

The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-fired Electric Power Generation

A GUIDANCE DOCUMENT

**Developed by the Canadian Council of Ministers of the Environment
Mercury Canada-wide Standards Development Committee
to guide data monitoring and reporting
under its 2002 - 2005 Mercury Electric Power Generation Workplan**

January, 2003

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The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-fired Electric Power Generation

1.0 Introduction

In order to permit a sound and scientific assessment of the mercury emissions from coal-fired electricity-generation in Canada, a national program of testing, analysis and verification called "The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-fired Electric Power Generation" will be conducted under the auspices of the *Canadian Council of Ministers of the Environment* (CCME). The main objective of the UDCP is to gather nationally consistent, comparable and compatible information upon which to base future standards development. More specifically, the program is designed to:

- compile quality assured and quality controlled inventories for mercury in coals, residues and flue gases - the assessment of mercury emissions should include analysis of all additives, for example limestone injection, and heavy fuel oil (HFO) where supplementary firing is used;
- develop reasonable correlations between mercury in feed coals, residues and flue gases for mass balance purposes; and
- use the above information to reasonably predict the mercury emission reductions that may be expected from various control technologies.

The testing program, to be conducted according to jurisdiction specific agreements between provincial governments and their utilities, will consist of the three major initiatives outlined below. It is anticipated that these initiatives will generate the data required to determine what mercury emissions reductions can reasonably be expected from coal-fired electric power plants and over what time frames. It is recognized, however, that operational realities and safety considerations may preclude the use of some of the recommended sampling procedures. In such cases, individual facilities are asked to propose alternatives in their individual test plans (described below) that will yield equivalent information.

It is anticipated that the information collected for the UDCP will be compiled into a national report and be made broadly available to the Canadian public. Details of how and when this is to be accomplished will be the subject of discussion between jurisdictions and the CCME. Inquiries regarding the UDCP should be directed to the UDCP Project Manager:

Mike Gilbertson, CCME Secretariat
123 Main Street, Suite 360
Winnipeg, MB., R3C 1A3

Phone: (204) 948-2032
FAX: (204) 948-2125
e-mail: mgilbertson@ccme.ca

2.0 Part I: General Plant Information

2.1 Background

Coal-fired thermal electric power plants in Canada burn a range of coal types. These plants vary in generation capacity, age, process equipment and pollution control technology. Such differences can have significant impacts on the quantities and species of mercury emitted to the atmosphere. Compiling facility specific configuration information, therefore, is an essential component of the UDCP that will aid in the evaluation of factors influencing mercury emissions. In addition, this information will be used in the evaluation of test plans (described below) to ensure fuels, residues and emissions data are nationally compatible, consistent and comparable.

2.2 Program Description

Individual utilities will be asked to provide the detailed identification, engineering and performance information specified in **Appendix A**.

2.3 Deliverables

Utilities will be asked to submit the information outlined in **Appendix A** to their jurisdictional representative. For the duration of the UDCP, utilities will be asked to update this information whenever modifications or changes are made which are likely to significantly impact mercury emissions or speciation (for example, configuration changes, modifications to boilers, burner equipment, and/or air pollution control devices).

3.0 Part II: Coal, Ash and Residues Sampling Program

3.1 Background

Weekly coal, ash and residues sampling/analyses programs from every electricity generating unit in Canada are designed to supply needed information on the mercury content of coal and residues found in Canadian coal-fired power plants. The collection of composite coal, ash and residue samples over the term of the UDCP will generate an essential portion of the data needed to make mass-balance estimates and predict the fate of mercury at each facility. In addition, analysis of all additives, for example limestone injection, and heavy fuel oil (HFO) where supplementary firing is used, will be important for mass-balances.

3.2 Program Description

Prior to commencing the sampling program, each utility will be asked to submit a site specific test plan outlining a coal (and supplementary fuels if applicable), ash and residue sampling program in accordance with the methodologies outlined below and as described in the guidance document for utility test plans (**Appendix B**). Again, it is recognized that operational realities may preclude the use of some of the recommended procedures. In such cases, individual facilities are asked to propose alternatives in their individual test plans that will yield equivalent information.

In order to minimize interlaboratory variances, utilities will be asked to utilize Natural Resources Canada's Canada Centre for Mineral and Energy Technology (CANMET) for their respective analyses while a 6-month, round-robin testing program is being conducted. Details regarding the Laboratory Verification and Round-Robin Testing Program can be found in **Appendix C**.

When the round-robin is complete, utilities may select their laboratories of choice from the approved list. It is anticipated that Part II will be carried out over a period of about 24 months.

3.2.1 Sampling and Analysis

Weekly composite coal samples are to be collected as-fired and must be reported with their corresponding feed rate. As-fired refers to the condition of the coal immediately upon entering the boiler. Any proposed deviation from this may require additional testing (i.e. on rejects) for the purposes of mass balance determination.

Fly ash composite samples must be obtained from either the hoppers of at least the first two fields of an electrostatic precipitator, each of the hoppers of a fabric filter, the ash silo, or the station prior to conveyance to the ash lagoon (i.e. prior to sluicing). Samples of bottom ash will be obtained once per week for the first three weeks of the UDCP. The requirement for bottom ash sampling will be reviewed at the end of this period with the objective of eliminating this requirement if results warrant.

At a minimum, all weekly composite samples must be comprised of samples taken over three separate days of the week in question. As far as possible, ash samples should correspond to the coal that is represented by the composite coal sample. It is recommended that split samples be obtained and stored in the event that future analyses are desired to re-evaluate program results.

Utilities will be asked to analyse weekly composite samples of coal, ash and any other significant residues for mercury, chlorine, inherent moisture (combined with air dry values to calculate total moisture) and sulphur. Coal samples will also be analysed for heat value while ash will be tested for unburned carbon. Proximate and ultimate analyses will be obtained for coal samples. Tests for moisture, sulphur and unburned carbon may be done using any standard analytical method and service provider the utility chooses. Mercury and chlorine contents, however, must be determined using laboratories whose performance has been assessed and approved during the UDCP Laboratory Round-robin Proficiency Testing Program. While not a mandatory part of the UDCP, it is recommended that utilities also analyze samples for other halides such as bromine and fluorine.

3.2.2 Sample Collection, Preparation and Analysis Methodologies

The methods below (Table 1) are recommended for collecting, preparing and analyzing samples for Part II of the UDCP.

Table 1: Recommended methods for sample preparation, collection and analysis

Activity	Sample	Procedures
Sample Collection	Coal	<u>ASTM D6315</u> - Standard Practice for Manual Sampling of Coal from Tops of Barges
		<u>ASTM D6610</u> - Standard Practice for Manual Sampling Coal from Surfaces of a Stockpile
		<u>ASTM D4915</u> - Standard Guide for Manual Sampling of Coal from Tops of Railroad Cars
		<u>ASTM D4916</u> - Standard Practice for Mechanical Auger Sampling
		<u>ASTM D6609</u> - Standard Guide for Part-Stream Sampling of Coal
		<u>ASTM D2234</u> - Standard Practice for Collection of a Gross Sample of Coal
		<u>ASTM D4702</u> - Standard Guide for Inspecting Crosscut, Sweep-Arm, and Auger Mechanical Coal-Sampling Systems for Conformance with Current

		ASTM Standards
		<u>ASTM D6518</u> - Standard Practice for Bias Testing a Mechanical Coal Sampling System
		<u>ASTM D4182</u> - Standard Practice for Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke
	Solid Residues	Standard practices for the sampling and preparation of residues have not been established.
Sample Preparation	Coal	<u>ASTM D2013</u> - Standard Practice of Preparing Coal Samples for Analysis
	Solid Residues	Standard practices for the preparation of residues have not been established. The recommended size reduction is 150-um (No. 100) U.S.A. standard sieve, prepared in an agate mortar and pestle.
Mercury Analysis	Coal	<u>ASTM D3684</u> - Standard Test Method for Total Mercury in Coal by the Oxygen Bomb Combustion/Atomic Absorption Method
		<u>ASTM D6414</u> - Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Acid Extraction or Wet Oxidation/Cold Vapor Atomic Adsorption
		<u>ASTM D6722</u> - Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Direct Combustion Analysis
		<u>EPA Method 7473</u> - Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry
	Solid Residues	<u>ASTM D6414</u> - Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Acid Extraction or Wet Oxidation/Cold Vapor Atomic Adsorption
		<u>ASTM D6722 (Draft Method)</u> - Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Direct Combustion Analysis
		<u>EPA Method 7473</u> - Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry
	Other Residues	<u>EPA 7471A</u> (Acid Extraction CVAA)
		<u>EPA 1631</u> (Acid Extraction CVAFS)
		Microwave Digestion CVAA
Chlorine Analysis	Coal	<u>ASTM D4208</u> - Bomb Combustion Ion Selective Electrode (ISE)
		Pyrohydrolysis / Ion Chromatography
		<u>ASTM D2361</u> - Standard Test Method for Chlorine in Coal
		<u>ASTM D6721</u> - Standard Test Method for Determination of Chlorine in Coal by Oxidative Hydrolysis Microcoulometry
		Instrumental Neutron Activation
	Solid Residues	Pyrohydrolysis / Ion Chromatography

Summaries of the ASTM methods outlined in Table 1 can be viewed at <http://www.astm.org/cgi-bin/SoftCart.exe/DATABASE.CART/MARKETINGCODES/PE2.htm?U+mystore+mnos7563> or by writing to:

ASTM
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959, USA
Phone: (610) 832-9585, Fax: (610) 832-9555

EPA method summaries can be obtained from <http://www.epa.gov/epahome/index/sources.htm>

3.3 Deliverables

Utility test plans should be submitted to their respective jurisdictional representatives. Feedback for utilities will be provided within 30 days. Thirty days will be allocated for test plan revisions (if necessary) prior to the commencement of the sampling program.

The sampling program is scheduled to run approximately 24 months. Utilities should provide data quarterly to the jurisdictional representative in hardcopy or spreadsheet format using reporting formats described in **Appendix D**.

4.0 Part III: Air Emissions Monitoring Program

4.1 Background

The UDCP will include the development of quality-assured speciated mercury emissions data for every electricity-generating unit in Canada. It is anticipated that the emissions data will be sufficient to determine facility-specific mass-balance relationships, predict the fate of the mercury in each facility, convert the weekly data collected in Part II to estimates of annual mercury emissions and predict the mercury emission reductions that may be expected from various control technologies.

4.2 Program Description

Each utility will submit a site-specific test plan outlining a speciated mercury emissions sampling and analysis program for its generating units in accordance with the methodologies described below and in the guidance document for the preparation of site-specific test plans (**Appendix E**). Again, it is recognized that operational realities may preclude the use of some of the recommended procedures. In such cases, individual facilities are asked to propose alternatives in their individual test plans that will yield equivalent information. The alternatives are to include the proposed use of proxy results or configurations in accordance with the requirements described in Section 5.0.

Ideally, Part III of the UDCP will consist of concurrent flue gas sampling upstream of the first emission control device (if applicable) and downstream of the last emission control device at each electricity generating unit to determine concentrations of elemental, reactive and particulate forms of mercury. At a minimum, facilities will be expected to obtain downstream measurements for each unit. Units should be operating under full load during the test period. The testing is to consist of three runs at each sampling location and is to be conducted in accordance with the approved Ontario Hydro Method (**Appendix F**). For each stack test, three as-fired coal samples will be collected and analyzed. Also, for each stack test, three combustion residue samples will be collected and analyzed. Coal and ash samples should be analyzed for the characteristics outlined in Part II using the procedures prescribed therein. Based on residence times, the emission testing and ash sampling should correspond, as closely as possible, to the coal that was sampled and was being burned during the test period. It is recommended that split samples be obtained and stored in the event that future analyses are desired to re-evaluate program results.

If coal samples are taken before coal rejects are removed, the rejects must also be analyzed for mercury content. It is imperative that the feed rates of the sampled coal (and rejects, if applicable) are known. Fly ash samples must be taken, at a minimum, from the hoppers of the first two fields of an electrostatic precipitator or from each of the hoppers of a fabric filter.

4.3 Deliverables

Utility test plans should be submitted to their respective jurisdictional representatives 60 days prior to testing. Feedback for utilities will be provided within 30 days of the receipt of the plans. The UDCP Part III is estimated to require 24 months to complete. Utilities will be expected to submit the results of stack testing to their respective jurisdictional representatives within 90 days of the completion of the test.

The results of flue gas, coal and residue sampling and analysis should be submitted in a final report following the format for test reports outlined in **Appendix E**. Reports will include an inventory of mercury in coal, combustion residues and speciated mercury air emissions. All stack testing is to be completed by June, 2004.

5.0 Proxies and Historical Data

5.1 Background

While some utilities will conduct stack tests for each electricity generating unit for the UDCP, some may have already completed tests that meet UDCP requirements. Preliminary short-term analysis of this existing data will be undertaken to evaluate its suitability for inclusion in the UDCP database.

In addition, it is recognized that some utilities may have generating units with configurations and feed coal sources that are equivalent for the purposes of mercury emissions and mass balance determinations. The equivalency, or “proxy” status, of these units will be assessed based on similarity of plant configurations, historical or current data for feed coals, residues and air emissions.

5.2 Program Description

Stack analyses conducted after 1996 and meeting the requirements outlined in Section 4.2 above may be used to partially fulfill the requirements of Part III of the UDCP. Re-testing using the approved Ontario Hydro Method (**Appendix F**) may be required to assure jurisdictions that no significant changes in emissions have occurred since the initial testing. Expert advice will be obtained on the suitability of existing data and the possible use of one plant configuration as a proxy for another.

5.3 Deliverables

Utilities should notify jurisdictions and the CCME (through the UDCP Project Manager) of the potential use of existing mercury emissions data or proxy units with the submission of their test plans. In the case of existing data, notification should be accompanied by the data in question, a detailed description of collection, analysis and QA/QC methods, and an indication of any changes to the unit capacity or configuration since the date of data collection. In the case of proxy determination, a detailed justification supporting the ‘similarity’ of unit configurations must be included. Any significant changes in the plant configuration during the UDCP will require a reassessment of the applicability of existing data and/or proxy status.

6.0 UDCP Summary

Tables 2, 3 and 4 below provide a synopsis of utility information requirements for UDCP Parts I, II and III. The testing programs may be modified or extended based on identified data gaps.

Table 2: Utility information requirement for UDCP Part I - General Plant Information

Information Requirement	Reporting to
Facility identification, performance and engineering information outlined in Appendix A	Jurisdictional representatives
Information on potential use of existing mercury emissions data or proxy units identified in section 5 above	Jurisdictional representatives, with updates if plant modifications are made during the course of the UDCP

Table 3: Utility information requirement for UDCP Part II - Coal, Ash and Residue Sampling Program

Source	Sampling Frequency	Sampling Locations	Sampling Methods	Analyses to be Conducted	Analysis Methods	Reporting
As-fired Coal	Weekly composite samples, comprised of samples taken on at least 3 separate days	Milled coal sent to boiler	Reference Section 3.2.2	mercury, chlorine, inherent moisture, total moisture, sulphur and heat value	Reference Section 3.2.2	Quarterly
Fly Ash	Weekly composite samples, comprised of samples taken on at least 3 separate days	The hoppers of the first two fields of an electrostatic precipitator, each of the hoppers of a fabric filter, the ash silo, or prior to conveyance to the ash lagoon (i.e. prior to sluicing)	Reference Section 3.2.2	mercury, chlorine, inherent moisture, total moisture, sulphur, and unburned carbon	Reference Section 3.2.2	Quarterly
Bottom Ash	Once per week for the first 3 weeks of sampling - further requirements will be reviewed	Before quenching		mercury	Reference Section 3.2.2	First Quarterly report

Table 4: Utility information requirement for UDCP Part III - Air Emissions Testing Program

Source	Number of Samples	Sampling Locations	Sampling Methods	Analyses to be Conducted	Analysis Methods	Reporting
As-fired Coal	3 samples for each stack test	Milled coal sent to boiler	Reference Section 3.2.2	mercury, chlorine, inherent moisture, total moisture, sulphur and heat value	Reference Section 3.2.2	Test report within 90 days of the completion of the test, and prior to November 1, 2004
Fly Ash	3 samples for each stack test	The hoppers of the first two fields of an electrostatic precipitator or each of the hoppers of a fabric filter (at a minimum)	Reference Section 3.2.2	mercury, chlorine, inherent moisture, total moisture, sulphur, and unburned carbon	Reference Section 3.2.2	Test report within 90 days of the completion of the test, and prior to November 1, 2004
Bottom Ash	3 samples for each stack test	Before quenching		mercury	Reference Section 3.2.2	Test report within 90 days of the completion of the test, and prior to November 1, 2004
Flue Gas	Three runs at each sampling location	Concurrent flue gas sampling upstream (if applicable) of the first emission control device and downstream of the last emission control device	Approved Ontario Hydro Method (Appendix F)	elemental, oxidized and particulate mercury. Where existing speciated data are acceptable, total mercury	Approved Ontario Hydro Method (Appendix F)	Test report within 90 days of the completion of the test, and prior to November 1, 2004

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APPENDIX A: General Plant Information

Contact Information

1. Facility name:

2. Street address of facility:

3. Mailing address of facility (if different from above):

4. Facility Latitude and Longitude:

Latitude _____ Longitude _____

5. Legal name of facility owner as incorporated under law:

6. Address of legal owner (Head Office):

7. Legal name of operator (if different from above):

8. Address of legal operator (if different from above):

9. Name and title of person(s) able to answer any technical questions regarding information provided in this form (please also include their phone numbers and e-mail addresses):

Plant History

10. Date of initial plant construction:

11. Using **table A-1**, provide a list of units, the dates of construction of each unit and projected dates of retirement of each unit at this facility.

12. In **table A-2**, list major modifications since initial start-up (i.e. changes resulting in a \pm 5% change in unit output).

Fuels and Residues

13. In **table A-3**, indicate the type of coal that is primarily utilized at each unit, its source and the amount burned during the 365 days prior to the completion of this form. Please indicate if coal mixing is occurring, or if different coals are fired sequentially.

14. In **table A-4**, provide proximate and ultimate analyses, heating value and mercury (Hg) content of the coal type identified in question 13 that is primarily utilized at each unit. (Proximate analysis is defined as the determination of moisture, volatile matter, and ash, and the calculation of fixed carbon by difference. Ultimate analysis from a dried sample is defined as the determination of carbon, hydrogen, sulphur, nitrogen and ash, and the calculation of oxygen by difference.) Also indicate the coal type for which the unit performance is optimized if different from coal currently combusted.

15. Using **table A-5**, describe fly ash and bottom ash disposal methods and provide the amount of ash generated at each unit for this year (over the 365 days prior to the completion of this form).

