

Hope Bay Mining Limited

# Doris North Gold Mine Project: Incinerator Stack Testing Compliance Report for Section 4 Item 30 of the Project Certificate



# **DORIS NORTH GOLD MINE PROJECT:**

## **Incinerator Stack Testing Compliance Report for Section 4 Item 30 of the Project Certificate**

**February 2010**  
Project #1009-04

**Prepared for:**



Hope Bay Mining Ltd.

**Prepared by:**



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Vancouver, British Columbia

**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Executive Summary**

## Executive Summary

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The following incinerator stack emissions monitoring requirements are outlined in the Doris North Gold Mine Project Certificate (NIRB No. 003, issued September 15, 2006): under Section 4.0, Item 30.

*Commentary: NIRB expects that Canada Wide Standards for Dioxins and Furans and the Canada Wide Standards for Mercury will apply and should be followed including stack testing of incinerators.*

This report is intended to meet the requirement outlined above.

In order to comply with Item 30 in Section 4.0 of the Project Certificate, Newmont Mining Corporation (Newmont) along with Rescan Environmental Services (Rescan) conducted the following activities in 2009:

- Collected measurements of dioxin and furan emissions from the Doris incinerator stack; and
- Collected measurements of mercury emissions from the Doris incinerator stack.

An incinerator stack emissions testing program was implemented in mid October 2009 to collect samples for dioxin, furan and mercury. The samples were analyzed at an accredited laboratory. These parameters were compared with the Canada Wide Standards for Dioxins and Furans and the Canada Wide Standards for Mercury. The Canada Wide Standards were developed by the Canadian Council for Ministers of the Environment (CCME). The Doris incinerator stack emissions monitoring results indicated that there were no exceedances for the mercury emissions; however, the dioxin and furan emissions exceeded the Canada Wide Standards.



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# **Incinerator Stack Testing Compliance**

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## **Abbreviations and Definitions**

# Abbreviations and Definitions

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<b>Amberlite XAD-2</b>	An amberlite adsorbent resin used to extract dioxin and furan from the stack gas emitted from a waste incinerator.
<b>Canada-wide Standards</b>	Objectives for maximum concentrations of criteria air contaminants in the atmosphere developed to ensure long-term protection of public health and the environment.
<b>CCME</b>	Canadian Council for Ministers of the Environment.
<b>Congener</b>	In chemistry, congeners are related chemicals, e.g., a derivative or an element in the same group of the periodic table. For example, there are 209 congeners of polychlorinated biphenyls (PCB) as well as 209 congeners of polybrominated diphenyl ethers (PBDE).
<b>Criteria Air Contaminants</b>	Contaminants for which environmental regulatory agencies have established ambient air concentration limits.
<b>Mercury</b>	Mercury is a natural and persistent bioaccumulative element which can be transported many miles in the atmosphere, mercury can have impacts many years and many miles removed from its original source. A common thread through all mercury impacts is that deposition to waterbodies from anthropogenic emissions poses a threat to human and ecosystem health. Mercury also enters into the environment through the disposal (e.g., land filling, incineration) of certain products. Products containing mercury include: auto parts, batteries, fluorescent bulbs, medical products, thermometers, and thermostats.
<b>PCDD</b>	<b>Polychlorinated dibenzodioxins (PCDDs)</b> , or simply <b>dioxins</b> , are a group of polyhalogenated compounds which are significant because they act as environmental pollutants. They are commonly referred to as dioxins for simplicity in scientific publications because every PCDD molecule contains a dioxin skeletal structure. Members of the PCDD family have been shown to bioaccumulate in humans and wildlife due to their lipophilic properties, and are known teratogens, mutagens, and suspected human carcinogens. They are organic compounds.
<b>PCDF</b>	<b>Polychlorinated dibenzofurans (PCDFs)</b> , or simply <b>furans</b> , are a group of halogenated organic compounds which are toxic environmental pollutants. They are known teratogens, mutagens, and suspected human carcinogens. PCDFs tend to co-occur with polychlorinated dibenzodioxins (PCDDs). PCDFs can be formed by pyrolysis or incineration at temperatures below 1200 °C of chlorine containing products, such as PVC, PCBs, and other organochlorides, or of non-chlorine containing products in the presence of chlorine donors.
<b>Standard (or reference) conditions for temperature and pressure</b>	In physical sciences, <b>standard conditions for temperature and pressure</b> (informally abbreviated as <b>STP</b> ) are standard sets of conditions for experimental measurements, to allow comparisons to be made between different sets of data. Environment Canada uses a standard reference pressure of 101.3 kPa and a standard reference temperature of 298 K (25 degrees Celsius). For atmospheric and ambient air sampling the US EPA uses 298K (25 degrees Celsius) and 760 mm Hg (101.3 kPa) to define standard conditions.
<b>Toxic Equivalency (TEQ)</b>	The combined toxicity of all 29 dioxins is called the "TEQ" (for Toxic Equivalency).
<b>Toxicity Equivalency Factor (TEF)</b>	Toxic Equivalency Factors (TEFs) are toxicity potency factors that are used by the World Health Organization (WHO) and by scientists and regulators globally as a consistent method to evaluate the toxicities of highly variable mixtures of dioxin compounds.
<b>US EPA</b>	United States Environmental Protection Agency. The USEPA has promulgated a variety of guidelines, objectives, and reference/standard methods for collection of air emissions samples from stationary sources such as waste incinerators.
<b>Waste incinerator</b>	A device, mechanism or structure constructed primarily to thermally treat (e.g., combust or pyrolyze) a waste for the purpose of reducing its volume, destroying a hazardous chemical present in the waste, or destroying pathogens present in the waste. This includes facilities where waste heat is recovered as a byproduct from the exhaust gases from an incinerator, but does not include industrial processes where fuel derived from waste is fired as an energy source as a matter incidental to the manufacture of the primary product.

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# **1. Introduction**

# 1. Introduction

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The following atmospheric monitoring requirements are outlined in Section 4.0 Item 30 of the Doris North Gold Mine Project Certificate (NIRB No. 003, issued September 15, 2006):

*Commentary: NIRB expects that Canada Wide Standards for Dioxins and Furans and the Canada Wide Standards for Mercury will apply and should be followed including stack testing of incinerators.*

This report is intended to meet the requirement outlined above.

In order to comply with Item 30 in Section 4.0 of the Project Certificate, Newmont Mining Corporation (Newmont) along with Rescan Environmental Services (Rescan) conducted the following activities in 2009:

- Collected dioxin and furan samples from the Doris camp incinerator; and
- Collected mercury samples from the Doris camp incinerator.

Prior to the beginning of the stack emissions testing program the general methodology was reviewed with Mr. Dave Fox (Air Protection Management Analyst North, Environment Canada, Yellowknife).

Chapter 2 of this report provides a description of the Doris incinerator and how it was being operated at the time of the stack emissions testing program in late September and early October 2009. Chapter 3 of this report provides the methods used for the dioxin and furan measurements, and Chapter 4 of this report provides the methods for the mercury measurements. Chapter 5 provides a brief discussion of the results for both dioxin/furan and mercury.



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## **2. Process Description**

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The incinerator operated at Hope Bay, Doris Camp is a Westland Incinerator, Model CY 100 CA-D-O two-stage incinerator. The incinerator utilizes a primary combustion chamber and secondary afterburner section, and is equipped with a six-metre (nominal) refractory lined stack. Plates 2.1-1 and 2.1-2 show the Doris incinerator from two different perspectives.



*Plate 2.1-1 The Doris incinerator viewed from the north, July 2009.*



*Plate 2.1-2 The Doris incinerator viewed from the east, July 2009.*

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Prior to waste introduction, the secondary chamber was pre-heated to approximately 900°C, and the primary chamber was pre-heated to 650°C. Once the primary and secondary chambers reached the set-point operating temperatures, waste was manually introduced to the primary chamber via the main charge door. Approximately 10 to 15 kg of waste comprised of one wet and one dry bag of “waste” were loaded at about 90 to 110 minute intervals. Incineration of the waste proceeded until the next waste charging sequence occurred. In general, the system was allowed to stabilize for about ten to fifteen minutes prior to the start of the stack emissions sampling. Appendix A contains the details of the operating conditions for the Doris waste incinerator. Plates 2.1-3 and 2.1-4 show the method used to introduce waste into the incinerator.

The stack emissions test schedule is summarized in Table 2.1-1.

**Table 2.1-1. Stack Emissions Test Schedule**

Date	Feed Rate (Nominal)	Test Matrix
September 29, 2009	10 kg/hr	one test for dioxin/furan
September 30, 2009	10 kg/hr	one mercury test, one dioxin/furan test, one mercury test
October 1, 2009	10 kg/hr	one dioxin/furan test, one mercury test

*note: O<sub>2</sub>/CO<sub>2</sub>, temperatures and stack gas velocities were measured throughout the test program using Environment Canada – Environmental Protection Service reference techniques*



*Plate 2.1-3. The incinerator operator adding waste to the Doris incinerator primary chamber, September 2009.*



*Plate 2.1-4. Two bags of camp waste were introduced into the Doris incinerator every 90 to 110 minutes during the September/October 2009 stack emissions testing campaign. The black bag contains “dry” waste (i.e., biosolids) from the on-site sewage treatment plant and the clear bag contains “wet” kitchen waste.*

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### **3. Dioxin and Furan**

### 3. Dioxin and Furan

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), commonly known as dioxins and furans, are toxic, persistent, bioaccumulative, and result predominantly from human activity. Due to their extraordinary environmental persistence and capacity to accumulate in biological tissues, dioxins and furans are slated for virtual elimination under the *Canadian Environmental Protection Act (CEPA)*, the federal *Toxic Substances Management Policy (TSMP)* and the *CCME Policy for the Management of Toxic Substances*.

The presence of dioxins and furans in the Canadian environment can be attributed to three principle sources: point source discharges (to water, air and soil), contamination from *in situ* dioxins and furans, and loadings from the long-range transportation of air pollutants.

Waste incineration has historically been responsible for a significant portion of the dioxins and furans emitted in Canada. The total release of dioxins and furans from this sector amounts to 22.5% of the total releases to the atmosphere. Improved exhaust gas controls to reduce emissions of acid gases and fine particulates or activated carbon injection systems have decreased emissions of dioxins and furans from the municipal solid waste (MSW) sector (CCME 2001). The Canada Wide Standard for Dioxin and Furans (Appendix B) indicate that the maximum allowable dioxin and furan (PCDD/PCDF) emissions are 80 picograms per reference cubic meter (pg/Rm<sup>3</sup>) of international toxic equivalents (I-TEQ) where a reference cubic meter is measured at 25°C and 101.3 kPa. In addition, it is standard practice to provide the dioxin and furan concentrations at 11% oxygen (O<sub>2</sub>).

The remainder of this chapter describes the methods that were used to sample and test the dioxin and furan emissions from the Doris incinerator stack.

#### 3.1 MONITORING METHODS

The sampling and analytical methods used for the dioxin and furan emissions testing conformed to the procedures outlined in the Environment Canada – Environmental Protection Service (EC-EPS 1989) emission monitoring reference method manuals (Appendix C). The specific methods used for the incinerator stack emissions testing program are summarized in Table 3.1-1.

**Table 3.1-1. Summary of Doris Incinerator Stack Emissions Testing Methods for Dioxin and Furan**

Parameter	Reference Method
Sample and velocity traverse points	EPS Method 8 A
Velocity and flowrate	EPS Method 8 B
Gas molecular weight (O <sub>2</sub> /CO <sub>2</sub> )	EPS Method 8 C
Stack gas moisture	EPS Method 8 D
Dioxin/furan	EPS Method 1/RM/2 with 1/RM/3 analysis

##### 3.1.1 Equipment Preparation Techniques

The preparation, cleaning, and proofing of the stack emissions sampling equipment and materials is an integral part of the quality assurance/quality control (QA/QC) program. Following are details of the cleaning and proofing of the equipment required for the dioxin and furan sampling equipment.

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### 3.1.1.1 *Glassware Cleaning*

1. Washed twice with industrial strength cleaner/detergent
2. Rinsed with generous amounts of deionized H<sub>2</sub>O
3. Rinsed three times with methylene chloride
4. Rinsed three times with hexane
5. Rinsed three times with acetone
6. Oven baked at 300°C overnight
7. Rinsed three times with hexane (saved for proofing)
8. Rinsed three times with acetone (saved for proofing)

### 3.1.1.2 *Amberlite XAD-2*

1. Rinsed and extracted with deionized H<sub>2</sub>O
2. Soxhlet extraction with methanol, methylene chloride and toluene (22 hrs each)
3. Nitrogen purge
4. Oven dried @ 50°C
5. Approximately 40-gram aliquot saved for proofing
6. Individual sample traps packed and spiked with surrogate regime

### 3.1.1.3 *Organic Filters*

1. Soxhlet extraction (16 hrs) with toluene
2. Nitrogen drying
3. Save 1 filter for proofing

### 3.1.1.4 *Other Glassware*

1. Hot detergent wash with brushing
2. Copious deionized H<sub>2</sub>O rinses

## 3.1.2 **Reference Methods for Dioxin and Furan Stack Emissions Sampling**

Following are brief descriptions of the reference method sampling techniques utilized to collect the dioxin and furan samples from the Doris incinerator stack. Isokinetic stack testing methods were used. Isokinetic stack testing withdraws the sample from the stack at the same flow rate at which the stack gas is moving through the stack. Isokinetic stack testing ensures that a representative sample of the stack gases is sampled.

### 3.1.2.1 *EPS Method 8a - Sampling Site and Traverse Points*

The stack sampling ports for the Doris Camp incinerator stack were located about seven stack diameters downstream and greater than two diameters upstream of the nearest flow disturbances. From these criteria, a measured stack diameter of 46 cm (18.0 inches), and Figure A-1 of EPS Method A, a 12-point sampling regime was established. The prescribed sample regime for each isokinetic stack

test included six sample points along two axes at 90° traverses. The test point locations for the Doris Camp incinerator stack are summarized in Table 3-1-2.

**Table 3.1-2. Doris Camp Incinerator Stack Testing Sample Points**

Point	Distance from Stack Wall (cm)
1	2.5
2	6.6
3	13.5
4	32.3
5	39.1
6	43.2

### 3.1.2.2 EPS Method 8b - Stack Gas Velocity and Volumetric Flowrate

At each traverse point a series of measurements including stack temperature, velocity pressure, static pressure, and sampling rate were recorded. Velocity and static pressures were measured with a calibrated S-type pitot tube mounted alongside the sample probe. Stack temperatures were measured with a calibrated K-type chromel-alumel thermocouple with a control console mounted digital readout. Cyclonic flow angles were measured using the null velocity technique.

### 3.1.2.3 EPS Method 8c - Molecular Weight by Gas Analysis

Stack gas molecular weight was determined by a series of grab samples that were analyzed on site for O<sub>2</sub> and CO<sub>2</sub> by Fyrite analyzers. Plate 3.1-1 shows a typical Fyrite analyzer being used to determine the O<sub>2</sub> and CO<sub>2</sub> content of the stack gas. A minimum of four to a maximum of eight grab samples were collected per test and the results averaged for insertion in the computer programs for result calculations and corrections to 11% O<sub>2</sub>. As a QA/QC component, calibrated portable O<sub>2</sub> analyzers were also used for oxygen analysis.

### 3.1.2.4 EPS Method 8d - Moisture Content

Stack gas moisture content was determined from the measured condensed water vapour that was collected in the impinger (cold box) section of the sampling trains, and the gas volume sampled corrected to standard conditions of 25°C and 101.3 kPa (dry).

### 3.1.2.5 EPS 1/RM/2 - Dioxin/Furan

The sample equipment for the dioxin and furan testing was assembled and leak checked at the laboratory the night prior to testing. Prior to sampling initiation, the stack train was assembled as shown in Figure 3.1-1 and leak checked to code specifications. The probe (quartz or glass lined) and filter module were heated to 120 +/- 15°C and crushed ice was placed around the impingers. Iced water was circulated in the condenser and in a cooling jacket around the XAD cartridge. Once the sampling system achieved the appropriate temperatures the probe tip was positioned at point No.1, isokinetic sampling was performed using the Ko orifice constant sampling procedure. A set of recordings was taken every four minutes until four sets of readings for each sample point of traverse one was achieved. The sample pump was shut off and the sample module with attached probe was withdrawn from the stack. The system was repositioned at point No. 1 of the next traverse and an additional 96 (6 points for 16 minutes each) minutes of sampling commenced. This regime was continued until both sample ports had been sampled. The total sample volume for each dioxin and furan test was 3.6 to 4.1 Rm<sup>3</sup> for the three tests.





*Plate 3.1-1. The O<sub>2</sub> and CO<sub>2</sub> concentration in the stack gas was determined using portable Fyrites analyzers.*

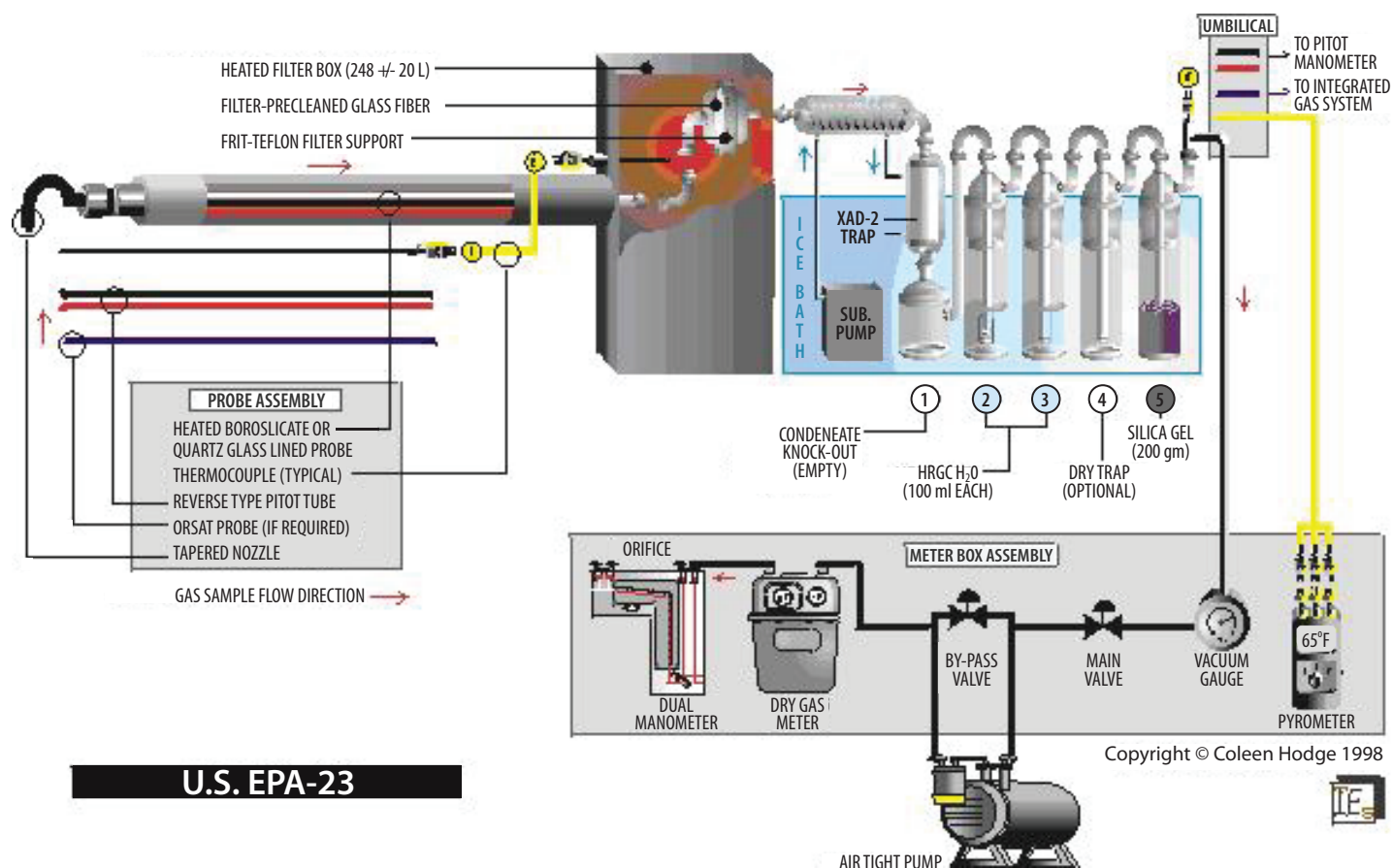
At the conclusion of the sampling, the train was checked for leaks and the probe was disassembled from the hot box/sample module. Any open ends of the sampling module and probe assembly were immediately sealed with pre-cleaned aluminium foil or teflon tape, and leak checks were conducted with only teflon tape touching the open ends.

At the conclusion of each test, the sample module and probe were lowered from the stack location and were in transport to the laboratory immediately. Approximately twenty minutes elapsed from sample conclusion to sample delivery at the sample recovery "laboratory".

### **3.1.3 Analytical and Sample Recovery Techniques**

Following sampling for dioxin and furan, the sample train was sealed and transported to the field laboratory for sample recovery. At the laboratory, the sample train was disassembled and six components were identified for each train. The recovery of each sample is described below:

1. **Sample Filter:** The exposed sample filter was removed from its holder with clean tweezers, placed on a sheet of aluminium foil, folded inside the foil and sealed in a glass petri dish. This was labelled component 1 of each test.
2. **Front/Back Half Washings:** This included a thorough acetone/hexane rinsing and brushing of the sample nozzle, probe liner, and connecting glassware prior to the filter. These washings were collected in a pre-cleaned one litre amber sample bottle with a teflon-lined lid. This was labelled component 2 of each test. The back half of the filter holder and glassware connecting the filter holder to the condenser were rinsed and soaked with acetone and hexane with the solvents added to the component 2 sample bottle.



Note: HRGC = high resolution gas chromatography.

XAD-2 = an amberlite adsorbent resin used to extract dioxin and furan from the incinerator stack gases.

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3. **Amberlite XAD-2 Resin Trap:** The resin trap was sealed with teflon tape, covered with aluminium foil and placed kept at about 4 °C prior to shipment to the analytical laboratory. This was labelled component 3 of each test.
4. **Impinger Condensate:** The condensate contained in the condensate trap, plus water and condensate from the impingers was measured for volume and collected in precleaned amber bottles. These samples were labelled component 4 for each test.
5. **Soak:** The glassware connecting the filter to the XAD module was soaked with hexane and acetone sequentially, three times, with all “soaks” and rinses collected in 1 litre amber sample bottles.
6. **Final Rinse:** All glassware was rinsed with hexane and acetone, sequentially three times into amber sample bottles.

All samples were labelled appropriately and placed in a cold room at 4°C until the analyses began. In addition, each bottle containing solvent was marked with the liquid level and the lid was sealed.

### 3.1.4 Organic Sample Analysis

The organic analysis of the sample train components involved an extremely complex series of procedures as detailed in the analytical manuals.

Following is a description, in very simplified terms, of the basic procedures used to process the sample train components.

Initially the sample components are separated into liquid (containers 2, 4, 5, 6) or solid phases (containers 1 and 3). Figure 3.1-2 summarizes the recovery procedures for semi-volatile organics. Solid samples are extracted with various solvents (usually toluene), sometimes under acid conditions. The liquid samples are extracted and concentrated with a rotary evaporator, with the final concentrate added to the filter and XAD components. At this point, an internal standard solution is added to the sample for QA/QC recovery determinations. Figure 3.1-3 summarizes the extraction and concentrating of the various train components.

The toluene rinse has internal standards added, with subsequent concentration by rotary evaporation. The extract volumes are fractionated, cleaned-up and analyzed by high-resolution gas chromatograph/mass spectrophotometer (GC/MS) analytical instrumentation. Figure 3.1-4 summarizes the analytical methodology for the dioxin and furan samples.

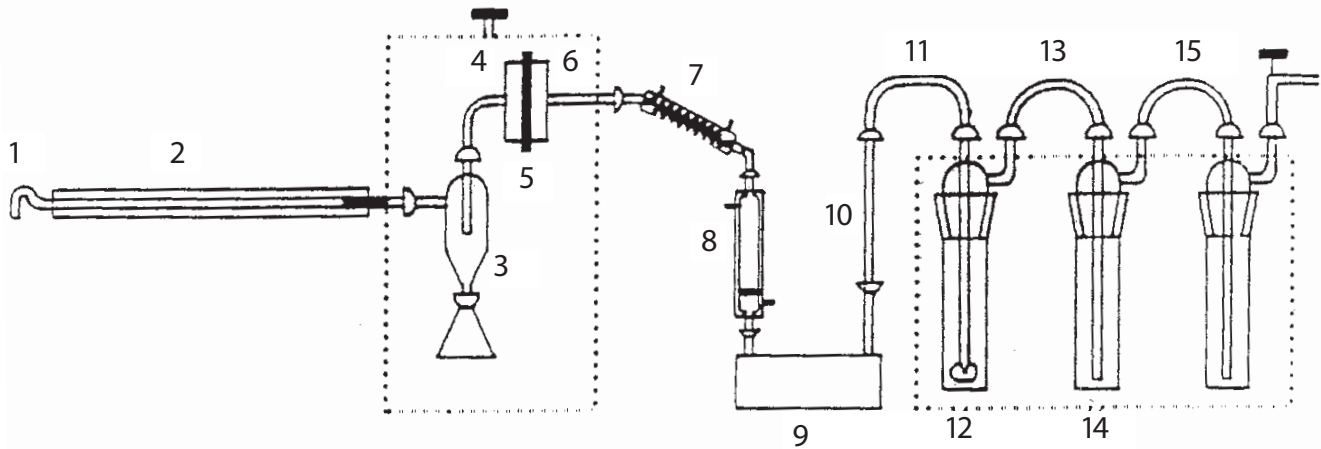
### 3.1.5 Quality Assurance/Quality Control (QA/QC) Techniques

The QA/QC component of this survey was designed to exceed the requirements normally instituted by the regulatory agency. Additionally, QA/QC of this survey was accomplished by the following mechanisms.

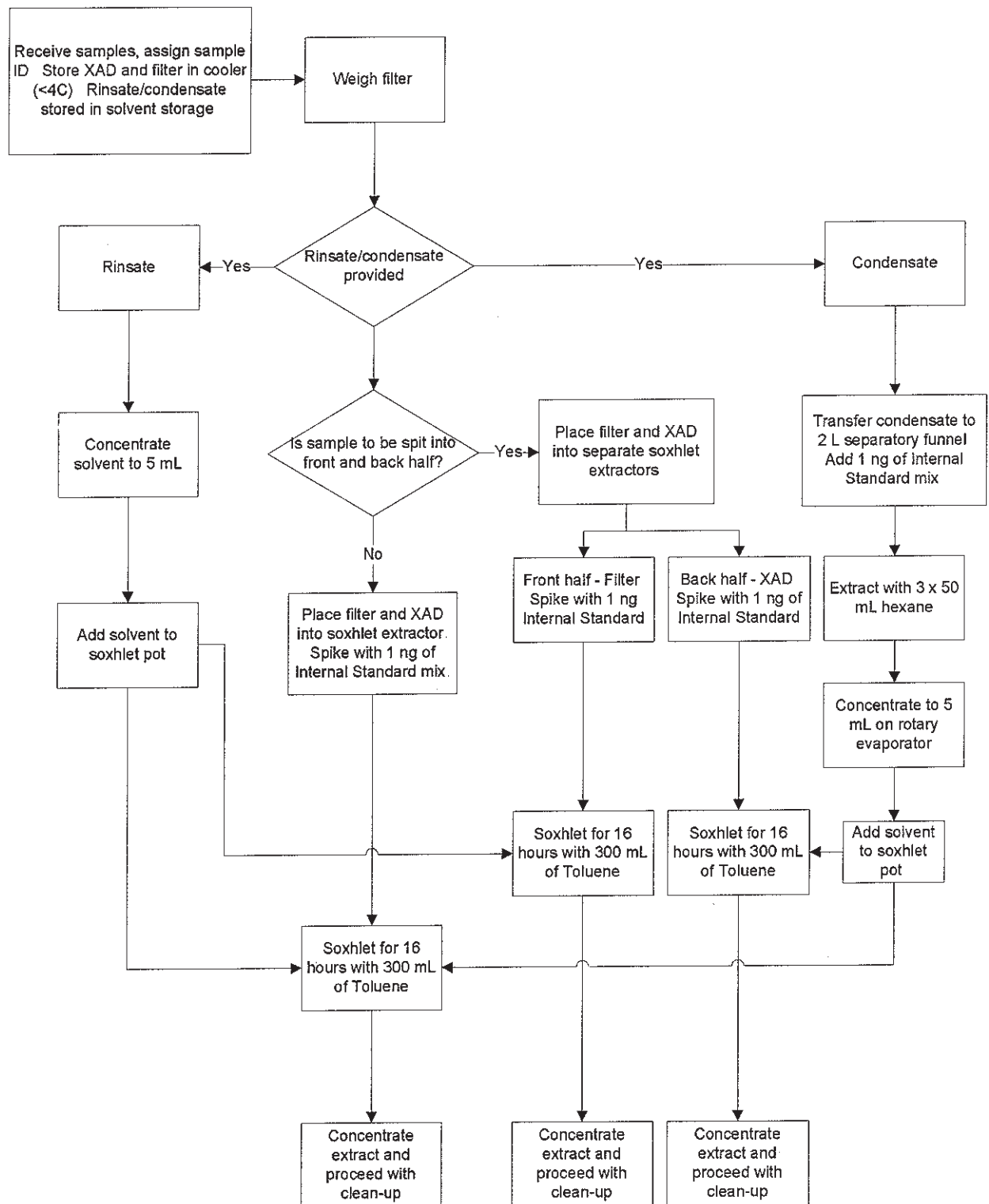
1. Pre and Post test leak checks
2. Calibration of volume measuring and monitoring instrumentation
3. Proofing of organic glassware and supplies (archived proofs)
4. Analysis of all blank solutions and materials
5. Spiking and recovery analysis of organic trains

6. Use of acid cleaned micro quartz filters
7. Duplicate analysis of selected samples
8. Reference material analysis with samples
9. Labelling and record-keeping
10. Surrogate spiking of dioxin trains using EPA protocols

A "Blank" test was conducted, with that sampled analysed in the same manner as test samples.

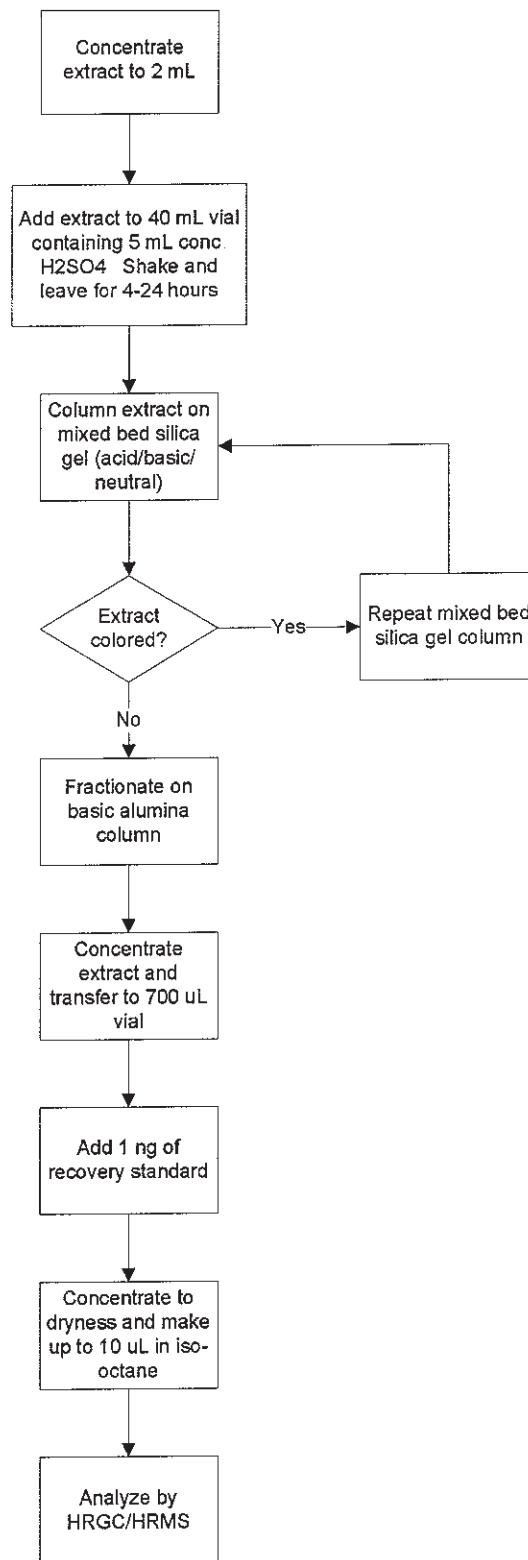


Container or Sample	Component (s)	Recovery Procedure
1	1,2,3,4	Wash and brush 3 times each with hexane (H) and acetone (A). Rinse 3 times each with H and A.
2	5	Remove carefully from holder. Place on pre-cleaned glass petri dish.
3	6,7	Soak 5 minutes each with H and A. Rinse 3 times each with H and A.
4	8	Cap ends and wrap in foil.
5	9,12	Empty contents into container and rinse each 3 times with HPLC water.
6	6 to 15 except 8	Rinse 3 times each with H and A.



**Extraction Schematic  
Diagram for Stack Samples**

Figure 3.1-3



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## **4. Mercury**



## 4. Mercury

Waste incineration has historically been responsible for a significant portion of the mercury emitted in Canada. Mercury is a natural and persistent bioaccumulative element that can be transported many miles in the atmosphere, mercury can have impacts many years and many miles removed from its original source. A common thread through all mercury impacts is that deposition to water bodies from anthropogenic emissions poses a threat to human and ecosystem health. Mercury also enters into the environment through the disposal (e.g., land filling, incineration) of certain products. Products containing mercury include: auto parts, batteries, fluorescent bulbs, medical products, thermometers, and thermostats.

The Canada Wide Standard for Mercury (CCME 2000, Appendix D) indicate that the maximum allowable mercury emissions are 20 micrograms per reference cubic metre ( $\mu\text{g}/\text{Rm}^3$ ) where a reference cubic metre is measured at 25°C and 101.3 kPa. In addition, it is standard practice to provide the mercury concentrations at 11% oxygen ( $\text{O}_2$ ).

The remainder of this chapter summarizes the methods used for mercury sampling.

### 4.1 MONITORING METHODS

The sampling and analytical methods used for the mercury emissions testing conformed to the procedures outlined in the Environment Canada – Environmental Protection Service (EC-EPS 1993) emission monitoring reference method manuals (Appendix C). The specific methods used for the incinerator stack emissions testing program are summarized in Table 4.1-1.

**Table 4.1-1. Summary of Doris Incinerator Stack Emissions Testing Methods for Mercury**

Parameter	Reference Method
Sample and velocity traverse points	EPS Method 8 A
Velocity and flowrate	EPS Method 8 B
Gas molecular weight ( $\text{O}_2/\text{CO}_2$ )	EPS Method 8 C
Stack gas moisture	EPS Method 8 D
Particulate Matter/Mercury	EPS Method 8 with US EPA Method 29 (metals)

Figure 4.1-1 summarizes the sampling equipment used to collect the mercury samples following the standard methods in the US EPA Method 29 Determination of Metal Emissions from Stationary Sources. Appendix E contains US EPA Method 29 Determination of Metal Emissions from Stationary Sources.

Similar to the dioxin and furan emissions sampling, the mercury emissions sampling was conducted using isokinetic sampling methods. Isokinetic sampling withdraws a sample from the stack at the same rate at which the air emissions are travelling in the stack. Isokinetic sampling removes a representative sample from the stack that is not biased by over or under sampling the actual stack gas.

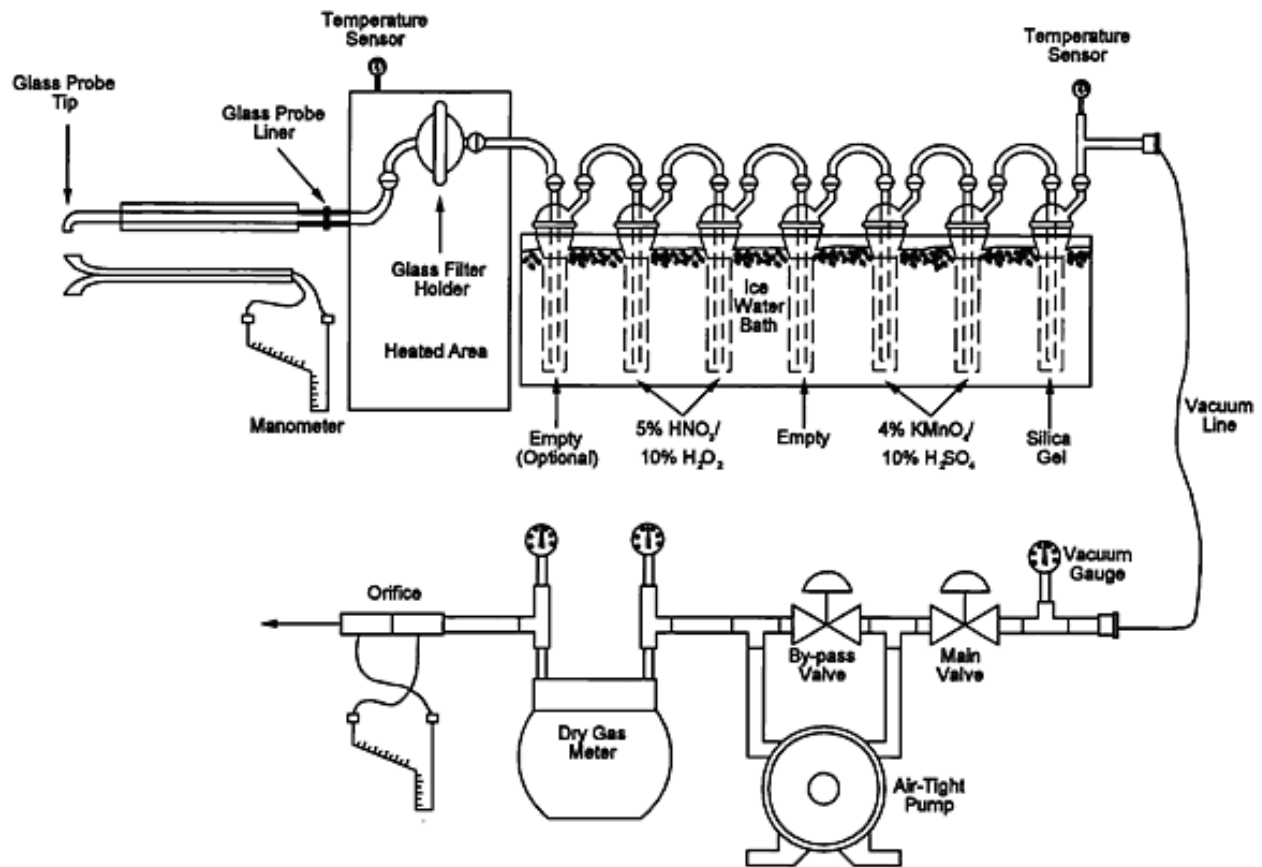


Figure 4.1-1

#### 4.1.1 Equipment Preparation Techniques

Following is a summary of the methods used to prepare the mercury stack emissions sampling train (i.e., test equipment).

##### 4.1.1.1 EPS Method 8/EPA 29 - Particulate, Hg

This train was a normal Method 29 train (Figure 4.1-1) except special (low metal) micro quartz glass filters were utilized and the impinger contents were:

- Impinger #1 100 ml 5% HNO<sub>3</sub> in 10% H<sub>2</sub>O<sub>2</sub>
- Impinger #2 100 ml 5% HNO<sub>3</sub> in 10% H<sub>2</sub>O<sub>2</sub>
- Impinger #3 Empty
- Impinger #4 100 ml 4% KMnO<sub>4</sub> in 10% H<sub>2</sub>SO<sub>4</sub>
- Impinger #5 100 ml 4% KMnO<sub>4</sub> in 10% H<sub>2</sub>SO<sub>4</sub>
- Impinger #6 100 ml distilled H<sub>2</sub>O
- Impinger #7 200 g silica gel

The train was operated isokinetically, sampling a total of 6 points on two 90° traverses for five minutes each, resulting in final sample volumes of about 1.5 Rm<sup>3</sup>. Data recordings were conducted at five-minute intervals. The train utilized a three-foot quartz probe and nozzle.

The methods used to prepare the glassware and filters are summarized below.

##### 4.1.1.2 Metal Train Glassware

1. Hot detergent wash with brushing
2. Rinse with 0.1 N HNO<sub>3</sub>
3. Copious rinsing with deionized H<sub>2</sub>O
4. Oven drying at 105°C

##### 4.1.1.3 Metal Train Filters

1. Overnight extraction with 1:1 nitric acid
2. Overnight rinsing with deionized H<sub>2</sub>O
3. Drying for 2 hrs @ 105°C, desiccation and weighing
4. Save 1 filter for blank

#### 4.1.2 Mercury Sample Recovery and Analysis

The particulate sample filters were removed from the cooled filter housing with teflon coated tweezers, with material retained on the gasket were recovered with a nylon brush and added to the filter. The filter was folded and placed in an identified plastic petri dish or envelope labelled "Container 1" with the date, time and the appropriate run number.

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Sequential rinses and brushings with acetone were used to clean up the probe and front half glassware from the mercury/particulate sample train. The resulting liquid was stored in Container 2.

Impingers 1 and 2 were measured for volume and transferred with about 100 ml of deionised water to a polyethylene sample container. Impingers 3, 4, and 5 were transferred to another polyethylene container using 100 ml potassium permanganate and water rinses. HCl rinses of the permanganate impingers were not conducted as visible deposits were effectively removed by the earlier rinsings.

Silica gel from the final impinger was transferred to its original container for final weighing. Blank filters and solutions for each component of the particulate mercury test were collected and labelled appropriately. A chain of custody was filled out and the samples were shipped to the analytical laboratory. All samples were labelled appropriately and placed in a cold room at 4°C until the analyses began.

### *4.1.2.1 Gravimetric Analysis*

At the A. Lanfranco and Associates Langley, BC laboratory, the sample filters were desiccated to a constant weight and weighed as per US EPA Method 5. The probe and front-half acetone rinsings were evaporated at about 70°C in tared, precleaned 250 ml glass beakers, with subsequent weighing to a constant weight. Blank filters and acetone were carried through the gravimetric process.

### *4.1.2.2 Mercury Analysis*

Following the gravimetric analysis, the filters and wash residues, along with the back half liquid samples were forwarded to Exova Laboratories in Surrey, BC for analysis of mercury. The samples and appropriate blanks were digested with acids and analyzed for mercury. Impingers 1, 2, 3, 4 and 5, for mercury, were analyzed at the Exova analytical laboratory using flameless atomic absorption.

## **4.1.3 Quality Assurance/Quality Control (QA/QC) Techniques**

The QA/QC component of this survey was designed to exceed the requirements normally instituted by the regulatory agency. Additionally, QA/QC of this survey was accomplished by the following mechanisms.

1. Pre and Post test leak checks
2. Calibration of volume measuring and monitoring instrumentation
3. Proofing of organic glassware and supplies (archived proofs)
4. Analysis of all blank solutions and materials
5. Spiking and recovery analysis of organic trains
6. Use of acid cleaned micro quartz filters
7. Duplicate analysis of selected samples
8. Reference material analysis with samples
9. Labelling and record-keeping
10. Surrogate spiking of dioxin trains using EPA protocols.

A "Blank" test was conducted, with that sampled analysed in the same manner as test samples.

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## **5. Results and Discussion**

## 5. Results and Discussion

Table 5.1-1 summarizes the Doris camp incinerator emissions testing results for mercury; the testing was done in triplicate to comply with the approved reference methods for source testing by Environment Canada – Environmental Protection Service (EC-EPS). Appendix F contains the report from the analytical laboratory for the mercury emissions from the incinerator. Appendix G summarizes the measured and calculated data collected during the stack emissions sampling.

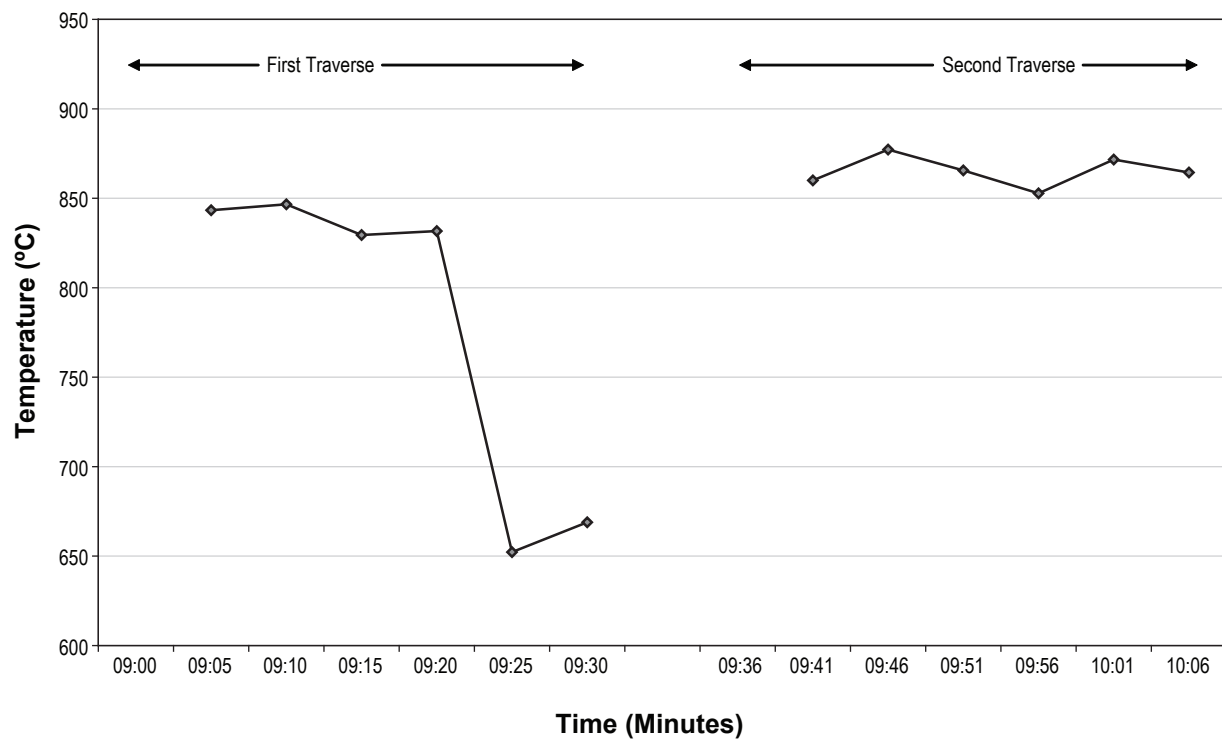
Figures 5.1-1 to 5.1-3 summarize the Doris camp incinerator stack gas temperatures during the three air emissions tests for mercury. Before the stack emissions testing began the manufacturer of the Doris camp incinerator (Westland Incinerators, Edmonton, AB) recommended that the temperature in the secondary chamber be maintained above 850°C. Generally the stack gas temperatures were at or above 850°C except for the last two sample points for run #1, traverse #1 when the stack gas temperature dropped to approximately 650°C.

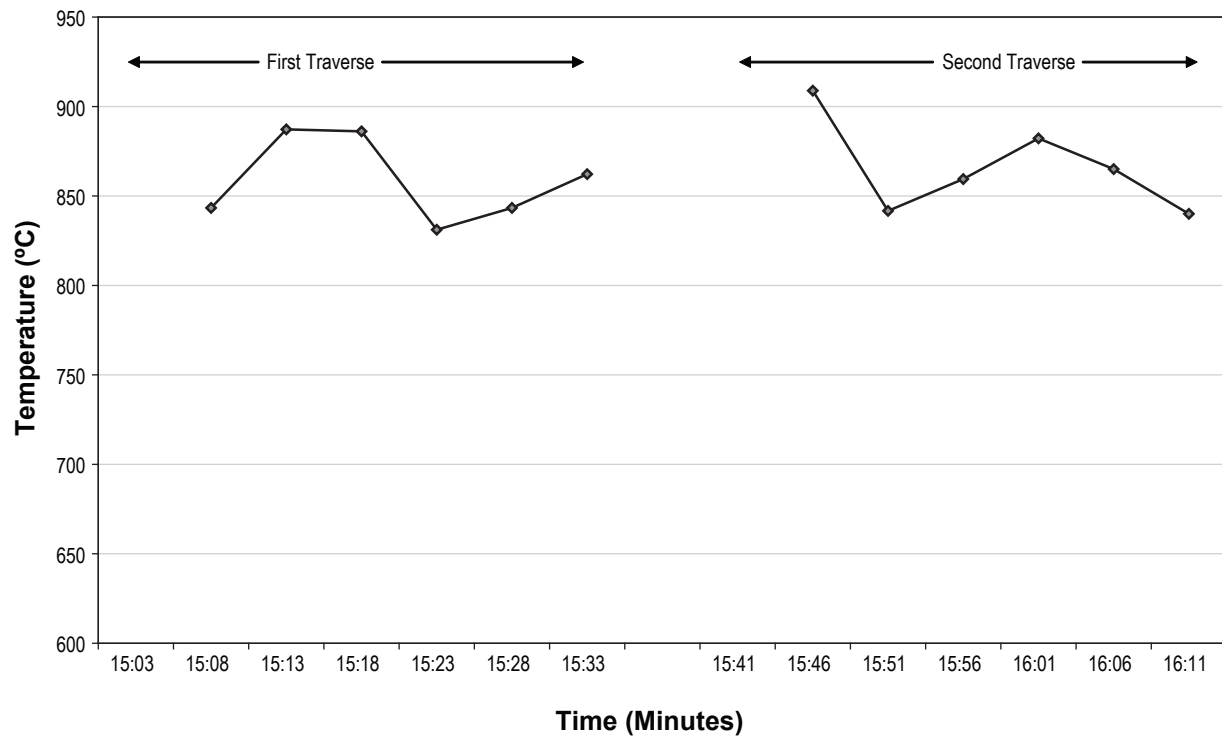
**Table 5.1-1. Doris Camp Incinerator Emissions Testing Results for Mercury and Particulate**

	Test 1	Test 2	Test 3	Average
Test Date	Sept. 30/09	Sept. 30/09	Oct. 1/09	
Test Time	09:00 - 10:06	15:03 - 16:11	14:08 - 15:13	
Duration (minutes)	60	60	60	60
<b>Parameter</b>				
Particulate (mg/Rm <sup>3</sup> )	4.3	6.9	26.8	12.7
Particulate (mg/Rm <sup>3</sup> @ 11% O <sub>2</sub> )	3.5	7.9	28.6	13.3
Particulate (kg/hr)	0.006	0.010	0.038	0.018
Particulate (kg/day)	0.1	0.2	0.9	0.4
<b>Mercury (ug/Rm<sup>3</sup> @ 11% O<sub>2</sub>)</b>	<b>0.2</b>	<b>0.5</b>	<b>2.3</b>	<b>1.0</b>
Flowrate (Rm <sup>3</sup> /min)	23.7	23.4	23.5	23.5
Flowrate (acm/min)	95.2	96.9	97.5	96.5
Average Stack Gas Temperature (°C)	822	863	876	853
O <sub>2</sub> (vol. % dry)	8.9	12.3	11.6	10.9
CO <sub>2</sub> (vol. % dry)	11.1	7.5	7.9	8.8
H <sub>2</sub> O (vol. %)	7.9	7.3	7.8	7.7
Isokinetic Variation (%)	103.9	99.8	102.0	102

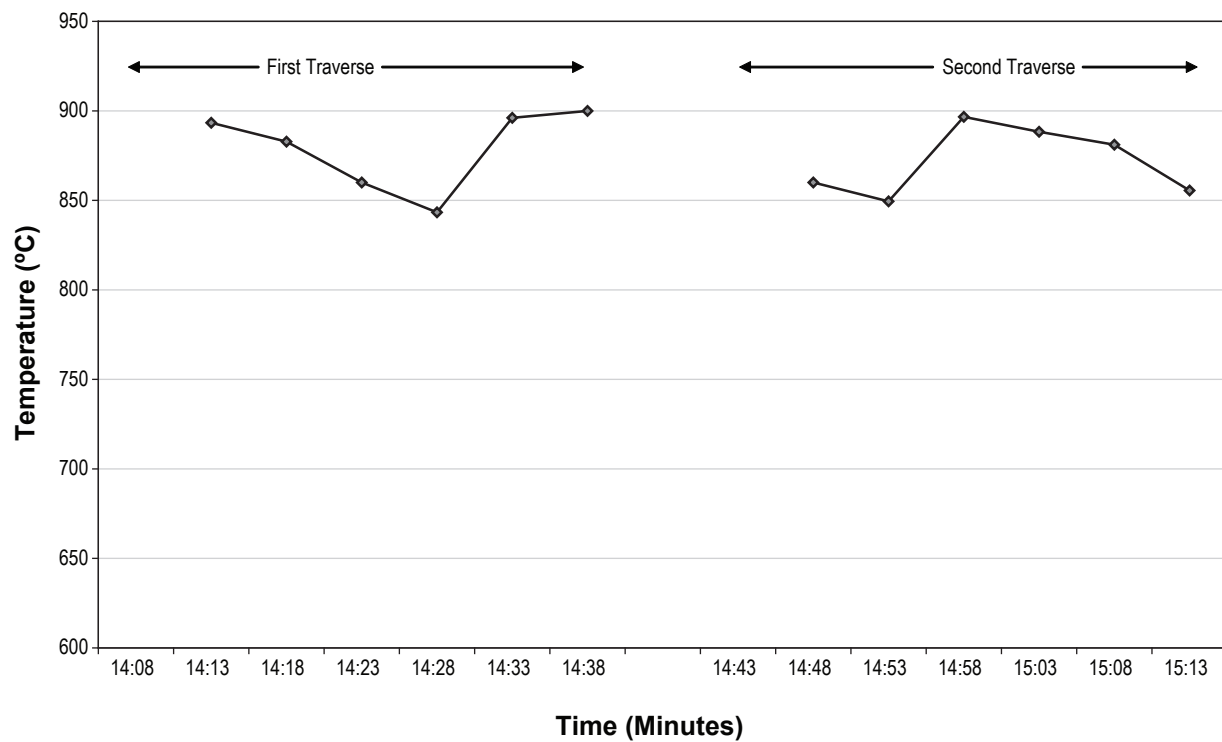
*Note: standard conditions are 25°C and 101.3 kPa.*

Table 5.1-2 summarizes the Doris camp incinerator emissions testing results for dioxin (PCDD) and furan (PCDF); the testing was done in triplicate to comply with the approved reference methods for source testing by Environment Canada – Environmental Protection Service (EC-EPS). Appendix H contains the report from the analytical laboratory for the dioxin and furan emissions from the incinerator.









