



May 10, 1999

Mr. Bill Maxwell
U.S. Environmental Protection Agency
Room 1114
411 West Chapel Hill
Durham, North Carolina 27701

Re: Results of Flue Gas Mercury Measurements
Lower Colorado River Authority
Sam K. Seymour Unit 3

Dear Mr. Maxwell:

Attached for your review is a report of flue gas mercury speciation measurements made on the Lower Colorado River Authority's (LCRA) Sam K. Seymour Unit 3. This sampling was conducted by Radian International on December 2 and 3, 1998, as part of a U.S. Department of Energy and Electric Power Research Institute study to evaluate flue gas mercury oxidation catalysts. The sampling was also conducted in a manner expected to meet the requirements of Part III the U.S. Environmental Protection Agency's (EPA) mercury Information Collection Request (ICR).

The LCRA is requesting written approval from the EPA to use the mercury sampling results contained in the attached report in lieu of conducting additional testing to meet the requirements of the ICR. As we discussed last week, the LCRA will delay the submittal of a site-specific test plan and a quality assurance project plan until you have reviewed the report and responded to this request. If EPA decides additional unit testing is still required, the LCRA will prepare the plans and submit them within approximately 30 days of receiving notice.

The LCRA appreciates your consideration of this request. If you have any questions or a need for additional information, please contact me at (512) 473-3272.

Sincerely,

Joe Bentley
GenCo Environmental Program Leader

Attachment

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RESULTS OF FLUE GAS MERCURY MEASUREMENTS

AT SAM K. SEYMOUR STATION, UNIT 3

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May 1999

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EXECUTIVE SUMMARY

Flue gas mercury speciation measurements were made on Unit 3 at the Lower Colorado River Authority's Sam K. Seymour Station on December 2 and 3, 1998. The sampling was conducted to determine if Unit 3 would be a suitable host site for a test program that is being co-funded by the U.S. Department of Energy and EPRI to evaluate flue gas mercury oxidation catalysts. However, the sampling was also conducted in a manner expected to satisfy the requirements of the U.S. Environmental Protection Agency's mercury Information Collection Request for flue gas sampling.

Three simultaneous sample runs were conducted using the Ontario Hydro (KCl) Method for measuring speciated mercury concentrations, at a location downstream of the south boiler induced draft fan (the FGD inlet) and at a second location downstream of the Unit 3 FGD system Module A. The results showed an average total mercury concentration of $12.0 \mu\text{g Hg}/\text{Nm}^3$ and an average gas phase elemental mercury concentration of $8.8 \mu\text{g Hg}/\text{Nm}^3$ in the flue gas upstream of the FGD system. These values correspond to an average mercury oxidation percentage of 27% at the FGD inlet.

Samples of the coal fired in Unit 3 were collected during each sampling run. The coal mercury content averaged 123 ppb (dry basis) in these three samples. Material balance calculations were conducted to compare the mercury in the fired coal versus that measured in the flue gas, and showed good closure (an average closure of 98%, with individual sample run closures of 65%, 110% and 121%).

The flue gas sample runs at the FGD outlet location indicated an average total mercury concentration of $9.3 \mu\text{g Hg}/\text{Nm}^3$ and an average gas phase elemental mercury concentration of $9.0 \mu\text{g Hg}/\text{Nm}^3$. As expected based on results from other sites, the FGD system on Unit 3 was observed to remove a high percentage (93%) of the oxidized mercury in the flue gas at the FGD inlet, and essentially none of the elemental mercury. The overall mercury removal across the FGD system averaged 23%.

There was only one problem during the sampling runs that resulted in a concern about data quality. The sampling train failed its post-run leak check after the first run at the FGD inlet location. At first it was thought that the leak developed when the sampling probe was removed from the duct at the end of the run. However, higher flue gas oxygen content and lower flue gas mercury concentrations measured for this run compared to those of the other two runs at this location suggest that the leak occurred while the sample was being collected. The first run

also showed the poorest closure in the coal mercury material balance calculations mentioned above (65%).

These results confirmed that Unit 3 at the Seymour Station would be a suitable host site for mercury oxidation catalyst testing, as the flue gas has an adequate elemental mercury concentration to support such testing.

1.0 INTRODUCTION

1.1 SUMMARY OF TEST PROGRAM

This report describes the results of flue gas mercury concentration and speciation measurements conducted by Radian International at Unit 3 of the Lower Colorado River Authority's (LCRA's) Sam K. Seymour, Jr. Station in December of 1998. These measurements were made primarily to determine if Unit 3 at Seymour Station would be a suitable host site for a project co-funded by the U.S. Department of Energy and EPRI (Contract No. DE-AC22-95PC95260) to investigate mercury oxidation catalysts.

However, the testing was also conducted in a manner that was expected to make the results suitable for reporting to the U.S. Environmental Protection Agency as part of the Information Collection Request for Electric Utility Steam Generating Unit Mercury Emissions Information Collection Effort (Mercury ICR). As such, the procedure used to make the flue gas mercury measurements was the "Standard Test Method for Elemental, Oxidized, Particle-Bound, and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)." Quality assurance/quality control (QA/QC) procedures employed were equivalent to what was expected to be specified in an EPA-approved Quality Assurance Project Plan.

Sam K. Seymour, Jr. Station is located approximately 9 miles southeast of LaGrange, Texas, adjacent to State Highway 71. Unit 3 is a dry-bottom, tangential-fired boiler that was built by Combustion Engineering (now part of ABB) and a steam turbine/generator set with a nameplate rating of 460 MW. The Unit 3 boiler typically fires a subbituminous coal from the Wyoming Powder River Basin.

The Unit 3 boiler has provisions for overfire air for NO_x controls, a cold-side ESP without flue gas conditioning for particulate control, and a wet flue gas desulfurization (FGD) unit for control of SO₂ emissions. The FGD system is the last air emissions control device installed in the flue gas stream from Unit 3, so the speciated mercury concentration testing was conducted on the FGD inlet and outlet streams. Because of duct configurations and sample port locations on Unit 3, the sampling was actually conducted on the outlet from one of two induced draft (ID) fans between the ESP and FGD inlet, and at the outlet of one of two operating FGD absorbers. The flue gas at these two locations was sampled simultaneously on three occasions over the time period December 2-3, 1998. Coal samples were also collected concurrent with each sampling period, for subsequent analyses of mercury content and other species.

1.2 KEY PERSONNEL

The key personnel that coordinated this sampling effort included:

- LCRA Project Manager - Bill Webb (409-249-8388)
- Radian Project Manager - Gary Blythe (512-419-5321)
- Radian Task Leader for Site Testing - Dr. Carl Richardson (512-419-5966)
- Radian Lead Source Sampler - Andrew O'Brien (512-419-5801)

2.0 PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 PROCESS DESCRIPTION AND OPERATION

As mentioned in the introduction, Unit 3 at the Seymour Station has a coal-fired boiler and a turbine/generator set with a nameplate rating of 460 MW. The boiler is a balanced-draft, tangential-fired unit built by Combustion Engineering. The boiler was designed to fire a relatively low-quality Texas lignite, that was to be mined near the plant site. However, the mine was never opened, and Unit 3 has fired Powder River Basin subbituminous coals since it was placed in service in 1988. Unit 3 has a base coal contract with the Cordero mine in Campbell County, Wyoming, but supplements that base contract with Powder River Basin coals purchased on the spot market.

Because the Powder River Basin coals are in general much higher in quality than the design lignite, the boiler and downstream air emissions control equipment are somewhat over-designed. For example, the Unit 3 boiler furnace is essentially the same size as those on Units 1 and 2, but Units 1 and 2 are 600-MW units that were designed to fire only Powder River Basin coals. The FGD system on Unit 3 was designed for a coal sulfur content of up to 10 lb SO₂ per million Btu heat input, but the actual coal sulfur level is less than 1 lb per million.

During the mercury speciation measurements, Unit 3 operated at net loads of 315 to 358 MW, which correspond to about 70 to 80% of full load. Coal sulfur levels were analyzed at 0.36 to 0.41% on an as-received basis, and the coal heat content was 9016 to 9245 Btu/lb on the same basis.

2.2 CONTROL EQUIPMENT DESCRIPTION

Figure 2-1 illustrates the flue gas path on Unit 3. The Unit 3 boiler has provisions for overfire air for NO_x controls, and a cold-side ESP supplied by Combustion Engineering for particulate control. The ESP operates with no flue gas conditioning. Flue gas leaving the ESP splits into two streams that go to two boiler induced-draft (ID) fans. From the ID fan outlets, the two streams rejoin and most of the flue gas goes to a wet flue gas desulfurization (FGD) system. The wet FGD system was also installed by Combustion Engineering, and uses spray absorbers for flue gas contactors. The FGD reagent is finely ground limestone slurry, and the FGD system operates in a forced oxidation mode to produce a gypsum byproduct. The FGD system has three absorbers installed, with two normally operating and a third on standby. The flue gas leaving the two absorbers is mixed with a small amount of ID fan exit gas that is bypassed. The bypass rate is adjusted to control the temperature of the stack gas to about 160°F while maintaining SO₂ emissions compliance. The amount of

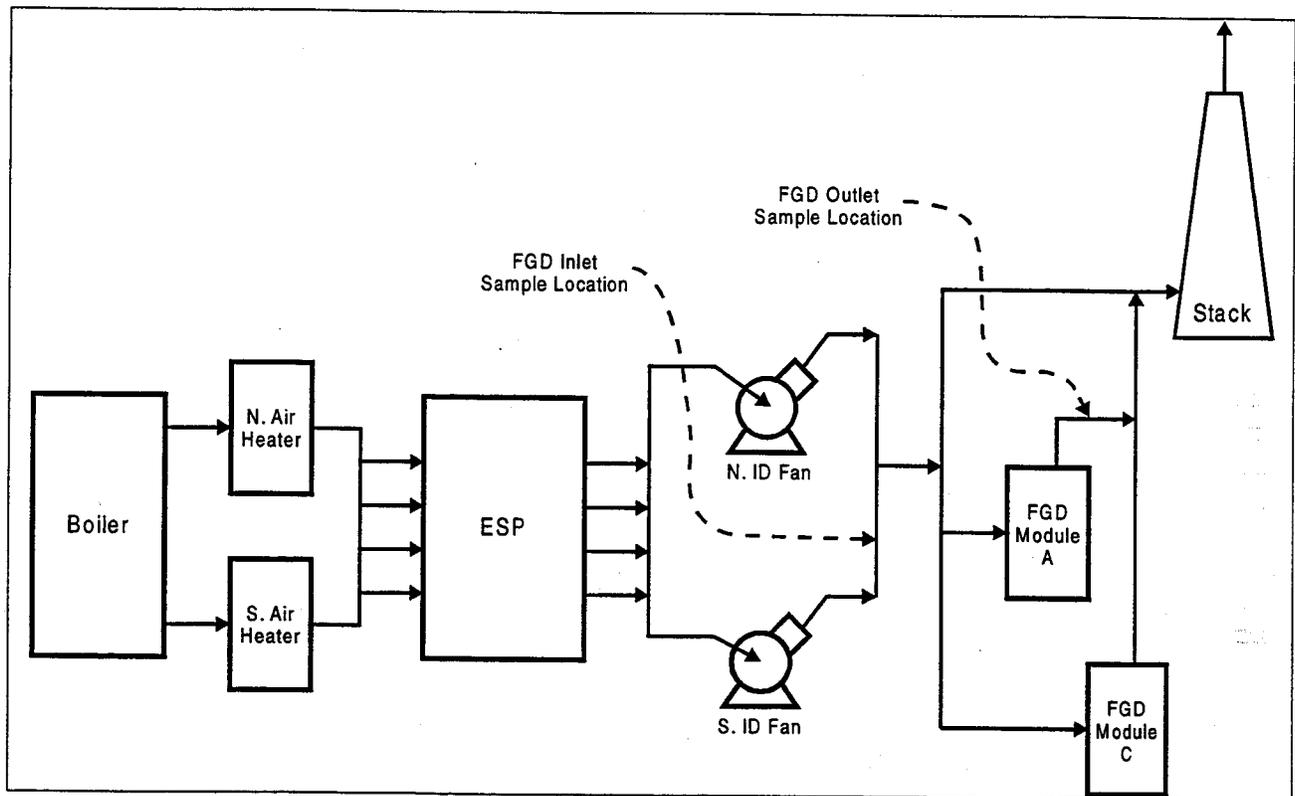


FIGURE 2-1. FLUE GAS PATH ON UNIT 3

bypass is typically about 15 to 20 percent of the total flow exiting the ID fans. The design SO_2 removal values for the FGD system were 90% SO_2 removal across the FGD system while treating 94% of the boiler flue gas.

