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March 3, 2000

FERCo-R743-Revised-tl2

Mr. William Grimley  
USEPA, EMC Building, Room 108  
4930 Old Page Road  
Durham, NC 27709

Dear Mr. Grimley,

Enclosed are five bound copies and one unbound copy of Fossil Energy Research Corp.'s Report No. FERCo R743-Revised entitled "**Mercury Speciation Stack Sampling Test Report: Coronado Unit 1.**"

We are submitting these copies on behalf of Salt River Project to meet the requirements of the information collection request.

Sincerely,

Fossil Energy Research Corp.



Mark D. McDannel, P.E.

MDM/jw

# MERCURY SPECIATION STACK SAMPLING TEST REPORT: CORONADO UNIT 1

March 2000

**Prepared by**

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**Prepared for**

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# TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
<b>1 INTRODUCTION</b> .....	<b>1-1</b>
1.1 Summary of Test Program.....	1-1
Purpose of Test.....	1-1
1.2 Key Personnel.....	1-3
<b>2 PLANT AND SAMPLING LOCATION DESCRIPTIONS</b> .....	<b>2-1</b>
2.1 Process and Control Equipment Description and Operation .....	2-1
2.2 Flue Gas Sampling Locations .....	2-3
Inlet Locations.....	2-3
Outlet Location.....	2-3
2.3 Coal Sampling Location .....	2-4
<b>3 SUMMARY AND DISCUSSION OF TEST RESULTS</b> .....	<b>3-1</b>
3.1 Objectives and Test Matrix.....	3-1
Objectives.....	3-1
Test Matrix .....	3-1
3.2 Field Test Changes and Problems.....	3-1
High Sample Volume on Run 1-Outlet.....	3-1
Lost Nozzle on Run 3-Outlet .....	3-2
Holding Time .....	3-2
Change of Analytical Method and Laboratory for Mercury in Coal.....	3-2
Lab Errors and Additional QA Analysis .....	3-2
3.3 Presentation of Results.....	3-3
<b>4 SAMPLING AND ANALYTICAL PROCEDURES</b> .....	<b>4-1</b>
4.1 Test Methods.....	4-1
Sample Recovery.....	4-4
Sample Digestion and Analysis.....	4-4
Handling of Non Detects.....	4-5
Auxiliary Flue Gas Measurements .....	4-7
Determination of Scrubber Efficiency and Stack Emissions .....	4-8
4.2 Process Data.....	4-9

**Table of Contents (continued)**

**5 INTERNAL QA/QC ACTIVITIES..... 5-1**

    5.1 QA/QC Problems..... 5-1

    5.2 QA Audits and Data Quality Objectives..... 5-1

    5.3 Comparison Analyses ..... 5-2

**APPENDIX A. RESULTS AND CALCULATIONS ..... A-1**

**APPENDIX B. RAW FIELD DATA AND CALIBRATION DATA SHEETS ..... B-1**

**APPENDIX C. CHAIN-OF-CUSTODY RECORDS..... C-1**

**APPENDIX D. ANALYTICAL LAB REPORTS ..... D-1**

**APPENDIX E. AUDIT DATA SHEETS..... E-1**

**APPENDIX F. LIST OF PARTICIPANTS ..... F-1**

**APPENDIX G. ADDITIONAL INFORMATION..... G-1**

# LIST OF TABLES

<u>SECTION</u>	<u>PAGE</u>
Table 1-1. Test Program Organization and Responsibilities.....	1-4
Table 2-1. Summary of Coronado Unit 1 Operation.....	2-2
Table 2-2. Coronado Unit 1 Sampling Location Descriptions.....	2-4
Table 3-1. Test Matrix for Mercury ICR Tests at Coronado 1 .....	3-3
Table 3-2. Coronado Unit 1 Sampling Times .....	3-4
Table 3-3. Coronado Unit 1 Sample Gas Conditions.....	3-5
Table 3-4. Coronado Unit 1 Mercury Speciation Results .....	3-6
Table 3-5. Coronado Unit 1 Mercury Removal Efficiency and Estimated Stack Concentrations.....	3-7
Table 4-1. Sample Train Components - Method 17 Configuration .....	4-3
Table 4-2. Sample Train Components - Method 5 Configuration .....	4-3
Table 5-1. Audit Samples for Ontario Hydro Mercury Speciation .....	5-1
Table 5-2. Data Quality Objectives for Flue Gas Mercury Analyses .....	5-2
Table 5-3. Results Evaluation and Verification Checklist .....	5-3
Table 5-4. Coronado 1 Sample Fraction Mercury Measurements .....	5-4
Table 5-5. Results of Independent QA Analyses of Coronado 1 Samples .....	5-5

# LIST OF FIGURES

<u>SECTION</u>	<u>PAGE</u>
Figure 1-1. Project Organization Chart .....	1-5
Figure 2-1. Coronado Unit 1 Schematic.....	2-1
Figure 2-2. Coronado Unit 1 Scrubber Layout .....	2-5
Figure 2-3. Coronado Unit 1 Inlet Sample Location Cross Section.....	2-6
Figure 2-4. Coronado Unit 1 Outlet Sample Location Cross Section.....	2-6
Figure 3-1. Mercury Speciation Across Coronado Unit 1 Scrubber .....	3-8
Figure 4-1. Schematic of the Mercury Speciation Sample Train (Method 5 option as used at the stack is shown; Method 17 in-stack filtration was used for the Inlet on Coronado 1).....	4-2
Figure 4-2. Sample Recovery Scheme for the Mercury Sampling Train.....	4-6

# 1

## INTRODUCTION

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Note: This revised report replaces the original Coronado 1 Test Report (Report No. FERCo-R743), which was submitted to the EPA in January, 2000. This version contains corrected mercury emission results, following discovery of errors in the original laboratory report. The author recommends that copies of the original report be disposed of.

### 1.1 Summary of Test Program

#### *Purpose of Test*

The United States Environmental Protection Agency (EPA) has implemented an Information Collection Request (ICR) aimed at characterizing mercury emissions from coal-fired power plants in the United States. As part of this ICR, the operators of selected coal-fired boilers were required to collect and analyze flue gas samples for particulate, elemental, and oxidized mercury.

Salt River Project's (SRP's) Coronado Unit 1 was selected at random by the EPA to provide speciated mercury emissions data, which will then be used to develop emission factors for boilers in its class.

Measurements collected were speciated mercury emissions at the wet scrubber outlet, speciated mercury concentrations at the scrubber inlet, and fuel mercury, chlorine, moisture, sulfur, ash, and heating value.

#### Test Unit

The test unit is Coronado 1. This unit is operated by Salt River Project, and is located in St. Johns, Arizona. The unit was selected by the EPA as part of the following category:

- Fuel type: subbituminous
- SO<sub>2</sub> control type: wet scrubber
- Particulate control type: hot side electrostatic precipitator (ESP)

The unit is rated at 435 MW gross. Coronado 1 is a Riley Stoker turbo-fired boiler, with overfire air for NO<sub>x</sub> control. It fires approximately 0.4% sulfur subbituminous coal. SO<sub>2</sub> emissions are controlled by horizontal weir scrubbers.

## Test Measurements

The program included the following tests, with triplicate sets of measurements performed simultaneously at each test location:

- Particulate, oxidized, and elemental mercury emissions at the exhaust of Scrubber Module 1B (the only operating module during the tests) per the Ontario Hydro mercury speciation method.
- Particulate, oxidized, and elemental mercury concentrations at one of two air preheater exit ducts. This location, referred to as the “inlet”, is downstream of the hot side electrostatic precipitators and upstream of the wet scrubber.
- Mercury and chlorine content of representative coal samples collected from the coal feeders.
- Coal moisture, sulfur, ash, and heating content.

## Responsible Organizations

Responsible organizations for this project are:

- Test site operator: Salt River Project
- Program sponsor: Electric Power Research Institute (EPRI)
- Sampling team: Fossil Energy Research Corp. under contract to EPRI, with Delta Air Quality Services as a major subcontractor
- Sample analysis: Philip Analytical Services (flue gas mercury, coal chlorine), Commercial Testing and Engineering (coal HHV, S, ash, moisture), Frontier Geosciences (coal mercury), University of North Dakota Energy and Environmental Research Center (QA analyses on flue gas samples)

## Dates of Test

The test program was conducted on October 18-19, 1999. Daily activities included:

- October 18: set up and conducted Run 1.
- October 19: conducted Runs 2 and 3; conducted field blanks.

## Document Description

This document is the test report for the Coronado Unit 1 mercury ICR testing. It has been prepared in accordance with Emission Measurement Center Guideline Document GD-043, as required in the ICR.

The work described here is based on the Coronado Unit 1 Test Plan (Report No. FERCo R678), the Coronado Unit 1 Quality Assurance Plan (Report No. FERCo R701), and the Coronado Unit 1 Test Plan Addendum (Report No. FERCo R723). These reports are available from SRP, the EPA or FERCo.

The Test Plan Addendum was prepared in response to initial EPA review of the Test Plan. The Test Plan Addendum was approved by Mr. William Grimley of the EPA. The QA Plan was approved by Ms. Lara Autry of the EPA prior to testing. EPA comments on the draft QA Plan were incorporated into the final version of the QA Plan.

