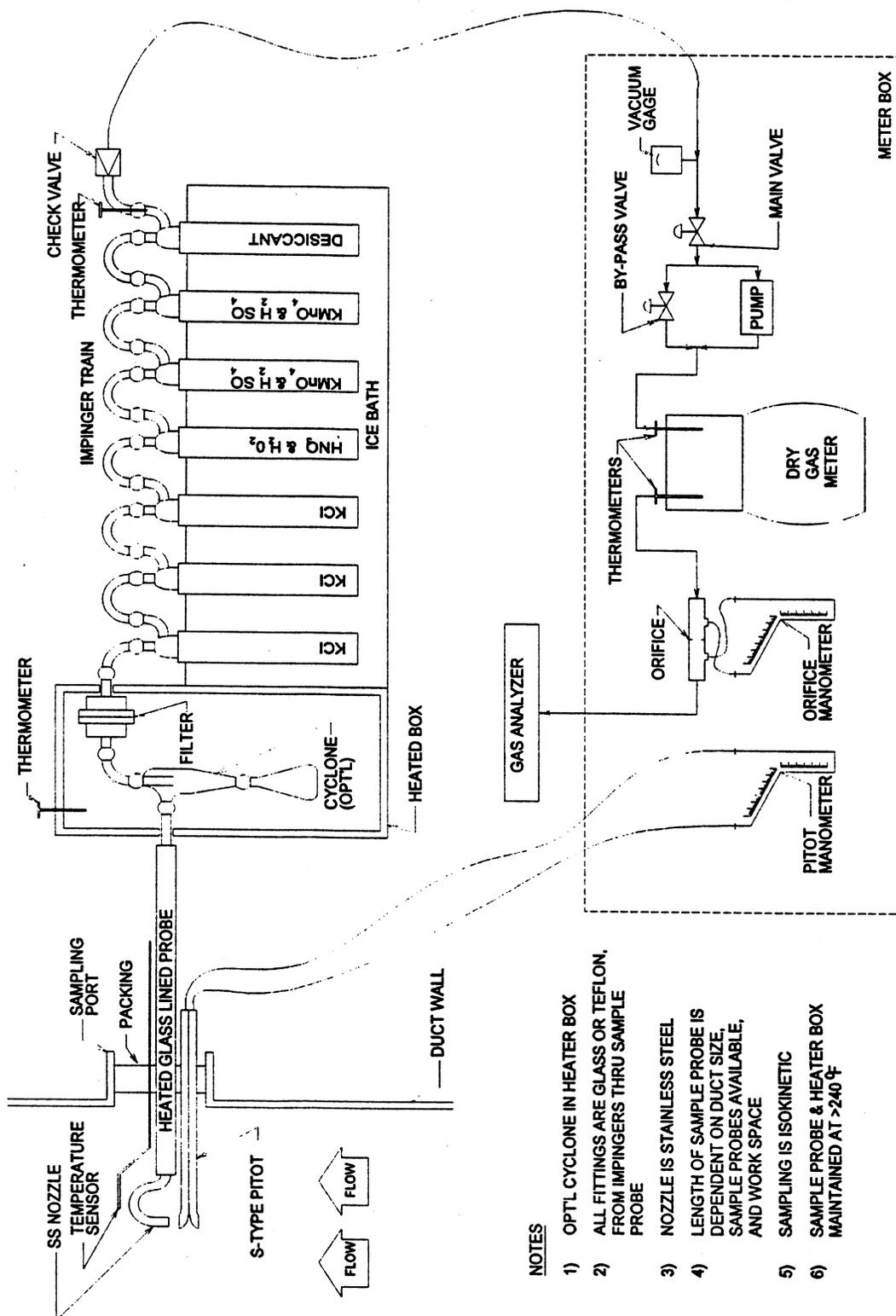


Figure 4-11. Ontario Hydro Sample Train.

