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Method 0060 or Method 29

Method for Determining Metals and Mercury Emissions in Stack Gas

Fundamentals of the Method for Analysis of Metallic Analytes in Method 0060 or Method 29 Multi-Metals Train (MMT) Samples

Collection and Analysis of Method 0060 or Method 29 Multi-Metals Train Samples

A U.S. EPA Method 0060 Multi-Metals Train (MMT) configuration is used to collect stack gas samples for the assessment of metals (including mercury) content. Stack gas samples are collected and the following MMT sample fractions are collected from the train after the completion of each run:

- Particulate Filter (metals and mercury)
- 0.1N HNO_3 Probe Rinse (metals and mercury)
- 5% HNO_3/10% H_2O_2 Impinger Solution (metals and mercury)
- Empty Impinger #4 (mercury only)
- 4% $KMnO_4$/10% H_2SO_4 Impingers (mercury only)
- 8N HCl Impinger Rinses (mercury only)

The particulate filter is combined with the 0.1N HNO_3 probe rinse after separate digestions using hydrofluoric acid to form a single Front half Composite sample. The first impinger is initially empty and is used to condense any excess moisture from the stack gas emissions. The second and third impingers contain 5% HNO_3/10% H_2O_2 for the sampling of total metals and mercury. The volume of the nitric acid/peroxide impinger composite is measured and processed through a total metals preparation using Methods 3050 and 3051, but without the incorporation of HF since these samples are considered to be an aqueous matrix. This composite is referred to as the Backhalf Composite. Analysis is completed using Method 6010B (ICAP). Mercury is determined in this composite by the preparation of a portion of the final digestate.

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Preparations and analyses for mercury is conducted using Method 7470A (Cold Vapor Atomic Absorption or CVAA). The train also includes impingers for trapping elemental mercury (Hg). The empty impinger, 4th in the train is analyzed separately by rinsing it with 0.1N HNO_3 and analyzing the rinsate for mercury. The two impingers containing 4% $KMnO_4$/10% H_2SO_4 that are placed behind the initially empty impinger in the sampling train are combined to form a mercury composite sample, which is prepared and analyzed by Method 7470A. The permanganate impinger glassware is given a final rinse in the field with 8N HCl to remove residues that collect on the glass walls of the impingers during the sampling. The residues are presumably forms of reduced $KMnO_4$, such as MnO_2, that are insoluble in the impinger reagent media, but are easily removed with the HCl. The 8N HCl sample was prepared and analyzed by Method 7470A for mercury.

Contamination Sources of Metals

Probably the most frequently encountered source of metals is the particulate filter due to poor selection. The particulate filter should be high purity quartz suitable to the use of metals sampling.

Another major source of background metals is the selection of bottles used during sample collection. Bottles, regardless of vendor or grade, should undergo the same glassware cleaning process as the MMT glassware. The cleaning steps are:

- Wash and rinse all glassware with hot soapy water and regular tap water
- Soak all glassware in a 10 percent HNO_3 solution (approximate) for four hours
- Rinse thoroughly with D.I. water, followed by acetone, and finally left to air dry
- Brushes and probe rams should be non-metallic and receive the same cleaning as the glassware.

Boston round amber bottles are the best style containers for liquid samples. Wide mouth packer bottles are prone to leakage.

Chemical reagents that make up the impinger solutions or used for glassware rinses should be of the highest purity available.

Method 0060 Train Metallic Analyte Data Qualifiers (Flags)

Laboratory-assigned data qualifiers (or data flags) are displayed with each metallic analyte when required. The data qualifiers are listed below with their corresponding definitions at the end of each run data set.

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Data Qualifiers for Metallic Analyte Results

Qualifier or Flag	Definition
"B"	The compound was detected at a concentration below the reporting limit or quantitation limit and lowest calibration point, but above the Method Detection Limit (MDL). The result is therefore considered an estimated value since the reported number is not bracketed by an upper and lower calibration point.
"J"	This compound was detected in the laboratory method blank that was processed as a companion sample in the sample delivery group (SDG) batch. Under these conditions, the value reported is considered an estimated value, since a portion of the analyte total may be derived from background sources.
"U"	This analyte was not detected down to the laboratory MDL when the analyses are conducted in such a way as to report data estimates below the reporting limit.
"<"	When listed, the less than (<) sign indicates that at least one sample fraction result is either a “non-detect” value down to the MDL of the measurement that carries, or an estimated “hit” value that is below the RDL. In either case, the final value included in the train total is the default RDL value and the actual value is known to be less than (<) the displayed result.