## **1.2 Key Personnel**

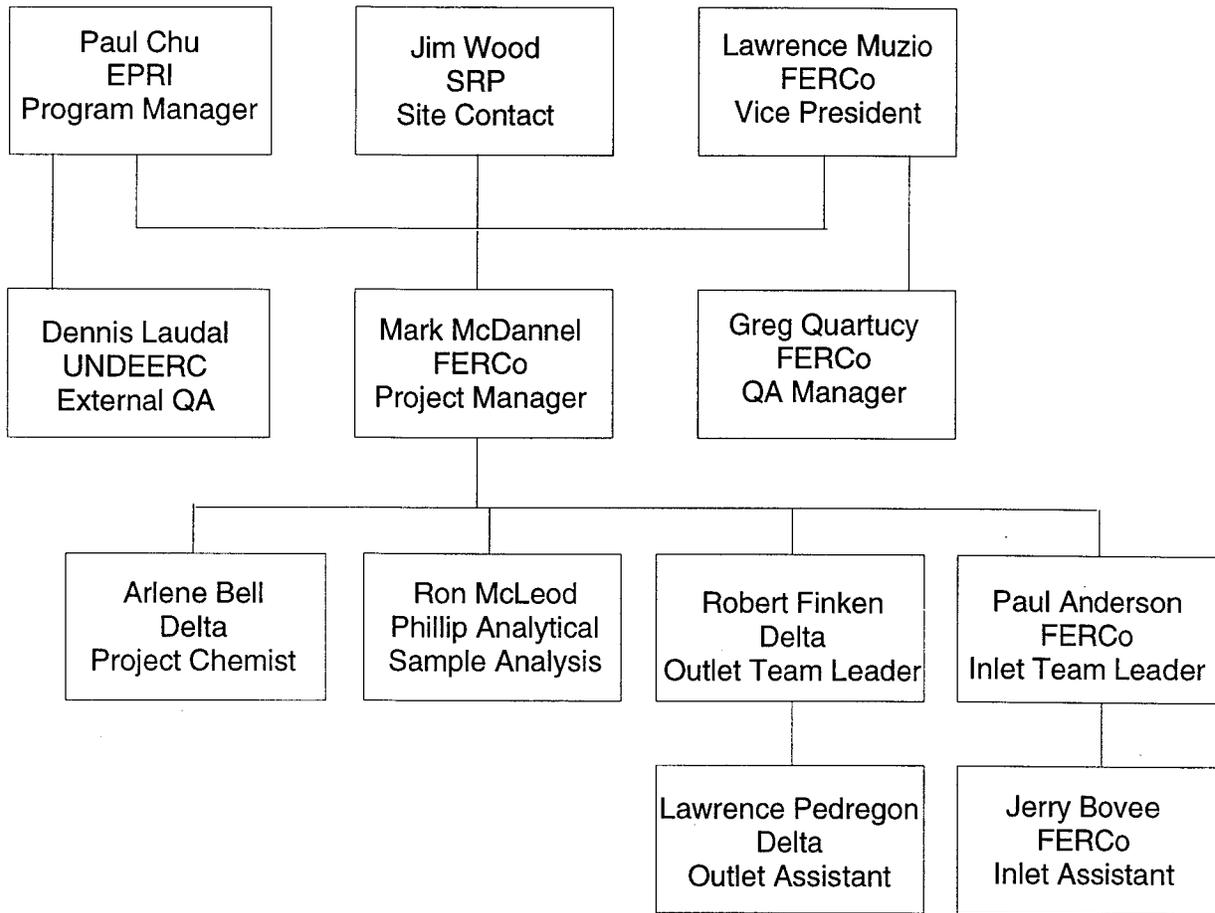
Table 1-1 lists the test program organization and key individuals with responsibilities, phone numbers, and e-mail addresses. A program organizational chart is shown in Figure 1-1.

The program was jointly funded by SRP and EPRI. FERCo was under contract to EPRI. The Project Quality Assurance Officer was Greg Quartucy of FERCo, who reported directly to Larry Muzio, FERCo's Vice President. External QA activities were performed by Dennis Laudal of UNDEERC. Mr. Laudal reported directly to Paul Chu of EPRI. Both UNDEERC and FERCo are contractors to EPRI. The reporting function from Mr. Laudal to Mr. Chu is considered to be external to FERCo's project.

Mr. Wood, Mr. McDannel, and Ms. Bell were all on-site for the testing. There were no observers from regulatory agencies.

**Table 1-1. Test Program Organization and Responsibilities**

Organization	Individual	Responsibility	Reports To	Phone Number	Fax Number	E-mail Address
Project Management and Oversight						
Electric Power Research Institute	Paul Chu	EPRI Project Manager	N/A	(650) 855-2812	(650) 855-2619	pchu@epri.com
FERCo	Lawrence Muzio	Vice President	N/A	(949) 859-4466	(949) 859-7916	lmuzio@ferco.com
FERCo	Greg Quartucy	QA Manager	Lawrence Muzio	(949) 859-4466	(949) 859-7916	gquartucy@ferco.com
Host Utility						
SRP	James Wood	Program Coordinator and Site Contact	N/A	(520) 337-4131 x2444	(520) 337-2961	JGWOOD@srpnet.com
FERCo/Delta Sampling Team						
FERCo	Mark McDannel	Program Manager	Paul Chu	(949) 859-4466	(949) 859-7916	mmcdannel@ferco.com
Delta	Arlene Bell	Project Chemist	Mark McDannel	(714) 279-6777	(714) 279-6781	deltaaqs@aol.com
Philip Environmental	Ron McLeod	Sample Analyses	Mark McDannel	(905) 332-8788	(905) 332-9169	rmcleod@philipinc.com
External QA/QC						
UNDEERC	Dennis Laudal	External QA/QC	Paul Chu	(701) 777-5138	(701) 777-5181	dlaudal@cerc.und.nodak.edu



**Figure 1-1. Project Organization Chart**



# 2

## PLANT AND SAMPLING LOCATION DESCRIPTIONS

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### 2.1 Process and Control Equipment Description and Operation

Coronado 1 is a Riley Stoker turbo-fired boiler rated at 435 MW gross. Figure 2-1 shows a schematic of the boiler and pollution control equipment, including sample points.

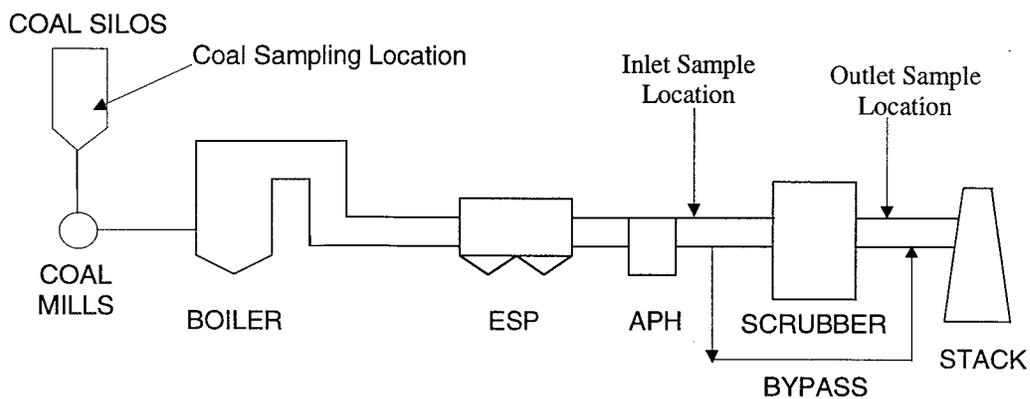


Figure 2-1. Coronado Unit 1 Schematic

Key unit parameters include:

- Unit capacity: 435 MW gross
- Boiler type: Riley Stoker, turbo-fired, balanced draft
- Fuel type: subbituminous, 0.4 – 0.5% S
- SO<sub>2</sub> control: limestone wet scrubber, horizontal weir design. There are two scrubber modules, designated 1A and 1B. One module is in operation at a time. There is a scrubber bypass system; the amount of flue gas scrubbed is controlled to meet SO<sub>2</sub> emission limits.
- Particulate control: hot side ESP, 99.7% efficiency
- NO<sub>x</sub> control: overfire air

Fuel samples were collected at the coal feeders ahead of the boiler, inlet samples were collected at one of the air preheater exit ducts, and outlet samples were collected at the outlet of scrubber module 1B.

The sample gas at the inlet is approximately 280°F. At the outlet the gas temperature is approximately 120°F, and the gas is saturated with moisture.

Unit operation during testing was at or near nominal full load, at steady state operation. Scrubber module 1B was in service, and its booster fan (which controls gas flow through the scrubber) was placed in manual control. Coal type, boiler operation, and control device operation were all within normal operating ranges.

Table 2-1 presents a summary of unit operation during the tests. Additional detailed unit data is included in Appendix G.

**Table 2-1. Summary of Coronado Unit 1 Operation**

Run No.	1	2	3	Average
Date, 1999	18-Oct	19-Oct	19-Oct	
Start time	1410	0809	1216	
Stop time	1658	1103	1456	
Unit load, MW gross	428	431	431	430
Coal mills in service	All 3	All 3	All 3	
Coal flow, klb/hr	406	405	415	409
Boiler O <sub>2</sub> , %	2.38	2.38	2.40	2.39
CEMS data				
CO <sub>2</sub> , % wet	13.24	13.54	13.58	13.45
SO <sub>2</sub> , lb/MMBtu	0.51	0.53	0.54	0.53
NO <sub>x</sub> , lb/MMBtu	0.50	0.50	0.51	0.50
Opacity, %	10	9	8	9
Stack flow, kwscfh	926	915	917	919
Stack temperature, F	215	216	229	220
ESP data				
Power level, kW	897	835	936	889
T/R sets in service	54	54	54	54
T/R sets out of service	0	0	0	0
Gas Temperature, F	649	660	674	661
FGD Data				
Module in service	B	B	B	
Inlet temperature	255	254	262	257
Outlet temperature	118	105	119	114
Pressure drop	2.1	2.6	2.0	2.2
Booster fan amps	189	187	184	187
Slurry flow rate	10,000	10,000	10,000	10,000

## 2.2 Flue Gas Sampling Locations

Table 2-2 presents a summary of key inlet and outlet sample location parameters. Individual discussions of the two locations are presented below.