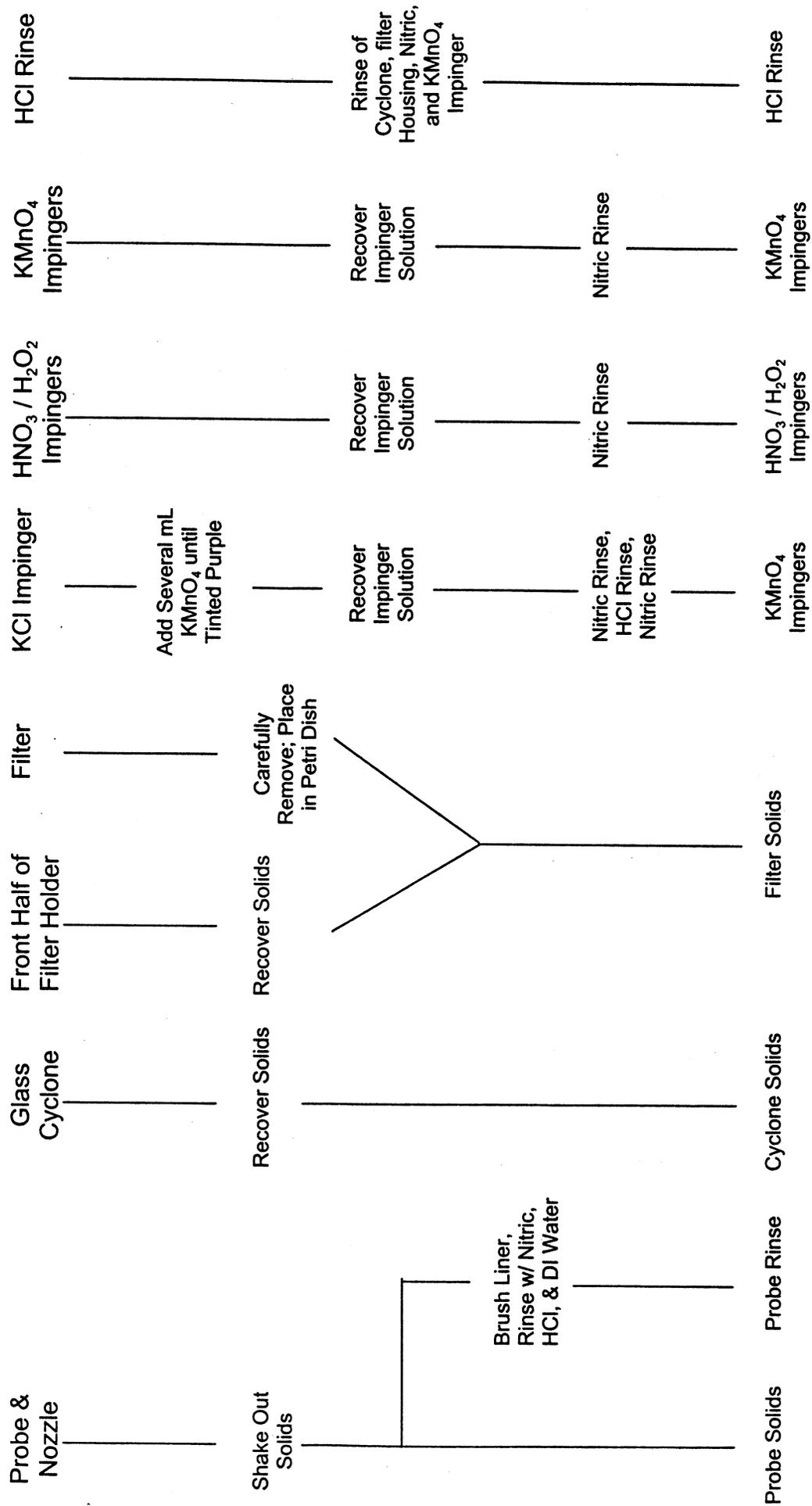


Figure 4-12. Ontario-Hydro Hg Sampling Recovery Schematic

**4.1.2 Coal Measurement**

Coal samples were collected in the manner described in Section 3.3.5. Care was taken to collect equal sample amounts from each of the four coal feeders. The composite coal sample was protected from the elements by being placed in a common 5-gallon bucket and sealed with an airtight lid. The entire unpulverized sample was sent to Hazen laboratories in order to be pulverized. Hazen also performed an ultimate, approximate, Btu and Cl analysis on the composite sample after pulverization. The pulverized coal sample was returned to ADA Technologies.

ADA Technologies then extracted 4 aliquot samples of the crushed coal to send to FGS for mercury analysis. The initial coal samples sent to FGS had quality control issues. Therefore the coal samples were re-analyzed for mercury. Coal sample analysis was completed via "Total Wet Oxidation with Perchloric Acid in Teflon Microwave Digestion Bombs" followed by detection via Modified US EPA Method 1631 (CVAFS). Calibration was verified using the referenced SRM recovery – NIST 1630. Results of this standard along with the original FGS coal mercury analyses results are located in Appendix F.

After receipt of the coal mercury analysis from FGS, ADA Technologies used a variety of customized data reduction spreadsheets in the calculation of the field sampling data and the Hg concentration data. The output from these spreadsheets is presented in Appendix F. The coal Hg is first corrected for air dry loss using the following equation:

$$\text{As Reported Coal Hg (ng/g)} * (1 - \% \text{ H}_2\text{O from air dry loss}) = \text{Coal Hg actual (ng/g)}$$

Using the coal ultimate analysis, the stoichiometric products are determined using the CRC 1990 definition of standard air. From this, the dry flue gas total mols are calculated. From these results, we calculate Hg,  $\mu\text{g}/\text{Nm}^3$ .

$$\frac{\text{Coal Hg (\#Hg / \#fuel)}}{\text{Total Mols, Dry (mol / \#fuel)}} \times \frac{35.3147 (\text{ft}^3 / \text{m}^3)}{380 (\text{ft}^3 / \# \text{mol})} \times 453.59 (\text{g} / \#) \times 1,000,000 (\mu\text{g} / \text{g})$$

### 4.1.3 Ash Measurement

ADA Technologies collected ash samples at the end of the testing day out of each of the 24 hoppers that are at the Comanche Unit #2 baghouse. The baghouse samples were kept separate from each other (not combined as the coal samples were) and labeled with the corresponding hopper number, date and time.

Eight of the 24 hopper ash samples were sent to FGS for analyses. The fly ash samples were digested via a 70:30 HNO<sub>3</sub>/H<sub>2</sub>SO<sub>4</sub> Hot Acid Reflux (Frontier GeoSciences Inc Standard Operating Procedure FGS-011) followed by detection via Modified US EPA Method 1631 (CVAFS). The results of the analyses are located in Appendix G.

After receipt of the ash mercury analysis from FGS, ADA Technologies used a variety of customized data reduction spreadsheets in the calculation of the field sampling data and the Hg concentration data. The output from these spreadsheets is presented in Appendix G. Since there were several ash samples analyzed, the ash Hg samples are first averaged to obtain an ash mercury value of 978 ng Hg/g ash. Using the coal ultimate analysis, the stoichiometric products are determined using the CRC 1990 definition of standard air. From this, the dry flue gas total mols are calculated. From these results, we calculate Hg, µg/Nm<sup>3</sup>:

$$\frac{\text{Hg (ng / g ash)}}{1000 \text{ (ng / } \mu\text{g)}} \times \frac{\text{Stoic. Products (\# ash / \# fuel)}}{\text{Total mols, Dry (\# mol / \# fuel)}} \times \frac{35.3 \text{ (ft}^3 \text{ / Nm}^3\text{)}}{380 \text{ (ft}^3 \text{ / \# mol)}} \times 453.59 \text{ (g / \#)}$$

### 4.2 Mercury Flue Gas Process Test Methods

The technical objective for this program was to complete a minimum of three paired flue gas Hg measurements across the baghouse of the Comanche Station Unit #2 using the Ontario Hydro Hg speciation sampling method. Assuming steady-state operating conditions and a consistent coal supply, our data quality objective was to provide flue average Hg-in-flue gas concentrations at both locations with a variability of <20%. A summary of the measurements made at the baghouse inlet and outlet is shown in Table 4-5.

Table 4-15. Ontario-Hydro Measurement Summary.

Location	Baghouse Inlet	Baghouse Outlet (Stack)
No. of Measurements	9 (6 isokinetic & 3 non-isokinetic)	4
Total Hg Conc., $\mu\text{g}/\text{m}^3$ (as measured)	9.83	3.58
Standard Deviation	1.71	0.47
% Relative St. Dev.	17.4	13.1

The accuracy of these measurements was assessed against our analytical criteria which is <5% error in the calibration range of our cold vapor atomic absorption analyzer (0.00 ng/mL blank and 5.00, 7.35, and 10.00 ng/mL standards), a <20% (or 1 ng/mL for samples less than 5 ng/mL of Hg concentration) difference in replicate samples analysis, and a 80% to 120% range for spike sample recoveries. A detailed description of the specific QA/QC procedures and results for this test program is presented in this section.