Power Generation

16. In **table A-6**, describe the type of boiler operating at each unit.
17. In **table A-7**, provide the current capacity and the net electricity generated annually for the period 1995-2002.

Process and Control Equipment Description

18. Using the format shown in figures 1 through 4, provide a diagram which shows the general equipment configuration at each unit at this facility.
19. Using **table A-8**, describe the air pollution control systems at each unit. List standard operating parameters and ranges. Indicate the maximum efficiency of these units and the typical operating efficiency.
20. In **table A-9**, indicate if there is physical space available to add additional air pollution control systems for the units at this facility.
21. In **table A-10**, Describe any additional operations which may affect pollution emissions (e.g., coal cleaning by supplier or on-site, reject separation)

Emissions

22. Using **table A-11**, indicate annual stack emissions of SO₂, NO_x, particulate matter (PM) and mercury (Hg) for each unit at this facility for the period from 1995 to 2002.
23. Please indicate the facility-specific identification number used for reporting to the National Pollutant Release Inventory (NPRI).

NPRI ID: _____

Table A-1: Unit Life span

Facility Name _____

Unit ID	Date of Construction	Projected Date of Retirement

Table A-2: Major Plant Modifications

Facility Name _____

Unit ID	Nature of Modification	Year of modification	Change in rated output (Mwe)

Table A-3: Primary coal type burned at each unit, source and annual amount burned (complete table 4 for each unit at this facility)

Facility Name _____

Unit ID _____

Coal Type	Coal Source (e.g., Country, Province, seam, mine mouth or shipment, etc.)	Tonnes of Coal Burned This Year (365 days prior to completion of this form)
<input type="checkbox"/> lignite <input type="checkbox"/> waste lignite		
<input type="checkbox"/> sub bituminous <input type="checkbox"/> waste sub bituminous		
<input type="checkbox"/> bituminous <input type="checkbox"/> waste bituminous (gob)		
<input type="checkbox"/> anthracite <input type="checkbox"/> waste anthracite (culm)		
<input type="checkbox"/> blend lignite % _____ waste lignite % _____ sub bituminous % _____ waste sub bituminous % _____ bituminous % _____ waste bituminous (gob) % _____ anthracite % _____ waste anthracite (culm) % _____		
<input type="checkbox"/> other - specify _____ _____ _____ _____ _____		

Note: if different coals are used, please describe if they are mixed or fired sequentially: _____

Table A-4: Characteristics of the primary coal type burned at each unit, and coal type for which the unit performance is optimized (complete table 5 for each unit at this facility)

Facility Name _____

Unit ID _____

Proximate Analysis of Primary Coal Type Burned	
Component	Weight (%)
Moisture	
Volatile Matter	
Fixed Carbon	
Ash	
Total	/100
Ultimate Analysis of Primary Coal Type Burned	
Component	Dry Weight (%)
Moisture	
Carbon	
Hydrogen	
Sulphur	
Nitrogen	
Oxygen	
Ash	
Total	/100
Higher Heating Value (HHV) of Primary Coal Type Burned (MJ/kg)	Hg Content (PPM) of Primary Coal Type Burned
Is the performance of this unit optimized for the above coal type ? (yes or no)	If the performance at this unit is not optimized for the above coal type, indicate the optimal coal type

Table A-5: Ash disposal methods and amount generated this year (365 days prior to completion of this form) - complete table 6 for each unit at this facility

Facility Name _____

Unit ID _____

Ash	Total Tonnes Generated during the 365 days prior to the completion of this form	Disposal Method and Amount (tonnes)		Amount Sent for Gypsum or Other Recovery Process	
		Wet	Dry	Process	Tonnes
Fly Ash					
Bottom Ash					

Table A-6: Boiler type at each unit

Facility Name _____

Unit ID	Boiler ID	Type ¹ of Boiler

¹ Examples: tangential-fired; cyclone; wall-fired; fluidized bed combustion (FBC)

Table A-7: Unit capacity and net electrical generation

Facility Name _____

Unit ID	Unit Capacity (MWe)	1995 (GWh)	1996 (GWh)	1997 (GWh)	1998 (GWh)	1999 (GWh)	2000 (GWh)	2001 (GWh)	2002 (GWh)

Table A-8: Pollution control devices present at each unit (complete table 9 for each unit at this facility)

Facility Name _____

Unit ID _____

Type of NO _x Control ¹	Standard Operating Parameter and Range	Standard Operating Parameter and Range	Maximum Efficiency	Typical Operating Efficiency
Type of SO ₂ Control ²	Standard Operating Parameter and Range	Standard Operating Parameter and Range	Maximum Efficiency	Typical Operating Efficiency
Type of Particulate Matter Control ³	Standard Operating Parameter and Range	Standard Operating Parameter and Range	Maximum Efficiency	Typical Operating Efficiency

¹ Examples: low-NO_x burners; selective catalytic reduction (SCR); selective non-catalytic reduction (SNCR)

² Examples: wet flue gas desulphurization (FGD; any type); dry scrubbing (any type); compliance (low sulphur) coal; FBC (any type)

³ Examples: fabric filter; cold-side electrostatic precipitator (ESP); hot-side ESP; cyclone

Table A-9: Space availability for additional air pollution control systems

Facility Name _____

Unit ID	Describe availability of physical space to add additional air pollution control systems

Table A-10: Additional operations which may affect pollution emissions

Facility Name _____

Operations

Table A-11: Annual stack pollutant emissions from each unit (complete table 12 for each unit at this facility)

Facility Name _____

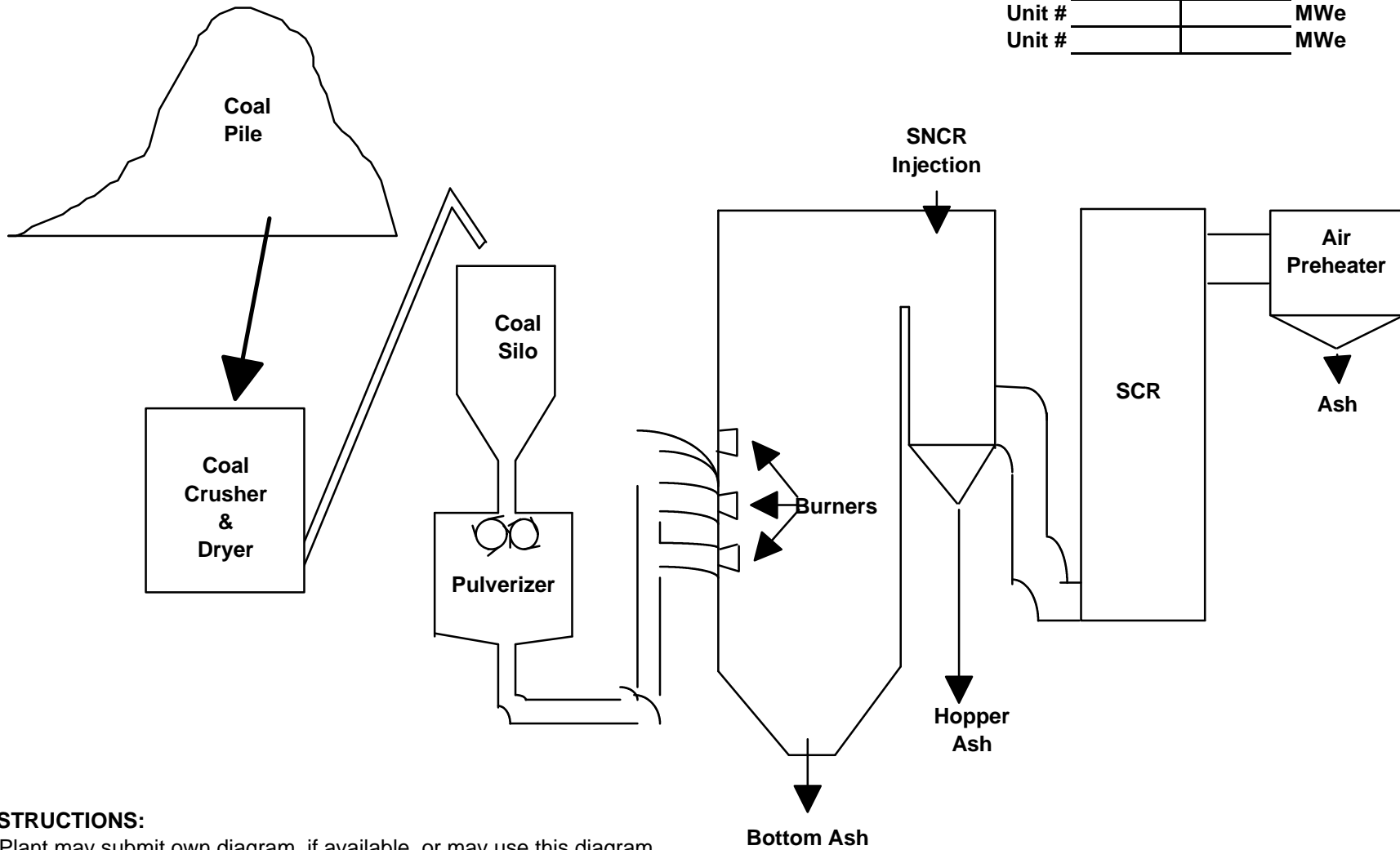
Unit ID _____

Pollutant	1995 (tonnes)	1996 (tonnes)	1997 (tonnes)	1998 (tonnes)	1999 (tonnes)	2000 (tonnes)	2001 (tonnes)	2002 (tonnes)
SO₂								
NO_x								
PM								
Hg (Total)								

Figure 1 Plant Configuration 1

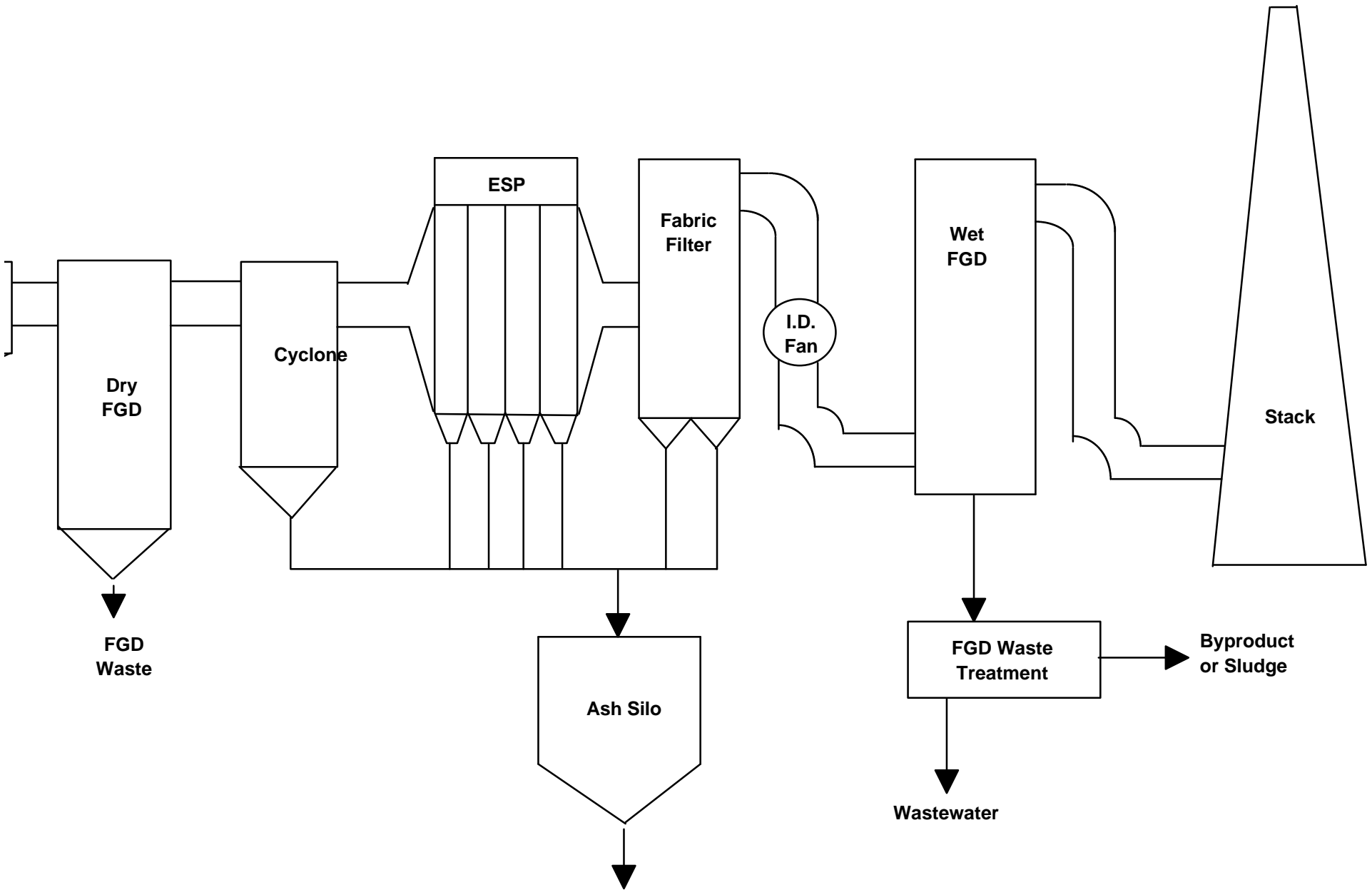
Facility Name _____

Unit #		MWe
Unit #		MWe
Unit #		MWe
Unit #		MWe

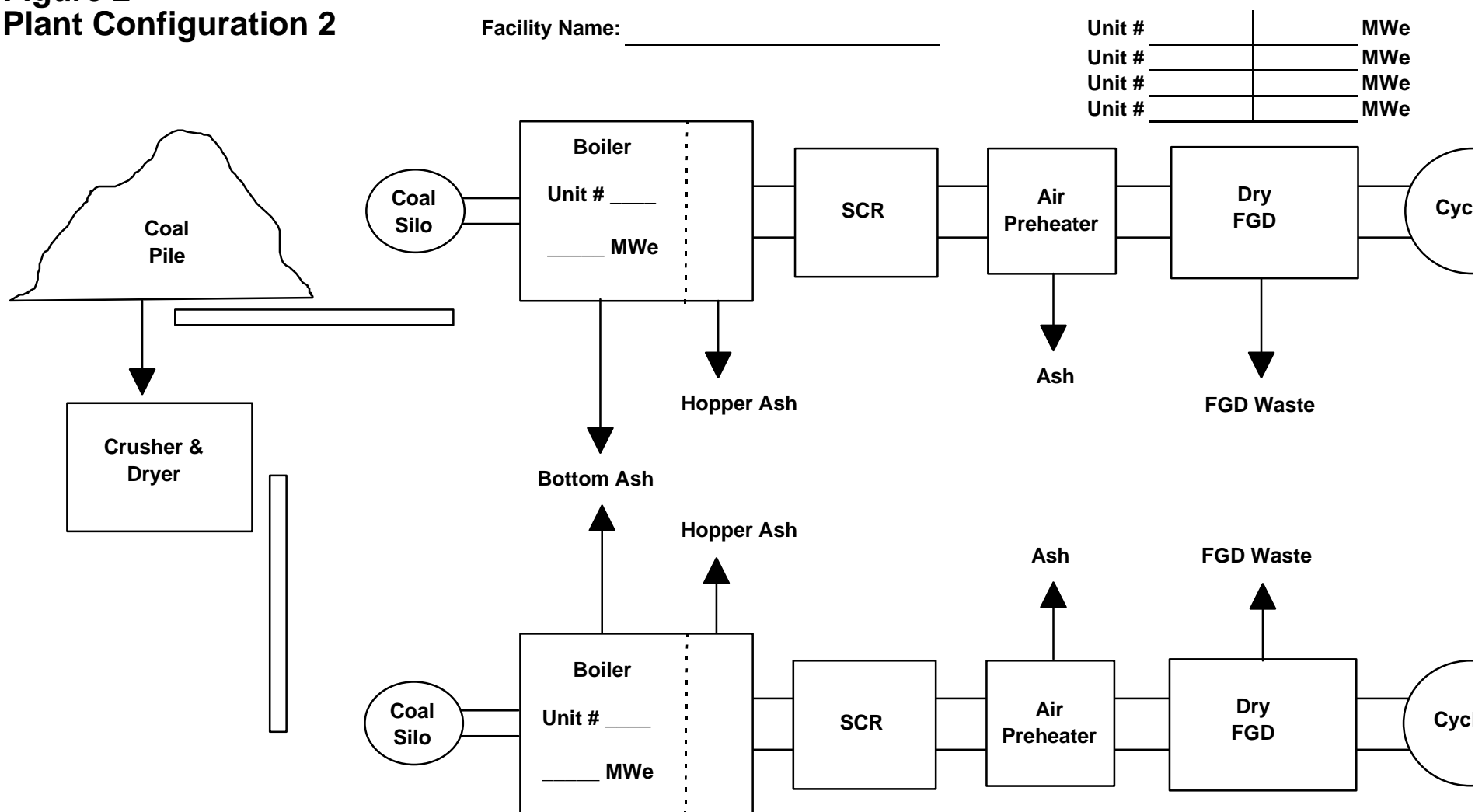


INSTRUCTIONS:

1. Plant may submit own diagram, if available, or may use this diagram.
2. Plant may submit more than one diagram.
3. Components not installed at plant should be deleted from this diagram.
4. Components installed at plant but not shown here should be added to this diagram.
5. Indicate on this diagram where sampling ports exist or are required for solid, liquid and air streams.
6. Indicate on this diagram the lengths and cross-sectional dimensions of ducting at existing or required sampling locations.