### ***Inlet Locations***

Figure 2-2 shows a schematic of the scrubber duct arrangement, and Figure 2-3 shows a cross section of the inlet sample ports.

The flue gas exits the boiler through two air preheaters, then goes through two induced draft fans into the bypass duct. The booster fan for the operating scrubber module pulls the gas to be treated, and the balance of the gas exhausts through the stack untreated.

The inlet samples were collected in the exit duct of air preheater (APH) 1B on Coronado 1. The APH exit duct was selected over the scrubber inlet duct because it offers a longer run of straight duct and the sample ports are more easily accessible.

Because of the number and location of the inlet ducts, it was not feasible to sample both air preheater exit ducts simultaneously with the outlet sample without adding an additional sample team. Sampling one of two ducts should adequately characterize mercury speciation at the inlet, and is consistent with ICR requirements.

The sample traverse scheme for Coronado 1 inlet was:

5 ports x 4 points/port x 7 ½ minutes/point = 150 minutes.

This location meets the requirements of EPA Method 1. A cyclonic flow check was done before testing. The average yaw angle was 5 degrees, with one point at 30 degrees and all other points at 10 degrees or less.

### ***Outlet Location***

Because most of the boiler flue gas is bypassed around the FGD system on Coronado 1, it is not possible to directly measure both stack emissions and scrubber removal efficiency. Sampling at the stack provides a direct measure of emissions but removal efficiency must be calculated, while the converse is true for sampling at the scrubber outlet.

For Coronado Unit 1, sampling was performed at the outlet because: (1) there are accessible outlet sample ports which allow direct measurement of scrubber removal, and (2) with most of the gas being bypassed, it would be difficult to precisely determine scrubber removal efficiency from stack measurements. Calculation procedures to determine control device efficiency and stack emissions are presented in Section 4.1.

The outlet samples were collected at the ports at the outlet of Module 1B. Module 1B was chosen rather than Module 1A because the 1A outlet duct has very little straight run. Since only one module operates at a time, all of the scrubbed gas on Coronado 1 was sampled. A cross section of the outlet location is shown in Figure 2-4.

The sample traverse scheme for Coronado 1 outlet was:

6 ports x 5 points/port x 5 minutes/point = 150 minutes.

This location does not meet the requirements of EPA Method 1. A cyclonic flow check was performed before testing, and the angle at all traverse points was less than 5 degrees.

**Table 2-2. Coronado Unit 1 Sampling Location Descriptions**

	<b>Inlet</b>	<b>Outlet</b>
Description	Air preheater exit/scrubber inlet	Scrubber 1B outlet
Elevation	Approximately 50'	Approximately 90'
Physical access	Ladder	Stairs, ladder
Side or top access	Top	Top
Round or rectangular	Rectangular	Rectangular
Port length (outside of port to inner stack wall)	18"	18"
Number/type of ports	Five 4-inch w/flanges	Six 4-inch w/flanges
Inside dimensions	16' 0" deep x 15' 0" wide Equivalent diameter 15.5 ft	12' 0 deep x 13' 0" wide Equivalent diameter 12.5 ft
Nearest upstream disturbance		
Disturbance	APH exit	45 deg bend in duct
Distance, ft	100'	24'
Distance, diameters	6.5	1.9
Nearest downstream disturbance		
Disturbance	30 deg jog in duct	Bypass duct junction
Distance, ft	20'	19'
Distance, diameters	1.3	1.5

### 2.3 Coal Sampling Location

Coal samples were collected from the silo just above the coal feeders to each individual mill. One one-pint jar sample was collected from each mill during the first and last hour of each test run, and all samples were composited. Samples were collected by Coronado station personnel.

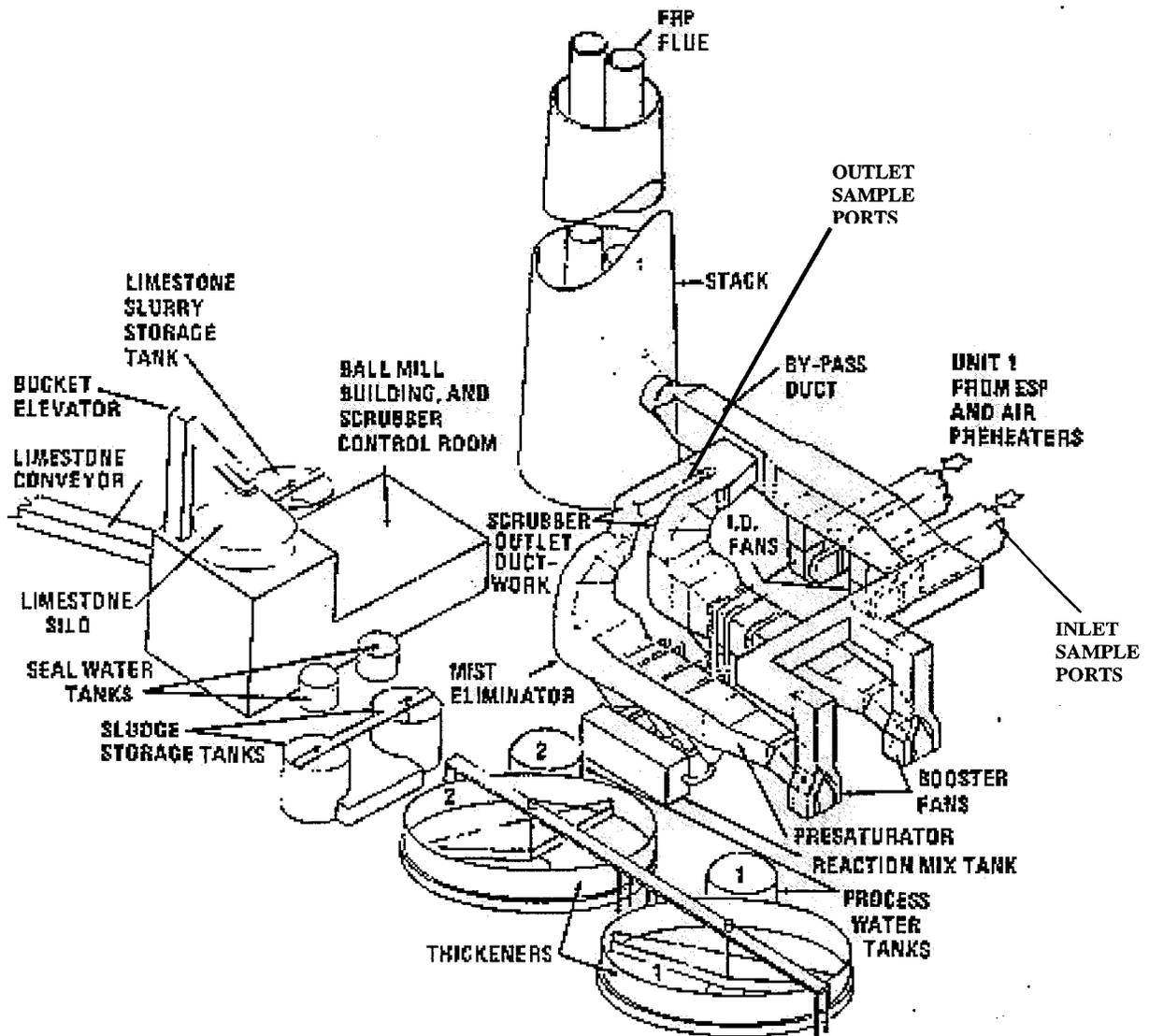
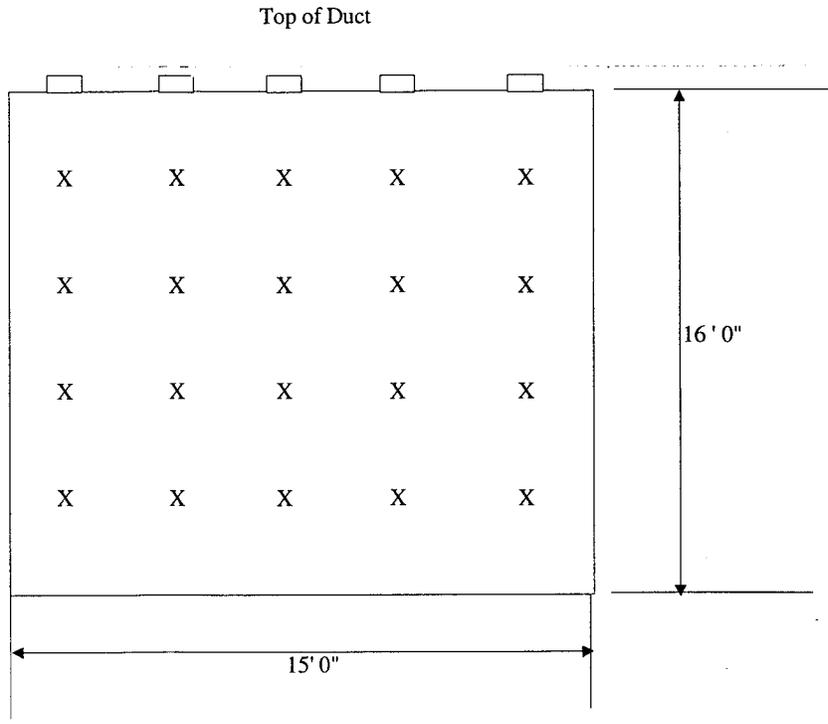
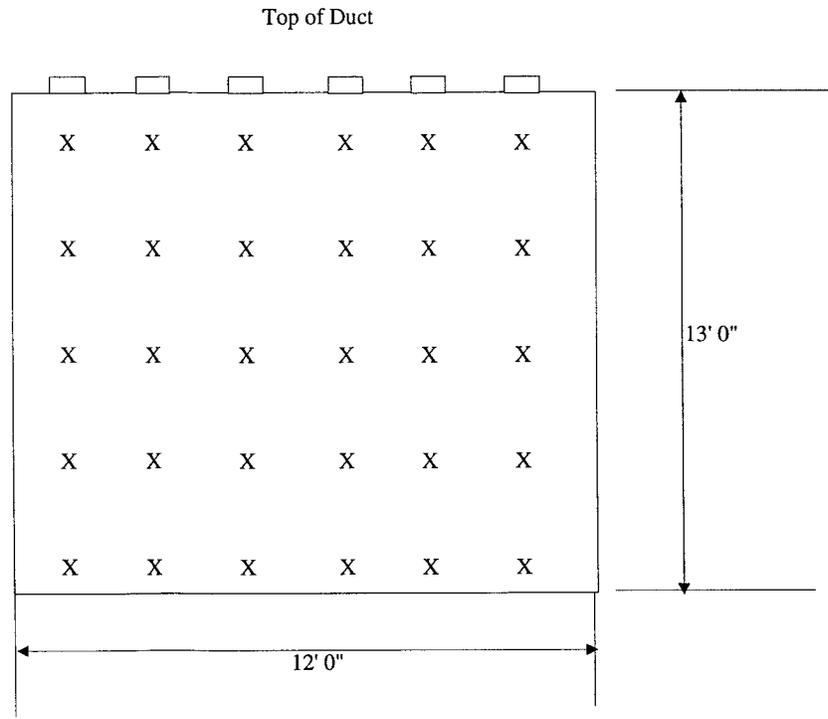