### General Program Procedures

The general QA/QC procedures for the sampling and analysis included the following:

- All sampling was conducted by personnel specifically trained and experienced in power plant air sampling methods, including the Ontario-Hydro Hg method,
- The use of standard sampling equipment maintained and calibrated as required,
- All sampling completed following a standard sampling protocol that was discussed with the project directors prior to the initial sampling,
- The use of consistent sample preparation and recovery procedures,
- Sample logging and tracking under the direction of sample team Group Leader,
- Individual calibration curves for each sample matrix,
- The analysis of NIST Standard Reference Material (SRM) and lab QC samples to verify calibration curve,

- Duplicate analysis of selected sample to assure repeatability,
- Analysis of selected "spiked" samples to assure sample recovery,
- Review of interim data to assure sample completeness,
- Use of computer spreadsheets to verify the accuracy of the calculations, and
- Review of sampling data by two senior professionals to help assure accuracy.

All of the QA/QC procedures were under the supervision of Mr. Matthew DeVito (412-854-6679). Mr DeVito is a Research Group Leader in the CONSOL Inc., Research and Development departments. Specific QA/QC responsibilities are illustrated in Table 4-16. A detailed discussion of these procedures is presented below.

**Table 4-16. QA/QC Responsibilities for Comanche Unit #2 Flue Gas Hg Measurements.**

<b>Task</b>	<b>Responsible Party</b>
<u>Sampling</u>	
Site Visit	Ron Oda
Site Specific Sample Plan (SSSP)	Ron Oda & Matt DeVito
Review of SSSP	Sheila Haythornwaite (ADA)
Equipment Prep	Orville Bedillion
Field Measurements	Ron Oda
Field Recoveries	Matt DeVito
<u>Sample Shipment</u>	
Field Log	Matt DeVito
Packing	Matt DeVito
Driving	Ron Oda
<u>Analysis</u>	
Sample Login	Matt DeVito & Carol Simmons
Sample Analysis	Dave Krofcheck
QC Checks	Dave Krofcheck
QC Review	Matt DeVito
<u>Data Reduction</u>	
Review of Field Sheets	Ron Oda
Review of Sampling Data	Ron Oda
Hg Emission Rates	Matt DeVito
<u>Reporting</u>	
Preparation	Ron Oda
Review	Matt DeVito

**4.2.1 Sampling and Analytical Personnel**

The sampling was conducted under the direction of Mr. DeVito with Mr. Ron Oda (Research Engineer) serving as the field team leader. Mr. DeVito is a charter member of the ASTM committee responsible for writing and evaluating the Ontario-Hydro Hg method. Mr. DeVito and Mr. Oda have extensive experience in coal-fired utility air sampling having conducted programs at over 20 different facilities. Mr. DeVito and Mr.

Oda have completed Hg measurement programs at 10 of these sites and have used the Ontario-Hydro draft methodology in five utility sampling programs and six in-house pilot scale test programs. In all of these program, CONSOL has utilized the same personnel for the equipment preparation and recovery, sampling, and analysis. A listing of these individuals with education and years of related experience is presented in Table 4-17.

**Table 4-17. CONSOL R&D Test Personnel**

Name	Education	Years of Experience	Previous Hg Programs	Responsibilities
Matthew DeVito	B.A. Education/Chemistry; M.S. Energy Resources	21	14	QA/QC, Sampling, Reporting
Ron Oda	B.S. Aerospace Eng; M.S. Applied Ocean Science	12	7	Sampling, Reporting
Vince Conrad	B.S. Chemistry Ph.D. Chemistry	18	18	Lab Manager
Robert Statnick	B.S. Chemistry Ph.D. Chemistry	27	14	Program Review
Orville Bedillion	High School	39	14	Sample Prep, & Recovery
Leonard Anthony	High School	28	14	Field Sampling
Dave Krofcheck	High School	25	25	Analysis
Carol Simmons	High School Vocational Courses	20	12	Sample Log-in

#### 4.2.2 Sample Equipment

The CONSOL test team used standard sampling equipment specific to EPA Methods 2, 5, 29, and the ASTM Ontario Hydro Hg Draft method. Most of the equipment was obtained from Graseby/Nutech and conforms to all stack sampling codes and specifications. The calibration procedures for this equipment and testing are displayed in Table 4-18.

Table 4-18. Summary of Equipment Calibration.

Equipment	Acceptance Criteria	Frequency & Method of Measurement
Wet Test Meter	2 cfm; accuracy within $\pm 1\%$	Reconditioned every 2 years
Dry Test Meters	Y and Delta H $\pm 5\%$	Calibrated vs. wet test meter annually and when post-check exceeds 5%
Thermometers	$\pm 3^\circ\text{F}$ with Glass Thermometer	Initially upon receiving
Probe Heaters	Capable of maintaining $248^\circ \pm 25^\circ\text{F}$ at sample flow rate	Field verified during every sampling
Barometer	$\pm 0.1$ in Hg	Calibrated with Hg column and field verified with electronic barometer
Nozzles	$\pm 0.004$ inches	Calibrated with micrometer; recalibrated when repairs have been completed
Pitot Tubes	Compliance with Method 2	Initially with in-house wind tunnel and after any tip repairs
Balance	$\pm 0.0003$ mg	Check with Class-S weights before daily use
Delta P Gauge	$\pm 0.001$ "H <sub>2</sub> O	Electronic unit factory calibrated

All of the equipment calibrations were completed by CONSOL sample personnel. Documentation of the most recent calibration data for the wet test meter, dry test meters, barometer, probe nozzles, and pitot tubes are included in Appendix L.

#### 4.2.3 Field Sampling

All flue gas sampling was conducted by CONSOL, Inc. using the procedures described in EPA Method 5 and the Ontario-Hydro Hg Speciation draft method. Any deviations or modifications to the draft Ontario Hydro method were discussed prior to sampling with the ADA project manager and are summarized in Section 3.3.

In addition to the sampling procedure mentioned in Section 3.3, the CONSOL test team followed a rigorous leak-check protocol to assure sample integrity. An initial leak check was completed as soon as the sampling train was assembled prior to system heat up. This enabled the sample team to find and correct any leaks on a cool system. Upon passing this check, the system was heated to the sampling temperature and another leak-check was conducted. A final leak check was completed at the completion of the sampling. Sampling at the baghouse inlet required that the complete sampling train be moved approximately 100' to accommodate both sample ducts. Additional leak-checks were conducted for these samples at the completion of sampling in the first duct, and then again after re-mobilizing at the second duct. A final leak-check was conducted at the end of the sampling.