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Method 0060 or Method 29

Metals and Mercury Emissions in Stack Gas

SAMPLE PRESERVATION AND HOLDING TIMES

The holding times and preservation techniques are either those recommended in Title 40 CFR Section 136.3, Table 11, "Required Containers, Preservation Techniques, and Holding Times," or those presented by EPA in Table 3-1 of the *Handbook - Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration* (EPA-625/6-89-023).

Measurement	Matrix	Preservation	Holding Time[a]
Metals	Particulate Filter	None required	6 months to analysis
	0.1N Nitric Acid (HNO_3) Probe Rinse	None required	6 months to analysis
	5% HNO_3/10% H_2O_2 Impinger Composite	None required	6 months to analysis
	Empty Impinger #4	None required	6 months to analysis
	4% $KMnO_4$/10% H_2SO_4 Impinger Composite	None required	6 months to analysis
	8N HCl Impinger Rinse	None required	6 months to analysis
Mercury	Particulate Filter	None required	28 days to analysis
	0.1N Nitric Acid (HNO_3) Probe Rinse	None required	28 days to analysis
	5% HNO_3/10% H_2O_2 Impinger Composite	None required	28 days to analysis
	Empty Impinger #4	None required	28 days to analysis
	4% $KMnO_4$/10% H_2SO_4 Impinger Composite	None required	28 days to analysis
	8N HCl Impinger Rinse	None required	28 days to analysis

[a] Holding times are calculated from the date of collection.

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Method 0060/29 Recommended Sample Collection Methods, Frequency, and Containers for a 3 Run CPT

Sample Name (Matrix)	Analysis	Type of Container(s)	Sampling Method	Sampling Frequency	Test Samples	Field QC Samples	Total Field Samples Collected
MMT Front-Half Composite (Filter and 0.1N nitric acid probe rinse)	Target metals and mercury	Petri dish, 250-mL Boston-round amber glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	2 reagent blanks, 1 blank train	6
MMT Nitric Acid Impinger Composite (5% nitric acid and 10% hydrogen peroxide impinger contents)	Metals and mercury	1 gallon amber Wheaton glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5
MMT Empty Impinger (Empty at start of test)	Mercury	250 mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 blank train	4
MMT Potassium Permanganate Impinger Composite (4% potassium permanganate and 10% sulfuric acid impinger composite and deionized water rinses)	Mercury	500 mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5
MMT 8N Hydrochloric Acid Impinger Rinse (8N HCl)	Mercury	250-mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ±0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5

[a] Method 29 is appropriate for sampling gases for metals. Taken from 40 CFR 60 Appendix A, "Determination of metals emissions from stationary sources".

[b] Method 0060 is appropriate for sampling gases for metals. Taken from "Determination of Metals in Stack Emissions". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996). U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