**Table 5.1-2. Doris Camp Incinerator Emissions Testing Results for Dioxin and Furan**

Component	TEF	Test 1 (Sept. 29/09)		Test 2 (Sept. 30/09)		Test 3 (Oct. 1/09)	
		Analyzed (ng)	TEQ (ng)	Analyzed (ng)	TEQ (ng)	Analyzed (ng)	TEQ (ng)
2378 TCDD	1.0000	0.1720	0.1720	0.2000	0.2000	0.1000	0.1000
12378 PCDD	0.5000	1.6000	0.8000	3.3000	1.6500	1.5000	0.7500
123478 HxCDD	0.1000	0.8900	0.0890	2.5000	0.2500	1.1000	0.1100
123678 HxCDD	0.1000	1.1000	0.1100	4.3000	0.4300	2.0000	0.2000
123789 HxCDD	0.1000	1.2000	0.1200	3.4000	0.3400	1.5000	0.1500
1234678 HpCDD	0.0100	4.7000	0.0470	19.0000	0.1900	9.8000	0.0980
OCDD	0.0010	1.8000	0.0018	10.0000	0.0100	3.8000	0.0038
2378 TCDF	0.1000	1.5000	0.1500	1.8000	0.1800	0.8000	0.0800
12378 PCDF	0.0500	1.9000	0.0950	3.6000	0.1800	1.7000	0.0850
23478 PCDF	0.5000	4.2000	2.1000	10.0000	5.0000	4.4000	2.2000
123478 HxCDF	0.1000	4.9000	0.4900	9.8000	0.9800	5.0000	0.5000
123678 HxCDF	0.1000	3.6000	0.3600	7.1000	0.7100	3.6000	0.3600
234678 HxCDF	0.1000	6.6000	0.6600	14.0000	1.4000	6.5000	0.6500
123789 HxCDF	0.1000	1.8000	0.1800	4.8000	0.4800	2.1000	0.2100
1234678 HpCDF	0.0100	10.0000	0.1000	22.0000	0.2200	11.0000	0.1100
1234789 HpCDF	0.0100	1.8000	0.0180	3.5000	0.0350	2.2000	0.0220
OCDF	0.0010	3.7000	0.0037	8.6000	0.0086	5.8000	0.0058
Summed PCDD & PCDF TEQ (ng)			5.50		12.26		5.63
Sample Volume (Rm <sup>3</sup> )			3.63		4.06		3.96
PCDD & PCDF TEQ ng/Rm <sup>3</sup>			1.51		3.02		1.42
PCDD & PCDF TEQ ng/Rm <sup>3</sup> (@11% O <sub>2</sub> )			1.59		3.29		1.64
<b>PCDD &amp; PCDF TEQ pg/Rm<sup>3</sup> (@11% O<sub>2</sub>)</b>			<b>1,590</b>		<b>3,290</b>		<b>1,640</b>
PCDD & PCDF TEQ grams/day			0.000046		0.000098		0.000046
Particulate mg/dscm (@ 11% O <sub>2</sub> )			3.5		7.9		28.6
Flowrate (Rm <sup>3</sup> /min)			20.95		22.56		22.27
Oxygen (Vol. %)			11.5		11.8		12.3
Carbon Dioxide (Vol. %)			8.5		7.8		7.3
Carbon Monoxide (ppm)			0 to 600		6 to 290		10 to 150
Moisture (Vol. %)			5.9		7.4		7.3
Average Stack Gas Temperature (°C)			881.1		855.8		873.1
Isokinetic Variation (%)			97.5		100.9		100.0

Notes: PCDD = **Polychlorinated dibenzodioxins (PCDDs)**, or simply **dioxins**.

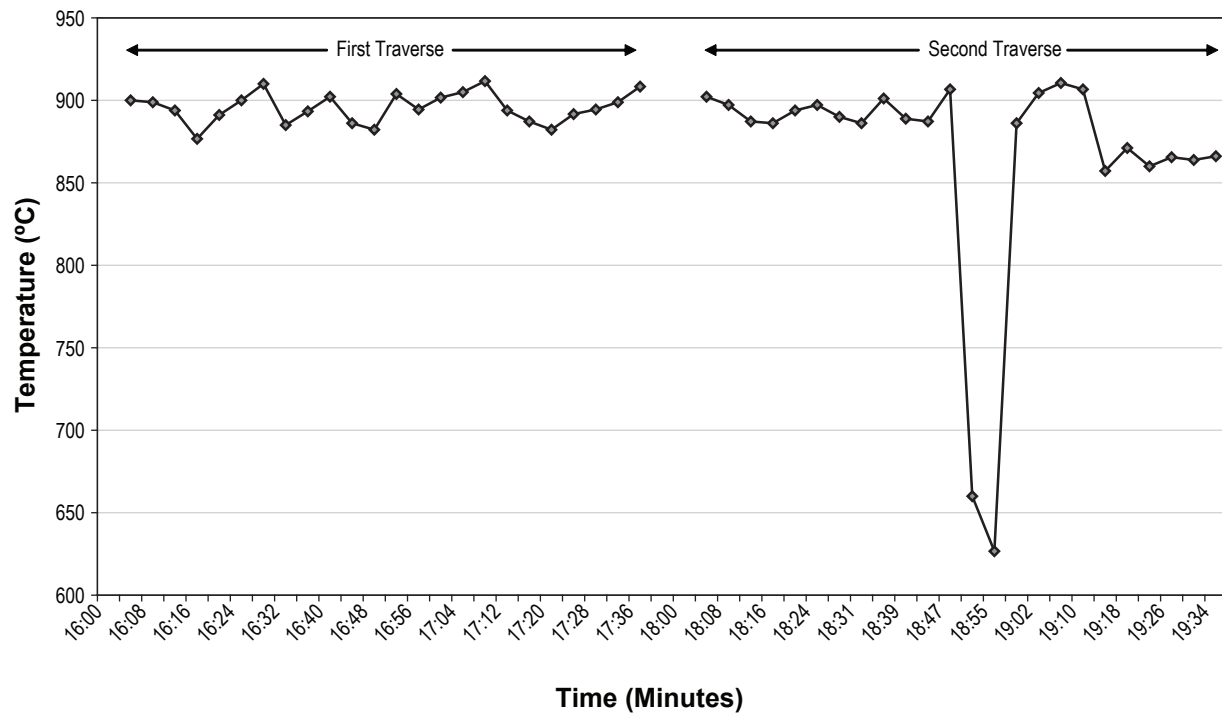
PCDF = **Polychlorinated dibenzofurans (PCDFs)**, or simply **furans**.

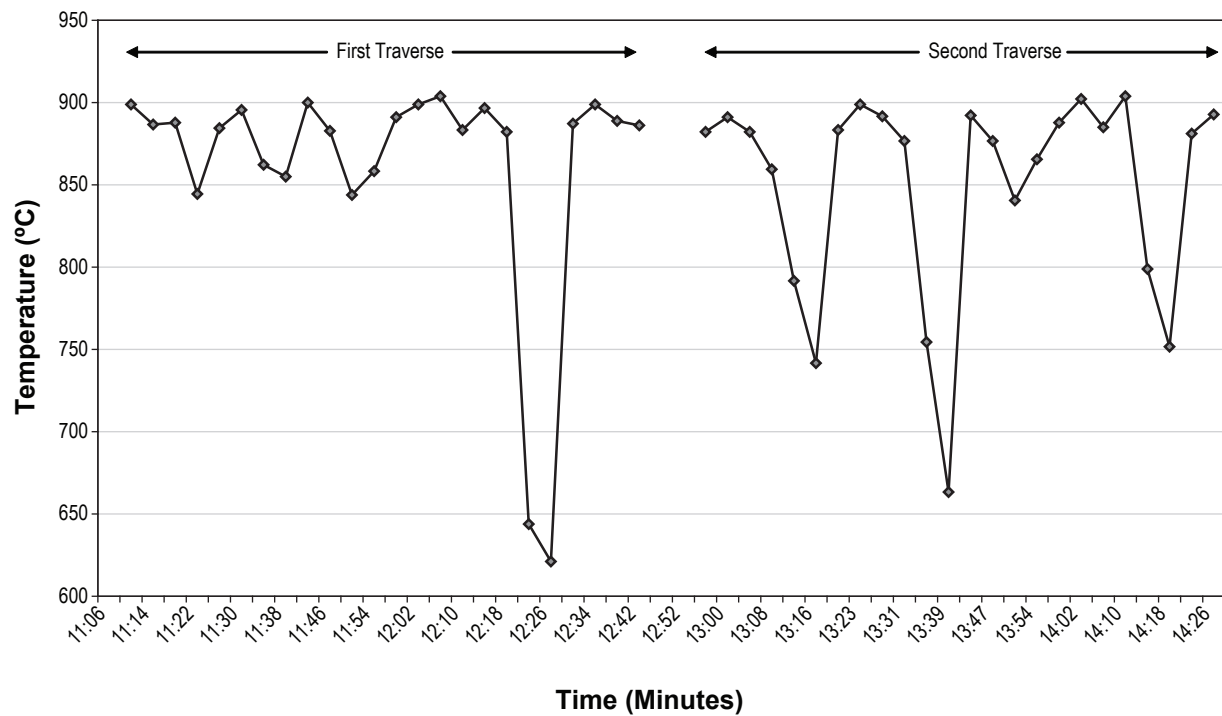
TEF = Toxic Equivalency Factor.

TEQ = The combined toxicity of all 29 dioxins is called the "TEQ" (for Toxic EQUIvalency).

dscm = dry standard cubic metre.

Figures 5.1-4 to 5.1-6 summarize the Doris camp incinerator stack gas temperatures during the three air emissions tests/runs for dioxin and furan. Before the stack emissions testing began the manufacturer of the Doris camp incinerator (Westland Incinerators, Edmonton, AB) recommended that the temperature in the secondary chamber be maintained above 850°C. Generally the stack gas temperatures were at or above 850°C for run #1, however during the second traverse the stack gas temperature dropped to approximately 650°C during two of the twenty four sample points. The stack gas temperatures for run #2 were more variable. During the first traverse there were 2 sample points with a stack gas temperature below 650°C. During the second traverse there were 6 sample points below 800°C. The stack gas temperatures for run #3 were all near 850°C or higher.





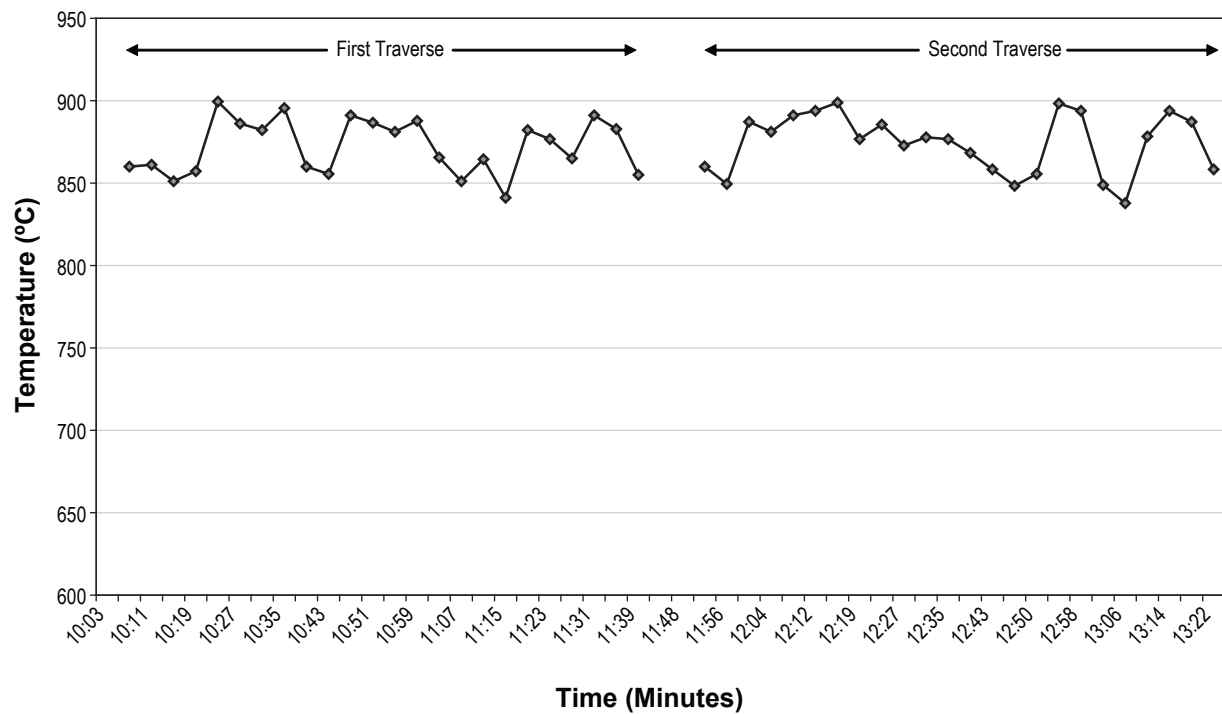


Table 5.1-3 summarizes the triplicate test averages for the dioxin/furan and mercury emissions from the Doris Camp incinerator stack on September 29 to October 1, 2009.

**Table 5.1-3. Summary of Doris Incinerator Stack Dioxin/Furan and Mercury Emissions**

Parameter and Units	Emission Rate	Canada Wide Standard
Dioxin/Furan (pg/Rm <sup>3</sup> TEQ @ 11% O <sub>2</sub> )	<b>2,170</b>	80
Mercury (ug/Rm <sup>3</sup> @ 11% O <sub>2</sub> )	1.0	20

*Note : Boldface results are exceeding the Canada Wide Standards*

Concentrations and flow rates are expressed at standard conditions of 25°C and 101.3 kPa (dry). Rm<sup>3</sup> = reference cubic metre. Most of the stack testing results were calculated using a "STACK" computer program developed for US EPA and Canadian requirements. Corrections to 11% O<sub>2</sub> were calculated by multiplying the determined stack concentrations by:

$$(20.9 - 11.0) / (20.9 - \text{measured O}_2)$$

The dioxin/furan results were recovery corrected according to surrogate recovery efficiencies determined for each organic analysis. The data from the mercury and dioxin/furan tests vary slightly for O<sub>2</sub>, temperature and flow rates due to the timing of each test.

The test results showed that the emissions from the Doris Camp incinerator were in compliance with the Canada Wide Standards (CWS) for mercury, but were not in compliance with the CWS for dioxin/furan. The average mercury emissions were 1.0 ug/Rm<sup>3</sup> @ 11% O<sub>2</sub>, compared to the CWS guideline of 20 ug/Rm<sup>3</sup> @ 11% O<sub>2</sub>. The average dioxin/furan emissions were 2,170 pg/Rm<sup>3</sup> @ 11% O<sub>2</sub>, compared to the CWS guideline of 80 pg/Rm<sup>3</sup> @ 11% O<sub>2</sub>. Mercury was found at levels significantly above method detection limits, and the results suggest that the waste feed mercury content is very low. It was noted that the temperature in the secondary chamber of the Doris incinerator for two of the three dioxin and furan tests/runs was not in the optimum range recommended by the manufacturer (i.e., > 850°C). During 2010 Hope Bay Mining Ltd. will be working with Environment Canada to develop more efficient waste management practices at the Doris North Gold Mine Project and intends to implement several best management practices (BMPs) to reduce the dioxin and furan emissions from the Doris incinerator.

Appendix I summarizes the quality assurance and quality control results (QA/QC) results for the Doris incinerator stack emissions testing program. The QA/QC program was successful in showing excellent analytical accuracy, in proving the avoidance of any significant sample contamination, and in maintaining leak free sampling procedures. The analysis of the dioxin/furan samples indicated complete recovery by virtue of excellent surrogate recoveries for the compounds of interest. The recovery of the five-labelled surrogates ranged from 71 to 118%. These recoveries comply with the US EPA Method 23 requirements of 70 to 130%. In addition, all data was recovery corrected for each congener. Recoveries of all internal standards ranged from 41 to 110% (except octa dioxin for Test 3) complying with EPS 1/RM/2 and EPA Method 23 requirements of 40 to 130% recovery.

There were no problems encountered in sample collection or analysis and validation of the field study is provided, in large part, by the analytical QA/QC program and the use of calibrated test equipment by qualified monitoring professionals. The test results, therefore, are reported with confidence and are considered an accurate representation of emission characteristics for the process conditions maintained on the test dates.

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## **References**

## References

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- Canadian Council of Ministers of the Environment, (CCME). 2001. Canada-Wide Standards for Dioxins and Furans, Canadian Council of Ministers of the Environment, Quebec City, QC.
- Canadian Council of Ministers of the Environment, (CCME). 2000. Canada-Wide Standards for Mercury Emissions, Canadian Council of Ministers of the Environment, Quebec City, QC.
- Environment Canada – Environmental Protection Service (EC-EPS). 1989. Reference Methods for Source Testing: Measurement of Releases of Selected Semi-volatile Organic Compounds from Stationary Sources. Reference Method EPS 1/RM/2. Environment Canada, Ottawa, ON.
- Environment Canada – Environmental Protection Service (EC-EPS). 1993. Reference Methods for Source Testing: Measurement of Releases of Particulate from Stationary Sources. Reference Method EPS 1/RM/8. Environment Canada, Ottawa, ON.



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## **Appendix A**

### **Doris Incinerator Operations Data**

Hope Bay Belt Project, Nunavut					
Hope Bay Mining Ltd. (Newmont)					
Doris incinerator stack emissions testing for dioxin/furan and mercury					
Project # 1009-004					
Incinerator process data collected during the stack emissions testing					
September 29th to October 1, 2009					
				Stack Gas	
				Temp	
		1st chamber	2nd chamber	at the sample	
		Temp	Temp	ports	
time	activity	(Celsius)	(Celsius)	(Celsius)	Notes
Tuesday September 29, 2009					
11:00	set-up	603	936	not available	no testing yet, just getting set up
13:00	charging	623	944	not available	2 bags added, 1 day, 1 wet
14:30	charging	596	932	not available	2 bags added, 1 day, 1 wet
15:55	charging	598	893	not available	2 bags added, 1 day, 1 wet
16:00	sampling				began first dioxin/furan test
16:14	monitoring	642	951	891	
17:10	charging	606	946	not available	2 bags added, 1 day, 1 wet
17:50	break				left site for supper at camp
18:10	monitoring	not available	not available	not available	for some reason the incinerator shut down, it came back on line at 18:50
19:00	sampling	not available	not available	not available	2 bags added, 1 day, 1 wet
19:13	monitoring	not available	not available	857	
19:21	monitoring	not available	not available	866	
19:32	sampling	not available	not available	not available	finished first dioxin/furan test
Wednesday September 30, 2009					
8:20	warm up	643	914	not available	operator had already warmed up the incinerator for approximately 1.4 hours
8:50	charging	590	871	not available	2 bags added, 1 day, 1 wet
9:00	sampling	not available	not available	804	began first mercury test
9:35	standby	not available	not available	not available	temporary shut down to change ports
9:38	sampling	not available	not available	860	began sampling again
10:25	sampling	not available	not available	not available	the first mercury test finished
10:45	misc	not available	not available	not available	called David Vokey on radio to see if the 2nd chamber should be shutting off for 1 minute every 5 to 8 minutes
10:57	charging	not available	not available	not available	2 bags added, 1 day, 1 wet
11:03	sampling	not available	not available	not available	began second dioxin/furan test
12:30	charging	not available	not available	not available	2 bags added, 1 day, 1 wet
12:43	sampling	not available	not available	not available	stopped testing temporarily to change sample ports
12:54	sampling	642	950	not available	began sampling again
14:00	charging	not available	not available	not available	2 bags added, 1 day, 1 wet
14:32	sampling	not available	not available	not available	finished second dioxin/furan test
14:53	charging	not available	not available	not available	2 bags added, 1 day, 1 wet
15:07	sampling	not available	910	not available	began second test for mercury
15:37	sampling	not available	not available	not available	shut down temporarily to change ports
16:14	sampling	not available	not available	not available	finished second test for mercury

<b>Thursday October 1, 2009</b>						
7:45	warm up	not available	not available	not available	operator began warming up incinerator	
9:50	charging	691	939	not available	2 bags added, 1 day, 1 wet	
10:03	sampling	not available	not available	not available	began third test for dioxin/furan	
10:40	sampling	611	901	854		
11:16	charging	617	897	886	2 bags added, 1 day, 1 wet	
11:40	sampling	not available	not available	not available	shut down temporarily to change ports	
11:47	sampling	669	910	868		
12:40	charging	652	919	868	2 bags added, 1 day, 1 wet	
13:23	sampling	not available	not available	not available	finished third test for dioxin/furan	
13:28	standby	630	925	865		
13:58	charging	not available	not available	not available	2 bags added, 1 day, 1 wet	
14:09	sampling	not available	not available	not available	began third test for mercury	
14:10	sampling	669	945	893		
14:38	sampling	not available	not available	not available	shut down temporarily to change ports	
14:47	sampling	600	928	875	began testing again	
15:13	sampling	not available	not available	not available	finished third test for mercury	
Notes:						
Low Set point for 1st chamber: 650 Celsius						
High Set point for 2nd chamber: 950 Celsius						

<b>Hope Bay Belt Project, Nunavut</b>									
<b>Hope Bay Mining Ltd. (Newmont)</b>									
<b>Doris incinerator stack emissions testing for dioxin/furan and mercury</b>									
<b>Project # 1009-004</b>									
<b>Incinerator Operating Guidelines from Westland Incinerators (Anil Chibber):</b>									
Hi Dan:									
I talked to Anil Chibber at Westland Incinerators this morning and discussed the requirements pre stack testing:									
1. Ensure the unit burner is working optimally (no apparent malfunctions).									
2. The incinerator operator should fire up the unit for <u>at least two (2) hours</u> before placing any garbage in it.									
3. Ensure that the temperature of the secondary chamber is > 850° C.									
4. Maximum of two (2) bags per load (when possible: one (1) wet + one (1) dry).									
5. Burn for 1.5 – 2.0 hrs before placing the next two (2) bags inside (ensure the temp is maintained > 850° C).									
6. Ensure no excessive volumes of plastics (including disposable razor cartridges).									
7. Ensure no aerosol cans.									
8. Recommends that the unit be monitored throughout the burn cycle to identify any potential drop in temperature.									
9. Recommends that a burner repair person be on standby should a problem with the unit be detected.									
With the above recommendations in place, Anil feels the unit should produce acceptable stack emission results.									
per email from Dave Vokey, September 24, 2009.									
<a href="mailto:David.Vokey@Newmont.com">David.Vokey@Newmont.com</a>									

[illegible]

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## **Appendix B**

### **Canada-wide Standards for Dioxins and Furans**

**Canadian Council of Ministers of the Environment**

***CANADA-WIDE STANDARDS***

***for***

***DIOXINS AND FURANS***

# CANADA-WIDE STANDARDS for Dioxins and Furans

## PREAMBLE

### Dioxins and Furans

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), commonly known as dioxins and furans, are toxic, persistent, bioaccumulative, and result predominantly from human activity. Due to their extraordinary environmental persistence and capacity to accumulate in biological tissues, dioxins and furans are slated for virtual elimination under the *Canadian Environmental Protection Act (CEPA)*, the federal *Toxic Substances Management Policy (TSMP)* and the *CCME Policy for the Management of Toxic Substances*.

The presence of dioxins and furans in the Canadian environment can be attributed to three principle sources: point source discharges (to water, air and soil), contamination from *in situ* dioxins and furans, and loadings from the long-range transportation of air pollutants (LRTAP).

LRTAP is the focus of multilateral conventions and bilateral accords at the international level.

Dioxin and furan contamination found in soil, water, sediments, and tissues (*in situ* contamination), is the subject of national guidelines for dioxins and furans. These guidelines outline ambient or “alert levels” which may be used by jurisdictions as benchmarks for the management and monitoring of dioxins and furans already present in the environment.

Point source discharges to water have been the target of aggressive federal and provincial regulation, as well as industry innovation and change. Discharges of dioxins & furans to the aquatic environment reached non-measurable levels in 1995.

### Development of the Canada-wide Standard

The Canada-wide Standards process has focussed on anthropogenic sources that are releasing dioxins and furans to the atmosphere and soil in a continuous process.

In January 1999, the Federal/Provincial Task Force on Dioxins and Furans released the *Dioxins and Furans and Hexachlorobenzene Inventory of Releases* which documented the current understanding of anthropogenic sources in Canada releasing dioxins and furans. The *Inventory of Releases* lists emissions from over 20 sectors by province and territory, and provides national summaries for each sector.

Initial efforts have focused on atmospheric releases, the most complete component of the Inventory. Six priority sectors, varying from regional to national in scope, accounting for about 80% of national emissions have been identified as priorities for early action. These are waste incineration (municipal solid waste, hazardous waste, sewage sludge and medical waste); burning salt laden wood in coastal pulp and paper boilers in British Columbia; residential wood combustion; iron sintering; electric arc furnace steel manufacturing; and conical municipal waste combustion in Newfoundland.



To date, CWSs have been developed for the coastal pulp and paper boiler and the incineration sectors. CWSs for the remaining priority sectors will be completed in 2001. Additional source sectors, many of which contribute very small amounts of dioxins and furans emissions, will also be addressed in 2001 as will releases to soil.

Development of CWSs for dioxins and furans has taken into consideration environmental benefits, available technologies, socio-economic impacts, opportunities for pollution prevention and collateral benefits from reductions in other pollutants.

In recognition of the ultimate goal of virtual elimination, pollution prevention is being encouraged as the preferred method for avoiding the creation of dioxins or reducing releases to the environment.

Wherever possible, work on the dioxins and furans CWSs has been coordinated with other ongoing processes (e.g. Mercury CWS and the Strategic Options Process). A multi-pollutant approach will be carried forward to the remaining sectors while ensuring that dioxins and furans issues are addressed and that the ultimate goal of virtual elimination is kept clearly in mind. Opportunities for a multi-pollutant approach will also be pursued as part of the implementation of the Dioxins and Furans Canada-wide Standard.

During development of the inventory, it was realized that the data on dioxins and furans is limited. The information in the dioxins and furans inventory will be refined and updated on a regular basis through a variety of sources including the National Pollutants Release Inventory (NPRI) as a means of tracking progress and as a means of identifying any future sources of releases that must be addressed.

## **PART 1:**

### **Pulp and Paper Boilers Burning Salt Laden Wood**

#### Rationale for standard

Unique to British Columbia, the burning of salt laden wood results in an annual release of 8.6 gTEQ/year to the atmosphere or 4.3 percent of the national total of dioxin and furans emissions documented in the inventory of releases prepared under the Canadian Environmental Protection Act.

As a result of mill closures and voluntary industry initiatives that have reduced atmospheric releases, the current total represents a 25% reduction from 1990 releases.

Dioxins and furans emitted from coastal pulp and paper mills are created through the burning of salt contaminated hogged fuel. Logs transported and stored in salt water take up chlorine into the bark. The bark is stripped from the logs and ground up to produce hogged fuel.

This material is then used as boiler fuel to produce heat and electrical energy for the pulp and paper process. Over 1.4 million oven dried tonnes of hogged fuel were used by the coastal pulp and paper industry in B.C. in 1998.

#### Nature and application:

The CWS for this sector consists of two components. The first component sets out numeric targets and timeframes for reducing emissions from new and existing boilers. This standard applies to boilers burning more than 10,000 oven dry metric tonnes per year of hogged fuel generated from wood transported or stored in salt water. All boilers currently reliant on hogged fuel generated from wood transported or stored in salt water currently consume in excess of 50,000 oven dry metric tonnes annually. As part of the implementation of this standard, procedures will be put in place to report on the salt content of the hogged fuel to ensure compliance with the standard.

The second component sets out a process for further examining pollution prevention opportunities to prevent the creation of dioxins and furans.

#### Numeric Target and Timeframe for Achieving Target

Dioxin and furan emissions will be less than 100 pg/m<sup>3</sup> TEQ for new boilers constructed after the effective date of this standard.

Dioxin and furan emissions will be less than 500 pg/m<sup>3</sup> TEQ for all existing boilers by 2006. “New” means a total replacement including firebox, heat transfer surfaces and air emission control equipment.

The standard for existing boilers is set pending the acquisition of further test data and controlled studies of boiler operation. Recognizing the ultimate objective of virtual elimination as set out in the Canadian Environmental Protection Act, the mill operators will voluntarily pursue further reductions in emissions during the period of the standard. In doing so the operators will conduct additional studies to identify the reasons for higher dioxin and furan emissions at some locations and explore and, as practicable, implement measures to achieve virtual elimination at all locations. Measures to be explored include physical and process modifications to prevent or reduce dioxin and furan formation as well as emission control upgrades and/or other pollution prevention measures.

Every boiler covered by this standard will be tested twice per year to determine the level of dioxin and furan air emissions for the years prior to 2003 and annually for the years 2003 and thereafter. Testing and reporting will be performed using methods and procedures acceptable to the responsible provincial ministry.

The standard for existing boilers will be reviewed in 2003 based on the results of the additional testing, the additional studies on dioxin and furan creation and opportunities to achieve virtual elimination and the examination of other pollution prevention opportunities.

#### Pollution Prevention Strategy

In addition to the continuing efforts of pulp and paper mill operators to capture emissions of dioxin and furans, emphasis will be placed on identifying and implementing opportunities to prevent the creation of dioxins and furans. A strategy identifying opportunities to eliminate the formation of dioxins and furans by the coastal pulp and paper industry will be developed through a multi-stakeholder process by December 31, 2001 to provide a framework for continual progress towards the elimination of dioxin and furans.

Recognizing that most opportunities for avoiding the creation of dioxins and furans fall beyond the exclusive influence of the coastal pulp and paper mill operators, preparation of this strategy must engage a wide range of stakeholders.

The range of issues to be addressed in developing the strategy could include:

- maximum allowable salt content for hogged fuel
- removal of chloride from logs
- hogged fuel washing and pressing
- options for blending hogged fuel of different salt levels
- alternatives to log handling, transportation and storage practices that rely on salt water
- impacts of the length of time entailed in transportation and storage on the salt content of hogged fuel
- inclusion of transportation modes and effects in eco-certification criteria
- in-plant opportunities to avoid creation of dioxins and furans
- alternative fuel opportunities and costs
- providing greater opportunities for market intervention by improving the understanding of the costs being imposed on the pulp and paper mills by current log handling and storage practices

## Waste Incineration

### Rationale for standard

Waste incineration has historically been responsible for a significant portion of the dioxins and furans emitted in Canada. The total release of dioxins and furans from this sector amounts to 44.9 g/ TEQ/y or 22.5% of the total releases to the atmosphere.

Improved exhaust gas controls to reduce emissions of acid gases and fine particulates or activated carbon injection systems have decreased emissions of both mercury and dioxins and furans from the municipal solid waste (MSW) sector. Dioxins and furans emissions from this sector are estimated to be approximately 8.4 g/yr. Many medical waste incinerators have closed for economic or environmental reasons. However, a range of medium-to small-sized facilities remain. Individually these are small sources, but as a sector they are significant, emitting an estimated 28.8 g/yr. Two additional incineration sectors, hazardous waste (7.6 g/yr) and sewage sludge (0.1 g/yr), are also addressed by the CWS.

A Canada-wide Standard for incineration of MSW in conical waste combusters in Newfoundland will be brought forward in 2001. Newfoundland has committed to reviewing the use of these facilities and to considering a phase-out strategy that will reduce emissions of dioxins and furans as well as mercury. These actions are also identified in the Mercury Canada-wide Standard accepted by the Council of Ministers in November 1999.

Actions to reduce national emissions require that any new facilities meet stringent limits, and that the bulk of the emissions from existing facilities be controlled through retrofits with control technology that is efficient at destroying dioxins and furans. Diverting waste from incinerators would result in less incineration overall and thus avoid creation of dioxins and furans. All facilities, and particularly smaller ones, may find that pollution prevention, waste segregation and diversion are options for either achieving the limit, or reducing “end-of-stack” expenditures, and during implementation all facilities should be encouraged to place a priority on reduced inputs rather than controlled releases.

### Definitions:

*Waste incinerator*: a device, mechanism or structure constructed primarily to thermally treat (e.g., combust or pyrolyze) a waste for the purpose of reducing its volume, destroying a hazardous chemical present in the waste, or destroying pathogens present in the waste. This includes facilities where waste heat is recovered as a byproduct from the exhaust gases from an incinerator, but does not include industrial processes where fuel derived from waste is fired as an energy source as a matter incidental to the manufacture of the primary product. For the purpose of the Dioxins and Furans CWS, conical waste combusters are considered separately from other incineration sectors.

*Municipal solid waste:* any waste which might normally be disposed of in a non-secure landfill site if not incinerated (i.e., including non-hazardous solid wastes regardless of origin), but is not intended to include “clean” wood waste. Clean wood waste means waste from woodworking or forest product operations where the wood waste has not been treated with preservative chemicals (e.g., pentachlorophenol) or decorative coatings.

*Medical waste:* any waste which includes as a component any Biomedical Waste as defined in the February 1992 CCME Guidelines for the Management of Biomedical Waste in Canada, with the exception that animal wastes derived from animal health care or veterinary research and teaching establishments are excluded.

*Determined efforts:* Determined efforts include the ongoing review of opportunities for reductions and implementation of in-plant changes and/or emissions control upgrades that are technically and economically feasible and which confer on-going reductions in emissions. Where possible, dioxin and furan emission reductions will be determined by way of a one-time stack test conducted after implementation of the measures. Where testing is not possible or will not provide reliable results, an audit of the dioxin and furan emission reductions associated with waste diversion or other measures is an acceptable alternative. Opportunities for regional consolidation and/or phase-out of smaller facilities may also be considered.

#### Nature and application:

Emission limits are expressed as a concentration in the exhaust gas exiting the stack of the facility. New or expanding facilities will be expected to comply immediately with the standard, and it will be up to individual jurisdictions to determine what constitutes a significant expansion to trigger the standard. The limits for existing facilities are capable of being met using generally available technology or waste diversion. Larger facilities will be subject to stack testing as described in Annex 1 to verify compliance with the limit. Smaller medical and municipal facilities will have the option of reporting on an audit of the dioxin and furan emission reductions associated with waste diversion or other measures or conducting a one-time stack test, to illustrate progress towards the standard.

#### Numeric targets:

The following standards are a step towards achieving virtual elimination for dioxins and furans.

For new or expanding facilities of any size, application of best available pollution prevention and control techniques, such as a waste diversion program, to achieve a maximum concentration<sup>1</sup> in the exhaust gases from the facility as follows:

Municipal waste incineration	80pg I-TEQ/m <sup>3</sup>
Medical waste incineration	80pg I-TEQ/m <sup>3</sup>
Hazardous waste incineration <sup>2</sup>	80pg I-TEQ/m <sup>3</sup>
Sewage sludge incineration	80pg I-TEQ/m <sup>3</sup>

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<sup>1</sup> Stack concentrations of dioxins and furans will be corrected to 11% oxygen content for reporting purposes.

<sup>2</sup> Hazardous waste incinerators include all facilities that burn hazardous waste including low level radioactive waste; however they do not include facilities that use waste derived fuel or used oil.

For existing facilities application of best available pollution prevention and control techniques, to achieve a maximum concentration<sup>1</sup> in the exhaust gases from the facility as follows:

Municipal waste incineration	
> 26 Tonnes/year <sup>3</sup>	80pg I-TEQ/m <sup>3</sup>
< 26 Tonnes/year <sup>4</sup>	80pg I-TEQ/m <sup>3</sup>
Medical waste incineration	
> 26 Tonnes/year <sup>3</sup>	80pg I-TEQ/m <sup>3</sup>
< 26 Tonnes/year <sup>4</sup>	80pg I-TEQ/m <sup>3</sup>
Hazardous waste incineration <sup>2</sup>	80 pg I-TEQ/m <sup>3</sup>
Sewage sludge incineration	100 pg I-TEQ/m <sup>3</sup>

Timeframe for achieving the targets:

Any new or expanding facility will be required to design for and achieve compliance immediately upon attaining normal full scale operation, compliance to be confirmed by annual stack testing..

Based on determined efforts in working towards virtual elimination, existing facilities will be required to meet the standards on the following schedule:

Municipal waste incineration	2006
Medical waste incineration	2006
Hazardous waste incineration	2006
Sewage sludge incineration	2005

Pollution Prevention Strategy:

In addition to the continuing efforts of waste incinerator operators to destroy or capture emissions of dioxin and furans, emphasis will be placed on identifying and implementing opportunities to prevent the creation of dioxins and furans as well as emissions of air pollutants and ash quality generally. As an initial action with shared responsibility by all jurisdictions, strategies identifying opportunities to minimize waste incineration emissions of air pollutants including dioxins and furans will be developed through a multi-stakeholder process by December 31, 2001 to provide a framework for continual progress towards the elimination of dioxin and furans.

Recognizing that many opportunities for minimizing air pollutant and ash emissions and specifically avoiding the creation of dioxins and furans fall beyond the exclusive influence of the operators of waste incinerators, preparation of this strategy must engage a wide range of stakeholders.

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<sup>3</sup> Larger facilities must achieve this stack concentration as confirmed by annual testing.

<sup>4</sup> Smaller facilities must make determined efforts to achieve this stack concentration.

The range of issues to be addressed in developing the strategy could include:

- waste diversion initiatives to minimize the generation of wastes destined for disposal (waste reduction, material reuse options)
- waste segregation initiatives aimed at materials with greater potential to generate emissions of dioxins and furans or other air pollutants of concern (e.g., mercury, other heavy metals) and aimed at diverting those wastes to recycling or other non-incineration disposal options
- combustion control strategies to optimize performance of existing combustors at destroying pollutants of concern
- use of alternative disposal or treatment technologies (e.g., anaerobic digestion of wastes with material recovery and combustion of biogas)

## **PART 2:**

### Reporting on Progress:

Ministers will receive reports on progress in achieving the CWS by jurisdictions in Spring 2004 and Spring 2008. Ministers will ensure that a single public report is prepared and posted on the CCME web site for public access. The report in 2004 will reflect interim progress on achieving the CWSs. Progress on both implementation of the numeric targets and the activities applied as part of the determined efforts provisions for smaller medical waste and municipal solid waste facilities will be documented. The 2008 report will evaluate whether targets have been met and the effectiveness of the determined efforts with respect to smaller facilities. More details on reporting are available in Annex 1.

Each jurisdiction will detail the means of ensuring achievement of the CWS in a manner consistent with the typical or desired programs for the affected facility/sector, so as not to impose an unnecessary level of reporting duplication.

With a view to continuous improvement towards the goal of virtual elimination, an evaluation of the Dioxin and Furan Canada-wide Standards will be presented to Ministers in Spring 2006. The evaluation will consider new scientific, technical and economic information and provide an assessment of the need to develop the next set of CWS targets and timelines to continue progress toward virtual elimination.

### **ADMINISTRATION:**

Jurisdictions will review and renew Part 2 and Annex 1 five years from coming into effect.

Any party may withdraw from these Canada-Wide Standards upon three month's notice.

These Canada-Wide Standards comes into effect on May 1, 2001.

# **Annex 1**

## **Dioxins and Furans CWS Reporting Framework**

### **Introduction**

Under the Harmonization Accord and its Canada-wide Environmental Standards Sub-Agreement, all jurisdictions are to report to the public and to Ministers on their progress towards achieving the CWSs for dioxins and furans.

This reporting framework is intended to provide a transparent and consistent mechanism for reporting by jurisdictions in a fashion which minimizes resource requirements for government and industry alike, while maximizing the availability of information on achievement of these standards.

The framework addresses:

- 1) frequency, timing and scope of reporting
- 2) guidance as to the means of determining compliance/achievement of the CWS
- 3) common measurement parameters for reporting purposes
- 4) data management and public reporting

### **Frequency, timing and scope of reporting**

The reporting schedule will be tied into assessing the performance of the governments in meeting the benchmarks and timelines relevant to the standards. A report in 2004 will provide a means for tracking interim progress and report on additional technical studies (e.g. technology feasibility and pollution prevention options for the coastal pulp & paper sector). The 2008 report will indicate compliance with the standards for the coastal pulp and paper boiler and incineration sectors.

Jurisdictions will submit sectoral data for inclusion in the progress reports in a timely manner. To report on achievement of the CWS, a data report along with an assessment of progress will be compiled into a single report for Ministers and a public version will be posted on the CCME web site for public access.

Reports will be limited to information on those facilities which are subject to achievement and/or compliance with the Canada-wide Standards as endorsed by the Ministers of the Environment May 1, 2001 and as implemented variously by the responsible jurisdictions or industries. This information is intended to show compliance rates and performance characteristics in a manner which documents sectoral performance as well as jurisdictional performance. It is not intended to provide a facility-by-facility record of performance.



## **Means of determining compliance/achievement of the CWS**

The Canada-wide Standards for dioxins and furans lend themselves to achievement through voluntary action, or through compliance with regulated or legally enforceable limits. As such, it is necessary to provide some means to ensure that a level playing field exists so that the numeric value provided in the CWS is applied equally or similarly in each jurisdiction. One means to do this is to require identical compliance procedures, but this may require that some jurisdictions apply compliance procedures for dioxins and furans CWSs that are different than those used for locally determined or regulated parameters such as SO<sub>2</sub>, PM, ammonia, etc. An example is where the dioxins and furans CWS is expressed as the average of 3 stack tests, whereas a jurisdiction may normally utilize the median value of 3 tests to determine compliance.

In an effort to streamline implementation, each jurisdiction will determine the exact means of ensuring compliance/achievement in a manner consistent with the typical or desired programs for the affected facility/sector. It is anticipated that minor variations in jurisdictional requirements will result in minimal variation across the country which is insignificant with respect to the overall reduction activities which range from 50-99% for various facilities.

## **Common measurement parameters for reporting purposes**

Each facility report will include specific measures corrected so as to be compatible and consistent for the purposes of public reporting. Dioxin and furan emissions must be corrected for the O<sub>2</sub> content of gases, to ensure compliance with the standards.

Each jurisdiction will determine the sector within which each subject facility will be reported. For example, a jurisdiction may determine that a small mixed waste incinerator (for example, burning both medical and municipal waste) may be subject to either standard, based upon the preponderance of waste (>50% as one type) or based upon the provincial designation of facility type. Sectoral assignments will be updated to reflect the most recent characteristics of the facility under consideration prior to reporting.

While little confusion is likely to exist over the implementation of dioxins and furans CWSs for “greenfield” facilities, it is possible that significantly expanded or modified facilities can/should be considered as new for the purposes of achievement/compliance with the dioxins and furans CWS. It will be the responsibility of the jurisdictions to determine at which point a facility no longer qualifies as an “existing” facility and must conform to the standard for “new or expanded” facilities as a result of significant modifications/alterations to the facility operations or physical plant.

Jurisdictions must report measurements that are below the detection limit in a consistent manner. These measurements should be reported as the limit of detection.

Large facilities will generally be required to perform stack tests at an annual frequency in order to demonstrate compliance. However, jurisdictions may vary the stack testing requirements for these facilities in cases where performance has been consistently demonstrated to be below the Level of Quantification (LoQ) as defined by Environment Canada. Where five years' data has been accumulated with all results reported below the LoQ, the stack testing frequency may be revised to a biennial schedule so long as all subsequent test results remain below the LoQ. For the purpose of reporting emissions, the most recent stack test results available should be used. Jurisdictions have the responsibility of deciding whether to implement this variance for all, some or none of the source types subject to these standards.

### **Data management and public reporting**

Reports on achieving the CWSs will include a data report and a report on achievement of the standards. Sectoral and jurisdictional specific data will be supplied in a spreadsheet format to facilitate reporting. A consolidated report will be made available to all jurisdictions and to the Ministers, along with the draft public report, prior to formal release of the public report. The public report will be released upon approval by the Council of Ministers.

Jurisdictions will provide a report in spreadsheet format so that the data report and report on achievement can be prepared along with the public report for review and approval. Reports will be prepared and distributed to all jurisdictions prior to review by Ministers. Along with the report on achievement, a draft public report will be provided for review and consideration prior to the Ministers' meeting at which public release is anticipated. That public report will be posted to the CCME web site upon approval by the Ministers. Jurisdictions are encouraged to provide reference to the CCME web site and/or pointers in their own web sites in order to ensure a single location for dioxins and furans CWSs reporting should errors/miscalculations have to be corrected at some time.

In addition to the consolidated public reporting on dioxins and furans CWSs, jurisdictions must provide a contact for facility-specific information in the advent that the public wishes to access compliance or achievement information. Such data will be supplied in a manner consistent with the normal data-reporting/compliance reporting procedures of the jurisdiction in question - the consolidated spreadsheet will not be made publicly available in that it may include proprietary (business) information.

**Canada-wide Standards for Dioxins and Furans  
Emissions from Waste Incinerators and  
Coastal Pulp and Paper Boilers**

Signed by:

British Columbia	Honourable Ian Waddell
Alberta	Honourable Lorne Taylor
Saskatchewan	Honourable Buckley Belanger
Manitoba	Honourable Oscar Lathlin
Ontario	Honourable Elizabeth Witmer
Environment Canada	Honourable David Anderson
New Brunswick	Honourable Kim Jardine
Nova Scotia	Honourable David Morse
Prince Edward Island	Honourable Chester Gillan
Newfoundland and Labrador	Honourable Ralph Wiseman Honourable Tom Lush
Yukon	Honourable Dale Eftoda
Northwest Territories	Honourable Joseph Handley
Nunavut	Honourable Olayuk Akesuk

Note: Québec has not endorsed the Canada-wide Accord on Environmental Harmonization or the Canada-wide Environmental Standards Sub-agreement.

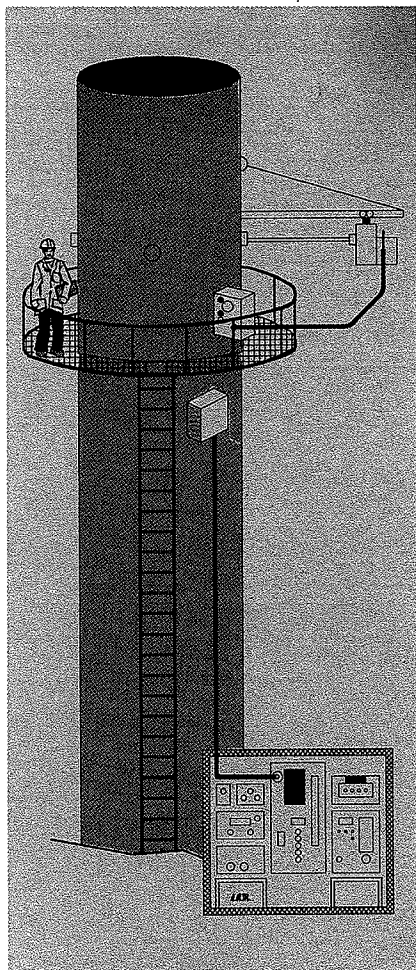
**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix C**

Environment Canada Emissions Monitoring Reference  
Methods

# Environmental Protection Series



## Reference Methods for Source Testing: Measurement of Releases of Particulate from Stationary Sources

Reference Method EPS 1/RM/8  
December 1993

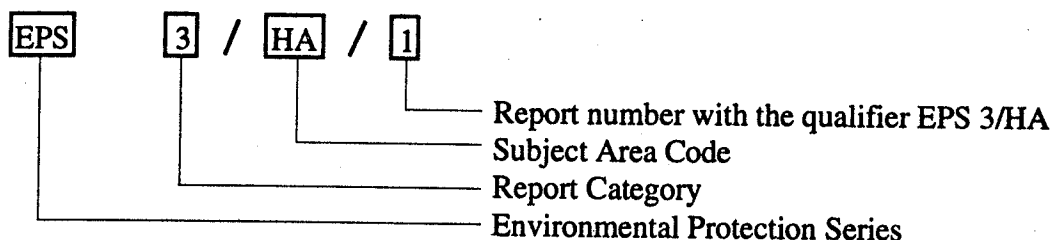
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Environment Canada  
Environnement Canada

# ENVIRONMENTAL PROTECTION SERIES

Sample Number:



## Categories

- 1 Regulations/Guidelines/Codes of Practice
- 2 Problem Assessments and Control Options
- 3 Research and Technology Development
- 4 Literature Reviews
- 5 Surveys
- 6 Social, Economic and Environmental Impact Assessments
- 7 Surveillance
- 8 Policy Proposals and Statements
- 9 Manuals

## Subject Areas

- |     |                                    |
|-----|------------------------------------|
| AG  | Agriculture                        |
| AN  | Anaerobic Technology               |
| AP  | Airborne Pollutants                |
| AT  | Aquatic Toxicity                   |
| CC  | Commercial Chemicals               |
| CE  | Consumers and the Environment      |
| CI  | Chemical Industries                |
| FA  | Federal Activities                 |
| FP  | Food Processing                    |
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| IC  | Inorganic Chemicals                |
| MA  | Marine Pollutants                  |
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| PG  | Power Generation                   |
| PN  | Petroleum and Natural Gas          |
| RA  | Refrigeration and Air Conditioning |
| RM  | Reference Methods                  |
| SF  | Surface Finishing                  |
| SP  | Oil and Chemical Spills            |
| SRM | Standard Reference Methods         |
| TS  | Transportation Systems             |
| TX  | Textiles                           |
| UP  | Urban Pollution                    |
| WP  | Wood Protection/Preservation       |

New subject areas and codes are introduced as they become necessary. A list of EPS reports may be obtained from Environmental Protection Publications, Conservation and Protection, Environment Canada, Ottawa, Ontario, Canada, K1A 0H3.

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# **Reference Method for Source Testing: Measurement of Releases of Particulate from Stationary Sources**

Environmental Technology Centre  
Technology Development Directorate  
Environmental Protection Service  
Environment Canada

Reference Method EPS 1/RM/8  
December 1993

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## Readers' Comments

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Inquiries pertaining to the use of this reference method should be directed to:

Environmental Technology Centre  
Technology Development Directorate  
Environment Canada  
Ottawa, Ontario  
K1A 0H3

Additional copies of this report may be obtained from:

Environmental Protection Publications  
Technology Development Directorate  
Environmental Protection Service  
Environment Canada  
Ottawa, Ontario  
K1A 0H3

## Foreword

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*The six test methods (A to F) presented in this report may be used either independently to determine certain characteristics of a gas stream (e.g., gas velocity, volumetric flow rate and moisture content), or in various combinations to determine particulate emissions. The combination of test methods to be used for a specific source will be given by Environment Canada or prescribed in guidelines and regulations applicable to the industry. The complexity of these methods warrants that personnel performing them be trained and experienced.*

*Note: Mention of trade names or commercial products does not constitute endorsement for use by Environment Canada.*

*This Reference Method EPS 1/RM/8 is a revised version of "Standard Reference Methods for Source Testing: Measurement of Emissions of Particulates from Stationary Sources," Report EPS 1-AP-74-1 February 1974.*

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## Section 1

# Method A: Determination of Sampling Site and Traverse Points

## 1.1 Applicability

This method is applicable to flowing gas streams in stacks or ducts. The method is not applicable as written when one or more of the following conditions exist:

- cyclonic flow (see Section 1.6 for confirmation of cyclonic flow);
- reverse flow (see Section 1.6 for confirmation of reverse flow);
- stack or duct diameters less than 0.30 m (1.0 ft);
- non-circular or non-rectangular stacks or ducts; and
- sampling site less than two stack or duct diameters downstream from a flow disturbance, or less than 0.5 stack or duct diameter upstream from a flow disturbance.

## 1.2 Principle

A sampling site and the number and location of traverse or sampling points are determined to optimize the extraction of a representative sample.

## 1.3 Location of Sampling Site

Select a site in a straight section of stack or duct located at least eight stack or duct

diameters downstream and two stack or duct diameters upstream of any flow disturbance such as a bend, expansion, contraction, visible flame, junction, or stack exit. In the case of rectangular stacks or ducts, an equivalent diameter ( $D_e$ ) shall be used in determining the downstream and upstream distances.

$$D_e = \frac{2 LW}{(L+W)}$$

where: L represents length, and  
W represents width.

In circular stacks or ducts, at least two sampling ports with a 90° separation are required. For particulate traverses, one diameter should be in the plane of an upstream flow disturbance.

For rectangular flow areas, ports are located on the most accessible face of the duct or flue. The number of ports will be determined by the total number of traverse points.

## 1.4 Determination of Traverse Points

When the sampling site is located at least eight diameters downstream and two diameters upstream from a flow disturbance, the required minimum number of traverse points for a circular or rectangular cross section is determined from Table A-1.



**Table A-1 Minimum Number of Traverse Points for Sampling Sites that Meet the Eight- and Two-diameter Criteria**

Stack or Duct Diameter (m)	Required Minimum Number of Traverse Points	
	Circular Duct	Rectangular Duct
>0.61	12	12
0.30 to 0.61	8	9

When the eight- and two-diameter criteria cannot be satisfied, the minimum number of traverse points is determined either from Figure A-1 for particulate sampling or from Figure A-2 for velocity measurement. Determine the number of diameters upstream and downstream from the sampling site to the flow disturbances. Then using either Figure A-1 or A-2, determine the minimum number of traverse points corresponding to: (a) the number of duct diameters upstream, and (b) the number of duct diameters downstream. Select the higher of the minimum numbers of traverse points from (a) and (b). In the case of circular cross sections, if the required number of points does not equal a multiple of four then the required number of points must be increased to the next greater multiple of four.