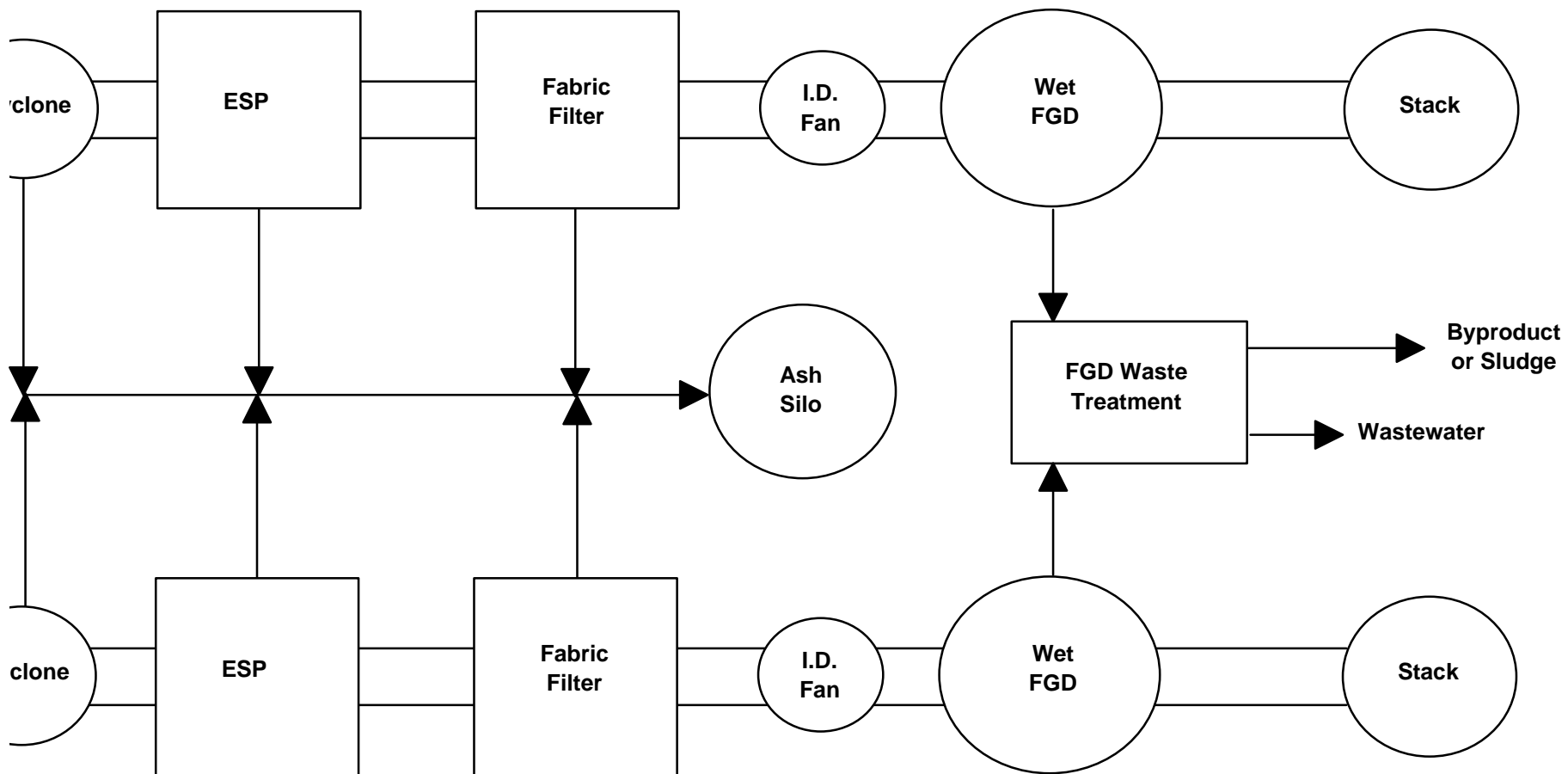


**Figure 2
Plant Configuration 2**

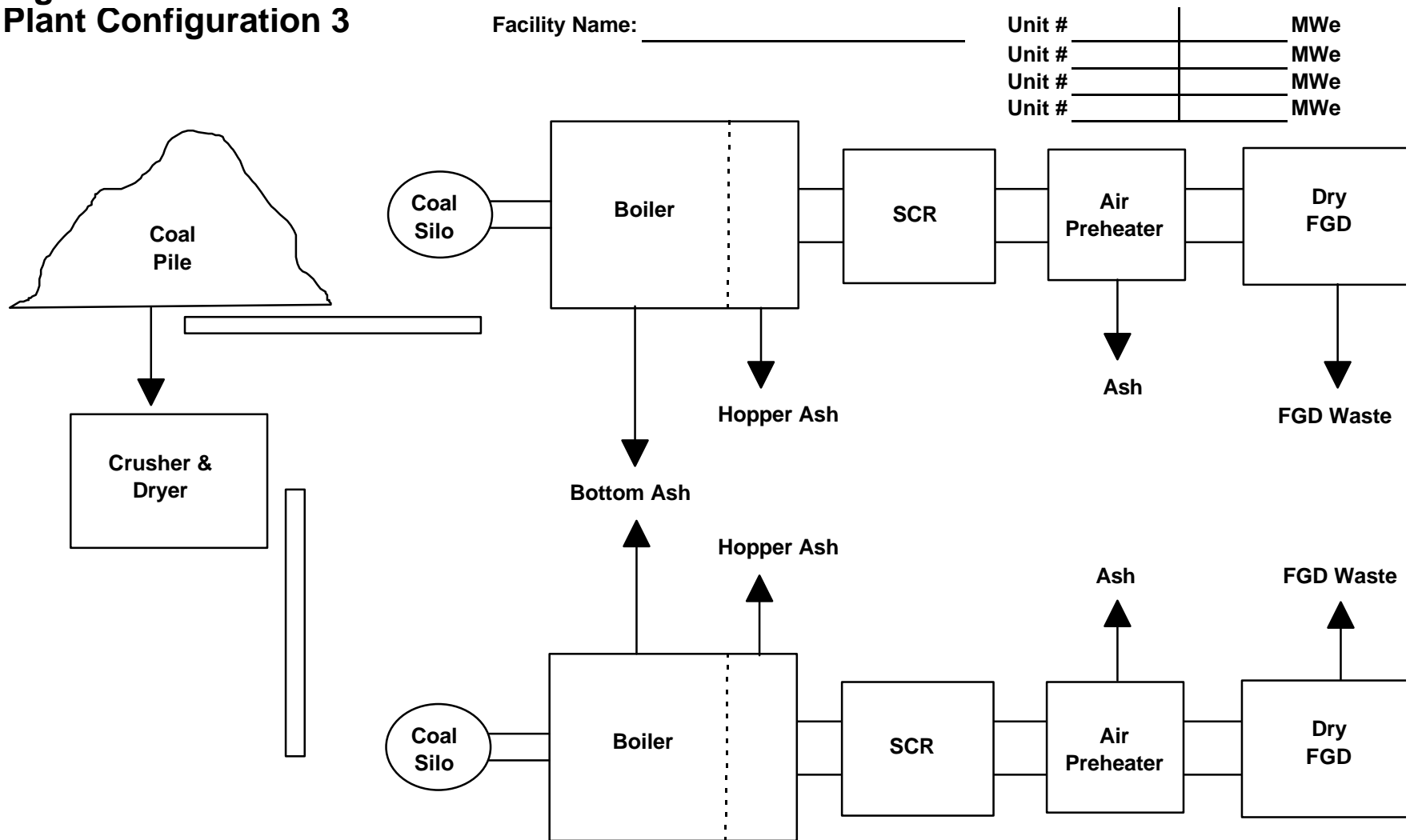


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2. Plant may submit more than one diagram.
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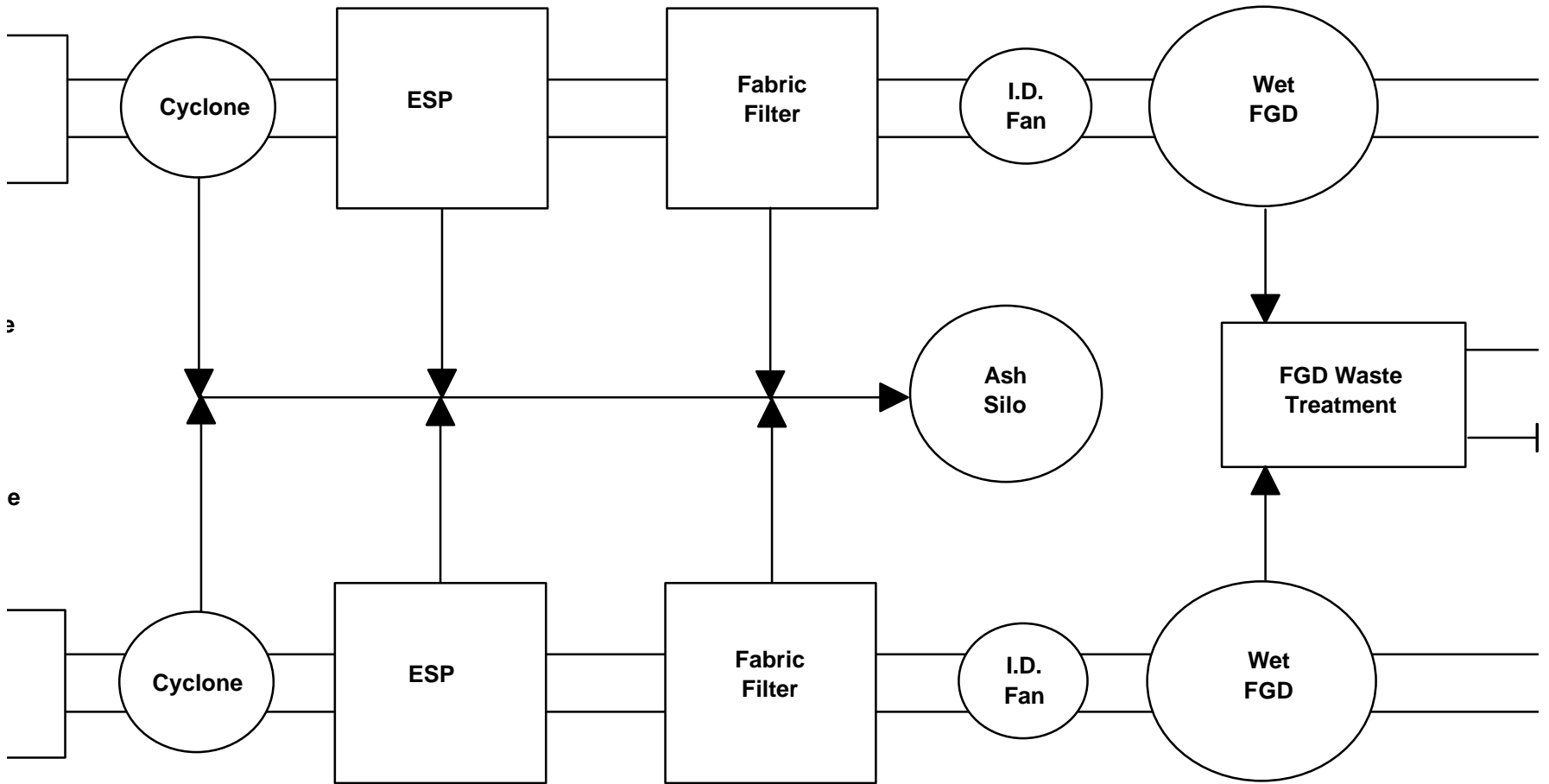


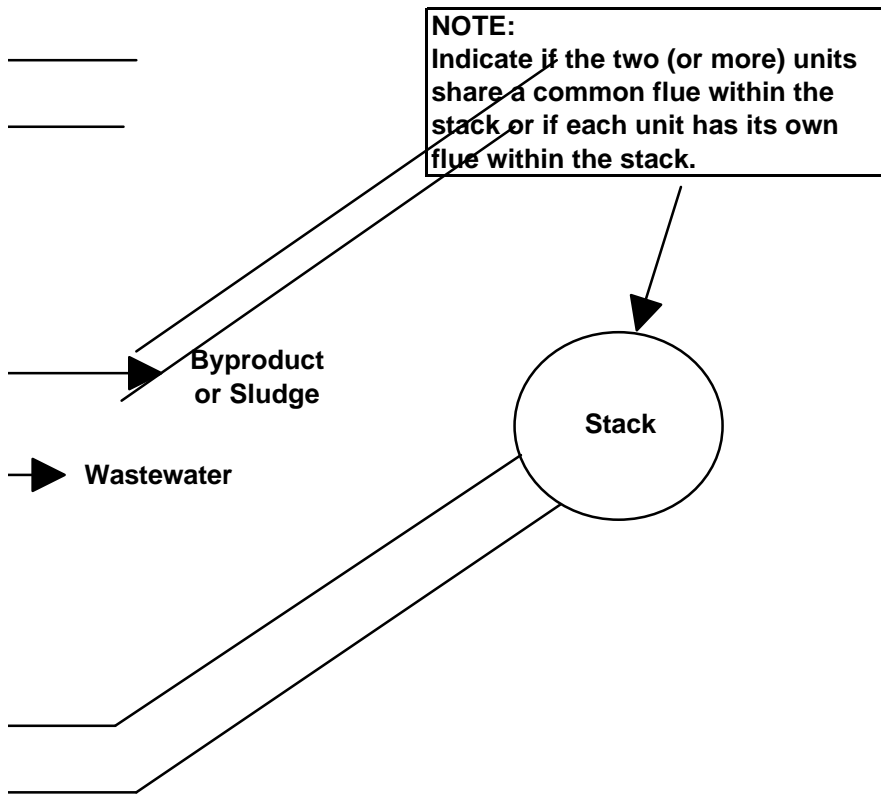
**Figure 3
Plant Configuration 3**



INSTRUCTIONS:

1. Plant may submit own diagram, if available, or may use this diagram.
2. Plant may submit more than one diagram.
3. Components not installed at plant should be deleted from this diagram.
4. Components installed at plant but not shown here should be added to this diagram.
5. Indicate on this diagram where sampling ports exist or are required for solid, liquid and air streams.
6. Indicate on this diagram the lengths and cross-sectional dimensions of ducting at existing or required sampling locations.

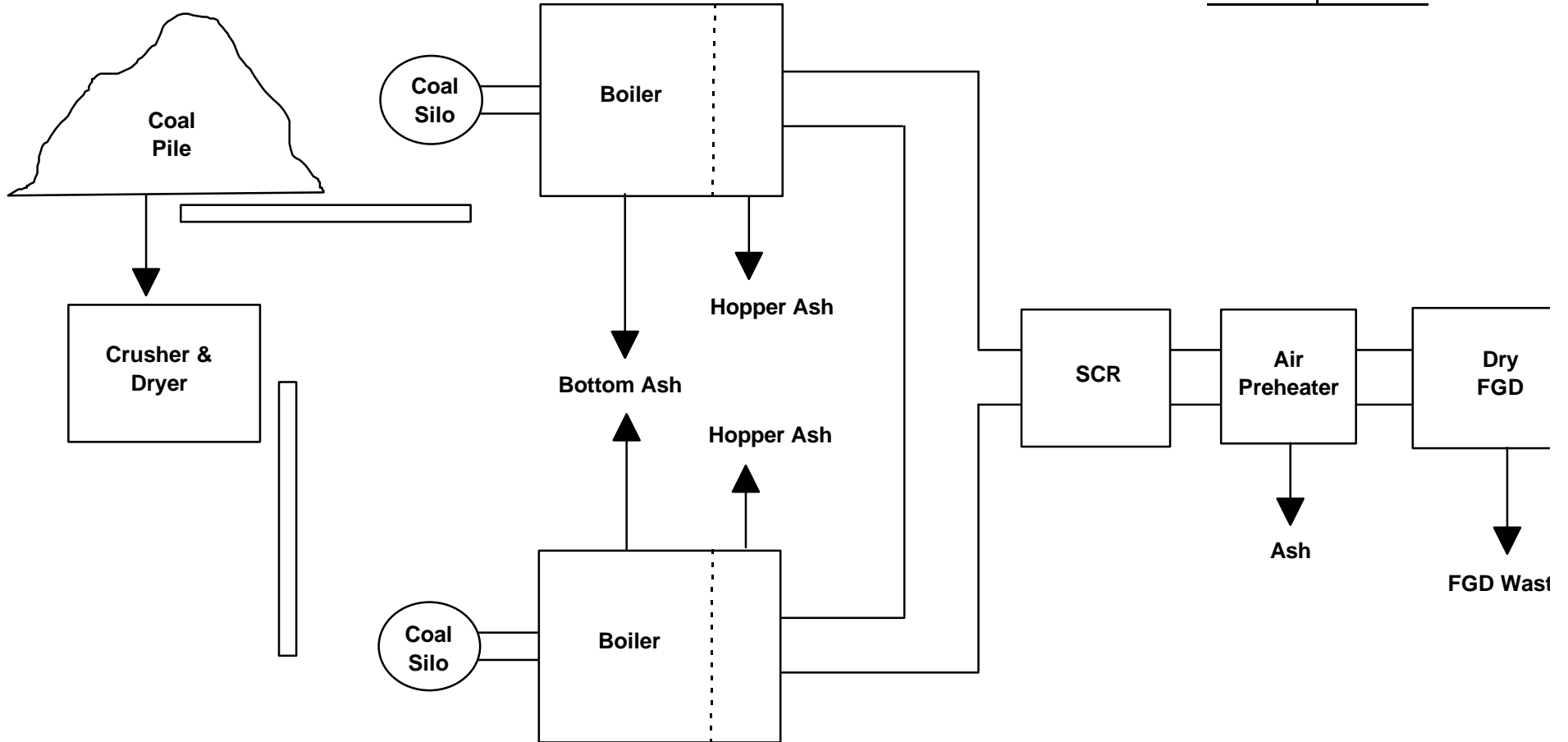




**Figure 4
Plant Configuration 4**

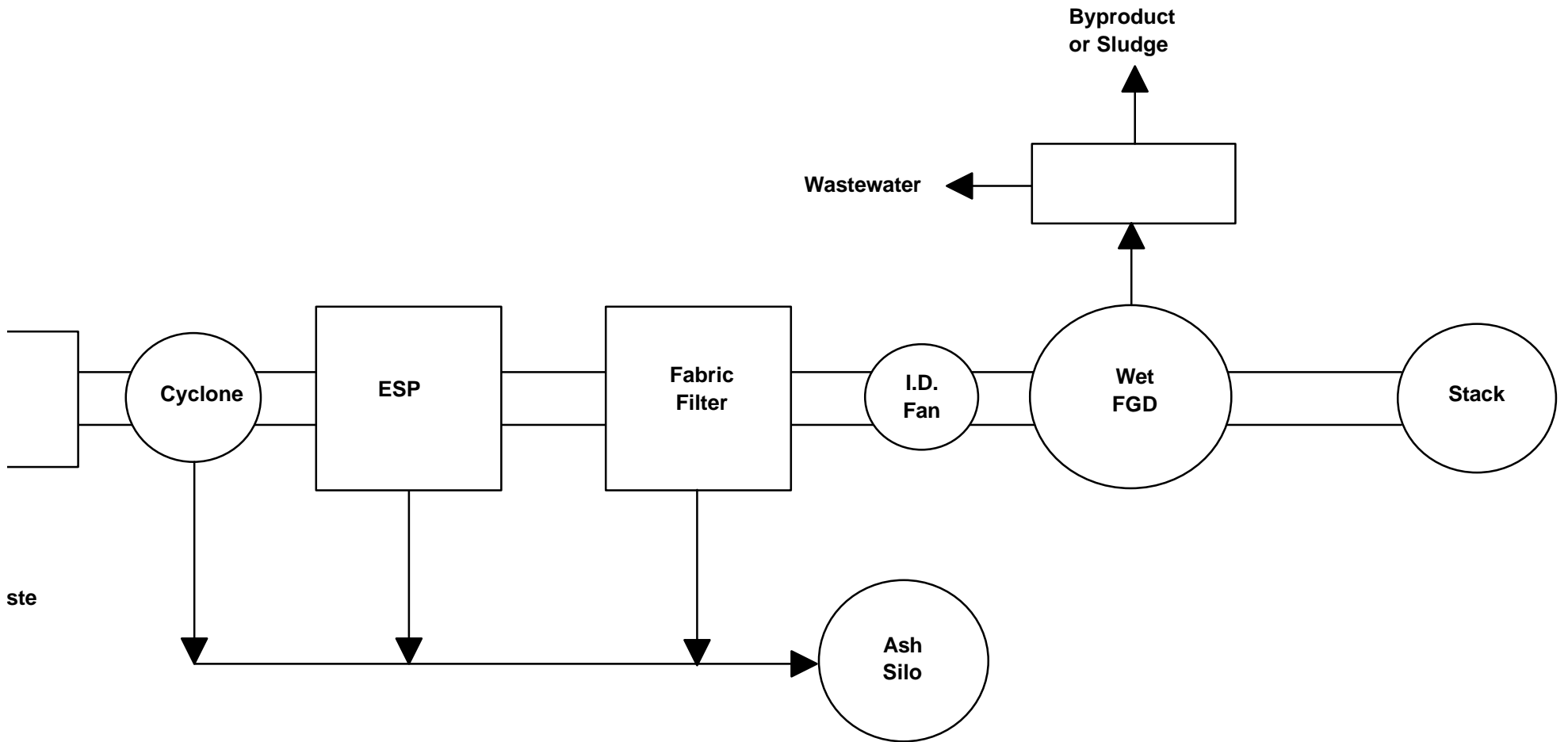
Facility Name: _____

Unit #		MWe
Unit #		MWe
Unit #		MWe
Unit #		MWe



INSTRUCTIONS:

1. Plant may submit own diagram, if available, or may use this diagram.
2. Plant may submit more than one diagram.
3. Components not installed at plant should be deleted from this diagram.
4. Components installed at plant but not shown here should be added to this diagram.
5. Indicate on this diagram where sampling ports exist or are required for solid, liquid and air streams.
6. Indicate on this diagram the lengths and cross-sectional dimensions of ducting at existing or required sampling locations.



ste

**The Canadian Uniform Data Collection Program (UDCP) for Mercury from
Coal-Fired Electric Power Generation**

**APPENDIX B: Preparation of Site-Specific Test Plans for the Coal,
Ash and Residue Sampling and Analysis Program**

Preface

The main purpose of the site-specific test plan is determine whether the sampling will meet the objectives of the “The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-fired Electric Power Generation”. The test plan will demonstrate that the sampling entity understands the sampling and analytical methods and has an approach that will be consistent with the national objectives and requirements. As numerous sources will be sampled under the UDCP, it is essential to adopt a consistent approach that will result in coal, ash and residue data that is consistent, compatible and comparable.

This document is an outline of the format for site-specific test plans for coal, ash and residues sampling and analysis that are to be submitted as part of the UDCP . A reasonable amount of time will be provided for the consultant/company to prepare the plan. A test plan does not necessarily have to be lengthy to be effective.

Table of Contents (for this document, and the test plan)

Site-specific test plans must include a table of contents containing, at a minimum, the items listed in the table of contents below. The essential information required in each section of the test plan is detailed in the following sections.

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6.1 QA/QC Procedure	5
6.2 Sample Handling, Identification and Custody	6
7.0 Qualifications	7
8.0 Report and Data Format	7

List of Figures

In this section, list the figures appearing throughout the test plan.

List of Tables

List the tables appearing throughout the test plan in this section.

1.0 Introduction

1.1 Purpose

In this section, provide a brief description of the facility and the purpose for the test program.

1.1 Objectives

In this section of the test plan, provide an overview of the facility test program and generating units to be tested. List the specific objectives and core components of the test program.

1.1 Contacts

In this section, list the key contacts (individuals/organizations) for the actual testing program. As a minimum, the contacts should include individual(s) from the company, the sampling consultant and the laboratory(s) performing the analyses. Contacts should be individuals in positions of authority and responsibility.

2.0 Process Description

2.1 Schematic of Unit(s) to be Tested

This area should include a detailed diagram for each unit(s) to be tested. In the diagrams, trace the processes from beginning to end, identifying major operations and equipment. Show only those flow streams that relate to the coal and residues sampling and analysis. *(Please note: utilities may wish to prepare this diagram with sufficient detail to be included in both UDCP Part II and Part III test plans.)*

2.2 Operating Conditions

For the unit(s) to be tested, describe process operating conditions, key parameters and standard operating ranges, control devices and operating schedules. Process operation should include operating conditions such as feed rates and materials, firing rates and fuels, temperatures, pressures, production rates, by-products, etc. Other process information should include the operating schedule (24 hours/day, 7 days per week, etc). Information on process operation will help to determine the sampling schedules and will also help to flag any potential conflicts in sampling timetables. Any maintenance completed on the process or control equipment should be identified. Steady process operation is also very important during the emission testing. *(Please note: utilities may wish to prepare this section with sufficient detail to be utilized for both UDCP Part II and Part III test plans.)*

3.0 Sampling Location Description

3.1 Coal, Ash and Residue Sampling Locations

A detailed description and diagram is required for each sampling location. Information should include:

- description of sampling locations
- access to the sampling site
- modifications, if any, that will be undertaken to facilitate sampling
- discussion of the representativeness of each sampling location

4.0 Methodologies

4.1 Coal, Ash and Residues Sampling and Analytical Methods

Describe the methods to be used for coal, ash and residues sampling and analysis. A description of the equipment and reagents, which can be inserted in the Appendix, should also be included.

4.2 Deviations

The program manager must approve, in advance, any proposed deviations from the prescribed methods. Although deviations may be unavoidable, they are discouraged as much as possible. It is critical that deviations are considered before the survey for the following reasons:

- To allow enough time to investigate the impact on sample representativeness
- To permit the consultant to take corrective action when a deviation is not approved.

5.0 Sampling Strategy and Schedule

5.1 Test Site Organization

Prepare a table showing the key tasks to be undertaken as part of the sampling program and the task leaders. In addition, prepare a table that lists sampling team assignments and responsibilities.

5.2 Test Matrix

Prepare a table with the following information:

- sampling locations
- sampling method
- analytical method
- analytical laboratory

5.3 Test Schedule

For each of the generating units being tested, describe the testing timeframes and schedules.

6.0 Quality Assurance/Quality Control

6.1 QA/QC Procedure

It is assumed that, where utilities have indicated they will follow one of the prescribed ASTM or EPA methods, they will also adhere to the corresponding quality assurance/quality control requirements of the standards. Where alternative procedures are proposed, however, the site-specific test plan must include information on all quality assurance and quality control (QA/QC) measures and procedures for the sampling program. This would typically include, but is not limited to, the following:

- Measures taken to avoid sample contamination such as pre-cleaning of the sample containers, reagent purity, proofing and handling of equipment in the field.
- Summary of acceptance criteria, control limits and corrective action
- Discussion of special QC procedures
- Data reduction techniques.
- Procedures for addressing and correcting problems in the field.
- External quality assurance and performance audits (control and reference materials).
- QC checklists must be included and can be added as an Appendix to the Test Plan.

6.2 Sample Handling, Identification and Custody

Provide a description of measures to maintain sample integrity such as sample identification and labelling, preservation of samples, container type, storage method, recovery facilities and chain-of-custody and tracking procedures. Provide details such as the names of person(s) responsible for handling and identifying samples. Also, sample log sheets and chain of custody forms should be included in an Appendix to the report.

7.0 Qualifications

As part of the test plan, describe the sampling team's and the analysis team's corporate history and experience and the qualifications of the staff participating in the emissions sampling and analysis.

8.0 Data Reporting and Format

Describe the manner in which the facility plans on submitting coal, ash and residues data in accordance with Part II of the UDCP. A potential reporting format is described in Appendix D.

The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-Fired Electric Power Generation

APPENDIX C: Laboratory Verification and Round Robin Testing

1.0 Background

The development of a credible mercury inventory requires that the accuracy and limits of detection of coal mercury analysis are known. Accordingly, the round robin component of the program seeks to:

- assess the proficiency of candidate laboratories in determining the mercury content in coal and ash samples,
- provide quality assurance for data gathered as part of the UDCP; and
- determine mercury and chlorine contents for coals and ash commonly found in Canadian electric power generating facilities.

This work will complement subsequent stack testing and solids sampling program components of the program.

2.0 Program Description

In order to improve mercury analysis and measurement, the program is using a two-stage approach. Broadly speaking, Phase I seeks to conduct a round-robin assessment of laboratories within Canada while Phase II will provide on-going quality assurance for the duration of this two-year data collection program as well as benchmark selected coals for mercury, chlorine and other coal-specific properties.

Approved methodologies for mercury and chlorine analysis are described in Section 3.2.2 of the UDCP.

It is anticipated that Phase I will require six months to complete. Well-characterized coal and ash reference materials will be sent to candidate laboratories for analysis of specific chemical characteristics, including, but not limited to: mercury; total chlorine; sulphur; moisture content; total carbon in ash. The results will be reviewed statistically to determine each lab's individual ability to analyze the necessary parameters in specific coals and ashes, the appropriateness of their respective methods for mercury determination, and their limits of detection. A prioritized list of the candidate labs with specific reference to their ability to analyze mercury and chlorine will then be compiled.

Phase II will provide two important components to the project: ongoing quality control and quality assurance for the duration of the UDCP and the opportunity to measure the mercury and chlorine concentrations of coal and ash commonly found in Canadian power generation. During Phase II, coal and ash sample suites will be sent quarterly for analysis to the laboratories approved in Phase I, as well as others who routinely participate in the Coal and Ash Sample

Proficiency Exchange (CANSPEX) (or equivalent) program. Again, part of the sample suite will consist of reference coals, similar to Phase I, that will be used to provide an on-going measure of laboratory performance. Utilities will also have the opportunity to nominate additional samples that, as mentioned, would be representative of coal and ash commonly found in Canadian operations. Results from the broader CANSPEX (or equivalent) network should provide a good statistical population from which to estimate mercury and chlorine contents in these coals. For more information on the CANSPEX program, please see <http://www.gai-online.com/CANSPEX.PDF>.

3.0 Deliverables

The project is estimated to require 6 months to complete Phase I and an additional 18 months to complete Phase II. The deliverables from this project will be:

- quarterly progress update reports,
- an interim report on Phase I, detailing the performance: multiple and single lab precision, recovery, lower quantitative limits, flexibility and economics, and
- a final report with statistical data on their performance as individuals and as a collective for both Phases, as well as a report on the benchmarking of the sampled coals used in Canada.

It is recommended that this program element begin in August, 2002 in order to identify suitable laboratories for the sampling and analysis data collection program.

The total cost of this contract will be based on the number of participating laboratories.

**The Canadian Uniform Data Collection Program (UDCP) for Mercury from
Coal-Fired Power Generation**

APPENDIX D: Data Fields for the Coal, Ash and Residue Sampling and Analysis Program

	Field Header	Field Description
1-8: Identification of the plant, its generating units and the sample period	1 Company Name	The name of the electricity generating company
	2 Plant Name	The name of the plant.
	3 Street	The street address of the plant
	4 City / Town	The city / town of the plant
	5 Province	The province of the plant
	6 Unit #	The unit for which coal / ash samples are taken(All units at this plant are to be sampled and analyzed.)
	7 Sample Period	The week for which sampling has occurred to obtain the composite samples described below.
	8 Unit Production	Unit production (MWh) for the sample period
9-49: Identification of the fuel / fuel blend burned in each generating unit for the sample period	9 Fuel Type 1	The primary coal / fuel burned.
	10 Fuel Type 1 %	The percentage of fuel type 1 in this sample.
	11 Coalfield 1	The coalfield from which fuel type 1 is derived
	12 Mine / Other 1	Name of mine or other location identification for fuel type 1
	13 Seam 1	The coal seam for which the fuel type 1 weekly sample is applicable (if available)
	14 Supplier 1	Name of fuel type 1 supplier
	15 Street 1	Street address of fuel type 1 supplier
	16 City 1	City of fuel type 1 supplier
	17 P / S 1	Province / State of fuel type 1 supplier
	18 PC / ZC 1	Postal Code / Zip Code of fuel type 1 supplier
	19 Country 1	Country of fuel type 1 supplier
	20 Fuel Type 2	The secondary coal / fuel burned. (if applicable)
	21 Fuel Type 2 %	The percentage of fuel type 2 in this sample
	22 Coalfield 2	The coalfield from which fuel type 2 is derived
	23 Mine / Other 2	Name of mine or other location identification for fuel type 2
	24 Seam 2	The coal seam for which the fuel type 2 weekly sample is applicable (if available)
	25 Supplier 2	Name of fuel type 2 supplier

26	Street 2	Street address of fuel type 2 supplier
27	City 2	City of fuel type 2 supplier
28	P / S 2	Province / State of fuel type 2 supplier
29	PC / ZC 2	Postal Code / Zip Code of fuel type 2 supplier
30	Country 2	Country of fuel type 2 supplier

31	Fuel Type 3	The tertiary coal / fuel burned. (if applicable)
32	Fuel Type 3 %	The percentage of fuel type 3 in this sample
33	Coalfield 3	The coalfield from which fuel type 3 is derived
34	Mine / Other 3	Name of mine or other location identification for fuel type 3
35	Seam 3	The coal seam for which the fuel type 3 weekly sample is applicable (if available)
36	Supplier 3	Name of fuel type 3 supplier
37	Street 3	Street address of fuel type 3 supplier
38	City 3	City of fuel type 3 supplier
39	P / S 3	Province / State of fuel type 3 supplier
40	PC / ZC 3	Postal Code / Zip Code of fuel type 3 supplier
41	Country 3	Country of fuel type 3 supplier

42	Other Additives	Other significant substances added to fuels, eg. limestone, SO ₂ sorbents
43	Mine	Name of mine or other location identification for the sorbent
44	Supplier	Name of additive supplier
45	Street	Street address of additive supplier
46	City	City of additive supplier
47	P / S	Province / State of additive supplier
48	PC / ZC	Postal Code / Zip Code of additive supplier
49	Country	Country of additive supplier

50-66: Fuel sampling and analyses

50	Fuel Sample ID	The sample ID is the analysis number that is used to identify the as-burned coal / fuel sample as it is analyzed by the testing laboratory.
51	Fuel consumption	The fuel use for the period (Kg)
52	Sulfur Fuel	The total sulfur content of the fuel sample, by percentage (by weight, dry basis).
53	MJ/kg Fuel	The higher heating value of the fuel sample, in megajoules per kilogram (MJ/kg - dry basis).
54	Ash Fuel	The ash content of the fuel sample, by percentage (by weight).
55	Ash Fuel Dry	The ash content of the fuel sample, by percentage (by weight, dry basis).
56	Moisture Fuel	The moisture content (by weight) of the fuel sample, by percentage.
57	Mercury Fuel	The mercury content of the fuel sample, in ppm (dry basis) or ND if below detection limits.
	Chlorine Fuel	The chlorine content of the coal / fuel sample, in ppm (dry basis) or ND if below detection limits.
58	VM Fuel	The volatile matter content (weight) of the coal/fuel sample, by percentage
59	Fixed Carbon Fuel	The fixed carbon content (weight) of the coal/fuel sample (by percent)
60	Dry Carbon Fuel	The carbon content of the coal/fuel sample (by percent, dry weight)

61	Hydrogen Fuel	The hydrogen content of the coal/fuel sample (by percent, dry weight)
62	Oxygen Fuel	The oxygen content of the coal/fuel sample (by percent, dry weight)
63	Nitrogen Fuel	The nitrogen content of the coal/fuel sample (by percent, dry weight)
64	Obtain M Fuel	The specific method(s) used by the electric utility company to obtain the coal/fuel sample (e.g., ASTM...).
65	Prepare M Fuel	The specific method(s) used by the testing laboratory to prepare the coal / fuel sample for analysis of mercury (e.g., ASTM...).
66	Analysis M Fuel	The specific method used by the testing laboratory to analyze the coal / fuel sample for mercury (e.g., EPA Method...).

67-77: Coal rejects sampling and analyses

67	Rejects Sample ID	The sample ID is the analysis number that is used to identify the coal rejects sample as it is analyzed by the testing laboratory.
68	Rejects Production	The total rejects generated, and represented by this sample, during the sampling period (Kg)
69	Sulfur Rejects	The total sulfur content of the coal rejects sample, by percentage (dry basis).
70	MJ/kg Rejects	The higher heating value of the coal rejects sample, in megajoules per kilogram (MJ/kg - dry basis).
71	Ash Rejects	The ash content of the coal rejects sample, by percentage (dry basis).
72	Moisture Rejects	The moisture content of the coal rejects sample, by percentage.
73	Mercury Rejects	The mercury content of the coal rejects sample, in ppm (dry basis) or ND if below detection limit.
74	Chlorine Rejects	The chlorine content of the coal rejects sample, in ppm (dry basis) or ND if below detection limit.
75	Obtain M Rejects	The specific method(s) used by the electric utility company to obtain the coal rejects sample (e.g., ASTM...).
76	Prepare M Rejects	The specific method(s) used by the testing laboratory to prepare the coal rejects sample for analysis of mercury (e.g., ASTM...).
77	Analysis M Rejects	The specific method used by the testing laboratory to analyze the coal rejects sample for mercury (e.g., EPA Method...).

78-87: Fly ash sampling and analysis

78	F-Ash Sample ID	The sample ID is the analysis number that is used to identify the fly ash sample as it is analyzed by the testing laboratory.
79	Amount of F-Ash	The amount of fly ash generated, and represented by this sample, during the sampling period (Kg)
80	Mercury F-Ash	The mercury content of the fly ash sample, in ppm (dry basis) or ND if below detection limit.
81	Carbon F-Ash	The unburned carbon content of the fly ash, by percentage

82	Chlorine F-Ash	The chlorine content of the fly ash sample, in ppm (dry basis) or ND if below detection limit.
83	Sulphur F-Ash	The total sulphur content of the fly ash sample, by percentage (dry basis).
84	Moisture F-Ash	The moisture content of the fly ash sample, by percentage.
85	Obtain M F-Ash	The specific method(s) used by the electric utility company to obtain the fly ash sample (e.g., ASTM...).
86	Prepare M F-Ash	The specific method(s) used by the testing laboratory to prepare the fly ash sample for analysis of mercury and LOI (e.g., ASTM...).
87	Analysis M F-Ash	The specific method used by the testing laboratory to analyze the fly ash sample for mercury and LOI (e.g., EPA Method...).