Figure 2-2. Coronado Unit 1 Scrubber Layout



**Figure 2-3. Coronado Unit 1 Inlet Sample Location Cross Section**



**Figure 2-4. Coronado Unit 1 Outlet Sample Location Cross Section**

# 3

## SUMMARY AND DISCUSSION OF TEST RESULTS

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### 3.1 Objectives and Test Matrix

#### *Objectives*

The objective of the program is to collect the information and measurements required by the EPA Mercury ICR. Specific objectives are:

- Quantify speciated mercury concentrations at the scrubber outlet, and estimate speciated mercury emissions at the stack.
- Quantify speciated mercury concentrations in the flue gas at the scrubber inlet.
- Quantify fuel mercury and chlorine content during the stack and inlet tests.
- Provide the above information for use in developing boiler-, fuel-, and control device-specific mercury emission factors.

#### *Test Matrix*

The test matrix is presented in Table 3-1, and actual test times are shown in Table 3-2. Table 3-1 includes a list of test methods used. In addition to speciated mercury, the flue gas measurements included moisture, stack gas flow, and O<sub>2</sub>/CO<sub>2</sub>.

### 3.2 Field Test Changes and Problems

#### *High Sample Volume on Run 1-Outlet*

The volume of sample collected on Run 1-Outlet was 2.72 standard cubic meters (scm), which is 9% higher than the Ontario Hydro Method upper limit of 2.5 scm. The target volume was exceeded because (1) the selected nozzle was targeted for the upper end of the allowable sampling rate range, and (2) duct flow was higher during the test than it was during the preliminary velocity traverse.

The sample volume upper limit was placed in the Ontario Hydro Method to avoid complete consumption of the KMnO<sub>4</sub> in Impingers 5, 6, and 7 by SO<sub>2</sub>. Since SO<sub>2</sub> levels at Coronado 1 outlet are low and since it was confirmed after the test that the H<sub>2</sub>SO<sub>4</sub> impingers were still purple, exceeding the 2.5 scm target is not believed to have any impact on the results.

This decision was confirmed on-site by a phone conversation with Richard Schultz of EERC, one of the authors of the Ontario Hydro Method.

### ***Lost Nozzle on Run 3-Outlet***

During removal of the sample probe from Port B on Run 3-Outlet, the sample nozzle caught on the inside lip of the port and was pulled off. The sample train was leak checked, the nozzle was replaced with a nozzle of the same size, the train was leak checked again, and the test was restarted. This should have no significant impact on the results, as the mass of fly ash lost in the nozzle is negligible and particulate mercury represented only 2% of total mercury at the outlet.

### ***Holding Time***

Due to delays and high work loads in the laboratory, not all the sample fractions were analyzed within the 45 day holding time specified in the Ontario Hydro Method. The filter fractions were analyzed 58 days after sampling, and the H<sub>2</sub>O<sub>2</sub> impingers were analyzed 51 days after sampling. The KCl and KMnO<sub>4</sub> impinger fractions were analyzed within 45 days.

This discrepancy is not considered to have any impact on the results. Dennis Laudal of the University of North Dakota (the author of the Ontario Hydro Method) indicates that they have performed stability studies showing that samples are stable for at least three months.

### ***Change of Analytical Method and Laboratory for Mercury in Coal***

The test plan called for coal mercury analysis to be performed by Philip Analytical, using EPA SW 846. However, the results for all three samples were not detected less than 0.04 ppm.

In an effort to achieve lower detection limits and be able to quantify mercury in the coal, splits of the samples were analyzed by Frontier Geosciences. The samples were digested by cold aqua regia (modified EPA 7371) and analyzed by cold vapor atomic fluorescence (modified EPA 1631). These methods provided detectable levels of mercury in the coal, and are used as the reported mercury values.

### ***Lab Errors and Additional QA Analysis***

Subsequent to submittal of the original Test Report to the EPA, two errors were discovered in the reported results: (1) the inlet KMnO<sub>4</sub> fractions were discovered to be high by a factor of 1.5, due to a systematic calculation error, and (2) upon re-analysis the KMnO<sub>4</sub> fraction for Run 2-Outlet was determined to be 2.6 micrograms rather than 4.5 micrograms.

In order to provide additional confidence in the revised results, splits of all KMnO<sub>4</sub> and KCl fractions were analyzed by EERC. These results confirmed all of the applicable Philip results, and are presented in Section 5.

### 3.3 Presentation of Results

The test results are presented in the following tables and figure:

- Table 3-3. Sample gas conditions.
- Table 3-4. Mercury concentration and speciation results.
- Table 3-5. Mercury removal across scrubber by species and estimated stack concentrations.
- Figure 3-1. Mercury speciation across scrubber.

**Table 3-1. Test Matrix for Mercury ICR Tests at Coronado 1**

Sampling Location	No. of Runs	Species Measured	Sampling Method	Sample Run Time	Analytical Method	Analytical Laboratory
Outlet	3	Speciated Hg	Ontario Hydro	150 min	Ontario Hydro	Philip Services
Outlet	3	Moisture	EPA 4	Concurrent	Gravimetric, compared with saturation value	FERCo
Outlet	3	Gas Flow	EPA 1/2	Concurrent	Pitot Traverse	FERCo
Outlet	3	O <sub>2</sub>	Batch Sample	Concurrent	Portable O <sub>2</sub>	FERCo
Outlet	3	CO <sub>2</sub>	N/A	Concurrent	Stoichiometric calculation	FERCo
Inlet	3	Speciated Hg	Ontario Hydro	150 min	Ontario Hydro	Philip Services
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric	FERCo
Inlet	3	Gas Flow	EPA 1/2	Concurrent	Pitot Traverse	FERCo
Inlet	3	O <sub>2</sub>	Batch Sample	Concurrent	Portable O <sub>2</sub>	FERCo
Inlet	3	CO <sub>2</sub>	N/A	Concurrent	Stoichiometric calculation	FERCo
Coal Feeders	3	Cl in coal	Modified ASTM D2234	1 grab sample per mill per run	EPA SW 846: 5050/9056 (Cl)	Philip
Coal Feeders	3	HHV, Ash, S, Moisture	Modified ASTM D2234	1 grab sample per mill per run	ASTM D514290	CTE
Coal Feeders	3	Hg in coal	Modified ASTM D2234	1 grab sample per mill per run	Modified EPA 7371/1631	Frontier Geosciences

**Table 3-2. Coronado Unit 1 Sampling Times**

	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>
Date, 1999	18-Oct	19-Oct	19-Oct
<b>Inlet Tests</b>			
Start time	1410	0809	1216
Stop time	1658	1103	1456
Total sample time, min	150	150	150
<b>Stack Tests</b>			
Start time	1410	0811	1219
Stop time	1657	1052	1502
Total sample time, min	150	150	150
Notes:			
1. Gas flow, moisture, O <sub>2</sub> were concurrent with mercury tests.			
2. Coal samples were collected during the first and last hour of each run.			

Results are calculated as  $\mu\text{g}/\text{scm}$  (at a reference temperature of 68°F), and normalized for dilution by converting to a  $\text{lb}/10^{12}$  Btu basis. This method allows direct comparison of inlet and stack results without incorporating uncertainties involved in gas flow measurement.