During the mercury speciation measurements, the FGD system operated with Modules A and C in service, and overall SO_2 removal levels of 81.2 to 88.4 percent. Note that these overall SO_2 removal levels include the effects of flue gas bypass. That is, the FGD outlet SO_2 concentration is measured at the stack, after the flue gas treated in the FGD system is mixed with the bypass gas. The FGD inlet sulfur levels ranged from 0.69 to 0.73 lb SO_2 per million Btu heat input, while the stack values ranged from 0.08 to 0.13 lb per million.

Analyses of coal samples collected during the mercury speciation measurements indicated sulfur levels in the range of 0.78 to 0.91 lb SO_2 per million Btu, which are higher than what was indicated by the continuous emissions monitoring (CEM) system at the FGD system inlet. This may indicate that a small percentage (10 to 15%) of the coal sulfur is retained on the alkaline ash produced by the coal through chemical reactions in the boiler and ESP.

The Unit 3 ESP operation was not monitored during the testing, but the ESP outlet particulate emissions continually remain below the regulated value of 0.03 lb particulate per million Btu of heat input, and the opacity is typically below 10 percent. The plant reports a typical outlet emission rate of 0.01 lb of particulate per million Btu of heat input on their 1997 Form EIA-767.

2.3 FLUE GAS SAMPLING LOCATIONS

The FGD system is the last air emissions control device installed in the flue gas stream from Unit 3, so the speciated mercury concentration testing was conducted on the FGD inlet and outlet streams. Because of duct configurations and sample port locations on Unit 3, the sampling was actually conducted on the outlet from one of two induced draft (ID) fans (the south fan) for the FGD inlet location, and at the outlet of one of two operating FGD absorbers (Module A) for the FGD system outlet location. These locations were identified previously on Figure 2-1.

2.3.1 FGD Inlet. The inlet location was chosen because existing ports on the south ID fan outlet duct were readily accessible, and because this was the very duct location that was proposed for conducting mercury oxidation testing during the DOE/EPRI project. A shortcoming of this location is that only half of the Unit 3 flue gas could be sampled from this duct run, as there is another identical duct run from the north ID fan that conveys the other half of the flue gas to a common plenum. The actual FGD inlet duct would have represented a challenging sampling location because of more difficult duct access, larger duct dimensions, and the fact that the sample ports are immediately downstream of a major flow disturbance. Those ports are immediately downstream (by less than one duct diameter) of the small plenum area where the flue gas from the two ID fans mix and the FGD inlet and bypass gas separate. It was expected that the gas flow at this location would be poorly distributed because of the significant flow disturbance immediately upstream. Because of the limited number of ports and limited clearance, it would not have been possible to do a multi-port traverse at this location.

It was decided that there would be little risk associated with sampling only half of the flue gas in the duct from the south ID fan. The Unit 3 boiler has a single furnace cavity (i.e. does not have a divided or "twin" furnace) and the tangential burner arrangement imparts a swirl to the furnace gas that promotes mixing of the gas before it splits into two streams downstream of the economizer. Therefore, it was decided that the flue gas from the south ID fan should be representative of the entire FGD inlet gas stream.

The south ID fan outlet duct is 16'-9" tall and 19'0" wide (17.8 ft equivalent diameter) in the horizontal run where the ports are located. The ports are 4-inch diameter pipe nipples fitted with 4-inch, 150-lb flanges on the ends. The distance from the flange face to the internal wall of the duct is 26 inches. Eight ports are equally spaced across the 19-ft duct width. The nearest upstream flow disturbance is a 90° elbow in the duct as it turns from vertical gas flow from the ID fan outlet to horizontal gas flow in the duct run sampled. This flow disturbance is 34 ft. (1.9 duct diameters) upstream of the sampling location. The nearest downstream location is the plenum area where the two ID fan streams mix; this disturbance is 8.3 ft. (0.47) duct diameters downstream of the sampling location.

Prior to sampling, a 64-point velocity/temperature traverse was conducted at this location according to Method 1 to measure the gas velocity and flow rate, and to determine a suitable point of average velocity. The Ontario Hydro method sampling was conducted at a single point of average velocity, rather than conducting a multi-port, multi-point traverse of the outlet duct as is recommended in the method. This decision was made because only 10-ft sampling probes and probe liners were readily available, and time constraints prohibited ordering additional equipment. The flue gas should be well mixed downstream of the ID fan, so this change should not have compromised data quality. Another change to the Ontario Hydro method was that previously unused, heat-traced Teflon tubing with glass sockets was used to transfer sample gas from the filter exit to the impinger train, rather than connecting the impinger train directly to the filter outlet. This was necessary because vertical sampling was conducted with 10-ft. probes and liners. It would have been impractical and unsafe to attach the impinger train directly to the filter exit.

A total of three sampling runs were conducted at this location, simultaneous with sampling runs at the FGD outlet location. Each run lasted 100 minutes, and collected approximately 1.5 dry standard cubic meters of gas.

2.3.2 FGD Outlet. The FGD outlet gas was sampled at the outlet duct of one of two operating FGD absorbers. There was some discussion as to whether the intent of the Information Collection Request was to measure stack gas, or the actual gas exiting the FGD system. The wording in the letter from EPA is ambiguous. The difference between the two locations is that the stack gas contains a mixture of FGD outlet gas and bypass gas, with the proportions of each possibly varying from day to day according to FGD system performance, coal sulfur and heat content, ambient temperatures, etc.

It was decided to sample the actual FGD system outlet gas, for several reasons. First is that measuring the FGD system outlet gas provides a measure of the true performance of the control device for removal of mercury species. Second, based on previous Radian experience, it was expected that the wet scrubbers would remove a high percentage of the oxidized mercury in the inlet gas, and little or none of the elemental mercury. Sampling the FGD system exit gas rather than the stack gas allowed this expectation to be confirmed, and provided a measure of the sampling data quality (because the elemental mercury concentrations in the inlet and outlet flue gas should be nearly equal). Sampling the stack gas, which is a mixture of the FGD inlet and outlet gases, would have confounded these effects. Finally, straightforward material and heat balances can be used to directly predict the mercury concentrations at the stack as the amount of bypass varies, from the measurements taken at the FGD inlet and outlet, so it was seen as less important to measure concentrations at that point.

Once it was decided to sample the FGD outlet gas rather than the stack gas, it became necessary to sample the outlet of one of two operating modules (Module A), because there are no ports for sampling the entire FGD outlet gas stream prior to mixing with bypassed gas. This was not seen as a significant compromise, as the two absorbers treat flue gas from a common inlet duct and operate at essentially identical conditions. Thus, the performance of one absorber should be representative of that of the entire FGD system.

The Module A outlet duct is 16'-9" square in cross section in the horizontal run where the ports are located. The ports are 4-inch diameter pipe nipples fitted with 4-inch, 150-lb flanges on the ends. The distance from the flange face to the internal wall of the duct is 26 inches. Eight ports are equally spaced across the 16'-9" duct width. The nearest upstream flow disturbance is a long-radius 90° elbow in the duct as it turns from vertical gas flow at the absorber mist eliminator outlet to horizontal gas flow in the duct run sampled. This flow disturbance is 28.8 ft. (1.7 duct diameters) upstream of the sampling location. The nearest downstream disturbance is the plenum where the absorber streams mix; this disturbance is 19 ft. (1.1 duct diameters) downstream of the sampling location.

Prior to sampling, a 64-point velocity/temperature traverse was conducted at this location according to Method 1 to measure the gas velocity and flow rate, and to determine a suitable point of average velocity. As described above for the FGD inlet location, the Ontario Hydro method sampling was conducted at a single point of average velocity, rather than conducting a multi-point, multi-point traverse of the outlet duct as is recommended in the method. This decision was made for the same reasons as

were describe above. Also as discussed for the FGD inlet location, previously unused, heat-traced Teflon tubing with glass sockets was used to transfer sample gas from the filter exit to the impinger train because of the vertical sampling configuration.

A total of three sampling runs were conducted at this location, simultaneous with sampling runs at the FGD inlet location. Each run lasted 100 minutes, and collected approximately 1.5 dry standard cubic meters of gas.

2.4 PROCESS SAMPLING LOCATIONS

The only process sampling conducted as part of this testing effort was to collect coal samples coincident with each gas sampling period. Coal sample aliquots were collected during each run from the Unit 3 autosampler.

3.0 SUMMARY AND DISCUSSION OF TEST RESULTS

3.1 OBJECTIVES AND TEST MATRIX

The purpose of this test program was twofold: 1) to verify that elemental mercury concentrations at the FGD inlet on Unit 3 of the Sam K. Seymour Station were high enough to support mercury oxidation catalyst testing at that site, and 2) to collect mercury concentration and speciation data at the FGD inlet and outlet in accordance with the requirements of the U.S. EPA mercury ICR, in anticipation of this site being selected for flue gas testing.

The specific objectives of the test program were to:

- Simultaneously measure flue gas flow rates and concentrations of elemental and oxidized forms of mercury in the flue gas at the Unit 3 FGD system inlet and outlet, and
- Measure mercury concentrations of coal fired during the test period.

Table 3-1 presents the sampling and analytical matrix and sampling log.