Mercury flue gas samples were extracted using the O-H method. The absorbing solutions were made fresh daily. The impingers were charged and the sampling components were transported to the required locations. The sampling trains were assembled, leak-checked, pre-heated, and again checked for leaks. After passing the leak-check procedure, the sampling probes were inserted into the respective duct and sampling was initiated. Single point, isokinetic sampling (except in the case of the non-isokinetic tests) was conducted simultaneously at inlet and outlet locations. Sampling was conducted for 85-140 minutes resulting in a sample volume of 2-3 normal cubic meters. Oxygen readings were monitored at the outlet of the sampling train using a Teledyne Model Max 5 portable analyzer (electrochemical O<sub>2</sub> sensor). At the completion, the sample trains were once again checked for leaks, purged for 10 min, then disassembled. The components were transported back to the lab trailer for recovery. The Hg concentrations of the individual impinger solutions were determined by cold vapor atomic absorption (CVAA) as specified in the methodology. An acid digestion followed by CVAA determined the concentration of Hg on the solids. All CVAA tests were conducted in the CONSOL corporate research lab in Library, PA.

All of the field data was recorded on standard forms which are included in Appendix C. The sampling data was reduced using standard "in-house" spreadsheets. Copies of these summary sheets are included in Appendix D.

#### **4.2.4 Flue Gas Sample Preparation and Recovery**

To assure consistency, all of the Ontario-Hydro train components were prepared and recovered by a single technician. Solutions were made fresh daily and impingers were charged no sooner than 16 hours prior to the sampling. The Hg trains were recovered

immediately at the completion of the sampling and the impinger contents were recovered and preserved (where appropriate) within 2 hours of completing the sampling. A schematic of the recovery scheme is presented in Figure 4-11. The lab technician responsible for this task used prepared recovery sheets that are included in Appendix H.

#### **4.2.5 Flue Gas Sample Logging and Tracking**

The sample logging and tracking activities were under the direct supervision of the Matthew DeVito. Each Hg train sample was recovered and the individual impinger volume and recovery data was reported on field recovery sheets. Mr. DeVito checked these sheets daily. At the completion of the final test and recovery, the samples were placed in protective cardboard shipping boxes and these boxes were placed in the CONSOL vehicle for transportation to the CONSOL R&D laboratory at Library, PA. This vehicle was locked during periods where it was unoccupied by CONSOL personnel. Upon arriving at the CONSOL site, the samples were transferred from the vehicle to the lab room where the Hg analyses were conducted. Each box was unpacked and the samples were placed in the receiving area and grouped by location and date. The samples were then crosschecked with the sample recovery sheets. No samples were lost or broken during transport. Mr. DeVito then completed CONSOL "sample log-in analytical request sheets", required for the lab accounting system. Analytical numbers were then assigned and Carol Simmons, Group Secretary, typed the samples and analytical requests into the lab database. The lab database program generates analytical labels and Mr. DeVito fixed these labels onto each individual sample bottle. Dave Krofchech, the lab technician responsible for the operation of the CVAA, then analyzed the samples. The analytical results are reported on the instrument printouts which also include the results of the calibration curve, blanks, QC checks, duplicate and spike analyses. These data were reviewed by the analyst and Mr. DeVito. Interim results were then reported on the sample log-in sheets. Upon final review, the Group Secretary types these results into the lab database.

The samples were under direct supervision of CONSOL personnel during every step of the sampling, recovery, shipping, analysis, and reporting process. Copies of the sample log-in and database log-in sheets are presented in Appendix I.

#### 4.2.6 Flue Gas Sample Analysis

The analytical phase for the Ontario-Hydro train samples consisted of eight sample runs. Where applicable each matrix was analyzed as an individual sample set. Each sample run consisted of the development of a calibration curve (absorbance versus Hg concentration in solution), checks of field and lab blanks, calibration checks with SRM (standard reference material) and lab standards, selected duplicates and selected sample spikes. The calibration standards were prepared using serial dilution of a  $1,000 \pm 3$   $\mu\text{g/mL}$  single element standard obtained from VHG Labs. The calibration curve was verified using SRM 1641C. The certified Hg concentration of SRM 1641C is  $1.47 \pm 0.04$  mg/L. This material was used to prepare a QC check of 7.35 ng/mL. Additional QC samples of 5.00 and 10.00 ng/mL were prepared from the VHG Hg standard to further verify the calibration curve. The sample spike solution was also prepared from the VHG standard. The certification sheets for these standards are contained in Appendix F. Graphical summaries of the calibration, blanks, and QC checks for each sample run are shown in Figures 4-13 through 4-19. Comprehensive summaries for these sample runs are shown in Appendix J.

A total of ~300 individual Hg determinations were completed. This total included 32 calibration standards, 59 blank samples, 33 SRM or lab QC checks, 10 sample spikes, and 16 duplicate analyses (samples representing pilot plant testing conducted on a flue gas slip-stream are included in this total and also included in the overall lab QA/QC activities). The average blank value was 0.12 ng/mL (ppb in solution) with 33 of the samples showing 0.00 ppb. The SRM and QC checks showed an average error of 1.6%. The eight duplicate analyses completed on the solid samples showed a percent difference of 11%. The duplicate analyses completed on the liquid samples (KCl and  $\text{KMnO}_4$  samples) showed a 14% difference.

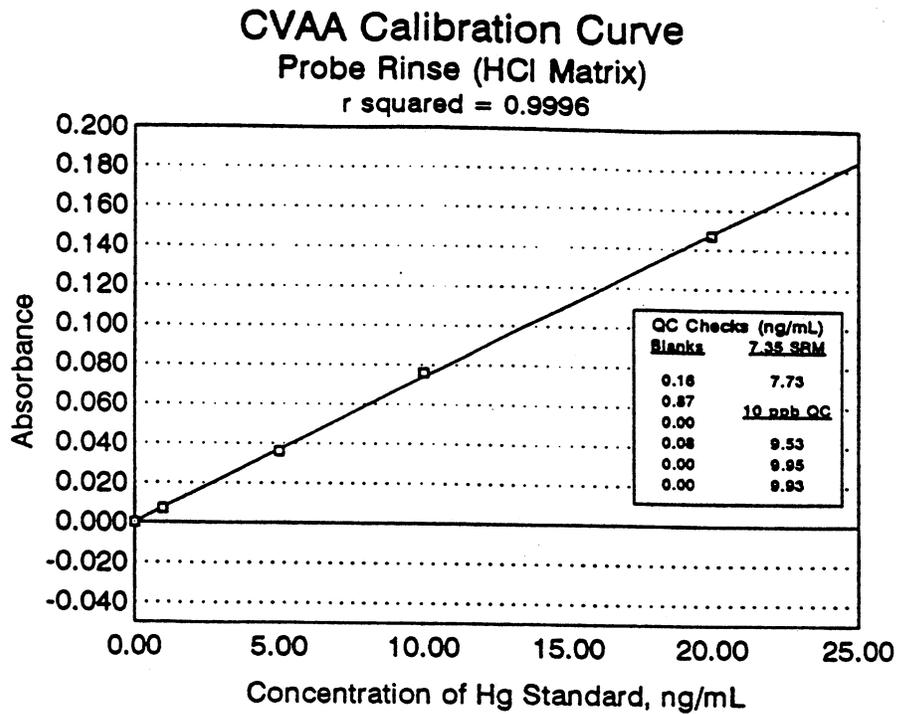


Figure 4-13. QC Data for CVAA Analysis, Probe Rinse.