Major observations that can be made from the results are:

1. Mercury is primarily in the elemental phase at both the inlet (67% of total mercury) and at the outlet (95% of total mercury). Oxidized mercury was 33% of the total at the inlet and 2% of the total at the outlet. There was no measurable particulate mercury at the inlet, and particulate mercury was 3% of total mercury at the outlet.
2. Mercury levels in the coal averaged  $3.1 \text{ lb}/10^{12}$  Btu, or 0.035 ppm. This is 48% higher than the ESP inlet concentration of  $2.1 \text{ lb}/10^{12}$  Btu.
3. Oxidized mercury was removed with 93% efficiency across the scrubber, elemental mercury increased by 43% across the scrubber, and particulate mercury increases from  $\text{ND}<0.04 \text{ lb}/10^{12}$  Btu to  $0.05 \text{ lb}/10^{12}$  Btu. The increase in particulate mercury is not considered significant relative to the uncertainty of the method. The increase in elemental mercury is at the outside boundary of the uncertainty of the method, and may or may not be significant.

**Table 3-3. Coronado Unit 1 Sample Gas Conditions**

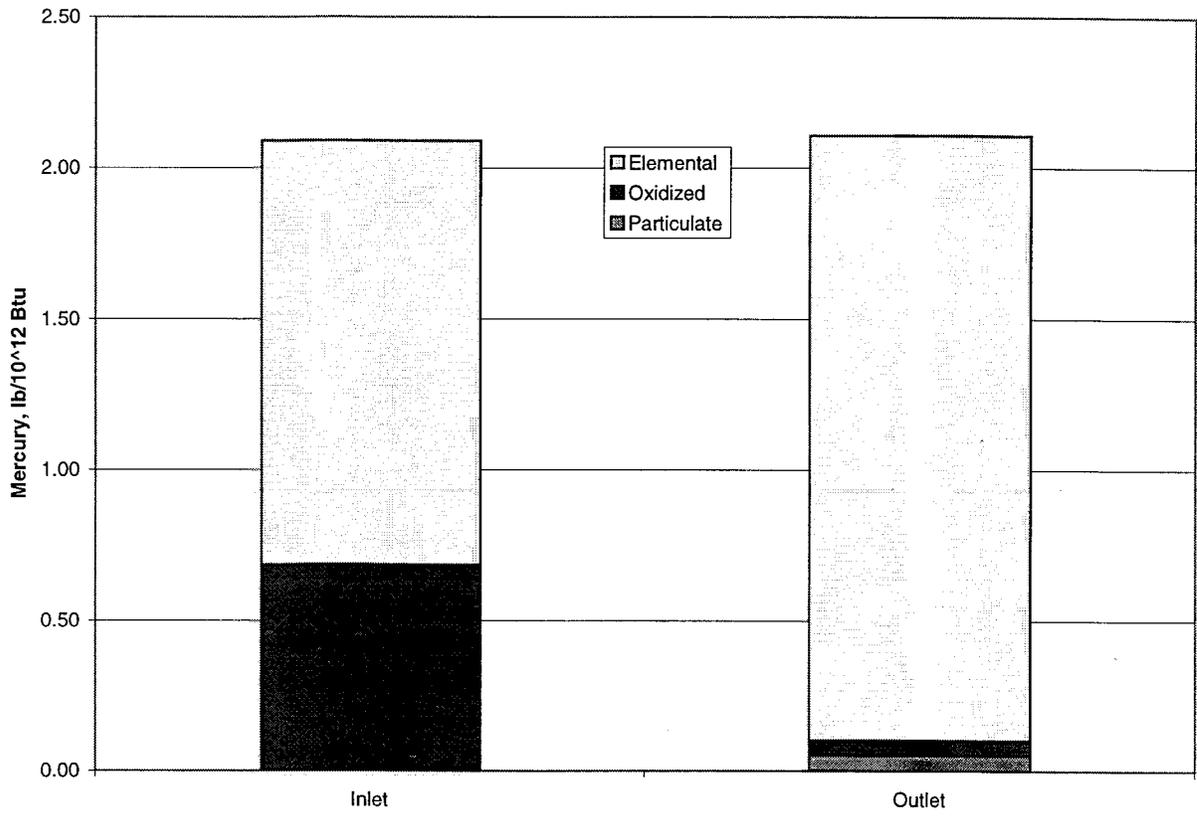
	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>	<b>Average</b>
<b>Test Date</b>	18-Oct	19-Oct	19-Oct	
<b>Inlet Gas Properties</b>				
Temperature, F	276	275	289	280
Gas flow, dscfm from pitot traverse x 2	827,435	823,298	819,326	823,353
Comparison gas flows, dscfm				
Calculated from fuel input and O <sub>2</sub>	840,343	820,185	813,979	824,836
O <sub>2</sub> , % dry	4.94	4.27	3.97	4.39
CO <sub>2</sub> , % dry	14.05	14.64	14.91	14.54
H <sub>2</sub> O, %	10.05	9.58	10.27	9.97
<b>Outlet Gas Properties</b>				
Temperature, F	119	117	120	119
Outlet gas flow, dscfm (pitot traverse)	351,474	326,866	321,550	333,297
O <sub>2</sub> , % dry	5.71	5.42	5.39	5.51
CO <sub>2</sub> , % dry	13.38	13.63	13.66	13.56
H <sub>2</sub> O, %	13.70	12.90	13.80	13.47
<b>Stack Gas Properties</b>				
Temperature, F	215	216	229	220
CEMS flow, dscfm	815,493	812,475	802,614	810,194
CO <sub>2</sub> , % dry	11.67	12.03	11.88	11.86
<b>Scrubber bypass, % of boiler gas flow</b>				
	57%	60%	60%	59%

**Table 3-4. Coronado Unit 1 Mercury Speciation Results**

	Run 1	Run 2	Run 3	Average
<b>Test Date</b>	18-Oct	19-Oct	19-Oct	
<b>Inlet Mercury Speciation</b>				
Particulate mercury				
ug/dscm	ND<0.05	ND<0.05	ND<0.05	ND<0.05
lb/10 <sup>12</sup> Btu	ND<0.04	ND<0.04	ND<0.04	ND<0.04
% of total Hg	0%	0%	0%	0%
Oxidized mercury				
ug/dscm	0.88	0.76	1.03	0.89
lb/10 <sup>12</sup> Btu	0.70	0.58	0.78	0.69
% of total Hg	31%	31%	37%	33%
Elemental mercury				
ug/dscm	1.95	1.73	1.77	1.82
lb/10 <sup>12</sup> Btu	1.56	1.32	1.33	1.40
% of total Hg	69%	69%	63%	67%
Total mercury				
ug/dscm	2.83	2.49	2.81	2.71
lb/10 <sup>12</sup> Btu	2.26	1.90	2.11	2.09
<b>Outlet Mercury Speciation</b>				
Particulate mercury				
ug/dscm	ND<0.026	0.073	0.097	0.061
lb/10 <sup>12</sup> Btu	ND<0.022	0.060	0.080	0.050
% of total Hg	0%	4%	3%	3%
Oxidized mercury				
ug/dscm	0.037	ND<0.061	0.115	0.061
lb/10 <sup>12</sup> Btu	0.031	ND<0.050	0.095	0.050
% of total Hg	1%	0%	4%	2%
Elemental mercury				
ug/dscm	3.02	1.58	2.67	2.42
lb/10 <sup>12</sup> Btu	2.53	1.30	2.19	2.01
% of total Hg	99%	96%	93%	95%
Total mercury				
ug/dscm	3.05	1.66	2.88	2.54
lb/10 <sup>12</sup> Btu	2.56	1.36	2.36	2.11
<b>Coal Analysis</b>				
Mercury, ppm dry	0.035	0.039	0.031	0.035
Mercury, lb/10 <sup>12</sup> Btu	3.1	3.4	2.8	3.1
Chlorine, ppm dry	100	ND<100	200	117
Moisture, %	13.3	13.6	13.5	13.5
Sulfur, % dry	0.53	0.42	0.51	0.49
Ash, % dry	17.8	17.1	18.3	17.7
HHV, Btu/lb as fired	9,704	9,881	9,738	9,774
Coal flow, lb/hr as fired	405,700	405,200	415,400	408,767
<b>Total Mercury Mass Rates</b>				
lb/hr input in coal	0.012	0.014	0.011	0.012
lb/hr at ESP outlet (total unit gas flow)	0.009	0.008	0.009	0.008
lb/hr at scrubber outlet (41% of unit gas flow)	0.004	0.002	0.003	0.003
lb/hr at stack	0.009	0.007	0.009	0.008

**Table 3-5. Coronado Unit 1 Mercury Removal Efficiency and Estimated Stack Concentrations**

	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>	<b>Average</b>
Date, 1999	18-Oct	19-Oct	19-Oct	
<b>Total mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	2.26	1.90	2.11	2.09
Outlet, lb/10 <sup>12</sup> Btu	2.56	1.36	2.36	2.10
Removal efficiency, %	-13%	28%	-12%	0%
Stack, lb/10 <sup>12</sup> Btu	2.39	1.69	2.21	2.10
<b>Particulate mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	ND<0.04	ND<0.04	ND<0.04	ND<0.04
Outlet, lb/10 <sup>12</sup> Btu	ND<0.02	0.06	0.08	0.05
Removal efficiency, %	N/A	N/A	N/A	N/A
Stack, lb/10 <sup>12</sup> Btu	N/A	N/A	N/A	N/A
<b>Oxidized mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	0.70	0.58	0.78	0.69
Outlet, lb/10 <sup>12</sup> Btu	0.03	ND<0.050	0.09	0.05
Removal efficiency, %	96%	>90%	88%	93%
Stack, lb/10 <sup>12</sup> Btu	0.41	0.37	0.50	0.43
<b>Elemental mercury</b>				
Inlet, lb/10 <sup>12</sup> Btu	1.56	1.32	1.33	1.40
Outlet, lb/10 <sup>12</sup> Btu	2.53	1.30	2.19	2.01
Removal efficiency, %	-62%	1%	-64%	-43%
Stack, lb/10 <sup>12</sup> Btu	1.98	1.31	1.68	1.66
Note: Outlet measurements and removal efficiencies are direct measurements.				
Stack values are calculated from inlet/outlet concentrations and inlet/outlet/stack flows.				