### **1.5 Location of Traverse Points**

For stacks or ducts with a circular cross section, locate the traverse points (as determined from Section 1.4) on two perpendicular diameters according to Table A-2. These points are located at the centroid of equal areas of the cross section.

The minimum distance between the stack wall and a traverse point shall be:

- (a) 2.5 cm (1.0 in) for stacks with diameters greater than 0.61 m (24 in); and

- (b) 1.3 cm (0.5 in) for stacks with diameters less than 0.61 m (24 in).

For rectangular cross sections, the area is divided into as many equal rectangular sections as there are sampling points (determined from Section 1.4). Locate the traverse points at the centroid of these rectangular sections. An example of the location of traverse points for a circular and rectangular stack is illustrated in Figure A-3.

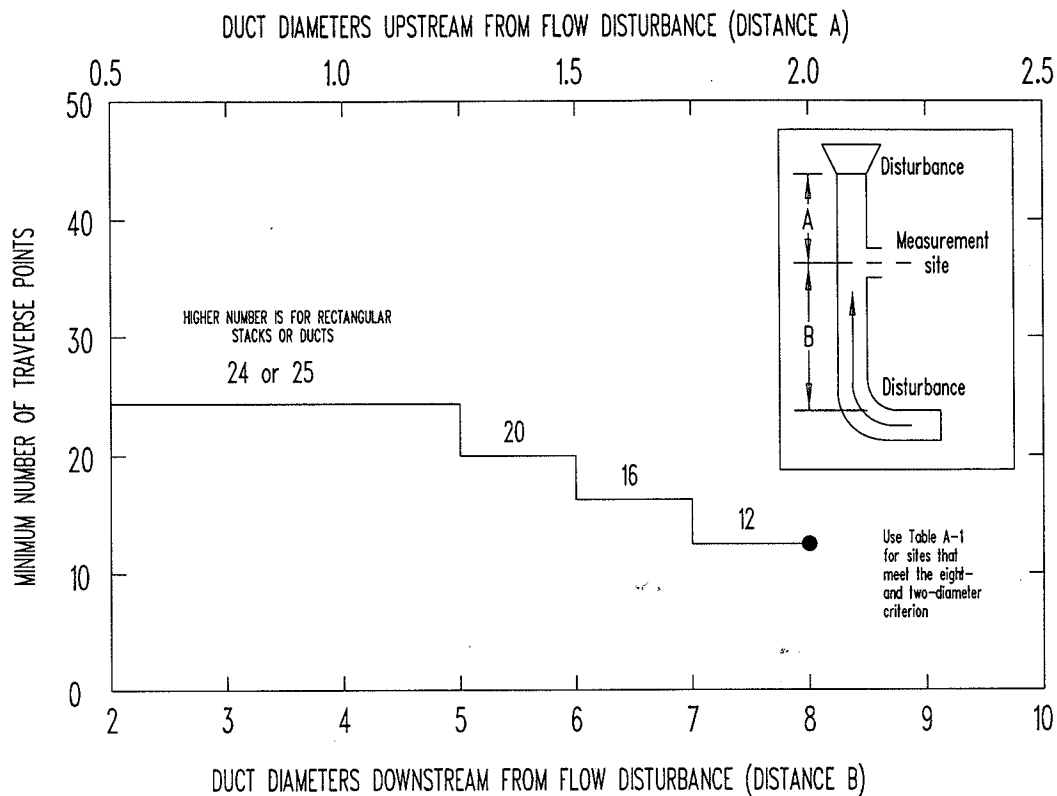
The cross-sectional layout of a rectangular duct shall be chosen such that the ratio of the length to the width is between 1.0 and 2.0.

## **1.6 Confirmation of Cyclonic and Reverse Flow**

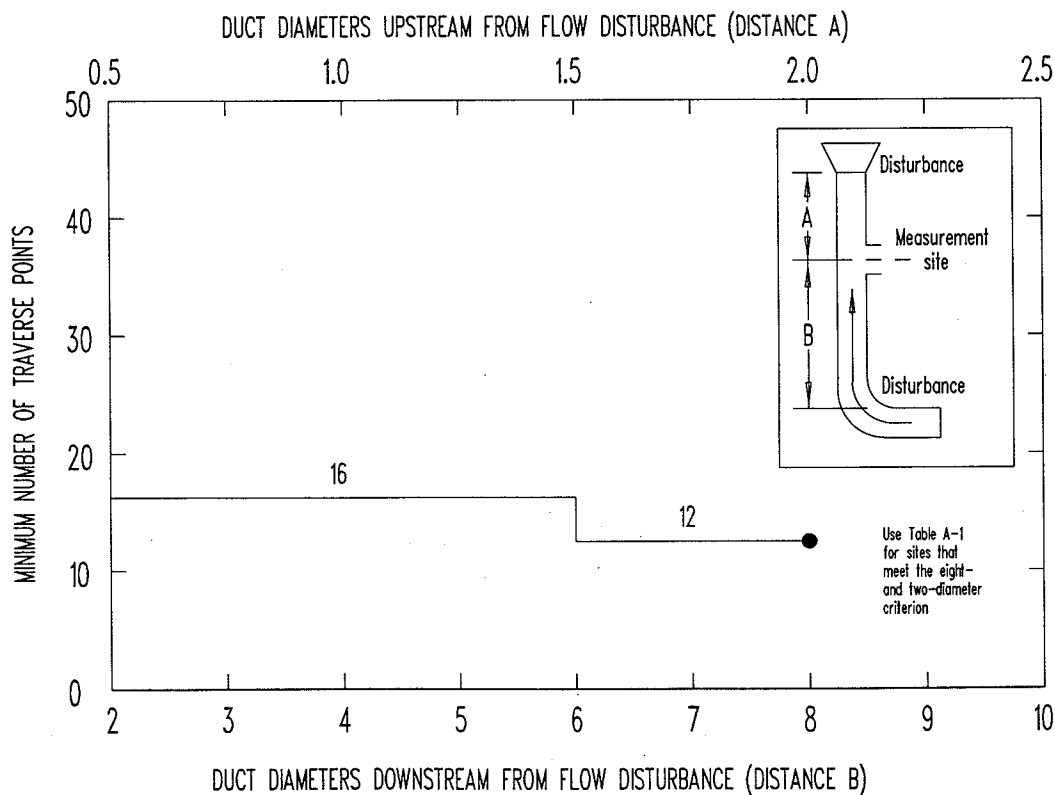
### **1.6.1 Cyclonic Flow**

The presence of cyclonic flow must be determined: (a) at a sampling site located after devices such as in-line turbine fans, cyclones, inertial demisters following venturi scrubbers; and (b) in stacks or ducts having tangential inlets or configurations which induce swirling flow. Cyclonic flow is verified by using the S-type pitot tube and manometer described in Method B and by following these procedures:

- level and zero the manometer;



**Figure A-1 Minimum Number of Traverse Points for Particulate Sampling**



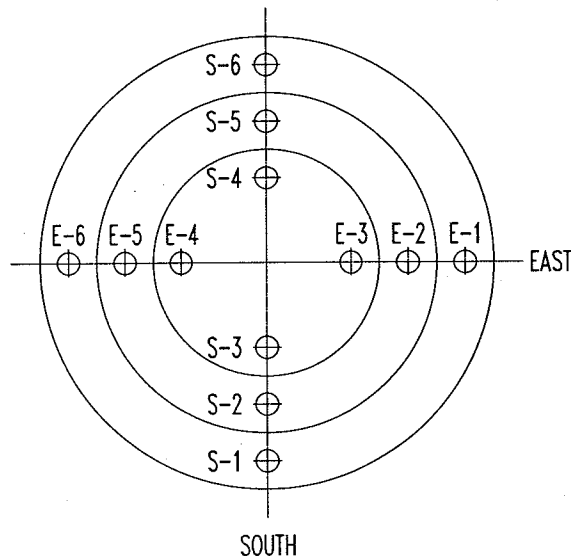
**Figure A-2 Minimum Number of Traverse Points for Velocity Measurement**



- position an S-type pitot tube at a traverse point such that the face-opening planes are parallel to the longitudinal axis (zero reference position) of the stack or duct;
- rotate the pitot (up to  $\pm 90^\circ$ ) about its longitudinal axis until the manometer  $\Delta p$  is zero; repeat this procedure for all traverse points; record all angles to the nearest degree; if the pitot does not have to be rotated to produce a null reading then an angle of  $0^\circ$  is recorded for that point; and
- calculate the arithmetic average for the absolute values of all the rotational angles; cyclonic flow is confirmed when the average rotational angle is greater than  $15^\circ$ .

### 1.6.2 Reverse Flow

The presence of reverse flow must be determined when an obstruction such as baffle plates is located inside the stack or duct, upstream of the sampling site. Reverse flow is confirmed when a negative velocity pressure is measured at one or more sampling points.



A	A-1 ⊕	A-2 ⊕	A-3 ⊕	A-4 ⊕
B	B-1 ⊕	B-2 ⊕	B-3 ⊕	B-4 ⊕
C	C-1 ⊕	C-2 ⊕	C-3 ⊕	C-4 ⊕

**Figure A-3 Location of Traverse Points on Circular and Rectangular Cross Sections Divided into Twelve Equal Areas**

## Section 2

# Method B: Determination of Stack Gas Velocity and Volumetric Flow Rate

## 2.1 Applicability

This method is **not** applicable when one or more of the following conditions exist:

- cyclonic or reverse flow has been confirmed (refer to Method A);
- gas velocity pressures are outside the normal operating range of the velocity measuring device; or
- velocity pressures fluctuate more than 20% of the average at each traverse point.

## 2.2 Principle

The average gas velocity in a stack or duct is determined from the gas density and from the measurement of velocity pressure with an S-type (Staustscheibe or reverse type) pitot tube. A standard pitot tube may be used where plugging of the tube openings due to particulate matter and/or moisture is not likely to occur. Stack gas volumetric flow rate is determined from measurements of stack gas velocity, temperature, absolute pressure, dry gas composition, moisture content, and stack diameter.

## 2.3 Apparatus

The following items are required:

**S-type pitot tube.** The S-type pitot tube should be constructed according to the specifications shown in Figure B-1. The face-opening planes shall be perpendicular

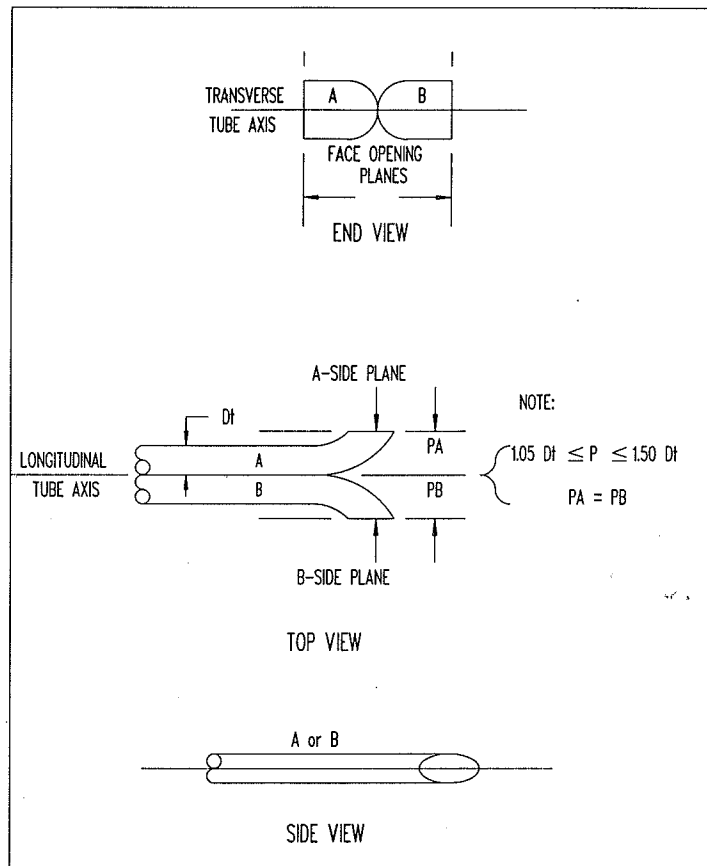
to the tube transverse axis and parallel to the longitudinal axis of the tube. Both legs shall be equal in length and the centrelines of the tubes shall be coincident when viewed from the sides. In other words,  $P_a = P_b$  and  $1.05 D_t \leq P \leq 1.50 D_t$  where  $D_t$  is between 0.48 and 0.95 cm ( $3/16$  to  $3/8$  in). Slight misalignments or deviations from the specifications in Figure B-1 are permitted, provided the deviations are recorded when the pitot tube is calibrated.

**Standard pitot tube.** A standard pitot tube may be used where particulate or condensates do not plug the static and pressure impact openings. The specifications of a standard pitot are shown in Figure B-2.

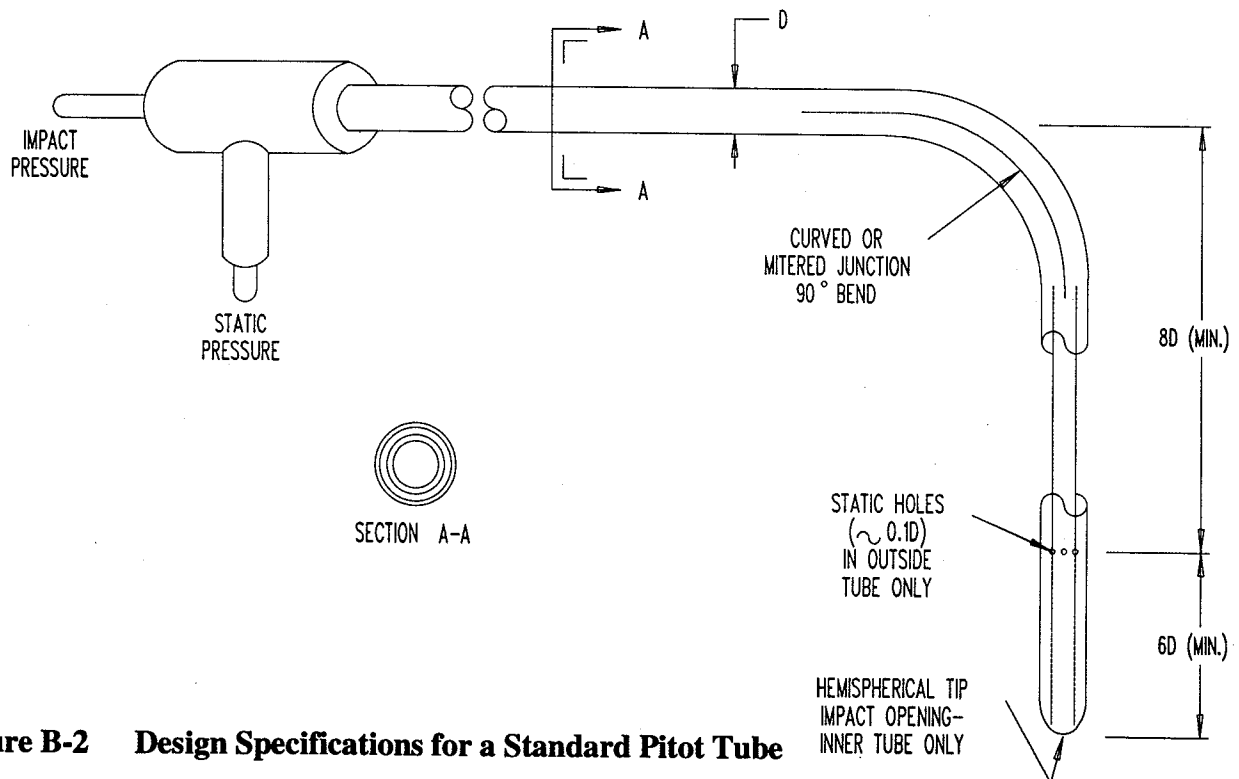
**Probe assembly.** In many cases an S-type pitot tube will be used in conjunction with a thermocouple and sampling probe. These assemblies should be constructed according to Figure B-3 to minimize the aerodynamic interference among the components of the assembly. The pitot tube coefficient for this assembly shall be determined by applying the calibration procedures described in Method F.

In the probe assembly, the tip of the temperature sensor must not contact any metal. The tip of the nozzle must extend at least two nozzle diameters below the tip of the upstream leg of the S-type pitot tube.

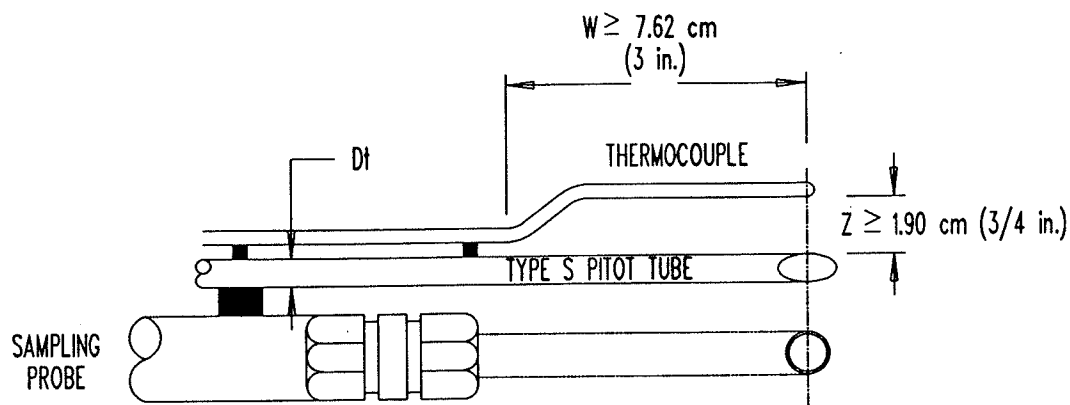
**Differential pressure indicator.** The device, such as an inclined manometer, must be capable of measuring the pitot tube



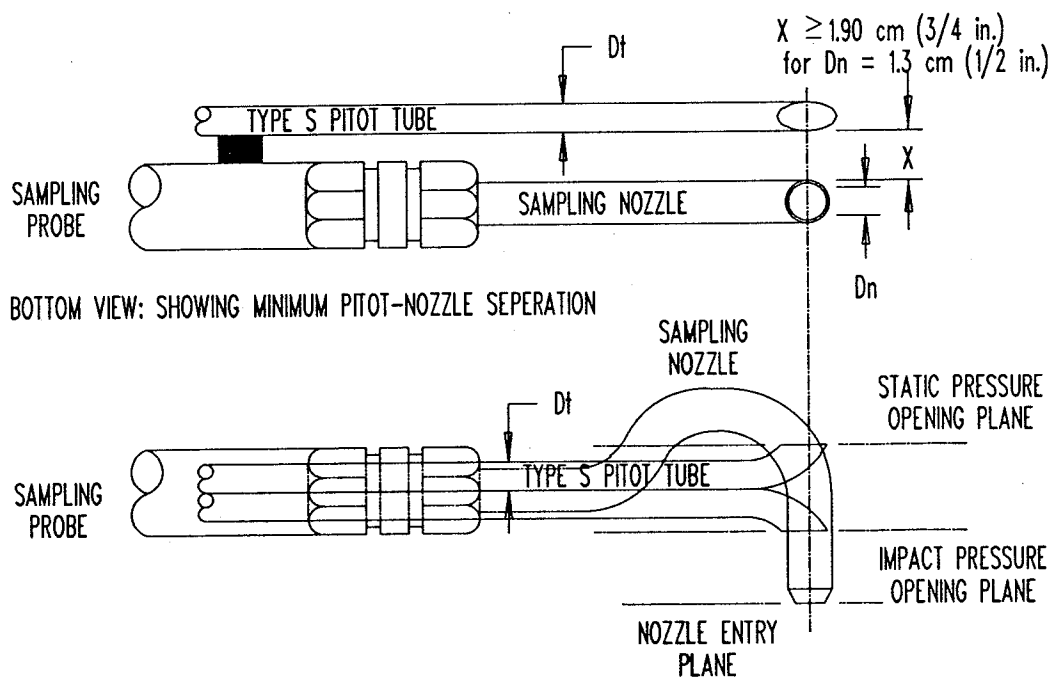
**Figure B-1 Design Specifications for an S-type Pitot Tube**



**Figure B-2 Design Specifications for a Standard Pitot Tube**



REQUIRED THERMOCOUPLE and PROBE PLACEMENT to PREVENT INTERFERENCE:  
 $D_t$  BETWEEN 0.48 and 0.95 cm (3/16 and 3/8 in.).



SIDE VIEW: TO PREVENT PITOT TUBE FROM INTERFERING WITH GAS FLOW STREAMLINES APPROACHING THE NOZZLE, THE IMPACT PRESSURE OPENING PLANE OF THE PITOT TUBE SHALL BE EVEN WITH OR DOWNSTREAM FROM THE NOZZLE ENTRY PLANE.

**Figure B-3 Configuration of Pitot Tube, Sampling Nozzle, and Thermocouple**

velocity pressure to within 0.1 mm (0.005 in) water (H<sub>2</sub>O) on the 0 to 25 mm (0 to 1 in) H<sub>2</sub>O scale, and 1 mm (0.05 in) H<sub>2</sub>O on the 25 to 250 mm (1 to 10 in) H<sub>2</sub>O scale. The device must be calibrated against a primary standard before the test.

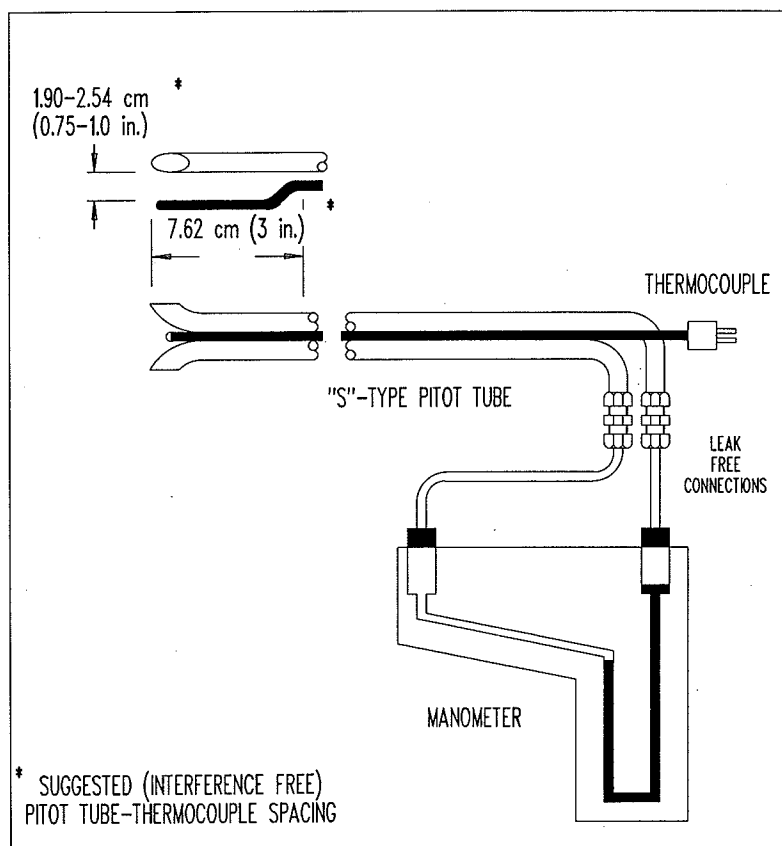
**Stack Temperature Sensor.** A calibrated thermocouple or other suitable temperature sensor capable of measuring the stack temperature to within 1.5% of the minimum absolute stack temperature. To minimize aerodynamic interaction, the thermocouple must be attached to the pitot tube as shown in Figure B-4. When stack temperatures exceeding 370°C (700°F) are encountered, appropriate shielding and aspiration shall be provided for the thermocouple to avoid heat radiation effects.

**Barometer.** A barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). The device must be calibrated against a primary standard before being used. Alternatively, the uncorrected atmospheric pressure provided by the local weather office may be used with an adjustment for the elevation of the sampling site.

**Gas density determination equipment.** The density of the stack gas shall be determined using Method C (dry molecular weight) and Method D (moisture content).

## 2.4 Procedure

Set up the apparatus as shown in Figure B-4 using an S-type pitot tube or probe assembly that has been calibrated according to Method F. The pitot tube must be used in the stack or duct in exactly the same



**Figure B-4 S-Type Pitot Tube and Manometer Assembly**



configuration as it was calibrated in the wind tunnel.

Leak check the pitot tube and lines by blowing into the pitot impact opening until 76 mm (3 in) H<sub>2</sub>O is registered on the differential pressure indicator such as an inclined manometer. Close off the impact opening. The pressure shall remain constant for at least 15 seconds. Similarly, leak check the static pressure side by applying a suction on the static opening until at least 76 mm (3 in) H<sub>2</sub>O is obtained on the manometer.

Level and zero the manometer. Since the manometer level and zero may drift due to vibrations and temperature changes, periodic checks must be made during the traverse. Record all relevant data in Figure B-5.

Measure the barometric pressure and record on the Velocity Traverse Data Sheet (Table B-1).

Measure the static pressure in the stack. Align the S-type pitot tube face-opening planes parallel to the gas flow. Seal the openings between the port and the pitot tube. Connect only one leg of the pitot tube to the manometer and vent the other side of the manometer to the atmosphere. Record the static pressure on the Velocity Traverse Data Sheet.

Measure the velocity pressure and temperature at each of the traverse points. Ensure that the face-opening planes of the S-type pitot tube are maintained perpendicular to the longitudinal axis of the

Plant _____	Stack Diameter (m) _____
Location _____	Barometric Pressure (kPa) _____
Test _____	Static Press. in Stack (mm H <sub>2</sub> O) _____
Date _____	S-type Pitot Coefficient _____
Time Started _____	Test Conducted by _____
Time Completed _____	

[illegible]

### Figure B-5 Velocity Traverse Data Sheet

stack or duct. The differential pressure gauge must be compatible with the range of measured  $\Delta p$  values. Record the velocity pressure and temperature at each point (Table B-1). When the velocity pressure fluctuates, an average velocity pressure must be recorded. However, if the velocity pressures fluctuate more than 20% around the estimated average, then the measurement is considered to be unacceptable. Determine the stack gas molecular weight and moisture content using Methods C and D, respectively.

## 2.5 Calculations

**Absolute stack gas pressure** at the sampling site is calculated using Equation B-1.

$$P_s = P_{bar} + \Delta p_s \quad \text{Equation B-1}$$

**Actual stack gas velocity** at each traverse point is calculated using Equation B-2.

$$U_s = 128.95 C_p \left( \frac{\Delta p T_s}{P_s M_s} \right)^{1/2} \quad \text{Equation B-2}$$

**The average stack gas volumetric flow rate** on a dry basis at reference conditions is calculated using Equation B-3.

$$Q_s = 3600 (U_s)_{avg} A_s (1 - B_{wo}) \frac{T_{ref} P_s}{(T_s)_{avg} P_{ref}} \quad \text{Equation B-3}$$

## 2.6 Nomenclature

$A_s$  - inside cross sectional area of stack or duct,  $m^2$

$B_{wo}$	- proportion by volume of water vapour in the stack gas, dimensionless
$C_p$	- pitot tube coefficient, dimensionless
$M_s$	- molecular weight of stack gas on a wet basis, kg/kmol
$\Delta p$	- stack gas velocity pressure, kPa
$\Delta p_s$	- stack static pressure at each traverse point, kPa
$P_{bar}$	- barometric pressure at sampling site, kPa
$P_{ref}$	- reference pressure, 101.3 kPa
$P_s$	- absolute stack gas pressure, kPa
$Q_s$	- volumetric stack gas flow rate on a dry basis at reference temperature and pressure conditions, $m^3/h$
$T_{ref}$	- reference temperature, 298 K
$T_s$	- stack gas temperature at each traverse point, K
$(T_s)_{avg}$	- arithmetic average of the stack gas temperatures, K
$U_s$	- stack gas velocity at each traverse point, m/s
$(U_s)_{avg}$	- arithmetic average of the stack gas velocities, m/s
128.95	- dimensional constant, $(m/s)[(kg/kmol)/K]^{1/2}$
3600	- conversion factor, seconds per hour, s/h

### Section 3

## Method C: Determination of Molecular Weight by Gas Analysis

### 3.1 Principle

An integrated or grab sample is extracted from a single point in the gas stream and analyzed for its components using an Orsat analyzer, a gas chromatograph, or calibrated continuous analyzers.

### 3.2 Apparatus

#### 3.2.1 Integrated Gas Sampling Train

The following items are required:

**Probe/Filter.** A probe made of stainless steel, Pyrex, or other corrosion-resistant material to extract the sample from the stack or duct being tested. A filter is used to remove particulate matter from the gas sample.

**Condenser/Water Trap.** A condenser and water trap, or impingers to remove and trap moisture contained in the gas sample.

**Control Valve.** A valve to control the flow of gas in the sampling system.

**Pump.** A leakless pump to withdraw the sample gas through the sample train.

**Rotameter.** A rotameter, or equivalent device to measure the sample gas flow in the train. The unit should measure flows up to 2 L/min.

**Sample Bag.** A flexible bag of a non-reactive material such as Tedlar or Teflon, with a total capacity of 1L for each

minute of anticipated sampling to hold the collected sample.

**S-type Pitot Tube and Differential Pressure Indicator.** A calibrated S-type pitot and a differential pressure device such as an inclined manometer to measure the gas velocity during sampling.

#### 3.2.2 Grab Sample Apparatus

The following items are required:

**Probe/Filter.** See Subsection 3.2.1.

**Pump.** A squeeze bulb with a one-way valve to withdraw a sample from the stack or duct under test.

**Analyzer.** An Orsat analyzer, a gas chromatograph, or calibrated continuous gas analyzers to analyze the major constituents in the gas sample. The components measured should include, as a minimum, carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), and nitrogen (N<sub>2</sub>) by difference. Where an additional gas may affect the molecular weight by greater than 2%, this component must be measured and the resulting concentration included in the calculation.

### 3.3 Procedures

#### 3.3.1 Integrated Sampling

Leakcheck the bag by connecting it to a water manometer and pressurizing the bag to 5 to 10 cm H<sub>2</sub>O. Allow to stand for 10 minutes. Repair or replace the bag if

there is a decrease in the manometer reading after this time period.

Evacuate the sample bag using the sample pump, then assemble the equipment as shown in Figure C-1, leaving the sample bag disconnected. Ensure that all connections are leak-free. Place the sample probe in the stack or duct under test, and allow at least five minutes for the probe and optional filter to reach stack temperature. Turn on the sample pump and allow sample gas to purge air out of the system for about two minutes using a flow of approximately 1L/min. Turn off the sample pump and connect the sample bag as in Figure C-1.

Turn on the sample pump and adjust the sample flow rate to one which will provide an adequate sample size, yet not overfill or burst the sample bag. The sampling rate,  $Q$ , is maintained proportional ( $K$ ) to the stack gas velocity,  $U_s$ , over the sampling period. Use Equation C-1 to calculate the sampling rate and adjust the control valve to maintain the sampling rate indicated by Equation C-1 throughout the test.

$$Q = K \cdot U_s$$

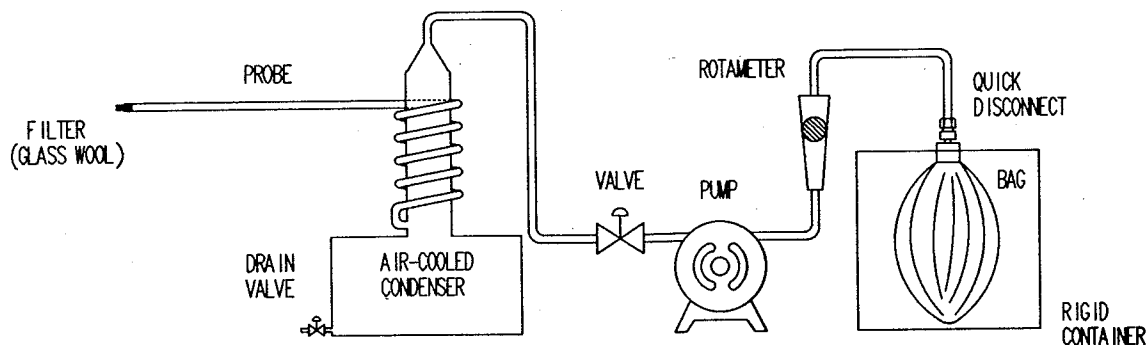
**Equation C-1**

At the completion of sampling, turn off the pump, then seal and disconnect the sample bag. Analyze the bag contents using the procedures provided in Section 3.4.

### 3.3.2 Grab Sampling

This procedure provides an analysis of the stack gas constituents which is not as complete as would be provided by the procedure given in Subsection 3.3.1 and should only be used when the previous procedure cannot be applied.

Set up the apparatus as shown in Figure C-2, but do not connect the analyzer(s). Ensure that all connections are leak-free. Place the probe in the duct or stack and purge the system with stack gas using the squeeze bulb for a minimum of one minute. Connect the analyzer(s) to the discharge side of the squeeze bulb. Follow the manufacturer's instructions regarding the recommended quantity of gas that should be provided to the analyzer(s) and any flow rate recommendations.



**Figure C-1 Integrated Gas Sampling Train**

### 3.3.3 Continuous Source Monitoring

A continuous (real-time) source monitoring system may be utilized to measure the concentrations of the major gas components. A monitoring system of this nature would normally be operated as part of a larger testing program. The setup and operation of such a system is beyond the scope of this document; therefore, specifics will not be provided in this method. Where data from such a system are used, the system must be calibrated using verified reference gases. A table of the calibration gases used must be provided as well as the procedures followed in calibrating and operating the system. A diagram of the continuous source monitoring system must also be supplied.

### 3.4 Analysis

Follow the manufacturer's instructions on the operation of the analytical apparatus utilized.

Calibrate the apparatus using verified reference gas cylinders prior to analyzing the

stack gas to ensure that the equipment is operating satisfactorily. Include this calibration data with the reported concentrations of the various components.

When following the integrated sampling procedure, determine the concentrations of the components immediately after sampling. Repeat the analysis of the bag sample until the analyses vary no more than 0.25% (v/v) for each component.

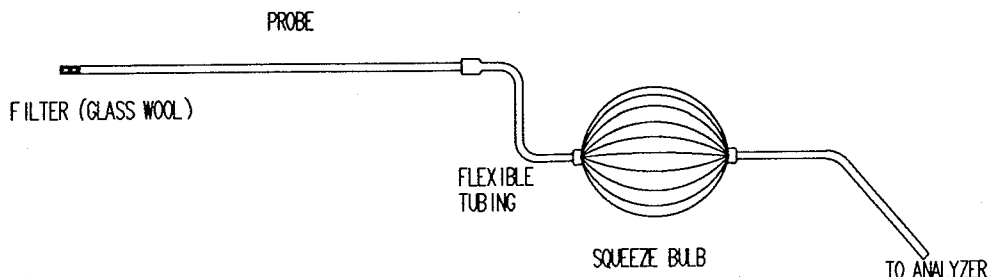
Using the grab sampling procedure, a set of analyses must be carried out at equal time intervals during testing; the time period between analyses not to exceed ten minutes.

### 3.5 Calculations

**Dry Molecular Weight ( $M_d$ ).** The dry molecular weight of the sample is calculated using Equation C-2.

$$M_d = 44(\%CO_2/100) + 28(\%N_2/100) + 32(\%O_2/100)$$

**Equation C-2**



**Figure C-2 Grab Sample Apparatus**

Equation C-2 assumes that the stack gas components measured include carbon dioxide (CO<sub>2</sub>), nitrogen (N<sub>2</sub>), and oxygen (O<sub>2</sub>). Where the concentrations of additional components are determined, the equation can be modified to include these components by adding extra terms to Equation C-2, as shown in Equation C-3.

$$M_d = M_d(\text{from Equation C-2}) + MW_i(\% i/100)$$

**Equation C-3**

**Wet Molecular Weight (M<sub>s</sub>).** The wet molecular weight is calculated using the dry molecular weight (M<sub>d</sub>) determined using Equation C-2 or C-3, and the stack gas moisture content (B<sub>wo</sub>) determined from Method D (Determination of Moisture Content).

$$M_s = M_d(1 - B_{wo}) + 18B_{wo}$$

**Equation C-4**

### 3.6 Nomenclature

B<sub>wo</sub> - proportion by volume of water vapour in the stack gases, dimensionless

%CO<sub>2</sub> - percent carbon dioxide by volume, dry basis

%i - percent of additional component, i, measured by volume, dry basis

K - proportionality, constant, (L/min) (s/m)

M<sub>d</sub> - molecular weight of gas on a dry basis, kg/kmol

M<sub>s</sub> - molecular weight of gas on a wet basis, kg/kmol

MW<sub>i</sub> - molecular weight of component i, kg/kmol

%N<sub>2</sub> - percent nitrogen by volume, by difference, dry basis

%O<sub>2</sub> - percent oxygen by volume, dry basis

Q - sampling rate indicated by the rotameter, L/min

U<sub>s</sub> - stack gas velocity, m/s

Plant: \_\_\_\_\_

Location: \_\_\_\_\_

Test: \_\_\_\_\_

Date: \_\_\_\_\_

Test Conducted by: \_\_\_\_\_

## A - GRAB SAMPLING RESULTS

Time	% O <sub>2</sub>	% CO <sub>2</sub>	% _____	% _____
AVERAGES				

(By difference) % N<sub>2</sub> = \_\_\_\_\_

## B - INTEGRATED SAMPLING RESULTS

% O<sub>2</sub> = \_\_\_\_\_% CO<sub>2</sub> = \_\_\_\_\_

% \_\_\_\_\_ = \_\_\_\_\_

% \_\_\_\_\_ = \_\_\_\_\_

Figure C-3 Molecular Weight Data Sheet

## Section 4

# Method D: Determination of Moisture Content

### 4.1 Principle

A gas sample is extracted from a single point in the enclosed gas stream being sampled. The moisture is condensed and its weight measured. This weight, together with the volume of gas sampled, enable the stack gas moisture content to be calculated. The moisture content of the gas leaving the final impinger is taken into consideration by assuming that the gas stream is saturated at the average impinger outlet temperature. The moisture content is calculated by using a psychrometric table relating temperature and water content.

If liquid water droplets are present, or are suspected, the moisture content of the gas should be determined using the stack gas temperature in association with psychrometric charts.

### 4.2 Apparatus

The following items are required:

**Probe.** A stainless-steel, Pyrex or other corrosion-resistant heated probe, equipped with a filter to remove particulate matter.

**Impingers.** Two 30-mL midget impingers.

**Ice-Bath.** An ice bath in which the midget impingers are immersed.

**Silica Gel Tube.** A tube, of approximately 250- to 500-mL capacity, containing indicating silica gel. The use of this tube is optional, as it serves to protect the pump and dry gas meter.

**Valve.** A valve to control the sample gas flow rate through the sampling train.

**Pump.** A leakless pump to pull the gas sample through the sampling train.

**Dry Gas Meter.** A calibrated dry gas meter capable of measuring the sample volume to within 1%. The meter should allow accurate volume determination for a sample flow rate of up to 5 L/min.

**Rotameter.** A calibrated rotameter or orifice meter to measure the instantaneous sample flow. The unit should be capable of indicating flows of up to 5 L/min.

**Graduated Cylinder.** A 25-mL graduated cylinder to measure the volume of water placed in the impingers.

**Barometer.** A barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). The device must be calibrated against a primary standard before use. Alternatively, the uncorrected atmospheric pressure provided by the local weather office may be used with an adjustment for the elevation of the sampling site.

**S-type Pitot Tube and Differential Pressure Indicator.** A calibrated S-type pitot and a differential pressure device, such as an inclined manometer to measure gas velocity during sampling.

**Scale or Balance.** A scale or balance, capable of measuring the weights of the impingers and their contents to within 0.1 g.



### 4.3 Procedure

Measure and place 5 mL of water into each of the two impingers. Weigh and record the initial weight of each of the two impingers to within 0.1 grams. Assemble the sampling train as indicated in Figure D-1, but do not connect the probe. Ensure that all connections are tight and the system is leak-free. Leak check the system by plugging the inlet to the first impinger, turning on the pump and observing the dry gas meter for any movement of the dial.

Connect the probe and place it in the stack at the desired sampling point. Seal the stack access port to prevent the influx of dilution air. Allow the probe to heat up sufficiently to prevent condensation ahead of the impingers. Turn on the sample pump and adjust the sample flow rate to approximately 2 L/min. Adjust the sampling rate, maintaining it proportional to changes in the

stack gas velocity using Equation D-1 (see Section 4.5 for nomenclature).

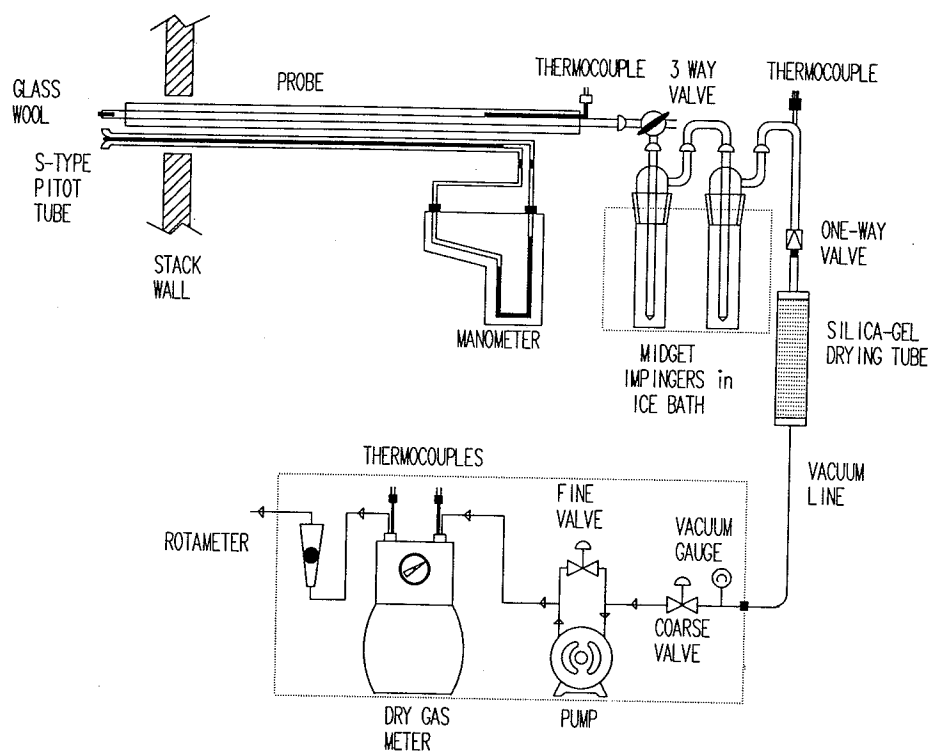
$$Q = K \cdot U_s$$

**Equation D-1**

Continue sampling for a minimum of 15 minutes, collecting a sample volume of at least 30 L, as indicated on the dry gas meter. Record on the Moisture Data Sheet (Figure D-2), at five-minute intervals, the temperature of the last impinger discharge, the sample flow rate, dry gas meter reading, and the temperatures at the inlet and outlet of the dry gas meter, if applicable. At the conclusion of sampling, measure the final weight of the two impingers with their contents to within 0.1 g.

### 4.4 Calculations

**Volume of Water Vapour Collected.** Use Equation D-2 to calculate the volume of



**Figure D-1 Moisture Sampling Train**

water vapour contained in the stack gas sample at reference temperature and pressure conditions.

$$(V_w)_{ref} = \frac{W_{H_2O} \times 10^{-3} RT_{ref}}{M_{H_2O} P_{ref}} \quad \text{Equation D-2}$$

**Sample Gas Volume.** Use Equation D-3 to calculate the sample gas volume at reference temperature and pressure conditions.

$$(V_m)_{ref} = \gamma V_m \frac{T_{ref} P_m}{(T_m)_{avg} P_{ref}} \quad \text{Equation D-3}$$

**Moisture Content of Final Impinger Outlet.** Using a psychrometric table, look up the corresponding partial pressure of water vapour at the average temperature at the outlet of the final impinger. Use Equation D-4 to calculate the volumetric proportion of water vapour in this gas.

$$B_{wm} = \frac{P_v}{P} \quad \text{Equation D-4}$$

**Moisture Content.** Use Equation D-5 to calculate the stack gas moisture content.

$$B_{wo} = \frac{(V_w)_{ref}}{(V_w)_{ref} + (V_m)_{ref}} + B_{wm} \quad \text{Equation D-5}$$

## 4.5 Nomenclature

$B_{wm}$  - proportion by volume of water vapour in gas stream leaving last impinger, dimensionless

$B_{wo}$  - proportion by volume of water vapour in the gas stream, dimensionless

$K$  - proportionality constant, (L/min)(s/m)

$M_{H_2O}$  - molecular weight of water, 18 kg/kmol

$P$  - absolute pressure at last impinger, approximated by the barometric pressure, kPa

$P_m$  - absolute pressure at dry gas meter, kPa

$P_{ref}$  - reference pressure, 101.3 kPa

$P_v$  - partial pressure of water vapour leaving the last impinger, kPa

$Q$  - sampling rate indicated by the rotameter, L/min

$R$  - universal gas constant, 8.31 (kPa)(m<sup>3</sup>)(kmol<sup>-1</sup>)(K<sup>-1</sup>)

$(T_m)_{avg}$  - arithmetic average of dry gas meter temperatures, K

$T_{ref}$  - reference temperature, 298K

$U_s$  - stack gas velocity, m/s

$V_m$  - volume of stack gas sample at dry gas meter conditions, m<sup>3</sup>

$(V_m)_{ref}$  - volume of stack gas sample at reference temperature and pressure conditions, m<sup>3</sup>

$(V_w)_{ref}$  - volume of water vapour at reference temperature and pressure conditions, m<sup>3</sup>

$W_{H_2O}$  - amount of moisture collected in the impingers, g

$\gamma$  - dry gas meter correction factor, dimensionless

Plant: \_\_\_\_\_

Location: \_\_\_\_\_

Test: \_\_\_\_\_

Date: \_\_\_\_\_

Test Conducted by: \_\_\_\_\_

Time	Meter Volume	Meter Temperature		Last Impinger Temperature	Rotameter Reading
		Inlet	Outlet		
AVERAGES					

Barometric pressure \_\_\_\_\_ kPa

Initial Weight of impingers and contents \_\_\_\_\_ g

Final Weight of impingers and contents \_\_\_\_\_ g

Difference ( $W_{H_2O}$ ) \_\_\_\_\_ g**Figure D-2 Moisture Data Sheet**

## Section 5

# Method E: Determination of Particulate Releases

## 5.1 Applicability

This method is used to measure the mass concentration and mass emission or release of particulate matter from enclosed gas streams of stationary sources.

Direct application of the procedures specified in this method may be limited by one or more of the following conditions:

- sample locations less than two stack diameters downstream or less than 0.5 stack diameter upstream of a flow disturbance;
- duct cross-sectional areas less than  $0.071 \text{ m}^2$  ( $113 \text{ in}^2$ ) or duct diameters less than 0.3 m (12 in);
- supersaturated gas streams with entrained liquid droplets;
- gas stream flow rates less than 3 m/s (10 ft/s) or greater than 30 m/s (100 ft/s);
- excessively high stack gas temperature that may cause damage to the sampling equipment even with the use of a water-cooled probe;
- gas streams containing corrosive or unstable components;
- cyclonic flow patterns within the gas stream; or
- rapid fluctuations in velocity, particulate loading, and/or temperature of the gas

stream due to uncontrollable process variations.

For compliance testing, possible modifications to allow sampling of sources exhibiting any of these characteristics may be approved in writing by the Minister of the Environment.

## 5.2 Principle

Particulate matter is withdrawn isokinetically from a number of sampling or traverse points in an enclosed gas stream. The particulate sample is collected in the nozzle, probe, cyclone (see Section 1.3), and on a glass fibre filter, all maintained at a temperature of  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ ) or at such other temperature as is necessary to prevent blinding of the filter from condensation. The particulate weight is determined gravimetrically after removal of uncombined water. Simultaneous determinations of the gas stream moisture content, velocity, temperature, and molecular weight allow calculations of the particulate concentration and the particulate mass emission or release rate to be made.

Sampling isokinetically means that the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sampling point.

## 5.3 Apparatus

### 5.3.1 Sample Collection (Figure E-1)

The following items are required for sample collection:

**Nozzle.** A button-hook type nozzle with sharp, tapered leading edges. The nozzle is usually made of 316 stainless steel or Incoloy 825, but quartz or other inert material may be used when high temperature or corrosive gases are encountered. The minimum inside diameter of the nozzle shall be 4.76 mm (3/16 in) and shall be determined using calipers.

**Probe.** A Pyrex or quartz glass liner. Where length or strength limitations preclude the use of a glass liner, a seamless tubing made from an inert and corrosion-resistant material such as 316 stainless steel, Incoloy 825 or Teflon may be used. The liner is encased in a stainless-steel tube with a heating and temperature indicating system capable of maintaining the exit gas temperature at  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ), or at such a temperature necessary to prevent condensation. A water-cooled probe should be used when very hot gases capable of damaging the nozzle/probe assembly are encountered.

**Pitot Tube.** An S-type (Stausscheibe) pitot tube attached to the probe. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other. The configuration of the probe assembly (pitot tube, nozzle, and thermocouple) is specified in Method B. The probe assembly shall be calibrated according to the calibration procedures specified in Method F.

**Stack Temperature Sensor.** A calibrated thermocouple or other suitable temperature sensor capable of measuring the stack temperature to within 1.5% of the minimum absolute stack temperature. To minimize aerodynamic interaction, the pitot tube, thermocouple, and probe should be configured as specified in Method B. When high temperature gases are encountered,

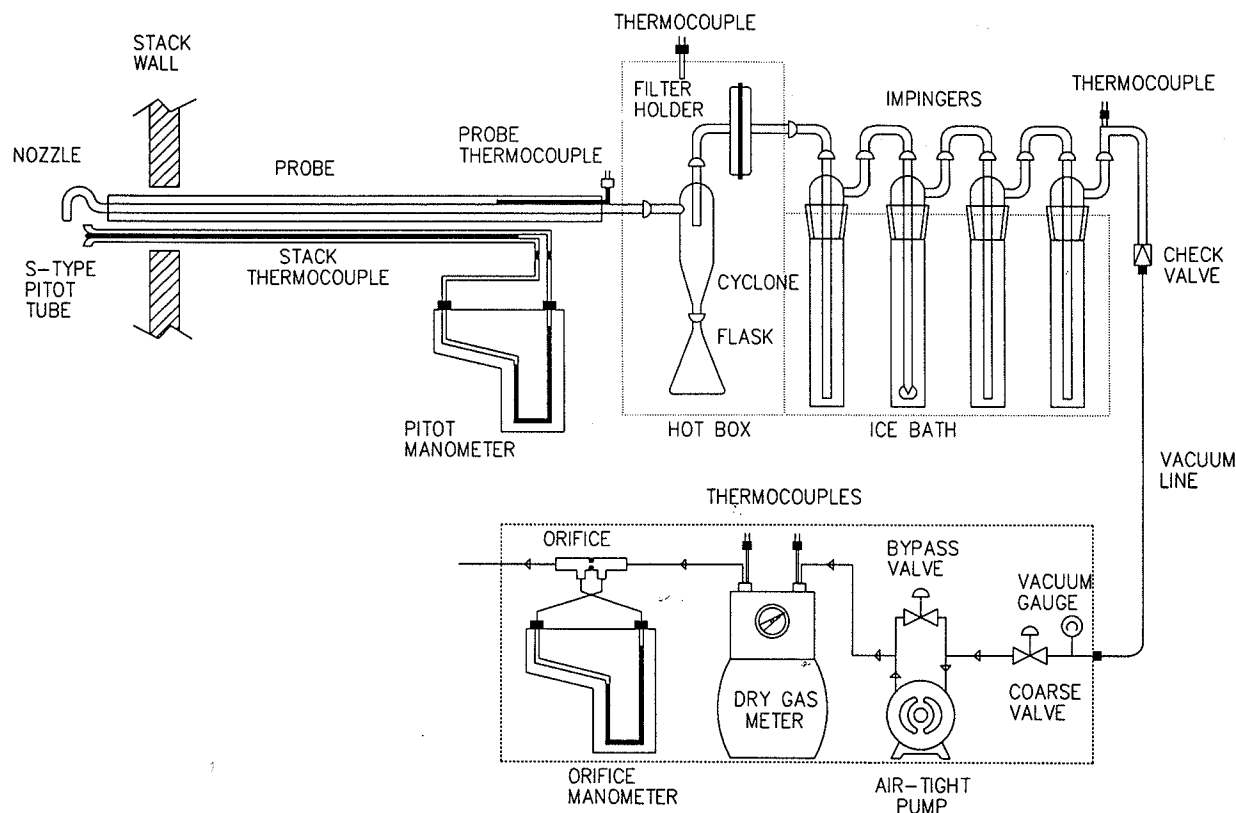
appropriate shielding and aspiration should be provided for the thermocouple to avoid heat radiation effects.

**Cyclone (Optional).** A miniature Pyrex cyclone following the sampling probe and preceding the filter is to be used if premature buildup of particulate matter on the filter medium is anticipated. The cyclone is located inside the filter compartment and is, therefore, maintained at the same temperature as the filter.

**Filter Holder.** A Pyrex filter holder with a porous, fritted glass filter support and a silicone rubber gasket. The filter support may also be made of stainless steel, Teflon, or other inert and corrosion-resistant material. The filter holder is located inside the filter compartment.

**Filter Compartment Heating System.** A heating system capable of maintaining the temperature in the filter holder compartment at  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ) or at such other temperature as is necessary to prevent blinding of the filter from condensation. A thermocouple or other temperature sensor is also required to measure the compartment temperature to within  $3^{\circ}\text{C}$  ( $5^{\circ}\text{F}$ ).

**Impingers.** Four Greenburg-Smith impingers are connected in series. The first, third, and fourth impingers are modified by replacing the tips and impaction plates of the standard design with a 13 mm (0.5 in) ID glass tube extending to within 13 mm (0.5 in) of the bottom of the impinger. The second impinger has the standard tip and impaction plate. The impingers are contained in an ice bath during sampling. The impingers may be replaced by any other suitable condenser providing that the condensed liquid is to be used for moisture determination only. A temperature sensor capable of measuring to within  $1^{\circ}\text{C}$  ( $2^{\circ}\text{F}$ )



**Figure E-1 Particulate Sampling Train**

shall be placed at the outlet of the last impinger.