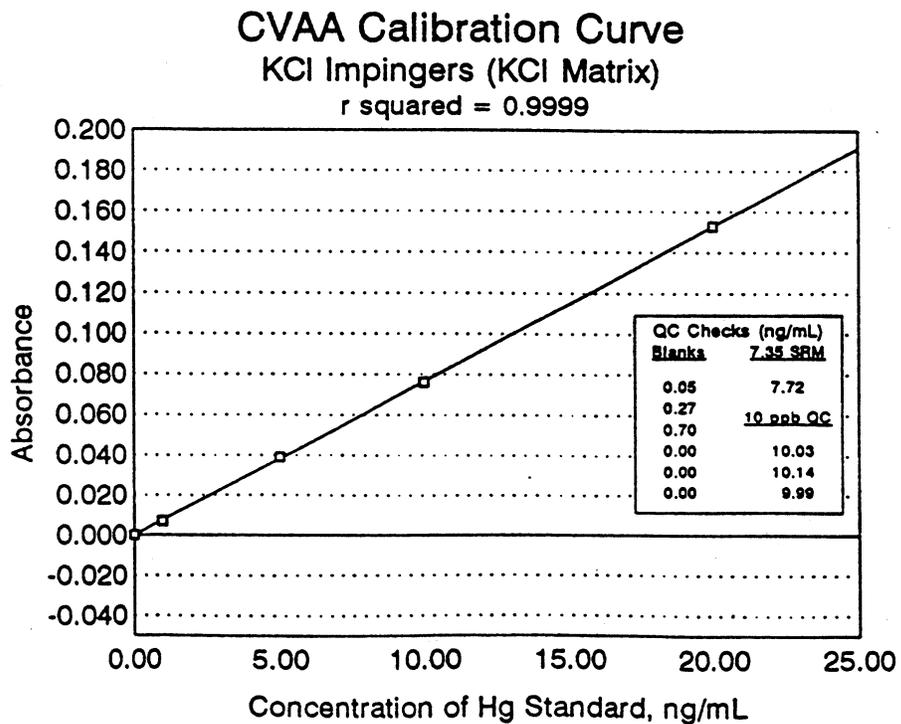


Figure 4-14. QC Data for CVAA Analysis, KCl Impinger.

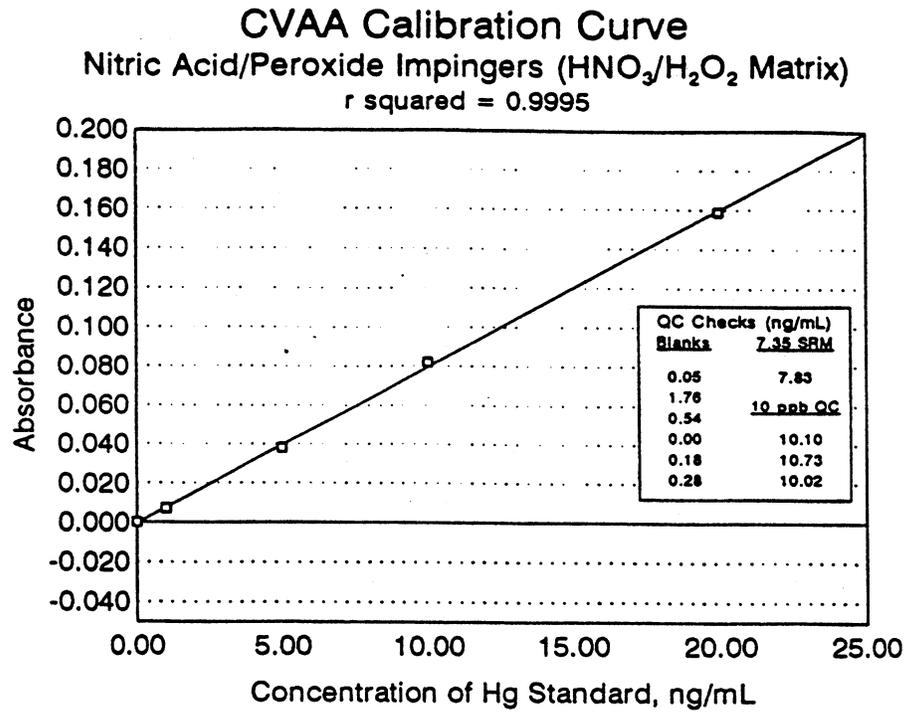


Figure 4-15. QC Data for CVAA Analysis, Nitric Acid/Peroxide Impinger.

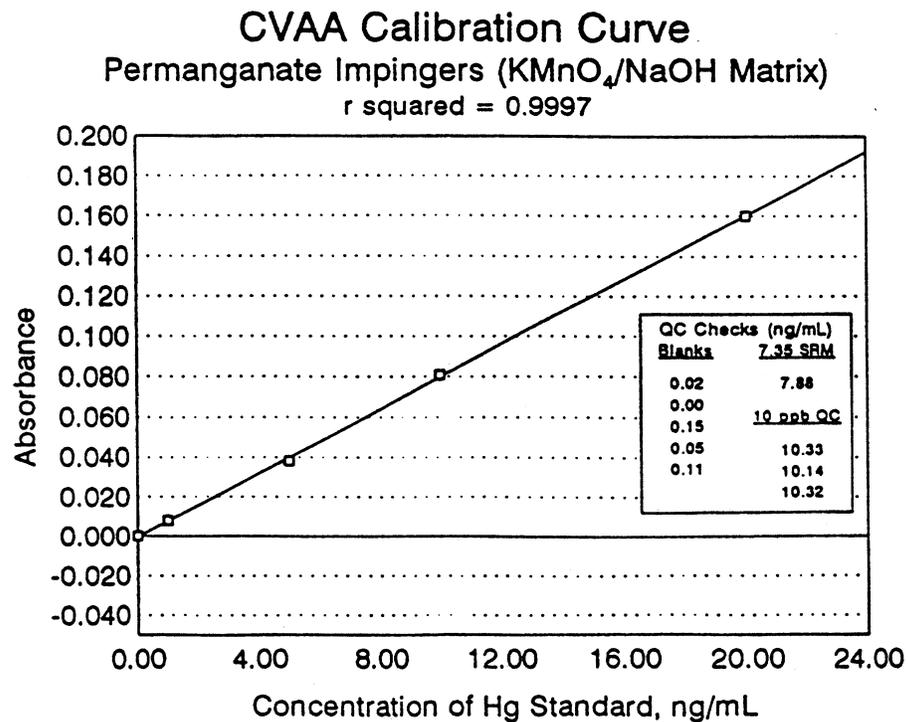


Figure 4-16. QC Data for CVAA Analysis, Potassium Permanganate Impinger.