**Figure 3-1. Mercury Speciation Across Coronado Unit 1 Scrubber**

# 4

## SAMPLING AND ANALYTICAL PROCEDURES

---

### 4.1 Test Methods

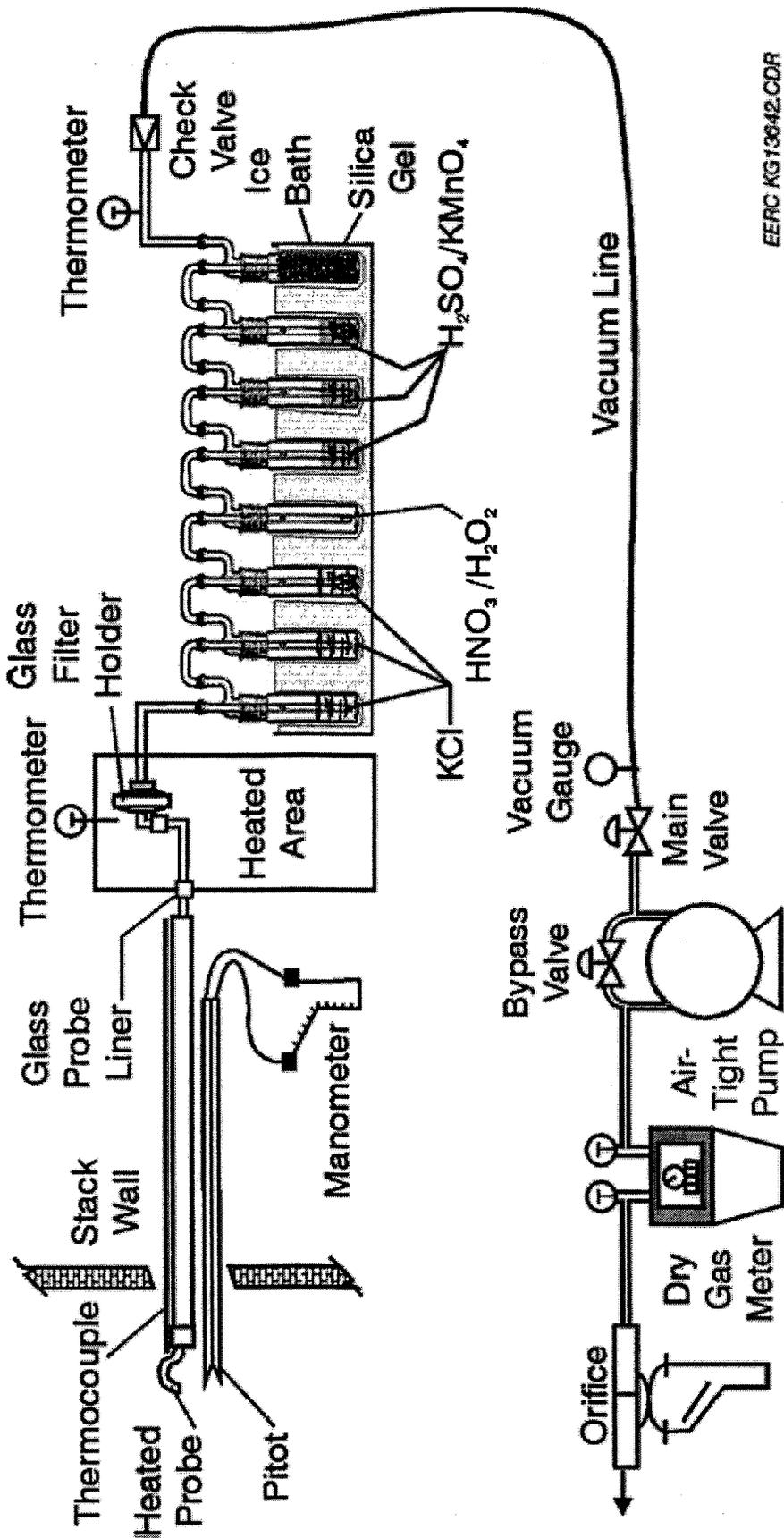
This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation required in EPA's ICR titled, "Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method)" dated April 8, 1999. The full text of the method was presented as Appendix A of the Test Plan.

Subsequent to submittal of the Test Plan, additional drafts of the Ontario Hydro Method were published. Wherever possible, the new features of these drafts were incorporated into the program.

Speciated mercury samples were collected in three test runs at the inlet and outlet of the control device. The inlet and outlet sampling were concurrent. A field blank was collected at each test location on October 19. The field blank consisted of assembling a sample train, transporting it to the sample location, conducting a leak check, letting the train sit for two to three hours, and then recovering the train as if it were a sample.

EPA methods to determine flue gas flow rate were used. EPA Reference Method 5 and 17 requirements for isokinetic sampling were followed. Each impinger was weighed before and after sampling to determine flue gas moisture content.

Figure 4-1 presents a schematic of the mercury speciation sample train, Table 4-1 presents a list of sample train components for the Method 17 configuration, and Table 4-2 presents a list of sample train components for the Method 5 configuration. The sampling train was set up with in-stack filtration (EPA Method 17 configuration) for the inlet location and external heated filtration (EPA Method 5 configuration) for the stack location.



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Figure 4-1. Schematic of the Mercury Speciation Sample Train (Method 5 option as used at the stack is shown; Method 17 in-stack filtration was used for the Inlet on Coronado 1)

**Table 4-1. Sample Train Components - Method 17 Configuration**

Component	Details
Nozzle	Glass.
Filter	Quartz thimble, in glass thimble holder.
Probe	Teflon, heated to minimum 120 C.
Connector line	Heated teflon line used to connect from probe to impingers. Heated to minimum 120 C.
Impingers 1, 2	1 mol/l KCl solution; modified Smith Greenburg (SG) impinger.
Impinger 3	1 mol/l KCl solution; standard Smith Greenburg impinger.
Impinger 4	5% nitric acid/10% hydrogen peroxide; modified SG impinger.
Impingers 5, 6	4% potassium permanganate/10% sulfuric acid; modified SG impinger.
Impinger 7	4% potassium permanganate/10% sulfuric acid; standard SG impinger.
Impinger 8	Silica gel; modified Smith Greenburg Impinger

**Table 4-2. Sample Train Components - Method 5 Configuration**

Component	Details
Nozzle	Glass
Probe	Glass, heated to minimum 120 C.
Filter	Quartz, in glass holder, heated to minimum 120 C.
Filter support	Teflon.
Connector line	Heated teflon line used to connect from filter outlet to impingers. Heated to minimum 120 C.
Impingers 1, 2	1 mol/l KCl solution; modified Smith Greenburg (SG) impinger.
Impinger 3	1 mol/l KCl solution; standard Smith Greenburg impinger.
Impinger 4	5% nitric acid/10% hydrogen peroxide; modified SG impinger.
Impingers 5, 6	4% potassium permanganate/10% sulfuric acid; modified SG impinger.
Impinger 7	4% potassium permanganate/10% sulfuric acid; standard SG impinger.
Impinger 8	Silica gel; modified Smith Greenburg Impinger

Sample was withdrawn from the flue gas stream isokinetically through the filtration system, which was followed by a series of impingers in an ice bath. Particulate-bound mercury was collected on the front half and filter; oxidized mercury was collected in impingers containing 1 N potassium chloride solution; and elemental mercury was collected in one impinger containing a 5% nitric acid and 10% peroxide solution, and in three impingers containing a solution of 10% sulfuric acid and 4% potassium permanganate. An impinger containing silica gel collected any remaining moisture.

The filter media was quartz fiber filters. At both the inlet and outlet quartz thimbles in a glass holders were used. At the inlet the probe included a heated teflon line; at the stack a heated glass probe was used. An additional heated teflon line was used to transport the flue gas from the end of the probe to the inlet of the first impinger. Both the probe and the line were heated to maintain a minimum gas temperature of 248°F.

Sample time at both the inlet and outlet was 150 minutes, with a target sample volume of 1 to 2.5 standard cubic meters.

### ***Sample Recovery***

Figure 4-2 is a schematic of the sample recovery procedure for the impinger train. The samples were recovered into precleaned glass bottles with vented teflon lined lids for shipment to the laboratory. The following sample fractions were recovered (specific rinse solutions are contained in the method):

1. The sample filter;
2. The front half rinse (includes all surfaces upstream of the filter)
3. Impinger 1 through 3 (KCl impingers) and rinses;
4. Impinger 4 (HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impinger) and rinses;
5. Impingers 5 through 7 (KMnO<sub>4</sub>/H<sub>2</sub>SO<sub>4</sub> impingers) and rinses;
6. Impinger 8 (silica gel impinger). Note this sample is weighed for moisture determination and is not included in the mercury analysis.