88-97: Bottom ash sampling and analyses

88	B-Ash Sample ID	The sample ID is the analysis number that is used to identify the bottom-ash sample as it is analyzed by the testing laboratory.
89	B-Ash Production Rate	The amount of bottom ash generated, and represented by this sample, during the sampling period (Kg)
90	Mercury B-Ash	The mercury content of the bottom ash sample, in ppm (dry basis) or ND if below detection limit.
91	Carbon B- Ash	The unburned carbon content of the bottom ash, by percentage
92	Chlorine B- Ash	The chlorine content of the bottom ash sample, in ppm (dry basis) or ND if below detection limit.
93	Sulphur B-Ash	The total sulphur content of the bottom ash sample, by percentage (dry basis).
94	Moisture B-Ash	The moisture content of the bottom ash sample, by percentage.
95	Obtain M B-Ash	The specific method(s) used by the electric utility company to obtain the bottom ash sample (e.g., ASTM...).
96	Prepare M B-Ash	The specific method(s) used by the testing laboratory to prepare the ash sample for analysis of mercury and LOI (e.g., ASTM...).
97	Analysis M B-Ash	The specific method used by the testing laboratory to analyze the bottom ash sample for mercury and LOI (e.g., EPA Method...).

98-99: These fields can be used to record useful information.

98	Acc-Prec Hg	Information on any evidence of accuracy and precision of analysis for mercury in fuel, rejects and ash can be entered here.
99	Notes	Any other useful notes about these analyses can be entered here.

100-123: Identification of the laboratory(ies) that performed the analyses and the aspects of the analyses performed by each lab.

100	Laboratory 1	Name of first laboratory performing analyses
101	Approved	Is this a UDCP approved laboratory?
102	Analyses 1	What elements of the analyses were carried out at laboratory 1?
103	Street L1	Street address of laboratory 1
104	City L1	City of laboratory 1
105	P / S L1	Province / State of laboratory 1
106	PC / ZC L1	Postal Code / Zip Code of laboratory 1

107	Country L1	Country of laboratory 1
108	Laboratory 2	Name of second laboratory performing analyses (if applicable)
109	Approved	Is this a UDCP approved laboratory?
110	Analyses 2	What elements of the analyses were carried out at laboratory 2?
111	Street L2	Street address of laboratory 2
112	City L2	City of laboratory 2
113	P / S L2	Province / State of laboratory 2
114	PC / ZC L2	Postal Code / Zip Code of laboratory 2
115	Country L2	Country of laboratory 2
116	Laboratory 3	Name of third laboratory performing analyses (if applicable)
117	Approval	Is this a UDCP approved laboratory?
118	Analyses 3	What elements of the analyses were carried out at laboratory 3?
119	Street L3	Street address of laboratory 3
120	City L3	City of laboratory 3
121	P / S L3	Province / State of laboratory 3
122	PC / ZC L3	Postal Code / Zip Code of laboratory 3
123	Country L3	Country of laboratory 3

**The Canadian Uniform Data Collection Program (UDCP) for Mercury from
Coal-Fired Electric Power Generation**

**APPENDIX E: Preparation of Site-Specific Test Plans for the Air
Emissions Testing Program**

Preface

The main purpose of the site-specific test plan is to determine whether the sampling will meet the objectives of the “The Canadian Uniform Data Collection Program (UDCP) for Mercury from Coal-fired Electric Power Generation”. The test plan will demonstrate that the sampling entity understands the sampling and analytical methods and has an approach that will be consistent with the national objectives and requirements. As numerous sources will be sampled under the UDCP, it is essential to adopt a consistent approach that will result in emission data that is consistent, compatible and comparable.

A requirement of the UDCP emission testing program is the collection of sufficient data to allow mercury mass balances to be performed, thereby providing a powerful QA/QC tool for assessing the success of the flue gas sampling. Details of this requirement are included and discussed in this Appendix.

This document is an outline of the format for site-specific test plans for flue gas sampling and analysis that are to be submitted as part of the UDCP. A reasonable amount of time will be provided for the consultant/company to prepare the test plan. A test plan does not necessarily have to be lengthy to be effective.

Table of Contents (for this document, and the test plan)

Site-specific test plans must include a table of contents containing, at a minimum, the items listed in the table of contents below. The essential information required in each section of the test plan is detailed in the following sections.

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List of Figures

In this section, list the figures appearing throughout the test plan.

List of Tables

List the tables appearing throughout the test plan in this section.

1.0 Introduction

1.1 Purpose

In this section, provide a brief description of the facility and the purpose for the test program.

1.2 Objectives

In this section of the test plan, provide an overview of the facility test program and generating units to be tested. List the specific objectives and core components of the test program.

1.3 Contacts

In this section, list the key contacts (individuals/organizations) for the actual testing program. As a minimum, the contacts should include individual(s) from the company, the sampling consultant and the laboratory(s) performing the analyses. Contacts should be individuals in positions of authority and responsibility.

2.0 Process Description

2.1 Schematic of Unit(s) to be Tested

This area should include a detailed diagram for each unit(s) to be tested. In the diagrams, trace the processes from beginning to end, identifying major operations and equipment. Show only those flow streams that relate to the emissions test. *(Please note: utilities may wish to prepare this diagram with sufficient detail to be included in both UDCP Part II and Part III test plans.)*

2.2 Operating Conditions

For the unit(s) to be tested, describe process operating conditions, key parameters and standard operating ranges, control devices and operating schedules. Process operation should include operating conditions such as feed rates and materials, firing rates and fuels, temperatures, pressures, production rates, by-products, etc. Other process information should include the operating schedule (24 hours/day, 7 days per week, etc). Information on process operation will help to determine the sampling schedules and will also help to flag any potential conflicts in sampling timetables. Any maintenance completed on the process or control equipment should be identified. Steady process operation at full load conditions is also very important during the emission testing. *(Please note: utilities may wish to prepare this section with sufficient detail to be included in both UDCP Part II and Part III test plans.)*

3.0 Sampling Location Description

3.1 Flue Gas Sampling Locations

Flue gas sampling must be conducted downstream of the last emission control device. Although generally not required, it may be necessary to also sample upstream of the first control device. For example, this could be required if suitable samples of the feed coal, along with the

coal feed rate, cannot be obtained. A detailed description and diagram is required for each sampling location. Information should include:

- duct lengths and cross-sectional
- direction of flow
- flow disturbances
- number and configuration of traverse points
- sampling port placement
- access to the sampling site
- discussion of the representativeness of each sampling location

3.2 Coal, Ash and Residue Sampling Locations

A detailed description and diagram is required for each sampling location. Information should include:

- description of sampling locations
- access to the sampling site
- modifications, if any, that will be undertaken to facilitate sampling
- discussion of the representativeness of each sampling location

4.0 Methodologies

4.1 Flue Gas Sampling and Analysis Methods

Describe the methods to be used for flue gas sampling and analysis. Include a schematic of the sampling train and a flow diagram or a description of the sample recovery procedure. As a minimum, sampling duration, train configuration, impinger contents, proofing or cleaning procedures and recovery and partitioning of the samples should be specified in this section. A description of the equipment and reagents, which can be inserted in the Appendix, should also be included.

4.2 Coal, Ash and Residue Sampling and Analysis

Describe the methods to be used for coal, ash and residues sampling and analysis. A description of the equipment and reagents, which can be inserted in the Appendix, should also be included. Note that it is essential to sample the coal as close as possible to where its feed rate is being measured, so that a dry basis coal feed rate can be determined (see Section 6.3, Mass Balances).

4.3 Deviations

The program manager must approve, in advance, any proposed deviations from the prescribed methods. Although deviations may be unavoidable, they are discouraged as much as possible. It is critical that deviations are considered before the survey for the following reasons:

- To allow enough time to investigate the impact of a deviation on the emission results.
- To permit the consultant to take corrective action when a deviation is not approved.

5.0 Sampling Strategy and Schedule

5.1 Test Site Organization

Prepare a table showing the key tasks to be undertaken as part of the sampling program and the task leaders. In addition, prepare a table that lists sampling team assignments and responsibilities.

5.2 Test Matrix

Prepare a table with the following information:

- sampling locations
- number of runs
- sampling method
- sample run times
- analytical methods
- analytical laboratory

5.3 Test Schedule

For each of the generating units being tested, describe the testing timeframes and schedules, including a summary of pre-test preparation activities.

6.0 Quality Assurance/Quality Control

6.1 QA/QC Procedures

It is assumed that, where utilities have indicated they will follow the prescribed Ontario Hydro Method, they will also adhere to the corresponding quality assurance/quality control requirements of that method. Where alternative procedures are proposed, however, the site-specific test plan must include information on all quality assurance and quality control (QA/QC) measures and procedures for the sampling program. This would typically include, but is not limited to, the following:

- Calibration methods and results for the sampling equipment such as dry gas meters, orifice meters, nozzle diameters, differential pressure gauges and temperature readers. The calibration results may be included in the Appendices.
- Measures taken to avoid sample contamination such as pre-cleaning of the glassware, reagent purity, proofing and handling of equipment in the field.
- Listing of blank solutions and trains.
- Summary of acceptance criteria, control limits and corrective action
- Discussion of special QC procedures, such as those contained in the prescribed Ontario Hydro Method
- Data reduction techniques.
- Procedures for addressing and correcting problems in the field.
- External quality assurance and performance audits (control and reference materials).
- QC checklists must be included and can be added as an Appendix to the Test Plan.

6.2 Sample Handling, Identification and Custody

Provide a description of measures to maintain sample integrity such as sample identification and labelling, preservation of samples, container type, storage method, recovery facilities and chain-of-custody and tracking procedures. Provide details such as the names of person(s) responsible

for handling and identifying samples. Also, sample log sheets and chain of custody forms should be included in an Appendix to the report.

6.3 Mass Balances

The UDCP emission testing protocol requires that sufficient data be collected to allow a mercury mass balance to be calculated for each test run. This requires that the mercury input to the boiler in the coal and the mercury retained in the bottom ash and fly ash (and any other significant residues) be known, in addition to the measured mercury emissions.

At a minimum, the following data are required to perform these mass balances:

- “As-fired” or “as-weighed” coal feed rate (Mg/hr)
- “As-fired” or “as-weighed” coal moisture, to give the dry basis coal feed rate (Mg/hr)
- Dry basis mercury (mg/kg) and ash (%) contents of the coal
- Dry basis mercury (mg/kg) and carbon (%) contents of the bottom ash and fly ash
- A best-estimate of the split between the bottom ash and fly ash fractions
- The total mercury emission rate (g/hr)

An example of the mass balance calculations is shown in Section 8.3.

7.0 Qualifications

As part of the test plan, describe the sampling team’s and the analysis team’s corporate history and experience and the qualifications of the staff participating in the emissions sampling and analysis.

8.0 Air Emissions Testing Program Report Format

For the purposes of commonality among the various Test Reports, and to facilitate a thorough, accurate and speedy review by the scientific authorities for the UDCP, it is strongly recommended that all reports be prepared using the following format. The final mercury emissions report format is described in the Table of Contents and template Tables on the following pages.

Note: The Site-Specific Test Plans for the UDCP Part III - Air Emissions Testing Program, as described previously in this Appendix, will contain elements which can be used as sections of the final test report.

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8.2 Format of Data Reporting Tables

Table 3-1 Test Matrix for the _____ Plant, Generating Unit # _____						
Sampling Location	No. of Runs	Parameters	Sampling Method	Sample Run Time (minutes)	Analytical Method	Analytical Laboratory
Downstream ¹		Speciated Hg				
Downstream		Moisture				
Downstream		Flow				
Downstream		O2/CO2				
Upstream ²		Speciated Hg				
Upstream		Moisture				
Upstream		Flow				
Upstream		O2/CO2				
Fuel Feed ³		Hg, Cl in Fuel				

1. Downstream refers to the location after the last pollution control device (usually in the stack).
2. Upstream refers to the location before the first pollution control device. These data will not normally be required for most emission test programs
3. Fuel Feed refers to “as-fired” or “as-weighed” coal sampling location

**Table 3-2
Summary of Results**

Sample location	Elemental Mercury (g/hr)	Oxidized Mercury (g/hr)	Particle-bound Mercury (g/hr)	Total Mercury (g/hr)
<u>Coal</u> Run 1 Run 2 Run 3 <u>Average</u>	Not Applicable	Not Applicable	Not Applicable	
<u>Bottom Ash</u> Run 1 Run 2 Run 3 <u>Average</u>	Not Applicable	Not Applicable	Not Applicable	
<u>Fly Ash</u> Run 1 Run 2 Run 3 <u>Average</u>	Not Applicable	Not Applicable	Not Applicable	
<u>Downstream Flue Gas</u> Run 1 Run 2 Run 3 <u>Average</u>				
<u>Upstream Flue Gas</u> (if applicable) Run 1 Run 2 Run 3 <u>Average</u>				

**Table 3-3
Comparison Of Volumetric Flow Rate Data**

Run No.	Upstream			Downstream			CEMS
	acm/min	scm/min	dscm/min	acm/min	scm/min	dscm/min	scm/min
Run 1							
Run 2							
Run 3							
Average							

acm/min – actual cubic metres per minute

scm/min – standard cubic metres per minute at 25°C and 101.3 kPa

dscm/min – dry standard cubic metres per minute at 25°C and 101.3 kPa

Note: Volumetric flow rate is a critical factor in calculating mass flow rates. Ideally, the volumetric flow rate (corrected to standard pressure and temperature) measured at the inlet to the pollution control device (upstream) should be the same as that measured at the stack (downstream), which should be the same as that measured by the CEMS. Table 3-3 allows a comparison of flow rates at these three locations. However, the comparisons will not be applicable if upstream sampling is not required and there are no CEMS in operation.

**Table 3-4
Downstream Individual Run Results**

Test Run Number:	1	2	3	Average
Source Condition				
Fuel Factor, dscm/10 ⁹ J				
Date				
Start Time				
End Time				
Elemental Mercury:				
µg detected				
µg/dscm				
g/hr				
g/109 J				
Oxidized Mercury:				
µg detected				
µg/dscm				
g/hr				
g/109 J				
Particle-bound Mercury:				
µg detected				
µg/dscm				
g/hr				
g/109 J				
Total Downstream Speciated Mercury				
µg/dscm				
g/hr				
g/109 J				
Average Gas Volumetric Flow Rate:				
@ Flue Conditions, acm/min				
@ Standard conditions, dscm/min				
Average Gas Temperature, oC				
Average Gas Velocity, m/s				
Flue Gas Moisture, % by volume				
Average Flue Pressure, mm Hg				
Barometric Pressure, mm Hg				
Average % CO ₂ by volume, dry basis				
Average % O ₂ by volume, dry basis				
% Excess Air				
Dry Molecular Wt. Of Gas, g/g-mole				
Gas Sample Volume, dscm				
Isokinetic Variance				

acm/min – actual cubic metres per minute

dscm – dry standard cubic metres at 25°C and 101.3 kPa

dscm/min – dry standard cubic metres per minute at 25°C and 101.3 kPa

**Table 3-5
Upstream Individual Run Results (if applicable)**

Test Run Number:	1	2	3	Average
Source Condition				
Fuel Factor, dscm/10 ⁹ J				
Date				
Start Time				
End Time				
Elemental Mercury:				
µg detected				
µg/dscm				
g/hr				
g/hr (based on outlet dscm/min)				
g/109 J				
Oxidized Mercury:				
µg detected				
µg/dscm				
g/hr				
g/hr (based on outlet dscm/min)				
g/109 J				
Particle-bound Mercury:				
µg detected				
µg/dscm				
g/hr				
g/hr (based on outlet dscm/min)				
g/109 J				
Total Upstream Speciated Mercury				
µg/dscm				
g/hr				
g/hr (based on outlet dscm/min)				
g/109 J				
Average Gas Volumetric Flow Rate:				
@ Flue Conditions, acm/min				
@ Standard conditions, dscm/min				
Average Gas Temperature, °C				
Average Gas Velocity, m/s				
Flue Gas Moisture, % by volume				
Average Flue Pressure, mm Hg				
Barometric Pressure, mm Hg				
Average % CO ₂ by volume, dry basis				
Average % O ₂ by volume, dry basis				
% Excess Air				
Dry Molecular Wt. Of Gas, g/g-mole				
Gas Sample Volume, dscm				
Isokinetic Variance				

acm/min – actual cubic metres per minute

dscm – dry standard cubic metres at 25°C and 101.3 kPa

dscm/min – dry standard cubic metres per minute at 25°C and 101.3 kPa

**Table 3-6
Coal Usage Results**

Test Run Number:	1	2	3	Average
Source Condition				
Date				
Start Time				
End Time				
Coal Properties:				
Carbon, % dry				
Hydrogen, % dry				
Nitrogen, % dry				
Sulphur, % dry				
Ash, % dry				
Oxygen, % dry (by difference)				
Moisture, %				
Higher Heating Value (HHV), MJ/kg				
Fd Factor O2 basis, dscm/109 J				
Fc Factor CO2 basis, scm/109 J				
Chloride, mg/kg dry				
Mercury, mg/kg dry				
Coal Consumption:				
Coal # 1, Mg/test				
Coal # 2, Mg/test				
Coal # 3, Mg/test				
Total Raw Coal Input, Mg/hr				
Total Coal Input, Mg/hr dry				
Total Mercury Available in Coal:				
Mercury, g/hr				
Mercury, g/109 J				

scm – standard cubic metres at 25°C and 101.3 kPa
dscm – dry standard cubic metres at 25°C and 101.3 kPa

Table 3-7 Coal Analysis Results (dry basis)				
Run No.	Moisture (%)	Mercury (mg/kg)	Chloride (mg/kg)	Ash Content (%)
1.				
2.				
3.				
Average				

Coal moistures are for the as-fired or as-weighed samples.