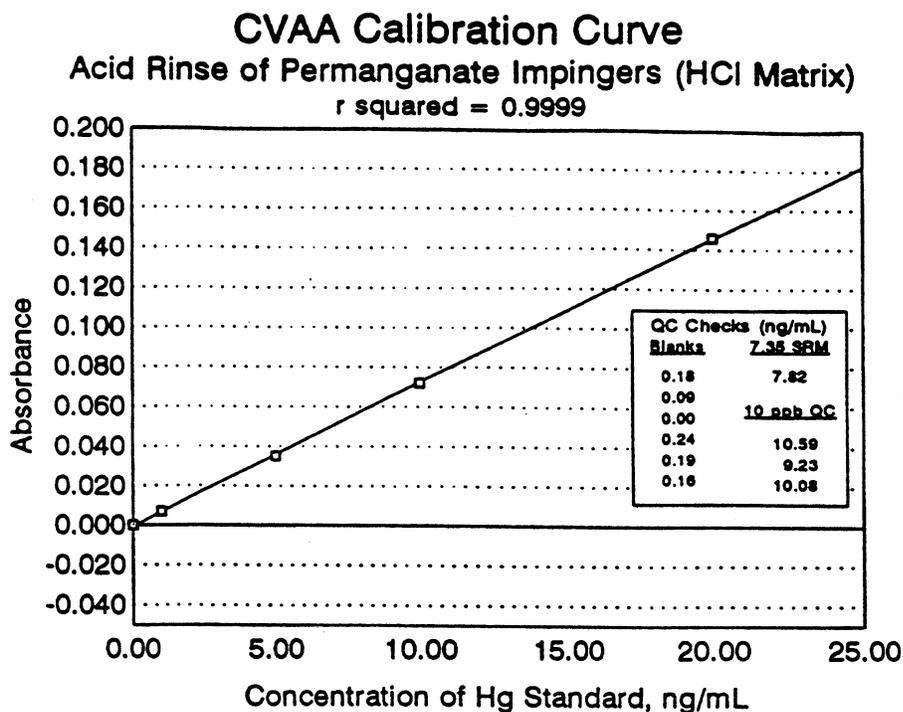


Figure 4-17. QC Data for CVAA Analysis, HCl Rinse.

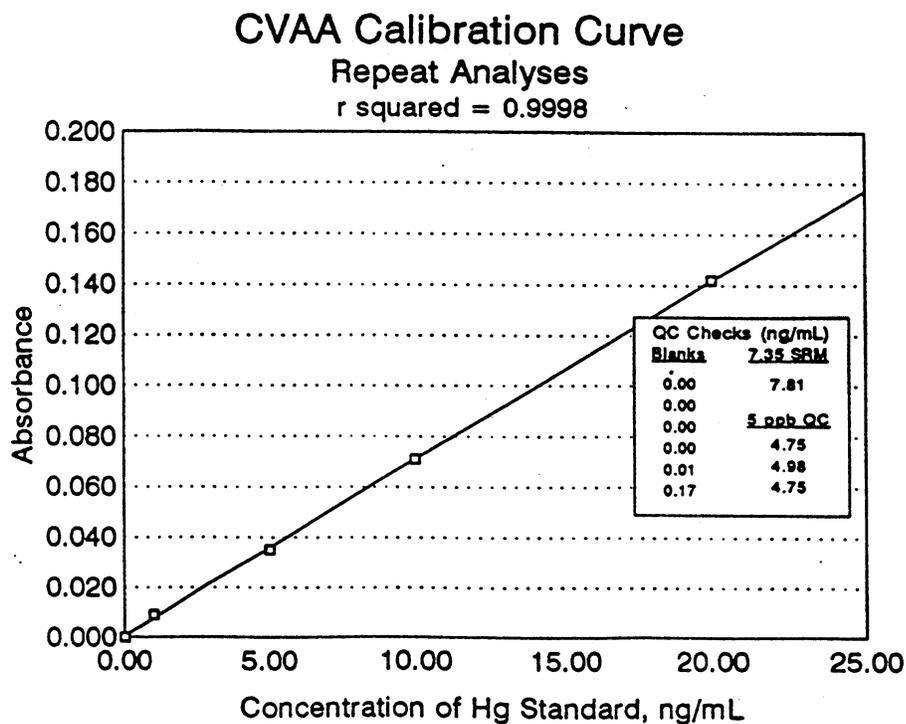
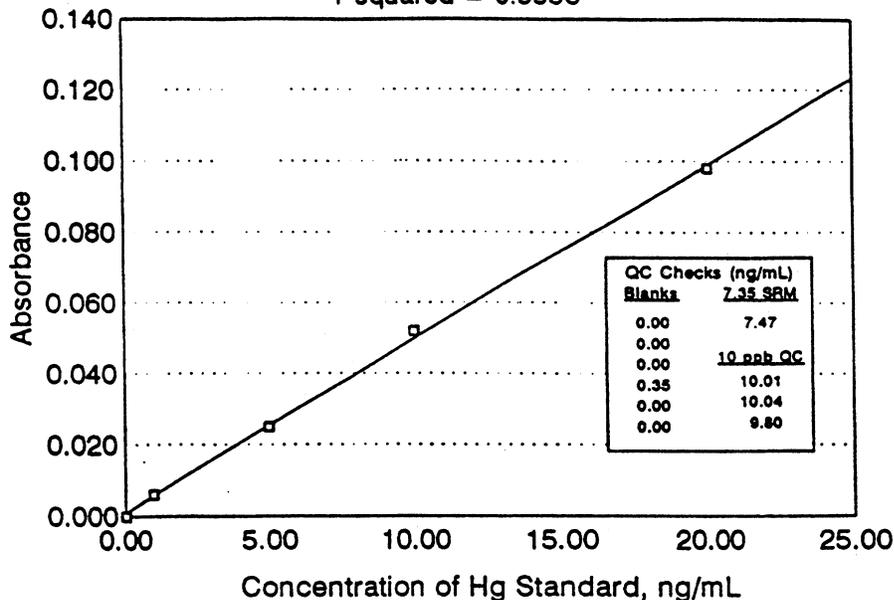


Figure 4-18. QC Data for CVAA Analysis, Impinger Repeats.