**Vacuum Pump.** A leakless vacuum pump capable of maintaining an isokinetic sampling rate while continuously withdrawing a portion of the stack gases through the sampling train. The pump intake vacuum is measured to within (13 mm Hg) (0.5 in Hg) by a vacuum gauge attached to the vacuum line connecting the pump to the last impinger outlet. The sample flow rate is controlled by a combination of the coarse and fine flow control valves.

**Metering System.** A calibrated dry gas meter with inlet and outlet temperature sensors, or one that is temperature-compensated. The meter shall be calibrated according to the procedures specified in Method F. The temperature sensors must be

capable of measuring the temperature to within 3°C (5°F).

**Orifice.** A calibrated orifice connected to the outlet of the dry gas meter. The orifice shall be calibrated according to the procedure specified in Method F.

**Differential Pressure Indicators.** The devices, such as inclined manometers, must be capable of measuring the pitot tube velocity pressure and the pressure drop across the orifice to within 0.1 mm (0.005 in) H<sub>2</sub>O on the 0 to 25 mm (0 to 1 in) H<sub>2</sub>O scale, and 1 mm (0.05 in) H<sub>2</sub>O on the 25 to 250 mm (1 to 10 in) H<sub>2</sub>O scale. The devices must be calibrated against a primary standard prior to the test.

**Barometer.** A barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 inch Hg). The device must

be calibrated against a primary standard prior to the test. Alternatively, the uncorrected atmospheric pressure provided by the local weather office may be used with an adjustment for the elevation of the sampling site. Deduct (2.5 mm Hg) per 30.5 m of elevation increase or add (2.5 mm Hg) per 30.5 m of elevation decrease.

### 5.3.2 Sample Recovery

The following items are required for sample recovery:

**Probe Brush.** A nylon bristle brush of a length and diameter suitable for cleaning the probe.

**Balances.** An analytical balance capable of measuring to within  $\pm 0.1$  mg or less and a trip or top loading balance capable of measuring to within  $\pm 0.5$  g or better.

**Miscellaneous.** Wash bottles, sufficient quantity of sample containers large enough to hold all washings and a petri dish for holding the filter sample. All items shall be made of material such as glass, Teflon, or polypropylene which are chemically inert to both the samples and the reagents used for sample recovery.

## 5.4 Reagents and Materials

All chemicals used shall be reagent grade. The quality of the distilled or deionized water shall conform to specifications for Type III water given by the ASTM<sup>(1)</sup>.

### 5.4.1 Sample Collection

The following items are required for sample collection:

**Filter.** A flash-fired glass fibre filter (organic binder removed) of a diameter compatible with the filter holder and capable of efficiently retaining particles as small as  $0.3 \mu\text{m}$  in accordance with ASTM Standard D2986-71<sup>(2)</sup>. The filter material must be chemically inert to stack gas components such as sulphur dioxide ( $\text{SO}_2$ ). Depending on the nature of the source and the analysis required, other types of filter media may be used, subject to Environment Canada approval. The filter must be desiccated to a constant weight before being used\*. Place the pre-weighed filter in a clean container to prevent contamination during transportation to the sampling site.

**Miscellaneous.** Distilled or deionized water, crushed ice, heat-stable silicone stopcock grease, and indicating-type 6-16 mesh silica gel that has been dried at  $180^\circ\text{C}$  ( $350^\circ\text{F}$ ) for two hours.

### 5.4.2 Sample Recovery

The following items are required for sample recovery:

**Acetone.** Reagent grade acetone with low residue ( $<0.001$  percent by weight).

**Water.** Distilled or deionized water.

\* **To Desiccate a filter to a Constant Weight.** Label and desiccate the filter for at least 24 hours using silica gel or equivalent at  $20 \pm 6^\circ\text{C}$  ( $68 \pm 10^\circ\text{F}$ ). Weigh the filter to the nearest 0.1 mg at intervals of six hours or more in a room where the relative humidity is 50% or less. The weighing must be completed within two minutes after the filter is removed from the desiccator. The constant weight is attained when the difference between two consecutive readings is less than 0.5 mg.

## 5.5 Procedures

### 5.5.1 Sample Collection

**Preliminary.** Select the sampling site and the minimum number of traverse points according to procedures described in Method A. In the absence of any previous knowledge of the stack variables, a preliminary test should be conducted to obtain the following data:

- Velocity profile across the stack (Method B)
- Stack temperature and pressure (Method B)
- Stack gas molecular weight (Method C)
- Stack gas moisture content (Method D)

Use the data to determine the largest nozzle size possible for isokinetic sampling. Recommended minimum nozzle size is 4.76 mm (3/16 in) ID.

Select a total sampling time so that the sampling time per traverse point is equal to or greater than five minutes. The sample should be taken over a continuous process operating period. The purpose of the test will determine the sample volume to be collected.

**Sampling Train Preparation.** Prepare the sampling train in a clean area to minimize contamination. Install the selected size nozzle on the probe. Mark the probe with heat-resistant tape to denote the location of each sampling point. Use a pair of tweezers to place the labelled and pre-weighed filter

Plant: \_\_\_\_\_  
 Location: \_\_\_\_\_  
 Test Number: \_\_\_\_\_  
 Date: \_\_\_\_\_  
 Test Conducted by: \_\_\_\_\_

Component	Content	Weight (g)
First impinger	~ 100 mL deionized or distilled water	Final _____ Initial _____ Gain _____ [a]
Second impinger	~ 100 mL deionized or distilled water	Final _____ Initial _____ Gain _____ [b]
Third impinger	Empty	Final _____ Initial _____ Gain _____ [c]
Fourth impinger	~ 200 g Silica Gel	Final _____ Initial _____ Gain _____ [d]

Weight of water collected = [a] + [b] + [c] + [d]

$W_{H_2O}$  = \_\_\_\_\_ g

**Figure E-2 Moisture Analysis Data Sheet**



in the filter holder. Place a known volume (approximately 100 mL) of deionized or distilled water in each of the first two impingers, leave the third impinger empty and place a known amount (approximately 200 g) of silica gel in the fourth. Record the volume or weight (to the nearest 0.5 mL or 0.5 g) of the content of each impinger on the Moisture Analysis Data Sheet (Figure E-2). Set up the sampling train as in Figure E-1. Adjust the filter compartment and probe heating systems to maintain a temperature of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ) or such other temperature as is necessary to prevent blinding of the filter due to condensation. In the presence of an acidic gas, such as  $\text{SO}_2$  in the source, both temperatures shall be maintained above the acid dew point of the gas stream.

Conduct a mandatory pre-test leak check of the sampling train by plugging the nozzle inlet and pulling a vacuum of 380 mm Hg (15 in. Hg) for at least one minute. The leakage rate must be less than 0.57 L/min ( $0.02 \text{ ft}^3/\text{min}$ ) or 4% of the estimated average sampling rate, whichever is less. Sampling cannot proceed until the leakage rate is acceptable. Record the actual leakage rate on the Particulate Sampling Data Sheet (Figure E-3). Place crushed ice and water in the impinger box before sampling.

**Sampling Train Operation.** To begin sampling, point the nozzle directly into the gas stream at the first traverse point. Using a nomograph or a programmable calculator, determine the orifice setting for isokinetic sampling. Immediately start the vacuum pump and adjust the sampling flow rate to isokinetic conditions. Sample for at least five minutes at each traverse point, the sampling time being the same for every point. Traverse the stack cross section and maintain isokinetic sampling throughout the test. Add more ice and water to the impinger

box, as required, to maintain the temperature of the last impinger exit in the range of  $0^{\circ}$  to  $20^{\circ}\text{C}$  ( $32^{\circ}$  to  $68^{\circ}\text{F}$ ).

Record instrumentation readings on the Particulate Sampling Data Sheet (Figure E-3) every five minutes, or at regular intervals that are consistent with the sampling duration established for each point, whichever is less. (For example: Five minutes of sample per traverse point will require readings being recorded every five minutes; six minutes per point, every three minutes; eight minutes per point, every four minutes; etc.) Readings must also be taken before and after a leak check and when sampling is halted.

When it is necessary to halt sampling temporarily, either to dismantle the sampling train during port changeover or to change a train component, turn off the pump and immediately withdraw the probe from the stack. Conduct a mandatory leak check on the sampling train by plugging the nozzle and pulling a vacuum equal to or greater than the maximum value observed during sampling. Record the actual leakage rate. If the leakage rate exceeds 0.57 L/min ( $0.02 \text{ ft}^3/\text{min}$ ) or 4% of the sampling flow rate, the test is invalid. If the leakage rate is acceptable, proceed with dismantling the sampling train or changing the train component. Before continuing with the test, conduct a mandatory leak check on the assembled train by following the pre-test leak check procedures used during sampling train preparation.

When the test is completed, conduct a mandatory post-test leak check on the sampling train by plugging the nozzle and pulling a vacuum equal to or greater than the maximum value observed during sampling. Record the actual leakage rate which must be less than 0.57 L/min ( $0.02 \text{ ft}^3/\text{min}$ ) or 4% of

1) \_\_\_\_\_ CFM @ \_\_\_\_\_ " Hg

2) \_\_\_\_\_ CFM @ \_\_\_\_\_ " Hg

[illegible]

### Figure E-3 Particulate Sampling Data Sheet

the sampling flow rate, whichever is less. If the leakage rate is acceptable, proceed with recovering the samples.

### 5.5.2 *Sample Recovery*

Disconnect the probe from the sampling train. Seal all openings. Exercise care in moving the train components from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Partition the train samples as follows:

**Filter (Container No. 1):** Use a pair of clean tweezers or a sharp knife to transfer the filter and any loose material adhering to the filter support into a petri dish. Label and seal the sample container.

**Nozzle, Probe Liner, Cyclone (if used), and Front-half of Filter-holder (Container No. 2):** Wash and brush the interior surfaces of the nozzle and probe with acetone. Place these washings into Container No. 2. Use acetone together with a brush or a rubber policeman to clean the inside surfaces of the cyclone (if used) and the front-half of the filter holder. Collect the acetone washings into Container No. 2. Seal and label the container and mark the liquid level.

**Acetone Blank (Container No. 3):** Place a known volume (approximately equal to that in Container No. 2) of acetone, taken directly from the wash bottle being used, into Container No. 3. Seal the sample container and label it "acetone blank". Mark the liquid level.

**Impingers (Container No. 4):** Measure the volume or weight (to the nearest 0.5 mL or 0.5 g) of the content of each impinger and record the results on the Moisture Analysis Data Sheet (Figure E-2). If chemical analysis of the impinger liquid is required, transfer the contents of the first three

impingers to Container No. 4. Rinse the inside surfaces of these impingers, all connectors, and the back-half of the filter holder with deionized or distilled water into the same sample container. Seal and label the container and mark the liquid level. Discard the spent silica gel.

### 5.5.3 *Sample Analysis*

All analyses shall be performed in a clean laboratory equipped with a fume hood. The relative humidity of the room in which weighing is performed should be maintained at or below 50%.

**Container No. 1 (Filter):** Transfer the filter and any loose particulate matter and filter material from the sample container to a tared weighing dish. Desiccate the sample to a constant weight (see note in Subsection 5.4.1) and record the result to the nearest 0.1 mg on the Particulate Analytical Data Sheet (Figure E-4).

**Container No. 2 (Acetone Washings):** Note the liquid level in the container (see Section 5.5.2) and determine if leakage occurred during transport. If there is a loss of the sample, the test is invalid. Transfer the sample from Container No. 2 into a small (less than 250 mL) tared beaker and evaporate the acetone washings to dryness at room temperature and pressure. Desiccate the sample to a constant weight and record the result (to the nearest 0.1 mg) on the Particulate Analytical Data Sheet (Figure E-4).

**Container No. 3 (Acetone Blank):** Note the liquid level and determine if leakage occurred during transport. Measure the volume of the acetone blank to the nearest 1 mL and enter the value on the Particulate Analytical Data Sheet (Figure E-4). Place the solution in a small tared beaker and evaporate the solution to dryness at room pressure and temperature (or at an elevated

temperature using a steam bath). Desiccate the acetone blank sample to a constant weight. Enter the results to the nearest 0.1 mg in the Particulate Analytical Data Sheet (Figure E-4).

**Container No. 4 (Impinger solution):** Note the liquid level and determine if leakage occurred during transport. Measure the volume of the solution to the nearest 1 mL and enter the value on the Particulate Analytical Data Sheet (Figure E-4). Perform chemical analysis on the impinger solutions as required.

### 5.6 Calculations (see Section 5.7 for Nomenclature)

To simplify record keeping during a test, field data may be entered in the units for which the sampling equipment is designed. These values must be converted, if necessary, to the numeric units specified in the equations where they are used.

**Volume of Stack Gas Sample:** Correct the total sample volume measured by the dry gas meter to reference temperature and pressure conditions (25°C and 101.3 kPa) using Equation E-1.

**Note:** For a temperature-compensated dry gas meter, the average dry gas meter temperature,  $(T_m)_{avg}$ , should be substituted by a constant value as specified by the manufacturer.

$$(V_m)_{ref} = \gamma V_m \frac{T_{ref} (P_{bar} + \Delta H_{avg})}{(T_m)_{avg} P_{ref}} \quad \text{Equation E-1}$$

**Volume of Water Vapour:** Calculate the volume of water vapour in the stack gas sample at reference temperature and pressure conditions using Equation E-2.

$$(V_w)_{ref} = \frac{W_{H_2O} \times 10^{-3} RT_{ref}}{M_{H_2O} P_{ref}} \quad \text{Equation E-2}$$

**Moisture Content of Stack Gas:** Calculate the volumetric fraction of water vapour in the stack gas at reference conditions using Equation E-3.

$$B_{wo} = \frac{(V_w)_{ref}}{(V_w)_{ref} + (V_m)_{ref}} \quad \text{Equation E-3}$$

For saturated or supersaturated stack gas, use a psychrometric chart to determine  $B_{wo}$ .

**Absolute Stack Gas Pressure:** Calculate the absolute stack gas pressure using Equation E-4.

$$P_s = P_{bar} + \Delta p_s \quad \text{Equation E-4}$$

**Stack Gas Molecular Weight:** Calculate the stack gas molecular weight on a wet basis using Equation E-5.

$$M_s = M_d(1-B_{wo}) + M_{H_2O} B_{wo} \quad \text{Equation E-5}$$

**Note:** Use Equation C-2 or C-3 of Method C to calculate  $M_d$ .

**Stack Gas Velocity:** Calculate the stack gas velocity measured at each traverse point using Equation E-6.

$$U_s = 128.95 C_p \left( \frac{\Delta p T_s}{P_s M_s} \right)^{1/2} \quad \text{Equation E-6}$$

**Volumetric Stack Gas Flow Rate:**

Calculate the average volumetric flow rate of the stack gas on a dry basis and at reference conditions using Equation E-7.

$$Q_s = 3600 (U_s)_{avg} A_s (1-B_{wo}) \frac{T_{ref} P_s}{(T_s)_{avg} P_{ref}} \quad \text{Equation E-7}$$

Plant \_\_\_\_\_  
 Location \_\_\_\_\_  
 Test Number \_\_\_\_\_  
 Date \_\_\_\_\_  
 Test Conducted By \_\_\_\_\_

# CONDITIONS OF SAMPLES AS RECEIVED:

- ▶ Container 1 (Filter # \_\_\_\_\_): Observation \_\_\_\_\_
- ▶ Container 2 (Acetone Washings of Nozzle, Probe Liner, Cyclone, and Front-half of Filter Holder:)
  - amount of leakage during transport \_\_\_\_\_ mL
  - amount of liquid in container \_\_\_\_\_ mL
- ▶ Container 3 (Acetone Blank)
  - amount of leakage during transport \_\_\_\_\_ mL
  - amount of liquid in container \_\_\_\_\_ mL
- ▶ Container 4 (First and Second Impinger Contents)\*
  - amount of leakage during transport \_\_\_\_\_ mL
  - amount of liquid in container \_\_\_\_\_ mL

\* only if analysis of the impinger liquid is required

SAMPLE ITEM	FINAL WEIGHT (mg)	TARE WEIGHT (mg)	WEIGHT OF PARTICULATE (mg)
Container 1 Filter # _____			[a]
Container 2 Acetone washings of nozzle, probe liner, cyclone, and front-half of filter holder			[b]
Container 3 Acetone blank (see note 1 below)			[c]

Total weight of particulates collected (see note 2 below) =  $[a] + \{[b] - [c]\}$   
 $W_p = \text{_____ mg}$

Note 1: The acetone used must have < 0.001 percent (by weight) residue, as specified in Section 5.4.2

Note 2: If (b-c) is less than zero,  $W_p = [a]$

**Figure E-4 Particulate Analytical Data Sheet**

**Weight of Particulate Sample:** Determine the weight of the particulate sample,  $W_p$ , from the sum of the particulate weights obtained from Containers No. 1 and 2 less the weight of the residue in the acetone blank, as shown in the Particulate Analytical Data Sheet (Figure E-4).

**Concentration of Particulate Matter:** Calculate the concentration of particulate matter in the stack gas using Equation E-8.

$$C_s = \frac{W_p}{(V_m)_{ref}} \quad \text{Equation E-8}$$

**Mass Emission Rate:** Calculate the mass emission or release rate of particulate matter using Equation E-9.

$$ER_p = 10^{-6} C_s Q_s \quad \text{Equation E-9}$$

**Isokineticity:** Calculate the isokineticity for each traverse point using Equation E-10.

$$I = \frac{\frac{(V_m)_j}{t} \frac{1}{(1 - B_{wo})} (P_{bar} + \Delta H) T_s}{6 \times 10^{-5} \left(\frac{\pi}{4}\right) (N_d)^2 \left(\frac{T_{mi} + T_{mo}}{2}\right) P_s U_s} \times 100$$

$$\text{Equation E-10}$$

A test shall be considered valid with respect to isokineticity (or isokinetic variation) providing that:

- (a) 90% or more of the isokineticity values,  $I$ , calculated for all traverse points are within the range 90 to 110%, i.e.,  $90\% \leq I \leq 110\%$ ; and
- (b) the arithmetic average of all the isokineticity values is within the range 90 to 110%, i.e.,  $90\% \leq I_{avg} \leq 110\%$ .

## 5.7 Nomenclature

$A_s$	- inside cross-sectional area of the stack, duct, or flue, $m^2$
$B_{wo}$	- volumetric fraction of water vapour in the stack gas, dimensionless
$C_p$	- S-type pitot tube coefficient, dimensionless
$C_s$	- concentration of particulate matter in the stack gas on a dry basis at reference temperature and pressure conditions, $mg/m^3$
$ER_p$	- mass emission rate of particulate matter, $kg/h$
$\Delta H_{avg}$	- average pressure drop across orifice meter, $kPa$
$\Delta H$	- pressure drop across orifice meter for each traverse point, $kPa$
$I_{avg}$	- the arithmetic average of all the isokineticity values for the test, %
$I$	- isokineticity i.e., the ratio of the sampling velocity through the nozzle to the velocity of the undisturbed gas stream at each traverse point, %
$j$	- the $j$ th traverse point, dimensionless
$10^{-3}$	- conversion factor, $kg/g$
128.95	- dimensional constant, $(m/s) [(kg/kmol)/K]^{1/2}$
3600	- conversion factor, $s/h$ (seconds/hour)
$10^{-6}$	- conversion factor, $kg/mg$

$6 \times 10^{-5}$	- conversion factor, $(\text{m}^2/\text{mm}^2)(\text{s}/\text{min})$	$T_{\text{mi}}$	- temperature at the dry gas meter inlet for each traverse point, K
$M_{\text{d}}$	- molecular weight of stack gases on a dry basis, kg/kmol	$T_{\text{mo}}$	- temperature at the dry gas meter outlet for each traverse point, K
$M_{\text{s}}$	- molecular weight of stack gases on a wet basis, kg/kmol	$(T_{\text{m}})_{\text{avg}}$	- arithmetic average of the dry gas meter temperatures, K
$M_{\text{H}_2\text{O}}$	- molecular weight of water, 18 kg/kmol	$T_{\text{ref}}$	- reference temperature, 298 K
$N_{\text{d}}$	- inside diameter of the sampling nozzle, mm	$U_{\text{s}}$	- stack gas velocity at each traverse point, m/s
$P_{\text{s}}$	- absolute stack gas pressure, kPa	$(U_{\text{s}})_{\text{avg}}$	- arithmetic average of the stack gas velocities, m/s
$P_{\text{bar}}$	- barometric pressure at the sampling site, kPa	$(V_{\text{w}})_{\text{ref}}$	- volume of water vapour in the stack gas sample at reference temperature and pressure conditions, $\text{m}^3$
$\Delta p$	- pitot tube velocity pressure reading at each traverse point, kPa	$V_{\text{m}}$	- volume of stack gas sample at dry gas meter conditions, $\text{m}^3$
$P_{\text{ref}}$	- reference pressure, 101.3 kPa	$(V_{\text{m}})_{\text{j}}$	- volume of stack gas sample at dry gas meter conditions for the $j^{\text{th}}$ traverse point, $\text{m}^3$
$\Delta p_{\text{s}}$	- static pressure of the stack gases, kPa	$(V_{\text{m}})_{\text{ref}}$	- volume of stack gas sample at reference temperature and pressure conditions, $\text{m}^3$
$Q_{\text{s}}$	- volumetric stack gas flow rate on a dry basis at reference temperature and reference pressure conditions, $\text{m}^3/\text{h}$	$W_{\text{H}_2\text{O}}$	- weight of water vapour condensed in the impingers, g
$R$	- universal gas constant, $8.31 (\text{kPa})(\text{m}^3)(\text{kmol}^{-1})(\text{K}^{-1})$	$W_{\text{p}}$	- total weight of particulate samples collected during the test run, mg
$t$	- sampling duration for each traverse point, min	$\gamma$	- dry gas meter calibration factor (ratio of the wet test meter volume to the dry test meter volume), dimensionless
$(T_{\text{s}})_{\text{avg}}$	- arithmetic average of the stack gas temperatures, K		
$T_{\text{s}}$	- stack gas temperature at each traverse point, K		

## Section 6

# Method F: Calibration Procedure for S-Type Pitot Tube, Dry Gas Meter and Orifice Meter

## 6.1 Calibration of S-Type Pitot Tube

### 6.1.1 Principle

The S-type pitot tube and/or probe assembly are calibrated in a wind tunnel against a standard pitot tube having a known coefficient. This pitot tube must be used in the same configuration as it is calibrated. Once calibrated, it is not necessary to calibrate an S-type pitot tube before every field use, except in the following cases:

- when it is to be used in compliance testing;
- when stubborn accretions of particulate matter have affected the area of the impact openings; and
- when the tube has sustained damage or undergone modifications.

In all cases, calibration data shall be recorded, dated, and signed by the party responsible for the calibration. For compliance testing, all calibration data must be submitted to Environment Canada for review and approval prior to testing.

### 6.1.2 Apparatus

The following items are required:

**Standard pitot tube.** A standard pitot tube to calibrate the S-type pitot tube. The standard pitot tube shall have a known coefficient in the range 0.98 to 1.0. The impact and static hole openings should be free of dirt. It is recommended that standard

pitot tubes used for calibrating other pitot tubes should not be used in dirty gas streams.

**S-type pitot tube.** Also known as the Stausscheibe or reverse type pitot tube. The S-type pitot tube may be used alone, attached to a temperature sensor, or mounted on a probe.

**Differential pressure indicators.** Two differential pressure devices such as inclined manometers to measure the velocity pressures to within 0.1 mm (0.005 in) H<sub>2</sub>O on the 0 to 25 mm (0 to 1.0 in) H<sub>2</sub>O range and 1 mm (0.05 in) H<sub>2</sub>O on the 25 to 250 mm (1 to 10 in) H<sub>2</sub>O range. Equivalent differential pressure devices such as micromanometers may be used provided that they are calibrated against a primary pressure standard before use.

**Wind tunnel.** A flowing gas stream confined in a duct with a constant cross section that is either circular or rectangular. The minimum cross-sectional area must be 5600 cm<sup>2</sup> (864 in<sup>2</sup>). The flow system must be capable of producing variable velocities [from 3 m/s to at least 20 m/s (10 to 60 ft/s)]. This generated velocity must have no temporal variations to ensure steady flow during calibration. The test site must be situated at least eight diameters downstream and two diameters upstream from a flow disturbance. Deviations from the eight- and two-diameter criteria are acceptable provided that it can be demonstrated that the flow is stable and parallel to the longitudinal axis of the duct. The test section of the wind tunnel shall contain two test ports, one each for the



standard and S-type pitot tubes. It is recommended that the working section be constructed of a transparent material to facilitate the positioning and alignment of the probe assembly and pitot tubes.

### 6.1.3 Procedure

Locate the two calibration points. The two points should be near the centre of the tunnel separated by at least two tunnel diameters distance. Before the S-type pitot tube (mounted or unmounted) is calibrated, measure the velocity pressures ( $\Delta p$ ) at each point to ensure that the velocities are the same. This may be done by measuring  $\Delta p$  with two standard pitot tubes simultaneously or by alternately measuring the  $\Delta p$  at each site with a single standard pitot tube. Position the standard pitot tube (reference) with known coefficient near the centre of the wind tunnel and position the S-type pitot tube a minimum of two tunnel diameters downstream of the reference. Ensure that both pitots are facing directly into the gas stream and that both ports are completely sealed. Connect the manometers to the pitot tubes and ensure that they are filled with the fluid of proper density. Inspect and leak check all pitot lines and level and zero the manometers.

Record the ambient temperature and barometric pressure. Turn on the wind tunnel fan and allow the flow to stabilize. Read the velocity pressure heads from the standard pitot tube ( $(\Delta p)_{std}$ ) and the S-type pitot tube  $\Delta p$  and record these values in Figure F-1. At least five velocities must be generated that cover the expected velocity range in the field.

In the case of single S-type pitot tubes with and without a mounted thermocouple, both legs of the pitot tube must be identified e.g., "A" and "B". It is not necessary to

calibrate with both legs facing the flowing stream; however, the pitot tube must be used in the field in the same way it was calibrated in the flowing gas stream. In the case of a probe assembly (pitot tube, thermocouple, and nozzle), it is recommended that the probe assembly be calibrated with the anticipated nozzle to be used in the field.

The wind tunnel must have a sufficiently large cross-sectional area in relation to the projected area of a probe assembly. The projected area (Figure F-2) of the probe must be at most 2% of the duct area to obtain reliable coefficients. When the projected area of the probe exceeds this criterion, the blockage area may be reduced by moving the probe back a few inches or by using a larger wind tunnel.

### 6.1.4 Calculations

**Pitot tube coefficient ( $C_p$ ).** Calculate the pitot tube coefficient for each wind tunnel setting using Equation F-1. The coefficient for the pitot tube is calculated by averaging the individual coefficients.

$$C_p = (C_p)_{std} \left( \frac{(\Delta p)_{std}}{\Delta p} \right)^{1/2} \quad \text{Equation F-1}$$

Calculate the absolute differences between the average pitot tube coefficient and the coefficient for each of the wind tunnel settings. The average of these differences must not exceed 0.01.

### 6.1.5 Nomenclature

- $C_p$  - coefficient of the S-type pitot tube (mounted or unmounted)
- $(C_p)_{std}$  - coefficient of the standard pitot tube (if unknown use 0.99)
- $\Delta p$  - velocity pressure measured by the S-type pitot tube, kPa

$(\Delta p)_{std}$  - velocity pressure measured by the standard pitot tube, kPa

## 6.2 Calibration of Dry Gas Meter and Orifice Meter

### 6.2.1 Principle

The dry gas meter and the orifice meter of the particulate sampling train shall be calibrated against a primary standard before each compliance testing sampling survey. The following procedure is based on a wet test meter that was calibrated against a primary standard.

### 6.2.2 Apparatus

A wet test meter or equivalent device capable of measuring the volume within (plus or minus) 1% accuracy at a rate approximating that of the dry gas meter (e.g., 30 L (1 ft<sup>3</sup>) per revolution) shall be used.

### 6.2.3 Procedure

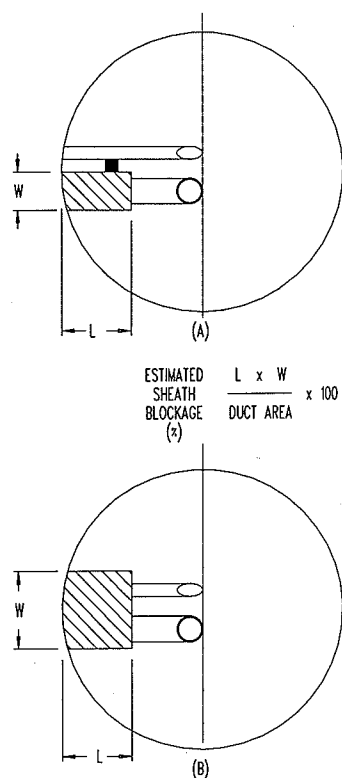
Before calibrating the dry gas and orifice meters, the pressure and vacuum side of the sampling console must be checked for leaks. The following procedure is recommended for leak checking that portion of the train

Date			Pitot Tube No.	
Location			T/C Attached?	
Operators			Probe Mounted?	
Ambient Temperature (°C)			Sheathed?	
Barometric Pressure (kPa)			Nozzle Diameter (mm)	
Wind Tunnel				
Wind Tunnel Setting (rpm)	Standard Pitot Tube Reading (mm H <sub>2</sub> O) $(\Delta p)_{std}$	S-type Pitot Tube Reading (mm H <sub>2</sub> O) $\Delta p$	Coefficient (C <sub>p</sub> ) $[(\Delta p)_{std} / \Delta p]^{1/2} (C_p)_{std}$	Difference $ \bar{C}_p - C_p $

Average \_\_\_\_\_

Average \_\_\_\_\_ Difference  $(\bar{C}_p)$

Figure F-1 Pitot Tube Calibration Data Sheet



**Figure F-2 Projected Area Models for Typical Probe Assemblies**

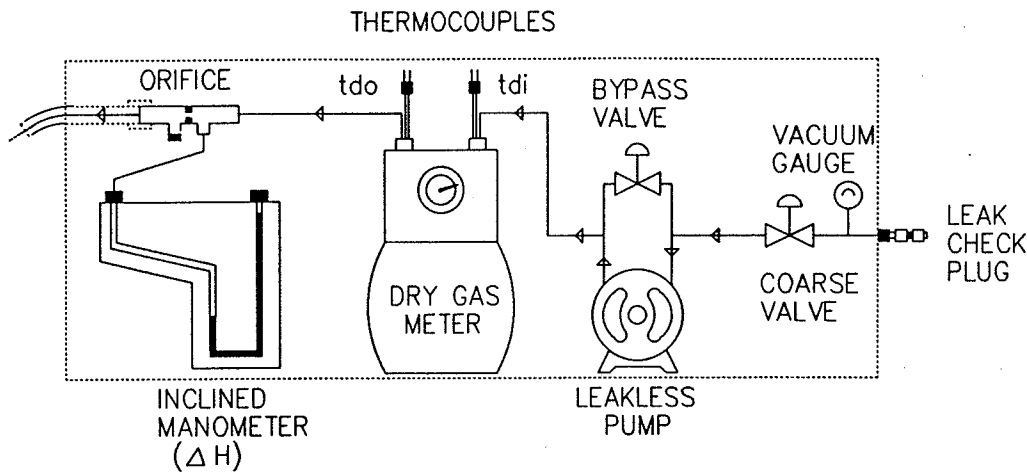
from the pump to the orifice meter (Figure F-3).

Close the main shut-off valve on the meter box. Attach a flexible tube to the orifice exhaust pipe. Disconnect and vent the low pressure side of the orifice manometer and plug or cap the low side of the orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in) H<sub>2</sub>O by blowing into the flexible tubing. Pinch the tubing and observe the manometer reading for one minute. A loss of pressure on the manometer indicates a leak between the pump and orifice meter. Correct the leak before calibration.

The vacuum or suction side of the pump may be checked by the following procedure. Plug the suction (sample) line going into the sampling console and open the coarse control valve. Start the pump and adjust the fine control (by-pass) valve to a vacuum of

380 mm (15 in) Hg. Check the dry gas meter needle for movement. No movement on the dial should be indicated following the initial purge of the line. Any leaks must be corrected before proceeding to the calibration procedure.

Connect the components as shown in Figure F-4. Level all components, zero all manometers, and inspect and leak check all sample lines. Operate the equipment with the orifice manometer set at 13 mm (0.5 in) H<sub>2</sub>O for at least 15 minutes to allow the pump to warm up and to permit all interior surfaces of the wet test meter to become wetted. Perform the tests by drawing the desired volume of air through the wet test meter at each of the orifice manometer settings. Record the dry test meter volumes, times, temperatures, and manometer readings in Figure F-5.



**Figure F-3 Leak Check of Meter Box**

#### 6.2.4 Calculations

##### Dry gas meter correction factor ( $\gamma$ ).

Calculate  $\gamma$ , the dry gas meter correction factor for each orifice setting by using Equation F-2.

$$\gamma = \frac{V_w}{V_d} \frac{(P_{bar} + \Delta m)}{(P_{bar} + \Delta H)} \frac{T_d}{T_w} \quad \text{Equation F-2}$$

where:  $T_d = \frac{T_{di} + T_{do}}{2}$

If the average value of  $\gamma$  is not within the range 0.95 to 1.05, the dry test meter must be adjusted internally and the calibration procedure repeated. Each value of  $\gamma$  must be within (plus or minus) 1.5% of the average value. If not, the calibration must be repeated.

**Orifice meter coefficient ( $K_o$ ).** The flow through an orifice meter for each orifice setting may be determined by using Equation F-3.

$$Q_m = K_o \left( \frac{\Delta H T_{do}}{(P_{bar} + \Delta H) M_s} \right)^{1/2} \quad \text{Equation F-3}$$

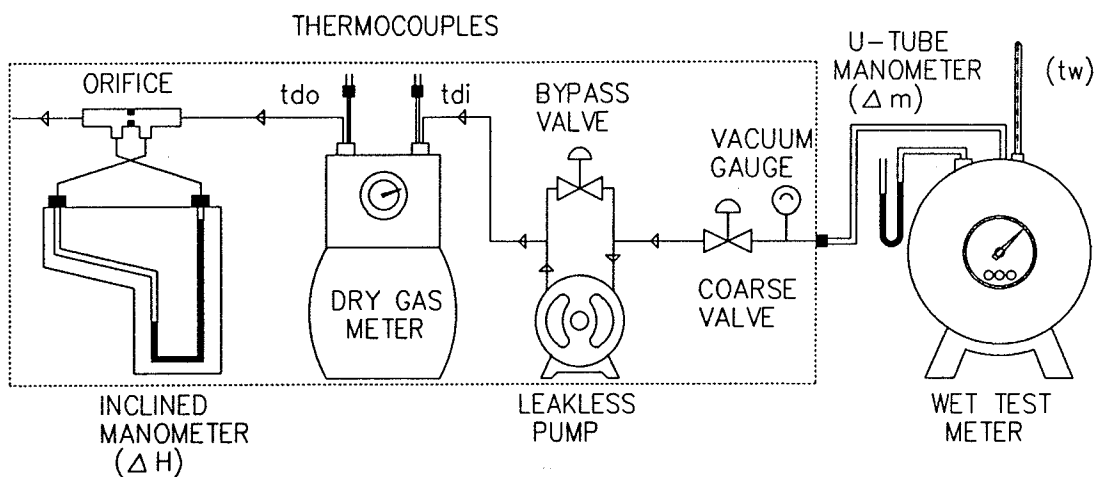
Alternately,  $Q_m$  may also be expressed in terms of the flow rate through the wet test meter using Equation F-4.

$$Q_m = \frac{V_w T_{do} (P_{bar} + \Delta m)}{\theta T_w (P_{bar} + \Delta H)} \quad \text{Equation F-4}$$

The  $K_o$  value for the orifice may be obtained by averaging the individual  $K_o$  at each orifice setting which are calculated by equating Equation F-3 to Equation F-4 and solving for  $K_o$ .

#### 6.2.5 Nomenclature

$B_{wo}$  - proportion by volume of water vapour (saturated) passing through the wet test meter calculated from partial pressure data published in technical handbooks, dimensionless



**Figure F-4 Assembly for Calibration of Dry Gas Meter and Orifice Meter**

$\Delta H$	- pressure drop across the orifice meter, kPa	$T_d$	- average temperature of dry gas meter, K
$K_o$	- orifice coefficient ( $\text{m}^3/\text{min}$ ) $[(\text{kg}/\text{kmol})/\text{K}]^{1/2}$	$T_{di}$	- dry gas meter inlet temperature, K
		$T_{do}$	- dry gas meter outlet temperature, K
$\Delta m$	- wet test meter manometer reading, kPa	$T_w$	- temperature of the wet test meter, K
$M_s$	- molecular weight of saturated air, kg/kmol	$V_d$	- actual volume drawn through dry gas meter, $\text{m}^3$
	$M_s = 28.95 (1 - B_{wo}) + 18B_{wo}$	$V_w$	- actual volume drawn through wet test meter, $\text{m}^3$
$P_{\text{bar}}$	- barometric pressure, kPa	$\gamma$	- the dry gas meter correction factor, dimensionless
$Q_m$	- flow rate through the orifice meter, $\text{m}^3/\text{min}$	$\theta$	- time, min.

Date \_\_\_\_\_ Barometric Pressure ( $P_{\text{bar}}$ ) \_\_\_\_\_ kPa

Dry Gas Meter No. \_\_\_\_\_ Sampling Train Type \_\_\_\_\_

Orifice Setting (mm H <sub>2</sub> O) $\Delta H$	Gas Volumes (m <sup>3</sup> )				Temperatures (°C)				Elapsed Time (min.) $\Theta$	Wet Test Meter Manometer Reading (mm H <sub>2</sub> O) $\Delta m$	Coefficients	
	* Wet Test Meter $V_w$	Dry Gas Meter			Wet Test Meter $T_w$	Dry Gas Meter					$K_o$	$\gamma$
		Finish	Start	Total, $V_d$		In $T_{di}$	Out $T_{do}$	Avg $T_d$				
10												
20												
25												
40												
50												
70												
Average												

\* The volume drawn through the wet test meter should be selected such that the elapsed time is between 5 and 10 minutes.

Figure F-5 Dry Gas Meter and Orifice Meter Calibration Data Sheet

## References

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1. Standard D1193-77 (Reapproved, 1983), "Standard Specification for Reagent Water", Annual Books of ASTM Standards, Volume 11.01 Water (1) (1987).
2. Standard D2986-71 (Reapproved, 1978), "Standard Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diethyl phthalate) Smoke Test", Annual Books of ASTM Standards, Volume 11.03 Atmospheric Analysis and Occupational Health and Safety (1990).

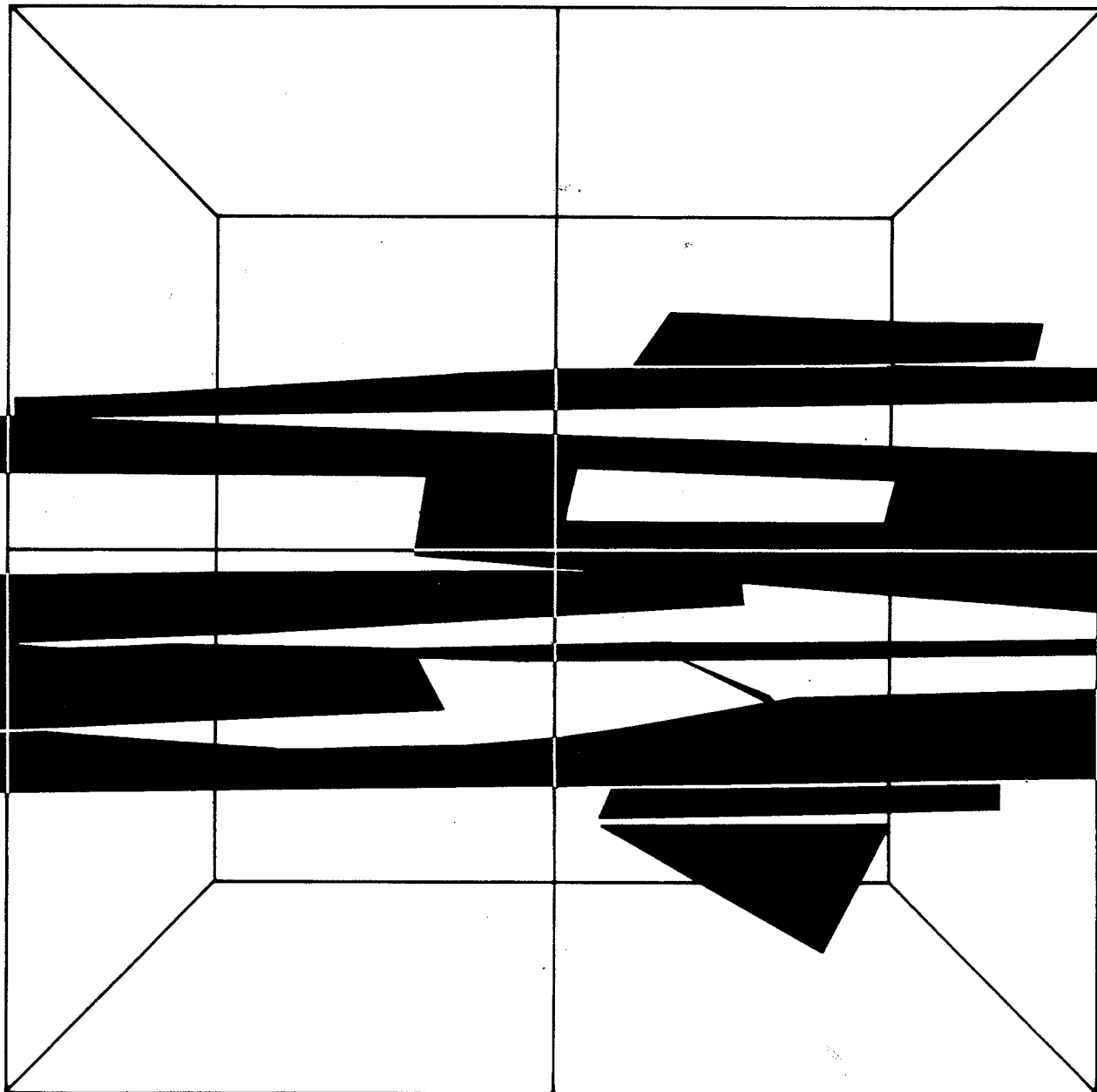
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1. Ministry of the Environment, Province of Ontario, "Source Testing Code (version #2)", Report # ARB-TDA-66-80 (November, 1980).
2. United States Environmental Protection Agency, Stationary Source Sampling Methods Notebook (NSPS and NESHAPS Methods), The McIlvaine Company (Compiled by Walter S. Smith, P.E., Entropy Environmentalists) (1984).

# Reference Method for Source Testing: Measurement of Releases of Selected Semi-volatile Organic Compounds from Stationary Sources

Report EPS 1/RM/2  
June 1989



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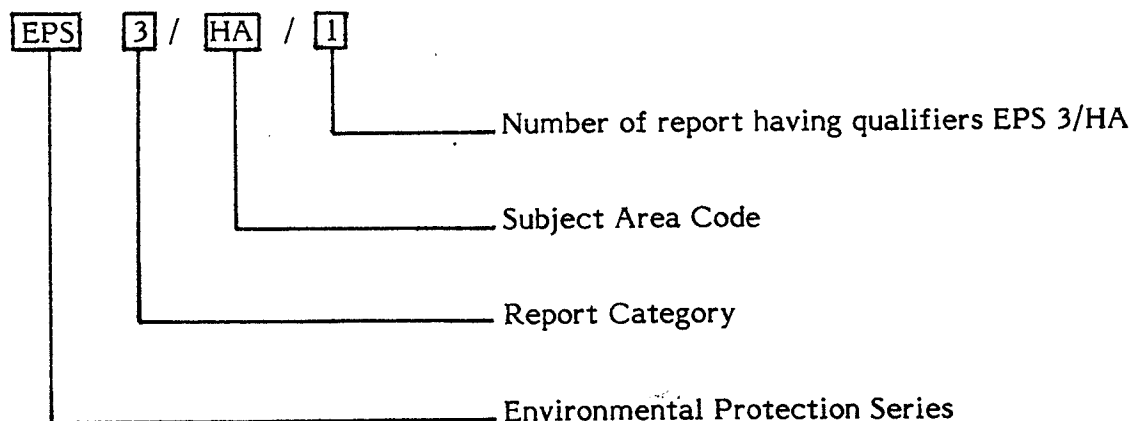
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**REFERENCE METHOD FOR SOURCE TESTING: MEASUREMENT OF RELEASES OF  
SELECTED SEMI-VOLATILE ORGANIC COMPOUNDS FROM STATIONARY SOURCES**

Pollution Measurement Division  
River Road Environmental Technology Centre  
Conservation and Protection  
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## FOREWORD

The Environment Canada Reference Method (RM) described in this document is used to measure the releases of selected semi-volatile organic compounds from stationary sources. This method is used in conjunction with those described in Environment Canada Report EPS 1-AP-74-1, "Standard Reference Method for Source Testing: Measurement of Emissions of Particulates from Stationary Sources", as amended. The complexity of method procedures warrants that personnel performing them be trained and experienced.

Application of this RM for compliance testing requires strict adherence to the method in all respects. Deviation from the method may invalidate the test results. Any changes in equipment, reagents, materials, procedures, or calculations from those specified in the RM must be approved in writing by Environment Canada prior to testing. If deviations are made without prior approval, the validities of the tests shall be determined by Environment Canada on a case-by-case basis.

NOTE: Mention of trade names or commercial products does not constitute endorsement for use by Environment Canada.

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## 1 APPLICABILITY

This Environment Canada Reference Method (RM) is applicable to the determination of emissions of polychlorinated dibenzo-*para*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), polychlorinated biphenyls (PCBs), and other semi-volatile organic compounds (SVOCs) from stationary sources. Semi-volatile organics are defined as compounds with boiling points greater than 100°C. The testing procedures in Method A,B,C,D and F, contained in Environment Canada Report EPS 1-AP-74-1, "Reference Methods for Source Testing: Measurement of Emissions of Particulates from Stationary Sources" (as amended), are to be used in conjunction with the SVOC method:

- Method A - Determination of Sampling Site and Traverse Points
- Method B - Determination of Stack Gas Velocity and Volumetric Flow Rates
- Method C - Determination of Molecular Weight by Gas Analysis
- Method D - Determination of Moisture Content
- Method F - Calibration Procedure for S-Type Pitot Tube, Dry Gas Meter and Orifice Meter.



## 2 PRINCIPLE

An integrated stack gas sample is isokinetically withdrawn from a number of traverse points along the stack cross section. Semi-volatile organic compounds associated with particulate matter are collected in the front-half components of the sampling train. Semi-volatile organic compounds not collected by the high efficiency glass or quartz fibre filter are adsorbed on a porous, polymeric resin, Amberlite XAD-2.

### 3 APPARATUS

#### 3.1 Sampling Train (Figure 1)

The following equipment is required:

Nozzle: A button-hook type nozzle with a sharp, tapered leading edge. The nozzle is usually made of 316 stainless steel or Incoloy 825, but quartz or equivalent material may be used when high temperature or corrosive gases are encountered. The minimum inside diameter of the nozzle shall be 4.76 mm (3/16 inch).

Probe: A borosilicate or quartz glass liner, encased in a stainless steel tube with a heating and temperature indicating system capable of maintaining the exit gas temperature at  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ), or at such a temperature necessary to prevent condensation. Where length or strength limitations preclude the use of a glass liner, a seamless tubing made from an inert material such as Teflon may be used. A water-cooled probe should be used when very hot gases which could damage the nozzle/probe assembly are encountered.

Pitot Tube: A calibrated S-type (Stausscheibe) pitot tube mounted on the probe assembly.

Stack Temperature Sensor: A thermocouple, or other suitable temperature sensor, mounted on the probe assembly, capable of measuring the stack temperature to within 1.5% of the minimum absolute stack temperature. When high temperature gases are encountered, appropriate shielding and aspiration should be provided for the thermocouple so that radiation effects are avoided.

Cyclone (Optional): A borosilicate miniature cyclone placed after the sampling probe and before the filter may be used to collect large particles. The cyclone prevents premature buildup of particulate matter on the filter medium and permits longer sampling periods. The cyclone is located inside the filter compartment and is kept at a temperature of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ).

Filter Holder: A borosilicate filter holder with an inert filter support. The filter support, which consists of a perforated disc, shall be constructed of Teflon to provide a seal against leakage at any point along the filter circumference. The filter holder is located inside the filter compartment and is maintained at a temperature of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ).

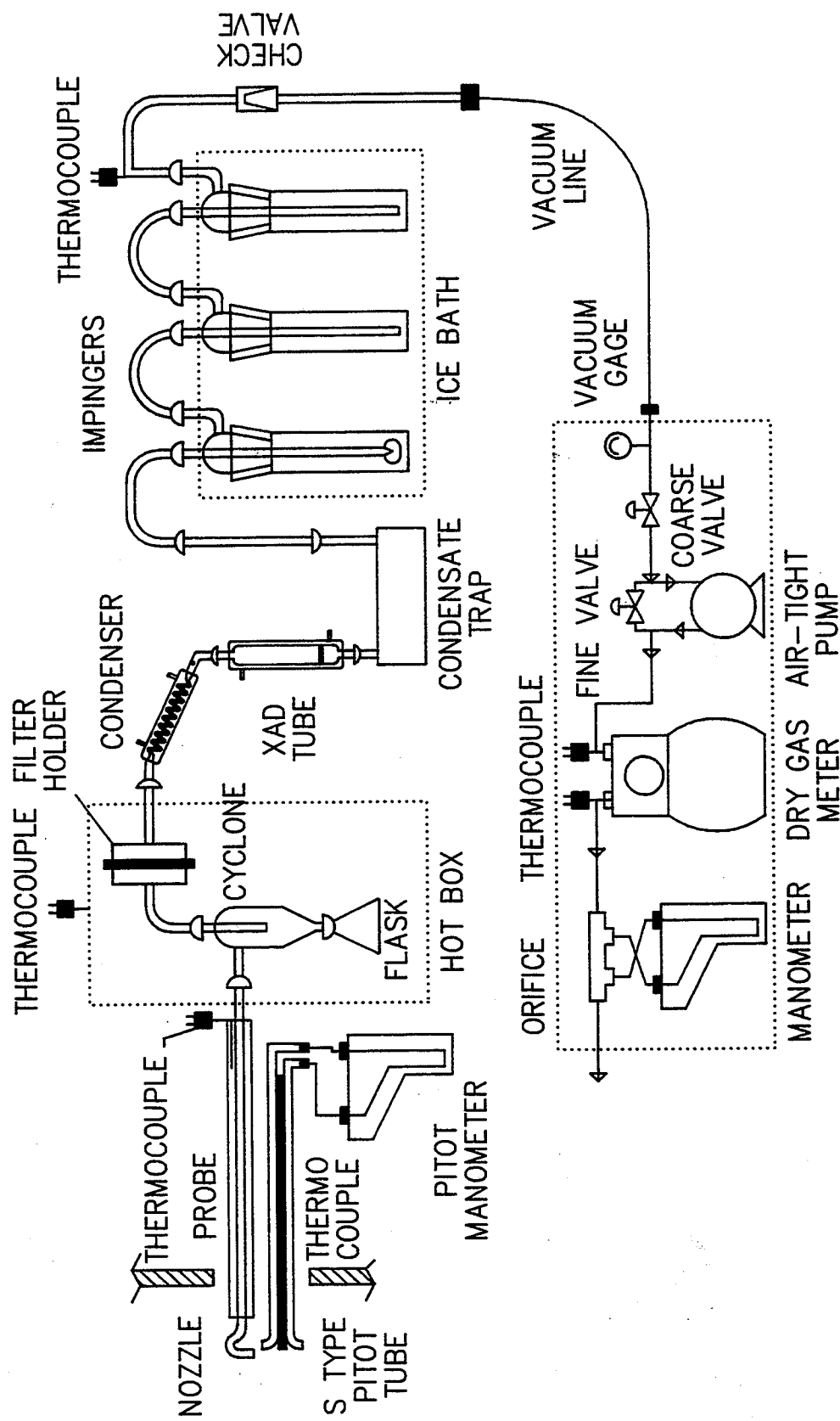


FIGURE 1 SEMI-VOLATILE ORGANICS SAMPLING TRAIN

Filter: A high-efficiency glass or quartz fibre filter, which contains no organic binders and is capable of at least 99.95% efficiency for 0.3  $\mu\text{m}$  dioctyl phthalate smoke particles as determined by ASTM Method D2986-71.

Filter Compartment Heating System: A heating system capable of maintaining the temperature in the filter holder compartment at  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ), or at such a temperature necessary to prevent blinding of the filter. A thermocouple or other temperature sensor must be able to read the filter compartment temperature to within  $3^{\circ}\text{C}$  ( $5^{\circ}\text{F}$ ).

Organic Sampling Module: This unit consists of a gas condensing coil, a sorbent trap and a condensate knockout trap. The condenser must be able to condition the stack gas leaving the back-half of the filter holder at a temperature below  $20^{\circ}\text{C}$  ( $68^{\circ}\text{F}$ ). The sorbent trap shall be sized to contain 30-40 g of porous polymeric resin, Amberlite XAD-2. The sorbent trap should contain a glass well to accommodate a thermocouple to permit monitoring of the temperature of the gas sample entering the sorbent. The organic sampling module shall be oriented so that the condensate flows vertically, downward through the XAD-2 resin, into the condensate trap. The condensate trap shall be suitably sized and designed to prevent bubbling and carryover of condensate into the impingers.

A peristaltic or submersible pump shall be used to circulate the coolant through the XAD-2 trap and condenser. Ice water from the impinger bath may be used as the coolant.

All components of the organic module (condenser, XAD-2 trap and condensate trap) shall be fabricated of borosilicate glass. These components are not available commercially, but they may be custom fabricated from available laboratory glassware.