Table 3-8 Ash Analysis Results (dry basis)				
Run No.	Bottom Ash		Fly Ash	
	Mercury (mg/kg)	Carbon (%)	Mercury (mg/kg)	Carbon (%)
1.				
2.				
3.				
Average				

**Table 5-1
Collection Efficiencies for Sampled Vapour Phase Mercury**

Run No.	Impinger	Mercury Detected (µg)	Recovery Pattern	Percent of Total Vapour Phase Mercury
1.	1. KCl		%	%
	2. KCl		%	
	3. KCl		%	%
	Total Oxidized			
	4. H2O2		%	%
	5. KMnO4		%	
	6. KMnO4		%	%
	7. KMnO4		%	
2.	Total Elemental			%
	1. KCl		%	
	2. KCl		%	%
	3. KCl		%	
	Total Oxidized			%
	4. H2O2		%	
	5. KMnO4		%	%
	6. KMnO4		%	
	7. KMnO4		%	%
	Total Elemental			
3.	1. KCl		%	%
	2. KCl		%	
	3. KCl		%	%
	Total Oxidized			
	4. H2O2		%	%
	5. KMnO4		%	
	6. KMnO4		%	%
	7. KMnO4		%	
	Total Elemental			%

Table 5-2 Reagent Blank				
Sample ID	Sample Fraction	Contents	Mercury (µg)	Detection Limit (µg)

Table 5-3 Blank Train Mercury Analysis				
Container #	Sample Fraction	Contents	Mercury (µg)	Detection Limit (µg)

Table 5-4 Spiked Mercury QA/QC Train Analysis and Spike Recovery Results				
QA/QC Mercury Train Impingers	Spiked Quantity of Mercury (µg)	Quantity of Mercury Found (µg)	Recovery of Spiked Mercury	
			Individual Impingers	Average for Solution Type
1. KCl 2. KCl 3. KCl			% % %	%
4. H2O2			%	%
5. KMnO4 6. KMnO4 7. KMnO4			% % %	%
Average Recovery for the Entire QA/QC Train			%	

8.3 Example Mass Balance Calculation

As-fired (as-weighed) coal feed rate	= 250.0 Mg/hr
As-fired (as-weighed) moisture content of the coal	= 15.0%
Therefore, dry-basis (DB) coal feed rate	= 212.5 Mg/hr
Mercury content of the coal (DB)	= 0.0750 mg/kg
Therefore, inlet mercury mass flow rate	= 212.5 x 0.0750 = 15.94 g/hr
Ash content of the coal (DB, carbon-free)	= 15.0%
Therefore, DB carbon-free ash mass flow rate	= 212.5 x 0.15 = 31.88 Mg/hr
DB carbon content of the bottom ash	= 5.00%
DB carbon content of the fly (ESP) ash	= 3.50%
Assuming a split of 25% bottom ash and 75% fly (ESP) ash, then the average carbon content of the total ash	= (0.25x5.00) + (0.75x3.50) = 3.88%
Therefore, total estimated ash mass flow rate, including carbon	= 31.88 / 0.9612 = 33.16 Mg/hr
At 25% of the total ash, bottom ash mass flow rate	= 8.290 Mg/hr
Bottom ash mercury content (DB)	= 0.0050 mg/kg
Therefore, mercury mass flow rate released in the bottom ash	= 8.290 x 0.0050 = 0.0414 g/hr
At 75% of the total ash, fly (ESP) ash mass flow rate	= 24.87 Mg/hr
Fly (ESP) ash mercury content (DB)	= 0.2000 mg/kg
Therefore, mercury mass flow rate released in the fly (ESP) ash	= 24.87 x 0.2000 = 4.974 g/hr
Therefore, total outlet mercury mass flow rate in the ash	= 0.0414 + 4.974 = 5.015 g/hr
Mercury emission rate in the flue gas	= 9.500 g/hr
Therefore, total outlet mercury mass flow rate	= 14.52 g/hr
Therefore, mercury mass balance, outlet versus inlet	= 14.52 / 15.94 x 100 = 91%
Of this, fraction found in the ash	= 31%
and fraction found in the flue gas	= 60%
Normalized to 100%, fraction found in the ash	= 35%
and fraction found in the flue gas	= 65%

Note: The data used in the above calculations are somewhat typical but are entirely fictional

**The Canadian Uniform Data Collection Program (UDCP) for Mercury from
Coal-fired Electric Power Generation**

**APPENDIX F: Measurement Of Speciated Mercury Emissions In
Stack Gas From Coal-Fired Electric Power Generating Stations:**

The Ontario Hydro Method

Prepared for the:

Canadian Uniform Data Collection Program

By:

Curtis Environmental Consulting

January 2003

Foreword

The stack gas sampling method described in this report is to be used for the determination of the concentrations and emission rates of the different chemical forms of mercury emitted from coal-fired electric power generating stations. The complexity of the procedure warrants that personnel performing the tests be trained and experienced in its use.

This version of the method is based on the original Ontario Hydro Method developed for measuring speciated mercury emissions (1). Precision and bias data for the method are based on US EPA Method 301 validation studies performed at the University of North Dakota on behalf of the US DOE and the US EPA (2). A modified version of the Ontario Hydro method has been published as an ASTM Standard (3) and is also presently listed as a US EPA Preliminary method (4).

Analytical procedures for the determination of mercury in the collected samples are not included in this document. The sample recovery procedure of the method results in impinger samples that a reputable laboratory should be able to analyze by standard CVAAS or CVAFS techniques for determining mercury in water samples. Similarly, the determination of the mercury content of particulate matter samples should pose no problems for most laboratories using standard analysis techniques. For further information regarding these analytical methods, refer to either of the ASTM or US EPA procedures referenced above, where they are fully documented.

It is recognized that this method has not been peer reviewed and may contain inconsistencies, errors, omissions or other points requiring clarification. All such requests for clarification or other observations should be forwarded to the UDCP Project Manager:

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1.0 Sampling Of Stack Gases For Speciated Mercury Emissions From Coal-Fired Electric Power Generating Stations

1.1 Scope

This method applies to the sampling of particulate-bound, oxidized and elemental mercury emitted to the atmosphere from the stacks and ducts of coal-fired electric power generating stations. Total mercury emissions are obtained from the sum of the three mercury species measured. The following stack sampling procedures, contained in the Environment Canada Standard Reference Method for Source Testing (5), form part of this method:

- Method A - Determination of Sampling Site and Traverse Points
- Method B - Determination of Stack Gas Velocity and Volumetric Flow Rate
- Method C - Determination of Molecular Weight by Gas Analysis
- Method D - Determination of Moisture Content
- Method E - Determination of Particulate Emissions
- Method F - Calibration Procedures for S-type Pitot Tube, Dry Gas Meter and Orifice Meter

It is assumed that the users of this method are familiar with the above stack gas sampling procedures, including all aspects of pre-test and post-test calibrations, data recording principles and calculations of standard stack gas parameters such as velocity, moisture content, molecular weight, volumetric flue gas flow rate, etc.

The method is applicable to concentrations of total mercury in flue gas ranging from approximately 0.5 to 100 $\mu\text{g}/\text{dscm}$, with the lower limit being based on a minimum laboratory detection limit of 0.01 μg of mercury per sample, or 0.0005 $\mu\text{g}/\text{mL}$ in the sample solutions.

Precision and bias data pertaining to the method are available in the ASTM version of the Ontario Hydro Method (4).

1.2 Principle

A representative composite stack gas sample is withdrawn isokinetically from a number of traverse points along the stack or duct cross section. Particulate-bound mercury present in the sample is collected on a filter, while the gaseous mercury is collected in a series of seven ice-cooled impingers. The first three of these impingers contain an aqueous solution of potassium chloride for trapping the oxidized mercury, which most likely exists as mercuric chloride in the stack gas. The last three contain an acidified solution of potassium permanganate that is capable of trapping the elemental mercury. The fourth impinger is placed between these two sets of three impingers and contains a solution of hydrogen peroxide in nitric acid. Its sole purpose is to protect the permanganate solutions from reaction with reducing agents in the flue gas, such as sulfur dioxide. Since all of the oxidized mercury has already been removed, any mercury found in this impinger is assigned to the elemental fraction of the catch.

1.3 Apparatus

1.3.1 Sampling Train (see Figure 1)

Most of the components listed below are based on those described in Method E for particulate emissions and in US EPA Method 29 (6) for the determination of metals emissions from stationary sources. In some cases, it may be preferable to use an in-stack filtration configuration, such as described in US EPA Method 17 (7).

Nozzle: A borosilicate or quartz glass nozzle of the buttonhook design, with a sharp, tapered leading edge. Nozzles constructed from other materials that are free from contamination and do not react with the sample may be used.

Probe: A borosilicate or quartz glass liner, with a heating and temperature indicating system capable of maintaining a temperature of at least 121°C (250°F) or that of the flue gas, whichever is greater. Teflon-lined probes may be used where length or strength limitations preclude the use of a glass liner, or if a US EPA Method 17 configuration is being used.

Pitot Tube: A calibrated S-type (Strausscheibe) pitot tube, or equivalent device, mounted on the probe assembly for monitoring stack gas velocity.

Stack Temperature Sensor: A thermocouple, or equivalent device, mounted on the probe assembly, capable of measuring stack temperatures to within 1.5% of the minimum absolute stack temperature. The temperature and corrosiveness of the stack gases will determine the type of thermocouple to be used.

Cyclone (optional): A borosilicate glass miniature cyclone following the sampling probe and preceding the filter may be used to collect larger particulates. The cyclone prevents the premature build-up of particulate matter on the filter medium, thereby minimizing absorption of gaseous mercury by the collected dust as well as allowing longer sampling runs than could otherwise be achieved. The cyclone must be maintained at a temperature of at least 121°C (250°F) or the temperature of the flue gas, whichever is greater, to avoid condensation. This option will generally be required only for sampling upstream of the first control device, before the majority of the particulate matter has been removed.

Filter Holder: A borosilicate glass or Teflon-coated stainless steel filter holder with an inert filter support, contained in a heated enclosure capable of maintaining a temperature of at least 121°C (250°F) or the temperature of the flue gas, whichever is greater. The filter support shall be constructed of Teflon or other non-metallic, non-contaminating material and a suitable gasket of Teflon or silicone rubber shall be used.

Filter: High purity quartz fibre filters, containing less than 0.2 µg/m² of mercury and with an efficiency of at least 99.95% for 0.3 µm diameter particles. The filters shall be free of organic binders and be inert to sulfur dioxide (SO₂) and sulfur trioxide (SO₃).

Note 1. *In some instances, it may be advantageous to use an in-stack filtering device, such as is described in US EPA Method 17. This precludes the need to heat the filter holder assembly. However, the connecting tubing from the filter holder to the inlet of the impingers must still be heated to at least 121°C (250°F) or the temperature of the flue gas, whichever is greater, to prevent condensation. The sample recovered from this tubing is added to that from the first impinger.*

Impingers: Eight Greenburg-Smith impingers, connected in series. All but the third impinger shall be modified by replacing the tip and impaction plate of the standard design with a 1.27 cm (0.5 inch) ID glass tube extending to 1.27 cm (0.5 inch) from the bottom of the flask. The third impinger shall have the standard tip and impaction plate.

***Note 2.** When flue gases with high moisture contents (> 20%) are sampled, it may be necessary to use either an oversized impinger in the first position, or to place an additional empty impinger at the beginning of the series. If the latter option is used, the contents of this impinger are recovered and added to the sample recovered from the impinger that would otherwise be the first.*

Vacuum Pump: A leakproof vacuum pump capable of maintaining an isokinetic sampling rate and continuously withdrawing a portion of the stack gases through the sampling train. The pump is connected to the outlet of the last impinger by a vacuum line containing a vacuum gauge to measure the pump intake vacuum to within 1 kPa (0.5 inch Hg) and a coarse adjustment valve to regulate sample flow. A bypass valve is connected across the vacuum pump to allow for fine control of the sample flow.

Dry Gas Meter: A calibrated dry gas meter, temperature compensated or equipped with inlet and outlet temperature indicators, for measuring the volume of flue gas sampled.

Orifice: A calibrated orifice connected to the outlet of the dry gas meter, for measuring the sampling rate of the flue gas.

Differential Pressure Gauges: Inclined manometers, or equivalent devices, for the measurement of the pitot tube velocity pressure and the pressure drop across the calibrated orifice. They must be capable of measuring to within 0.001 kPa (0.005 inch H₂O) on the 0 to 0.25 kPa (0 to 1 inch H₂O) scale and 0.01 kPa (0.05 inch H₂O) on the 0.25 to 2.5 kPa (1 to 10 inch H₂O) scale.

Barometer: A barometer capable of measuring the atmospheric pressure to within 0.35 kPa (0.1 inch Hg).

1.3.2 Sample Recovery Apparatus

Probe Brush: A nylon or Teflon bristle brush with no exposed metallic parts and of a length and diameter suitable for cleaning the interior surface of the probe liner.

Balance: A top-loading balance capable of weighing to the nearest 0.1 g with a capacity of at least 1,500 g.

Sample Storage Containers: Petri dishes for storing and transporting filters. Pre-cleaned clear glass storage bottles (preferably wide-mouth), with Teflon-lined lids and of sufficient capacity (at least 500 mL) to hold each sample solution plus associated rinses.

Miscellaneous: Polypropylene or Teflon wash bottles, polypropylene or Teflon-coated tweezers, dropper-type pipettes, various sized volumetric flasks and graduated cylinders.

1.4 Reagents

All chemicals used shall be reagent grade (8). Water shall be deionized and conform to the specifications for Type II water given by ASTM (9).

1.4.1 Sampling Train Reagents

Deionized Water: For preparing the following solutions.

Potassium Chloride (1 mol/L): Dissolve 74.55 g of potassium chloride (KCl) in water and dilute to one litre in a volumetric flask.

Hydrogen peroxide (30% v/v):

Nitric acid (10% v/v): Add 100 mL of concentrated nitric acid (HNO₃) to approximately 800 mL of water in a one litre volumetric flask and dilute to volume with water.

Sulfuric Acid (50% v/v): While stirring, slowly add 500 mL of concentrated sulfuric acid (H₂SO₄) to approximately 400 mL of water. Allow the solution to cool to room temperature and transfer to a one litre volumetric flask. Dilute to volume with water.

Potassium Permanganate (5% w/v): Dissolve 50.0 g of potassium permanganate (KMnO₄) in about 800 mL of water and stir the contents for about two hours to allow for complete dissolution. Dilute to one litre with water in a one litre volumetric flask and store in an amber glass bottle, preferably in a dark place. This solution can be kept for the duration of a single stack test program (usually about one week), but it must be thoroughly mixed prior to each use. A fresh batch must be prepared for each stack test program.

Indicating Type Silica Gel (6-16 mesh): Dried at 177°C (350°F) for 2 hours, or used fresh.

Crushed Ice: For maintaining the impinger bath at the required temperature.

1.4.2 Sample Recovery Reagents

Deionized Water: For preparing solutions and rinsing glassware.

Nitric Acid Rinse (0.1 mol/L): Add 6.3 mL of concentrated nitric acid to approximately 900 mL of water and dilute to one litre with water.

Potassium Dichromate (5% w/v): Dissolve 25 g of potassium dichromate (K₂Cr₂O₇) in 500 mL of water.

Sodium Chloride (10% w/v): Dissolve 100 g of sodium chloride (NaCl) in water and dilute to one litre (see below).

Hydroxylamine Sulfate (15% w/v): Dissolve 15.0 g of hydroxylamine sulfate in 100 mL of 10% w/v sodium chloride solution.

Hydroxylamine Sulfate (1.5% w/v): Dilute 10 mL of the 15% w/v hydroxylamine sulfate solution to 100 mL with water.

1.5 Procedures

1.5.1 Cleaning of Glassware

Prior to any field work, all glassware must be cleaned according to the guidelines outlined in Section 5.1.1 of US EPA Method 29. Briefly, this consists of hot soapy water washing and tap water rinses prior to soaking in 10% w/v nitric acid solution for at least 4 hours. This is followed by water rinses and finally an acetone rinse before drying.

1.5.2 Sample Collection

Preliminary: Select a suitable sampling site and determine the minimum number of sampling points according to the procedures described in Report EPS 1-AP-74-1 (Method A). Determine the stack pressure, temperature, moisture and range of velocity pressures. Use this information to determine the size of nozzle required for isokinetic sampling. Recommended minimum nozzle size is 4.76 mm (3/16 inch) ID.

Sampling Time and Volume: The total sampling time for this method should not exceed 3 hours and the sampling volume should be restricted to not more than 4 m³ (dry, at reference conditions). This minimizes any reaction between dissolved sulfur dioxide and the oxidized mercury collected in the first three impingers. That reaction can cause a partial reduction from the oxidized form to elemental mercury, which is then collected in subsequent impingers and causes a bias in the speciation results.

Preparation of Sampling Train: A detailed step-by-step method summary for these procedures is given in Appendix I. Assembly of the entire train must be performed in a clean area, free from contamination. Place the filter in the filter holder and cap the holder openings. Using a graduated cylinder, transfer 100 mL of 1 mol/L KCl solution to each of the first three impingers. Mix 50 mL of 30% v/v hydrogen peroxide (H₂O₂) with 50 mL of 10% v/v HNO₃ solution and place in the fourth impinger (see Note 3 below). For each of the next three impingers, mix 80 mL of 5% w/v KMnO₄ with 20 mL of 50% v/v H₂SO₄ in a measuring cylinder and add to the impinger. Mix the contents. Add approximately 200g of silica gel to the last impinger. Weigh all impingers and record these values as well as the solution volumes on the Moisture Analysis Data Sheet (for example, see Figure 2). Set up the train as shown in Figure 1.

Note 3. *The above procedure results in a hydrogen peroxide (H₂O₂) concentration of about 15% v/v, a concentration that will be required only for very high SO₂ sources. Any excess H₂O₂ remaining at the end of the sampling run must be destroyed prior to analysis for mercury, either during sample recovery or in the analytical laboratory. This is a tedious process. It is therefore useful to pre-calculate the concentration of H₂O₂ required to react with the expected quantity of SO₂ in the flue gas to be sampled. This ensures that the H₂O₂ will be depleted at the end of the sampling, or that only a small excess remains which can easily be destroyed during sample recovery. When less than 50 mL of the H₂O₂ is used, the volume is made up to 100 mL with water.*

Adjust the heating system to provide a temperature at the probe and filter of at least 121°C (250°F) or the temperature of the stack gas, whichever is greater. Plug the nozzle inlet and leak

check the system at this temperature by drawing a 51 kPa (15 inch Hg) vacuum. A leakage rate not in excess of 0.6 L/min (0.02 ft³/min) is acceptable. Record the actual leak rate (Leak Check 1) on the Sampling Data Sheet (for example, see Method E). Also, leak check the pitot tube lines at this time to ensure that they are leak free.

Place crushed ice around the impingers. Add more ice during sampling to keep the temperature of the gases leaving the last impinger as low as possible, and always below 21°C (70°F). Temperatures above 21°C (70°F) may result in damage to the dry gas meter from either moisture condensation or excessive heat.