TABLE 3-1. SAMPLING MATRIX

| RUN NO. DATE | SAMPLE TYPE | TEST METHOD | LOCATION/CLOCK TIME/SAMPLING TIME (MINUTES) | |
|-----------------|---|------------------------------|---|-------------------------|
| | | | FGD INLET | FGD OUTLET |
| VT1 12/2/98 | Velocity Traverse | Method 1 and 2 | 0930-1115 105 | 1040-1230 110 |
| 1 12/2/98 | Hg ⁰ and Hg ⁺² , PM, Moisture O ₂ /CO ₂ | Ontario Hydro Method 3 | 1425-1605 100 100 | 1425-1605 100 100 |
| 2 12/3/98 | Hg ⁰ and Hg ⁺² , PM, Moisture O ₂ /CO ₂ | Ontario Hydro Method 3 | 0935-1115 100 100 | 0935-1115 100 100 |
| 3 12/3/98 | Hg ⁰ and Hg ⁺² , PM, Moisture O ₂ /CO ₂ | Ontario Hydro Method 3 | 1305-1445 100 100 | 1305-1445 100 100 |

3.2 FIELD TEST CHANGES AND PROBLEMS

There were no field test changes and only one problem worth noting. The sampling train leak rate after Run 1 at the FGD Inlet exceeded the allowable 0.02 cubic feet per minute. This was

apparently due to a fitting coming loose where the Teflon transfer tubing connected to the filter exit. It was speculated that the fitting came loose while the probe was being removed from the duct at the end of the run, and that the leak therefore should not have affected the run data quality. However, the flue gas oxygen content measured by Method 3 for this run was higher than for the other two runs at this location, by 1.0 to 1.5 percentage points. The flue gas CO₂ content values measured by the plant CEM do not indicate that the actual flue gas oxygen concentrations should have been higher during this run, so this is an indication of a leak during the sampling run. Also, the moisture content measured for this run was lower than for the subsequent runs by 2 to 3 percentage points, which is another indicator of a leak. Finally, the mercury concentrations measured for this run were lower than for the other two runs, which provides a third indicator of a leak in the sample train.

Otherwise, the percent isokinetics for all three runs at the inlet and the outlet locations fell within the required $\pm 10\%$, and all three runs at each location went the planned 100 minutes. The plant operation was relatively stable during each run. The biggest unit load change during the run periods was 39 MW (-10%), while the FGD system removal percentage varied by less than 6 percentage points over the duration of any of the runs.

3.3 PRESENTATION OF RESULTS

Plant operating data were collected for the times sampling was conducted. Key operating variables are summarized in Table 3-2. The coal fired during this period was a Powder River Basin blend. Three coal samples from the time of this sampling showed an average heat content of 9122 Btu/lb and a sulfur content of 0.38 wt.%, both on an as-received basis. The coal samples also showed an average mercury content of 123 ppb (dry weight basis) and an average chloride content of 20 ppm (dry weight basis). The coal sample results are summarized in Table 3-3.

Tables 3-4 and 3-5 summarize the results of the velocity traverse data collected on December 2. The tables also note the port and sampling depth of average velocity that were used for the mercury sampling. The data in Table 3-4 show that the gas velocity at the FGD inlet location was slightly biased towards the upper right-hand portion of the duct cross-section, looking in the direction of gas flow. This represents the outer portion of the duct relative to turns made by the gas upstream of this point, so this bias could be expected. The flow near the center of the duct was evenly distributed, though, and any of several

TABLE 3-2. SEYMOUR UNIT 3 OPERATING CONDITIONS

| PARAMETER | RUN 1 | | RUN 2 | | RUN 3 | |
|--|---------|-----------|---------|-----------|---------|-----------|
| | AVERAGE | RANGE | AVERAGE | RANGE | AVERAGE | RANGE |
| Unit Load, net MW | 333 | 315-344 | 358 | 339-378 | 323 | 318-328 |
| FGD Inlet SO ₂ , lb/MM Btu | 0.71 | 0.70-0.72 | 0.71 | 0.69-0.73 | 0.71 | 0.70-0.72 |
| FGD Outlet SO ₂ , lb/MM Btu | 0.13 | 0.13 | 0.11 | 0.08-0.12 | 0.11 | 0.11 |
| FGD SO ₂ Removal, % | 81.4 | 81.2-81.7 | 84.9 | 82.4-88.4 | 84.6 | 84.6-84.7 |
| FGD Inlet CO ₂ , % | 11.5 | 11.4-11.6 | 11.5 | 11.3-11.7 | 11.3 | 11.3 |
| Stack NO _x Conc., lb/MM Btu | 0.28 | 0.26-0.29 | 0.31 | 0.30-0.31 | 0.28 | 0.28 |

TABLE 3-3. COAL ANALYSIS RESULTS

| PARAMETER | RUN 1 | RUN 2 | RUN 3 | AVERAGE |
|--|-------|-------|-------|---------|
| Heat Content, Btu/lb (as received) | 9016 | 9104 | 9245 | 9122 |
| Ultimate Analysis (Wt. % as received): | | | | |
| Moisture | 23.42 | 23.48 | 22.73 | 23.21 |
| Carbon | 52.96 | 53.30 | 53.82 | 53.36 |
| Hydrogen | 3.72 | 3.70 | 3.42 | 3.61 |
| Nitrogen | 1.44 | 1.45 | 0.77 | 1.22 |
| Sulfur | 0.41 | 0.36 | 0.36 | 0.38 |
| Ash | 5.55 | 5.29 | 6.07 | 5.64 |
| Oxygen (difference) | 12.50 | 12.42 | 12.83 | 12.58 |
| Sulfur content, lb SO ₂ /MM Btu | 0.91 | 0.79 | 0.78 | 0.83 |
| Mercury (ppb, dry basis) | 139 | 115 | 114 | 123 |
| Chlorine (ppm, dry basis) | 22 | 19 | 19 | 20 |

**TABLE 3-4. FGD INLET LOCATION VELOCITY TRAVERSE DATA
(GAS FLOW INTO PAGE)**

| SAMPLE POINT DEPTH (IN.) | PORT NUMBER/VELOCITY PRESSURE (IN. H ₂ O)/TEMPERATURE (°F) | | | | | | | |
|-----------------------------------|---|--------|--------|--------|--------|--------|--------|--------|
| | PORT 1 | PORT 2 | PORT 3 | PORT 4 | PORT 5 | PORT 6 | PORT 7 | PORT 8 |
| 38.6 | 0.17 | 0.24 | 0.25 | 0.24 | 0.30 | 0.30 | 0.34 | 0.26 |
| | 301 | 301 | 300 | 295 | 295 | 292 | 290 | 288 |
| 63.7 | 0.22 | 0.21 | 0.24 | 0.22 | 0.29 | 0.33 | 0.37 | 0.33 |
| | 303 | 302 | 301 | 298 | 297 | 293 | 291 | 288 |
| 88.8 | 0.23 | 0.20 | 0.21 | 0.23* | 0.28 | 0.34 | 0.39 | 0.37 |
| | 303 | 302 | 301 | 298 | 297 | 294 | 291 | 289 |
| 113.9 | 0.17 | 0.18 | 0.19 | 0.23 | 0.23 | 0.32 | 0.40 | 0.37 |
| | 303 | 302 | 301 | 299 | 297 | 294 | 291 | 289 |
| 139.1 | 0.18 | 0.18 | 0.16 | 0.19 | 0.22 | 0.29 | 0.36 | 0.34 |
| | 303 | 302 | 300 | 299 | 297 | 295 | 292 | 289 |
| 164.2 | 0.18 | 0.15 | 0.13 | 0.18 | 0.20 | 0.26 | 0.30 | 0.34 |
| | 302 | 302 | 300 | 298 | 296 | 295 | 292 | 289 |
| 189.3 | 0.17 | 0.15 | 0.13 | 0.15 | 0.16 | 0.24 | 0.25 | 0.30 |
| | 302 | 302 | 300 | 298 | 296 | 295 | 292 | 289 |
| 214.4 | 0.14 | 0.13 | 0.13 | 0.12 | 0.13 | 0.25 | 0.25 | 0.30 |
| | 301 | 302 | 300 | 297 | 295 | 293 | 292 | 289 |
| Duct Average | 0.23 | | | | | | | |
| | 297 | | | | | | | |

* Port/sampling depth of average velocity used for Ontario Hydro Method mercury speciation sampling.

**TABLE 3-5. FGD OUTLET LOCATION VELOCITY TRAVERSE DATA
(GAS FLOW INTO PAGE)**

| SAMPLE POINT DEPTH (IN.) | PORT NUMBER/VELOCITY PRESSURE (IN. H ₂ O)/TEMPERATURE (°F) | | | | | | | |
|-----------------------------|---|--------|--------|--------|--------|--------|--------|--------|
| | PORT 1 | PORT 2 | PORT 3 | PORT 4 | PORT 5 | PORT 6 | PORT 7 | PORT 8 |
| 38.6 | 0.53 | 0.64 | 0.61 | 0.63 | 0.57 | 0.34 | 0.73 | 0.68 |
| | 138 | 139 | 141 | 136 | 136 | 136 | 143 | 140 |
| 63.7 | 0.59 | 0.65 | 0.53 | 0.53 | 0.58 | 0.60 | 0.61 | 0.66 |
| | 137 | 138 | 140 | 135 | 135 | 136 | 143 | 140 |
| 88.8 | 0.52 | 0.58 | 0.41 | 0.48 | 0.49 | 0.50 | 0.48 | 0.62 |
| | 137 | 136 | 138 | 130 | 135 | 134 | 143 | 141 |
| 113.9 | 0.41 | 0.47 | 0.30 | 0.32 | 0.34* | 0.43 | 0.40 | 0.45 |
| | 137 | 135 | 137 | 129 | 134 | 134 | 141 | 142 |
| 139.1 | 0.40 | 0.27 | 0.22 | 0.17 | 0.18 | 0.22 | 0.18 | 0.23 |
| | 138 | 133 | 136 | 132 | 134 | 132 | 138 | 143 |
| 164.2 | 0.20 | 0.18 | 0.14 | 0.11 | 0.15 | 0.20 | 0.24 | 0.20 |
| | 137 | 134 | 136 | 132 | 134 | 129 | 135 | 144 |
| 189.3 | 0.13 | 0.07 | 0.08 | 0.09 | 0.14 | 0.23 | 0.15 | 0.18 |
| | 135 | 134 | 137 | 131 | 134 | 133 | 135 | 144 |
| 214.4 | 0.04 | 0.12 | 0.10 | 0.07 | 0.07 | 0.15 | 0.11 | 0.13 |
| | 135 | 136 | 139 | 134 | 136 | 135 | 138 | 146 |
| Duct Average | 0.34 | | | | | | | |
| | 137 | | | | | | | |

* Port/sampling depth of average velocity used for Ontario Hydro Method mercury speciation sampling.

points of average velocity could have been selected for the single-point sampling to be conducted.

For the FGD outlet location, the data in Table 3-5 show that the flue gas flow is strongly biased towards the top of the duct, with little bias left to right. This is also expected, as the most recent flow disturbance upstream of this point is a 90° turn from vertical to horizontal flow as the gas exits the FGD absorber mist eliminator. The top of the duct represents the outside of that turn. Because the flow in this location was more highly stratified, the point selected for the Ontario Hydro method sampling was the only point that exactly represented the average velocity.

Table 3-6 summarizes the results of the measurements made by the Ontario Hydro method at the FGD inlet and outlet. Results are shown for both gas phase and particulate phase mercury, although the particulate phase results show almost negligible mercury content at these two locations. Tables 3-7 and 3-8 provide more information about the results of the individual sample runs.