### CVAA Calibration Curve

Solids Analyses - 1st Set

r squared = 0.9998



### CVAA Calibration Curve

Solids Analyses - 2nd Set

r squared = 0.9996

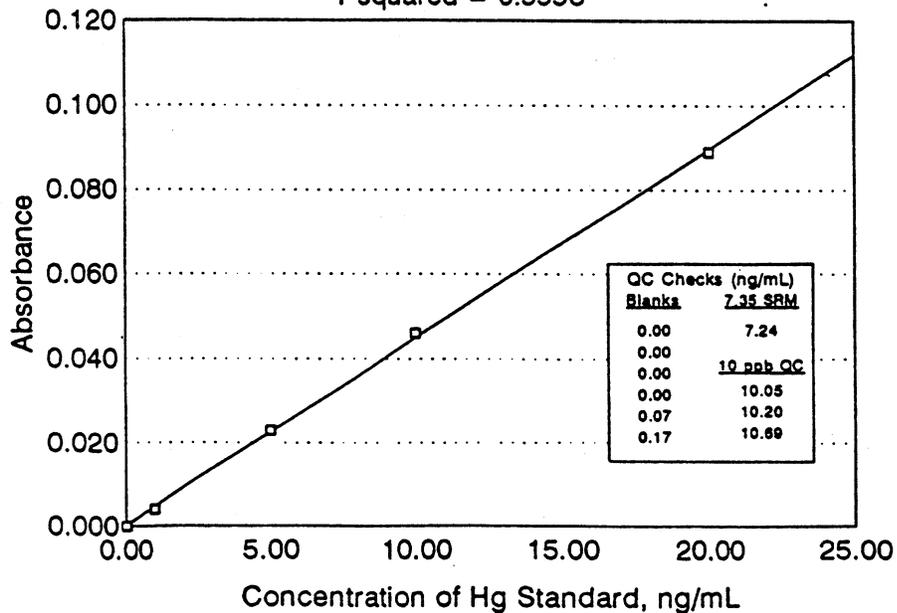


Figure 4-19. QC Data for CVAA Analysis, Solid Samples.

#### 4.2.7 Flue Gas Duplicate Analyses

Duplicate analyses were completed on selected samples to determine the repeatability and ultimately the uncertainty for the Hg measurements. The results of the duplicate analyses for the train components containing significant levels of Hg (solids, KCl and  $\text{KMnO}_4$  impingers) are shown in Table 4-19, 4-20 and 4-21.

**Table 4-19. Summary of Duplicate Solids Hg Analysis**

Matrix	Lab ID Number	Rep #1	Rep #2	% Diff.
		(ppm weight basis)		
Filter Solids	985667	0.26	0.24	8.0
Cyclone Solids	985671	0.10	0.11	9.5
Probe Solids	985675	0.29	0.26	10.9
Cyclone Solids	985676	0.14	0.14	0.0
Cyclone Solids	985682	0.15	0.14	6.9
Probe Solids	985684	0.29	0.25	14.8
Cyclone Solids	985689	0.11	0.10	9.5
Cyclone Solids	985696	0.10	0.13	26.1
Average	-----	-----	-----	10.7

The average % difference for the eight pairs of duplicates is 11%. The Hg concentration attributed to solids at the baghouse inlet averaged  $3.00 \mu\text{g}/\text{m}^3$  for the isokinetically collected samples and  $1.60 \mu\text{g}/\text{m}^3$  for the "reverse nozzle" samples. An 11% variability in the repeat solids analysis represents just 0.18 to  $0.33 \mu\text{g}/\text{m}^3$  uncertainty on an individual sample basis.

Table 4-20. Summary of Duplicate KCl Impinger Hg Analysis.

Matrix	Lab ID Number	Rep #1	Rep #2	Absolute Difference	% Diff.
KCl Impinger	985786	1.96	2.34	0.38	17.7
KCl Impinger	985801	2.22	1.17	1.05	62.0
KCl Impinger	985806	4.36	3.02	1.34	36.3
KCl Impinger	985831	2.53	2.70	0.17	6.5
KCl Impinger	985846	4.84	5.79	0.95	17.9
Average	----	----	----	0.78	28.1

The average % difference for selected sample duplicates was 28%. While this is slightly higher than our data quality objective of 20%, the average absolute difference was 0.78 ng/mL. This is within our data quality objectives for samples containing less than 5 ng/mL of Hg. In theory, the KCl impingers collect the oxidized Hg species. The observed flue gas speciation at the baghouse inlet showed a oxidized Hg concentration of 1.95  $\mu\text{g}/\text{m}^3$ . A 28% uncertainty in sample duplicates represents an absolute uncertainty in the reported oxidized concentration at the baghouse inlet of 0.55  $\mu\text{g}/\text{m}^3$ . The observed flue gas speciation at the baghouse outlet showed a oxidized Hg concentration of 3.14  $\mu\text{g}/\text{m}^3$ . A 28% uncertainty in sample duplicates represents an absolute uncertainty in the reported oxidized concentration at the baghouse outlet of 0.88  $\mu\text{g}/\text{m}^3$ . The high average percent difference is a results of the repeats for sample ID# 985801. For this particular sample, the Hg concentration was just slightly above our detection limit. Our lab protocol is capable of reproducing results within 1.0 ng/mL. In cases where the sample concentration is low (<5 ng/mL), analytical repeatability of <20% is difficult to obtain.

**Table 4-21.** Summary of Duplicate KMnO<sub>4</sub> Impinger Hg Analysis.

Matrix	Lab ID Number	Rep #1	Rep #2	Absolute Difference	% Diff.
			(ng/mL)		
KMnO <sub>4</sub> Impinger	985803	16.0	15.1	0.9	5.8
KMnO <sub>4</sub> Impinger	985808	20.3	20.9	0.6	2.9
KMnO <sub>4</sub> Impinger	985838	19.4	24.7	5.3	24.0
KMnO <sub>4</sub> Impinger	985847	27.0	23.7	3.3	13.0
KMnO <sub>4</sub> Impinger	985853	20.2	22.2	2.0	9.4
Average	----	----	----	2.42	11.0

The average % difference for selected sample duplicates was 11%. This is well within our data quality objective of 20%. In theory, the KMnO<sub>4</sub> impingers collect the elemental Hg species. The observed flue gas speciation at the baghouse inlet showed an elemental Hg concentration of 5-6 µg/m<sup>3</sup>. An 11% uncertainty in sample duplicates represents an absolute uncertainty in the reported oxidized concentration at the baghouse inlet of 0.5 to 0.6 µg/m<sup>3</sup>. The observed flue gas speciation at the baghouse outlet showed a oxidized Hg concentration of 0.44 µg/m<sup>3</sup>. An 11% uncertainty in sample duplicates represents an absolute uncertainty in the reported oxidized concentration at the baghouse inlet of 0.05 µg/m<sup>3</sup>.