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Method 0060 or Method 29

Metals and Mercury Emissions in Stack Gas

Recommended Quality Measurements for a 3 Run CPT

Analytical Parameter (Analysis)	Sample Name or Type	Total No. of Field Samples	Analytical Procedure Description (Method)	Laboratory QC Measurement Type	Frequency of Applied QC Measurement Type	Total No. of Laboratory QC Measurements	Field QC Measurement Type	Total No. of Field QC Samples	Total No. of Laboratory Analyses[a]
Metals	MMT Front half Composite (Filter and 0.1N Nitric Acid Probe Rinse)	3	Digestion, ICP (EPA Method 29, SW-6010/6020)	PDS	Every Front half composite	3	Reagent Blank (1 filter and 1 0.1N nitric acid probe rinse solution	2	8
	MMT Backhalf Composite (5% Nitric Acid and 10% Hydrogen Peroxide)	3	Digestion, ICP (EPA Method 29, SW-6010/6020)	PDS	One Backhalf per test condition	1	Reagent Blank (5% nitric acid and 10% hydrogen peroxide solution)	1	5
Mercury	MMT Front half Composite (Filter and 0.1N Nitric Acid Probe Rinse)	3	Digestion, CVAA (EPA Method 29, SW-7470)	PDS	Every Front half composite	3	Reagent Blank (1 filter and 1 0.1N nitric acid probe rinse solution)	2	8
	MMT Backhalf Composite (5% Nitric Acid and 10% Hydrogen Peroxide)	3	Digestion, CVAA (EPA Method 29, SW-7470)	MS/MSD	One set per test condition	2	Reagent Blank (5% nitric acid and 10% hydrogen peroxide solution)	1	6
	Impinger 4 (empty)	3	Digestion, CVAA (EPA Method 29, SW-7470)	MS/MSD	One set per test conditions	2	Reagent blank (0.1N impinger rinse solution)	1	6

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Analytical Parameter (Analysis)	Sample Name or Type	Total No. of Field Samples	Analytical Procedure Description (Method)	Laboratory QC Measurement Type	Frequency of Applied QC Measurement Type	Total No. of Laboratory QC Measurements	Field QC Measurement Type	Total No. of Field QC Samples	Total No. of Laboratory Analyses[a]
	4% Potassium Permanganate and 10% Sulfuric Acid Impinger Composite	3	Digestion, CVAA (EPA Method 29, SW-7470)	MS/MSD	One set per test conditions	2	Reagent blank (4% potassium permanganate and 10% sulfuric acid impinger solution)	1	6
Mercury (continued)	8N hydrogen chloride impinger rinse samples	3	Digestion, CVAA (EPA Method 29, SW-7470)	MS/MSD	One set per test conditions	2	Reagent blank (8N hydrogen chloride impinger rinse solution)	1	6

Analytical Quality Control Checks, Frequencies, Target Acceptance Criteria, and Corrective Action

Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
Metals by ICP (Method 6010B)	Initial Calibration Verification (ICV/ICB)	Beginning of analysis sequence	ICV • 90 to 110% recovery • %RSD of at least two exposures < 5% ICB • Absolute value of concentration < RL	(1) Correct problem (2) Recalibrate
	Continuing Calibration Verification (CCV/CCB)	Before and after sample analysis, and after every ten (10) samples	CCV • 90 to 110% recovery • %RSD of at least two exposures < 5% CCB • Absolute value of concentration < RL	(1) Correct problem (2) Recalibrate (3) Reanalyze affected samples
	Laboratory Method Blanks	Once per digestion batch (maximum 20 samples)	Target analyte concentrations • Absolute value of concentration < RL	Flag data associated with method blanks
	Laboratory Control Sample/Laboratory Control Sample duplicate (LCS/LCSD)	Once per digestion batch (maximum 20 samples) LCSD not required if MS/MSD performed	Accuracy • %Recovery: 80 – 120% Precision (if applicable) • RPD ≤ 20%	(1) Flag LCS/LCSD data (2) Discuss in final report

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Parameter/Method	QC Check	Frequency	Target Criteria	Corrective Action
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis	One per 20 samples per matrix (excluding filters) • If MS/MSD not performed, LCSD is required.	Accuracy • %Recovery: 75 – 125% Precision (if applicable) • RPD ≤ 20%	(1) Flag MS/MSD data (2) Discuss in final report
	Post-digestion spikes	One per 20 samples per client's request if MS/MSD not performed	75 - 125% recovery	(1) Flag PDS data (2) Discuss in final report
Mercury by CVAA (combined Methods 7470/7471)	Initial Calibration (minimum five (5) standards and one (1) blank)	Daily, before sample analysis	Fit of standard curve • Correlation coefficient ≥ 0.995	Repeat calibration
	Initial Calibration Verification (ICV/ICB)	Beginning of analytical sequence	ICV • 90 to 110% recovery ICB • Absolute value of concentration < RL	(1) Correct problem (2) Recalibrate
	Continuing Calibration Verification (CCV/CCB)	Before and after sample analysis, and after every ten (10) samples	CCV • 80 to 120% recovery CCB • Absolute value of concentration < RL	(1) Correct problem (2) Recalibrate (3) Reanalyze affected samples
	Laboratory Method Blanks	Once per digestion batch (maximum 20 samples)	Target analyte concentrations • Absolute value of concentration < RL	Flag data associated with method blanks, reprep and reanalyze if enough sample remaining
	Laboratory Control Sample/Laboratory Control Sample duplicate (LCS/LCSD)	Once per digestion batch (maximum 20 samples) LCSD not required if MS/MSD performed	Accuracy • %Recovery: 80 – 120% Precision (if applicable) • RPD ≤ 20%	(1) Flag LCS/LCSD data (2) Discuss in final report
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis	One per 20 samples per matrix (excluding filters) • If MSD not performed, LCSD is required.	Accuracy • %Recovery: 80 – 120% Precision (if applicable) • RPD ≤ 20%	(1) Flag MS/MSD data (2) Discuss in final report