Impingers: Three Greenburg-Smith impingers are connected in series. The second and third impingers are modified by replacing the tips and impaction plates of the standard design with a 13 mm (0.5 inch) inside diameter glass tube extending to within 13 mm (0.5 inch) from the bottom of the impinger. The first impinger has the standard tip and impaction plate. The impingers are contained in an ice bath during sampling. The first impinger shall contain a known volume of ethylene glycol. The second shall remain empty and the third shall contain a known weight of silica gel.

Vacuum Pump: A leakproof vacuum pump capable of maintaining an isokinetic sampling rate while continuously withdrawing a portion of the stack gases through the sampling

train is used. The pump intake vacuum is measured to within 13 mm mercury (Hg) (0.5 inch Hg) by a vacuum gauge which is attached to the vacuum line connecting the pump to the last impinger outlet. The sample flow rate is controlled with both coarse-and fine-flow control valves.

Dry Gas Meter: A calibrated dry gas meter with inlet and outlet temperature sensors, or one that is temperature-compensated. The sensors must be capable of measuring the temperatures to within 3°C (5°F).

Orifice: A calibrated orifice connected to the outlet of the dry gas meter is required.

Differential Pressure Gauges: Differential pressure gauges such as an inclined manometer, or a device of equal sensitivity, which are capable of measuring the pitot tube velocity pressure and the pressure drop across the orifice to within 0.13 mm (0.005 inch) H<sub>2</sub>O on the 0 to 25 mm (0 to 1 inch) H<sub>2</sub>O scale, and readable to the nearest 1.3 mm (0.05 inch) H<sub>2</sub>O for  $\Delta P$  values greater than 25 mm H<sub>2</sub>O.

A more sensitive device shall be required when  $\Delta P$  values are less than 1.3 mm H<sub>2</sub>O (0.05 inch H<sub>2</sub>O).

Barometer: A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 inch Hg). Alternatively, the atmospheric pressure provided by the local weather office may be used with an adjustment for the elevation of the sampling site. Deduct 2.5 mm Hg (0.1 inch Hg) per 30.5 m (100 feet) of elevation increase or add 2.5 mm Hg (0.1 inch Hg) per 30.5 m (100 feet) of elevation decrease.

### 3.2 Sample Preparation and Recovery

Probe Brush: A nylon bristle brush of a length and diameter suitable for cleaning the probe. The brushes shall be cleaned prior to field use and shall have stainless steel or Teflon extensions.

Wash Bottles: Teflon wash bottles are necessary to dispense the recovery solvents. Polyethylene wash bottles shall not be used as the organic solvents stored in the bottle may extract organic contaminants.

Sample Storage Containers: Wide-mouthed amber glass bottles to store the hexane and acetone rinse. The screw caps shall be lined with Teflon lid liners. Both the amber glass bottles and the Teflon lid liners shall be cleaned. The amber glass bottles shall be proofed prior to field use. Both 500-mL and 1000-mL capacity bottles are required.

Petri Dishes: Glass petri dishes to store and transport the filters. The glass petri dishes shall be cleaned before field use.

Teflon Lid Liners: Teflon sheet to line the inside of the screw caps. The lid liners shall be cleaned prior to field use.

Teflon Tape and Sleeves: Teflon tape is necessary to seal the circumference of the filter holder and the glass ball joints. Both 13-mm (1/2-inch) and 25-mm (1-inch) wide tape is required. The Teflon sleeves are used to seal the tapered impinger joints.

Glass Wool: Glass or quartz wool to plug the unfritted end of the sorbent trap. The wool shall be cleaned prior to field use.

Aluminum Foil: Aluminium foil to wrap the loaded filter and to cover the open ends of the train glassware. The foil shall be cleaned before use.

Miscellaneous: Miscellaneous glassware such as graduated cylinders, glass plugs and caps and funnels. This glassware shall be cleaned prior to field use. Tweezers (stainless steel or Teflon) and disposable plastic gloves are also required. Do not use latex gloves or gloves with powdered inside surfaces.

## REAGENTS

All reagents used for organic testing shall be analyzed for organic contaminants prior to sampling. These reagents include the cleaning and rinsing solvents, High Purity Liquid Chromatography (HPLC) water, ethylene glycol and the XAD-2 resin.

Solvents: Solvents used for sampling shall be "Distilled-in-Glass" grade or "Pesticide Grade". These include methylene chloride (dichloromethane) hexane and acetone. Solvents may be screened by lot or batch number. Solvents from different batches will require a separate analysis.

HPLC Water: HPLC water for sample recovery is required.

Ethylene Glycol: Distilled-in-glass grade ethylene glycol shall be added to the first impinger. The glycol serves as a backup collection medium should organics break through the XAD-2 resin.

Amberlite XAD-2 Resin: Porous polymeric resin, XAD-2, is necessary to trap the gaseous semi-volatile organic compounds. The resin shall be cleaned and proofed before field use. Once cleaned, the XAD-2 should be stored in wide-mouthed amber glass jars with Teflon-lined caps and wrapped in aluminum foil. Resin not used within four weeks of preparation should be re-cleaned. The analytical procedure and acceptance criteria are described in the method specified in the Chemical Analysis Section. All XAD-2 traps (including the blank train and reagent blanks) must be loaded at the same time in a clean environment.

Ice: Crushed ice is required to maintain the impinger bath at a constant temperature.

Silica Gel: Indicating-type 6-16 mesh silica gel that has been dried at 180°C (356°F) for two hours is required.

## 5 PROCEDURES

### 5.1 Cleaning and Proofing

Before the field tests, all train glassware, amber storage bottles, Teflon lid liners, probe brushes, glass wool, petri dishes and aluminum foil must be cleaned as described in Table 1. The cleaning procedures also apply to miscellaneous items such as graduated cylinders, plugs and caps, funnels and tweezers. Other cleaning procedures are recognized, but the components must be proofed according to the procedures detailed in Table 1 and pass the acceptance criteria.

#### **Caution: Handle all Solvents in a Well-Ventilated Area**

The effectiveness of the cleaning procedures is verified by proofing selected components and reagents. Proofing ensures that the glassware, reagents and recovery solvents are free of pollutants prior to sampling. All components coming into contact with the stack gas or recovery samples must be cleaned, but it is not necessary to proof all the cleaned components. The proofing samples are analyzed as specified in Section 6, using High Resolution Gas Chromatography/Low Resolution Mass Spectrography. The components and reagents that require proofing are as follows:

Train Glassware: Glassware cannot be re-used in the field with semi-volatile organic sampling. As a result, one complete set of pre-cleaned and proofed glassware must be allowed for each stack test. In addition, one complete set of glassware must also be allowed for the blank train. Assemble the complete sets of train glassware that will be needed and identify each set. Following the cleaning procedure described in Table 1, rinse each component from a train set three times each with hexane and acetone. Combine the rinses from each train into a pre-cleaned amber bottle with a pre-cleaned Teflon lid liner. Label the container. This sample constitutes the proof rinse for that train set. Repeat the above procedure for the other train sets and the blank train. Submit the proof rinses (one bottle per train) to the analytical lab for analysis.

Amber Glass Storage Bottles: Following the cleaning procedure, select three or four bottles from each box (48 bottles). Rinse these bottles three times each with hexane and acetone. Combine rinsings from each bottle in a pre-cleaned amber bottle with a pre-cleaned Teflon lid liner. Label the container and submit the proof rinse to the analytical laboratory. One proof rinse shall be submitted for each box of bottles.



TABLE 1 - CLEANING AND PROOFING PROCEDURES

Component	Cleaning	Proofing
Glassware including Teflon Filter Supports* Probe Brushes* Petri dishes* Glass plugs* Glass caps* Graduated Cylinders* Funnels* Tweezers*	<ul style="list-style-type: none"> <li>- Soak in RBS solution at 70°C for at least two hours. Scrub glassware with a brush. Discard RBS solution after each batch.</li> <li>- Wash twice in dishwasher with lab dish detergent.</li> <li>- Rinse with generous amounts of deionized water.</li> <li>- Rinse each component three times with methylene chloride.</li> <li>- Rinse each component three times with hexane.</li> <li>- Rinse each component three times with acetone.</li> <li>- Seal all open ends with pre-cleaned aluminum foil.</li> </ul>	<ul style="list-style-type: none"> <li>- Assemble as many complete sets of trains as glassware allows.</li> <li>- Identify each train set.</li> <li>- Wash each component from each train three times each with hexane and acetone. Combine all rinses from each train into a pre-cleaned amber bottle with pre-cleaned Teflon lid liner. (Do not use methylene chloride)</li> <li>- Submit one proof rinse for each train.</li> </ul>
Amber glass bottles	<ul style="list-style-type: none"> <li>- As above except that no scrubbing with brushing is required.</li> </ul>	<ul style="list-style-type: none"> <li>- Select 3-4 bottles from each box (48 bottles). Rinse three times each with hexane and acetone. Store rinsings from each bottle in a pre-cleaned amber bottle with pre-cleaned Teflon lid liner.</li> </ul>
Teflon lid liners, Aluminum foil	<ul style="list-style-type: none"> <li>- Cut to size - Soak at least three hours in methylene chloride. (Cover beaker, replace MC daily)</li> <li>- Rinse three times with hexane.</li> <li>- Rinse three times with acetone. (Note: Handle with tweezers only).</li> </ul>	Proofing not required.
Glass wool	<ul style="list-style-type: none"> <li>- Elute in a column with 500 mL hexane followed by 500 mL methylene chloride. Oven dry for 8 hours at 225°C.</li> </ul>	Proofing not required.
Amberlite XAD-2	<ul style="list-style-type: none"> <li>- Rinse with deionized water.</li> <li>- Sequential soxhlet extraction with methanol, methylene chloride and cyclohexane (20 hours each). Rinse with hexane (5 cycles).</li> <li>- Oven dry at 50°C for four hours in shallow trays lined with pre-cleaned aluminum foil.</li> <li>- Store in tightly-sealed, pre-cleaned glass bottles.</li> </ul>	<ul style="list-style-type: none"> <li>- Submit a 30 g aliquot from each drying tray for proofing analysis.</li> </ul>

\* Proofing not required for these components.

Notes:

- all reagents distilled-in-glass grade
- avoid latex gloves or gloves with powdered inside surface
- all squeeze bottles made out of Teflon
- handle all solvents in a well-ventilated area

Amberlite XAD-2 Resin: A 30 g aliquot of each cleaned batch is taken and analyzed. This procedure is described in greater detail in the analytical method specified in Section 6.

Hexane and Acetone: Submit a sample of each solvent in a pre-cleaned amber bottle with a pre-cleaned Teflon lid liner. Label each container, including the batch or lot number. Take an aliquot of each solvent to form a combined hexane/acetone proof rinse. Submit the combined rinse to the analytical lab for analysis.

Ethylene Glycol and HPLC Water: Submit a sample of each reagent in a pre-cleaned amber bottle with a pre-cleaned Teflon lid liner. Label each container and include the batch or lot number. Take an aliquot of each reagent to form a combined glycol/HPLC water proof rinse. Submit the combined rinse to the analytical lab for analysis.

## 5.2 Sample Collection

Preliminary: Select the sampling site and the minimum number of traverse points according to procedures described in Method A of Environment Canada Report EPS 1-AP-74-1, "Reference Method of Source Testing: Measurement of Emissions of Particulates from Stationary Sources" (as amended). Without previous knowledge of the stack variables, a preliminary test should be conducted to obtain the following data:

- velocity profile across the stack (Method B)
- stack temperature and pressure (Method B)
- stack gas molecular weight (Method C)
- stack gas moisture content (Method D)

Use the information to select the largest nozzle possible for isokinetic sampling. The recommended minimum nozzle size is 4.76 mm (3/16 inch) inside diameter. A nozzle size should be selected such that 3 to 4 m<sup>3</sup> (105.9 - 141.3 ft<sup>3</sup>) of dry standard sample gas will be collected over an approximate four-hour sampling period.

Select a suitable probe length that will permit access to all the sampling points.

Select a total sampling time so that the sampling time per traverse point is equal to or greater than five minutes.

Sampling Train Preparation: Prepare the sampling train in a clean area to minimize contamination. Install the selected nozzle on the probe. Mark the probe to denote the

location of each sampling point. Before preparing and assembling the train glassware, the components should be rinsed once each with hexane and acetone. Discard the rinses.

Carefully wrap the filter support with 25 mm (1-inch) Teflon tape along the circumference to provide a leak-free seal between both halves of the filter holder. Using a pair of tweezers, remove the filter from its petri dish, inspect for flaws and place on the perforated Teflon filter support. It is recommended that the filter assembly be "leak-checked" at 381 mm Hg (15 inches Hg) prior to assembly at the sampling site.

Add approximately 100 mL of ethylene glycol to the first impinger and about 200-300 g of silica gel to the third impinger. Weigh the condenser, XAD-2 trap, condensate trap and the three impingers to the nearest 0.5 g. Record each weight on the Moisture Analysis Data Sheet (Figure 2).

During the assembly of the train, the joints must be sealed by wrapping the ball joints with 13-mm (0.5-inch) Teflon tape. Do not use vacuum grease to seal the joints. The configuration of the glassware will determine to what extent the train can be assembled in the field lab. From train assembly to extraction of the stack sample, the open glassware joints must be sealed with pre-cleaned glass plugs or caps or aluminum foil at all times.

At the sampling site, set up the train as shown in Figure 1. Connect all temperature sensors and pitot lines. Adjust the filter compartment and probe heating systems to maintain a temperature of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ). Connect the cooling system. Do not add the water or ice until the pre-test leak check is completed.

Once the filter compartment and probe have reached the necessary temperatures, conduct a leak check on the sampling train. This is done by plugging the nozzle inlet and pulling a vacuum of 381 mm Hg (15 inches Hg). A leakage rate of less than 0.57 L/min (0.02 ft<sup>3</sup>/min) or four percent of the estimated average sampling rate, whichever is less, is acceptable. Unacceptable leakage rates must be corrected. Record the leakage rate and the vacuum on the Organics Sampling Data Sheet (Figure 3). Place water and crushed ice in the impinger bath and turn on the coolant recirculating pump for the condenser coil and XAD-2 tube. During testing, the temperature of the XAD-2 must not exceed 20°C (68°F) for effective removal of the semi-volatile organic species. At all other times the XAD-2 must not be exposed to temperatures above 50°C (122°F) to avoid thermal degradation.

Sampling Train Operation: Verify the heating and cooling systems and check that the probe and pitot tube are properly aligned. Level and zero the manometer. Clean the

Plant \_\_\_\_\_  
 Location \_\_\_\_\_  
 Run Number \_\_\_\_\_  
 Date \_\_\_\_\_  
 Run Conducted By \_\_\_\_\_

Component	Contents	Weight (g)
Condenser	Empty	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>a</i>
Resin Trap	XAD-2	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>b</i>
Condensate Trap	Empty	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>c</i>
First Impinger	100 mL Ethylene Glycol	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>d</i>
Second Impinger	Empty	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>e</i>
Third Impinger	~200 g Silica Gel	<i>Final</i> _____ <i>Initial</i> _____ <i>Gain</i> _____ <i>f</i>

Weight of water collected =  $a + b + c + d + e + f$

$W_{H_2O}$  = \_\_\_\_\_ grams

FIGURE 2 MOISTURE ANALYSIS DATA SHEET

access port to avoid extraneous pick up of deposited material. To begin sampling, point the nozzle directly into the gas stream at the first traverse point. Block off the opening between the probe assembly and the access ports. With a nomograph or a programmable calculator, determine the orifice setting for isokinetic sampling. Record the initial dry gas meter reading. Immediately start the vacuum pump and adjust the sampling flow rate to isokinetic conditions. Sample for at least five minutes at each traverse point, the sampling time being the same for every point. Traverse the stack cross section and maintain isokinetic sampling ( $\pm 10\%$ ) throughout the test. Record instrumentation readings on the Organics Sampling Data Sheet (Figure 3) every five minutes, or at regular intervals that are consistent with the sampling duration established for each point, whichever is less. To simplify recording in the field, values may be entered in units for which the sampling equipment is designed. These values may then be converted (if necessary) to the metric units specified in the equations where they are used. Readings must also be taken before and after a "leak-check" and when sampling is halted. Record all sampling interruptions.

During sampling, verify the heating and cooling systems. The temperature of the filter enclosure and probe must be  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ). The XAD-2 temperature must be below  $20^{\circ}\text{C}$ . Verify the alignment of the probe and pitot tube to the gas stream. Check the level and zero of the manometer and adjust if necessary. Verify that the condensate volume is increasing as the run progresses. If the sampling system vacuum becomes so high that it is difficult to maintain isokinetic sampling the filter holder and/or sorbent tube must be replaced. If this occurs, a "leak-check" must be conducted before and after the replacement of the above components.

When it is necessary to halt sampling temporarily either to dismantle the sampling train during port change-over or to change a train component, turn off the pump and immediately withdraw the probe from the stack. Conduct a mandatory "leak-check" on the sampling train by plugging the nozzle and pulling a vacuum equal to or greater than the maximum value observed during sampling. Record the actual leakage rate. If the leakage rate exceeds  $0.57 \text{ L/min}$  ( $0.02 \text{ ft}^3/\text{min}$ ) or four percent of the sampling flowrate, whichever is less, consult Environment Canada regarding the validity of the test. If the leakage rate is acceptable, proceed with dismantling the sampling train or changing the train component. Before continuing the test, conduct a "leak-check" on the assembled train by following the pre-test "leak-check" procedures used during sampling train preparation.



When the test is completed, conduct a mandatory post-test check on the sampling train by plugging the nozzle and pulling a vacuum equal to or greater than the maximum value observed during sampling. Record the actual leakage rate which must be less than either 0.57 L/min (0.02 ft<sup>3</sup>/min) or four percent of the sampling flow rate during the test, whichever is less. If the leakage rates are acceptable, proceed with the recovery of the samples.

When the test is finished, disconnect the probe and set it aside to cool. Seal both ends and take care not to lose any material in the probe. Disconnect the filter (and cyclone, if used) and seal both ends with pre-cleaned glass plugs or caps, or aluminum foil. Disconnect the XAD-2 and condenser. Seal both ends. Seal the ends of the impinger train. When transporting the sampling train components to the on-site lab, take care to minimize the possibility of sample loss or contamination. Samples must be recovered in a clean area.

**Blank Train:** One blank train must be submitted for every group of three sample trains. The blank train is handled in the same manner as the loaded train except that no stack gases are drawn through the train. However, a volume of ambient air equal to that drawn during the pre- and post-traverse "leak-checks" must be drawn through the blank train. Hence, on the day that the blank train will be submitted, it is necessary to record all volumes drawn into the sampling train during all "leak-checks". It is not necessary to heat the filter components of the blank train during sampling or to conduct "leak-checks" on the blank train. The sample recovery procedures for the loaded trains are also applicable to the blank train.

### 5.3 Sample Recovery

**Nozzle, Probe, Cyclone and Front-Half of the Filter Holder:** Wipe exterior surfaces of the nozzle and probe to remove excess particulate matter. Quantitatively recover the particulate matter and condensibles from the nozzle, probe, cyclone (if used), by-pass connector (if cyclone is not used) and the front-half of the filter holder by washing (with brush) these components with hexane and acetone. Wash and brush each component three times with hexane and acetone. Finally, rinse each component three times each with hexane and acetone. Store all rinses in a wide mouth amber glass bottle (pre-cleaned, with a pre-cleaned Teflon lid liner). Mark the liquid level on the outside of the bottle and label the container. Place the labelled container in a sealed clear plastic bag.

**Note:** The extension rod (stainless steel or Teflon) of the probe brush used to wash the probe liner shall be wiped with the recovery solvents prior to sample recovery.

Filter: Carefully remove the exposed filter from the filter holder and place on pre-cleaned aluminum foil with exposed side up. Carefully transfer any loose particulate matter or filter fibres adhering to the support of the filter holder with a dry nylon bristle brush or sharp blade. Fold the filter in half and crimp the foil to close the edges. Place in a pre-cleaned glass petri dish. Label the petri dish and seal with 25-mm (one inch) wide Teflon tape around the circumference. The petri dish may also be sealed with aluminum foil.

Back-half of Filter Holder and Condenser: Drain all the cooling water from the condenser and wipe the outside of the condenser to remove excess water. Weigh the condenser and record the weight on the Moisture Analysis Data Sheet. Soak each of these components five minutes each with hexane and acetone. Rinse each component three times each with hexane and acetone. Store the soak and rinses in a wide-mouth amber bottle (pre-cleaned, with a pre-cleaned Teflon lid liner). Mark the liquid level on the outside of the bottle and label the container. Place the labelled container in a sealed clear plastic bag.

XAD-2 Tube: Remove the aluminum foil on the tube and drain all the cooling water. Wipe the outside of the tube to remove excess moisture. Weigh the XAD-2 tube and record the weight on the Moisture Analysis Data Sheet. Seal both ends of the XAD-2 tube with pre-cleaned glass caps or plugs. Wrap the whole tube with aluminum foil and label the container.

Condensate Trap and Ethylene Glycol Impinger: Wipe the outside of these components to remove excess moisture. Weigh each component and record the weights on the Moisture Analysis Data Sheet. Empty each container into a pre-cleaned amber glass bottle with a pre-cleaned Teflon lid liner. Rinse each component three times each with HPLC water into the same container. Mark the liquid level on the outside of the bottle and label the container. Place the labelled container in a sealed clear plastic bag.

Second and Third Impingers: Wipe the excess moisture from the outside of these impingers and weigh each impinger. Record each weight on the Moisture Analysis Data Sheet.

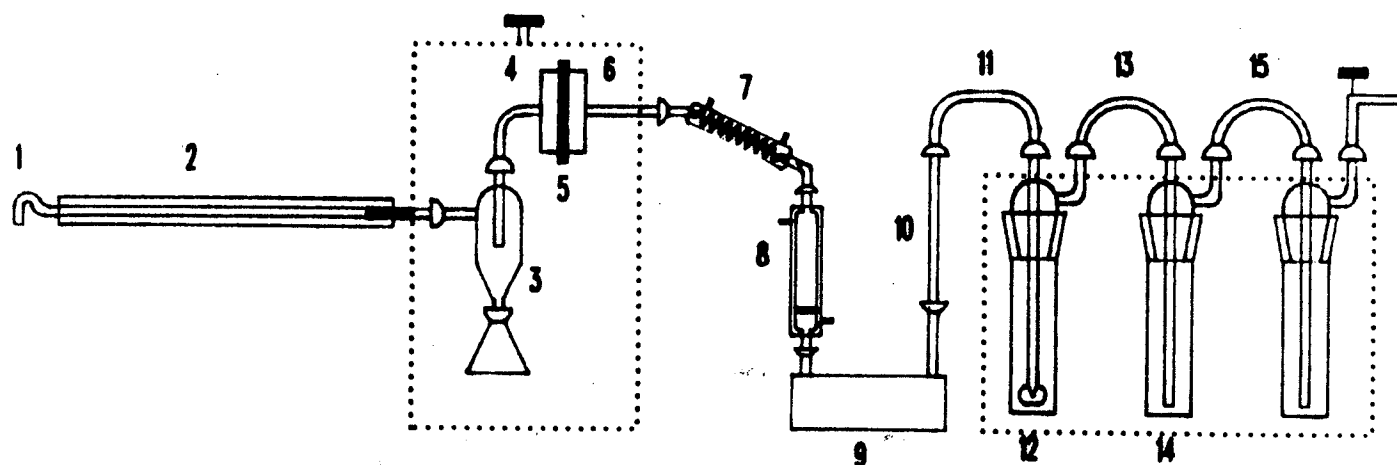
All Back-Half Glassware excluding XAD-2 Tube: Rinse each component including connectors three times each with hexane and acetone into a pre-cleaned glass amber bottle with a pre-cleaned Teflon lid liner. Mark the liquid level on the outside of the



bottle and label the container. Place the labelled container in a sealed clear plastic bag. The sample recovery procedures for the front- and back-half components are illustrated in Figure 4.

Sample Handling: Following the recovery of the train components, all samples must be kept cool (approximately 4°C). They may be stored in a refrigerator or on ice in an insulated chest during field storage and transport to the analytical laboratory.

Before transporting the insulated chest, it is critical to ensure that the containers are well-packed to minimize sample loss. Many materials are available to protect the glass container against shock. Extra care should be taken when packing the samples.



Container or Sample	Component(s)	Recovery Procedure
1	1, 2, 3, 4	Wash and brush 3 times each with hexane (H) and acetone (A). Rinse 3 times each with H and A.
2	5	Remove carefully from holder. Place on pre-cleaned foil. Fold in half. Place in pre-cleaned glass petri dish.
3	6, 7	Soak 5 minutes each with H and A. Rinse 3 times each with H and A.
4	8	Cap ends and wrap in foil.
5	9, 12	Empty contents into container and rinse each 3 times with HPLC water.
6	6 to 15 except 8	Rinse 3 times each with H and A.

Mark liquid levels on all bottles.

All sample containers are pre-cleaned amber glass bottles with pre-cleaned Teflon lid liners.

FIGURE 4 SEMI-VOLATILE ORGANICS RECOVERY PROCEDURES

## 6 CHEMICAL ANALYSIS

The amount of PCBs, PCDDs and PCDFs in the test samples shall be determined by the Chemical Analysis procedures found in the Environment Canada document, "A Method for the Analysis of Polychlorinated Dibenzo-*para*-Dioxins (PCDDs), Polychlorinated Dibenzofurans (PCDFs), and Polychlorinated Biphenyls (PCBs) in samples from the Incineration of PCB Waste", Chemistry Division, Conservation and Protection, EPS-1/RM/3, 1989. The analytical results are to be used in Equation 10, Equation 11 and Figure 5.

## 7 CALCULATIONS

To simplify recording during a test, field data may be entered in the units for which the sampling equipment is designed. These values must be converted, if necessary, to the metric units specified in the applicable equations.

### 7.1 Equations (see Section 7.2 for Nomenclature)

Dry Gas Meter Volume: The total sample volume measured by the dry gas meter is corrected to reference temperature and pressure conditions (25°C and 101.3 kPa) using *Equation 1*. For a temperature-compensated dry gas meter ( $T_m$ )<sub>avg</sub> should be substituted by the temperature specified by the manufacturer.

Equation 1

$$(V_m)_{\text{ref}} = \gamma V_m \frac{T_{\text{ref}}(P_{\text{bar}} + \Delta H_{\text{avg}})}{(T_m)_{\text{avg}} P_{\text{ref}}}$$

Volume of Water Vapour: The volume of water vapour in the sack gas sample expressed at reference conditions (25°C and 101.3Kpa) is calculated by using *Equation 2*.

Equation 2

$$(V_m)_{\text{ref}} = \frac{W_{\text{H}_2\text{O}} 10^{-3} R T_{\text{ref}}}{M_{\text{H}_2\text{O}} P_{\text{ref}}}$$

Moisture Content: The volumetric fraction of water vapour in the stack gas expressed at reference conditions (25°C and 101.3 Kpa) is calculated using *Equation 3*.

Equation 3

$$B_{\text{wo}} = \frac{(V_w)_{\text{ref}}}{(V_w)_{\text{ref}} + (V_m)_{\text{ref}}}$$

For a saturated or supersaturated stack, use a psychrometric chart to determine  $B_{\text{wo}}$ .

Absolute Stack Gas Pressure: The absolute stack gas pressure is calculated using Equation 4.

Equation 4

$$P_s = P_{\text{bar}} + \Delta P_s$$

Stack Gas Molecular Weight: The stack gas molecular weight on a wet basis is calculated using Equation 5.

Equation 5

$$M_s = M_d (1 - B_{wo}) + 18 B_{wo}$$

Stack Gas Velocity: The stack gas velocity measured at each traverse point is calculated using Equation 6.

Equation 6

$$U_s = 128.95 C_p \left( \frac{\Delta p T_s}{P_s M_s} \right)^{1/2}$$

The average stack gas velocity  $((U_s)_{\text{avg}})$  and stack gas temperature  $((T_s)_{\text{avg}})$  for the test run are determined by averaging the velocities and temperatures respectively measured at the traverse points.

Volumetric Stack Gas Flow Rate: The average volumetric stack gas flow rate expressed on a dry basis and at reference conditions (25°C and 101.3 kPa) is calculated using Equation 7.

Equation 7

$$Q_s = 3600 (U_s)_{\text{avg}} A_s (1 - B_{wo}) \frac{T_{\text{ref}} P_s}{(T_s)_{\text{avg}} P_{\text{ref}}}$$

Isokinetic Variation: The isokinetic variation for each traverse points is calculated using Equation 8.

Equation 8

$$I = \frac{Y \frac{(V_m)_j}{t} \frac{1}{(1 - B_{wo})} (P_{\text{bar}} + \Delta H) T_s}{4.71 \times 10^{-5} (N_d)^2 \left( \frac{T_{mi} + T_{mo}}{2} \right) P_s U_s} \times 100$$

A test shall be considered valid when no more than 10% of the isokinetic values fall outside the range 90% to 110% and the average for all the traverse points  $I_{avg}$  falls within the range 90% to 110%.

Correction Factor: The pollutant concentrations expressed in this method must be corrected to a basis of 11% O<sub>2</sub>. The correction factor to express concentrations at 11% O<sub>2</sub> is calculated using *Equation 9*.

Equation 9

$$f_{11\% \text{ O}_2} = \frac{9.9}{20.9 - \% \text{O}_2}$$

Polychlorinated Biphenyl Emission Rates: The emission rate of polychlorinated biphenyls is calculated using *Equation 10*.

Equation 10

$$ER_{PCBs} = \frac{W_{PCBs} Q_s}{(V_m)_{ref}}$$

Concentrations of 2,3,7,8-TCDD Equivalent PCDDs and PCDFs: The concentration of 2,3,7,8-TCDD equivalent PCDDs and PCDFs corrected to 11% O<sub>2</sub> is calculated using *Equation 11*. The weight of 2,3,7,8-TCDD equivalent PCDDs and PCDFs is determined from Figure 5.

Equation 11

$$C_{2,3,7,8\text{-T4CDD}'} = \frac{W_{2,3,7,8\text{-T4CDD}'} f_{11\% \text{ O}_2}}{(V_m)_{ref}}$$

## 7.2 Nomenclature

$A_s$	- inside cross-sectional area of stack or duct, m <sup>2</sup>
$B_{wo}$	- volumetric fraction of water vapour in the stack gas, dimensionless
$C_{2,3,7,8\text{-T4CDD}'}$	- concentration of 2,3,7,8-T4CDD equivalent PCDDs and PCDFs on a dry basis at reference temperature and pressure conditions corrected to 11% O <sub>2</sub> , ng/m <sup>3</sup> at 11% O <sub>2</sub>
$C_p$	- S-type pitot tube coefficient, dimensionless
$ER_{PCBs}$	- emission rate of polychlorinated biphenyls, µg/h

$\Delta H_{avg}$	- average pressure drop across orifice meter, kPa
$\Delta H$	- pressure drop across orifice meter for each traverse point, kPa
$I_{avg}$	- average isokineticity for the test, (%)
$I$	- isokineticity i.e., the ratio of the sampling velocity through the nozzle to the velocity of the undisturbed gas stream at each traverse point, dimensionless, (%)
$j$	- traverse point number, dimensionless
$M_d$	- molecular weight of stack gases on a dry basis, kg/kmol
$M_s$	- molecular weight of stack gases on a wet basis, kg/kmol
$M_{H_2O}$	- molecular weight of water, 18 kg/kmol
$N_d$	- inside diameter of the sampling nozzle, mm
$f_{11\%O_2}$	- correction factor to convert concentrations at dry and reference temperature and pressure conditions to 11% O <sub>2</sub> , dimensionless
%O <sub>2</sub>	- concentration of oxygen in the stack gas (dry basis), % by volume
$P_s$	- absolute stack gas pressure, kPa
$P_{bar}$	- barometric pressure at the sampling site, kPa
$\Delta p$	- pitot-tube velocity pressure reading at each traverse point, kPa
$P_{ref}$	- reference pressure, 101.3 kPa
$\Delta p_s$	- static pressure of the stack gas, kPa
$Q_s$	- volumetric stack gas flow rate on a dry basis at reference temperature and reference pressure conditions, m <sup>3</sup> /h
$R$	- universal gas constant, 8.31 kPa m <sup>3</sup> (kmol·K) <sup>-1</sup>
$t$	- sampling duration for each traverse point, min
$(T_s)_{avg}$	- average of the absolute stack gas temperatures, K
$T_s$	- absolute stack gas temperature at each traverse point, K
$(T_m)_{avg}$	- average of the absolute dry gas meter temperatures, K
$T_{mi}$	- absolute temperature at the dry gas meter inlet for each traverse point, K
$T_{mo}$	- absolute temperature at the dry gas meter outlet for each traverse point, K
$T_{ref}$	- reference temperature, 298 K
$(U_s)_{avg}$	- average stack gas velocity, m/s
$U_s$	- stack gas velocity measured at each traverse point, m/s
$V_m$	- volume of stack gas sample at dry gas meter conditions, m <sup>3</sup>
$(V_m)_j$	- volume of stack gas sample at dry gas meter conditions for each traverse point, m <sup>3</sup>
$(V_m)_{ref}$	- volume of stack gas sample at reference conditions, m <sup>3</sup>

CONGENER	TRAIN CATCH		TOXICITY FACTOR	=	EQUIVALENT TRAIN CATCH
<b>PCDDs</b>					
1. 2,3,7,8-T4CDD	<input type="text"/>	X	1.0	=	<input type="text"/>
2. 1,2,3,7,8-P5CDD	<input type="text"/>	X	0.5	=	<input type="text"/>
3. 1,2,3,4,7,8-H6CDD	<input type="text"/>	X	0.1	=	<input type="text"/>
4. 1,2,3,6,7,8-H6CDD	<input type="text"/>	X	0.1	=	<input type="text"/>
5. 1,2,3,7,8,9-H6CDD	<input type="text"/>	X	0.1	=	<input type="text"/>
6. 1,2,3,4,6,7,8-H7CDD	<input type="text"/>	X	0.01	=	<input type="text"/>
7. O8CDD	<input type="text"/>	X	0.001	=	<input type="text"/>
<b>PCDFs</b>					
8. 2,3,7,8-T4CDF	<input type="text"/>	X	0.1	=	<input type="text"/>
9. 1,2,3,7,8-P5CDF	<input type="text"/>	X	0.01	=	<input type="text"/>
10. 2,3,4,7,8-P5CDF	<input type="text"/>	X	0.5	=	<input type="text"/>
11. 1,2,3,4,7,8-H6CDF	<input type="text"/>	X	0.1	=	<input type="text"/>
12. 1,2,3,6,7,8-H6CDF	<input type="text"/>	X	0.1	=	<input type="text"/>
13. 1,2,3,7,8,9-H6CDF	<input type="text"/>	X	0.1	=	<input type="text"/>
14. 2,3,4,6,7,8-H6CDF	<input type="text"/>	X	0.1	=	<input type="text"/>
15. 1,2,3,4,6,7,8-H7CDF	<input type="text"/>	X	0.01	=	<input type="text"/>
16. 2,3,4,6,7,8,9-H7CDF	<input type="text"/>	X	0.01	=	<input type="text"/>
17. O8CDF	<input type="text"/>	X	0.001	=	<input type="text"/>
$W_{2,3,7,8-T4CDD'} =$ 2,3,7,8-T4CDD Equivalent PCDDs and PCDFs (add 1 to 17)					<input type="text"/>

FIGURE 5

CALCULATION OF 2,3,7,8-T4CDD EQUIVALENT PCDDs AND PCDFs



$(V_w)_{ref}$	- volume of water vapour in the stack gas sample at reference conditions, $m^3$
$W_{H_2O}$	- weight of water vapour condensed in the impingers, g
$W_{PCBs}$	- weight of polychlorinated biphenyls collected in the semi-volatile organics sampling train, mg
$W_{2,3,7,8-T4CDD}$	- weight of 2,3,7,8-TCDD equivalent PCDDs and PCDFs collected in the semi-volatile organics sampling train, ng
$Y$	- dry gas meter calibration factor (ratio of the wet test meter volume to the dry test meter volume) dimensionless
128.95	- proportionality constant $(m/s)(K-l \cdot kg/kmol)^{1/2}$

## REFERENCES

1. Environment Canada, "Standard Reference Method for Source Testing: Measurement of Emissions of Particulates from Stationary Sources", Report EPS 1-AP-74-1 February 1974 (as amended).
2. Bursey, J., M. Hartman, J. Homolya, R. McAllister, J. McGaughey, D. Wagoner. "Laboratory and Field Evaluation of the Semi-VOST Method, Volume II: Appendices", EPA/600/4-85/0756 NTIS No. PB 86-123569 (November 1985).

**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix D**

### **Canada-wide Standards for Mercury**

**Canadian Council of Ministers of the Environment**

***CANADA-WIDE STANDARDS***

***for***

***MERCURY EMISSIONS***

# **CANADA-WIDE STANDARDS for MERCURY EMISSIONS**

## **PREAMBLE**

The Canadian Council of Ministers of the Environment determined that mercury levels in fish and wildlife across Canada warrant additional efforts to reduce atmospheric emissions derived from both deliberate use of mercury and from incidental releases of mercury.

Restrictions on the human consumption of fish in order to safeguard the health of both high fish consumers (sustenance and commercial fishers) and sensitive populations (infants, children and women of childbearing age) are widespread. Some of these restrictions are derived from lakes naturally high in mercury, others in lakes and rivers contaminated by historical point source discharges and still others in waters remote from identifiable sources. Traditional lifestyles may be profoundly influenced by mercury contamination.

Mercury levels in fish pose an additional, largely unquantified risk to fish-eating wildlife. Isolated examples of toxicity to loons and otters suggest the potential for large scale and/or widespread impacts. Just as mercury levels in fish affect their consumption by humans, some levels in fish may affect wildlife which consume them. In both instances, the mercury causing the impacts is derived from both natural and anthropogenic sources.

The combined impacts of mercury contamination in Canada are difficult to quantify. The exact proportion of the impact which can be ascribed to natural mercury and to past and present anthropogenic releases cannot presently be quantified. Because it is a natural and persistent bioaccumulative element which can be transported many miles in the atmosphere, mercury can have impacts many years and many miles removed from its original source. A common thread through all mercury impacts is that deposition to waterbodies from anthropogenic emissions poses a threat to human and ecosystem health, and that reduced deposition will contribute, in time, to reduced impacts.

Under a variety of regional, national, binational and international programs, treaties and agreements, mercury has been consistently targeted for emission reductions. Such a policy position is consistent with the CCME Policy for the Management of Toxic Substances which identifies that mercury shall be managed through its lifecycle to minimize releases. This is consistent with the precautionary approach endorsed in the Harmonization Accord and Canada Wide Standards Sub-agreement. Ministers of the Environment have thus agreed to undertake and promote cost-effective actions to achieve further precautionary reductions in anthropogenic emissions (releases to the air) of mercury.

## **Canada-wide Standards for Mercury Emissions**

The Canada-wide Standards development process reviewed the nature of the mercury issue in Canada, and concluded that two distinctive source categories were amenable to further actions, namely life-cycle management of products containing mercury to minimize releases, and reduction or minimization actions for major point source emissions of incidental mercury.

Based upon available inventory information, it is estimated that of the year 2000 national mercury emissions (12 T/yr) three sectors will contribute the bulk of these emissions, namely the base metal smelting sector (2.57 T/yr), waste incineration sector (1.2 T/yr) and coal-fired electricity generation sector (1.1 T/yr). Standards have been developed for two of these three sectors that reflect room for improvement that is significant and cost-effective. Standards have been provided also that will be used to guide the development of new facilities should they be constructed. Efforts to develop a standard for the electricity generation sector have been complicated and progress has been delayed such that a workplan to develop standards for this sector will not be completed until early in 2000. Life-cycle standards for select mercury-containing products will also be completed early in 2000.

## **PART 1:**

### **Base metal smelting**

#### Rationale for standard

The base metal smelting sector has historically been responsible for much of the mercury emitted in Canada. However the voluntary application of a number of process changes and stack treatments/scrubbers have combined to reduce mercury emissions from this sector by more than 90% since 1988. Due to reductions from this sector, Canada has complied with its obligations under the United Nations Economic Commission for Europe Heavy Metals Protocol. Despite this substantial progress additional reductions are possible. As of 2000, the mercury emissions from base metal smelting remain the single largest emission sector in Canada at 2.8 T/yr. Under the federal Strategic Options Process (SOP), industry and government recommended development by CCME of “environmental source performance guidelines” that reflect application of best available techniques. By following this approach, Canada’s domestic program will be consistent with international objectives for this industry.

#### Nature and application:

Based upon the performance of various technologies and practices as demonstrated at existing facilities in Canada, and in consideration of the recommendations made in the federal SOP for this sector, a two-part standard is recommended. This standard reflects the application of “best available techniques” on a facility-specific basis, and a uniform reporting mechanism based upon environmental source performance (atmospheric emission) guidelines. Standards are suggested for both existing facilities, to reflect actions taken to reduce emissions of mercury, and for new facilities, to ensure that smelters utilize the best available techniques to avoid or reduce metals emissions generally and mercury emissions specifically.

#### Numeric Targets:

For existing facilities: application by all primary zinc, lead and copper smelters of best available pollution prevention and control techniques economically achievable to achieve an environmental source performance (atmospheric emission) guideline of 2 g Hg/tonne total production of finished metals.

For new and expanding facilities: application of best available pollution prevention and control techniques to minimize mercury emissions throughout the life-cycle of the minerals in question to achieve an environmental source performance (atmospheric emission) guideline of 0.2 g Hg/tonne production of finished zinc, nickel and lead, and 1 g Hg/tonne of finished copper, and consideration of a mercury offset<sup>1</sup> program to ensure no “net” emission increases occur.

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<sup>1</sup> A new facility will recover and retire an amount of mercury equivalent to their annual emissions.

## Canada-wide Standards for Mercury Emissions

### Timeframe for achieving the targets:

Existing facilities will be expected to make a determined effort<sup>2</sup> to meet this standard by 2008, coincident with implementation of the federal Strategic Options Report, while any new facility will be required to design for and achieve compliance immediately upon full scale operation. Jurisdictions will evaluate changes and upgrades to existing facilities to ensure they constitute determined efforts.

## Waste Incineration

### Rationale for standard

Waste incineration<sup>3</sup> has historically been responsible for a significant portion of the mercury emitted in Canada, however reductions in emissions have been apparent. Improved exhaust gas controls to reduce emissions of acid gases and fine particulates or activated carbon injection systems have decreased emissions of both mercury and dioxins and furans from the municipal solid waste<sup>4</sup> sector. At the same time, action has been taken by many product manufacturers to reduce the mercury content of consumer goods which could end their life cycle in domestic solid waste (e.g., alkaline batteries) and thus have reduced the mercury available in the waste stream. Mercury from this sector is estimated to be 446 kg/year. Many medical waste incinerators<sup>5</sup> have closed for economic or environmental reasons, but a range of medium- to small-sized facilities remain which alone are small sources, but as a sector are considerable, emitting an estimated 250 kg/yr. Two sectors in which emission reductions are not apparent, hazardous waste<sup>6</sup> (550 kg/yr) and sewage sludge (285 kg/yr) incineration, can achieve reductions either through source control or gas-controls. Control of mercury emissions has been recognized in a variety of jurisdictions, including some Canadian Provinces, with emission standards being developed by the Eastern Canadian Premiers and New England Governors (ECP-NEG), or the United Nations Economic

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<sup>2</sup> Determined efforts include the ongoing review of opportunities for reductions and implementation of in-plant changes and/or emissions control upgrades that are technically and economically feasible and which confer on-going reductions in emissions

<sup>3</sup> For the purpose of the Mercury CWS, an incinerator shall be considered to be a device, mechanism or structure constructed primarily to thermally treat (e.g., combust or pyrolyze) a waste for the purpose of reducing its volume, destroying a hazardous chemical present in the waste, or destroying pathogens present in the waste. This includes facilities where waste heat is recovered as a byproduct from the exhaust gases from an incinerator, but does not include industrial processes where fuel derived from waste is fired as an energy source as a matter incidental to the manufacture of the primary product.

<sup>4</sup> For the purpose of the Mercury CWS, municipal solid waste shall be taken to include any waste which might normally be disposed of in a non-secure landfill site if not incinerated (i.e., including non-hazardous solid wastes regardless of origin), but is not intended to include “clean” wood waste. Clean wood waste means waste from woodworking or forest product operations where the wood waste has not been treated with preservative chemicals (e.g., pentachlorophenol) or decorative coatings.

<sup>5</sup> For the purpose of the Mercury CWS, medical waste is any waste which includes as a component any Biomedical Waste as defined in the February 1992 CCME *Guidelines for the Management of Biomedical Waste in Canada*, with the exception that animal wastes derived from animal health care or veterinary research and teaching establishments are excluded.

<sup>6</sup> hazardous waste incinerators do not include facilities that use waste derived fuel.



## Canada-wide Standards for Mercury Emissions

Commission for Europe (UNECE) providing a basis for evaluating possible Canada-wide Standards.

Actions to reduce national emissions below the anticipated 2000 rate of 1.5 T/yr require that any new facilities meet stringent limits, and that the bulk of the emissions from existing facilities be controlled through retrofits with control technology that is efficient at removing mercury, or the mercury containing waste be diverted from incinerators. All facilities, and particularly smaller ones, may find that pollution prevention, waste segregation and diversion are options for either achieving the limit, or reducing “end-of-stack” expenditures, and during implementation all facilities should be encouraged to place a priority on reduced inputs rather than controlled releases. Attainment of the numeric standards adopted by the ECP-NEG for application to large municipal, and sludge incinerators, and by the UNECE for hazardous waste incineration is suggested as cost-effective means of reducing emissions from this sector. A review of the ECP-NEG limit for medical incinerators concluded that larger medical incinerators actually combust a considerable amount of municipal waste, and as such it is recommended they comply with the same limit as the municipal sector.

### Nature and application:

Emission limits are expressed as a concentration in the exhaust gas exiting the stack of the facility. New or expanding facilities will be expected to comply immediately with the standard, and it will be up to individual jurisdictions to determine what constitutes a significant expansion to trigger the standard. The limits for existing facilities are capable of being met using generally available technology (or waste diversion). Larger facilities will be subject to annual stack testing to verify compliance with the limit and smaller (medical, municipal) facilities will have the option of reporting on a successful mercury diversion plan or of conducting a one-time stack test, to illustrate progress towards the standard.

### Numeric targets:

For new or expanding facilities of any size, application of best available pollution prevention and control techniques, such as a mercury waste diversion program, to achieve a maximum concentration<sup>7</sup> in the exhaust gases from the facility as follows:

Municipal waste incineration <sup>8,9</sup>	20 µg/Rm <sup>3</sup>
Medical waste incineration	20 µg/Rm <sup>3</sup>
Hazardous waste incineration	50 µg/Rm <sup>3</sup>
Sewage sludge incineration	70 µg/Rm <sup>3</sup>

For existing facilities application of best available pollution prevention and control techniques, to achieve a maximum concentration<sup>7</sup> in the exhaust gases from the facility as follows:

Municipal waste incineration <sup>8</sup>	
> 120 Tonnes/year <sup>9</sup>	20 µg/Rm <sup>3</sup>
< 120 Tonnes/year <sup>10</sup>	20 µg/Rm <sup>3</sup>

## Canada-wide Standards for Mercury Emissions

Medical waste incineration	
> 120 Tonnes/year <sup>9</sup>	20 µg/Rm <sup>3</sup>
< 120 Tonnes/year <sup>10</sup>	40 µg/Rm <sup>3</sup>
Hazardous waste incineration	50 µg/Rm <sup>3</sup>
Sewage sludge incineration	70 µg/Rm <sup>3</sup>

<sup>7</sup> Stack concentrations of mercury will be corrected to 11% oxygen content for reporting purposes.

<sup>8</sup> Conical waste combustors are under separate consideration since the proposed standard for municipal waste incinerators cannot be achieved with these burners. Newfoundland will review the use of conical waste combustors and consider a phase-out strategy that will reduce mercury emissions. Such a strategy would be developed in conjunction with the Canada-wide Standard for dioxins and furans.

<sup>9</sup> Larger facilities must achieve this stack concentration as confirmed by annual testing.

<sup>10</sup> Smaller facilities must make determined efforts<sup>2</sup> to achieve this stack concentration. The effectiveness of the pollution prevention measures will be established by way of a one-time stack test conducted after implementation of the plan or by the provision of an inventory documenting an audit of a waste diversion program, which is deemed an acceptable substitute.

### Timeframe for achieving the targets:

Any new or expanding facility will be required to design for and achieve compliance immediately upon attaining normal full scale operation, compliance to be confirmed by annual stack testing or an equivalent emission rate as confirmed by an audit of a waste diversion program.

Existing facilities will endeavour to meet the standards on the following schedule:

Municipal waste incineration	2006
Medical waste incineration	2006
Hazardous waste incineration	2003
Sewage sludge incineration	2005

## **PART 2:**

### REPORTING ON PROGRESS:

Ministers will receive reports by jurisdictions in 2004, 2007 and 2010, and will ensure that a single public report is prepared and posted on the CCME web site for public access. The 2010 report will include an evaluation of these standards and a recommendation whether changes should be considered.

These reports will be accompanied by other information on additional outcomes, activities, research or issues which are relevant to the mercury CWSs and/or sector under consideration. Examples of such reporting includes speciation measures relevant to design of stack control measures, other environmental programs with implications for compliance with the standards,

## **Canada-wide Standards for Mercury Emissions**

etc..

Each jurisdiction will detail the means of ensuring compliance/achievement in a manner consistent with the typical or desired programs for the affected facility/sector, so as not to impose an unnecessary level of reporting duplication upon the jurisdictions. In those sectors where the CWS calls for determined efforts, jurisdictions will report on their evaluation of these efforts.

During the years prior to the date for achievement/compliance of a standard, jurisdictions will provide information explaining the status of their implementation of each mercury CWS so that a consolidated summary of jurisdictional progress can be prepared for the public. More details and a mock-up report are available in Annex 1.

### **ADMINISTRATION:**

Jurisdictions will review and renew Part 2 and Annex 1 five years from coming into effect.

Any party may withdraw from these Canada-Wide Standards upon three month's notice.

These Canada-Wide Standards come into effect for each jurisdiction on the date of signature by the jurisdiction.

# **Annex 1**

## **Mercury Reporting Framework**

### **Introduction**

Under the Harmonization Accord and its Canada-wide Environmental Standards Sub-Agreement, all jurisdictions are to report to the public and to Ministers on their progress towards achieving the CWSs for mercury.

This reporting framework is intended to provide a transparent and consistent mechanism for reporting by jurisdictions in a fashion which minimizes resource requirements for government and industry alike, while maximizing the availability of information on achievement of these standards.

The framework addresses:

- 1) frequency, timing and scope of reporting
- 2) guidance as to the means of determining compliance/achievement of the CWS
- 3) common measurement parameters for reporting purposes
- 4) data management and public reporting

### **Frequency, timing and scope of reporting**

There will be reporting on a schedule which is tied into assessing the performance of the governments in meeting the benchmarks and timelines relevant to the standards. A report issued in 2004 will include compliance by one incineration sector and progress in all others towards implementation, the report in 2007 compliance by all incineration sectors and progress for base metal smelting, and the third report in 2010 an overall evaluation of compliance for all standards and any recommendations for revisions. Jurisdictions will submit spreadsheets which will contain all relevant information necessary for a single public report to be posted on the CCME web site for public access.

Reports will be limited to information on those facilities which are subject to achievement and/or compliance with the Canada-wide Standards as endorsed by the Ministers of the Environment (insert date) and as implemented variously by the responsible jurisdictions or industries. This information is intended to show compliance rates and performance characteristics in a manner which documents sectoral performance as well as jurisdictional performance. It is not intended to provide a facility-by-facility record of performance.

### **Means of determining compliance/achievement of the CWS**

The Canada-wide Standards for mercury lend themselves to achievement through voluntary action, or through compliance with regulated or legally enforceable limits. As such, it is necessary to provide some means to ensure that a level playing field exists so that the numeric value provided in the CWS is applied equally or similarly in each jurisdictions. One means to do this is to require identical compliance procedures, but this may require that some jurisdictions apply compliance procedures for mercury CWSs that are different than those used for locally determined or regulated parameters such as SO<sub>2</sub>, PM, ammonia, etc. An example is where the mercury CWS is expressed as the average of 3 stack tests, whereas a jurisdiction may normally utilize the median value of 3 tests to determine compliance.

In an effort to streamline implementation, each jurisdiction will determine the exact means of ensuring compliance/achievement in a manner consistent with the typical or desired programs for the affected facility/sector. It is anticipated that minor variations in jurisdictional requirements will result in minimal variation across the country which is insignificant with respect to the overall reduction activities which range from 50-99% for various facilities.

### **Common measurement parameters for reporting purposes**

Each facility report will include specific measures corrected so as to be compatible and consistent for the purposes of public reporting. Mercury emissions must be corrected for the O<sub>2</sub> content of gases, to ensure compliance with the standards.

Each jurisdiction will determine the sector within which each subject facility will be reported. For example, a jurisdiction may determine that a small mixed waste incinerator (for example, burning both medical and municipal waste) may be subject to either standard, based upon the preponderance of waste (>50% as one type) or based upon the provincial designation of facility type. Sectoral assignments will be updated to reflect the most recent characteristics of the facility under consideration prior to reporting.

While little confusion is likely to exist over the implementation of mercury CWSs for “greenfield” facilities, it is possible that significantly expanded or modified facilities can/should be considered as new for the purposes of achievement/compliance with the mercury CWS. It will be the responsibility of the jurisdictions to determine at which point a facility no longer qualifies as an “existing” facility and must conform to the standard for “new or expanded” facilities as a result of significant modifications/alterations to the facility operations or physical plant.

### **Data management and public reporting**

Facility-specific information will be supplied in a spreadsheet format to facilitate reporting. A consolidated data-report and achievement/compliance report will be made available to all jurisdictions and to the Ministers, along with the draft public report, prior to formal release of the public report. The public report will be released upon approval by the Ministers of the

## **Canada-wide Standards for Mercury Emissions**

Environment.