#### 4.2.8 Flue Gas Analysis Spike Recovery

Selected samples were spiked with a 5 ppb Hg standard and then re-analyzed to determine the percent spike recovery. The results of these QA/QC procedures are shown on Table 4-22.

**Table 4-22. Analytical Spike Sample Recovery Summary.**

Matrix	Lab ID Number	Concentration [ ppb ]	Concentration with 5 ppb Spike [ ppb ]	Recovery [ % ]
Probe Rinse	985800	0.65	5.22	91
KCl Impinger	985801	0.22	5.01	96
Nitric Impinger	985802	0.07	4.97	98
KMnO <sub>4</sub> Impinger	985803	0.78	6.38	112
HCl Rinse	985804	0.26	5.06	96
Probe Rinse	985840	0.61	4.91	86
KCl Impinger	985841	0.47	5.37	98
Nitric Impinger	985842	0.25	5.11	97
KMnO <sub>4</sub> Impinger	985843	2.74	7.65	98
HCl Rinse	985844	0.28	4.78	90
AVERAGE				96

The average spike recovery was 96%. This is well within the data quality objectives of 20%.

#### 4.2.9 Flue Gas Hg Concentration Detection Limits

The flue gas Hg concentration is calculated using the following equation:

$$\text{Hg [ } \mu\text{g/m}^3 \text{ ]} = \frac{C_{\text{imp}} \times V_{\text{imp}}}{V_{\text{gas}} \times 1000} \quad \text{Equation 1}$$

where:  $C_{\text{imp}}$  = Hg concentration of impinger solution [ ng/mL (ppb) ]

$V_{\text{imp}}$  = Liquid volume of impinger solution [ mL ]

$V_{\text{gas}}$  = Flue gas sample volume [ dry standard m<sup>3</sup> ]

1000 = Conversion factor [1000 ng per  $\mu\text{g}$  ]

From equation 1, the flue gas Hg detection limit is enhanced with increased flue gas sample volume, smaller liquid volumes, and smaller analytical detection limits. The CVAA is calibrated between 0 and 20 ng/mL. Over this range the calibration coefficient

between absorbance and concentration is linear. The lowest standard used in the calibration curve is 1.00 ng/mL. While it is acceptable to interpolate between 0 and 1 ng/mL, we have more confidence using 0.5 ng/mL as the lowest measurable concentration. This lower limit of quantification (LLQ) is constant for all of the impinger matrices. The prescribed sampling and recovery procedures result in final impinger volumes varying between 70 and 700 mL. Due to sampling constraints, the volume of flue gas sampled varied between 1.2 and 2.6 dscm. These variables result in sample-specific flue gas detection limits. With this noted, the minimum flue gas Hg detection limit for each sample matrix are listed in Table 4-23.

**Table 4-23. Minimum Hg in Flue Gas Detection Limits.**

Matrix	Typical Liquid Volume [ mL ]	Typical Gas Volume [ dscm ]	Flue Gas Hg Detection Limit [ $\mu\text{g}/\text{m}^3$ ]
Probe Rinse	125	1.2	0.05
KCl Impinger	600	1.2	0.25
HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> Impingers	175	1.2	0.07
KMnO <sub>4</sub> Impingers	250	1.2	0.10
HCl Rinse	100	1.2	0.04

The KCl matrix has the highest detection limit. This is due to the fact that this matrix has the largest solution volume. All of the measured flue gas concentrations for the KCl fractions were 3 to 12 times greater than the detection limit. The flue gas detection limit for the other matrices is low enough to be insignificant in the flue gas calculations.

#### 4.2.10 Flue Gas Review of Interim Data

Interim data was reviewed during every phase of the project to assure sample completeness and accuracy. The field sampling results were reviewed at the completion of each flue gas measurement and gas flow rate, dscf of sample volume, % moisture in flue gas, and % Isokinetics were calculated to verify sample quality. Upon inspection of the impinger recovery data for the second stack measurement, it was evident that some "back-flushing" had occurred. With this knowledge, an immediate decision was made to obtain an additional stack measurement. Upon reviewing the calculated Hg speciation data for all four stack measurements, the back-flush apparently had no impact.

The laboratory results were reviewed on a daily basis while the analysis were in progress. These results were used in data reduction spreadsheets to identify possible inconsistencies with the Hg flue gas data. As appropriate, suspect samples were re-analyzed.

#### 4.2.11 Flue Gas Computerized Data Reduction

The CONSOL field sampling team used a variety of customized data reduction spreadsheets in the calculation of the field sampling data and the Hg concentration data. The output from these spreadsheets is presented in Appendix K.

#### 4.2.12 Data Review

The field data and Hg sampling results were reviewed and checked by Mr. DeVito and Mr. Oda. Mr. Oda prepared the final report to ADA with assistance by Mr. DeVito. Dr. Robert Statnick also reviewed the final report prior to distribution to ADA Technologies, Inc.

### 5.0 Internal QA/QC Activities

As mentioned in Section 4.2, process test methods, all flue gas samples were logged and tracked under the direct supervision of Matthew DeVito. All flue gas samples were logged as returned by CONSOL's laboratory accounting system. Copies of the flue gas sample login and database login sheets are presented in Appendix I.

ADA Technologies collected coal and ash samples during testing. A complete set of coal and ash logs can be found in Appendices A and F. A complete set of ash logs can be found in Appendices B and F.

Hazen laboratories performed the coal pulverization and ultimate, proximate, BTU and Cl analyses. The chain of custody sheet can be found in Appendix F. Please note that while the coal chain of custody has not been signed as returned to ADA Technologies, Sheila Haythornwaite accepted receipt. E-mail was used to confirm sample custody of the ash and coal samples sent to FGS for Hg analysis. Copies of these e-mails are no longer available.

### **5.1 QA/QC Problems**

There are no QA/QC problems to report.

### **5.2 QA Audits**

There were no QA audits performed during testing. All of the flue gas sampling equipment calibrations were completed by CONSOL sample personnel. These results were discussed in Section 4.2.2.