TABLE 3-6. FLUE GAS MERCURY RESULTS BY THE ONTARIO HYDRO METHOD, AVERAGE OF THREE SAMPLING RUNS

| SAMPLE | OXIDIZED MERCURY ($\mu\text{g}/\text{Nm}^3$) | ELEMENTAL MERCURY ($\mu\text{g}/\text{Nm}^3$) | TOTAL MERCURY ($\mu\text{g}/\text{Nm}^3$) | MERCURY OXIDATION (%) |
|---------------------------------|---|--|--|-----------------------|
| FGD Inlet (Gas Phase): | | | | |
| Run 1 | 2.17 | 6.57 | 8.73 | 24.8 |
| Run 2 | 3.29 | 10.55 | 13.84 | 23.8 |
| Run 3 | 4.19 | 9.30 | 13.49 | 31.1 |
| Average | 3.22 | 8.81 | 12.02 | 26.6 |
| FGD Inlet (Particulate Phase): | | | | |
| Run 1 | - | - | 0.02 | - |
| Run 2 | - | - | <0.01 | - |
| Run 3 | - | - | <0.01 | - |
| Average | - | - | 0.01 | - |
| FGD Outlet (Gas Phase): | | | | |
| Run 1 | 0.17 | 8.85 | 9.01 | 1.8 |
| Run 2 | 0.21 | 9.63 | 9.84 | 2.1 |
| Run 3 | 0.25 | 8.66 | 8.91 | 2.8 |
| Average | 0.21 | 9.04 | 9.25 | 2.3 |
| FGD Outlet (Particulate Phase): | | | | |
| Run 1 | - | - | 0.04 | - |
| Run 2 | - | - | 0.08 | - |
| Run 3 | - | - | 0.04 | - |
| Average | - | - | 0.05 | - |

TABLE 3-7. FGD INLET SAMPLE RUN DATA

| RUN NO. | 1 | 2 | 3 | AVERAGE |
|---|-----------|-----------|-----------|---------|
| Date | 12/2/1998 | 12/3/1998 | 12/3/1998 | - |
| Start Time | 1425 | 935 | 1305 | - |
| End Time | 1605 | 1115 | 1445 | - |
| Operator | CSG | CSG | CSG | - |
| Initial Leak Rate | 0.003@17 | 0.002@15 | 0.002@15 | - |
| Final Leak Rate | >0.02 | 0.004@7 | 0.002@5 | - |
| Cross-Sectional Area (ft^2) | 318.3 | 318.3 | 318.3 | - |
| Pitot Tube Correction Factor (C_p) | 0.84 | 0.84 | 0.84 | - |
| Dry Gas Meter Calibration (Y_d) | 0.995 | 0.995 | 0.995 | - |
| Nozzle Diameter (in.) | 0.278 | 0.278 | 0.278 | - |
| Barometric Pressure (in. Hg) | 29.5 | 29.5 | 29.5 | 29.5 |
| Static Pressure (in. H_2O) | 2.5 | 2.5 | 2.5 | 2.5 |

TABLE 3-7 (CONTINUED)

| RUN NO. | 1 | 2 | 3 | AVERAGE |
|--|---------|---------|---------|---------|
| Meter Volume (acf) | 58.612 | 63.272 | 55.435 | 59.106 |
| Meter Volume (acm) | 1.660 | 1.792 | 1.570 | 1.674 |
| Average Square Root of ΔP | 0.550 | 0.589 | 0.500 | 0.546 |
| Average Delta H (in. H ₂ O) | 1.14 | 1.40 | 1.03 | 1.19 |
| Average Stack Temperature (°F) | 305.3 | 301.2 | 296.4 | 301.0 |
| Average Dry Gas Meter Temp (°F) | 91.5 | 82.8 | 87.6 | 87.3 |
| Test Duration (min) | 100.0 | 100.0 | 100.0 | 100.0 |
| Condensed Water (g) | 128.5 | 196.3 | 158.1 | 160.9 |
| Filter Weight Gain (g) | 0.0091 | 0.0091 | 0.0077 | 0.0086 |
| % CO ₂ | 13.0 | 14.0 | 13.0 | 13.3 |
| % O ₂ | 8.0 | 6.5 | 7.0 | 7.2 |
| % N ₂ | 79.0 | 79.5 | 80.0 | 79.5 |
| Meter Volume (dscf) | 55.207 | 60.590 | 52.572 | 56.123 |
| Meter Volume (Ncm) | 1.457 | 1.599 | 1.387 | 1.481 |
| Flue Gas Moisture - Measured (%) | 9.9 | 13.3 | 12.4 | 11.9 |
| F.G. Molecular Wt. (Wet) (g/g-mole) | 29.2 | 28.8 | 28.8 | 28.9 |
| Absolute Stack Pressure (in. Hg) | 29.7 | 29.7 | 29.7 | 29.7 |
| Absolute Stack Temperature (°R) | 765.3 | 761.2 | 756.4 | 761.0 |
| Average Gas Velocity (ft/sec) | 37.1 | 39.9 | 33.8 | 36.9 |
| Avg Flow Rate (acfm) | 709,001 | 761,623 | 644,611 | 705,078 |
| Avg Flow Rate (dscfm) | 437,077 | 454,233 | 390,903 | 427,405 |
| Avg Flow Rate (acmm) | 20,079 | 21,569 | 18,255 | 19,968 |
| Avg Flow Rate (Ncmm) | 11,532 | 11,985 | 10,314 | 11,277 |
| Isokinetic Sampling Rate (%) | 95.4 | 100.7 | 101.6 | 99.2 |

TABLE 3-8. FGD OUTLET SAMPLE RUN DATA

| RUN NO. | 1 | 2 | 3 | AVERAGE |
|---|-----------|-----------|-----------|---------|
| Date | 12/2/1998 | 12/3/1998 | 12/3/1998 | - |
| Start Time | 1425 | 935 | 1305 | - |
| End Time | 1605 | 1115 | 1445 | - |
| Operator | KS,AO | AO,KS | KS,AO | - |
| Initial Leak Rate | 0.008@20 | 0.004@15 | 0.004@15 | - |
| Final Leak Rate | 0.008@10 | 0.002@5 | 0.003@7.5 | - |
| Cross-Sectional Area (ft ²) | 280.6 | 280.6 | 280.6 | - |
| Pitot Tube Correction Factor (Cp) | 0.84 | 0.84 | 0.84 | - |
| Dry Gas Meter Calibration (Yd) | 0.999 | 0.999 | 0.999 | - |
| Nozzle Diameter (in.) | 0.274 | 0.274 | 0.274 | - |
| Barometric Pressure (in. Hg) | 29.5 | 29.5 | 29.5 | - |
| Static Pressure (in. H ₂ O) | -0.55 | -0.55 | -0.55 | - |
| Meter Volume (acf) | 61.578 | 62.239 | 60.404 | 61.407 |
| Meter Volume (acm) | 1.744 | 1.763 | 1.711 | 1.739 |
| Average Square Root of ΔP | 0.562 | 0.563 | 0.518 | 0.548 |
| Average Delta H (in. H ₂ O) | 1.38 | 1.44 | 1.25 | 1.36 |
| Average Stack Temperature (°F) | 134.7 | 129.1 | 133.0 | 132.3 |
| Average Dry Gas Meter Temp (°F) | 94.3 | 85.3 | 87.7 | 89.1 |
| Test Duration (min) | 100.0 | 100.0 | 100.0 | 100.0 |
| Condensed Water (g) | 263.0 | 318.2 | 268.6 | 283.3 |
| Filter Weight Gain (g) | 0.0039 | -0.0030* | 0.0024 | - |
| % CO ₂ | 11.0 | 12.0 | 12.0 | 11.7 |
| % O ₂ | 8.0 | 8.0 | 8.0 | 8.0 |
| % N ₂ | 81.0 | 80.0 | 80.0 | 80.3 |
| Meter Volume (dscf) | 57.974 | 59.572 | 57.535 | 58.360 |
| Meter Volume (Ncm) | 1.530 | 1.572 | 1.518 | 1.540 |
| Flue Gas Moisture - Saturation (%) | 17.4 | 15.0 | 16.6 | 16.4 |
| Flue Gas Moisture - Measured (%) | 17.6 | 20.1 | 18.1 | 18.6 |
| F.G. Moisture for Calculations (%) | 17.4 | 15.0 | 16.6 | 16.3 |
| F.G. Molecular Wt. (Wet) (g/g-mole) | 28.0 | 28.4 | 28.2 | 28.2 |

TABLE 3-8 (CONTINUED)

| RUN NO. | 1 | 2 | 3 | AVERAGE |
|----------------------------------|----------|----------|----------|----------------|
| Absolute Stack Pressure (in. Hg) | 29.5 | 29.5 | 29.5 | 29.5 |
| Absolute Stack Temperature (°R) | 594.7 | 589.1 | 593.0 | 592.3 |
| Average Gas Velocity (ft/sec) | 34.3 | 33.9 | 31.4 | 33.2 |
| Avg Flow Rate (acfm) | 577,084 | 571,056 | 529,022 | 559,054 |
| Avg Flow Rate (dscfm) | 416,529 | 428,168 | 386,439 | 410,379 |
| Avg Flow Rate (acmm) | 16,343 | 16,172 | 14,982 | 15,832 |
| Avg Flow Rate (Ncmm) | 10,990 | 11,297 | 10,196 | 10,828 |
| Isokinetic Sampling Rate (%) | 95.4 | 95.3 | 102.0 | 97.6 |

*Filter was not completely recovered.

An average total gas phase mercury concentration of 12.0 µg/Nm³ was measured at the FGD inlet location, and the gas phase elemental mercury concentration was 8.8 µg/Nm³. The corresponding mercury oxidation percentage was 27%. The gas phase elemental mercury concentration measured at the FGD outlet (9.0 µg/Nm³) was virtually the same as the concentration measured at the inlet. This provides additional validation of the inlet data, as no removal of elemental mercury was expected across the FGD system and the two concentrations were expected to be almost identical. The gas phase concentration of oxidized mercury at the FGD outlet location was only 0.21 µg/Nm³. The observed removal of oxidized mercury across the FGD system was about 93%, which is within the range measured previously for other FGD systems.

The gas phase mercury concentration at the FGD inlet location measured on the first run was lower than in the other two runs. As was mentioned above in Section 3.2, it is possible that the sampling system developed a leak during the run, as the sample train failed a leak check test at the end of the run.

A mass balance calculation was made to compare the amount of mercury measured in the coal fired versus the amount of mercury accounted for in the flue gas (assuming the flue gas flow rate from the north ID fan was equal to that measured for the south ID fan). Because of the potential for sample-to-sample variability for both the Ontario Hydro method and in the coal sample and analysis, it was decided to use test average values for the material balance rather than calculating balances for each individual run. This material balance calculation is summarized in Table 3-9. The material balance indicated that 0.0371 lb/hr

TABLE 3-9. MERCURY BALANCE BASED ON AVERAGE ONTARIO HYDRO METHOD AND COAL ANALYSES RESULTS

| PARAMETER | VALUE |
|--|-------------|
| Average coal mercury content, mg/kg (as received basis) | 0.094 |
| Amount of coal fired, tons/hr | 197 |
| Mercury in coal fired, lb/hr | 0.0371 |
| Average total mercury concentration in FGD inlet flue gas, $\mu\text{g}/\text{Nm}^3$ | 12.0 |
| Flue gas flow rate at ESP outlet, dscfm@68°F* | 855,000 |
| Total mercury in flue gas, lb/hr | 0.0358 |
| Average mercury concentration in fly ash samples, mg/kg | 0.020 |
| Fly ash collection rate, tons/hr** | 8.9 |
| Mercury in fly ash collected, lb/hr | 0.0004 |
| Mercury in bottom ash, lb/hr | 0 (assumed) |
| Total mercury accounted for in flue gas and fly ash, lb/hr | 0.0362 |
| Mercury material balance closure, % of coal mercury accounted for | 97.7 |

*Assumes flue gas flow rate through North ID fan is equal to the rate measured from the South ID fan.