Jurisdictions will provide a report in spreadsheet format prior to September 30 so that the consolidated spreadsheet can be prepared along with the public report (draft) for review and approval. A consolidated spreadsheet will be prepared and distributed to all jurisdictions within 30 days of receipt of the final jurisdictional spreadsheet. Along with the consolidated spreadsheet a draft public report will be provided for review and consideration prior to the Ministers' meeting at which public release is anticipated. That public report (draft format attached) will be posted to the CCME web site upon approval by the Ministers. Jurisdictions are encouraged to provide reference to the CCME web site and/or pointers in their own web sites in order to ensure a single location for mercury CWSs reporting should errors/miscalculations have to be corrected at some time.

In addition to the consolidated public reporting on mercury CWSs, jurisdictions must provide a contact for facility-specific information in the advent that the public wishes to access compliance or achievement information. Such data will be supplied in a manner consistent with the normal data-reporting/compliance reporting procedures of the jurisdiction in question - the consolidated spreadsheet will not be made publicly available in that it may include proprietary (business) information.

## Canada-wide Standards for Mercury Emissions

Example public report format only

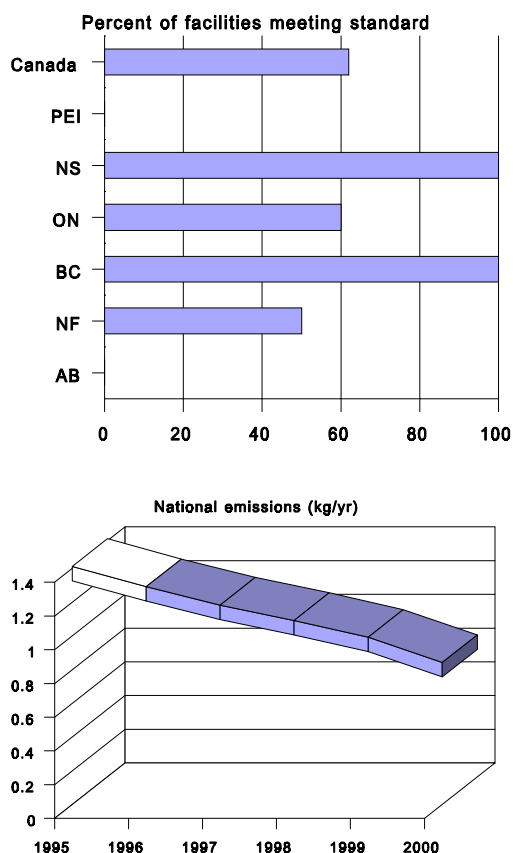
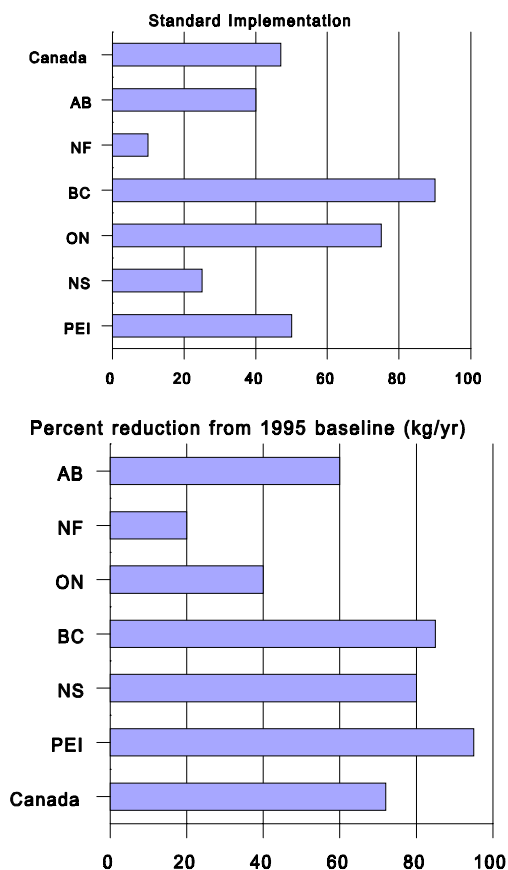
### CWS-Hg for Municipal Solid Waste Incinerators in Canada (This report covers those processing more than 120 Tonnes/yr only)

#### Report overview:

This is a consolidated report on the achievement of or compliance with Canada-Wide Standard for mercury emissions from the Municipal Solid Waste Incineration sector in Canada. Several facilities located in the Province of Quebec are not addressed in this report. This report provides an progress indicator for the jurisdictional activities to implement the standards, an indicator for facility compliance and/or achievement with the standard, a consolidated graphic showing the net reductions in emissions from this sector against two baseline years, and a national summary for the sector.

#### Sector overview:

The incineration of solid waste is utilized for two reasons in Canada - either to reduce volumes to minimize landfilling, or to achieve generate electricity (energy from waste). During the development of the Canada-wide Standard for this sector, a total of 13 major facilities and more than 100 minor facilities were evaluated. A baseline year of 2000 has been selected from the original total of 13 major facilities located in 7 jurisdictions all facilities remain in operation.



## **Canada-wide Standard for Mercury Emissions**

Signed by:

British Columbia	Honourable Joan Sawicki
Alberta	Honourable Halvar Johnson
Saskatchewan	Honourable Buckley Belanger
Manitoba	Honourable Oscar Lathlin
Ontario	Honourable Dan Newman
Environment Canada	Honourable David Anderson
New Brunswick	Honourable Kim Jardine
Nova Scotia	Honourable Michael Baker
Prince Edward Island	Honourable Kevin MacAdam
Newfoundland and Labrador	Honourable Oliver Langdon Honourable Walter Noel
Yukon	Honourable Dale Eftoda
Northwest Territories	Honourable Joseph Handley
Nunavut	Honourable Peter Kilabuk

Note: Québec has not endorsed the Canada-wide Accord on Environmental Harmonization or the Canada-wide Environmental Standards Sub-agreement.



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## **Appendix E**

U.S. EPA Method 29 Metals from Stationary Sources

**METHOD 29 - DETERMINATION OF METALS EMISSIONS  
FROM STATIONARY SOURCES**

**NOTE:** This method does not include all of the specifications (e.g. equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 5 and Method 12.

*1.0 Scope and Application.*

1.1 Analytes.

Analyte	CAS No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-2
Barium (Ba)	7440-39-3
Beryllium (Be)	7440-41-7
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Manganese (Mn)	7439-96-5
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Phosphorus (P)	7723-14-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4

Analyte	CAS No.
Thallium (Tl)	7440-28-0
Zinc (Zn)	7440-66-6

1.2 Applicability. This method is applicable to the determination of metals emissions from stationary sources. This method may be used to determine particulate emissions in addition to the metals emissions if the prescribed procedures and precautions are followed.

1.2.1 Hg emissions can be measured, alternatively, using EPA Method 101A of Appendix B, 40 CFR Part 61. Method 101-A measures only Hg but it can be of special interest to sources which need to measure both Hg and Mn emissions.

## 2.0 Summary of Method.

2.1 Principle. A stack sample is withdrawn isokinetically from the source, particulate emissions are collected in the probe and on a heated filter, and gaseous emissions are then collected in an aqueous acidic solution of hydrogen peroxide (analyzed for all metals including Hg) and an aqueous acidic solution of potassium permanganate (analyzed only for Hg). The recovered samples are digested, and appropriate fractions are analyzed for Hg by cold vapor atomic absorption spectroscopy (CVAAS) and for Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, P, Se, Ag, Tl, and Zn by inductively coupled argon plasma emission spectroscopy

(ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of Sb, As, Cd, Co, Pb, Se, and Tl if these elements require greater analytical sensitivity than can be obtained by ICAP. If one so chooses, AAS may be used for analysis of all listed metals if the resulting in-stack method detection limits meet the goal of the testing program. Similarly, inductively coupled plasma-mass spectroscopy (ICP-MS) may be used for analysis of Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl and Zn.

### 3.0 *Definitions.* [Reserved]

### 4.0 *Interferences.*

4.1 Iron (Fe) can be a spectral interference during the analysis of As, Cr, and Cd by ICAP. Aluminum (Al) can be a spectral interference during the analysis of As and Pb by ICAP. Generally, these interferences can be reduced by diluting the analytical sample, but such dilution raises the in-stack detection limits. Background and overlap corrections may be used to adjust for spectral interferences. Refer to Method 6010 of Reference 2 in Section 16.0 or the other analytical methods used for details on potential interferences to this method. For all GFAAS analyses, use matrix modifiers to limit interferences, and matrix match all standards.

## 5.0 Safety.

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Nitric Acid ( $\text{HNO}_3$ ). Highly corrosive to eyes, skin, nose, and lungs. Vapors cause bronchitis, pneumonia, or edema of lungs. Reaction to inhalation may be delayed as long as 30 hours and still be fatal. Provide ventilation to limit exposure. Strong oxidizer. Hazardous reaction may occur with organic materials such as solvents.

5.2.2 Sulfuric Acid ( $\text{H}_2\text{SO}_4$ ). Rapidly destructive to body tissue. Will cause third degree burns. Eye damage may result in blindness. Inhalation may be fatal from spasm of

the larynx, usually within 30 minutes. May cause lung tissue damage with edema. 1 mg/m<sup>3</sup> for 8 hours will cause lung damage or, in higher concentrations, death. Provide ventilation to limit inhalation. Reacts violently with metals and organics.

5.2.3 Hydrochloric Acid (HCl). Highly corrosive liquid with toxic vapors. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent can be lethal to humans in a few minutes. Provide ventilation to limit exposure. Reacts with metals, producing hydrogen gas.

5.2.4 Hydrofluoric Acid (HF). Highly corrosive to eyes, skin, nose, throat, and lungs. Reaction to exposure may be delayed by 24 hours or more. Provide ventilation to limit exposure.

5.2.5 Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>). Irritating to eyes, skin, nose, and lungs. 30% H<sub>2</sub>O<sub>2</sub> is a strong oxidizing agent. Avoid contact with skin, eyes, and combustible material. Wear gloves when handling.

5.2.6 Potassium Permanganate (KMnO<sub>4</sub>). Caustic, strong oxidizer. Avoid bodily contact with.

5.2.7 Potassium Persulfate. Strong oxidizer. Avoid bodily contact with. Keep containers well closed and in a cool place.

5.3 Reaction Pressure. Due to the potential reaction of the potassium permanganate with the acid, there could be pressure buildup in the acidic  $\text{KMnO}_4$  absorbing solution storage bottle. Therefore these bottles shall not be fully filled and shall be vented to relieve excess pressure and prevent explosion potentials. Venting is required, but not in a manner that will allow contamination of the solution. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

#### *6.0 Equipment and Supplies.*

6.1 Sampling. A schematic of the sampling train is shown in Figure 29-1. It has general similarities to the Method 5 train.

6.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as Method 5, Sections 6.1.1.1 and 6.1.1.2, except that glass nozzles are required unless alternate tips are constructed of materials that are free from contamination and will not interfere with the sample. If a probe tip other than glass is used, no correction to the sample test results to compensate for the nozzle's effect on the sample is allowed. Probe fittings of plastic such as Teflon, polypropylene, etc. are recommended instead of metal fittings to prevent contamination. If one

chooses to do so, a single glass piece consisting of a combined probe tip and probe liner may be used.

6.1.2 Pitot Tube and Differential Pressure Gauge. Same as Method 2, Sections 6.1 and 6.2, respectively.

6.1.3 Filter Holder. Glass, same as Method 5, Section 6.1.1.5, except use a Teflon filter support or other non-metallic, non-contaminating support in place of the glass frit.

6.1.4 Filter Heating System. Same as Method 5, Section 6.1.1.6.

6.1.5 Condenser. Use the following system for condensing and collecting gaseous metals and determining the moisture content of the stack gas. The condensing system shall consist of four to seven impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. Use the first impinger as a moisture trap. The second impinger (which is the first  $\text{HNO}_3/\text{H}_2\text{O}_2$  impinger) shall be identical to the first impinger in Method 5. The third impinger (which is the second  $\text{HNO}_3/\text{H}_2\text{O}_2$  impinger) shall be a Greenburg Smith impinger with the standard tip as described for the second impinger in Method 5, Section 6.1.1.8. The fourth (empty) impinger and the fifth and sixth (both acidified  $\text{KMnO}_4$ ) impingers are the same as the first impinger in Method 5. Place a temperature sensor capable of measuring to within  $1^\circ\text{C}$  ( $2^\circ\text{F}$ ) at the



outlet of the last impinger. If no Hg analysis is planned, then the fourth, fifth, and sixth impingers are not used.

6.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 6.1.1.9, 6.1.2, and 6.1.3, respectively.

6.1.7 Teflon Tape. For capping openings and sealing connections, if necessary, on the sampling train.

6.2 Sample Recovery. Same as Method 5, Sections 6.2.1 through 6.2.8 (Probe-Liner and Probe-Nozzle Brushes or Swabs, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

6.2.1 Non-metallic Probe-Liner and Probe-Nozzle Brushes or Swabs. Use non-metallic probe-liner and probe-nozzle brushes or swabs for quantitative recovery of materials collected in the front-half of the sampling train.

6.2.2 Sample Storage Containers. Use glass bottles (see Section 8.1 of this Method) with Teflon-lined caps that are non-reactive to the oxidizing solutions, with capacities of 1000- and 500-ml, for storage of acidified  $\text{KMnO}_4$ -containing samples and blanks. Glass or polyethylene bottles may be used for other sample types.

6.2.3 Graduated Cylinder. Glass or equivalent.

6.2.4 Funnel. Glass or equivalent.

6.2.5 Labels. For identifying samples.

6.2.6 Polypropylene Tweezers and/or Plastic Gloves.

For recovery of the filter from the sampling train filter holder.

6.3 Sample Preparation and Analysis.

6.3.1 Volumetric Flasks, 100-ml, 250-ml, and 1000-ml.

For preparation of standards and sample dilutions.

6.3.2 Graduated Cylinders. For preparation of reagents.

6.3.3 Parr Bombs or Microwave Pressure Relief Vessels with Capping Station (CEM Corporation model or equivalent). For sample digestion.

6.3.4 Beakers and Watch Glasses. 250-ml beakers, with watch glass covers, for sample digestion.

6.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

6.3.6 Filter Funnels. For holding filter paper.

6.3.7 Disposable Pasteur Pipets and Bulbs.

6.3.8 Volumetric Pipets.

6.3.9 Analytical Balance. Accurate to within 0.1 mg.

6.3.10 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures, respectively.

6.3.11 Hot Plates.

6.3.12 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

6.3.12.1 Graphite Furnace Attachment. With Sb, As, Cd, Co, Pb, Se, and Tl hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). Same as Reference 2 in Section 16.0. Methods 7041 (Sb), 7060 (As), 7131 (Cd), 7201 (Co), 7421 (Pb), 7740 (Se), and 7841 (Tl).

6.3.12.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL, an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp shall be capable of raising the temperature at the quartz cell by 10°C above ambient, so that no condensation forms on the wall of the quartz cell. Same as Method 7470 in Reference 2 in Section 16.0. See **NOTE 2:** Section 11.1.3 for other acceptable approaches for analysis of Hg in which analytical detection limits of 0.002 ng/ml were obtained.

6.3.13 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. Same as EPA Method 6010 in Reference 2 in Section 16.0.

6.3.14 Inductively Coupled Plasma-Mass Spectrometer. Same as EPA Method 6020 in Reference 2 in Section 16.0.

## 7.0 *Reagents and Standards.*

7.1 Unless otherwise indicated, it is intended that all reagents conform to the specifications established by

the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

## 7.2 Sampling Reagents.

7.2.1 Sample Filters. Without organic binders. The filters shall contain less than  $1.3 \mu\text{g}/\text{in.}^2$  of each of the metals to be measured. Analytical results provided by filter manufacturers stating metals content of the filters are acceptable. However, if no such results are available, analyze filter blanks for each target metal prior to emission testing. Quartz fiber filters meeting these requirements are recommended. However, if glass fiber filters become available which meet these requirements, they may be used. Filter efficiencies and unreactiveness to sulfur dioxide ( $\text{SO}_2$ ) or sulfur trioxide ( $\text{SO}_3$ ) shall be as described in Section 7.1.1 of Method 5.

7.2.2 Water. To conform to ASTM Specification D1193-77 or 91, Type II (incorporated by reference -- see §60.17). If necessary, analyze the water for all target metals prior to field use. All target metals should be less than 1 ng/ml.

7.2.3  $\text{HNO}_3$ , Concentrated. Baker Instra-analyzed or equivalent.

7.2.4  $\text{HCl}$ , Concentrated. Baker Instra-analyzed or equivalent.

7.2.5  $\text{H}_2\text{O}_2$ , 30 Percent (V/V).

7.2.6  $\text{KMnO}_4$ .

7.2.7  $\text{H}_2\text{SO}_4$ , Concentrated.

7.2.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 7.1.2 and 7.1.4, respectively.

### 7.3 Pretest Preparation of Sampling Reagents.

7.3.1  $\text{HNO}_3/\text{H}_2\text{O}_2$  Absorbing Solution, 5 Percent  $\text{HNO}_3$ /10 Percent  $\text{H}_2\text{O}_2$ . Add carefully with stirring 50 ml of concentrated  $\text{HNO}_3$  to a 1000-ml volumetric flask containing approximately 500 ml of water, and then add carefully with stirring 333 ml of 30 percent  $\text{H}_2\text{O}_2$ . Dilute to volume with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

7.3.2 Acidic  $\text{KMnO}_4$  Absorbing Solution, 4 Percent  $\text{KMnO}_4$  (W/V), 10 Percent  $\text{H}_2\text{SO}_4$  (V/V). Prepare fresh daily. Mix carefully, with stirring, 100 ml of concentrated  $\text{H}_2\text{SO}_4$  into approximately 800 ml of water, and add water with stirring to make a volume of 1 liter: this solution is 10 percent  $\text{H}_2\text{SO}_4$  (V/V). Dissolve, with stirring, 40 g of  $\text{KMnO}_4$  into 10 percent  $\text{H}_2\text{SO}_4$  (V/V) and add 10 percent  $\text{H}_2\text{SO}_4$  (V/V) with stirring to make a volume of 1 liter. Prepare and store in glass bottles to prevent degradation. This reagent shall contain less than 2 ng/ml of Hg.

**Precaution:** To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper.

7.3.3  $\text{HNO}_3$ , 0.1 N. Add with stirring 6.3 ml of concentrated  $\text{HNO}_3$  (70 percent) to a flask containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

7.3.4  $\text{HCl}$ , 8 N. Carefully add with stirring 690 ml of concentrated  $\text{HCl}$  to a flask containing 250 ml of water. Dilute to 1000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of Hg.

#### 7.4 Glassware Cleaning Reagents.

7.4.1  $\text{HNO}_3$ , Concentrated. Fisher ACS grade or equivalent.

7.4.2 Water. To conform to ASTM Specifications D1193, Type II.

7.4.3  $\text{HNO}_3$ , 10 Percent (V/V). Add with stirring 500 ml of concentrated  $\text{HNO}_3$  to a flask containing approximately 4000 ml of water. Dilute to 5000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

7.5 Sample Digestion and Analysis Reagents. The metals standards, except Hg, may also be made from solid

chemicals as described in Reference 3 in Section 16.0.

Refer to References 1, 2, or 5 in Section 16.0 for additional information on Hg standards. The 1000 µg/ml Hg stock solution standard may be made according to Section 7.2.7 of Method 101A.

7.5.1 HCl, Concentrated.

7.5.2 HF, Concentrated.

7.5.3 HNO<sub>3</sub>, Concentrated. Baker Instra-analyzed or equivalent.

7.5.4 HNO<sub>3</sub>, 50 Percent (V/V). Add with stirring 125 ml of concentrated HNO<sub>3</sub> to 100 ml of water. Dilute to 250 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

7.5.5 HNO<sub>3</sub>, 5 Percent (V/V). Add with stirring 50 ml of concentrated HNO<sub>3</sub> to 800 ml of water. Dilute to 1000 ml with water. Mix well. This reagent shall contain less than 2 ng/ml of each target metal.

7.5.6 Water. To conform to ASTM Specifications D1193, Type II.

7.5.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See Reference 2 In Section 16.0 for preparation.

7.5.8 Stannous Chloride. See Reference 2 in Section 16.0 for preparation.

7.5.9 KMnO<sub>4</sub>, 5 Percent (W/V). See Reference 2 in Section 16.0 for preparation.

7.5.10  $\text{H}_2\text{SO}_4$ , Concentrated.

7.5.11 Potassium Persulfate, 5 Percent (W/V). See Reference 2 in Section 16.0 for preparation.

7.5.12 Nickel Nitrate,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ .

7.5.13 Lanthanum Oxide,  $\text{La}_2\text{O}_3$ .

7.5.14 Hg Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.15 Pb Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.16 As Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.17 Cd Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.18 Cr Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.19 Sb Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.20 Ba Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.21 Be Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.22 Co Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.23 Cu Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.24 Mn Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.25 Ni Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.26 P Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.27 Se Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.28 Ag Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.29 Tl Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.30 Zn Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.31 Al Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .

7.5.32 Fe Standard (AAS Grade), 1000  $\mu\text{g}/\text{ml}$ .



### 7.5.33 Hg Standards and Quality Control Samples.

Prepare fresh weekly a 10 µg/ml intermediate Hg standard by adding 5 ml of 1000 µg/ml Hg stock solution prepared according to Method 101A to a 500-ml volumetric flask; dilute with stirring to 500 ml by first carefully adding 20 ml of 15 percent HNO<sub>3</sub> and then adding water to the 500-ml volume. Mix well. Prepare a 200 ng/ml working Hg standard solution fresh daily: add 5 ml of the 10 µg/ml intermediate standard to a 250-ml volumetric flask, and dilute to 250 ml with 5 ml of 4 percent KMnO<sub>4</sub>, 5 ml of 15 percent HNO<sub>3</sub>, and then water. Mix well. Use at least five separate aliquots of the working Hg standard solution and a blank to prepare the standard curve. These aliquots and blank shall contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution containing 0, 200, 400, 600, 800, and 1000 ng Hg, respectively. Prepare quality control samples by making a separate 10 µg/ml standard and diluting until in the calibration range.

### 7.5.34 ICAP Standards and Quality Control Samples.

Calibration standards for ICAP analysis can be combined into four different mixed standard solutions as follows:

#### MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

Solution	Elements
I	As, Be, Cd, Mn, Pb, Se, Zn

II	Ba, Co, Cu, Fe
III	Al, Cr, Ni
IV	Ag, P, Sb, Tl

---

Prepare these standards by combining and diluting the appropriate volumes of the 1000 µg/ml solutions with 5 percent HNO<sub>3</sub>. A minimum of one standard and a blank can be used to form each calibration curve. However, prepare a separate quality control sample spiked with known amounts of the target metals in quantities in the mid-range of the calibration curve. Suggested standard levels are 25 µg/ml for Al, Cr and Pb, 15 µg/ml for Fe, and 10 µg/ml for the remaining elements. Prepare any standards containing less than 1 µg/ml of metal on a daily basis. Standards containing greater than 1 µg/ml of metal should be stable for a minimum of 1 to 2 weeks. For ICP-MS, follow Method 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i).

7.5.35 GFAAS Standards. Sb, As, Cd, Co, Pb, Se, and Tl. Prepare a 10 µg/ml standard by adding 1 ml of 1000 µg/ml standard to a 100-ml volumetric flask. Dilute with stirring to 100 ml with 10 percent HNO<sub>3</sub>. For GFAAS, matrix match the standards. Prepare a 100 ng/ml standard by adding 1 ml of the 10 µg/ml standard to a 100-ml volumetric flask,

and dilute to 100 ml with the appropriate matrix solution. Prepare other standards by diluting the 100 ng/ml standards. Use at least five standards to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Prepare quality control samples by making a separate 10 µg/ml standard and diluting until it is in the range of the samples. Prepare any standards containing less than 1 µg/ml of metal on a daily basis. Standards containing greater than 1 µg/ml of metal should be stable for a minimum of 1 to 2 weeks.

#### 7.5.36 Matrix Modifiers.

7.5.36.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  or other nickel compound suitable for preparation of this matrix modifier in approximately 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

7.5.36.2 Nickel Nitrate, 0.1 Percent (V/V). Dilute 10 ml of 1 percent nickel nitrate solution to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during GFAAS analysis for As.

7.5.36.3 Lanthanum. Carefully dissolve 0.5864 g of  $\text{La}_2\text{O}_3$  in 10 ml of concentrated  $\text{HNO}_3$ , and dilute the solution by adding it with stirring to approximately 50 ml of water. Dilute to 100 ml with water, and mix well. Inject an equal

amount of sample and this modifier into the graphite furnace during GFAAS analysis for Pb.

7.5.37 Whatman 40 and 541 Filter Papers (or equivalent). For filtration of digested samples.

## *8.0 Sample Collection, Preservation, Transport, and Storage.*

8.1 Sampling. The complexity of this method is such that, to obtain reliable results, both testers and analysts must be trained and experienced with the test procedures, including source sampling; reagent preparation and handling; sample handling; safety equipment and procedures; analytical calculations; reporting; and the specific procedural descriptions throughout this method.

8.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 8.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. First, rinse all sampling train glassware with hot tap water and then wash in hot soapy water. Next, rinse glassware three times with tap water, followed by three additional rinses with water. Then soak all glassware in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinse three times with water, rinse a final time with acetone, and allow to air

dry. Cover all glassware openings where contamination can occur until the sampling train is assembled for sampling.

8.1.2 Preliminary Determinations. Same as Method 5, Section 8.1.2.

8.1.3 Preparation of Sampling Train.

8.1.3.1 Set up the sampling train as shown in Figure 29-1. Follow the same general procedures given in Method 5, Section 8.3, except place 100 ml of the  $\text{HNO}_3/\text{H}_2\text{O}_2$  solution (Section 7.3.1 of this method) in each of the second and third impingers as shown in Figure 29-1. Place 100 ml of the acidic  $\text{KMnO}_4$  absorbing solution (Section 7.3.2 of this method) in each of the fifth and sixth impingers as shown in Figure 29-1, and transfer approximately 200 to 300 g of pre-weighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to final train assembly.

8.1.3.2 Based on the specific source sampling conditions, the use of an empty first impinger can be eliminated if the moisture to be collected in the impingers will be less than approximately 100 ml.

8.1.3.3 If Hg analysis will not be performed, the fourth, fifth, and sixth impingers as shown in Figure 29-1 are not required.

8.1.3.4 To insure leak-free sampling train connections and to prevent possible sample contamination problems, use

Teflon tape or other non-contaminating material instead of silicone grease.

**Precaution:** Exercise extreme care to prevent contamination within the train. Prevent the acidic  $\text{KMnO}_4$  from contacting any glassware that contains sample material to be analyzed for Mn. Prevent acidic  $\text{H}_2\text{O}_2$  from mixing with the acidic  $\text{KMnO}_4$ .

8.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 8.4.2 (Pretest Leak-Check), Section 8.4.3 (Leak-Checks During the Sample Run), and Section 8.4.4 (Post-Test Leak-Checks).

8.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 8.5. When sampling for Hg, use a procedure analogous to that described in Section 8.1 of Method 101A, 40 CFR Part 61, Appendix B, if necessary to maintain the desired color in the last acidified permanganate impinger. For each run, record the data required on a data sheet such as the one shown in Figure 5-3 of Method 5.

8.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 12.11.

8.2 Sample Recovery.

8.2.1 Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period. The

probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling; a vacuum can form in the filter holder with the undesired result of drawing liquid from the impingers onto the filter.

8.2.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the impinger. Cap the filter holder outlet and impinger inlet. Use non-contaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon® tape to close these openings.

8.2.3 Alternatively, the following procedure may be used to disassemble the train before the probe and filter holder/oven are completely cooled: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

8.2.4 Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. Take special precautions to assure that all the items necessary for recovery do not contaminate the samples. The sample is recovered and treated as follows (see schematic in Figures 29-2a and 29-2b):

8.2.5 Container No. 1 (Sample Filter). Carefully remove the filter from the filter holder and place it in its labeled petri dish container. To handle the filter, use either acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water and dried. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metal-containing materials when recovering this train. Seal the labeled petri dish.

8.2.6 Container No. 2 (Acetone Rinse). Perform this procedure only if a determination of particulate emissions is to be made. Quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting,



probe liner, and front half of the filter holder by washing these components with a total of 100 ml of acetone, while simultaneously taking great care to see that no dust on the outside of the probe or other surfaces gets in the sample. The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis.

8.2.6.1 Carefully remove the probe nozzle, and clean the inside surface by rinsing with acetone from a wash bottle while brushing with a non-metallic brush. Brush until the acetone rinse shows no visible particles, then make a final rinse of the inside surface with acetone.

8.2.6.2 Brush and rinse the sample exposed inside parts of the probe fitting with acetone in a similar way until no visible particles remain. Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a non-metallic probe brush. Hold the probe in an inclined position, squirt

acetone into the upper end as the probe brush is being pushed with a twisting action three times through the probe. Hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

8.2.6.3 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination. Clean the inside of the front-half of the filter holder by rubbing the surfaces with a non-metallic brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid so that acetone will not leak out when shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Clearly label the container to identify its contents.

8.2.7 Container No. 3 (Probe Rinse). Keep the probe assembly clean and free from contamination during the probe rinse. Rinse the probe nozzle and fitting, probe liner, and front-half of the filter holder thoroughly with a total of 100 ml of 0.1 N  $\text{HNO}_3$ , and place the wash into a sample storage container. Perform the rinses as applicable and generally as described in Method 12, Section 8.7.1. Record the volume of the rinses. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during transport. Seal the container, and clearly label the contents. Finally, rinse the nozzle, probe liner, and front-half of the filter holder with water followed by acetone, and discard these rinses.

**NOTE:** The use of a total of exactly 100 ml is necessary for the subsequent blank correction procedures.

8.2.8 Container No. 4 (Impingers 1 through 3, Moisture Knockout Impinger, when used,  $\text{HNO}_3/\text{H}_2\text{O}_2$  Impingers Contents and Rinses). Due to the potentially large quantity of liquid involved, the tester may place the impinger solutions from impingers 1 through 3 in more than one container, if necessary. Measure the liquid in the first three impingers to within 0.5 ml using a graduated cylinder. Record the volume. This information is required to calculate the

moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N  $\text{HNO}_3$  using the procedure as applicable in Method 12, Section 8.7.3.

**NOTE:** The use of exactly 100 ml of 0.1 N  $\text{HNO}_3$  rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the final total volume. Mark the height of the fluid level, seal the container, and clearly label the contents.

8.2.9 Container Nos. 5A (0.1 N  $\text{HNO}_3$ ), 5B ( $\text{KMnO}_4/\text{H}_2\text{SO}_4$  absorbing solution), and 5C (8 N  $\text{HCl}$  rinse and dilution).

8.2.9.1 When sampling for Hg, pour all the liquid from the impinger (normally impinger No. 4) that immediately preceded the two permanganate impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place the liquid in Container No. 5A. Rinse the impinger with exactly 100 ml of 0.1 N  $\text{HNO}_3$  and place this rinse in Container No. 5A.

8.2.9.2 Pour all the liquid from the two permanganate impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place this

acidic  $\text{KMnO}_4$  solution into Container No. 5B. Using a total of exactly 100 ml of fresh acidified  $\text{KMnO}_4$  solution for all rinses (approximately 33 ml per rinse), rinse the two permanganate impingers and connecting glassware a minimum of three times. Pour the rinses into Container No. 5B, carefully assuring transfer of all loose precipitated materials from the two impingers. Similarly, using 100 ml total of water, rinse the permanganate impingers and connecting glass a minimum of three times, and pour the rinses into Container 5B, carefully assuring transfer of any loose precipitated material. Mark the height of the fluid level, and clearly label the contents. Read the **Precaution:** in Section 7.3.2.

**NOTE:** Due to the potential reaction of  $\text{KMnO}_4$  with acid, pressure buildup can occur in the sample storage bottles. Do not fill these bottles completely and take precautions to relieve excess pressure. A No. 70-72 hole drilled in the container cap and Teflon liner has been used successfully.

8.2.9.3 If no visible deposits remain after the water rinse, no further rinse is necessary. However, if deposits remain on the impinger surfaces, wash them with 25 ml of 8 N  $\text{HCl}$ , and place the wash in a separate sample container labeled No. 5C containing 200 ml of water. First, place 200

ml of water in the container. Then wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing both permanganate impingers combined. Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse. Finally, pour the 25 ml of 8 N HCl rinse carefully into the container. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport.

8.2.10 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal it. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that might adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

8.2.11 Container No. 7 (Acetone Blank). If particulate emissions are to be determined, at least once during each field test, place a 100-ml portion of the acetone used in the sample recovery process into a container labeled No. 7. Seal the container.

8.2.12 Container No. 8A (0.1 N HNO<sub>3</sub> Blank). At least once during each field test, place 300 ml of the 0.1 N HNO<sub>3</sub> solution used in the sample recovery process into a container labeled No. 8A. Seal the container.

8.2.13 Container No. 8B (Water Blank). At least once during each field test, place 100 ml of the water used in the sample recovery process into a container labeled No. 8B. Seal the container.

8.2.14 Container No. 9 (5 Percent HNO<sub>3</sub>/10 Percent H<sub>2</sub>O<sub>2</sub> Blank). At least once during each field test, place 200 ml of the 5 Percent HNO<sub>3</sub>/10 Percent H<sub>2</sub>O<sub>2</sub> solution used as the nitric acid impinger reagent into a container labeled No. 9. Seal the container.

8.2.15 Container No. 10 (Acidified KMnO<sub>4</sub> Blank). At least once during each field test, place 100 ml of the acidified KMnO<sub>4</sub> solution used as the impinger solution and in the sample recovery process into a container labeled No. 10. Prepare the container as described in Section 8.2.9.2.

Read the **Precaution:** in Section 7.3.2 and read the **NOTE** in Section 8.2.9.2.

8.2.16 Container No. 11 (8 N HCl Blank). At least once during each field test, place 200 ml of water into a sample container labeled No. 11. Then carefully add with stirring 25 ml of 8 N HCl. Mix well and seal the container.

8.2.17 Container No. 12 (Sample Filter Blank). Once during each field test, place into a petri dish labeled No. 12 three unused blank filters from the same lot as the sampling filters. Seal the petri dish.

8.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure 29-3.

8.3.1 Container No. 1 (Sample Filter).

8.3.1.1 If particulate emissions are being determined, first desiccate the filter and filter catch without added heat (do not heat the filters to speed the drying) and weigh to a constant weight as described in Section 11.2.1 of Method 5.



8.3.1.2 Following this procedure, or initially, if particulate emissions are not being determined in addition to metals analysis, divide the filter with its filter catch into portions containing approximately 0.5 g each. Place the pieces in the analyst's choice of either individual microwave pressure relief vessels or Parr Bombs. Add 6 ml of concentrated  $\text{HNO}_3$  and 4 ml of concentrated HF to each vessel. For microwave heating, microwave the samples for approximately 12 to 15 minutes total heating time as follows: heat for 2 to 3 minutes, then turn off the microwave for 2 to 3 minutes, then heat for 2 to 3 minutes, etc., continue this alternation until the 12 to 15 minutes total heating time are completed (this procedure should comprise approximately 24 to 30 minutes at 600 watts). Microwave heating times are approximate and are dependent upon the number of samples being digested simultaneously. Sufficient heating is evidenced by sorbent reflux within the vessel. For conventional heating, heat the Parr Bombs at  $140^\circ\text{C}$  ( $285^\circ\text{F}$ ) for 6 hours. Then cool the samples to room temperature, and combine with the acid digested probe rinse as required in Section 8.3.3.

8.3.1.3 If the sampling train includes an optional glass cyclone in front of the filter, prepare and digest the cyclone catch by the procedures described in Section 8.3.1.2

and then combine the digestate with the digested filter sample.

8.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically within 1 ml or gravimetrically within 0.5 g. Transfer the contents to an acid-cleaned, tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. If particulate emissions are being determined, desiccate for 24 hours without added heat, weigh to a constant weight according to the procedures described in Section 11.2.1 of Method 5, and report the results to the nearest 0.1 mg. Redissolve the residue with 10 ml of concentrated  $\text{HNO}_3$ . Quantitatively combine the resultant sample, including all liquid and any particulate matter, with Container No. 3 before beginning Section 8.3.3.

8.3.3 Container No. 3 (Probe Rinse). Verify that the pH of this sample is 2 or lower. If it is not, acidify the sample by careful addition with stirring of concentrated  $\text{HNO}_3$  to pH 2. Use water to rinse the sample into a beaker, and cover the beaker with a ribbed watch glass. Reduce the

sample volume to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Digest the sample in microwave vessels or Parr Bombs by quantitatively transferring the sample to the vessel or bomb, carefully adding the 6 ml of concentrated  $\text{HNO}_3$ , 4 ml of concentrated HF, and then continuing to follow the procedures described in Section 8.3.1.2. Then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 8.3.1.2. The resultant combined sample is referred to as "Sample Fraction 1". Filter the combined sample using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. This diluted sample is "Analytical Fraction 1". Measure and record the volume of Analytical Fraction 1 to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as "Analytical Fraction 1B". Label the remaining 250-ml portion as "Analytical Fraction 1A". Analytical Fraction 1A is used for ICAP or AAS analysis for all desired metals except Hg. Analytical Fraction 1B is used for the determination of front-half Hg.

8.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample to within 0.5 ml and label it "Sample Fraction 2". Remove a 75- to 100-ml aliquot for Hg analysis and label the aliquot "Analytical Fraction 2B". Label the remaining portion of Container

No. 4 as "Sample Fraction 2A". Sample Fraction 2A defines the volume of Analytical Fraction 2A prior to digestion. All of Sample Fraction 2A is digested to produce "Analytical Fraction 2A". Analytical Fraction 2A defines the volume of Sample Fraction 2A after its digestion and the volume of Analytical Fraction 2A is normally 150 ml. Analytical Fraction 2A is analyzed for all metals except Hg. Verify that the pH of Sample Fraction 2A is 2 or lower. If necessary, use concentrated  $\text{HNO}_3$  by careful addition and stirring to lower Sample Fraction 2A to pH 2. Use water to rinse Sample Fraction 2A into a beaker and then cover the beaker with a ribbed watchglass. Reduce Sample Fraction 2A to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Then follow either of the digestion procedures described in Sections 8.3.4.1 or 8.3.4.2.

8.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent  $\text{HNO}_3$ , and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent  $\text{H}_2\text{O}_2$  and heat for 10 more minutes. Add 50 ml of hot water, and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution produces Analytical Fraction 2A. Measure and record the volume to within 0.1 ml.

8.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent  $\text{HNO}_3$  and heat for 6 minutes total heating time in alternations of 1 to 2 minutes at 600 Watts followed by 1 to 2 minutes with no power, etc., similar to the procedure described in Section 8.3.1. Allow the sample to cool. Add 10 ml of 3 percent  $\text{H}_2\text{O}_2$  and heat for 2 more minutes. Add 50 ml of hot water, and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution produces Analytical Fraction 2A. Measure and record the volume to within 0.1 ml.

**NOTE:** All microwave heating times given are approximate and are dependent upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.

8.3.5 Container No. 5A (Impinger 4), Container Nos. 5B and 5C (Impingers 5 and 6). Keep the samples in Containers Nos. 5A, 5B, and 5C separate from each other. Measure and record the volume of 5A to within 0.5 ml. Label the contents of Container No. 5A to be Analytical Fraction 3A. To remove any brown  $\text{MnO}_2$  precipitate from the contents of Container No. 5B, filter its contents through Whatman 40

filter paper into a 500 ml volumetric flask and dilute to volume with water. Save the filter for digestion of the brown  $\text{MnO}_2$  precipitate. Label the 500 ml filtrate from Container No. 5B to be Analytical Fraction 3B. Analyze Analytical Fraction 3B for Hg within 48 hours of the filtration step. Place the saved filter, which was used to remove the brown  $\text{MnO}_2$  precipitate, into an appropriately sized vented container, which will allow release of any gases including chlorine formed when the filter is digested. In a laboratory hood which will remove any gas produced by the digestion of the  $\text{MnO}_2$ , add 25 ml of 8 N HCl to the filter and allow to digest for a minimum of 24 hours at room temperature. Filter the contents of Container No. 5C through a Whatman 40 filter into a 500-ml volumetric flask. Then filter the result of the digestion of the brown  $\text{MnO}_2$  from Container No. 5B through a Whatman 40 filter into the same 500-ml volumetric flask, and dilute and mix well to volume with water. Discard the Whatman 40 filter. Mark this combined 500-ml dilute HCl solution as Analytical Fraction 3C.

8.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

9.0 *Quality Control.*

9.1 Field Reagent Blanks, if analyzed. Perform the digestion and analysis of the blanks in Container Nos. 7 through 12 that were produced in Sections 8.2.11 through 8.2.17, respectively. For Hg field reagent blanks, use a 10 ml aliquot for digestion and analysis.

9.1.1 Digest and analyze one of the filters from Container No. 12 per Section 8.3.1, 100 ml from Container No. 7 per Section 8.3.2, and 100 ml from Container No. 8A per Section 8.3.3. This step produces blanks for Analytical Fractions 1A and 1B.

9.1.2 Combine 100 ml of Container No. 8A with 200 ml from Container No. 9, and digest and analyze the resultant volume per Section 8.3.4. This step produces blanks for Analytical Fractions 2A and 2B.

9.1.3 Digest and analyze a 100-ml portion of Container No. 8A to produce a blank for Analytical Fraction 3A.

9.1.4 Combine 100 ml from Container No. 10 with 33 ml from Container No. 8B to produce a blank for Analytical Fraction 3B. Filter the resultant 133 ml as described for Container No. 5B in Section 8.3.5, except do not dilute the 133 ml. Analyze this blank for Hg within 48 hr of the filtration step, and use 400 ml as the blank volume when calculating the blank mass value. Use the actual volumes of the other analytical blanks when calculating their mass values.

9.1.5 Digest the filter that was used to remove any brown  $\text{MnO}_2$  precipitate from the blank for Analytical Fraction 3B by the same procedure as described in Section 8.3.5 for the similar sample filter. Filter the digestate and the contents of Container No. 11 through Whatman 40 paper into a 500-ml volumetric flask, and dilute to volume with water. These steps produce a blank for Analytical Fraction 3C.

9.1.6 Analyze the blanks for Analytical Fraction Blanks 1A and 2A per Section 11.1.1 and/or Section 11.1.2. Analyze the blanks for Analytical Fractions 1B, 2B, 3A, 3B, and 3C per Section 11.1.3. Analysis of the blank for Analytical Fraction 1A produces the front-half reagent blank correction values for the desired metals except for Hg; Analysis of the blank for Analytical Fraction 1B produces the front-half reagent blank correction value for Hg. Analysis of the blank for Analytical Fraction 2A produces the back-half reagent blank correction values for all of the desired metals except for Hg, while separate analyses of the blanks for Analytical Fractions 2B, 3A, 3B, and 3C produce the back-half reagent blank correction value for Hg.

9.2 Quality Control Samples. Analyze the following quality control samples.

9.2.1 ICAP and ICP-MS Analysis. Follow the respective quality control descriptions in Section 8 of Methods 6010



and 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i). For the purposes of a source test that consists of three sample runs, modify those requirements to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (analyze by Method of Standard Additions unless within 25 percent), one quality control sample to check the accuracy of the calibration standards (required to be within 25 percent of calibration), and one duplicate analysis (required to be within 20 percent of average or repeat all analyses).

9.2.2 Direct Aspiration AAS and/or GFAAS Analysis for Sb, As, Ba, Be, Cd, Cu, Cr, Co, Pb, Ni, Mn, Hg, P, Se, Ag, Tl, and Zn. Analyze all samples in duplicate. Perform a matrix spike on at least one front-half sample and one back-half sample, or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the Method of Standard Additions. Analyze a quality control sample to check the accuracy of the calibration standards. If the results are not within 20 percent, repeat the calibration.

9.2.3 CVAAS Analysis for Hg. Analyze all samples in duplicate. Analyze a quality control sample to check the

accuracy of the calibration standards (if not within 15 percent, repeat calibration). Perform a matrix spike on one sample (if not within 25 percent, analyze all samples by the Method of Standard Additions). Additional information on quality control can be obtained from Method 7470 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i), or in Standard Methods for Water and Wastewater Method 303F.

#### *10.0 Calibration and Standardization.*

**NOTE:** Maintain a laboratory log of all calibrations.

10.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 10.1); Pitot Tube (Section 10.2); Metering System (Section 10.3); Probe Heater (Section 10.4); Temperature Sensors (Section 10.5); Leak-Check of the Metering System (Section 8.4.1); and Barometer (Section 10.6).

10.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in Section 7.5. Profile and calibrate the instrument according to the manufacturer's recommended procedures using those standards. Check the calibration once per hour. If the instrument does not reproduce the standard concentrations within 10 percent,

perform the complete calibration procedures. Perform ICP-MS analysis by following Method 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i).

10.3 Atomic Absorption Spectrometer - Direct Aspiration AAS, GFAAS, and CVAAS analyses. Prepare the standards as outlined in Section 7.5 and use them to calibrate the spectrometer. Calibration procedures are also outlined in the EPA methods referred to in Table 29-2 and in Method 7470 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i), or in Standard Methods for Water and Wastewater Method 303F (for Hg). Run each standard curve in duplicate and use the mean values to calculate the calibration line. Recalibrate the instrument approximately once every 10 to 12 samples.

#### *11.0 Analytical Procedure.*

11.1 Sample Analysis. For each sampling train sample run, seven individual analytical samples are generated; two for all desired metals except Hg, and five for Hg. A schematic identifying each sample container and the prescribed analytical preparation and analysis scheme is shown in Figure 29-3. The first two analytical samples, labeled Analytical Fractions 1A and 1B, consist of the

digested samples from the front-half of the train.

Analytical Fraction 1A is for ICAP, ICP-MS or AAS analysis as described in Sections 11.1.1 and 11.1.2, respectively.

Analytical Fraction 1B is for front-half Hg analysis as described in Section 11.1.3. The contents of the back-half of the train are used to prepare the third through seventh analytical samples. The third and fourth analytical samples, labeled Analytical Fractions 2A and 2B, contain the samples from the moisture removal impinger No. 1, if used, and  $\text{HNO}_3/\text{H}_2\text{O}_2$  impingers Nos. 2 and 3. Analytical Fraction 2A is for ICAP, ICP-MS or AAS analysis for target metals, except Hg. Analytical Fraction 2B is for analysis for Hg. The fifth through seventh analytical samples, labeled Analytical Fractions 3A, 3B, and 3C, consist of the impinger contents and rinses from the empty impinger No. 4 and the  $\text{H}_2\text{SO}_4/\text{KMnO}_4$  Impingers Nos. 5 and 6. These analytical samples are for analysis for Hg as described in Section 11.1.3. The total back-half Hg catch is determined from the sum of Analytical Fractions 2B, 3A, 3B, and 3C. Analytical Fractions 1A and 2A can be combined proportionally prior to analysis.

11.1.1 ICAP and ICP-MS Analysis. Analyze Analytical Fractions 1A and 2A by ICAP using Method 6010 or Method 200.7 (40 CFR 136, Appendix C). Calibrate the ICAP, and set up an analysis program as described in Method 6010 or Method

200.7. Follow the quality control procedures described in Section 9.2.1. Recommended wavelengths for analysis are as shown in Table 29-2. These wavelengths represent the best combination of specificity and potential detection limit. Other wavelengths may be substituted if they can provide the needed specificity and detection limit, and are treated with the same corrective techniques for spectral interference. Initially, analyze all samples for the target metals (except Hg) plus Fe and Al. If Fe and Al are present, the sample might have to be diluted so that each of these elements is at a concentration of less than 50 ppm so as to reduce their spectral interferences on As, Cd, Cr, and Pb. Perform ICP-MS analysis by following Method 6020 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i).

**NOTE:** When analyzing samples in a HF matrix, an alumina torch should be used; since all front-half samples will contain HF, use an alumina torch.

11.1.2 AAS by Direct Aspiration and/or GFAAS. If analysis of metals in Analytical Fractions 1A and 2A by using GFAAS or direct aspiration AAS is needed, use Table 29-3 to determine which techniques and procedures to apply for each target metal. Use Table 29-3, if necessary, to

determine techniques for minimization of interferences. Calibrate the instrument according to Section 10.3 and follow the quality control procedures specified in Section 9.2.2.

11.1.3 CVAAS Hg analysis. Analyze Analytical Fractions 1B, 2B, 3A, 3B, and 3C separately for Hg using CVAAS following the method outlined in Method 7470 in EPA Publication SW-846 Third Edition (November 1986) including updates I, II, IIA, IIB and III, as incorporated by reference in §60.17(i), or in Standard Methods for Water and Wastewater Analysis, 15th Edition, Method 303F, or, optionally using **NOTE No. 2** at the end of this section. Set up the calibration curve (zero to 1000 ng) as described in Method 7470 or similar to Method 303F using 300-ml BOD bottles instead of Erlenmeyers. Perform the following for each Hg analysis. From each original sample, select and record an aliquot in the size range from 1 ml to 10 ml. If no prior knowledge of the expected amount of Hg in the sample exists, a 5 ml aliquot is suggested for the first dilution to 100 ml (see **NOTE No. 1** at end of this section). The total amount of Hg in the aliquot shall be less than 1 µg and within the range (zero to 1000 ng) of the calibration curve. Place the sample aliquot into a separate 300-ml BOD bottle, and add enough water to make a total volume of 100

ml. Next add to it sequentially the sample digestion solutions and perform the sample preparation described in the procedures of Method 7470 or Method 303F. (See **NOTE No. 2** at the end of this section). If the maximum readings are off-scale (because Hg in the aliquot exceeded the calibration range; including the situation where only a 1-ml aliquot of the original sample was digested), then dilute the original sample (or a portion of it) with 0.15 percent  $\text{HNO}_3$  (1.5 ml concentrated  $\text{HNO}_3$  per liter aqueous solution) so that when a 1- to 10-ml aliquot of the "0.15  $\text{HNO}_3$  percent dilution of the original sample" is digested and analyzed by the procedures described above, it will yield an analysis within the range of the calibration curve.

**NOTE No. 1:** When Hg levels in the sample fractions are below the in-stack detection limit given in Table 29-1, select a 10 ml aliquot for digestion and analysis as described.

**NOTE No. 2:** Optionally, Hg can be analyzed by using the CVAAS analytical procedures given by some instrument manufacturer's directions. These include calibration and quality control procedures for the Leeman Model PS200, the Perkin Elmer FIAS systems, and similar models, if available, of other instrument manufacturers. For digestion and analyses by these instruments, perform the following two

steps: (1), Digest the sample aliquot through the addition of the aqueous hydroxylamine hydrochloride/sodium chloride solution the same as described in this section: (The Leeman, Perkin Elmer, and similar instruments described in this note add automatically the necessary stannous chloride solution during the automated analysis of Hg.); (2), Upon completion of the digestion described in (1), analyze the sample according to the instrument manufacturer's directions. This approach allows multiple (including duplicate) automated analyses of a digested sample aliquot.

## *12.0 Data Analysis and Calculations.*

### 12.1 Nomenclature.

- A = Analytical detection limit,  $\mu\text{g/ml}$ .
- B = Liquid volume of digested sample prior to aliquotting for analysis, ml.
- C = Stack sample gas volume,  $\text{dscm}^3$ .
- $C_{a1}$  = Concentration of metal in Analytical Fraction 1A as read from the standard curve,  $\mu\text{g/ml}$ .
- $C_{a2}$  = Concentration of metal in Analytical Fraction 2A as read from the standard curve, ( $\mu\text{g/ml}$ ).
- $C_s$  = Concentration of a metal in the stack gas,  $\text{mg/dscm}$ .



- D = In-stack detection limit,  $\mu\text{g}/\text{m}^3$ .
- $F_a$  = Aliquot factor, volume of Sample Fraction 2 divided by volume of Sample Fraction 2A (see Section 8.3.4.)
- $F_d$  = Dilution factor ( $F_d$  = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument to produce the reading  $C_{a1}$ . For example, if a 2 ml aliquot of Analytical Fraction 1A is diluted to 10 ml to place it in the calibration range,  $F_d = 5$ ).
- $\text{Hg}_{bh}$  = Total mass of Hg collected in the back-half of the sampling train,  $\mu\text{g}$ .
- $\text{Hg}_{bh2}$  = Total mass of Hg collected in Sample Fraction 2,  $\mu\text{g}$ .
- $\text{Hg}_{bh3(A,B,C)}$  = Total mass of Hg collected separately in Fraction 3A, 3B, or 3C,  $\mu\text{g}$ .
- $\text{Hg}_{bhb}$  = Blank correction value for mass of Hg detected in back-half field reagent blanks,  $\mu\text{g}$ .
- $\text{Hg}_{fh}$  = Total mass of Hg collected in the front-half of the sampling train (Sample Fraction 1),  $\mu\text{g}$ .

- $Hg_{fhh}$  = Blank correction value for mass of Hg detected in front-half field reagent blank,  $\mu g$ .
- $Hg_t$  = Total mass of Hg collected in the sampling train,  $\mu g$ .
- $M_{bh}$  = Total mass of each metal (except Hg) collected in the back-half of the sampling train (Sample Fraction 2),  $\mu g$ .
- $M_{bhb}$  = Blank correction value for mass of metal detected in back-half field reagent blank,  $\mu g$ .
- $M_{fh}$  = Total mass of each metal (except Hg) collected in the front half of the sampling train (Sample Fraction 1),  $\mu g$ .
- $M_{fhh}$  = Blank correction value for mass of metal detected in front-half field reagent blank,  $\mu g$ .
- $M_t$  = Total mass of each metal (separately stated for each metal) collected in the sampling train,  $\mu g$ .
- $M_t$  = Total mass of that metal collected in the sampling train,  $\mu g$ ; (substitute  $Hg_t$  for  $M_t$  for the Hg calculation).
- $Q_{bh2}$  = Quantity of Hg,  $\mu g$ , TOTAL in the ALIQUOT of Analytical Fraction 2B selected for

digestion and analysis . **NOTE:** For example, if a 10 ml aliquot of Analytical Fraction 2B is taken and digested and analyzed (according to Section 11.1.3 and its **NOTES** Nos. 1 and 2), then calculate and use the total amount of Hg in the 10 ml aliquot for  $Q_{bh2}$ .