Operation of Sampling Train: Verify that the heating system is providing a temperature of at least 121°C (250°F) at the filter and the probe outlet. To begin sampling, position the sampling nozzle at the first sampling point (traverse point). Point the nozzle directly into the approaching gas stream and secure the entire apparatus. Immediately start the vacuum pump and adjust the sampling flow rate to isokinetic conditions. Sample for at least 5 minutes at each sampling point; sampling time must be the same for each point and at least 1.7 m³ (60 ft³) but not more than 4 m³ (140 ft³) of sample gas must be collected during the run. Maintain isokinetic sampling throughout the sampling period by making the necessary adjustments in the sampling flow rate as stack conditions change, or as the build-up particulate matter on the filter affects the flow. Computer programs, sampling rate equations or nomographs are available, or can be constructed, to aid in the rapid adjustment of the sampling rate. For each run, record the data required on the Sampling Data Sheet.

Note 4. *If the source being sampled is under negative pressure, it is necessary to start the vacuum pump at a low flow rate before inserting the probe into the gas stream, and to then adjust the sampling flow rate. Similarly, a low flow rate should be maintained at the end of the sampling time until the probe has been withdrawn, before turning off the vacuum pump.*

To simplify recording of data in the field, values may be entered in the units for which the sampling equipment is designed. These values must then be converted to the metric units specified in the equations where they are used. Instrument readings shall be recorded at intervals that are consistent with the time duration established for each point. Readings must be taken at least once at each traverse point and the time between readings must not exceed 5 minutes. Record any sampling interruptions on the reverse side of the Sampling Data Sheet. When the traverse is completed, turn off the vacuum pump and record the final instrument readings. It is recommended that the leak check procedure described in the next paragraph be followed before transferring the sampling apparatus to any additional port(s). Any excessive leaks may then be corrected before the continuation of sampling. This procedure is mandatory whenever it becomes necessary to disconnect or change sampling train components. Transfer the sampling apparatus to any additional sampling port(s) and repeat the sampling procedure.

Remove the sampling apparatus from the stack port when the test is completed. Perform a final leak check by plugging the nozzle and drawing a vacuum equal to the maximum vacuum observed during sampling. Record this result (Leak Check 2) on the Sampling Data Sheet. If the leakage rate exceeds 0.6 L/min (0.02 ft³/min), locate and record the cause on the data sheet and consult the appropriate regulatory agency regarding the validity of the test. Leak check the pitot tube lines again to ensure they have remained leak free and the measured velocity head data are valid.

Disconnect the probe and set it aside to cool. Cap the probe openings and remove the probe and impinger assemblies to a clean area for sample recovery. Exercise care when transporting the sampling train components to minimize the possibility of sample loss or contamination.

1.5.3 Sample Recovery

A detailed step-by-step method summary for these procedures is given in Appendix I.

1.5.3.1 Nozzle, Probe Liner, and Filter. Carefully remove the filter from its holder with tweezers and transfer it to a pre-weighed petri dish labelled as Container No. 1. Brush any loose particulate and filter material into the petri dish. If a miniature cyclone was used ahead of the filter, transfer the particulate from the cyclone to Container No. 1. Use a non-metallic brush if necessary.

Using the probe brush, wash the nozzle, the inside of the probe liner and the front-half of the filter holder with 0.1 mol/L HNO₃ rinse solution. Add these washings to a sample bottle labelled as Container No. 2. If the miniature cyclone was used ahead of the filter, wash the interior surfaces with 0.1 mol/L HNO₃ rinse solution, using a rubber policeman to remove the particulate matter adhering to the walls, and add these washings to Container No. 2. Perform a final rinse of all components with water and add to Container No. 2.

Note 5. *If the particulate emission rate is also being determined, it will be necessary to do an acetone rinse of the nozzle, probe liner and front-half of the filter holder prior to the above 0.1 mol/L HNO₃ washes. In that case, the acetone rinse would be placed in Container No. 2a and the nitric acid rinse would be placed in Container No. 2b.*

1.5.3.2 Impingers. Remove the impingers from the ice bath and dry their exterior surfaces. Weigh each impinger and record its weight on the Moisture Analysis Data Sheet.

Potassium Chloride Impingers (oxidized mercury): This recovery procedure is carried out separately for each of the three KCl impingers, with each one being recovered as a separate sample. Carefully add small amounts of 5% w/v KMnO₄ solution to the contents of each of the three impingers, mixing well between additions, until a pale pink colour persists.

Note 6. *This step should be carried out for all three impingers as soon as possible after completion of sampling and disassembly of the sampling train, preferably preceding the recovery of the probe, nozzle and filter assembly described above. This minimizes possible losses of elemental mercury that might be formed by the reaction of oxidized mercury with dissolved SO₂ in the impinger solutions.*

Note 7. *It is very important that great care be taken during this step to avoid the formation of a brown precipitate of MnO₂. This can occur if the KMnO₄ solution is added too quickly or a large excess is added. This then requires the careful addition of 1.5% w/v hydroxylamine solution to just dissolve the solid material, avoiding a large excess, followed by further careful addition of the KMnO₄ solution to give the desired pink colour.*

Add 5 mL of 5% w/v K₂Cr₂O₇ solution to the impingers, ensuring that a distinct yellow colour persists. Pour the contents of the first impinger into a pre-weighed sample bottle, labelled as Container No. 3. Rinse the impinger three times with approximately 30 mL of 10% v/v HNO₃ solution per rinse, and add to Container No. 3 (this ensures the final pH will be < 2). Rinse the impinger three times with water and also add to Container No. 3. Rinse the leading U-tube and

the back-half of the filter holder with both 10% v/v HNO₃ solution and water and add to Container No. 3. Add additional K₂Cr₂O₇ solution if necessary. Seal the container and record its weight on a Sample Recovery Data Sheet.

Note 8. *If the US EPA Method 17 in-stack filtering procedure was used, the connecting tubing is also recovered by rinsing with both 10% v/v HNO₃ solution and water and adding these rinses to Container No. 3.*

Except for the back-half of the filter holder washings, repeat this procedure for the second impinger to give sample Container No. 4 and for the third impinger to give sample Container No. 5.

Hydrogen Peroxide/Nitric Acid Impinger (elemental mercury): Add small amounts of 5% w/v KMnO₄ solution to the contents of the impinger, mixing well between additions, until a pale pink colour persists. Add 5 mL of 5% w/v K₂Cr₂O₇ solution to the impingers, ensuring that a distinct yellow colour persists. Pour the contents of the impinger into a pre-weighed sample bottle, labelled as Container No. 6. Rinse the impinger three times with approximately 30 mL of 0.1 mol/L HNO₃ solution per rinse, and add to Container No. 6. Rinse the impinger three times with water and also add to Container No. 6. Rinse the leading U-tube with both 0.1 mol/L HNO₃ solution and water and add to Container No. 6. Add additional K₂Cr₂O₇ solution if necessary. Seal the container and record its weight on the Sample Recovery Data Sheet.

Note 9. *If the concentration of the H₂O₂ has not been reduced from 15% v/v and this sample is being submitted directly to a laboratory for destruction of the excess H₂O₂ prior to analysis, the first two steps of this section may be omitted.*

Potassium Permanganate Impingers (elemental mercury): This recovery procedure is carried out separately for each of the three KMnO₄ impingers, with each one being recovered as a separate sample. Note and record the colour of the impinger contents (purple, brown, colourless). Rinse the leading U-tube of the fifth impinger with a small portion of the dilute 1.5% w/v hydroxylamine solution to dissolve any brown solid material, and add to the contents of the impinger. Using a dropper, slowly add small portions of 15% w/v hydroxylamine solution, rinsing down the sides and the stem of the impinger. Mix thoroughly between additions. Continue this process until the KMnO₄ solution just becomes colourless and any solid material on the walls and stem of the impinger has dissolved. Care must be taken to avoid adding a large excess of the hydroxylamine. Add small portions of 5% w/v KMnO₄ solution, mixing well between additions, until a pale pink colour persists. Add 5 mL of 5% w/v K₂Cr₂O₇ solution to the impinger, ensuring that a distinct yellow colour persists. Pour the contents of this impinger into a pre-weighed sample bottle labelled as Container No. 7. Rinse the impinger and its leading U-tube three times with 0.1 mol/L HNO₃ solution and three times with water and also add these rinses to the Container No. 7. Add additional K₂Cr₂O₇ solution if necessary. Seal the container and record its weight on the Sample Recovery Data Sheet.

Repeat this procedure for the sixth impinger to give sample container No. 8 and for the seventh impinger to give sample Container No. 9. Note that the trailing U-tube from the seventh impinger is also rinsed with 0.1 mol/L HNO₃ solution and water, with these washings being added to sample Container No. 9

Silica Gel Impinger: Note the colour and relative proportions of the silica gel (i.e. half blue, half pink). Once the weight of this impinger has been recorded, its contents can be discarded, or regenerated for future use.

1.6 Quality Assurance/Quality Control Procedures

1.6.1 Reagent Blanks

At least once per set of tests, the following blanks shall be prepared and submitted for mercury analysis. Alternatively, a complete blank train can be prepared and recovered as described above, and the samples submitted for mercury analysis.

Filter Blank: Place an unused filter in a petri dish labelled as Container No. 10.

Potassium Chloride Blank: Measure 100 mL of 1 mol/L KCl solution into a pre-weighed sample bottle labelled as Container No. 11. Add 90 mL of 10% v/v HNO₃ solution and 90 mL of water to the container and mix the contents. Add small portions of 5% w/v KMnO₄ solution, mixing well between additions, until a pale pink colour persists. Add 5 mL of 5% w/v K₂Cr₂O₇ solution to give a distinct yellow colour. Seal and record the weight of the container.

Hydrogen Peroxide/Nitric Acid Blank: In a measuring cylinder, mix 50 mL of 30% v/v H₂O₂ with 50 mL of 10% v/v HNO₃ solution and pour into a pre-weighed sample bottle labelled as Container No. 12. If less than 50 mL of the H₂O₂ was used in the fourth impinger during sampling, use the same volume for this blank and make up to 100 mL with water. Add 90 mL of 0.1 mol/L HNO₃ solution and 90 mL of water, and mix the contents. Add small amounts of 5% w/v KMnO₄ solution, mixing well between additions, until a pale pink colour persists. Add 5 mL of 5% w/v K₂Cr₂O₇ solution to give a distinct yellow colour. Seal the container and record its weight. As described previously in Note 9, the addition of the KMnO₄ and K₂Cr₂O₇ solutions may be omitted if the concentration of the H₂O₂ has not been reduced from 15% v/v and this blank is being submitted directly to a laboratory for destruction of the excess H₂O₂ prior to analysis.

Potassium Permanganate Blank: In a measuring cylinder, mix 80 mL of 5% w/v KMnO₄ solution with 20 mL of 50% v/v H₂SO₄ solution and add to a pre-weighed sample bottle labelled as Container No. 13. Using a dropper, slowly add small portions of 15% w/v hydroxylamine solution, mixing thoroughly between additions. Continue this process until the KMnO₄ solution just becomes colourless and any solid material that may have formed has dissolved. Care must be taken to avoid adding a large excess of the hydroxylamine. Add small portions of 5% w/v KMnO₄ solution, mixing well between additions, until a pale pink colour persists. Add 5 mL of 5% w/v K₂Cr₂O₇ solution to the impinger to give a distinct yellow colour. Seal and record the weight of the container.

1.6.2 Spiked Matrix QA/QC Samples

At least once per set of tests, a set of five QA/QC spiked matrix samples representing each of the three types of impinger solutions shall be prepared and submitted for mercury analysis. If deemed desirable, a blank filter can also be spiked and submitted for analysis at the same time. Alternatively, a complete train of seven impingers can be prepared and the filter and each impinger spiked with a known quantity of mercury. This train is then recovered as described previously and the samples submitted for mercury analysis.

Prepare a 1000 mg/L mercury standard by dissolving 1.3535 g of mercuric chloride (HgCl₂) in 0.1 mol/L HNO₃ solution and diluting to one litre with the same solvent. Alternatively, a commercially available 1000 mg/L mercury standard can be used for this purpose. Dilute 2.0

mL of the 1000 mg/L standard to one litre in a volumetric flask with 0.1 mol/L HNO₃ solution, adding 5 mL of 5% w/v K₂Cr₂O₇ solution before making up to the mark. Each millilitre of this QA/QC mercury standard contains 2.0 µg of mercury. Aliquots of between 1 to 10 mL can then be used to add between 2 to 20 µg of mercury to the QA/QC spiked matrix samples.

Prepare two solutions of potassium chloride, one hydrogen peroxide/nitric acid solution and two potassium permanganate solutions, as described above in Section 1.6.1 for the reagent blanks. Prior to sealing and weighing the containers, accurately pipette a known volume of the QA/QC mercury standard into each container. Record these spike volumes. Use different volumes for each type of matrix, and for each sample of the same matrix, attempting to simulate the expected distribution of mercury in the actual samples. These samples are labelled as Container No 14 to 18, respectively.

1.6.3 Collection Patterns of Samples

Since each of the seven impinger samples from each test run is analyzed separately, the results can be used to give information on the success of the sample collection. For example, if the total mercury found in the three KCl impingers was found to be distributed as 90% in the first, 8% in the second and only 2% in the third, one would be confident that the sample had been successfully trapped by those impingers. By contrast, a collection pattern of 40%, 30% and 30% would strongly indicate that sample break-through had occurred and the sampling was not successful.

Following receipt of the sample analysis results, assemble the data in groups of three KCl impingers and three KMnO₄ impingers. Calculate the collection pattern for each set. Use these data to assess the success of the sample collection. Although this is a subjective judgement, results with greater than 15% of the catch in the third impinger should generally be considered suspect.

The fourth impinger catch, while forming part of the total elemental mercury found, does not significantly affect the KMnO₄ collection pattern. Generally this quantity of mercury is small, at less than 2% of the total elemental mercury.

1.6.4 Mass Balances

Although not part of the stack testing methodology *per se*, the UDCP emission testing protocol requires that a mass balance calculation be included in the QA/QC procedures for each total mercury emission result. This requires that the mercury input to the boiler in the coal and the mercury retained in the bottom ash and fly ash (and any other significant residues) also be known. An example of these calculations using typical but fictional data is shown in Appendix II. Generally, a mass balance closure of between 80% to 120% would be considered an acceptable result in support of the measured emission rate. Note that these calculations also give an estimation of the fraction of mercury retained in the plant *versus* that emitted in the stack gas. These data are essential for estimating power plant emissions bases on mercury in coal analyses.

2.0 Analysis Of Mercury In The Samples From The Stack Tests

Full details of the analytical procedures for determining the mercury content of the stack test samples are not included in this document. The sample recovery procedure of the method results in impinger samples that any reputable laboratory should be able to analyze by standard CVAAS or CVAFS techniques for determining mercury in water samples. However, since only the weight of each sample has been measured, as opposed to its volume, it is important that the laboratory be requested to either measure the volume of each sample prior to its analysis, or to determine the specific gravity of each type of matrix. Either procedure will then allow the total mercury content of each sample to be calculated from its mercury concentration.

The determination of the mercury content of particulate matter samples should also pose no problems for most laboratories using standard analysis techniques. For further information regarding these analytical methods, refer to either of the ASTM or US EPA procedures referenced below, where they are fully documented.

It is essential that the laboratory be able to achieve a minimum detection limit of 0.1 μg of mercury per sample, or 0.00005 $\mu\text{g}/\text{mL}$ in solution, to allow the method to be used for very low mercury emission sources.

References

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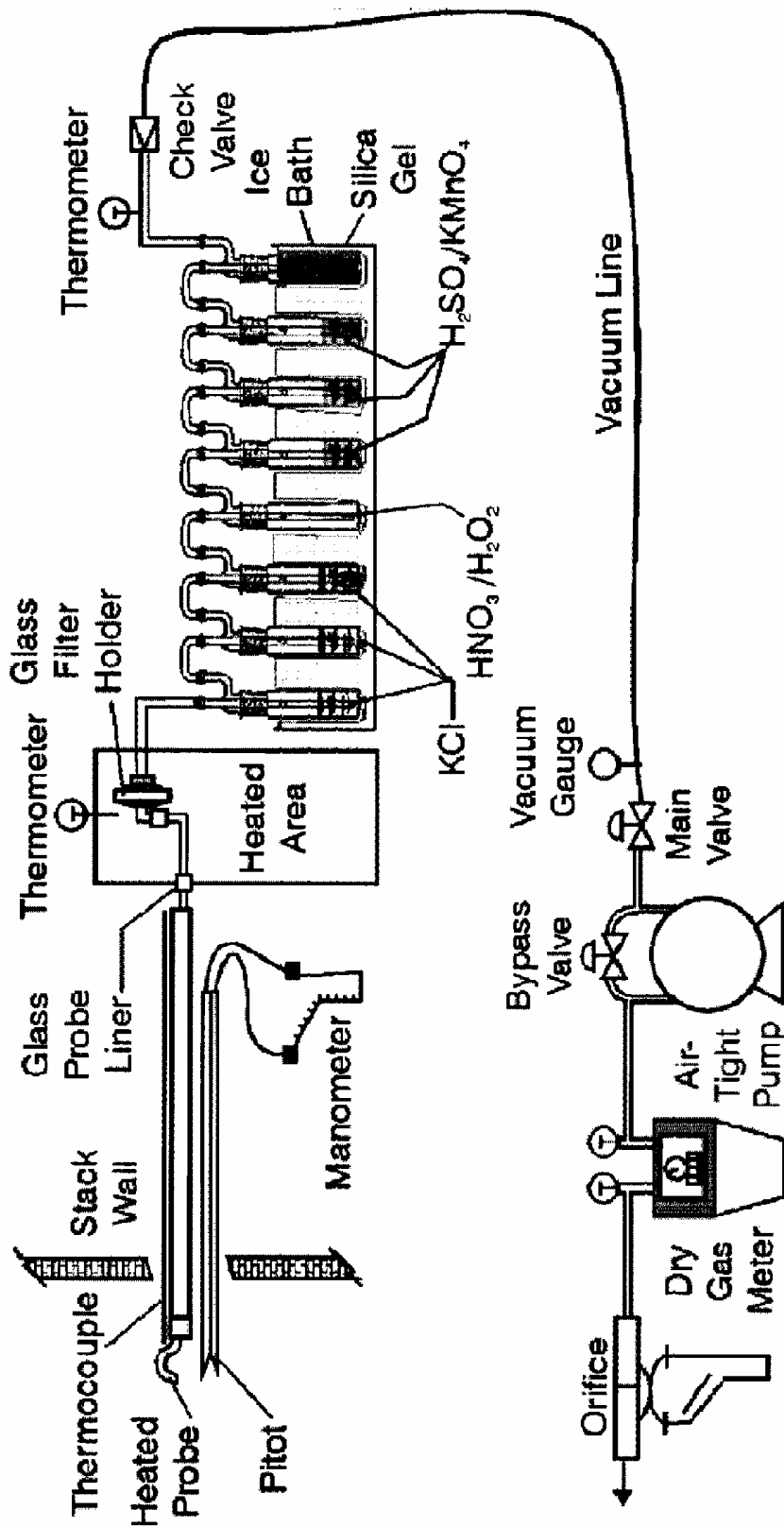


Figure 1. Schematic of Mercury Speciation Sampling Train

FIGURE 2

MOISTURE ANALYSIS DATA SHEET

Plant
 Location
 Test Number
 Test Conducted by

IMPINGER NUMBER	IMPINGER CONTENTS	MOISTURE GAIN (g.)	
		Final Initial Gain	
1	KCl (100 mL)	Final Initial Gain	(a)
2	KCl (100 mL)	Final Initial Gain	(b)
3	KCl (100 mL)	Final Initial Gain	(c)
4	H ₂ O ₂ /HNO ₃ (100 mL)	Final Initial Gain	(d)
5	KMnO ₄ /H ₂ SO ₄ (100 mL)	Final Initial Gain	(e)
6	KMnO ₄ /H ₂ SO ₄ (100 mL)	Final Initial Gain	(f)
7	KMnO ₄ /H ₂ SO ₄ (100 mL)	Final Initial Gain	(g)
8	Silica Gel (200 g)	Final Initial Gain	(h)

Weight of water collected $W_{H_2O} = a + b + c + d + e + f + g + h$
 $W_{H_2O} = g$

Appendices

- I. Step-by-Step Method Summary
- II. Example Mass Balance Calculation

APPENDIX I

Ontario Hydro Method for the Measurement of Speciated Mercury Emissions: Sample Train Loading and Recovery Procedures

1.0 Scope

This method describes the chemical solutions required, and the impinger loading and sample recovery instructions for the Ontario Hydro Method sampling train, designed for the collection and speciation of mercury emissions from stationary sources.