### ***Sample Digestion and Analysis***

The sample fractions were digested and analyzed as specified in the method and summarized below:

#### **Ash Sample (Containers 1 and 2)**

If the particulate catch is greater than 1 gram (as would be the case at most particulate control device inlet locations), an aliquot of the particulate collected on the filter is digested by microwave digestion.

### KCl Impingers (Container 3)

The impingers are digested using H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, and KMnO<sub>4</sub> solutions as specified in the method.

### KNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub> Impinger (Container 4)

The impinger solution is digested using HCl and KMnO<sub>4</sub> solutions as specified in the method.

### H<sub>2</sub>SO<sub>4</sub>-KMnO<sub>4</sub> Impingers (Container 5)

The impinger solution is digested using hydroxylamine sulfate as specified in the method.

### Analysis

Each digested fraction is analyzed in duplicate for total mercury by cold vapor atomic absorption (CVAAS). CVAAS is a method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorbency is measured as a function of mercury concentration. A soda-lime trap and a magnesium perchlorate trap must be used to precondition the gas before it enters the absorption cell.

### ***Handling of Non Detects***

This section addresses how data was handled in cases where no mercury was detected in an analytical fraction.

*A single analytical fraction representing a subset of a mercury species is not detected.* When more than one sample component is analyzed to determine a mercury species and one fraction is not detected, it is counted as zero. This occurred on all of the samples for elemental mercury, which is the sum of the mercury collected in the HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impinger and the H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> impingers. For example, on Test 3-Outlet the H<sub>2</sub>O<sub>2</sub> fraction was ND<0.25 µg and the KMnO<sub>4</sub> fraction was 4.4 µg. Elemental mercury was reported as 4.4 µg.

*Mercury is detected on one or two of three runs.* If mercury is detected on one or two of three runs, average mercury is calculated as the average of the detected value(s) and half of the detection limits for the non detect(s).

For example, the particulate mercury results for the three outlet tests (in units of lb/10<sup>12</sup> Btu) are ND<0.022, 0.060, and 0.080. The average using half the detection limit is (0.022/2 + 0.060 + 0.080)/3, or 0.050.

1. Rinse filter holder and connector with 0.1 N HNO<sub>3</sub>.
2. Add H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> to each impinger bottle until purple color remains.
3. Rinse with 0.1 N HNO<sub>3</sub>.
4. Rinse with 8N HCl if brown residue remains.
5. Final rinse with 0.1 N HNO<sub>3</sub>.

Rinse Bottles Sparingly with  
 - 0.1N HNO<sub>3</sub>  
 8N HCl  
 - 0.1N HNO<sub>3</sub>

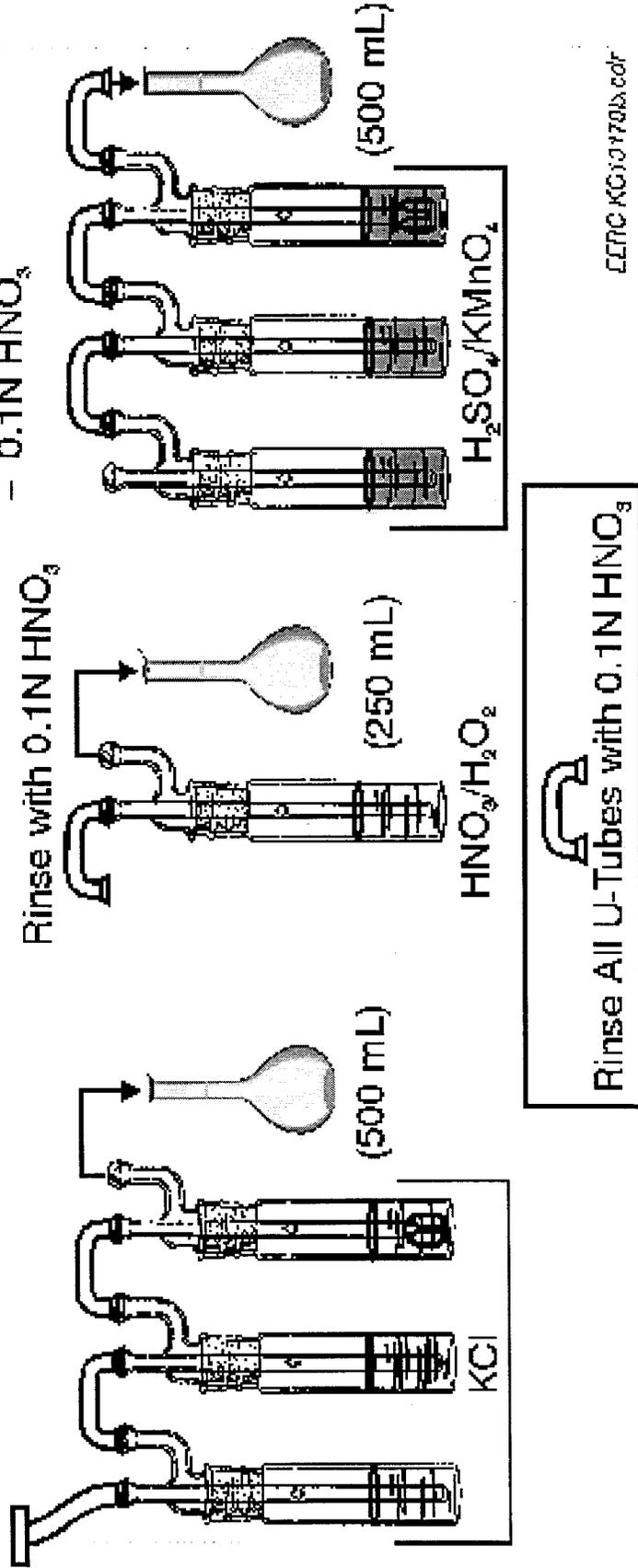


Figure 4-2. Sample Recovery Scheme for the Mercury Sampling Train

*No mercury is detected for a species on all three test runs.* When all three test runs show no detectable levels of mercury for a mercury species, that mercury species is reported as not detected at less than highest detection limit. For example, the results for the three inlet particulate mercury runs were all ND<0.04 lb/10<sup>12</sup> Btu. The average is reported as ND<0.04 lb/10<sup>12</sup> Btu.

In summing up individual species to determine total mercury, a value of zero is used for non-detected species. For example, the average inlet mercury values (in lb/10<sup>12</sup> Btu) were ND<0.04 for particulate mercury, 0.69 for oxidized mercury, and 1.40 for elemental mercury. Total mercury is reported as 0.69 + 1.40, or 2.09.

In calculating the percentage of mercury in each species, a value of zero is used for the non-detected species. For the example listed in the preceding paragraph, the results are reported as 0% particulate mercury, 33% oxidized mercury, and 67% elemental mercury.

### ***Auxiliary Flue Gas Measurements***

Auxiliary flue gas measurements performed were flue gas flow rate per EPA Methods 1 and 2 (pitot traverse), O<sub>2</sub> by portable O<sub>2</sub> analyzer (as described below), and H<sub>2</sub>O by EPA Method 4 (condensation/gravimetric analysis). These measurements were collected as integral parts of all mercury speciation test runs at both the inlet and stack locations.

#### **Outlet Moisture**

Measured moisture values at the outlet were compared with saturation moistures for each test, and found to be 0.3 to 1.6% higher than saturation moisture. This excess may be due to collection of liquid water droplets. In accordance with EPA guidelines, saturation moisture was used for determination of gas density, calculation of isokinetic sample rates, and standard duct gas flow rates.

#### **Inlet Flow Determination**

Inlet gas flow rate was measured by the pitot traverse conducted as part of the mercury test. Total boiler gas flow was determined as twice the measured flow, since one of two ducts was sampled.

#### **Outlet Flow Determination**

Outlet flow was measured by the pitot traverse conducted as part of the mercury test. This flow represents all of the scrubbed gas from the boiler.

#### **Comparative Flow Rate Calculations**

As a QA indicator, additional flow rate determinations were done. At the inlet, exhaust gas flow was calculated based on boiler fuel input and oxygen (F<sub>d</sub>) F factors. The plant CEMS stack flow rate is also presented.

## Alternate Methodology for O<sub>2</sub>/CO<sub>2</sub> Determination

As an alternate to conventional Orsat analysis, the following procedure was used for determination of O<sub>2</sub> and CO<sub>2</sub> content.

O<sub>2</sub> determination. O<sub>2</sub> was measured by a portable O<sub>2</sub> analyzer using an electrochemical cell. The gas sample for the portable analyzer was drawn through a tube inserted in the exit gas of the sample gas meter. This provides direct analysis of the gas sampled for the mercury test. Care was taken that the O<sub>2</sub> sample tube was not inserted so far that it interfered with the meter orifice pressure differential reading. Calibration procedures for the portable analyzer included:

1. At the beginning of the test day, the instrument was calibrated on ambient air. As-found readings were then taken using zero gas and an EPA Protocol 1 mid scale O<sub>2</sub> calibration gas (40 to 60% of the span used to collect readings). If these as found readings were within 2% of span, the data was acceptable. If the readings were outside of these ranges, the O<sub>2</sub> cell was replaced, the instrument was repaired, or an alternate instrument was used.
2. During testing, the calibration of the instrument was checked on ambient air every three or four sample points. If the as-read value on air had drifted more than 0.2% O<sub>2</sub> (0.8% of scale), the instrument was recalibrated.
3. At the end of the test day, the calibration error step described in Step 1 above was repeated.