**Estimated as 80% of the ash in the coal fired.

of mercury is introduced from the coal, while 0.0362 lb/hr was measured in the flue gas and fly ash. This represents 98% recovery of the coal mercury, which is excellent recovery for a trace species being measured in a full-scale unit. The material balance closures for the individual sampling runs (not shown in the table) were more varied, ranging from 64.5% to 120.8%. As might have been anticipated, the low closure was for the first FGD inlet sampling run, where the total mercury concentration measured appeared to be low, possibly due to a leak in the sampling train.

The Ontario Hydro method particulate filters were weighed and used to calculate an apparent mass loading at the FGD inlet and outlet locations. These data are summarized in Table 3-10. Because the ducts were not traversed, the calculated mass loadings may not be accurate. Also, the Ontario Hydro method does not include recovery and weighing of the mass of material collected from the sampling probe rinse; the values in Table 3-10 reflect only the filter weight gain data. However, the values indicate that the ESP on Unit 3 is very efficient. The average value indicated at the FGD inlet location, of 0.00420 g/Nm³, is equivalent to an emission rate of 0.0035 lb of particulate per million Btu of heat input. At the FGD outlet location, the particulate loading was so low that the weight gain by the

TABLE 3-10. APPARENT PARTICULATE MASS LOADING VALUES FOR THE ONTARIO HYDRO METHOD SAMPLING RUNS (ALL VALUES ARE CORRECTED FOR THE WEIGHT GAIN MEASURED BY A FIELD BLANK FILTER)

| SAMPLING RUN | MASS LOADING (g/Nm³) |
|---------------------|--|
| FGD Inlet: | |
| Run 1 | 0.00459 |
| Run 2 | 0.00419 |
| Run 3 | 0.00382 |
| Average | 0.00420 |
| FGD Outlet: | |
| Run 1 | 0.00091 |
| Run 2 | -0.00350* |
| Run 3 | -0.00007 |
| Average | 0.00042 |

*Filter was not recovered completely, value was not included in the average.

filters was difficult to quantify. It appears that the particulate removal efficiency across the FGD system is on the order of 75% or better.

4.0 SAMPLING AND ANALYTICAL PROCEDURES

4.1 Test Methods

This section describes the test methods used to obtain flue gas samples and process data. The procedures used for the analytical determination of process samples are also summarized. Copies of all applicable test methods are provided in Appendix A.

4.1.1 EPA Method 1 - Sample and Velocity Traverses for Stationary Sources. Flue gas velocity at the FGD absorber inlet and outlet locations was determined using the EPA Method 1 sampling procedure. An 8x8 matrix was used in the velocity traverse. The objective of the preliminary traverse was to determine the point(s) of average flow in each duct. This information was used to determine the location Ontario Hydro samples were extracted from. A copy of EPA Method 1 is included in Appendix A.

4.1.2 EPA Method 2 - Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube). EPA Method 2 was followed as outlined in 40 CFR Part 60- Appendix A throughout the test program for determination of flue gas velocity and volumetric flow rate. A copy of Method 2 is provided in Appendix A.

4.1.3 EPA Method 3 - Gas Analysis for the Determination of Dry Molecular Weight. Oxygen and carbon dioxide concentrations in the tested flue gas were measured using Fyrite combustion gas analyzers as referenced in EPA Method 3, Section 2.3. Nitrogen was calculated by difference, as outlined in the method. Manufacturer guidelines for operation and maintenance were adhered to throughout the test program. A copy of Method 3 is provided in Appendix A.

4.1.4 Ontario Hydro - Determination of Elemental, Oxidized, Particle-Bound, and Total Mercury Emissions from Coal-Fired Stationary Sources. The draft Ontario Hydro Method was used to collect simultaneous mercury speciation samples at the FGD absorber inlet and outlet locations. A copy of the draft procedure followed is provided in Appendix A. This sampling procedure is very similar to 40 CFR Part 60 Appendix A, Method 29 with the following noted exceptions:

- The gas sample is maintained through the probe/filter system to within $\pm 27^{\circ}\text{F}$ of the flue gas temperature, rather than at $248 \pm 25^{\circ}\text{F}$ as stated in EPA Method 29. At no time is the probe allowed to be at a temperature lower than 248°F with the Ontario Hydro method.

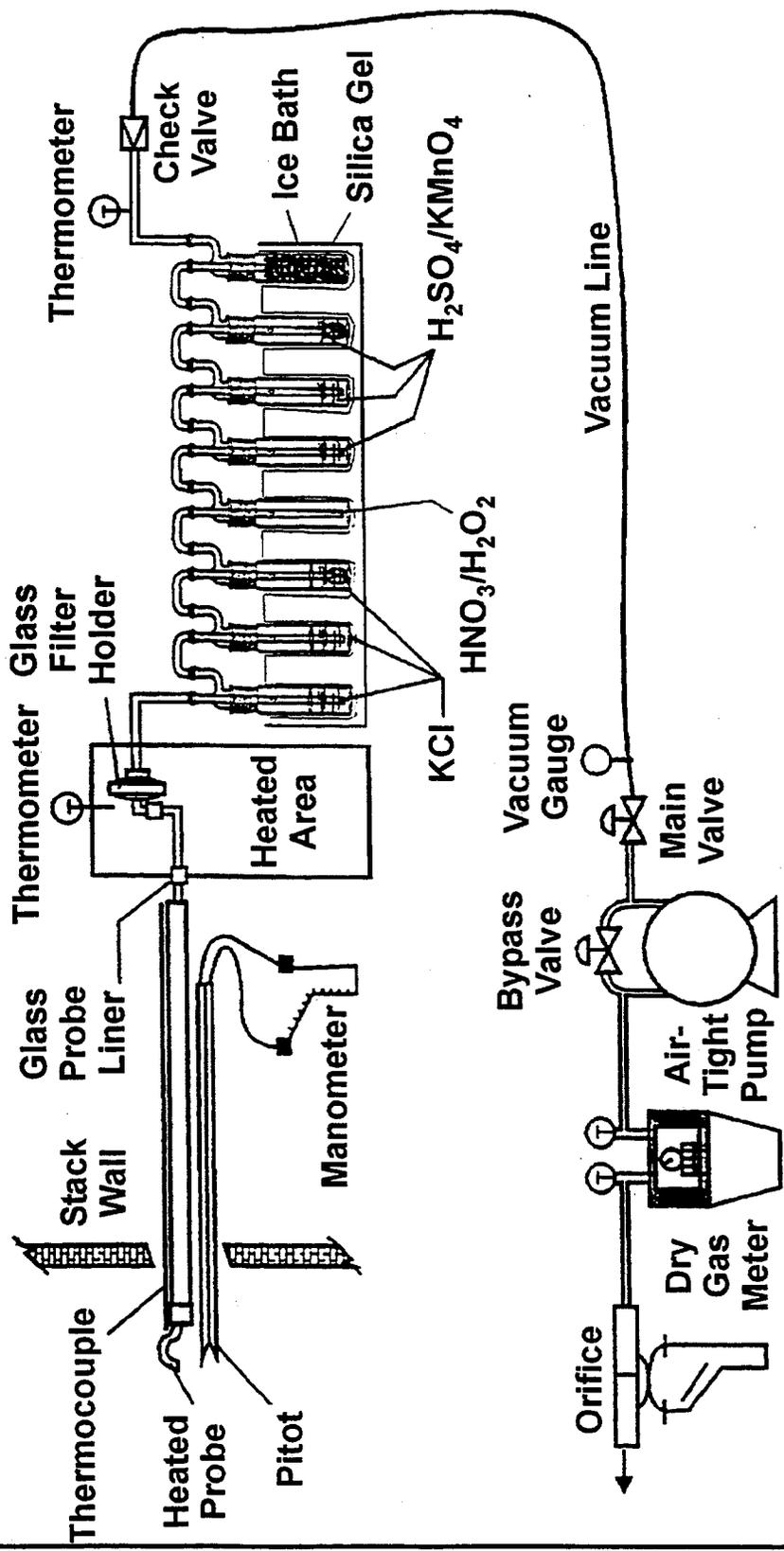
- The impinger train (condenser) consists of eight (8) impingers in series, as opposed to a maximum of seven (7) as stated in EPA Method 29. Of the seven impingers, only one (1) is of the Greenburg-Smith design in EPA Method 29 (impinger number 2) whereas the Ontario Hydro train contains two of this design (impingers 3 and 7).
- The first set of impingers (1-3) contain a 1M KCl absorbing solution rather than a $\text{HNO}_3/\text{H}_2\text{O}_2$ solution. Also, the impinger separating the two different absorbing solutions is empty in Method 29 but is charged with $\text{HNO}_3/\text{H}_2\text{O}_2$ in the Ontario Hydro method.
- EPA Method 29 has a provision for an optional acetone probe/nozzle/front half rinse if particulate emissions are to be determined. The Ontario Hydro method calls for only a 0.1 N HNO_3 rinse.

Figure 4-1 is a schematic of the draft Ontario Hydro mercury sampling train. Figures 4-2 and 4-3 illustrate the sample recovery scheme used for the front and back portions of the sampling train, respectively. During the recovery process, sufficient KMnO_4 solution was added to each KCl impinger solution to maintain a purple color. HCl rinses were not required for the KCl or $\text{HNO}_3/\text{H}_2\text{O}_2$ impingers since no residue was observed.

One modification made to the draft Ontario Hydro method was the separate collection and analysis of the HCl rinse of the KMnO_4 impingers; the draft method calls for this rinse to be added directly to the recovered permanganate sample. Previous Radian testing has shown the HCl rinse to typically contain very low levels of mercury. This rinse therefore acts as a diluent to the KMnO_4 samples, thus lowering the concentration in the sample; this can increase analytical variability associated with measuring low levels of mercury in solution. A second concern with adding the HCl rinse to the permanganate samples is possible matrix effects caused by variability associated with different rinse volumes of the concentrated acid from sample to sample.