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Method 0060/29 Recommended Sample Collection Methods, Frequency, and Containers for a 3 Run CPT

Sample Name (Matrix)	Analysis	Type of Container(s)	Sampling Method	Sampling Frequency	Test Samples	Field QC Samples	Total Field Samples Collected
MMT Front-Half Composite (Filter and 0.1N nitric acid probe rinse)	Target metals and mercury	Petri dish, 250-mL Boston-round amber glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	2 reagent blanks, 1 blank train	6
MMT Nitric Acid Impinger Composite (5% nitric acid and 10% hydrogen peroxide impinger contents)	Metals and mercury	1 gallon amber Wheaton glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5
MMT Empty Impinger (Empty at start of test)	Mercury	250 mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 blank train	4
MMT Potassium Permanganate Impinger Composite (4% potassium permanganate and 10% sulfuric acid impinger composite and deionized water rinses)	Mercury	500 mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ≤0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5
MMT 8N Hydrochloric Acid Impinger Rinse (8N HCl)	Mercury	250-mL amber Boston-round glass	Method 29[a] Method 0060[b]	Collect 2 m^3 at a sampling rate of ±0.75 m^3/hr.	3	1 reagent blank, 1 blank train	5

[a] Method 29 is appropriate for sampling gases for metals. Taken from 40 CFR 60 Appendix A, "Determination of metals emissions from stationary sources".

[b] Method 0060 is appropriate for sampling gases for metals. Taken from "Determination of Metals in Stack Emissions". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996). U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

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Method 0060 or Method 29

Metals and Mercury Emissions in Stack Gas

Sampling and Field Procedure for Metals in Stack Gases

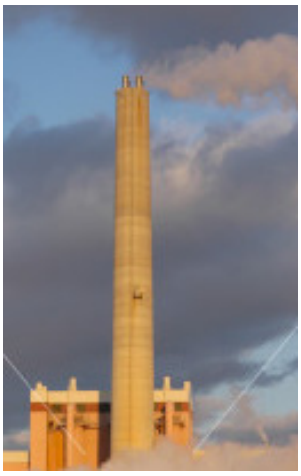
Sample Name: Stack Gas Multi-Metals Train (MMT)

Note: The recommended sample names for these samples are:

- Particulate Filter
- 0.1N Nitric Acid (HNO_3) Probe Rinse
- 5% HNO_3/10% H_2O_2 Impinger Composite
- Empty Impinger #4
- 4% $KMnO_4$/10% H_2SO_4 Impinger Composite
- 8N HCl Impinger Rinse

The following metals are routinely analyzed for in the MMT:

- Antimony (Sb)
- Arsenic (As)
- Barium (Ba)
- Beryllium (Be)
- Cadmium (Cd)
- Calcium (Ca)
- Chromium (Cr)
- Iron (Fe)
- Lead (Pb)
- Manganese (Mn)
- Mercury (Hg)
- Magnesium (Mg)
- Nickel (Ni)
- Phosphorus (P)
- Potassium (K)
- Selenium (Se)
- Silver (Ag)
- Sodium (Na)
- Strontium (Sr)
- Thallium (Tl)
- Tin (Sn)
- Titanium (Ti)
- Vanadium (V)