$Q_{bh3(A,B,C)}$  = Quantity of Hg,  $\mu\text{g}$ , TOTAL, separately, in the ALIQUOT of Analytical Fraction 3A, 3B, or 3C selected for digestion and analysis (see **NOTES** in Sections 12.7.1 and 12.7.2 describing the quantity "Q" and calculate similarly).

$Q_{fh}$  = Quantity of Hg,  $\mu\text{g}$ , TOTAL in the ALIQUOT of Analytical Fraction 1B selected for digestion and analysis . **NOTE:** For example, if a 10 ml aliquot of Analytical Fraction 1B is taken and digested and analyzed (according to Section 11.1.3 and its **NOTES** Nos. 1 and 2), then calculate and use the total amount of Hg in the 10 ml aliquot for  $Q_{fh}$ .

$V_a$  = Total volume of digested sample solution (Analytical Fraction 2A), ml (see

Section 8.3.4.1 or 8.3.4.2, as applicable).

$V_{f1B}$  = Volume of aliquot of Analytical Fraction 1B analyzed, ml. **NOTE:** For example, if a 1 ml aliquot of Analytical Fraction 1B was diluted to 50 ml with 0.15 percent  $\text{HNO}_3$  as described in Section 11.1.3 to bring it into the proper analytical range, and then 1 ml of that 50-ml was digested according to Section 11.1.3 and analyzed,  $V_{f1B}$  would be 0.02 ml.

$V_{f2B}$  = Volume of Analytical Fraction 2B analyzed, ml. **NOTE:** For example, if 1 ml of Analytical Fraction 2B was diluted to 10 ml with 0.15 percent  $\text{HNO}_3$  as described in Section 11.1.3 to bring it into the proper analytical range, and then 5 ml of that 10 ml was analyzed,  $V_{f2B}$  would be 0.5 ml.

$V_{f3(A,B,C)}$  = Volume, separately, of Analytical Fraction 3A, 3B, or 3C analyzed, ml (see previous notes in Sections 12.7.1 and 12.7.2, describing the quantity "V" and calculate similarly).

$V_{m(std)}$  = Volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

$V_{soln,1}$  = Total volume of digested sample solution (Analytical Fraction 1), ml.

$V_{soln,1}$  = Total volume of Analytical Fraction 1, ml.

$V_{soln,2}$  = Total volume of Sample Fraction 2, ml.

$V_{soln,3(A,B,C)}$  = Total volume, separately, of Analytical Fraction 3A, 3B, or 3C, ml.

$K_4$  =  $10^{-3}$  mg/ $\mu$ g.

12.2 Dry Gas Volume. Using the data from this test, calculate  $V_{m(std)}$ , the dry gas sample volume at standard conditions as outlined in Section 12.3 of Method 5.

12.3 Volume of Water Vapor and Moisture Content. Using the total volume of condensate collected during the source sampling, calculate the volume of water vapor  $V_{w(std)}$  and the moisture content  $B_{ws}$  of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

12.4 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

12.5 In-Stack Detection Limits. Calculate the in-stack method detection limits shown in Table 29-4 using the conditions described in Section 13.3.1 as follows:

$$A \times \frac{B}{C} = D \quad \text{Eq. 29-1}$$

## 12.6 Metals (Except Hg) in Source Sample.

12.6.1 Analytical Fraction 1A, Front-Half, Metals (except Hg). Calculate separately the amount of each metal collected in Sample Fraction 1 of the sampling train using the following equation:

$$M_{fh} = C_{a1} F_d V_{soln,1} \quad \text{Eq. 29-2}$$

**NOTE:** If Analytical Fractions 1A and 2A are combined, use proportional aliquots. Then make appropriate changes in Equations 29-2 through 29-4 to reflect this approach.

12.6.2 Analytical Fraction 2A, Back-Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 2 of the sampling train using the following equation:

$$M_{bh} = C_{a2} F_a V_a \quad \text{Eq. 29-3}$$

12.6.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{fh} - M_{fhb}) + (M_{bh} - M_{bhb}) \quad \text{Eq. 29-4}$$

**NOTE:** If the measured blank value for the front half ( $M_{f_{hb}}$ ) is in the range 0.0 to "A"  $\mu\text{g}$  [where "A"  $\mu\text{g}$  equals the value determined by multiplying 1.4  $\mu\text{g}/\text{in.}^2$  times the actual area in  $\text{in.}^2$  of the sample filter], use  $M_{f_{hb}}$  to correct the emission sample value ( $M_{f_{h}}$ ); if  $M_{f_{hb}}$  exceeds "A"  $\mu\text{g}$ , use the greater of I or II:

I. "A"  $\mu\text{g}$ .

II. the lesser of (a)  $M_{f_{hb}}$ , or (b) 5 percent of  $M_{f_{h}}$ .

If the measured blank value for the back-half ( $M_{b_{hb}}$ ) is in the range 0.0 to 1  $\mu\text{g}$ , use  $M_{b_{hb}}$  to correct the emission sample value ( $M_{b_{h}}$ ); if  $M_{b_{hb}}$  exceeds 1  $\mu\text{g}$ , use the greater of I or II:

I. 1  $\mu\text{g}$ .

II. the lesser of (a)  $M_{b_{hb}}$ , or (b) 5 percent of  $M_{b_{h}}$ .

12.7 Hg in Source Sample.

12.7.1 Analytical Fraction 1B; Front-Half Hg.

Calculate the amount of Hg collected in the front-half, Sample Fraction 1, of the sampling train by using Equation 29-5:

$$\text{Hg}_{f_{h}} = \frac{Q_{f_{h}}}{V_{f1B}} (V_{\text{soln},1}) \quad \text{Eq. 29-5}$$

12.7.2 Analytical Fractions 2B, 3A, 3B, and 3C; Back Half Hg.

12.7.2.1 Calculate the amount of Hg collected in Sample Fraction 2 by using Equation 29-6:

$$\text{Hg}_{\text{bh2}} = \frac{Q_{\text{bh2}}}{V_{\text{f2B}}} (V_{\text{soln},2}) \quad \text{Eq. 29-6}$$

12.7.2.2 Calculate each of the back-half Hg values for Analytical Fractions 3A, 3B, and 3C by using Equation 29-7:

$$\text{Hg}_{\text{bh3(A,B,C)}} = \frac{Q_{\text{bh3(A,B,C)}}}{V_{\text{f3(A,B,C)}}} (V_{\text{soln},3(A,B,C)}) \quad \text{Eq. 29-7}$$

12.7.2.3 Calculate the total amount of Hg collected in the back-half of the sampling train by using Equation 29-8:

$$\text{Hg}_{\text{bh}} = \text{Hg}_{\text{bh2}} + \text{Hg}_{\text{bh3A}} + \text{Hg}_{\text{bh3B}} + \text{Hg}_{\text{bh3C}} \quad \text{Eq. 29-8}$$

12.7.3 Total Train Hg Catch. Calculate the total amount of Hg collected in the sampling train by using Equation 29-9:

$$\text{Hg}_{\text{t}} = (\text{Hg}_{\text{fh}} - \text{Hg}_{\text{fhh}}) + (\text{Hg}_{\text{bh}} - \text{Hg}_{\text{bhb}}) \quad \text{Eq. 29-9}$$

**NOTE:** If the total of the measured blank values ( $\text{Hg}_{\text{fhh}} + \text{Hg}_{\text{bhb}}$ ) is in the range of 0.0 to 0.6  $\mu\text{g}$ , then use the total to correct the sample value ( $\text{Hg}_{\text{fh}} + \text{Hg}_{\text{bh}}$ ); if it exceeds 0.6  $\mu\text{g}$ , use the greater of I. or II:

I. 0.6  $\mu\text{g}$ .



II. the lesser of (a)  $(Hg_{fhh} + Hg_{bhh})$ , or (b) 5 percent of the sample value  $(Hg_{fh} + Hg_{bh})$ .

#### 12.8 Individual Metal Concentrations in Stack Gas.

Calculate the concentration of each metal in the stack gas (dry basis, adjusted to standard conditions) by using Equation 29-10:

$$C_s = \frac{K_4 M_t}{V_{m(std)}} \quad \text{Eq. 29-10}$$

#### 12.9 Isokinetic Variation and Acceptable Results.

Same as Method 5, Sections 12.11 and 12.12, respectively.

#### 13.0 Method Performance.

13.1 Range. For the analysis described and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per ml (ng/ml) to micrograms per ml ( $\mu\text{g/ml}$ ) range in the final analytical solution can be analyzed using this method. Samples containing greater than approximately 50  $\mu\text{g/ml}$  As, Cr, or Pb should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20  $\mu\text{g/ml}$  of Cd should be diluted to that level before analysis.

#### 13.2 Analytical Detection Limits.

**NOTE:** See Section 13.3 for the description of in-stack detection limits.

13.2.1 ICAP analytical detection limits for the sample solutions (based on SW-846, Method 6010) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Co (7 ng/ml), Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15 ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Tl (40 ng/ml), and Zn (2 ng/ml). ICP-MS analytical detection limits (based on SW-846, Method 6020) are lower generally by a factor of ten or more. Be is lower by a factor of three. The actual sample analytical detection limits are sample dependent and may vary due to the sample matrix.

13.2.2 The analytical detection limits for analysis by direct aspiration AAS (based on SW-846, Method 7000 series) are approximately as follow: Sb (200 ng/ml), As (2 ng/ml), Ba (100 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Co (50 ng/ml), Cu (20 ng/ml), Pb (100 ng/ml), Mn (10 ng/ml), Ni (40 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml), and Zn (5 ng/ml).

13.2.3 The detection limit for Hg by CVAAS (on the resultant volume of the digestion of the aliquots taken for Hg analyses) can be approximately 0.02 to 0.2 ng/ml, depending upon the type of CVAAS analytical instrument used.

13.2.4 The use of GFAAS can enhance the detection limits compared to direct aspiration AAS as follows: Sb (3 ng/ml), As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Co (1 ng/ml), Pb (1 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml).

### 13.3 In-stack Detection Limits.

13.3.1 For test planning purposes in-stack detection limits can be developed by using the following information: (1) the procedures described in this method, (2) the analytical detection limits described in Section 13.2 and in SW-846, (3) the normal volumes of 300 ml (Analytical Fraction 1) for the front-half and 150 ml (Analytical Fraction 2A) for the back-half samples, and (4) a stack gas sample volume of 1.25 m<sup>3</sup>. The resultant in-stack method detection limits for the above set of conditions are presented in Table 29-1 and were calculated by using Eq. 29-1 shown in Section 12.5.

13.3.2 To ensure optimum precision/resolution in the analyses, the target concentrations of metals in the analytical solutions should be at least ten times their respective analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, these concentrations can be as low as approximately three times the respective analytical detection limits without seriously impairing the precision of the analyses. On at least one sample run in the source

test, and for each metal analyzed, perform either repetitive analyses, Method of Standard Additions, serial dilution, or matrix spike addition, etc., to document the quality of the data.

13.3.3 Actual in-stack method detection limits are based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be improved over those shown in Table 29-1 for a specific test by either increasing the sampled stack gas volume, reducing the total volume of the digested samples, improving the analytical detection limits, or any combination of the three. For extremely low levels of Hg only, the aliquot size selected for digestion and analysis can be increased to as much as 10 ml, thus improving the in-stack detection limit by a factor of ten compared to a 1 ml aliquot size.

13.3.3.1 A nominal one hour sampling run will collect a stack gas sampling volume of about 1.25 m<sup>3</sup>. If the sampling time is increased to four hours and 5 m<sup>3</sup> are collected, the in-stack method detection limits would be improved by a factor of four compared to the values shown in Table 29-1.

13.3.3.2 The in-stack detection limits assume that all of the sample is digested and the final liquid volumes for analysis are the normal values of 300 ml for Analytical

Fraction 1, and 150 ml for Analytical Fraction 2A. If the volume of Analytical Fraction 1 is reduced from 300 to 30 ml, the in-stack detection limits for that fraction of the sample would be improved by a factor of ten. If the volume of Analytical Fraction 2A is reduced from 150 to 25 ml, the in-stack detection limits for that fraction of the sample would be improved by a factor of six. Matrix effect checks are necessary on sample analyses and typically are of much greater significance for samples that have been concentrated to less than the normal original sample volume. Reduction of Analytical Fractions 1 and 2A to volumes of less than 30 and 25 ml, respectively, could interfere with the redissolving of the residue and could increase interference by other compounds to an intolerable level.

13.3.3.3 When both of the modifications described in Sections 13.3.3.1 and 13.3.3.2 are used simultaneously on one sample, the resultant improvements are multiplicative. For example, an increase in stack gas volume by a factor of four and a reduction in the total liquid sample digested volume of both Analytical Fractions 1 and 2A by a factor of six would result in an improvement by a factor of twenty-four of the in-stack method detection limit.

13.4 Precision. The precision (relative standard deviation) for each metal detected in a method development

test performed at a sewage sludge incinerator were found to be as follows:

Sb (12.7 percent), As (13.5 percent), Ba (20.6 percent), Cd (11.5 percent), Cr (11.2 percent), Cu (11.5 percent), Pb (11.6 percent), P (14.6 percent), Se (15.3 percent), Tl (12.3 percent), and Zn (11.8 percent). The precision for Ni was 7.7 percent for another test conducted at a source simulator. Be, Mn, and Ag were not detected in the tests. However, based on the analytical detection limits of the ICAP for these metals, their precisions could be similar to those for the other metals when detected at similar levels.

*14.0 Pollution Prevention.* [Reserved]

*15.0 Waste Management.* [Reserved]

*16.0 References.*

1. Method 303F in Standard Methods for the Examination of Water Wastewater, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street N.W., Washington, D.C. 20036.

2. EPA Methods 6010, 6020, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition, November 1986, with updates I, II, IIA, IIB and III. Office of Solid Waste and Emergency Response, U. S. Environmental Protection Agency, Washington, D.C. 20460.

3. EPA Method 200.7, Code of Federal Regulations, Title 40, Part 136, Appendix C. July 1, 1987.

4. EPA Methods 1 through 5, Code of Federal Regulations, Title 40, Part 60, Appendix A, July 1, 1991.

5. EPA Method 101A, Code of Federal Regulations, Title 40, Part 61, Appendix B, July 1, 1991.

*17.0 Tables, Diagrams, Flowcharts, and Validation Data.*

**TABLE 29-1. IN-STACK METHOD DETECTION LIMITS ( $\mu\text{g}/\text{m}^3$ )  
FOR THE FRONT-HALF, THE BACK-HALF, AND THE  
TOTAL SAMPLING TRAIN USING ICAP, GFAAS, AND CVAAS.**

Metal	Front-half: Probe and Filter	Back-half: Impingers 1-3	Back-half: Impingers 4-6 <sup>a</sup>	Total Train:
Antimony	<sup>1</sup> 7.7 (0.7)	<sup>1</sup> 3.8 (0.4)		<sup>1</sup> 11.5 (1.1)
Arsenic	<sup>1</sup> 12.7 (0.3)	<sup>1</sup> 6.4 (0.1)		<sup>1</sup> 19.1 (0.4)
Barium	0.5	0.3		0.8
Beryllium	<sup>1</sup> 0.07 (0.05)	<sup>1</sup> 0.04 (0.03)		<sup>1</sup> 0.11 (0.08)
Cadmium	<sup>1</sup> 1.0 (0.02)	<sup>1</sup> 0.5 (0.01)		<sup>1</sup> 1.5 (0.03)
Chromium	<sup>1</sup> 1.7 (0.2)	<sup>1</sup> 0.8 (0.1)		<sup>1</sup> 2.5 (0.3)
Cobalt	<sup>1</sup> 1.7 (0.2)	<sup>1</sup> 0.8 (0.1)		<sup>1</sup> 2.5 (0.3)
Copper	1.4	0.7		2.1
Lead	<sup>1</sup> 10.1 (0.2)	<sup>1</sup> 5.0 (0.1)		<sup>1</sup> 15.1 (0.3)
Manganese	<sup>1</sup> 0.5 (0.2)	<sup>1</sup> 0.2 (0.1)		<sup>1</sup> 0.7 (0.3)
Mercury	<sup>2</sup> 0.06	<sup>2</sup> 0.3	<sup>2</sup> 0.2	<sup>2</sup> 0.56
Nickel	3.6	1.8		5.4
Phosphorus	18	9		27
Selenium	<sup>1</sup> 18 (0.5)	<sup>1</sup> 9 (0.3)		<sup>1</sup> 27 (0.8)
Silver	1.7	0.9 (0.7)		2.6
Thallium	<sup>1</sup> 9.6 (0.2)	<sup>1</sup> 4.8 (0.1)		<sup>1</sup> 14.4 (0.3)
Zinc	0.5	0.3		0.8

<sup>a</sup>Mercury analysis only.

<sup>1</sup>Detection limit when analyzed by ICAP or GFAAS as shown in parentheses (see Section 11.1.2).

<sup>2</sup>Detection limit when analyzed by CVAAS, estimated for Back-half and Total Train. See Sections 13.2 and 11.1.3.

Note: Actual method in-stack detection limits may vary from these values, as described in Section 13.3.3.



**TABLE 29-2. RECOMMENDED WAVELENGTHS FOR ICAP ANALYSIS**

Analyte	Wavelength (nm)
Aluminum (Al)	308.215
Antimony (Sb)	206.833
Arsenic (As)	193.696
Barium (Ba)	455.403
Beryllium (Be)	313.042
Cadmium (Cd)	226.502
Chromium (Cr)	267.716
Cobalt (Co)	228.616
Copper (Cu)	328.754
Iron (Fe)	259.940
Lead (Pb)	220.353
Manganese (Mn)	257.610
Nickel (Ni)	231.604
Phosphorus (P)	214.914
Selenium (Se)	196.026
Silver (Ag)	328.068
Thallium (Tl)	190.864
Zinc (Zn)	213.856

TABLE 29-3. APPLICABLE TECHNIQUES, METHODS AND MINIMIZATION OF INTERFERENCES FOR AAS ANALYSIS.

Metal	Technique	SW-846 <sup>1</sup> Method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Fe	Aspiration	7380	248.3	Contamination	Great care taken to avoid contamination
Pb	Aspiration	7420	283.3	217.0 nm alternate	Background correction required
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 µl of phosphorus acid to 1 ml of prepared sample in sampler cup
Mn	Aspiration	7460	279.5	403.1 nm alternate	Background correction required
Ni	Aspiration	7520	232.0	352.4 nm alternate Fe, Co, and Cr	Background correction required Matrix matching or nitrous-oxide/acetylene flame
				Nonlinear response	Sample dilution or use 352.3 nm line
Se	Furnace	7740	196.0	Volatility	Spike samples and reference materials and add nickel nitrate to minimize volatilization
				Adsorption & scatter	Background correction is required and Zeeman background correction can be useful
Ag	Aspiration	7760	328.1	Adsorption & scatter AgCl insoluble	Background correction is required Avoid hydrochloric acid unless silver is in solution as a chloride complex Sample and standards monitored for aspiration rate
Tl	Aspiration	7840	276.8		Background correction is required Hydrochloric acid should not be used
Tl	Furnace	7841	276.8	Hydrochloric acid or chloride	Background correction is required Verify that losses are not occurring for volatilization by spiked samples or standard addition; Palladium is a suitable matrix modifier
Zn	Aspiration	7950	213.9	High Si, Cu, & P Contamination	Strontium removes Cu and phosphate Great care taken to avoid contamination

TABLE 29-3. Continued.

Metal	Technique	SW-846 <sup>1</sup> Method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Sb	Aspiration	7040	217.6	1000 mg/ml Pb, Ni, Cu, or acid	Use secondary wavelength of 231.1 nm; match sample & standards acid concentration or use nitrous oxide/acetylene flame
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction
As	Furnace	7060	193.7	Arsenic Volatilization Aluminum	Spike samples and add nickel nitrate solution to digestates prior to analysis Use Zeeman background correction
Ba	Aspiration	7080	553.6	Calcium  Barium Ionization	High hollow cathode current and narrow band set 2 ml of KCl per 100 ml of sample
Be	Aspiration	7090	234.9	500 ppm Al High Mg and Si	Add 0.1% fluoride
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects
Cd	Aspiration	7130	228.8	Absorption and light scattering	Background correction is required
Cd	Furnace	7131	228.8	As above Excess Chloride Pipet Tips	As above Ammonium phosphate used as a matrix modifier Use cadmium-free tips
Cr	Aspiration	7190	357.9	Alkali metal	KCl ionization suppressant in samples and standards-- Consult mfgs' literature
Co	Furnace	7201	240.7	Excess chloride	Use Method of Standard Additions
Cr	Furnace	7191	357.9	200 mg/L Ca and P	All calcium nitrate for a known constant effect and to eliminate effect of phosphate
Cu	Aspiration	7210	324.7	Absorption and Scatter	Consult manufacturer's manual

<sup>1</sup>Refer to EPA publication SW-846 (Reference 2 in Section 16.0).

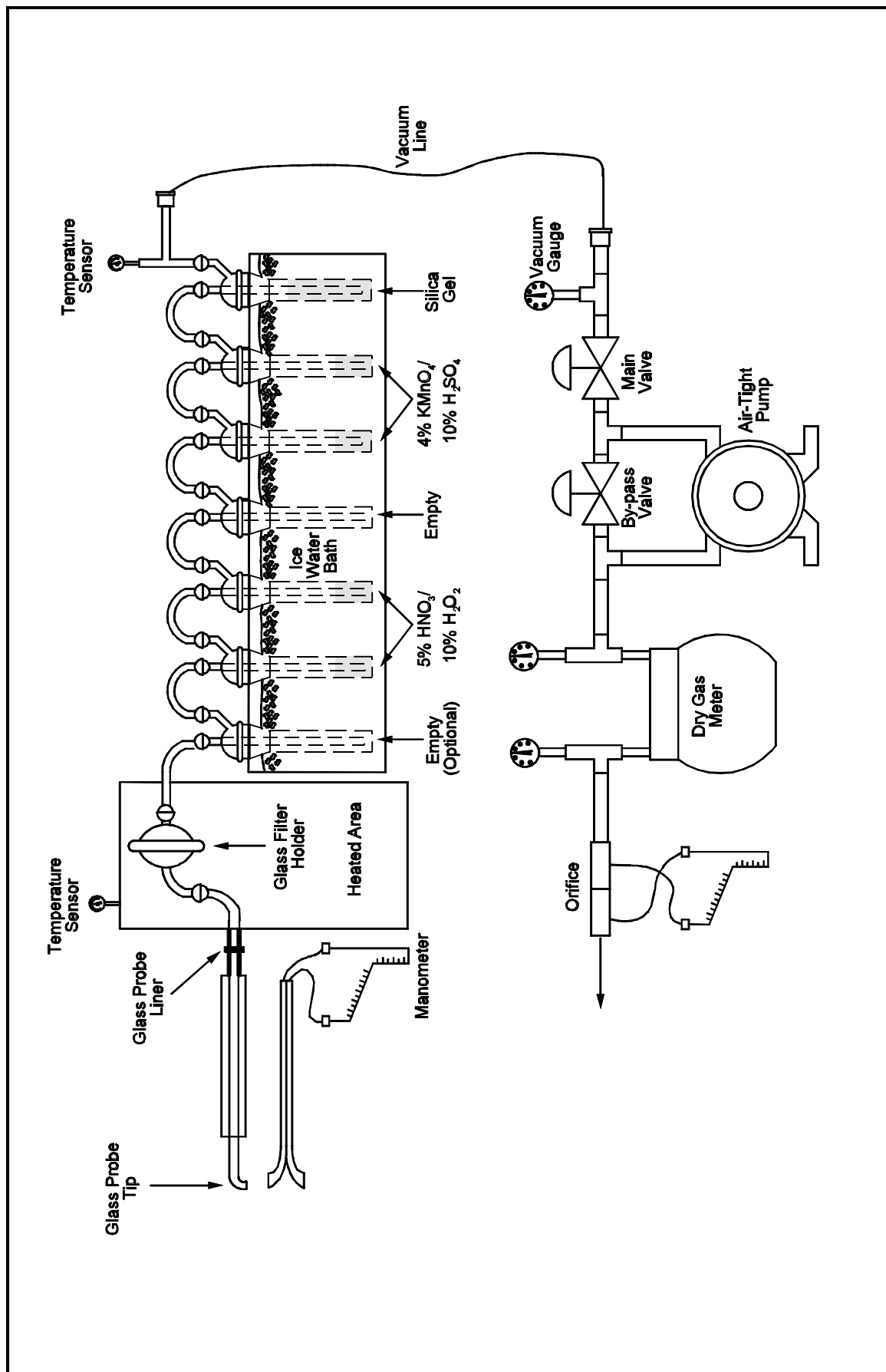


Figure 29-1. Sampling Train.

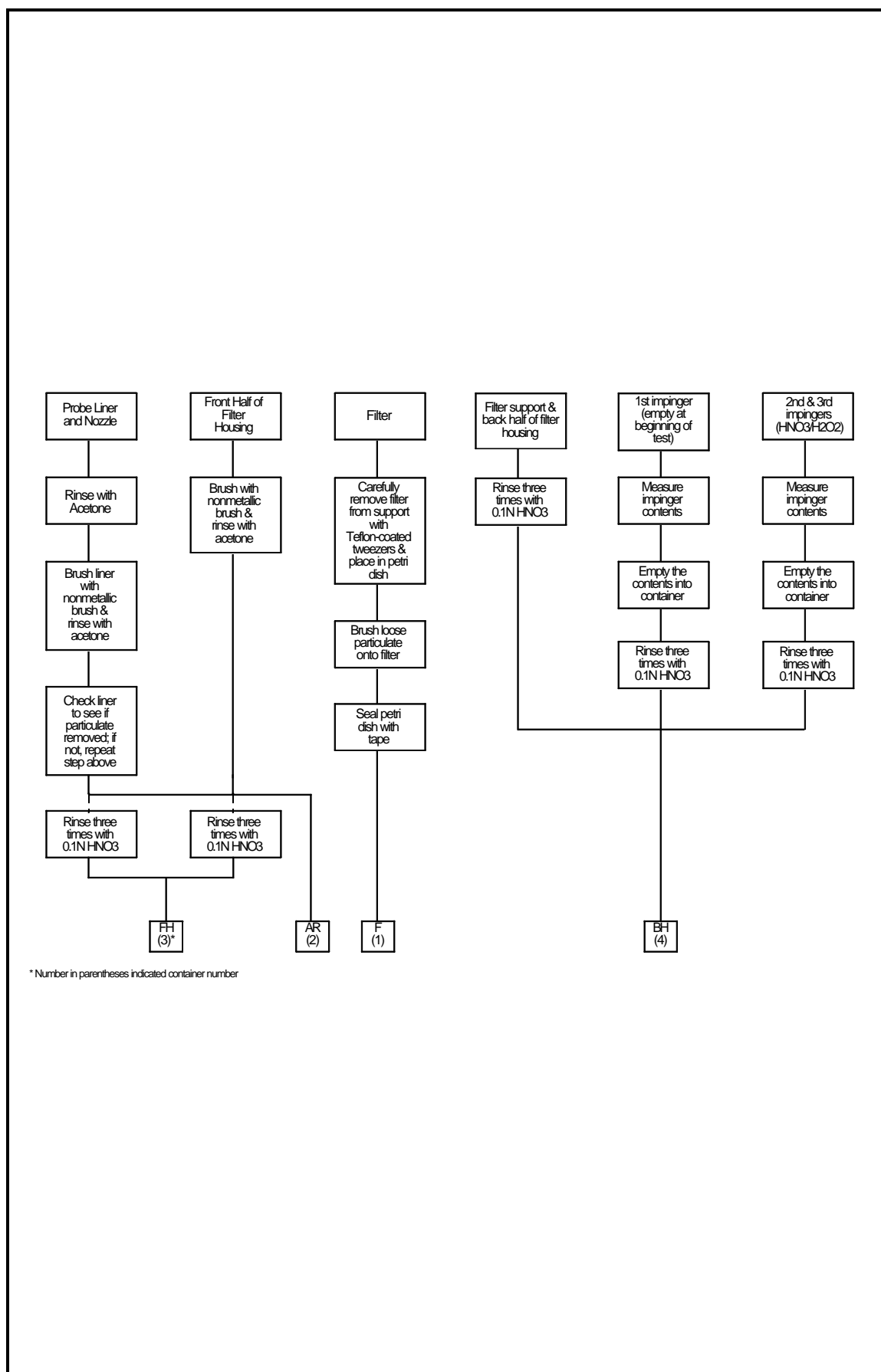


Figure 29-2a. Sample Recovery Scheme.

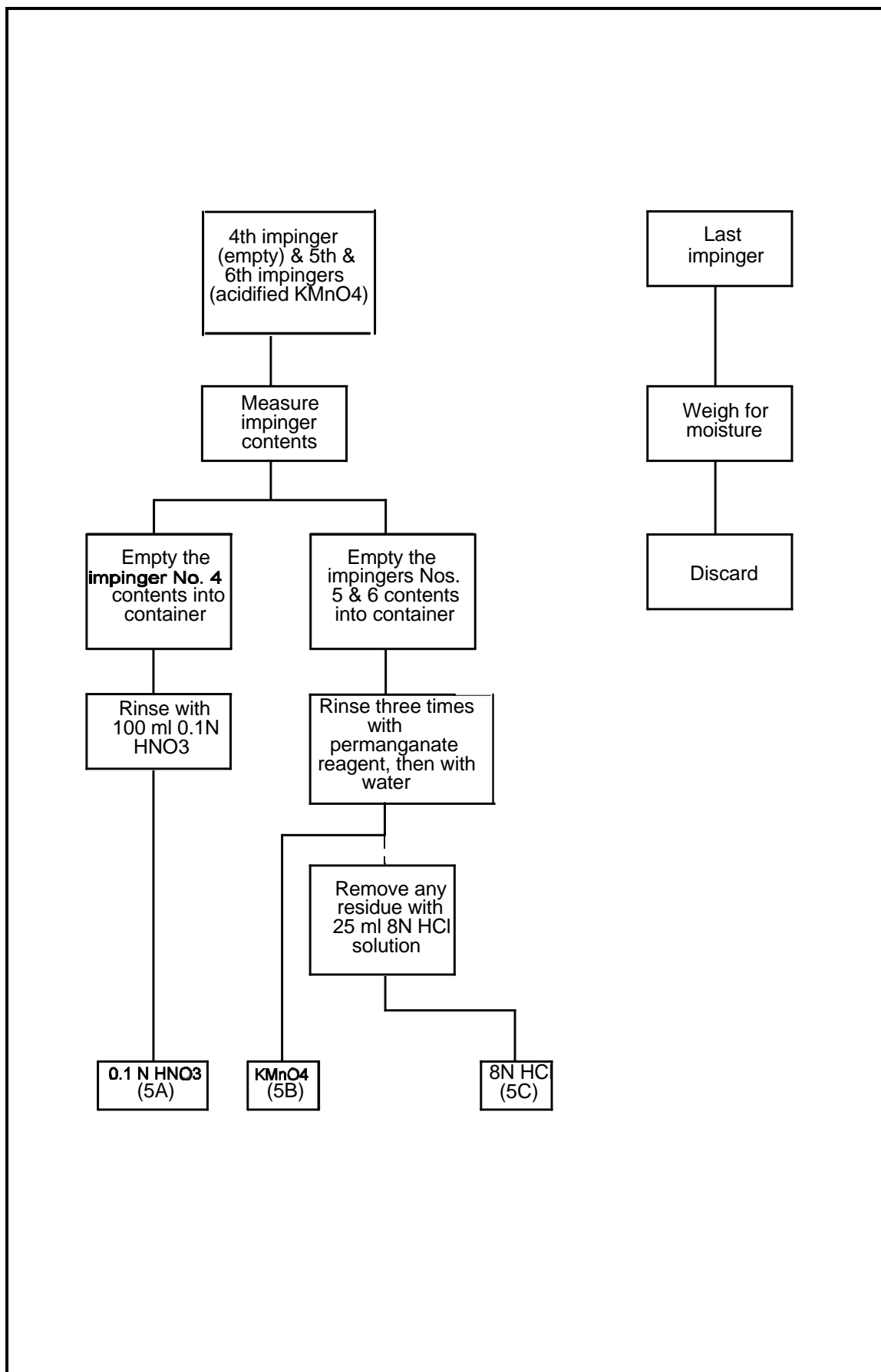


Figure 29-2b. Sample Recovery Scheme.

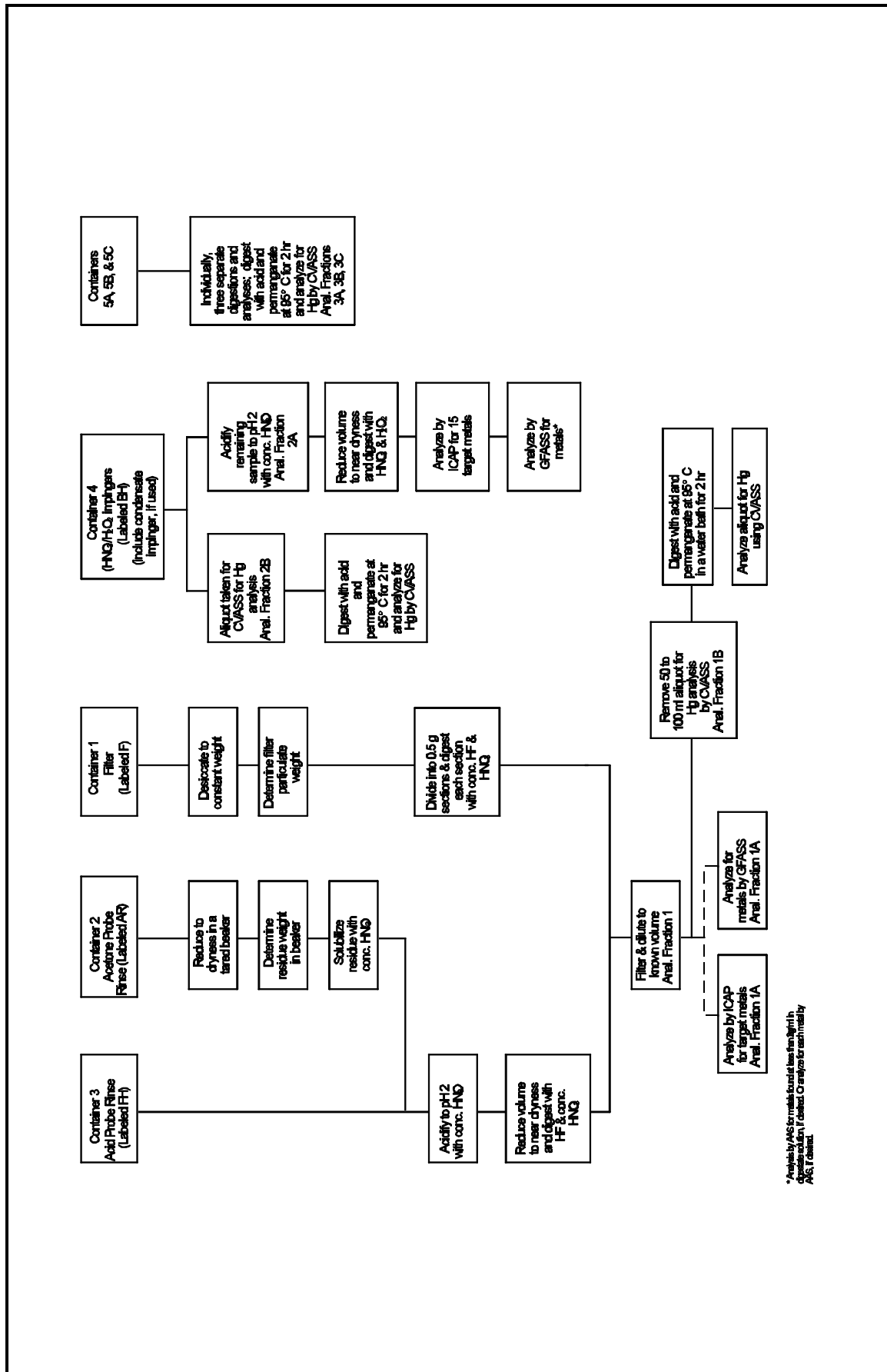


Figure 29-3. Sample Preparation and Analysis Scheme.

**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix F**

### **Mercury Laboratory Reports**



A. Lanfranco And Associates Inc.

# **STANDARD VOLUME / GAS CONCENTRATION WORKSHEET**

<b>Plant:</b>	Newmont Doris Camp		<b>Tested for:</b>	Hg
<b>Location:</b>	Incin. Stack		<b>DGM ID:</b>	BEI - 2
<b>Barometric:</b>	29.7		<b>DGM Y:</b>	0.9851
<b>Date:</b>	Sept. 30 - Oct. 1 2009			

DRY GAS METER					CONSTANTS			RESULTS			11.0		
RUN	TIME	Reading (ft <sup>3</sup> )	Temp In (Avg. oF)	Temp Out (Avg. oF)	Avg. Delta H (inches H <sub>2</sub> O)	Y Factor	Pb (in. Hg)	Volume Std. (m <sup>3</sup> std.)	Lab Result (ug of Hg)	Concentration (ug Hg/m3)	Oxygen (%)	Mercury Conc. @ 11% O2 (ug/Sm <sup>3</sup> )	
	Sept. 30												
1	Sept. 30	9:00	689.6000	80.0	61.0	2.5	0.9851	29.70	1.53280	0.38	0.25	8.9	<b>0.20</b>
		10:06	743.9500	-	-	-	-	-					
	Sept. 30												
2	Sept. 30	15:03	890.0000	82.0	61.0	2.4	0.9851	29.75	1.44837	0.57	0.39	12.3	<b>0.45</b>
		16:11	941.3800	-	-	-	-	-					
	Oct. 1												
3	Oct. 1	14:08	87.2000	80.0	59.0	2.3	0.9851	30.05	1.47949	3.20	2.16	11.6	<b>2.31</b>
		15:13	138.9800	-	-	-	-	-					

## Report Transmission Cover Page

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Attn: Al LanFranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Approval Status: Approved  
Invoice Frequency: by Lot  
COD Status:  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

Contact & Affiliation	Address	Delivery Commitments	
Al LanFranco A. Lanfranco & Associates Inc.	#101, 9488 - 189 Street Surrey, British Columbia V4N 4W7 Phone: (604) 881-2582 Fax: (604) 881-2581 Email: <a href="mailto:lanfranco@telus.net">lanfranco@telus.net</a>	On [Lot Verification] send (COA) by Email - Multiple Reports On [Report Approval] send (COC, Test Report) by Automated Fax On [Lot Approval and Final Test Report Approval] send (Invoice) by Post On [Lot Approval and Final Test Report Approval] send (COC, Test Report) by Email - Merge Reports	M

### Notes To Clients:

## Analytical Report

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al Lanfranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

		Reference Number	707266-1	707266-2	707266-3
		Sample Date			
		Sample Time			
		Sample Location			
		Sample Description	BLK - Beaker & Filter (M-53)	Run 1 - Beaker & Filer (M-2)	Run 2 - Beaker & Filer (M-3)
		Matrix	Stack Samples	Stack Samples	Stack Samples
Analyte	Units	Results	Results	Results	Nominal Detection Limit
<b>Air Quality Metals</b>					
Mercury	Strong Acid Extractable ug	<0.002	<0.002	<0.002	0.005

## Analytical Report

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al LanFranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

		Reference Number	707266-4	707266-5	707266-6
		Sample Date			
		Sample Time			
		Sample Location			
		Sample Description	Run 3 - Beaker & Filer (M-6)	BLK Bottle	Run 1-2 Bottles
		Matrix	Stack Samples	Stack Samples	Stack Samples
Analyte	Units	Results	Results	Results	Nominal Detection Limit
<b>Air Quality Metals</b>					
Mercury	Strong Acid Extractable	ug	<0.002	<0.002	<0.002 0.005

## Analytical Report

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al LanFranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

		Reference Number	707266-7	707266-8	707266-9	
		Sample Date				
		Sample Time				
		Sample Location				
		Sample Description	Run 2-2 Bottles	Run 3-2 Bottles	BLK - Hg	
		Matrix	Stack Samples	Stack Samples	Stack Samples	
Analyte		Units	Results	Results	Results	Nominal Detection Limit
<b>Air Quality Metals</b>						
Mercury	Strong Acid Extractable	ug	<0.002	<0.002		0.005
Volume	Sample	mL			890	
Volume	aliquot volume	mL			25	
Volume	Final	mL			40	
Mercury	As Tested	ug/L			<0.05	0.05
Dilution Factor	As Tested				1.00	
Mercury	Total	ug/sample			<0.07	

## Analytical Report

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al LanFranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

		Reference Number	707266-10	707266-11	707266-12	Nominal Detection Limit
		Sample Date				
		Sample Time				
		Sample Location				
		Sample Description	Run 1 - Hg	Run 2 - Hg	Run 3 - Hg	
		Matrix	Stack Samples	Stack Samples	Stack Samples	
Analyte		Units	Results	Results	Results	
<b>Air Quality Metals</b>						
Volume	Sample	mL	910	885	878	
Volume	aliquot volume	mL	25	25	25	
Volume	Final	mL	40	40	40	
Mercury	As Tested	ug/L	0.26	0.40	2.31	0.05
Dilution Factor	As Tested		1.00	1.00	1.00	
Mercury	Total	ug/sample	0.38	0.57	3.2	

Approved by:   
Andrew Garrard, BSc  
General Manager

## Analytical Report

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al LanFranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

Approved by:



Andrew Garrard, BSc  
General Manager

## Methodology and Notes

Bill To:	A. Lanfranco & Associates	Project:		Lot ID:	<b>707266</b>
Report To:	A. Lanfranco & Associates	ID:	Neumont	Control Number:	A089905
	#101, 9488 - 189 Street	Name:	Doris Bay Camp	Date Received:	Oct 8, 2009
	Surrey, BC, Canada	Location:	Sept 29 - Oct 1/09	Date Reported:	Oct 20, 2009
	V4N 4W7	LSD:		Report Number:	1259403
Attn:	Al LanFranco	P.O.:			
Sampled By:		Acct code:			
Company:					

## Method of Analysis

Method Name	Reference	Method	Date Analysis Started	Location
Mercury in Air (Surrey)	APHA	* Cold Vapour Atomic Absorption Spectrometric Method, 3112 B	19-Oct-09	Exova Surrey
Metals (Strong Acid Leachable) in air	APHA	* Cold Vapour Atomic Absorption Spectrometric Method, 3112 B	19-Oct-09	Exova Surrey

*\* Laboratory method(s) based on reference method*

## References

APHA      Standard Methods for the Examination of Water and Wastewater

## Comments:

Please direct any inquiries regarding this report to our Client Services group.

Results relate only to samples as submitted.

The test report shall not be reproduced except in full, without the written approval of the laboratory.



## Quality Control

Bill To: A. Lanfranco & Associates  
Report To: A. Lanfranco & Associates  
#101, 9488 - 189 Street  
Surrey, BC, Canada  
V4N 4W7  
Attn: Al Lanfranco  
Sampled By:  
Company:

Project:  
ID: Neumont  
Name: Doris Bay Camp  
Location: Sept 29 - Oct 1/09  
LSD:  
P.O.:  
Acct code:

Lot ID: **707266**  
Control Number: A089905  
Date Received: Oct 8, 2009  
Date Reported: Oct 20, 2009  
Report Number: 1259403

## Air Quality Metals

Blanks		Units	Measured	Lower Limit	Upper Limit	Passed QC	
Mercury		ug/L	<0.05	-0.05	0.05	yes	
Date Acquired:		October 19, 2009					
Calibration Check		Units	% Recovery	Lower Limit	Upper Limit	Passed QC	
Mercury		ug/L	102.60	80	120	yes	
Date Acquired:		October 19, 2009					
Mercury		ug/L	60.59	80	120	yes	
Date Acquired:		October 19, 2009					
Mercury		ug/L	113.37	85	115	yes	
Date Acquired:		October 19, 2009					
Certified Reference Material		Units	Measured	Target	Lower Limit	Upper Limit	Passed QC
Mercury		ug/sample	0.035	0.018	0.015	0.021	yes
Date Acquired:		October 19, 2009					
Replicates		Units	Replicate 1	Replicate 2	% RSD Criteria	Absolute Criteria	Passed QC
Mercury		ug/L	2.31	2.35	30	0.05	yes
Date Acquired:		October 19, 2009					

NOTE: Proper completion of this form is required in order to proceed with analysis  
See reverse for your nearest Bodycote location and proper sampling protocol

<b>Billing Address:</b>		<b>Copy of Report To:</b>		<b>Copy of Invoice:</b> <input type="checkbox"/>	
Company: <i>C. Janfranco + ASSUE</i>		Company:		Mail Invoice to this address for approval <input type="checkbox"/>	
Address:		Address:			
Attention:		Attention: <i>Same</i>		Report Results:	
Phone: <i>604-881-2582</i>		Phone:		Fax <input type="checkbox"/>	
Fax:		Fax:		Mail <input type="checkbox"/>	
Cell:		Cell: <i>Lot # 707266</i>		Courier <input type="checkbox"/>	
e-mail:		e-mail:		e-mail <input type="checkbox"/>	
QA/QC Report <input type="checkbox"/>		Report Results:		e-Service <input type="checkbox"/>	
Fax <input type="checkbox"/>					
Mail <input type="checkbox"/>					
Courier <input type="checkbox"/>					
e-mail <input type="checkbox"/>					
e-Service <input type="checkbox"/>					

<b>Information to be included on Report and Invoice</b>	<b>Rush</b> Please contact the laboratory to confirm rush dates and times before submitting samples.	<b>Sample Custody (Please Print)</b>
	Upon filling out this section, client accepts that surcharges will be attached to this analysis	Sampled by:
Project ID: <i>Newmont</i>	RUSH <input type="checkbox"/> All analysis <input type="checkbox"/> As indicated <input type="checkbox"/>	Company: _____ Signature: _____
Project Name: <i>Doris Bay Camp</i>	Date Required: _____	I authorize Bodycote to proceed with the work indicated on this form:
Project Location: <i>Sept. 29 - Oct. 1/09</i>	Signature: _____	Date: _____ Initial: _____
Legal Location: _____	Bodycote Authorization: _____	Received by: <i>mn</i> Sample Temp. _____ °C
PO#: _____		Waybill # _____ Date: <i>Oct 8/09</i>
Proj. Acct. Code: _____		Company: <i>Hand</i> Time: <i>500</i>
Agreement ID: _____		

<b>Special Instructions / Comments</b>	<b>FOR LAB USE ONLY</b>
<i>Please Report ug/sample Hg Only !!</i> <i>Report high + low. spike Recovery</i>	Condition of containers / coolers upon arrival at lab
	<input type="checkbox"/> Check here if Bodycote is required to report results directly to a regulatory body (Please include contact information) <input type="checkbox"/> Check here if you're testing POTABLE WATER for <b>HUMAN CONSUMPTION</b> .

	Sample Identification	Location	Depth IN CM M	Date/Time Sampled	Matrix	Sampling Method	Number of Containers	Enter tests above (✓ relevant samples below)									
1	BLK Beaker + Filter (M-53)			Metals			1	✓									
2	BLK Bottle			Metals			1	✓									
3	Run 1 Beaker + Filter (M-2)			Metals			1	✓									
4	Run 2 Beaker + Filter (M-3)			Metals			1	✓									
5	Run 3 Beaker + Filter (M-6)			Metals			1	✓									
6	Run 1-2 Bottles			Metals			2	✓									
7	Run 2-2 Bottles			Metals			2	✓									
8	Run 3-2 Bottles			Metals			2	✓									
9																	
10	BLK - Hg	1 Bottle		Hg			1	✓									
11	Run -1 Hg			Hg			2	✓									
12	Run -2 Hg			Hg			2	✓									
13	Run -3 Hg			Hg			2	✓									
14																	
15																	

NOTE: All hazardous samples must be labeled according to WHMIS guidelines.