Figure 4-4 depicts the analysis scheme used for the Ontario Hydro samples. Impinger solutions were treated as called for in the draft method. Sample aliquots were digested using a modified version of Method 7470, as indicated in the draft Ontario Hydro method. One modification made was that the concentration of the hydroxylamine solution was twice that recommended in the method which serves to make the reduction of potassium permanganate more efficient. A copy of EPA Method 7470 is included in Appendix A. The digested samples were analyzed for mercury by cold vapor atomic absorption (CVAA) spectroscopy using a Perkin Elmer Flow Injection Mercury System (FIMS). The Radian International

DRAFT



EERC KG 13642.CDR

FIGURE 4-1. DRAFT ONTARIO HYDRO SAMPLING TRAIN

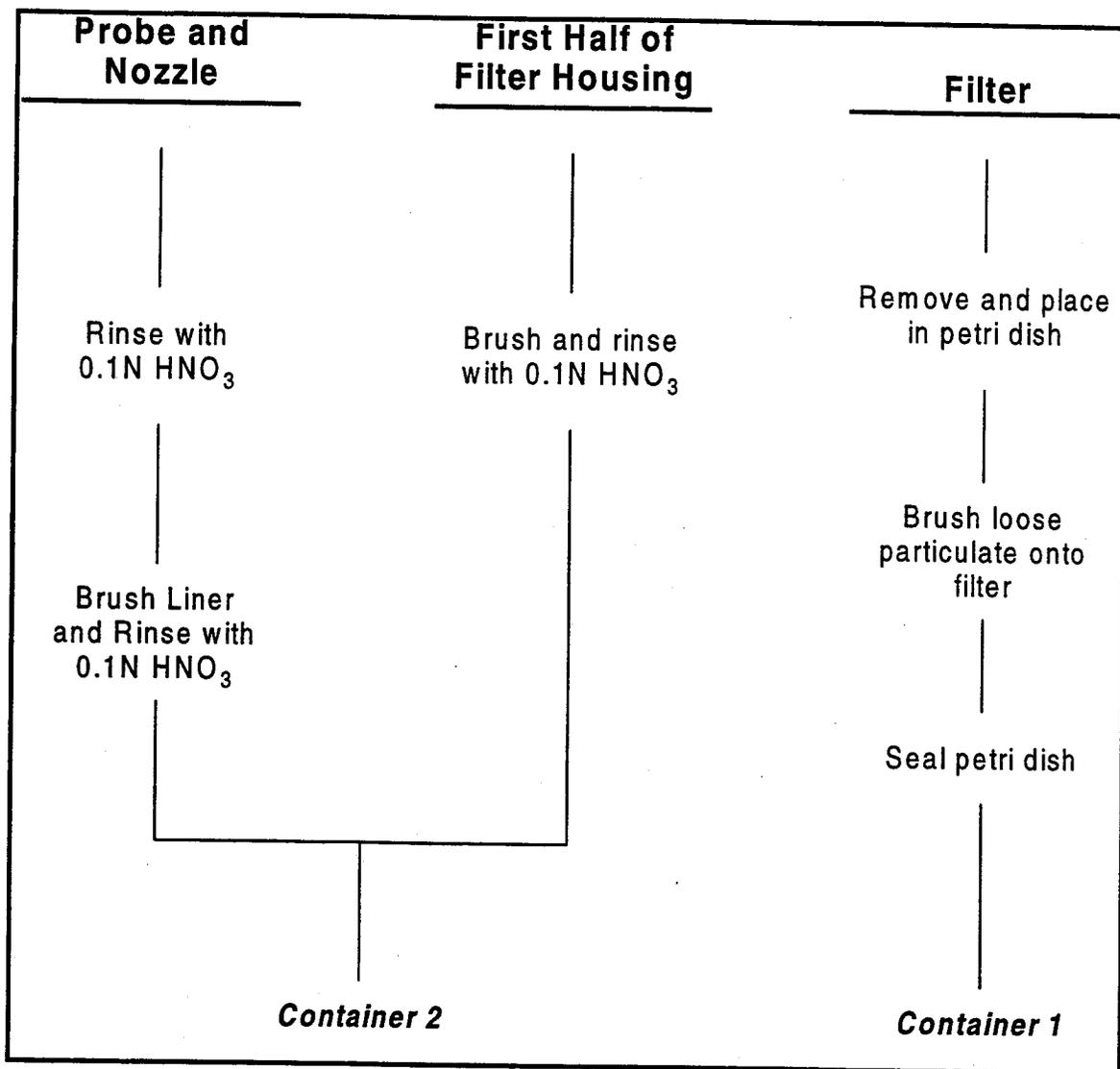
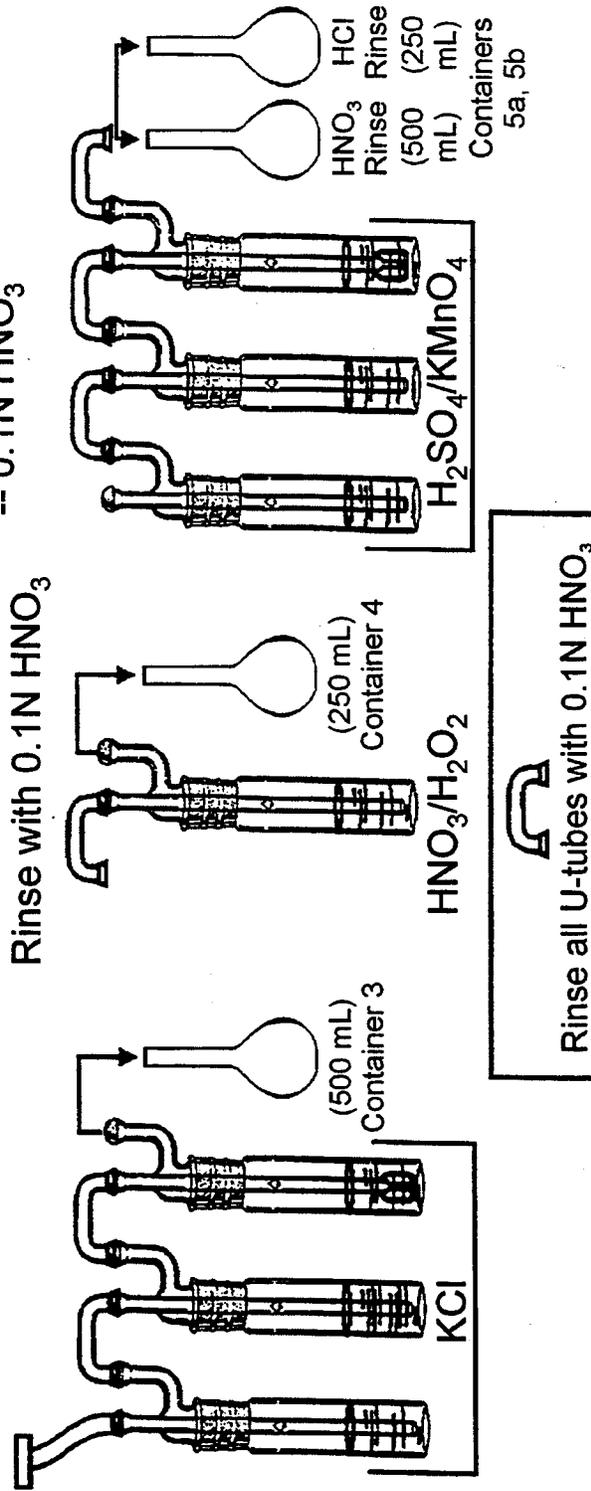


FIGURE 4-2. RECOVERY SCHEME OF FRONT PORTION OF ONTARIO HYDRO SAMPLE TRAIN

DRAFT

1. Rinse filter holder and connector with 0.1N HNO₃.
2. Add H₂SO₄/KMnO₄ to each impinger bottle until purple color remains.
3. Rinse with 0.1N HNO₃.
4. Rinse with 8N HCl if brown residue remains.
5. Final rinse with 0.1N HNO₃.