Sampler: Stack Sampling Engineer

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Process Sample Location: Stack Sampling Platform

Sampling & Health & Safety Equipment: Sampling and safety equipment is as follows:

- U.S. EPA Method 5 Multi-Metals Sampling Train
- Glass petri dish with particulate filter (PallFlex Tissue Quartz, 2500QAT-UP, 82.6 mm)
- Impinger chemical reagents
- Glass sample containers with Teflon®-lined lids
- Latex gloves
- Graduated cylinder
- Safety glasses or face shield
- Gloves and other safety equipment as required

The following containers are appropriate for the MMT samples:

- Petri Dish (Particulate Filter)
- 250 mL glass Boston Round (0.1N Nitric Acid Probe and Nozzle Rinse)
- 1 Liter amber glass Boston Round (Condensate and 5% HNO_3/10% H_2O_2 Impinger Composites)
- 250 mL glass Boston Round (0.1N HNO_3 rinse of Impinger #4)
- 500 mL glass Boston Round (4% $KMnO_4$/10% H_2SO_4 Impinger Composite)
- 250 mL glass Boston Round (8N HCl Impinger Rinse)

Note: Sample containers and particulate filters can be a significant source of metal's background. EPA Level III bottles should be used to collect samples. The narrow neck style is recommended since it is designed to seal liquids in whereas a widemouth design is prone to leak, and can contaminate the sample at the lid by allowing solutions to get behind the Teflon® insert. The narrow neck style have build in seals in the lids. Bottles and Petri dishes should be made of glass and cleaned by the glassware cleaning procedure using nitric acid (HNO_3).

Particulate filters should be made of high purity quartz and not glass filters. High background of metals is a problem with most filters other than high purity quartz.

Sample Collection Frequency: Continuously for approximately three (3) hours during each sampling run. Sampling volume will be 2 m^3 at a rate not to exceed 0.75 m^3/hour.

Sampling Procedures: The sampling train is assembled with a particulate filter; an empty first impinger; 100 ml of a 5% HNO_3/10% H_2O_2 solution in the second and third impingers; 100 ml of a 4% $KMnO_4$/10% H_2SO_4 in the fifth and sixth impingers, and silica gel in the seventh impinger. The fourth impinger is intentionally left empty. The impinger containing the indicating silica gel will be initially weighed to the nearest 0.5 gram.

An initial traverse will be made with a pitot tube at each sample port following U.S. EPA Methods 1 and 2 to establish the stack velocity profile, temperature, and flow rate, and to check for cyclonic air flow. Sample point location will be in accordance with U.S. EPA Method 1. The total sampling time during a run

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will be approximately 4 hours with a nominal 2 dry standard cubic meters of sample collected. Larger volumes may be collected if lower in stack detection limits are needed. U.S. EPA Method 5 procedures are followed for pre-test and post-test leak checks, isokinetic sampling rate, and filter change-outs (if needed), port changes, and data recording.

The sampling train will be leak tested according to U.S. EPA Method 5 protocols. A Teflon® plug or a sampler's thumb covered with Teflon® tape will be placed over the end of the nozzle to ensure that no contaminants are transferred to the train during nozzle leak checks.

Sample Recovery - The probe section of the sampling train will be removed after the final leak check has passed the required criteria. The probe and the filter impinger assembly of the sampling train are transferred intact to the cleanup area for sample recovery as follows:

- The particulate filter is removed from its holder and carefully placed into its original glass petri dish, which is sealed with Teflon® tape and sealed in a Ziploc® bag for shipping.

NOTE: The acetone rinsing procedure will be eliminated when the determination of particulate emissions are not part of the MMT.

- The internal surfaces of the nozzle, probe, and front half of the filter holder are cleaned by repeated rinsing with a 0.1N HNO_3 solution, brushed, followed by a final HNO_3 rinse. These nitric acid rinsates are collected together in a separate pre-labeled, numbered sample container.

- The volumetric contents of the first three impingers (#1, #2, & #3) are measured to the nearest 0.5 milliliter individually and delivered into a single, numbered, pre-labeled sample container. The impingers are then rinsed with the 5% HNO_3/10% H_2O_2 solution which is then added to the sample collection bottle.