2.0 Personnel Qualifications/Experience

Laboratory experience in the handling of chemicals and preparation of solutions is essential, especially for performing the sample recovery procedure. Ability to follow specific detailed instructions is essential. Precision measurements and accurate data records are important.

3.0 Apparatus/Equipment/Materials

3.1 **Fumehood**

Workspace in a fumehood, suitable for use in preparing chemical solutions.

3.2 **Balance**

A top loading balance capable of measuring mass to the nearest 0.1 g is required for measuring the masses of chemicals, containers and samples. A balance capable of measuring mass to the nearest 0.0001 g is required for measuring the mass of the filter.

3.3 **Chemicals and Solutions**

The following chemicals and solutions are required for this method:

- Potassium Chloride 1 mol/L KCl
- Potassium Permanganate 5% w/v KMnO₄
- Potassium Dichromate 5% w/v K₂Cr₂O₇
- Sodium Chloride 10% w/v NaCl (see below)
- Hydroxylamine Sulfate 15% w/v (NH₂OH)₂ •H₂SO₄ in 10% w/v NaCl
- Hydroxylamine Sulfate 1.5% w/v (NH₂OH)₂ •H₂SO₄ in 1% w/v NaCl
- Nitric Acid 10% v/v HNO₃ and 0.1 N HNO₃
- Sulphuric Acid 50% v/v H₂SO₄
- Hydrogen Peroxide 30% v/v H₂O₂
- Acetone (CH₃)₂CO
- Indicating Silica Gel (coarse) For moisture capture

Note: All chemicals must be of a grade suitable for mercury testing, such as Fisher Brand Certified Grade or Trace Metal Grade.

3.4 **Glassware for Preparation of Chemical Solutions**

Glass graduated cylinders are required for preparation of chemical solutions. Generally, 100 mL, 250 mL, 500 mL and 1 L sizes are used. Volumetric flasks of 250 mL, 500 mL and 1 L capacities are also required. Larger quantities of prepared solutions are stored in 2 L and 4 L glass jugs, which are suitable for transport to

field test sites. The stock 5% w/v $KMnO_4$ solution should be stored in amber glass, or be wrapped in aluminum foil to protect it from light.

3.5 Sampling Train

The sampling train consists of the following components:

- pitot tube/thermocouple assembly (attached to probe)
- probe liner, borosilicate or quartz glass
- probe nozzle, borosilicate or quartz glass
- filter holder, glass/Teflon
- filter hot-box/impinger cold-box assembly
- eight glass impingers connected in series with leak-free ground glass fittings

3.6 Filters

Quartz fibre filters (110 mm diameter), free of organic binders, shall be used.

3.7 Impinger Stand

Two impinger stands, each capable of supporting at least 8 impingers.

3.8 Sample Bottles

Use 500 mL clean clear glass bottles with Teflon lined caps. VWR "TraceClean Tall Wide-Mouth" pre-cleaned bottles meet or exceed the performance-based quality standards of the US EPA. If pre-cleaned bottles are not available, sample bottles must be thoroughly rinsed three times with 10% nitric acid, then three times with deionized water.

3.9 Petri Dish

New or pre-cleaned (as sample bottles above) polystyrene, 150 mm diameter, petri dishes are required for storing the sample filters both before and after sampling.

3.10 Polypropylene or Teflon-Coated Tweezers

For transferring the filter from the sampling train filter holder to the petri dish.

3.11 Teflon Probe Brush

A probe brush made from a length of Teflon tubing that is cut at one end to create bristles and is long enough to extend down the inner length of the probe being used (or a commercially available equivalent). This is required for the quantitative recovery of mercury and any particulate matter from the nozzle and probe liner.

3.12 Labels

Labels for identifying samples with information such as test site, test number and sample number.

3.13 Wash Bottles and Pipettes

Polypropylene or Teflon wash bottles and dropper-type pipettes are required for the addition of reagents and washing of equipment during sample recovery.

3.14 Teflon Tape

Teflon tape is required for capping off the open ends of the sample train prior to the sampling and again at its completion, prior to sample recovery. It may also be used to ensure leak free seals on sample bottles.

4.0 Calibration/Check Standards

A laboratory log of all calibration records must be maintained. The dry gas meters and the pitot tube should be calibrated before and after each use, or at least every 6 months. The train components (probe nozzle, pitot tube, metering system, probe heater, temperature gauges and barometer) are calibrated according to the appropriate sections of US EPA Method 5. If one or more flue gas analyzers are used to measure the composition of the flue gas for molecular weight determinations, they must be calibrated prior to each use with standard calibration gas mixtures.

5.0 Test Method Laboratory Procedures

5.1 Loading Sample Train

5.1.1 Filter

Measure and record the mass of an empty, labelled petri dish to the nearest 0.0001g. Place a quartz fibre filter that has been dried and stored in a desiccator into the petri dish and record the combined mass to 0.0001g. Prior to the test, load the filter into the sampling train's glass filter holder assembly.

5.1.2 Loading Impingers #1, #2 and #3

Measure and record the mass of each empty impinger to the nearest 0.1g (#1 and #2 are modified, #3 is standard).

Pour 100 mL of 1.0 mol/L potassium chloride (KCl) solution into each impinger. Weigh and record to the nearest 0.1g.

5.1.3 Loading Impinger #4 (for sampling in high SO₂ conditions)

Measure and record the mass of the empty impinger to the nearest 0.1g (#4 is modified).

In a graduated cylinder that holds at least 100 mL, pour 50 mL of 30% ^{v/v} hydrogen peroxide (H₂O₂). Add 50 mL of 10% ^{v/v} nitric acid (HNO₃) to make a total of 100 mL of solution. Pour into Impinger #4. Swirl the contents to ensure the solution is well mixed. Weigh and record to the nearest 0.1g.

Note: *The above procedure results in a solution with a very high H₂O₂ concentration of 15% ^{v/v}. Any excess H₂O₂ remaining at the completion of sampling must be destroyed prior to analysis for mercury, either during sample recovery or in the analytical laboratory. This is a very tedious process. Given that the sole purpose of this solution is to protect the contents of Impinger #5 (KMnO₄) from being reduced by SO₂, it is useful to pre-calculate the concentration of the H₂O₂ required to react with the expected quantity of SO₂ in the flue gas to be sampled. This ensures that the H₂O₂ will either be depleted just prior to completion of the sampling, or that only a relatively small quantity will remain which can be easily reduced by the addition of a small volume of 5% ^{w/v} KMnO₄ solution*

5.1.4 Loading Impingers #5, #6 and #7

Measure and record the mass of each empty impinger to the nearest 0.1 g (all are modified). In a graduated cylinder that holds at least 100 mL, pour 80 mL of 5% ^{w/v} potassium permanganate (KMnO₄) solution. Add 20 mL of 50% ^{v/v} sulfuric acid (H₂SO₄) to make a total of 100 mL of solution. Pour this mixture into Impinger #5. Swirl the contents to ensure the solution is well mixed. Repeat for Impingers #6 and #7. Weigh each impinger and record to the nearest 0.1g.

5.1.5 Loading Last Impinger

Measure and record the mass of an empty modified impinger to the nearest 0.1g. Partially fill impinger (about 2/3 full) with fresh coarse indicating silica gel. Weigh to nearest 0.1g and record.

5.1.6 Sample Train Assembly

Connect the series of impingers with glass U-tubes, after applying an adequate amount of silicone vacuum grease to the ball and socket joints. Alternatively, Teflon tape or impingers pre-fitted with Teflon O-rings can be used. Secure the joints with metal clamps and tighten. Seal open ends with Teflon tape. Cover the assembled train to protect it from light that may cause degradation of the potassium permanganate solution.

5.2 Sample Train Recovery

5.2.1 Disassembly of Sample Train

After stack sampling is complete and the train has been sealed and transported back to the on-site sample recovery facility, carefully remove the clamps and disassemble the sampling train. Dry the exterior surfaces of the impingers and wipe away any vacuum grease (if it was used) from the ground glass fittings. Weigh and record the mass of each impinger, then place it in the impinger stand, along with its corresponding leading U-tube.

5.2.2 Filter Recovery

Allow the glass filter holder assembly to cool and then carefully remove the filter with the tweezers and place it in a pre-weighed petri dish labelled as Sample #1. Brush any loose particulate or filter material into the container. Dry in a desiccator, then measure and record the combined mass to the nearest 0.0001 g.

5.2.3 Acetone Probe Rinse

Let the probe cool and then rinse the nozzle and probe liner with 100 mL of acetone into a pre-weighed glass bottle. Brush the inside of the probe liner with a Teflon probe brush while rinsing the probe. In the lab, rinse the front half of the filter holder with acetone and add to the sample bottle. This sample will later be transferred to a pre-weighed beaker and covered with a ribbed watch glass, which is then placed in a fumehood to allow the acetone to evaporate. Once dry, weigh and then cover the beaker securely with parafilm. This dry sample will be labelled as Sample #2(a), and its mass will be combined with that for Sample #1 to determine the total mass of particulate matter collected.

5.2.4 Nitric Acid (0.1 mol/L) Probe Rinse

Following the acetone rinse, rinse the nozzle and probe liner with 100 mL of 0.1 mol/L nitric acid into a pre-weighed clear glass bottle, again brushing with the Teflon probe brush. Perform a final rinse and brushing with deionized water and also add to the bottle. In the lab, rinse the front half of the filter holder with 0.1 mol/L nitric acid and then deionized water and add to the sample bottle. Weigh and record as Sample #2(b).

5.2.5 Recovery of Potassium Chloride Impingers - Oxidized Mercury

Sample #3: Impinger #1

Carefully add small amounts of 5% w/v potassium permanganate (KMnO₄) solution to the contents of the impinger, mixing well between additions, until a pale pink colour persists.

Note: It is very important that great care be taken during this step to avoid the formation of a brown precipitate of MnO₂. This can occur if the KMnO₄ solution is added too quickly or a large excess is added. This then requires the careful addition of the dilute 1.5% w/v hydroxylamine solution to just dissolve the solid material, avoiding a large excess, followed by further careful addition of KMnO₄ solution to give the desired pink colour.

Add 5% w/v potassium dichromate (K₂Cr₂O₇) solution (approximately 5 mL) to impinger contents so that a distinct yellow colour persists.

Pour impinger contents into a pre-weighed clear glass bottle.

Rinse Impinger #1 three times with approximately 30mL of 10% v/v HNO₃ per rinse, and add to the sample bottle. This will ensure that the final pH will be <2.

Rinse Impinger #1 three times with deionized water and add to sample bottle.

Rinse the leading U-tube with both 10% v/v HNO₃ and deionized water and also add to the sample bottle.

Add additional K₂Cr₂O₇ solution if necessary. Weigh and record as Sample #3.

Repeat the above procedure with Impinger #2 to give Sample #4 and with Impinger #3 to give Sample #5.

Note: The addition of the KMnO₄ solution in Step 5.2.5 above should be done as soon as possible after completion of sampling and the sampling train has been disassembled (Step 5.2.1). This minimizes possible losses of elemental mercury that might be formed by the reaction of the oxidized species with dissolved SO₂ in the impinger solutions. Where possible, this action should precede Steps 5.2.2 to 5.2.4.

5.2.6 Recovery of Hydrogen Peroxide Impinger - Elemental Mercury

Sample #6: Impinger #4

Add small amounts of 5% w/v potassium permanganate (KMnO₄) solution to the contents of the impinger, mixing well between additions, until a pale pink colour persists.

Add 5% w/v potassium dichromate (K₂Cr₂O₇) solution (approximately 5 mL) to the impinger contents so that a distinct yellow colour persists.

Pour impinger contents into a pre-weighed clear glass bottle.

Rinse Impinger #4 three times with approximately 30 mL of 0.1 N HNO₃ per rinse, and add to sample bottle.

Rinse the impinger with deionized water three times and add to bottle. Rinse the leading U-tube with nitric acid and deionized water and add to the bottle. Weigh and record as Sample #6.

Note: If the concentration of the hydrogen peroxide has not been reduced from 15% v/v and this sample is being submitted directly to the laboratory for destruction of the excess peroxide prior to analysis, the first two steps of this section can be omitted.

5.2.7 Potassium Permanganate Impingers - Elemental Mercury

Sample #7: Impinger #5

Note and record the colour of the solution (purple, brown, colourless).

Rinse the leading U-tube with a small portion of the dilute 1.5% w/v hydroxylamine sulfate in 1.0% w/v sodium chloride solution to dissolve any brown solid material, and add to the contents of the impinger.

Using a wash bottle or dropper-type pipette, carefully add small portions of 15% w/v hydroxylamine sulfate 10% w/v sodium chloride solution, rinsing down the sides and stem of the impinger. Mix the solution thoroughly between additions.

Continue this process until the potassium permanganate solution in the impinger becomes colourless and the solid material on the walls and stem of the impinger has dissolved. Avoid adding a large excess of the hydroxylamine. Then add small portions of 5% w/v potassium permanganate solution to the contents of the impinger, mixing well between additions, until a pale pink colour persists.

Add 5% w/v potassium dichromate ($K_2Cr_2O_7$) solution (approximately 5 mL) to impinger contents so that a distinct yellow colour persists.

Pour the contents into a pre-weighed 500 mL clear glass bottle.

Rinse Impinger #5 and the U-tube three times with approximately 30 mL of 0.1 mol/L HNO_3 per rinse, and add to the bottle.

Rinse the impinger and U-tube three times with deionized water and add to bottle.

Add additional $K_2Cr_2O_7$ solution to sample bottle if necessary. Weigh and record as Sample #7.

Repeat this procedure with Impinger #6 to give Sample #8 and with Impinger #7 to give Sample #9. Note that for Impinger #7 there are both the leading and following U-tubes to be included in the sample.

5.2.8 Moisture Collection

Note colour and relative portions of the silica gel (i.e. half pink, half blue).

After the weight of Impinger #7 has been recorded, empty out and discard or regenerate the used silica gel.

Determine the mass difference for each impinger (before test versus after test) and add the results together to calculate total moisture gained.

6.0 Quality Control

All data are recorded on data sheets that are checked, signed and kept for future reference. Reagent blanks and filter blanks are submitted for mercury analysis. In addition, samples can be recovered from a blank train that is run at the test site in the leak check area and submitted for analysis.

Spiked blank impinger solution samples that are recovered on site are used for control samples.

7.0 Records/Reports

Excel spreadsheet can be used for final calculations. A final report is issued to the client.

8.0 Other Requirements

Job Safety Analysis forms relating to this work should be prepared and reviewed by all participating personnel.

9.0 Bibliography

US EPA Preliminary Method 003

US EPA Method 5

Ontario Ministry of the Environment Source Testing Code

Manufacture's Isokinetic Stack Sampling Equipment Operating Manual

APPENDIX II

Example Mass Balance Calculation

As-fired (as-weighed) coal feed rate	= 250.0 Mg/hr
As-fired (as-weighed) moisture content of the coal	= 15.0%
Therefore, dry-basis (DB) coal feed rate	= 212.5 Mg/hr
Mercury content of the coal (DB)	= 0.0750 mg/kg
Therefore, inlet mercury mass flow rate	= 212.5 x 0.0750 = 15.94 g/hr
Ash content of the coal (DB, carbon-free)	= 15.0%
Therefore, DB carbon-free ash mass flow rate	= 212.5 x 0.15 = 31.88 Mg/hr
DB carbon content of the bottom ash	= 5.00%
DB carbon content of the fly (ESP) ash	= 3.50%
Assuming a split of 25% bottom ash and 75% fly (ESP) ash, then the average carbon content of the total ash	= (0.25x5.00) + (0.75x3.50) = 3.88%
Therefore, total estimated ash mass flow rate, including carbon	= 31.88 / 0.9612 = 33.16 Mg/hr
At 25% of the total ash, bottom ash mass flow rate	= 8.290 Mg/hr
Bottom ash mercury content (DB)	= 0.0050 mg/kg
Therefore, mercury mass flow rate released in the bottom ash	= 8.290 x 0.0050 = 0.0414 g/hr
At 75% of the total ash, fly (ESP) ash mass flow rate	= 24.87 Mg/hr
Fly (ESP) ash mercury content (DB)	= 0.2000 mg/kg
Therefore, mercury mass flow rate released in the fly (ESP) ash	= 24.87 x 0.2000 = 4.974 g/hr
Therefore, total outlet mercury mass flow rate in the ash	= 0.0414 + 4.974 = 5.015 g/hr
Mercury emission rate in the flue gas	= 9.500 g/hr
Therefore, total outlet mercury mass flow rate	= 14.52 g/hr
Therefore, mercury mass balance, outlet <i>versus</i> inlet	= 14.52 / 15.94 x 100 = 91%
Of this, fraction found in the ash	= 31%
and fraction found in the flue gas	= 60%
Normalized to 100%, fraction found in the ash	= 35%
and fraction found in the flue gas	= 65%

Note: The data used in the above calculations are somewhat typical but are entirely fictional