Rinse bottles sparingly with
-- 0.1N HNO₃
-- 8N HCl
-- 0.1N HNO₃



EERC KG 13178b.CDR

FIGURE 4-3. RECOVERY SCHEME FOR ONTARIO HYDRO IMPINGERS

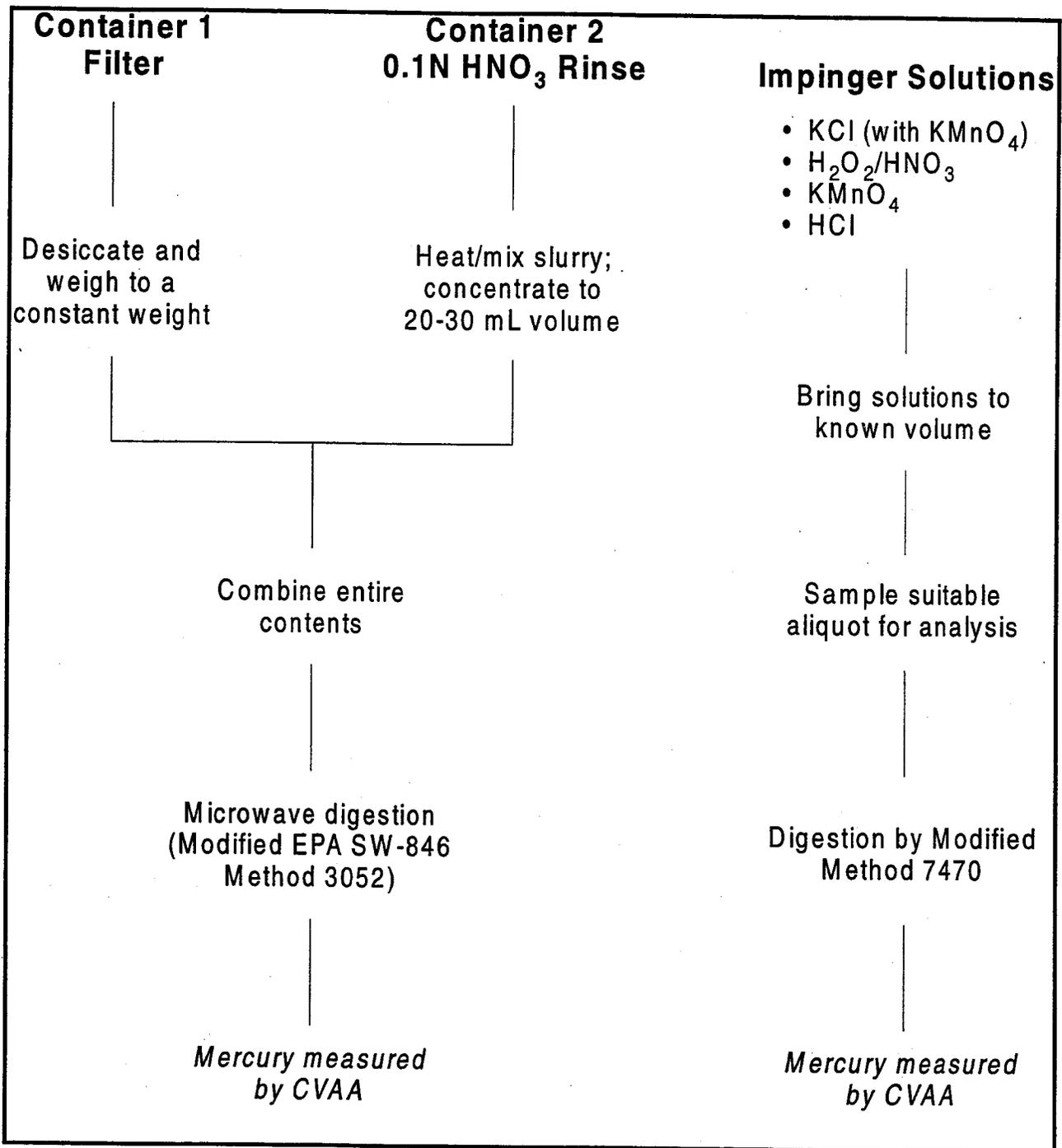


Figure 4-4. ANALYTICAL SCHEME FOR ONTARIO HYDRO SAMPLES

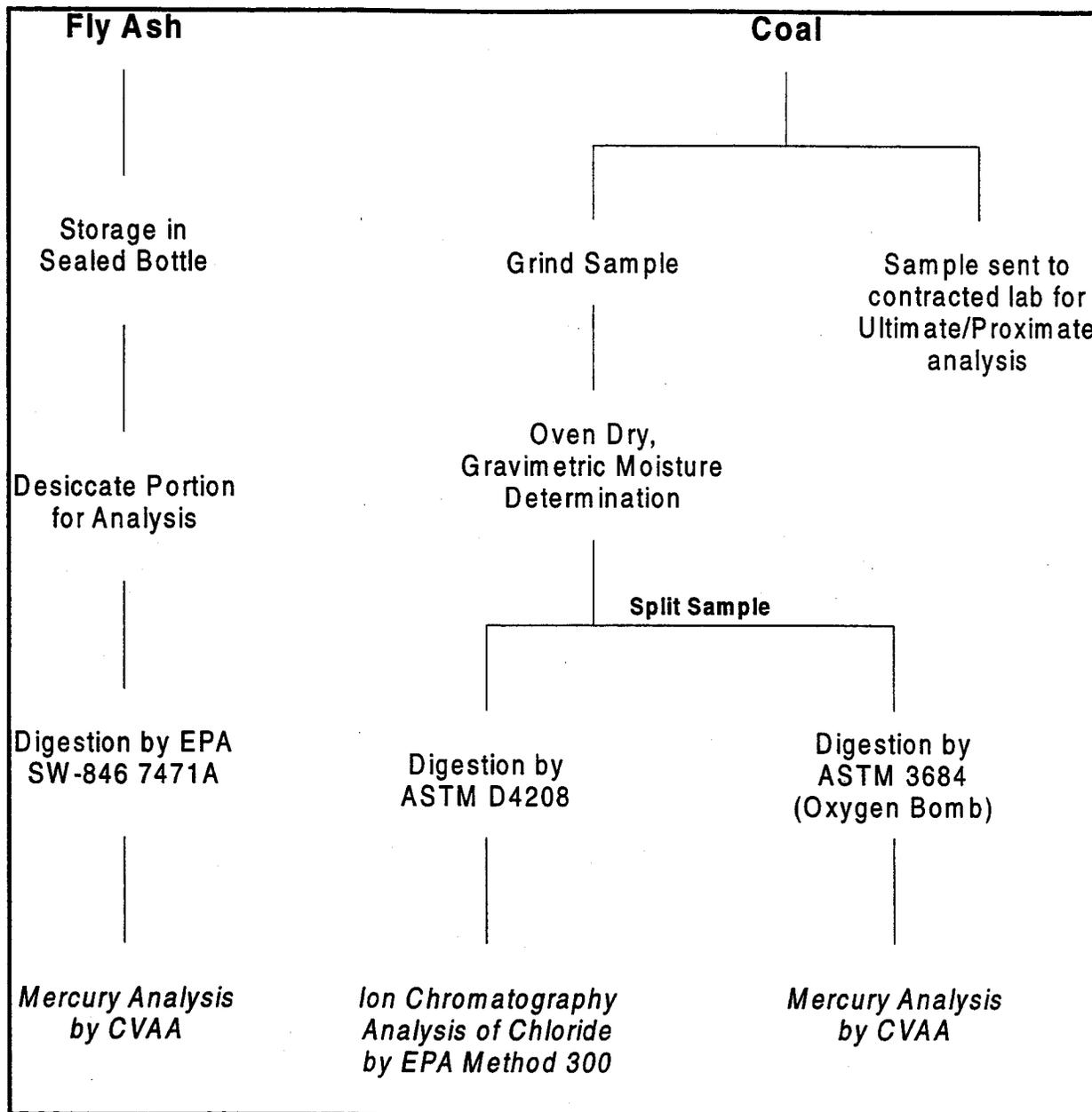


FIGURE 4-5. ANALYTICAL SCHEME FOR SOLID PROCESS SAMPLES

standard operating procedure (SOP) for mercury analysis by CVAA is given in Appendix A.

The mass of filter-collected particulate was determined as indicated in Figure 4-4. Since a nitric acid probe rinse is called for (instead of one with acetone), the resulting rinse solution was not totally evaporated; thus the mass of particulate in the probe rinse was not specifically determined. The entire contents of the filter and probe rinse were added together, digested using a microwave method and analyzed by CVAA for mercury; this provided a value for total particulate mercury in the Ontario Hydro samples. The microwave digestion procedure was carried out according to the instrument manufacturer based upon EPA Method 3052.

4.1.5. Analysis of Solid Process Samples. Coal and fly ash process samples were obtained concurrently with the Ontario Hydro samples as outlined in Section 2.4. Figure 4-5 illustrates the analytical scheme for these samples. All analyses were carried out as defined in the respective test methods. The following exceptions were noted:

- A modification was made to the procedure for measuring coal chloride in order to decrease the analytical detection limit. This included the use of an ion chromatographic determination of chloride (EPA Method 300) as opposed to using an ion selective electrode as indicated in ASTM D4208.
- Prior to mercury analyses of solid samples Method 7471A, the samples were digested with nitric acid and sulfuric acids. This is consistent with the alternative digestion procedure listed in the method (Section 7.2).
- When preparing solid samples for analysis, triplicate 0.2-g portions were not weighed out but rather a single 0.5 - 1.0 g aliquot of the sample. According to the technical representative on the EPA MICE line, this satisfies the intent of the method (to collect a homogeneous sample) and is what is expected to be published in SW-846 Update IV.

4.2 PROCESS TEST METHODS

4.2.1 Procedures for Process Stream and Control Equipment Data. Most of the Unit 3 boiler data were collected from the plant distributed control system (DCS). Hourly average data were reported by the DCS for the following parameters:

- Unit load (MW)
- Net Plant Heat Rate (Btu/kw-hr)

- FGD SO₂ removal (%)
- FGD inlet and stack CO₂ concentrations (%)
- FGD inlet and stack SO₂ concentrations (lb/MMBtu)
- Stack NO_x concentrations (lb/MMBtu)

Hourly averages for these parameters that most closely corresponded to the sample run periods were averaged to produce run average values. The CO₂, SO₂ and NO_x concentration data were all based on measurements made by the plant continuous emissions monitoring system.

Design information for the Unit 3 boiler and emissions control equipment was taken from the 1997 Form EIA-767 submitted by LCRA for the Seymour Station.

4.2.2 Equipment Calibration. The following equipment calibration methods were utilized during the test program to ensure the collection of accurate data.

4.2.2.1 Type S Pitot Tube Calibration. The EPA guidelines were followed for the use of Type S pitot tubes, as presented in section 3.1.1 of EPA Document 600/4-77-027b. Prior to the sampling program, all pitot tubes to be used were inspected in accordance with EPA specifications. At both sampling locations the pitot tube coefficient was 0.84.

4.2.2.2 Sampling Nozzle Calibration. In order to perform isokinetic sampling, the sampling nozzle cross-sectional area must be precisely known. All nozzles used for isokinetic sampling were calibrated according to the procedure outlined in Section 3.4.2 of EPA Document 600/4-77-027b. This procedure requires that three measurements be made on different cross-sections of the nozzle. Measurements were made to the nearest 0.001 inch, using a Vernier caliper. Nozzles were only considered acceptable for sampling if the difference between any two cross-sectional measurements was less than 0.004 inches. All nozzle calibrations are recorded on field sampling data sheets.

4.2.2.3 Temperature Measuring Device Calibration. All thermocouple temperature sensors for field sampling were calibrated at a single point against a NIST-traceable, mercury-in-glass thermometer. The linearity was demonstrated using a traceable precision voltage generator.

4.2.2.4 Dry Gas Meter Calibration. Dry gas meters are used in all sampling trains to control the sampling rate and to record the volume of gas sampled. All dry gas meters used in the sampling program were calibrated against traceable critical orifices prior to use. A calibration overview is outlined below:

- A five-point calibration, using five different orifices, is performed semi-annually on each dry gas meter.
- A three-point calibration is performed as a post-test calibration check. These calibrations must agree within 5% of the five-point calibration.

The calibration procedure is as follows:

- A positive pressure leak-check of the dry gas meter is performed prior to calibration. The system is placed under approximately ten inches of water pressure, and a gauge oil manometer is used to detect any pressure decrease. If any pressure decrease is detected over a one-minute period, the leak will be repaired before calibrations commence.
- Also prior to calibration, the pump is run for at least five minutes to allow the dry gas meter to warm up.
- Once warmed up, the critical orifice is attached, and ambient air is pulled through the dry gas meter at rate controlled by the orifice for ten minutes.
- After ten minutes, the valve is closed and the volume of gas read by the meter is compared to known volume of gas passed through the critical orifice.
- Duplicate calibrations are performed using each of the five orifices.
- Additional calibrations and maintenance (if necessary) are performed until the results vary by 2% or less.

4.2.2.5 Calibration of Analytical Instrumentation. All laboratory mercury measurements were preceded by instrument calibrations. Analytical calibration standards were analyzed beginning with the blank and proceeding to the highest standard. Linear regression was used to define the calibration curves. The regression line was constructed using concentration as the abscissa and mean response as the ordinate. The blank was included as $X=0$. For acceptance (i.e., for analyses to proceed) the correlation coefficient of the standard curve must be ≥ 0.995 . Likewise, the calculated intercept value must be between 0.001 and -0.001 absorbance units. A detailed description of the calibration procedure is given in the CVAA method included in Appendix A.

5.0 INTERNAL QA/QC ACTIVITIES

5.1 QA/QC PROBLEMS

No problems were encountered with sample identification or custody during this test program.

One sampling problem was encountered while obtaining the Ontario Hydro samples. The sampling train leak rate measured after Run 1 at the FGD Inlet location exceeded the allowable 0.02 cubic feet per minute. It was determined that this leak was caused by a tube fitting that came loose. Although it was initially believed that this problem occurred as the sample probe was lifted out of the flue gas duct, oxygen and mercury results suggested that some leakage into the sample train occurred during the run.

One QC problem was observed during the analytical determination of mercury in the Ontario Hydro filter and probe rinse samples. The analytical spike recovery for the analysis showed 120% recovery falling outside the accepted range of 85 - 115%. This QA check involves the addition of a known mercury standard to a digested sample prior to analysis. It is believed that the results obtained were still valid since all of the other QA checks, including the matrix spike, fell well within the accepted ranges. Also, since the filter and probe rinse samples represented a very small fraction of the total mercury measured (Table 3-6) it was decided that a repeat analytical determination of these samples was not justified.

5.2 QA AUDITS

5.2.1. QA/QC Checks For Field Samples. Table 5-1 lists the quality assurance samples obtained for checks on the process samples obtained at Sam Seymour Station. Field blank samples were included for both the FGD absorber inlet and outlet locations. The objective of these samples was to account for any "background" mercury associated with the reagent solutions or holding vessels used in the sampling or storage of the samples. These samples were obtained by assembling Ontario Hydro sampling trains, including filter and all impingers, in the same manner as with each sample run. After a minimum of 60 minutes the field blank trains were recovered in the same manner as the gas sampling trains. This included the probe and nozzle rinse performed at the sampling location. All impingers were recovered and stored in a similar manner as those used for actual gas samples.

The field blank samples were analyzed for mercury along with the respective process samples. In the case of the KCl and KMnO₄,

impinger samples, the field blank solutions were used as the matrix in the CVAA calibration in order to eliminate the effect of matrix effects during the analysis; the calibrant solutions thus possessed the same matrix as the samples. Radian has shown this method to produce better results for mercury determinations than analyses in which calibrants are prepared in water. For analyses in which the field blank solution was not used as the calibrant matrix, the value of the analyzed field blank sample was subtracted from the values obtained for each sample.

TABLE 5-1. QA SAMPLES OBTAINED FOR CHECKS ON PROCESS SAMPLES

| FIELD SAMPLE TYPE | QA SAMPLE | FREQUENCY | ACTION TAKEN |
|---------------------------|------------------------|-----------------------|------------------------------|
| Ontario Hydro Gas Samples | Field Blank | 1 per sample location | Analyze all samples |
| | Reagent Blanks | 1 per reagent type | Hold all; selective analysis |
| Process Coal Samples | SARM 20 Control Sample | 1 per program | Run with all coal analyses |

Reagent blank samples were also obtained for each solution used in the gas sampling program. This included all impinger and rinse solutions. The reagent blank solutions were held and analyzed for mercury as deemed necessary.