CO<sub>2</sub> determination. CO<sub>2</sub> is used only for molecular weight determination. At the stack, CO<sub>2</sub> readings were taken from the plant CEMS.

At the outlet and inlet, the CO<sub>2</sub> was calculated by stoichiometric calculations, using standard F factors. It was not possible to calculate CO<sub>2</sub> at these locations by dilution calculations because there was no O<sub>2</sub> measurement available from the stack.

### ***Determination of Scrubber Efficiency and Stack Emissions***

A fraction of the flue gas at Coronado 1 is scrubbed in order to meet SO<sub>2</sub> emission limits, and the balance bypasses the scrubber. This section presents the calculation procedures used to determine scrubber efficiency and to estimate stack emissions.

#### Scrubber Efficiency Determination

Scrubber removal efficiency was calculated according to Equation 1 below:

$$(1) \quad E = 1 - C_{\text{out}}/C_{\text{in}}$$

Where,

E = Scrubber removal efficiency

C<sub>out</sub> = Measured concentration at scrubber outlet

$C_{in}$  = Measured concentration at scrubber inlet

It is important that the inlet and outlet values be corrected for air leakage to provide results on a consistent basis. For this program, the correction was achieved by calculating mercury concentration in units of  $lb/10^{12}$  Btu.

### Stack Emission Estimates

The stack gas concentration was calculated as shown in Equation 2:

$$(2) \quad C_{stack} = (BF \times C_{in}) + ((1-BF) \times C_{out})$$

Where,

$C_{stack}$  = Estimated concentration at stack

BF = fraction of gas bypassed, unitless. For Coronado 1, the bypass fraction was determined from the measured gas flow at the outlet and the CEMS stack flow reading. Calculations are shown in Appendix A.

Note that this procedure is different than the heat and mass balance approach discussed in the test plan. This procedure was used because it is a more direct approach. Calculations using the heat and mass balance approach are shown in Appendix A as a check, and resulted in an average bypass of 65% compared to 59% by the gas flow method.

The stack mass emissions were calculated as shown in Equation 3:

$$(3) \quad M_{stack} = (BF \times M_{in}) + M_{out}$$

Where M = mass flow, lb/hr, and subscripts denote location

## 4.2 Process Data

Process data was collected on computer logs set up by station personnel. Data collected included key boiler, scrubber, and ESP operating parameters, and all CEMS data.

Prior to and during each test, unit operation was assessed by station personnel to assure that operating conditions were within project target ranges.



# 5

## INTERNAL QA/QC ACTIVITIES

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### 5.1 QA/QC Problems

There were no sampling related QA/QC problems. All  $\text{KMnO}_4$  impingers were purple at the conclusion of each test.

### 5.2 QA Audits and Data Quality Objectives

QA audit samples were analyzed as specified in the Ontario Hydro Method and listed in Table 5-1. Data quality objectives are listed in Table 5-2. Table 5-3 presents audit results and compares data quality results with data quality objectives. Table 5-4 presents individual mercury fraction mass measurements, along with field blank results.

**Table 5-1. Audit Samples for Ontario Hydro Mercury Speciation**

Audit Sample	Acceptance Criteria and Frequency	Reference
Known reagent spike	Every 10 samples.	Ontario Hydro Section 13.4.1
Certified reference ash	One per program.	Ontario Hydro Section 13.4.1

All data quality objectives were met, with the following exceptions:

1. The sample volume for Run 1-Outlet was 2.7 standard cubic meters (scm), exceeding the method target range of 1.0-2.5 scm. This was discussed in Section 3.2, and is considered to have no impact on the results.
2. The range of results for outlet particulate mercury exceeded the target of +/- 35% from the mean. This is because the absolute values of outlet particulate mercury were low; the scatter on an absolute basis is less than  $0.05 \text{ lb}/10^{12} \text{ Btu}$ , and is considered small.
3. The range of results for outlet oxidized mercury exceeded the target of +/- 35% from the mean. This is because the absolute values of outlet oxidized mercury were low; the scatter on an absolute basis is less than  $0.04 \text{ lb}/10^{12} \text{ Btu}$ , and is considered small.

**Table 5-2. Data Quality Objectives for Flue Gas Mercury Analyses**

<i>Measure</i>	<i>Objective</i>	<i>Approach</i>
Accuracy	≤10% of sample value or ≤10x instrument detection limit	Reagent blanks-analyze one blank per batch of each reagent
Accuracy	Field blank ≤30% of sample value, or no greater than reagent blank; whichever is higher	Collect and analyze one field blank at inlet and one at outlet; criteria evaluated for each mercury species
Accuracy	±10% of nominal value	One known reagent spike every ten samples
Precision, lab analysis	≤10% RPD	All laboratory samples analyzed in duplicate, every 10th sample analyzed in triplicate
Completeness	≥95%	Failed or incomplete tests to be repeated, if possible and practical

### 5.3 Comparison Analyses

As an independent Quality Assurance check of the data, KCl and KMnO<sub>4</sub> samples were analyzed by the University of North Dakota Energy and Environmental Research Center (EERC). These results, shown in Table 5-5, indicate excellent agreement between the laboratories.

**Table 5-3. Results Evaluation and Verification Checklist**

<b>Measure</b>	<b>Objective</b>	<b>Result</b>
<i>Unit Operation</i>		
Unit operating conditions	No unusual conditions	Steady, normal operation
Air pollution control device operation	No unusual conditions	Steady, normal operation
<i>Sample Train Information</i>		
Trains leak checked before/after each test	<0.02 cfm	All tests passed
Pitot probes leak checked	Zero leakage	All tests passed
Probe, line, and filter temperature maintained	Minimum 120 C	All tests passed
Sample rate isokinetics	90-110%	98-106% at inlet 100-103% at outlet
Sample volume	1-2.5 std cubic meters	1.4-1.5 m <sup>3</sup> at inlet 2.7 m <sup>3</sup> on Run 1-Out, 1.6-1.7 m <sup>3</sup> for other outlet runs
Post-test color of permanganate impingers	Purple	All tests passed
<i>Results/lab QA</i>		
Flow rate for triplicate runs	All runs w/in 10% of mean	W/in 1% at inlet W/in 5% at outlet
Stack temperature for triplicate runs	All runs w/in 5% of mean	W/in 2% at inlet W/in 1% at outlet
Total mercury for triplicate runs	All runs w/in 35% of mean	W/in 9% at inlet W/in 35% at outlet
Particulate mercury	All runs w/in 35% of mean	Not detected at inlet One run ND at 60% below mean at outlet
Oxidized mercury	All runs w/in 35% of mean	W/in 16% at inlet One run 80% high and one run 38% low at outlet
Elemental mercury	All runs w/in 35% of mean	W/in 14% at inlet W/in 35% at outlet
Sample and blank spikes	W/in 10% of value	All tests passed
Field blanks	<30% of measured values	All tests passed

**Table 5-4. Coronado 1 Sample Fraction Mercury Measurements**

	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>	<b>Average</b>	<b>Field blank</b>	<b>Field blank/ sample, %</b>
<b>Inlet, µg/sample</b>						
Filter/probe wash (particulate Hg)	ND<0.070	ND<0.070	ND<0.070	ND<0.070	ND<0.070	ND
KCl fraction (oxidized Hg)	1.3	1.1	1.4	1.3	ND<0.10	ND
H <sub>2</sub> O <sub>2</sub> fraction (elemental Hg)	ND<0.25	ND<0.25	ND<0.25	ND<0.25	ND<0.050	ND
KMnO <sub>4</sub> fraction (elemental Hg)	2.9	2.5	2.4	2.6	ND<0.050	ND
<b>Outlet, µg/sample</b>						
Filter/probe wash (particulate Hg)	ND<0.070	0.12	0.16	0.11	ND<0.070	ND
KCl fraction (oxidized Hg)	0.10	ND<0.10	0.19	0.11	ND<0.10	ND
H <sub>2</sub> O <sub>2</sub> fraction (elemental Hg)	ND<0.25	ND<0.25	ND<0.25	ND<0.25	ND<0.050	ND
KMnO <sub>4</sub> fraction (elemental Hg)	8.2	2.6	4.4	5.1	ND<0.050	ND

**Table 5-5. Results of Independent QA Analyses of Coronado 1 Samples**

Run No.	1	2	3	Average
Date, 1999	18-Oct	19-Oct	19-Oct	
<i>Inlet laboratory mercury results, µg/sample</i>				
KCl fraction by Philip	1.3	1.1	1.4	1.3
KCl fraction by EERC	1.3	1.1	1.4	1.3
KMnO <sub>4</sub> fraction by Philip	2.9	2.5	2.4	2.6
KMnO <sub>4</sub> fraction by EERC	3.6	2.5	2.7	2.9
<i>Stack laboratory mercury results, µg/sample</i>				
KCl fraction by Philip	0.10	ND<0.10	0.19	0.10
KCl fraction by EERC	0.17	0.17	0.27	0.20
KMnO <sub>4</sub> fraction by Philip	8.2	2.6	4.4	5.1
KMnO <sub>4</sub> fraction by EERC	7.8	2.5	3.9	4.7
<i>Total mercury mass rates</i>				
Inlet lb/hr by Philip	0.0087	0.0077	0.0086	0.0083
Inlet lb/hr by EERC	0.0102	0.0078	0.0092	0.0091
Stack lb/hr by Philip	0.0090	0.0066	0.0086	0.0081
Stack lb/hr by EERC	0.0097	0.0066	0.0087	0.0083