- The volumetric contents of impinger #4 is measured to the nearest 0.5 milliliter using a graduated cylinder and delivered to a single numbered, pre-labeled sample container. The 4th impinger is then rinsed with a 100 mL volume of 0.1N HNO_3 and added to the sample container. This sample will be analyzed separately for mercury (Hg).

- The volumetric contents of the fifth and sixth impinger catches are measured to the nearest 0.5 milliliter individually and delivered into a single, numbered, pre-labeled sample container. The impingers are then rinsed with the 4% $KMnO_4$/10% H_2SO_4 solution followed by a rinse with 100 mL of DI water which are then added to the sample collection bottle. Each 100 mL portion is sufficient to afford 3 rinses of each impinger. This sample composite will be analyzed separately for mercury (Hg).

- If no visible deposits remain after the H_2O rinse, then no further rinse is needed. However, if deposits or coloration remain on the impinger surfaces,

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rinse all surfaces of the 5th and 6th impingers with 100 mLs of 8N HCl taking care to contact all sides of the glassware. Finally, deliver the 100 mLs of acid to a numbered, pre-labeled sample container.

- The silica gel contents of the seventh silica gel impinger are weighed to the nearest 0.5 g for determining the amount of moisture collected. Note the color of the indicating silica gel and make a notation of its condition.

Quality Control:

A complete multi-metals blank train should be prepared once during the test burn series, set up near the base of the stack in a manner similar to the actual multi-metals sampling train. All associated leak checks should be conducted on the blank train. The blank train should remain sealed with its filter box and probe heated at that location during one test run. The blank train samples will be recovered using the same procedures as those described for the actual sampling trains.

Glassware Preparations Prior to Tests:

The glassware for performing the MMT metals emissions tests is prone to display memory effects for several metals unless it is carefully prepared before testing. MMT glassware for the actual testing and for the blank train (including spare parts, bottles, and Petri dishes) should be prepared by the following metals removal procedure:

- Wash and rinse all glassware with hot soapy water and regular tap water
- Soak all glassware in a 10 percent HNO_3 solution (approximate) for four hours
- Rinse thoroughly with D.I. water, followed by acetone, and finally left to air dry
- Brushes and probe rams should be non-metallic and receive the same cleaning as the glassware

Reagent blanks for each of the following solutions or media should be collected as individual samples and submitted to the laboratory for analysis:

- Particulate Filter
- 0.1 N HNO_3 Probe and Nozzle Rinse Solution
- 5% HNO_3/10% H_2O_2 Impinger Solution
- 4% $KMnO_4$/10% H_2SO_4 Impinger Solution
- 8N HCl Impinger Rinse Solution

All bottles containing liquid samples should be marked on the outside of the container to show the height of fluid level so that leakage during shipment can be demonstrated not to have occurred.

Method References:

U.S. EPA Methods 1, 2, 3, and 5, Appendix A, Reference Methods, New Source Performance Standards, 40 CFR 60.

Method 29 - 40CFR 60 Appendix A, "Determination of Metals Emissions from Stationary Sources".

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Method 0060 – "Determination of Metals In Stack Emissions". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

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Analysis of Multi-Metals Train (MMT) Samples

Sample Name: Multi-Metals Train (Method 29/0060) Samples

Sample Holding Time: 6 months to analysis from date of sample collection, 28 days for mercury analysis

Analysis Procedures: ***Front Half Sample Preparations:***

- MMT Front Half Composite (Particulate filter and 0.1N HNO_3 probe and nozzle rinses)

The 0.1N HNO_3 probe rinse sample and particulate filter are digested separately with hydrofluoric acid (HF) and nitric acid (HNO_3). The digestates are combined and analyzed by CVAA (Method 7470) and by ICAP (Method 6010/6020) for the following metals:

- Aluminum (Al)
- Antimony (Sb)
- Arsenic (As)
- Barium (Ba)
- Beryllium (Be)
- Cadmium (Cd)
- Calcium (Ca)
- Chromium (Cr)
- Cobalt (Co)
- Copper (Cu)
- Iron (Fe)
- Lead (Pb)
- Magnesium (Mg)
- Manganese (Mn)
- Mercury (Hg)
- Nickel (Ni)
- Phosphorus (P)
- Potassium (K)
- Selenium (Se)
- Silver (Ag)
- Sodium (Na)
- Strontium (Sr)
- Thallium (Tl)
- Tin (Sn)
- Titanium (Ti)
- Vanadium (V)
- Zinc (Zn)

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Back Half Sample Preparations:

- MMT Back Half Nitric Acid Impinger Composite (5% HNO_3/10% H_2O_2)

The back half impinger composite sample is prepared by total metals digestion (Method 0060). The digestate is then analyzed by CVAA (Method 7470) and by ICAP (Method 6010/6020) for the following metals:

- Aluminum (Al)
- Antimony (Sb)
- Arsenic (As)
- Barium (Ba)
- Beryllium (Be)
- Cadmium (Cd)
- Calcium (Ca)
- Chromium (Cr)
- Cobalt (Co)
- Copper (Cu)
- Iron (Fe)
- Lead (Pb)
- Magnesium (Mg)
- Manganese (Mn)
- Mercury (Hg)
- Nickel (Ni)
- Phosphorus (P)
- Potassium (K)
- Selenium (Se)
- Silver (Ag)
- Sodium (Na)
- Strontium (Sr)
- Thallium (Tl)
- Tin (Sn)
- Titanium (Ti)
- Vanadium (V)
- Zinc (Zn)

Mercury Impinger Samples Preparations:

- 4th Impinger (Empty at the start of sampling)

- 4% $KMnO_4$/10% H_2SO_4 Impingers

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- 8N HCl Impinger Rinse

These samples are prepared by mercury (Hg) digestion (Method 29/0060). Analyze a portion of the digestate for Hg by cold vapor atomic absorption (Method 7470).

For each of the metals analyzed, a total multi-metals train content will be reported by summarizing the total ηg in the front half and the total ηg in the back half samples.

Method References:

Method 0060 - "Determination of Metals in Stack Emissions". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 6010 - "Inductively Coupled Plasma - Atomic Emission Spectrometry". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 29 - "Determination of metals emissions from stationary sources", 40 CFR 60 Appendix A.

Method 6020 - "Inductively Coupled Plasma-Mass Spectrometry". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 7470 - "Mercury in Liquid Waste (Manual Cold-Vapor Technique)". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

EPA Method 5, Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.

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THE LEADER IN ENVIRONMENTAL TESTING

Analysis of Metals in Liquid Waste Feeds and Process Samples

Sample Name:	Primary and Secondary Liquid Waste Feeds Makeup Water, and Metals Spiking Solutions
Sample Holding Time:	Analyze within six (6) months of the date of sample collection. Mercury is analyzed within 28 days of sample collection.
Analysis Procedures:	Analyze by CVAA (Method 7470) and by ICAP (Method 6010/6020) for the following metals:

- Aluminum (Al)
- Antimony (Sb)
- Arsenic (As)
- Barium (Ba)
- Beryllium (Be)
- Cadmium (Cd)
- Calcium (Ca)
- Chromium (Cr)
- Cobalt (Co)
- Copper (Cu)
- Iron (Fe)
- Lead (Pb)
- Magnesium (Mg)
- Manganese (Mn)
- Mercury (Hg)
- Nickel (Ni)
- Phosphorus (P)
- Potassium (K)
- Selenium (Se)
- Silver (Ag)
- Sodium (Na)
- Strontium (Sr)
- Thallium (Tl)
- Tin (Sn)
- Titanium (Ti)
- Vanadium (V)
- Zinc (Zn)

Method References:	Method 3010 - "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January

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1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 6010 - "Inductively Coupled Plasma-Atomic Emission Spectrometry". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 6020 - "Inductively Coupled Plasma-Mass Spectrometry". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

Method 7470 - "Mercury in Liquid Waste (Manual Cold-Vapor Technique)". Taken from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846, Third Edition, September 1986. Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IIIA (April 1998). USEPA, OSWER, Washington, D.C. 20460.

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