A standard coal sample (SARM 20) was obtained as a control during determinations of mercury in the process coal. This sample was supplied by Brammer Standard (ISO 9002 Certificate # R-021) and contains a mercury concentration of 0.25 µg/g. Although the chloride content of this sample is not certified, previous Radian testing has shown its level to be between 50 - 60 ppm. The SARM 20 sample was included with all coal mercury and chloride analyses.

5.2.2 QA/QC Audits For Analytical Determinations. Mercury analyses of the Ontario Hydro and process solid samples were carried out at Radian International's fixed-price analytical services (FPAS) laboratories. Mercury analyses were carried out using cold vapor atomic absorption (CVAA) spectroscopy. The QA/QC audits and specifications for Radian's CVAA analysis of mercury are listed in Tables 5-2 through 5-4. Table 5-2 lists detection and reporting limits for mercury in different matrices, Table 5-3 lists accuracy and precision requirements, and Table 5-4 summarizes calibration and QC checks. A detailed summary of the analytical procedure, including all QA audits, is given in Appendix A.

TABLE 5-2. PROTOCOL REQUIRED DETECTION LIMITS AND REPORTING LIMITS

| ANALYTE | AQUEOUS | | SOLID | | MICROWAVE ^b | | TCLP | |
|---------|-------------|------------------------|---------------------------|--------------------------------------|------------------------|--------------------------------------|-------------|------------------------|
| | PRDL (mg/L) | REPORTING LIMIT (mg/L) | PRDL (mg/kg) ^a | REPORTING LIMIT (mg/kg) ^a | PRDL (mg/kg) | REPORTING LIMIT (mg/kg) ^b | PRDL (mg/L) | REPORTING LIMIT (mg/L) |
| Mercury | 0.00020 | 0.00020 | 0.020 | 0.020 | 0.20 | 0.20 | 0.0010 | 0.0010 |

^aNominal value. Solid digestion assumes 0.5 g (dry weight) sample digested with a final volume of 50 mL.

^bNominal value. Microwave digestion assumes a 0.1 g (dry weight) sample digested with a final volume of 100 mL.

TABLE 5-3. ACCURACY AND PRECISION REQUIREMENTS

| ANALYTE | LCS TOLERANCE (%) ^a | | MATRIX SPIKE TOLERANCE (%) | | ANALYTICAL SPIKE TOLERANCE (%) |
|---------|--------------------------------|-----------------|----------------------------|------------------------------|--------------------------------|
| | RECOVERY | PRECISION (RPD) | RECOVERY ^b | PRECISION (RPD) ^c | |
| Mercury | 82 - 123 | <13 | 70-130 | <25 | 85-115 |

^aLCS tolerances statistically derived from historical data from Radian's Laboratory.

^bMatrix spike accuracy tolerances are adopted from EPA Method 245.1 Matrix spike tolerances also used for media spikes and for analytical spikes when no sample is available for MS/MSD.

^cAs adopted from USACE EM 200-1-1.

TABLE 5-4. SUMMARY OF CALIBRATION AND QC ANALYSES

| CALIBRATION AND QC ANALYSES | DESCRIPTION | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION |
|--|--|---|---|---|
| Initial Calibration | A blank + 6-point calibration; for concentrations see Table 6 | Daily before any other analysis | Correlation coefficient ≥ 0.995 ; calculated intercept = 0.000 ± 0.001 absorbance units | Identify and reanalyze outlying point(s); recalculate curve using repeated points |
| Demonstration of Capability (DOC - Initial) | Quadruplicate analysis of LCS; for concentration see Table 7 | For each new instrument and each new analyst prior to analyzing samples | 1. Accuracy: See Table 5-3 for limits 2. Precision: See Table 5-3 for s limits | 1. Accuracy: a. Verify calibration with 2nd source standard and repeat DOC b. If still out, repeat multipoint calibration 2. Precision: a. Repeat DOC b. If still out, identify and correct source of excess variability |
| Demonstration of Capability (DOC - on-going) | Control charts generated by instrument and by analyst | Semi-annually | 25 of the last 30 points must be within statistical control. Control limits must be within LCS tolerances | 1. Analyst technique will be evaluated 2. Analyze DOC as described above |
| Initial Calibration Verification (ICV) | Primary source calibration standard with final digestate concentration at 0.010 mg/L | Daily before batch one is analyzed | 90-110% of true value | Accuracy: 1. Repeat calibration verification 2. If still out, identify and correct problem, run calibration verification again; if still out, recalibrate |
| Initial Calibration Blank (ICB) | Deionized water with digestion reagents | After initial calibration verification | Within \pm PRDL | 1. Reanalyze calibration blank 2. If still out, recalibrate |

FIGURE 5-4. (CONTINUED)

| CALIBRATION AND QC ANALYSES | DESCRIPTION | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION |
|-----------------------------|---|---|---|--|
| Method Blank | Deionized water with digestion reagents and analyzed as a sample | Once for each preparative and analytical batch | Measured concentrations must be <PRDL | Blank Contamination: 1. Reanalyze method blank; 2. If method blank is still contaminated, identify and correct source of contamination, then reprepare samples with new method blank |
| LCS/LCSD | Deionized water with digestion reagents, spiked with second source standard, and analyzed with samples For standard concentration, see Table 7 | One LCS/LCSD pair for each preparative and analytical batch | 1. Accuracy: See Table 2 LCS tolerances (see Appendix A) 2. Precision: See Table 2 LCS tolerances (see Appendix A) | 1. Accuracy: Reanalyze LCS/LCSD. If recovery is still out, stop, identify, and correct problem before proceeding. If digestion batch is to be reprepared, write Level 1 QCER. If samples can't be redigested, contact CSC and write Level 2 QCER 2. Precision: Reanalyze LCS/LCSD. If recovery is still out, stop, identify, and correct problem before proceeding. |

FIGURE 5-4. (CONTINUED)

| CALIBRATION AND QC ANALYSES | DESCRIPTION | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION |
|-----------------------------|--|---|--|--|
| MS/MSD | <p>Two aliquots of a native sample spiked with a standard solution.</p> <p>For concentration see Table 7</p> | One MS/MSD pair for each preparative and analytical batch | <p>1. Accuracy: See Table 2 MS tolerances (see Appendix A)</p> <p>2. Precision: See Table 2 MS tolerances (see Appendix A)</p> | <p>1. Accuracy:</p> <ul style="list-style-type: none"> a. If there is an assignable cause, and LCS/LCSD are within tolerance, flag data and contact CSC; write a Level 2 QCER b. If no assignable cause, run analytical spike c. If analytical spike fails, and LCS/LCSD results are within tolerance, flag MS/MSD results as matrix interference, write Level 2 QCER <p>2. Precision:</p> <ul style="list-style-type: none"> a. If there is an assignable cause, and LCS/LCSD are within tolerance, flag data and contact CSC; write Level 2 QCER b. If there is no assignable cause, analyze analytical spike |

FIGURE 5-4. (CONTINUED)

| CALIBRATION AND QC ANALYSES | DESCRIPTION | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION |
|---|--|---|--|---|
| Continuing Calibration Verification (CCV) | Primary source calibration standard with final digestate concentration at 0.010 mg/L | After 10th sample and at the end of each analytical batch | 90-110% of true value | Accuracy: 1. Repeat calibration verification 2. If still out, identify and correct problem, then reanalyze all samples analyzed since last passing calibration verification |
| Continuing Calibration Blank (CCB) | Deionized water with digestion reagents | Following each continuing calibration verification and at the end of each analytical batch | Within \pm PRDL | 1. Reanalyze calibration blank 2. If still out, correct the problem and reanalyze all samples since last passing calibration blank |
| Serial Dilution | A five-fold (1+4) dilution of a sample digestate prior to analysis | Once per analytical batch | Within 10% of the undiluted value if parent sample is $>5x$ PRDL | Perform analytical spike |
| Analytical Spike | Sample digestate spiked with a 0.020 mg/L calibration standard prior to analysis | 1. When serial dilution fails or if parent sample $<5x$ PRDL. 2. When MS/MSD fails without assignable cause. | 85% - 115% recovery | 1. Flag data 2. Contact CSC 3. Write Level 2 QCER |

Commercial liquid standards containing 1000 ppm mercury were used to prepare the calibrant (standard) and QC samples. Table 5-5 lists the mercury standards used.

TABLE 5-5. MERCURY STANDARDS USED IN ANALYTICAL DETERMINATIONS

| QA SOLUTION | MERCURY CONCENTRATION | MANUFACTURER | LOT NO. |
|-------------|-----------------------|--------------|------------|
| Standard | 1000 ppm | E.M. Science | A7045027 |
| QC | 1000 ppm | Mallinckrodt | H548KJBJ-P |

5.2.3 Results of QA/QC Audits. The results of the QA/QC checks performed during analysis of the Ontario Hydro impingers and the solid process samples are listed in Tables 5-6 and 5-7, respectively. These results indicate that, with the exception of one check (see Section 5-1), all values fell within the acceptable limits.

TABLE 5-6. QA/QC RESULTS FOR MERCURY ANALYSES OF IMPINGER SOLUTIONS

| QA CHECK | ACCEPTED RANGE | QA/QC RESULTS FOR DIFFERENT ONTARIO HYDRO SAMPLES | | | | |
|------------------|----------------|---|-------------------------------|-------------------|---------|----------------|
| | | KCl | H ₂ O ₂ | KMnO ₄ | HCl | Filter/ PNR |
| Reagent Blank | - | <0* | ND | <0* | ND | - |
| Field Blank | - | 0** | 0.19 µg | 0** | 0.05 µg | 0.02 µg |
| Initial QC | 90 - 110% | 100.6 | 102.0 | 102.8 | 102.0 | 100.0 |
| Final QC | 90 - 110% | 100.8 | 105.0 | 103.0 | 105.0 | 102.0 |
| Matrix Spike | 70 - 130% | 104.2 | 110.0 | 83.9 | 111.0 | 102.0 |
| Analytical Spike | 85 - 115% | 103.2 | 111.0 | 95.1 | 111.0 | 120.0 |
| RPD | <10% | 0.29 | 3.6 | 5.4 | 3.6 | 1.8 |

* - Result indicated reagent blank lower than field blank (calibration performed in field blank matrix).

** - Calibration performed in Field Blank matrix.

TABLE 5-7. QA/QC RESULTS FOR MERCURY ANALYSES OF PROCESS SOLIDS

| QA CHECK | ACCEPTED RANGE | QA/QC RESULTS FOR MERCURY ANALYSES | |
|------------------|----------------|------------------------------------|---------------|
| | | FLY ASH | COAL |
| Method Blank | <PRDL* | 0.007 mg/kg | 0.007 mg/kg |
| Initial QC | 90 - 110% | 101.0 | 100.0 |
| Final QC | 90 - 110% | 102.0 | 95.0 |
| Matrix Spike | 70 - 130% | 100.0 | 101.0, 105.0 |
| Analytical Spike | 85 - 115% | 109.0 | 98.0 |
| RPD | <13% | 0.0 | 7.6 |
| Standard Sample* | ± 20% actual** | NA | 0.232 (92.8%) |

* - PRDL for mercury = 0.20 mg/kg.

** - SARM 20 coal sample (mercury concentration = 0.25 µg/g).