Page \_\_\_\_ of \_\_\_\_

**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix G**

### **Measured and Calculated Data**

Standard Conditions Set To:			77 oF	25 oC	536.67 oR	Other Constants and Conversions:	459.67 oR
			29.92 in.Hg	101.325 kPa			35.315 cf
			0.048 * ml H2O = Vcond	(use 0.04707 for 68 oF)		from mg/m to divide by	2288.3519 gr/cf
				(use 0.04800 for 75 oF)			
Correct To:	0 % CO2	For no correction enter 0 or -1					
	11 % O2						

**Client:**  
**Jobsite:**  
**Source:**

Newmont, Hope Bay  
Doris Camp  
Incinerator stack

**Date:**  
**Run:**  
**Run Time:**

Sept. 29/09  
1 PCDD/PCDF  
16:00 - 19:34

**Concentration:**

0.00 mg/dscm  
0.00 mg/Acm

0.0000 gr/dscf  
0.0000 gr/Acf

0.00 mg/dscm (@ 11% O2)

0.0000 gr/dscf (@ 11% O2)

**Emission Rate:**

0.00 Kg/hr

0.000 lb/hr

**Sample Gas Volume:**  
**Total Sample Time:**

3.6318 dscm  
192.0 minutes

128.258 dscf

**Average Isokineticity:**

97.5 %

Flue Gas Characteristics

**Moisture:**

5.94 %

**Temperature**

881.1 oC

1618.1 oF

**Flow**

20.9 dscm/min  
0.35 dscm/sec  
85.8 Acm/min

740 dscf/min  
12.3 dscf/sec  
3031 Acf/min

**Velocity**

8.714 m/sec

28.59 f/sec

**Gas Analysis**

11.50 % O2

8.46 % CO2

29.814 Mol. Wt (g/gmole) Dry

29.112 Mol. Wt (g/gmole) Wet

**\* Standard Conditions:**

Metric: 25 deg C, 101.325 kPa  
Imperial: 77 deg F, 29.92 in.Hg

**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator stack

**Date:** Sept. 29/09  
**Run:** 1 PCDD/PCDF  
**Run Time:** 16:00 - 19:34

Control Unit (Y) 0.9851  
 Nozzle Diameter (in.) 0.5480  
 Pitot Factor 0.8568  
 Baro. Press. (in. Hg) 30.05  
 Static Press. (in. Hg) 0.04  
 Stack Height (ft) 50  
 Stack Diameter (in.) 18.0  
 Stack Area (sq.ft.) 1.767  
 Minutes Per Reading 4.0  
 Minutes Per Point 16.0

**Collection:**  
 Filter (grams) 0.0000  
 Washings (grams) 0.0000  
 Impinger (grams) 0.0000  
**Total (grams) 0.0000**

**Gas Analysis (Vol. %):**  

	CO2	O2
Trav 1	8.75	11.25
Trav 2	8.17	11.75
<b>Average =</b>	<b>8.46</b>	<b>11.50</b>

**Condensate Collection:**  
 Impinger 1 (grams) 136.0  
 Impinger 2 (grams) 16.0  
 Impinger 3 (grams) 5.0  
 Impinger 4 (grams) 11.8  
**Total Gain (grams) 168.8**

Traverse	Point	Time (min.)	Dry Gas Meter (ft3)	Pitot ΔP (in. H2O)	Orifice ΔH (in. H2O)	Dry Gas Temperature Inlet (oF)	Dry Gas Temperature Outlet (oF)	Vacuum (in. Hg.)	XAD Exit (oF)	Stack (oF)	Wall Dist. (in.)	Isokin. (%)
1	1	0.0	557.400									
		4.0	560.290	0.080	1.60	66	50	2	36	1652	0.8	98.0
		8.0	563.190	0.080	1.60	74	56	2	35	1650	0.8	96.9
		12.0	566.120	0.080	1.60	80	60	2	35	1641	0.8	96.8
		16.0	568.890	0.070	1.40	83	61	2	37	1610	0.8	96.7
	2	20.0	571.620	0.070	1.40	85	62	2	37	1636	2.6	95.6
		24.0	574.410	0.070	1.40	86	63	2	38	1652	2.6	97.9
		28.0	577.220	0.070	1.40	89	64	2	38	1670	2.6	98.7
		32.0	580.200	0.080	1.65	90	64	2	38	1625	2.6	96.8
	3	36.0	583.220	0.080	1.65	92	66	3	38	1640	5.3	98.1
		40.0	586.250	0.080	1.65	93	68	3	39	1656	5.3	98.5
		44.0	589.090	0.070	1.45	95	70	3	39	1627	5.3	97.6
		48.0	591.890	0.070	1.45	97	71	3	39	1620	5.3	95.8
	4	52.0	594.680	0.070	1.45	97	73	4	39	1659	12.7	96.2
		56.0	597.300	0.060	1.25	98	74	4	39	1642	12.7	97.0
		60.0	599.920	0.060	1.25	98	75	5	38	1655	12.7	97.2
		64.0	602.550	0.060	1.25	99	77	5	38	1661	12.7	97.4
	5	68.0	604.950	0.050	1.15	100	78	5	38	1673	15.4	97.5
		72.0	607.350	0.050	1.15	100	79	5	38	1641	15.4	96.6
		76.0	609.740	0.050	1.15	100	80	4	38	1629	15.4	95.9
		80.0	612.580	0.070	1.56	101	81	4	40	1620	15.4	96.0
	6	84.0	615.410	0.070	1.56	101	81	5	40	1637	17.2	96.0
		88.0	618.050	0.060	1.35	102	81	5	40	1642	17.2	96.8
		92.0	620.860	0.070	1.56	102	81	5	40	1650	17.2	95.6
		96.0	623.700	0.070	1.56	102	81	5	40	1667	17.2	97.0
		0.0	623.700									
2	1	4.0	626.060	0.050	1.15	90	74	5	38	1656	0.8	96.7
		8.0	628.650	0.060	1.35	93	77	5	38	1647	0.8	96.2
		12.0	631.270	0.060	1.35	94	80	5	39	1629	0.8	96.5
		16.0	633.660	0.050	1.15	95	80	5	39	1627	0.8	96.3
	2	20.0	636.500	0.070	1.56	95	80	5	40	1641	2.6	97.1
		24.0	639.520	0.080	1.80	96	80	5	40	1647	2.6	96.7
		28.0	642.560	0.080	1.80	96	80	5	41	1634	2.6	97.0
		32.0	645.580	0.080	1.80	97	80	5	40	1627	2.6	96.1
	3	36.0	648.200	0.060	1.38	98	80	5	40	1654	5.3	96.7
		40.0	650.840	0.060	1.38	98	80	5	40	1632	5.3	97.0
		44.0	653.240	0.050	1.15	99	80	5	40	1629	5.3	96.4
		48.0	655.620	0.050	1.15	100	80	5	40	1664	5.3	96.3
	4	52.0	658.620	0.050	1.45	100	80	5	38	1220	12.7	108.0
		56.0	661.600	0.050	1.45	100	80	5	38	1160	12.7	105.3
		60.0	664.400	0.070	1.56	92	77	5	38	1627	12.7	95.9
		64.0	667.400	0.080	1.80	95	78	5	38	1660	12.7	96.6
	5	68.0	670.420	0.080	1.80	96	78	5	38	1671	15.4	97.4
		72.0	673.050	0.060	1.35	96	78	5	39	1664	15.4	97.7
		76.0	675.810	0.060	1.35	96	80	5	39	1575	15.4	100.2
		80.0	678.560	0.060	1.45	97	81	5	39	1600	15.4	100.3
	6	84.0	681.000	0.050	1.25	97	80	5	39	1580	17.2	97.0
		88.0	683.490	0.050	1.25	96	80	5	39	1590	17.2	99.3
		92.0	685.970	0.050	1.25	96	80	5	39	1587	17.2	98.9
		96.0	688.450	0.050	1.25	96	80	5	40	1591	17.2	99.0
			<b>Average:</b>	0.065	1.432	94.5	75.0	4.3	38.7	1618.1		97.5

**Calculation Section: Do Not Touch**

Pm: 30.155  
 Ps: 30.053  
 AN (ft2): 0.00163790  
 Vcond: 8.1024  
 Bwo: 0.0594

Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)
66.300			96.0
	32.187	31.529	
	32.171	31.186	
	32.103	31.078	
	29.807	28.825	
	29.994	28.684	
	30.108	29.483	
	30.236	29.836	
	31.980	30.963	
	32.095	31.487	
	32.217	31.743	
	29.929	29.222	
	29.879	28.635	
	30.158	29.014	
	27.808	26.965	
	27.894	27.107	
	27.934	27.213	
	25.572	24.922	
	25.379	24.526	
	25.307	24.262	
	29.879	28.683	
	30.001	28.815	
	27.808	26.906	
	30.094	28.763	
	30.215	29.304	
64.750			96.0
	25.470	24.626	
	27.841	26.775	
	27.722	26.756	
	25.295	24.350	
	30.029	29.158	
	32.149	31.084	
	32.049	31.097	
	31.996	30.761	
	27.888	26.979	
	27.742	26.902	
	25.307	24.386	
	25.518	24.565	
	22.694	22.509	
	22.285	23.476	
	29.929	28.713	
	32.248	31.154	
	32.331	31.495	
	27.954	27.308	
	27.362	27.407	
	27.529	27.599	
	25.008	24.261	
	25.069	24.902	
	25.051	24.766	
	25.076	24.814	
131.050	28.5895	27.8541	192.0

Standard Conditions Set To:		77 oF	25 oC	536.67 oR	her Constants and Conversions:	459.67 oR
		29.92 in.Hg	101.325 kPa			35.315 cf
Correct To:	0 % CO2 11 % O2	0.048 * ml H2O = Vcond		(use 0.04707 for 68 oF) (use 0.04800 for 75 oF)	from mg/m to divide by	2288.3519 gr/cf
For no correction enter 0 or -1						

<b>Client:</b>	Newmont, Hope Bay	<b>Date:</b>	Sept. 30/09
<b>Jobsite:</b>	Doris Camp	<b>Run:</b>	2 PCDD/PCDF
<b>Source:</b>	Incinerator stack	<b>Run Time:</b>	11:06 - 14:26

<b>Concentration:</b>	0.00 mg/dscm	0.0000 gr/dscf
	0.00 mg/Acm	0.0000 gr/Acf
	0.00 mg/dscm (@ 11% O2)	0.0000 gr/dscf (@ 11% O2)

<b>Emission Rate:</b>	0.00 Kg/hr	0.000 lb/hr
-----------------------	------------	-------------

<b>Sample Gas Volume:</b>	4.0640 dscm	143.521 dscf
<b>Total Sample Time:</b>	192.0 minutes	

<b>Average Isokineticity:</b>	100.9 %
-------------------------------	---------

**Flue Gas Characteristics**

<b>Moisture:</b>	7.43 %	
<b>Temperature</b>	855.8 oC	1572.4 oF
<b>Flow</b>	22.6 dscm/min	797 dscf/min
	0.38 dscm/sec	13.3 dscf/sec
	92.8 Acm/min	3277 Acf/min
<b>Velocity</b>	9.420 m/sec	30.91 f/sec
<b>Gas Analysis</b>	11.82 % O2	7.76 % CO2
	29.713 Mol. Wt (g/gmole) Dry	28.843 Mol. Wt (g/gmole) Wet

\* **Standard Conditions:** Metric: 25 deg C, 101.325 kPa  
Imperial: 77 deg F, 29.92 in.Hg

**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator stack

**Date:** Sept. 30/09  
**Run:** 2 PCDD/PCDF  
**Run Time:** 11:06 - 14:26

**Control Unit (Y)** 0.9851  
**Nozzle Diameter (in.)** 0.5480  
**Pitot Factor** 0.8568  
**Baro. Press. (in. Hg)** 29.75  
**Static Press. (in. Hg)** 0.04  
**Stack Height (ft)** 50  
**Stack Diameter (in.)** 18.0  
**Stack Area (sq.ft.)** 1.767  
**Minutes Per Reading** 4.0  
**Minutes Per Point** 16.0

**Collection:**  
**Filter (grams)** 0.0000  
**Washings (grams)** 0.0000  
**Impinger (grams)** 0.0000  
**Total (grams)** 0.0000

**Gas Analysis (Vol. %):**  

	CO2	O2
Trav 1	7.13	12.63
Trav 2	8.38	11.00

**Average =** 7.76 11.82

**Condensate Collection:**  
Impinger 1 (grams) 210.0  
Impinger 2 (grams) 14.0  
Impinger 3 (grams) 4.0  
Impinger 4 (grams) 12.1

**Total Gain (grams)** 240.1

#### Calculation Section: Do Not Touch

Pm: 29.873  
Ps: 29.753  
AN (ft2): 0.00163790  
Vcond: 11.5248  
Bwo: 0.0743

Traverse	Point	Time (min.)	Dry Gas Meter (ft3)	Pitot ^P (in. H2O)	Orifice ^H (in. H2O)	Dry Gas Temperature Inlet (oF)	Dry Gas Temperature Outlet (oF)	Vacuum (in. Hg.)	XAD Exit (oF)	Stack (oF)	Wall Dist. (in.)	Isokin. (%)
		0.0	744.955									
1	1	4.0	747.880	0.080	1.68	64	52	5	36	1650	0.8	99.7
		8.0	750.860	0.080	1.68	77	53	5	37	1628	0.8	99.7
		12.0	753.880	0.080	1.68	80	55	5	37	1630	0.8	100.7
		16.0	757.220	0.090	2.10	81	55	5	37	1552	0.8	103.0
	2	20.0	760.450	0.090	2.10	82	56	6	36	1624	2.6	101.2
		24.0	763.410	0.080	1.68	82	56	6	36	1644	2.6	98.7
		28.0	766.130	0.060	1.29	82	56	6	36	1584	2.6	103.1
		32.0	768.860	0.060	1.29	82	56	6	36	1571	2.6	103.2
	3	36.0	771.840	0.080	1.70	82	58	5	36	1652	5.3	99.4
		40.0	774.850	0.080	1.70	83	58	5	36	1621	5.3	99.5
		44.0	777.540	0.060	1.29	83	58	5	36	1551	5.3	100.9
		48.0	780.230	0.060	1.29	84	58	5	36	1577	5.3	101.4
	4	52.0	783.050	0.070	1.46	82	59	5	37	1636	12.7	100.0
		56.0	786.040	0.080	1.67	82	60	5	37	1650	12.7	99.5
		60.0	789.020	0.080	1.67	82	60	5	37	1659	12.7	99.3
		64.0	792.010	0.080	1.67	82	60	5	37	1622	12.7	98.8
	5	68.0	794.810	0.070	1.45	82	60	5	37	1646	15.4	99.4
		72.0	797.630	0.070	1.45	82	60	5	37	1620	15.4	99.5
		76.0	801.570	0.090	2.61	83	61	5	38	1191	15.4	109.4
		80.0	805.520	0.090	2.61	83	61	5	38	1150	15.4	108.3
	6	84.0	808.080	0.060	1.25	84	62	5	38	1629	17.2	97.4
		88.0	810.860	0.070	1.50	86	62	5	38	1650	17.2	98.3
		92.0	813.460	0.060	1.25	86	62	5	37	1632	17.2	98.8
		96.0	816.050	0.060	1.25	86	62	5	37	1627	17.2	98.3
		0.0	816.050									
2	1	4.0	819.050	0.080	1.70	67	59	6	36	1620	0.8	100.6
		8.0	822.040	0.080	1.70	77	59	6	37	1636	0.8	99.7
		12.0	824.660	0.060	1.75	80	60	6	37	1620	0.8	100.1
		16.0	827.330	0.060	1.25	83	60	6	38	1579	0.8	100.6
	2	20.0	830.400	0.070	1.25	84	61	6	38	1457	2.6	103.7
		24.0	833.860	0.080	1.47	85	61	6	39	1367	2.6	106.6
		28.0	836.900	0.080	1.70	85	61	6	39	1622	2.6	100.1
		32.0	839.910	0.080	1.76	85	61	6	38	1650	2.6	99.8
	3	36.0	843.110	0.090	1.76	87	62	5	38	1637	5.3	99.4
		40.0	846.310	0.090	1.90	88	62	5	38	1610	5.3	98.7
		44.0	849.560	0.070	1.90	89	62	5	39	1390	5.3	107.4
		48.0	853.020	0.070	1.65	89	62	5	39	1226	5.3	109.1
	4	52.0	856.000	0.080	1.75	90	63	6	40	1638	12.7	97.9
		56.0	859.240	0.090	1.75	90	64	6	40	1610	12.7	99.5
		60.0	862.380	0.080	1.90	92	66	6	41	1545	12.7	100.4
		64.0	865.510	0.080	1.80	92	67	6	41	1590	12.7	101.0
	5	68.0	868.510	0.080	1.90	90	67	6	41	1630	15.4	98.0
		72.0	871.540	0.080	1.70	90	67	6	39	1656	15.4	99.5
		76.0	874.760	0.090	1.70	88	67	6	39	1625	15.4	99.2
		80.0	877.960	0.090	1.90	88	67	6	39	1659	15.4	99.4
	6	84.0	880.820	0.060	1.90	88	66	6	39	1470	17.2	104.0
		88.0	883.820	0.060	1.40	88	66	6	39	1385	17.2	106.5
		92.0	886.650	0.070	1.50	88	66	6	38	1618	17.2	98.7
		96.0	889.640	0.080	1.70	88	66	6	38	1639	17.2	98.1
			<b>Average:</b>	0.076	1.667	84.0	60.9	5.5	37.8	1572.4		100.9

Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)
71.095			96.0
	32.484	32.402	
	32.314	32.231	
	32.329	32.540	
	33.644	34.647	
	34.241	34.640	
	32.437	32.016	
	27.688	28.554	
	27.600	28.476	
	32.499	32.295	
	32.260	32.111	
	27.464	27.704	
	27.641	28.036	
	30.285	30.283	
	32.484	32.310	
	32.553	32.339	
	32.267	31.881	
	30.357	30.183	
	30.169	30.023	
	30.476	33.326	
	30.095	32.581	
	27.991	27.257	
	30.386	29.859	
	28.011	27.670	
	27.978	27.498	
73.590			96.0
	32.252	32.448	
	32.376	32.280	
	27.931	27.967	
	27.654	27.826	
	28.962	30.023	
	30.226	32.236	
	32.267	32.295	
	32.484	32.411	
	34.348	34.148	
	34.126	33.689	
	28.452	30.549	
	27.161	29.621	
	32.391	31.696	
	34.126	33.970	
	31.665	31.781	
	32.018	32.353	
	32.329	31.681	
	32.530	32.380	
	34.249	33.969	
	34.527	34.326	
	26.905	27.968	
	26.306	28.010	
	30.154	29.768	
	32.399	31.784	
144.685	30.9060	31.1675	192.0

Standard Conditions Set To:			77 oF	25 oC	536.67 oR	Other Constants and Conversions:	459.67 oR
			29.92 in.Hg	101.325 kPa			35.315 cf
Correct To:			0.048 * ml H2O = Vcond		(use 0.04707 for 68 oF) (use 0.04800 for 75 oF)		from mg/m to divide by 2288.3519 gr/cf
	0 % CO2	For no correction enter 0 or -1					
	11 % O2						

Client:

Jobsite:

Source:

Newmont, Hope Bay

Doris Camp

Incinerator stack

Date:

Run:

Run Time:

Oct. 1/09

3 PCDD/PCDF

10:03 - 13:22

Concentration:

0.00 mg/dscm

0.0000 gr/dscf

0.00 mg/Acm

0.0000 gr/Acf

0.00 mg/dscm (@ 11% O2)

0.0000 gr/dscf (@ 11% O2)

Emission Rate:

0.00 Kg/hr

0.000 lb/hr

Sample Gas Volume:

3.9619 dscm

139.913 dscf

Total Sample Time:

192.0 minutes

Average Isokineticity:

100.0 %

Flue Gas Characteristics

Moisture:

7.33 %

Temperature

873.1 oC

1603.6 oF

Flow

22.3 dscm/min

786 dscf/min

0.37 dscm/sec

13.1 dscf/sec

92.0 Acm/min

3248 Act/min

Velocity

9.336 m/sec

30.63 f/sec

Gas Analysis

12.34 % O2

7.25 % CO2

29.653 Mol. Wt (g/gmole) Dry

28.799 Mol. Wt (g/gmole) Wet

\* Standard Conditions:

Metric: 25 deg C, 101.325 kPa

Imperial: 77 deg F, 29.92 in.Hg



**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator stack

**Date:** Oct. 1/09  
**Run:** 3 PCDD/PCDF  
**Run Time:** 10:03 - 13:22

**Control Unit (Y)** 0.9851  
**Nozzle Diameter (in.)** 0.5480  
**Pitot Factor** 0.8568  
**Baro. Press. (in. Hg)** 30.05  
**Static Press. (in. Hg)** 0.04  
**Stack Height (ft)** 50  
**Stack Diameter (in.)** 18.0  
**Stack Area (sq.ft.)** 1.767  
**Minutes Per Reading** 4.0  
**Minutes Per Point** 16.0

**Collection:**  
**Filter (grams)** 0.0000  
**Washings (grams)** 0.0000  
**Impinger (grams)** 0.0000  
**Total (grams)** 0.0000

**Gas Analysis (Vol. %):**

	CO2	O2
Trav 1	7.50	12.00
Trav 2	7.00	12.67

**Average = 7.25 12.34**

**Condensate Collection:**

Impinger 1 (grams)	210.0
Impinger 2 (grams)	6.0
Impinger 3 (grams)	1.0
Impinger 4 (grams)	13.5

**Total Gain (grams) 230.5**

**Calculation Section: Do Not Touch**

Pm: 30.170  
Ps: 30.053  
AN (ft2): 0.00163790  
Vcond: 11.0640  
Bwo 0.0733

Traverse	Point	Time (min.)	Dry Gas Meter (ft3)	Pitot ^P (in. H2O)	Orifice ^H (in. H2O)	Dry Gas Temperature Inlet (oF)	Dry Gas Temperature Outlet (oF)	Vacuum (in. Hg.)	XAD Exit (oF)	Stack (oF)	Wall Dist. (in.)	Isokin. (%)
1	0	0.0	946.400									
		4.0	949.440	0.080	1.84	50	42	5	35	1580	0.8	104.7
		8.0	952.460	0.080	1.84	66	46	5	36	1582	0.8	102.1
		12.0	955.710	0.090	2.07	71	48	5	35	1564	0.8	102.5
		16.0	958.950	0.090	2.07	75	49	5	35	1575	0.8	101.9
	1	20.0	962.070	0.090	1.90	77	51	6	35	1651	2.6	99.5
		24.0	964.820	0.070	1.50	79	53	6	35	1627	2.6	98.5
		28.0	967.620	0.070	1.50	80	56	6	35	1620	2.6	99.7
		32.0	970.810	0.090	1.88	80	57	6	36	1644	2.6	100.7
	2	36.0	973.980	0.080	1.70	82	59	5	36	1580	5.3	104.1
		40.0	977.100	0.080	1.70	82	61	5	36	1572	5.3	102.1
		44.0	979.990	0.070	1.50	84	63	5	36	1636	5.3	102.2
		48.0	983.010	0.080	1.70	85	64	5	35	1628	5.3	99.6
	3	52.0	986.020	0.080	1.70	85	64	5	35	1618	12.7	99.0
		56.0	988.810	0.070	1.50	85	65	5	36	1630	12.7	98.3
		60.0	991.960	0.080	1.85	85	65	5	36	1590	12.7	102.9
		64.0	994.900	0.070	1.65	86	66	5	37	1564	12.7	101.8
	4	68.0	998.060	0.080	1.85	85	66	6	37	1588	15.4	103.1
		72.0	1000.790	0.060	1.40	85	66	6	36	1546	15.4	101.6
		76.0	1003.500	0.060	1.40	85	66	5	36	1620	15.4	102.7
		80.0	1006.150	0.060	1.30	85	66	5	36	1610	15.4	100.2
	5	84.0	1008.790	0.060	1.40	85	66	5	36	1589	17.2	99.3
		88.0	1011.580	0.070	1.50	86	67	5	38	1636	17.2	98.1
		92.0	1014.380	0.070	1.50	86	67	5	38	1621	17.2	98.1
		96.0	1017.260	0.070	1.61	86	67	5	38	1571	17.2	99.7
		0.0	1017.260									
2	0	4.0	1020.200	0.070	1.61	70	64	6	38	1580	0.8	103.9
		8.0	1023.110	0.070	1.61	73	67	6	39	1561	0.8	101.8
		12.0	1025.720	0.060	1.26	79	66	6	39	1629	0.8	99.7
		16.0	1028.340	0.060	1.26	85	66	6	38	1618	0.8	99.2
	1	20.0	1031.330	0.080	1.68	86	67	6	36	1636	2.6	98.4
		24.0	1034.330	0.080	1.68	87	68	6	36	1641	2.6	98.7
		28.0	1037.550	0.090	1.90	89	69	6	36	1650	2.6	99.9
		32.0	1040.740	0.090	1.90	91	69	6	36	1610	2.6	97.8
	2	36.0	1043.770	0.080	1.68	93	70	6	35	1626	5.3	98.6
		40.0	1046.780	0.080	1.68	94	71	6	35	1603	5.3	97.2
		44.0	1049.620	0.070	1.55	94	72	6	35	1612	5.3	98.1
		48.0	1052.470	0.070	1.55	95	72	6	35	1610	5.3	98.4
	3	52.0	1055.560	0.080	1.85	95	73	6	34	1595	12.7	99.4
		56.0	1058.630	0.080	1.85	96	74	6	36	1577	12.7	98.1
		60.0	1061.950	0.090	2.07	94	74	6	36	1559	12.7	99.8
		64.0	1065.260	0.090	2.07	95	74	6	38	1572	12.7	99.8
	4	68.0	1068.050	0.070	1.50	95	74	6	38	1649	15.4	97.0
		72.0	1070.870	0.070	1.50	95	74	6	39	1641	15.4	97.9
		76.0	1073.350	0.050	1.20	95	74	6	40	1560	15.4	99.8
		80.0	1075.840	0.050	1.25	94	74	6	38	1540	15.4	99.8
	5	84.0	1078.550	0.060	1.40	95	74	6	38	1613	17.2	100.9
		88.0	1081.130	0.060	1.40	95	74	6	38	1641	17.2	96.7
		92.0	1083.940	0.070	1.50	94	74	6	37	1629	17.2	97.3
		96.0	1086.880	0.070	1.60	94	74	6	37	1577	17.2	100.6
			<b>Average:</b>	0.074	1.634	85.6	65.6	5.6	36.5	1603.6		100.0

Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)
70.860			96.0
	31.804	33.305	
	31.820	32.476	
	33.601	34.427	
	33.692	34.342	
	34.316	34.160	
	30.091	29.625	
	30.040	29.948	
	34.259	34.513	
	31.804	33.113	
	31.742	32.402	
	30.156	30.827	
	32.176	32.047	
	32.099	31.788	
	30.113	29.592	
	31.882	32.799	
	29.633	30.153	
	31.867	32.840	
	27.313	27.759	
	27.812	28.572	
	27.745	27.799	
	27.604	27.420	
	30.156	29.594	
	30.048	29.488	
	29.684	29.609	
69.620			96.0
	29.750	30.908	
	29.611	30.136	
	27.872	27.784	
	27.799	27.588	
	32.238	31.730	
	32.276	31.852	
	34.307	34.257	
	33.981	33.233	
	32.161	31.705	
	31.983	31.091	
	29.983	29.426	
	29.968	29.474	
	31.921	31.719	
	31.781	31.180	
	33.559	33.501	
	33.667	33.584	
	30.249	29.340	
	30.192	29.543	
	25.020	24.961	
	24.896	24.839	
	27.765	28.005	
	27.952	27.022	
	30.105	29.297	
	29.728	29.897	
140.480	30.6296	30.6389	192.0

Standard Conditions Set To:		77 oF	25 oC	536.67	459.67 oR
		29.92 in.Hg	101.325 kPa		35.315 cf
Correct To:		0 % CO2	For no correction enter 0 or -1		2288.3519 gr/cf
		11 % O2			(use 0.04707 for 68 oF (use 0.04800 for 75 oF)

<b>Client:</b>	Newmont, Hope Bay	<b>Date:</b>	Sept. 30/09
<b>Jobsite:</b>	Doris Camp	<b>Run:</b>	1 Partic/ Metals
<b>Source:</b>	Incinerator Stack	<b>Run Time:</b>	09:00 - 10:06

<b>Particulate Concentration:</b>	<b>4.3 mg/dscm</b>	0.0019 gr/dscf
	1.1 mg/Acm	0.0005 gr/Acf
	3.5 mg/dscm (@ 11% O2)	0.0015 gr/dscf (@ 11% O2)
<b>Emission Rate:</b>	0.01 Kg/hr	0.013 lb/hr
<b>Sample Gas Volume:</b>	1.5329 dscm	54.135 dscf
<b>Total Sample Time:</b>	60.0 minutes	
<b>Average Isokineticity:</b>	103.9 %	

**Flue Gas Characteristics**

<b>Moisture:</b>	7.93 %	
<b>Temperature</b>	822.0 oC	1511.6 oF
<b>Flow</b>	23.7 dscm/min	837 dscf/min
	0.39 dscm/sec	13.9 dscf/sec
	95.2 Acm/min	3362 Act/min
<b>Velocity</b>	9.665 m/sec	31.71 f/sec
<b>Gas Analysis</b>	8.88 % O2	11.13 % CO2
	30.135 Mol. Wt (g/gmole) Dry	29.173 Mol. Wt (g/gmole) Wet

**\* Standard Conditions:**

Metric:	25 deg C, 101.325 kPa
Imperial:	77 deg F, 29.92 in.Hg

**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator Stack

**Date:** Sept. 30/09  
**Run:** 1 Partic/ Metals  
**Run Time:** 09:00 - 10:06

**Control Unit (Y)** 0.9851  
**Nozzle Diameter (in.)** 0.5783  
**Pitot Factor** 0.8568  
**Baro. Press. (in. Hg)** 29.70  
**Static Press. (in. H2O)** 0.04  
**Stack Height (ft)** 50  
**Stack Diameter (in.)** 18.0  
**Stack Area (sq.ft.)** 1.767  
**Minutes Per Reading** 5.0  
**Minutes Per Point** 5.0

**Gas Analysis (Vol. %):**

	CO2	O2
	7.00	13.00
	13.00	7.00
	11.00	9.00
	13.50	6.50
<b>Average =</b>	<b>11.13</b>	<b>8.88</b>

**Condensate Collection:**

Impinger 1 (grams)	50.0
Impinger 2 (grams)	27.0
Impinger 3 (grams)	5.0
Impinger 4 (grams)	4.0
Impinger 5 (grams)	2.0
Impinger 6 (grams)	9.1
<b>Total Gain (grams)</b>	<b>97.1</b>

**Calculation Section: Do Not Touch**

Pm: 29.881  
Ps: 29.703  
AN (ft2): 0.00182404  
Vcond: 4.6608  
Bwo: 0.0793

**Collection:**

<b>Filter (grams)</b>	<b>0.0048</b>
<b>Washings (grams)</b>	<b>0.0018</b>
<b>Impinger (grams)</b>	<b>0.0000</b>
<b>Total (grams)</b>	<b>0.0066</b>

Traverse	Point	Time (min.)	Dry Gas Meter Pitot ΔP (ft3)	Orifice ΔH (in. H2O)	Dry Gas Temperature Inlet (oF)	Dry Gas Temperature Outlet (oF)	Stack (oF)	Wall Dist. (in.)	Isokin. (%)	Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)	
1		0.0	689.600							28.620			30.0	
	1	5.0	693.950	0.080	2.35	66	60	1550	0.8	104.2	31.551	32.889		
	2	10.0	698.300	0.080	2.35	73	61	1556	2.6	103.6	31.598	32.737		
	3	15.0	703.200	0.100	2.95	77	61	1525	5.3	103.3	35.055	36.225		
	4	20.0	707.580	0.080	2.35	80	62	1529	12.7	102.8	31.386	32.276		
	5	25.0	713.060	0.095	2.64	82	62	1206	15.4	107.9	31.301	33.784		
	6	30.0	718.220	0.090	2.85	85	62	1236	17.2	105.1	30.739	32.309		
2		0.0	718.220							25.730			30.0	
	1	5.0	722.850	0.090	2.63	71	60	1580	0.8	105.0	33.714	35.384		
	2	10.0	727.460	0.090	2.63	82	61	1611	2.6	104.1	33.969	35.363		
	3	15.0	731.790	0.080	2.40	83	61	1590	5.3	103.0	31.863	32.829		
	4	20.0	735.860	0.070	2.15	85	61	1567	12.7	102.7	29.638	30.435		
	5	25.0	739.910	0.070	2.15	86	61	1601	15.4	102.9	29.885	30.765		
	6	30.0	743.950	0.070	2.15	86	62	1588	17.2	102.3	29.791	30.467		
			Average:	0.083	2.467	79.7	61.2	1511.6		103.9	54.350	31.7075	32.9553	60.0

\* **Standard Conditions:** Metric: 25 deg C, 101.325 kPa  
Imperial: 77 deg F, 29.92 in.Hg

**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator Stack

**Date:** Sept. 30/09  
**Run:** 2 Partic/ Metals  
**Run Time:** 15:03 - 16:11

Control Unit (Y) 0.9851  
 Nozzle Diameter (in.) 0.5783  
 Pitot Factor 0.8568  
 Baro. Press. (in. Hg) 29.75  
 Static Press. (in. H2O) 0.04  
 Stack Height (ft) 50  
 Stack Diameter (in.) 18.0  
 Stack Area (sq.ft.) 1.767  
 Minutes Per Reading 5.0  
 Minutes Per Point 5.0

**Gas Analysis (Vol. %):**

	CO2	O2
	7.50	12.00
	6.00	14.00
	7.50	12.00
	9.00	11.00
<b>Average =</b>	<b>7.50</b>	<b>12.25</b>

**Condensate Collection:**

Impinger 1 (grams)	73.0
Impinger 2 (grams)	6.0
Impinger 3 (grams)	3.0
Impinger 4 (grams)	1.0
Impinger 5 (grams)	1.0
Impinger 6 (grams)	8.9
<b>Total Gain (grams)</b>	<b>84.0</b>

**Collection:**

Filter (grams)	0.0065
Washings (grams)	0.0036
Impinger (grams)	0.0000
<b>Total (grams)</b>	<b>0.0101</b>

**Calculation Section: Do Not Touch**

Pm: 29.923  
 Ps: 29.753  
 AN (ft2): 0.00182404  
 Vcond: 4.0320  
 Bwo: 0.0730

Traverse	Point	Time (min.)	Dry Gas Meter Pitot ^P (ft3) (in. H2O)	Orifice ^H (in. H2O)	Dry Gas Temperature Inlet (oF) Outlet (oF)	Stack (oF)	Wall Dist. (in.)	Isokin. (%)	Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)		
1		0.0	890.000						25.280			30.0		
	1	5.0	893.790	0.060	1.75	60	52	1550	0.8	104.9	27.460	28.806		
	2	10.0	897.100	0.080	2.25	74	52	1629	2.6	79.9	32.325	25.829		
	3	15.0	901.550	0.090	2.55	79	52	1627	5.3	100.8	34.270	34.551		
	4	20.0	906.200	0.090	2.65	82	58	1528	12.7	102.0	33.447	34.107		
	5	25.0	910.880	0.090	2.62	86	60	1550	15.4	102.6	33.631	34.509		
	6	30.0	915.280	0.080	2.32	88	62	1584	17.2	102.7	31.975	32.846		
2		0.0	915.280						26.100			30.0		
	1	5.0	919.500	0.080	2.25	72	62	1668	0.8	102.0	32.626	33.289		
	2	10.0	924.170	0.090	2.65	84	64	1547	2.6	102.1	33.606	34.322		
	3	15.0	928.760	0.090	2.60	88	65	1579	5.3	100.7	33.873	34.108		
	4	20.0	933.000	0.080	2.25	89	66	1620	12.7	99.4	32.255	32.054		
	5	25.0	937.290	0.080	2.32	90	66	1589	15.4	99.7	32.014	31.924		
	6	30.0	941.380	0.070	2.05	91	67	1544	17.2	100.3	29.616	29.692		
			Average:	0.082	2.355	81.9	60.5	1584.6		99.8	51.380	32.2582	32.1699	60.0

**Date:** Oct. 1/09  
**Run:** 3 Partic/ Metals  
**Run Time:** 14:08 - 15:13

\* **Standard Conditions:** Metric: 25 deg C, 101.325 kPa  
Imperial: 77 deg F, 29.92 in.Hg

**Client:** Newmont, Hope Bay  
**Jobsite:** Doris Camp  
**Source:** Incinerator Stack

**Date:** Oct. 1/09  
**Run:** 3 Partic/ Metals  
**Run Time:** 14:08 - 15:13

**Control Unit (Y)** 0.9851  
**Nozzle Diameter (in.)** 0.5783  
**Pitot Factor** 0.8568  
**Baro. Press. (in. Hg)** 30.05  
**Static Press. (in. H2O)** 0.04  
**Stack Height (ft)** 50  
**Stack Diameter (in.)** 18.0  
**Stack Area (sq.ft.)** 1.767  
**Minutes Per Reading** 5.0  
**Minutes Per Point** 5.0

**Gas Analysis (Vol. %):**

CO2	O2
8.00	11.50
6.50	12.50
8.00	11.50
9.00	11.00
<b>Average = 7.88</b>	<b>11.63</b>

**Condensate Collection:**

Impinger 1 (grams)	50.0
Impinger 2 (grams)	24.0
Impinger 3 (grams)	1.0
Impinger 4 (grams)	7.0
Impinger 5 (grams)	1.0
Impinger 6 (grams)	8.7
<b>Total Gain (grams)</b>	<b>91.7</b>

**Collection:**

<b>Filter (grams)</b>	<b>0.0157</b>
<b>Washings (grams)</b>	<b>0.0240</b>
<b>Impinger (grams)</b>	<b>0.0000</b>
<b>Total (grams)</b>	<b>0.0397</b>

**Calculation Section: Do Not Touch**

Pm: 30.222  
Ps: 30.053  
AN (ft2): 0.00182404  
Vcond: 4.4016  
Bwo: 0.0777

Traverse	Point	Time (min.)	Dry Gas Meter (ft3)	Pitot ^P (in. H2O)	Orifice ^H (in. H2O)	Dry Gas Temperature		Stack (oF)	Wall Dist. (in.)	Isokin. (%)	Sample Volume (ft3)	Velocity Stack (ft/sec)	Velocity Nozzle (ft/sec)	Time Per Traverse (min.)
						Inlet (oF)	Outlet (oF)							
1	1	0.0	87.200								25.460	34.217	35.103	30.0
	2	5.0	91.650	0.090	2.52	72	54	1640	0.8	102.6				
	3	10.0	96.110	0.090	2.44	76	55	1621	2.6	101.8				
	4	15.0	100.180	0.070	2.05	79	56	1580	5.3	103.8				
	5	20.0	104.270	0.070	2.05	81	58	1550	12.7	103.2				
	6	25.0	108.460	0.080	2.25	83	59	1645	15.4	101.0				
		30.0	112.660	0.080	2.20	84	60	1652	17.2	101.2		32.299	32.610	
												32.352	32.731	
		0.0	112.660								26.320	31.796	33.626	30.0
2	1	5.0	117.050	0.080	2.34	69	57	1580	0.8	105.8				
	2	10.0	121.700	0.090	2.65	80	59	1561	2.6	103.9				
	3	15.0	126.150	0.090	2.52	83	61	1646	5.3	101.0				
	4	20.0	130.360	0.090	2.52	84	61	1631	12.7	95.1				
	5	25.0	134.650	0.080	2.26	85	61	1618	15.4	102.3				
		30.0	138.980	0.080	2.32	85	61	1572	17.2	102.1		32.091	32.837	
												31.734	32.414	
			<b>Average:</b>	0.083	2.343	80.1	58.5	1608.0		102.0	51.780	32.4827	33.1106	60.0

**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix H**

### **Dioxin and Furan Laboratory Results**



**SAMPLE RECEIPT FORM / CHEMICAL ANALYSIS FORM**

FILE #: PR91136

CLIENT: A. Lanfranco & Assoc.  
#101 – 9488 189 Street  
Surrey, BC  
V4N 4W7  
Phone – 604-881-2582  
Fax - 604-881-2581

**RECEIVED BY:** P.Caines      **DATE/TIME:** October 6, 2009 (9:00 a.m.)

**CONDITION:** intact, 10°C

**LABELLING:** all bottles, filters and XAD columns labelled

<u># of Samples</u>	<u>Sample Type</u>	<u>Sample (Client Codes)</u>	<u>Lab Codes</u>
5	XAD, filter, impinger, probe, final rinse	Newmont Blank	PR91136
5	XAD, filter, impinger, probe, final rinse	Newmont Run#1	PR91137
5	XAD, filter, impinger, probe, final rinse	Newmont Run #2	PR91138
5	XAD, filter, impinger, probe, final rinse	Newmont Run #3	PR91139

**STORAGE:** stored at 4 °C

**ANALYTES:** HRGC/HRMS analysis for dioxins/furans

**SPECIAL INSTRUCTIONS:** none

**METHODOLOGY**

Reference Method: Dioxin: SOP LAB01; Environment Canada 1-RM-3; EPA Method 23

Data summarized in Data Report Attached

Report sent to: Al Lanfranco

Date: October 22, 2009

Comments: Dioxin/Furans: The recovery of Internal Standard -OCDD (31%) was low for Run 3. All other QA/QC data associated with this analysis were acceptable according to the reference method.

---

David Hope PChem, CEO



**Acronyms used in reporting dioxins and furans:**

TCDD = Tetrachlorodibenzo-*p*-dioxin  
 PeCDD = Pentachlorodibenzo-*p*-dioxin  
 HxCDD = Hexachlorodibenzo-*p*-dioxin  
 HpCDD = Heptachlorodibenzo-*p*-dioxin  
 OCDD = Octachlorodibenzo-*p*-dioxin

TCDF = Tetrachlorodibenzofuran  
 PeCDF = Pentachlorodibenzofuran  
 HxCDF = Hexachlorodibenzofuran  
 HpCDF = Heptachlorodibenzofuran  
 OCDF = Octachlorodibenzofuran

**Acceptable recoveries for surrogates EPA Method 23**

	<b>Min (%)</b>	<b>Max (%)</b>
-2,3,7,8-TCDD	70	130
-2,3,4,7,8-PeCDF	70	130
-1,2,3,4,7,8-HxCDD	70	130
-1,2,3,4,7,8-HxCDF	70	130
-1,2,3,4,7,8,9-HpCDF	70	130

**Acceptable recoveries for Internal Standards**

	<b>EPA Method 23</b>		<b>Env. Can. 1-RM-3</b>	
	<b>Min (%)</b>	<b>Max (%)</b>	<b>Min (%)</b>	<b>Max (%)</b>
-2,3,7,8-TCDD	40	130	40	130
-1,2,3,7,8-PeCDD	40	130	40	130
-1,2,3,6,7,8-HxCDD	40	130	40	130
-1,2,3,4,6,7,8-HpCDD	25	130	40	130
-OCDD	25	130	40	130
-2,3,7,8-TCDF	40	130	40	130
-1,2,3,7,8-PeCDF	40	130		
-1,2,3,6,7,8-HxCDF	40	130		
-1,2,3,4,6,7,8-HpCDF	25	130		

# METHOD 23/1-RM-3 DATA REPORT

Client: A. Lanfranco & Assoc  
Client ID: BLANK - Newmont  
PRL ID: PR91136

Sample Date: -  
Date Extracted: 7-Oct-09  
Date Analysed: 19-Oct-09  
Filter Wt.: 0.43g

DIOXINS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDD	ND	2	
Total TCDD	5.3	2	2
1,2,3,7,8-PeCDD	ND	4	
Total PeCDD	23	4	3
1,2,3,4,7,8-HxCDD	ND	4	
1,2,3,6,7,8-HxCDD	ND	4	
1,2,3,7,8,9-HxCDD	6.7	4	
Total HxCDD	110	4	2
1,2,3,4,6,7,8-HpCDD	30	4	
Total HpCDD	30	4	1
OCDD	30	15	1
Total Dioxin TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	1
ND	1
ND	0.2
ND	0.2
0.67	0.67
0.3	0.3
0.03	0.03
1.0	3.4

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	1
ND	2
ND	0.2
ND	0.2
0.67	0.67
0.3	0.3
0.003	0.003
1.0	4.4

FURANS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDF	3.7	2	
Total TCDF	81	2	6
1,2,3,7,8-PeCDF	ND	4	
2,3,4,7,8-PeCDF	17	4	
Total PeCDF	51	4	2
1,2,3,4,7,8-HxCDF	14	4	
1,2,3,6,7,8-HxCDF	9.7	4	
1,2,3,7,8,9-HxCDF	7.8	4	
2,3,4,6,7,8-HxCDF	24	4	
Total HxCDF	87	4	5
1,2,3,4,6,7,8-HpCDF	36	4	
1,2,3,4,7,8,9-HpCDF	8.7	4	
Total HpCDF	45	4	2
OCDF	27	15	1
Total Furan TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
0.37	0.37
ND	0.1
8.5	8.5
1.4	1.4
0.97	0.97
0.78	0.78
2.4	2.4
0.36	0.36
0.087	0.087
0.027	0.027
15	15

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
0.37	0.37
ND	0.1
8.5	8.5
1.4	1.4
0.97	0.97
0.78	0.78
2.4	2.4
0.36	0.36
0.087	0.087
0.0027	0.0027
15	15

Total PCDD/PCDF Toxic Equivalent (pg)
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16	18
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16	19
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## Surrogate Recoveries (%)

<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	108
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	88
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	84
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	79
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	85

ND - none detected NDR - none detected based on peak ratio

## Internal Standards (%)

<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	67
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	81
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	91
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	56
<sup>13</sup> C <sub>12</sub> -OCDD	43
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	62
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	66
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	97
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	57

Patrick Pond



# METHOD 23/1-RM-3 DATA REPORT

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Client: A. Lanfranco & Assoc  
Client ID: Run 1 - Newmont  
PRL ID: PR91137

Sample Date: 29-Sep-09  
Date Extracted: 7-Oct-09  
Date Analysed: 19-Oct-09  
Filter Wt.: 0.45g

DIOXINS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDD	172	2	
Total TCDD	9900	2	4
1,2,3,7,8-PeCDD	1600	4	
Total PeCDD	15000	4	7
1,2,3,4,7,8-HxCDD	890	4	
1,2,3,6,7,8-HxCDD	1100	4	
1,2,3,7,8,9-HxCDD	1200	4	
Total HxCDD	29000	4	4
1,2,3,4,6,7,8-HpCDD	4700	4	
Total HpCDD	13000	4	2
OCDD	1800	15	1
Total Dioxin TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
172	172
800	800
89	89
110	110
120	120
47	47
1.8	1.8
1340	1340

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
172	172
1600	1600
89	89
110	110
120	120
47	47
0.18	0.18
2138	2138

FURANS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDF	1500	2	
Total TCDF	78000	2	14
1,2,3,7,8-PeCDF	1900	4	
2,3,4,7,8-PeCDF	4200	4	
Total PeCDF	37000	4	6
1,2,3,4,7,8-HxCDF	4900	4	
1,2,3,6,7,8-HxCDF	3600	4	
1,2,3,7,8,9-HxCDF	1800	4	
2,3,4,6,7,8-HxCDF	6600	4	
Total HxCDF	41000	4	7
1,2,3,4,6,7,8-HpCDF	10000	4	
1,2,3,4,7,8,9-HpCDF	1800	4	
Total HpCDF	17000	4	3
OCDF	3700	15	1
Total Furan TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
150	150
95	95
2100	2100
490	490
360	360
180	180
660	660
100	100
18	18
3.7	3.7
4157	4157

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
150	150
95	95
2100	2100
490	490
360	360
180	180
660	660
100	100
18	18
0.37	0.37
4153	4153

Total PCDD/PCDF Toxic Equivalent (pg)
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5497	5497
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6292	6292
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## Surrogate Recoveries (%)

<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	118
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	80
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	86
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	87
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	87

ND - none detected NDR - none detected based on peak ratio

## Internal Standards (%)

<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	80
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	88
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	71
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	52
<sup>13</sup> C <sub>12</sub> -OCDD	54
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	63
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	81
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	76
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	49

Patrick Pond



Pacific Rim Laboratories Inc. #103 - 19575 55A Avenue, Surrey, B.C. V3S 8P8

Tel: +604-532-8711 Fax: +604-532-8712 Email: info@pacificrimlabs.com

Form Name: DOC22 Data Report Dx 15-Feb-07 DGH

# METHOD 23/1-RM-3 DATA REPORT

Page 5 of 8

Client: A. Lanfranco & Assoc  
Client ID: Run 2 - Newmont  
PRL ID: PR91138

Sample Date: 30-Sep-09  
Date Extracted: 7-Oct-09  
Date Analysed: 19-Oct-09  
Filter Wt.: 0.46g

DIOXINS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDD	200	2	
Total TCDD	17000	2	7
1,2,3,7,8-PeCDD	3300	4	
Total PeCDD	33000	4	7
1,2,3,4,7,8-HxCDD	2500	4	
1,2,3,6,7,8-HxCDD	4300	4	
1,2,3,7,8,9-HxCDD	3400	4	
Total HxCDD	84000	4	4
1,2,3,4,6,7,8-HpCDD	19000	4	
Total HpCDD	54000	4	2
OCDD	10000	15	1
Total Dioxin TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
200	200
1650	1650
250	250
430	430
340	340
190	190
10	10
3070	3070

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
200	200
3300	3300
250	250
430	430
340	340
190	190
1	1
4711	4711

FURANS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDF	1800	2	
Total TCDF	91000	2	9
1,2,3,7,8-PeCDF	3600	4	
2,3,4,7,8-PeCDF	10000	4	
Total PeCDF	64000	4	3
1,2,3,4,7,8-HxCDF	9800	4	
1,2,3,6,7,8-HxCDF	7100	4	
1,2,3,7,8,9-HxCDF	4800	4	
2,3,4,6,7,8-HxCDF	14000	4	
Total HxCDF	78000	4	7
1,2,3,4,6,7,8-HpCDF	22000	4	
1,2,3,4,7,8,9-HpCDF	3500	4	
Total HpCDF	35000	4	3
OCDF	8600	15	1
Total Furan TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
180	180
180	180
5000	5000
980	980
710	710
480	480
1400	1400
220	220
35	35
8.6	8.6
9194	9194

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
180	180
180	180
5000	5000
980	980
710	710
480	480
1400	1400
220	220
35	35
0.86	0.86
9186	9186

Total PCDD/PCDF Toxic Equivalent (pg)
---------------------------------------

12264	12264
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13897	13897
-------	-------

## Surrogate Recoveries (%)

<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	117
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	98
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	91
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	86
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	82

ND - none detected NDR - none detected based on peak ratio

## Internal Standards (%)

<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	63
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	110
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	83
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	66
<sup>13</sup> C <sub>12</sub> -OCDD	41
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	60
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	83
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	81
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	64

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Form Name: DOC22 Data Report Dx 15-Feb-07 DGH

# METHOD 23/1-RM-3 DATA REPORT

Page 6 of 8

Client: A. Lanfranco & Assoc  
Client ID: Run 3 - Newmont  
PRL ID: PR91139

Sample Date: 1-Oct-09  
Date Extracted: 7-Oct-09  
Date Analysed: 19-Oct-09  
Filter Wt.: 0.45g

DIOXINS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDD	100	2	
Total TCDD	8700	2	6
1,2,3,7,8-PeCDD	1500	4	
Total PeCDD	15000	4	4
1,2,3,4,7,8-HxCDD	1100	4	
1,2,3,6,7,8-HxCDD	2000	4	
1,2,3,7,8,9-HxCDD	1500	4	
Total HxCDD	53000	4	4
1,2,3,4,6,7,8-HpCDD	9800	4	
Total HpCDD	28000	4	2
OCDD	3800	15	1
Total Dioxin TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
100	100
750	750
110	110
200	200
150	150
98	98
3.8	3.8
1412	1412

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
100	100
1500	1500
110	110
200	200
150	150
98	98
0.38	0.38
2158	2158

FURANS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDF	800	2	
Total TCDF	44000	2	12
1,2,3,7,8-PeCDF	1700	4	
2,3,4,7,8-PeCDF	4400	4	
Total PeCDF	27000	4	3
1,2,3,4,7,8-HxCDF	5000	4	
1,2,3,6,7,8-HxCDF	3600	4	
1,2,3,7,8,9-HxCDF	2100	4	
2,3,4,6,7,8-HxCDF	6500	4	
Total HxCDF	40000	4	7
1,2,3,4,6,7,8-HpCDF	11000	4	
1,2,3,4,7,8,9-HpCDF	2200	4	
Total HpCDF	23000	4	3
OCDF	5800	15	1
Total Furan TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
80	80
85	85
2200	2200
500	500
360	360
210	210
650	650
110	110
22	22
5.8	5.8
4223	4223

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
80	80
85	85
2200	2200
500	500
360	360
210	210
650	650
110	110
22	22
0.58	0.58
4218	4218

Total PCDD/PCDF Toxic Equivalent (pg)
---------------------------------------

5635	5635
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6376	6376
------	------

## Surrogate Recoveries (%)

<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	112
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	87
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	84
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	83
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	71

ND - none detected NDR - none detected based on peak ratio

## Internal Standards (%)

<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	77
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	96
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	74
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	42
<sup>13</sup> C <sub>12</sub> -OCDD	31
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	71
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	84
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	80
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	44

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Form Name: DOC22 Data Report Dx 15-Feb-07 DGH

# METHOD 23/1-RM-3 QC REPORT - BLANK

Page 7 of 8

Client: A. Lanfranco & Assoc  
Client ID: Lab Blank  
PRL ID: DF09429B

Sample Date: n/a  
Date Extracted: 7-Oct-09  
Date Analysed: 19-Oct-09

DIOXINS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDD	ND	2	
Total TCDD	ND	2	0
1,2,3,7,8-PeCDD	ND	4	
Total PeCDD	ND	4	0
1,2,3,4,7,8-HxCDD	ND	4	
1,2,3,6,7,8-HxCDD	ND	4	
1,2,3,7,8,9-HxCDD	ND	4	
Total HxCDD	ND	4	0
1,2,3,4,6,7,8-HpCDD	ND	4	
Total HpCDD	ND	4	0
OCDD	32	15	1
Total Dioxin TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	1
ND	1
ND	0.2
ND	0.2
ND	0.2
ND	0.02
0.032	0.032
0.032	2.7

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	1
ND	2
ND	0.2
ND	0.2
ND	0.2
ND	0.02
0.0032	0.0032
0.0032	3.6

FURANS			
Congeners	pg	DL pg	# of peaks
2,3,7,8-TCDF	ND	2	
Total TCDF	ND	2	0
1,2,3,7,8-PeCDF	ND	4	
2,3,4,7,8-PeCDF	ND	4	
Total PeCDF	ND	4	0
1,2,3,4,7,8-HxCDF	ND	4	
1,2,3,6,7,8-HxCDF	ND	4	
1,2,3,7,8,9-HxCDF	ND	4	
2,3,4,6,7,8-HxCDF	ND	4	
Total HxCDF	ND	4	0
1,2,3,4,6,7,8-HpCDF	ND	4	
1,2,3,4,7,8,9-HpCDF	ND	4	
Total HpCDF	ND	4	0
OCDF	ND	15	0
Total Furan TEQ			

I-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	0.1
ND	0.1
ND	1
ND	0.2
ND	0.2
ND	0.2
ND	0.2
ND	0.02
ND	0.02
ND	0.0075
0.00	2.0

WHO-TEQs	
(ND=0)	(ND=½DL)
pg	pg
ND	0.1
ND	0.1
ND	1
ND	0.2
ND	0.2
ND	0.2
ND	0.2
ND	0.02
ND	0.02
ND	0.00075
0.00	2.0

Total PCDD/PCDF Toxic Equivalent (pg)
---------------------------------------

0.032	4.7
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0.0032	5.7
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ND - none detected NDR - none detected based on peak ratio

## Internal Standards (%)

<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	79
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	102
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	109
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	75
<sup>13</sup> C <sub>12</sub> -OCDD	50
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	75
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	86
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	118
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	75



nd

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Form Name: DOC22 Data Report Dx 15-Feb-07 DGH

# QC REPORT - SPIKE

Client: A. Lanfranco & Assoc  
 Client ID: MATRIX SPIKE  
 PRL ID: DF09430S

Contact: Al Lanfranco  
 Date Extracted: 7-Oct-09  
 Date Analysed: 19-Oct-09

DIOXINS		Acceptable Recovery			Pass/Fail
Congeners	LOF pg	Recovery %	Min %	Max %	
2,3,7,8-TCDD	200	88	80	120	Pass
1,2,3,7,8-PeCDD	1000	95	80	120	Pass
1,2,3,4,7,8-HxCDD	1000	113	80	120	Pass
1,2,3,6,7,8-HxCDD	1000	93	80	120	Pass
1,2,3,7,8,9-HxCDD	1000	105	80	120	Pass
1,2,3,4,6,7,8-HpCDD	1000	89	80	120	Pass
OCDD	2000	96	80	120	Pass

Surrogate Recoveries %
70
106
-
90
-
78
46

FURANS		Acceptable Recovery			Pass/Fail
Congeners	LOF pg	Recovery %	Min %	Max %	
2,3,7,8-TCDF	200	86	80	120	Pass
1,2,3,7,8-PeCDF	1000	89	80	120	Pass
2,3,4,7,8-PeCDF	1000	82	80	120	Pass
1,2,3,4,7,8-HxCDF	1000	113	80	120	Pass
1,2,3,6,7,8-HxCDF	1000	97	80	120	Pass
1,2,3,7,8,9-HxCDF	1000	81	80	120	Pass
2,3,4,6,7,8-HxCDF	1000	89	80	120	Pass
1,2,3,4,6,7,8-HpCDF	1000	98	80	120	Pass
1,2,3,4,7,8,9-HpCDF	1000	83	80	120	Pass
OCDF	2000	85	80	120	Pass

Surrogate Recoveries %
72
98
-
-
90
-
-
76
-
-

LOF - Level of Fortification

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**DORIS NORTH GOLD MINE PROJECT**  
**Incinerator Stack Testing Compliance Report for**  
**Section 4 Item 30 of the Project Certificate**

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## **Appendix I**

### **Quality Assurance Quality Control (QAQC) Results**

# Quality Assurance/Quality Control Program Results

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## **Pre and Post Test Leak Checks**

Each test is required to be leak checked prior to, and following the test. The leak checks must show less than 0.02 cubic feet per minute (cfm). All tests passed the code leak check requirements. Evidence of the leak checks is shown on each data sheet.

## **Equipment Calibrations**

All emission monitoring equipment used for the Doris camp incinerator emission monitoring was calibrated to Environment Canada specifications. Dry gas meters, pitot tubes and temperature measuring devices were calibrated within four months of the test date.

## **Proofing of Dioxin Glassware and Supplies**

Although not required by Nunavut/GNWT, it is our practice to verify that the glassware and sorbent used in dioxin tests is free of contamination. Proofs of the glassware and XAD are implied by the very low Blank sample results.

## **Analysis of Blank Materials and Reagents**

All blank materials and reagents yielded very low or non-detectable levels of target species.

## **Spiking and Recovery of Dioxin/Furan Surrogates**

The recovery of the five labelled surrogates ranged from 71 to 118. These recoveries comply with EPA Method 23 requirements of 70 to 130%. In addition, all data was recovery corrected for each congener. Recoveries of all internal standards ranged from 41 to 110%, complying with EPS 1/RM/2 and Method 23 requirements of 40 to 130% recovery (except for 31% recovery of Octa dioxin for Run 3 only).

## **Spiking and Recovery Assessments of Inorganic Samples**

Blanks of all reagents used for sample collection were spiked to known contaminant concentrations and analyzed with the source samples. Normally a high and low spike was conducted. The various recoveries are reported on the analytical data in Appendix 2. In summary the results are:

	<b>Spike 1</b>	<b>Spike 2</b>	<b>Spike 3</b>
Hg	95 % recovery	62* % recovery	88% recovery

*\* Hg was not detectable in this component of any sample*

## **Chain of Custody**

All samples were in the possession of the stack test team until relinquishing to the courier/shipping companies used. The samples were inspected on arrival, and shipping containers were observed to be sealed on arrival, with no apparent tampering or sample loss in shipment.