



**TEST REPORT**

**2019 EMISSIONS COMPLIANCE TEST PROGRAM**

**GREAT LAKES WATER AUTHORITY  
WASTEWATER RESOURCE RECOVERY FACILITY**

**PREPARED FOR:** Great Lakes Water Authority  
9300 W. Jefferson Avenue  
Detroit, Michigan 48209

**CONCERNING:** Compliance Emissions Test  
Seven (7) multiple hearth incinerators  
40 CFR Part 60 Subpart Mmmm

ROP No. MI-ROP-B2103-2014d  
**State Registration No. B2103**

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CK Project No. 5471

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## TEST SUMMARY

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Source Tested: **7 Multiple Hearth Incinerators**

Test Methods: **1, 2, 3A, 4, 6C, 7E, 10, 22, 23, 29, 5/26A**

Renewable Operating Permit: **ROP No. MI-ROP-B2103-2014d**

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Test Dates: **June 18-23, 2019**



## **2.0 PROCESS DESCRIPTION**

GLWA operates a flexible group. The flexible group covers all sewage sludge incinerators subject to the 40 CFR Part 60, Subpart M emissions guidelines through Rule 972. Seven (7) MHIs are included in this group that require testing, they include: EUINC07, EUINC08, EUINC09, EUINC10, EUINC12, EUINC13, EUINC14.

Sludge is dewatered with belt filter presses and conveyed to the multiple hearth furnaces with belt conveyors. The sludge conveyors are equipped with weigh scales for continuous monitoring of the amount of sludge being incinerated. The dewatered sludge is introduced at the top hearth and rabbled down through successive hearths in a spiral path. The moisture in the sludge is evaporated in the upper hearths as hot combustion gases traveling concurrently from the middle hearths where combustion takes place. The maximum feed rate is 3.2 dry tons per hour at 25% solids and 75% volatiles condition. It is a continuous feed process. Under normal operating conditions each incinerator runs between 2.0 and 2.5 dry tons per hour with temperature of the solids between 50 and 80 °F. The furnace is equipped with auxiliary natural gas burners at hearths 2, 4, 6, 8, 10, and 12. The firing rate of the burners is modulated by a central control system to sustain the desired hearth temperatures.

Each air pollution control system is comprised of a Double Zero Hearth afterburner section of Hearths 1 and 2, a quench section, and EnviroCare® Venturi-Pak (venturi throat sections and mist eliminator) scrubber system. The total pressure drop across the wet scrubber ranges between 25 and 40 inches of water column (in. wc). The total scrubber water flow should be greater than 1330 gallons per minute (gpm). Exhaust gases pass through this MHI via an induced draft (ID) fan and exit the scrubber at 100-150 °F.

### **2.1 Process Monitoring**

An appointed facility personnel recorded and monitored key process parameter. The process parameters monitored during each test, at a minimum, consisted of the following:

- Biosolids Feed Rate (wet tons/hr)
- Biosolid Cake Solids (%)
- Biosolids Feed Rate (dry tons/hr)
- Afterburner Exit Temp (°F)
- Total Scrubber Water Flow (gal/min)
- Total Scrubber Pressure Drop (in. wc)
- Scrubber Water Outlet pH



<b>CEMS</b>				
<b>US EPA Method</b>	<b>Pollutant</b>	<b># of Runs</b>	<b>Length of Run</b>	<b>Emission Limit</b>
3A	Oxygen/Carbon Dioxide (O <sub>2</sub> /CO <sub>2</sub> )	3	60 mins	N/A
6C	Sulfur Dioxide (SO <sub>2</sub> )	3	60 mins	26 ppmvd @ 7% O <sub>2</sub>
7E	Nitrogen Oxides (NO <sub>x</sub> )	3	60 mins	220 ppmvd @ 7% O <sub>2</sub>
10	Carbon Monoxide (CO)	3	60 mins	3,800 ppmvd @ 7% O <sub>2</sub>

\*Note: Method 5 and 26A trains were combined.

**Table 3-3**  
Test Matrix – Ash Handling System

<b>US EPA Method</b>	<b>Pollutant</b>	<b># of Runs</b>	<b>Length of Run</b>	<b>Emission Limit</b>
22	Fugitive Emissions	3	60 mins	Visible emissions of combustion ash from an ash conveying system (including conveyor transfer points) for no more than 5% of the hourly observation period

Facility personnel were also responsible for collecting a composite sludge sample during each units' metals and PCDD/PCDF testing. CK sent out one (1) sample for each unit to determine percent solids from collected sludge samples in accordance with Method SM2540B (Standard Methods) and ASTM Method D3684-01, respectively.



**TABLE 3-4 SUMMARY OF RESULTS - EUINC07**

Test Run No.		Run 1	Run 2	Run 3		Facility Permit Limits
Date		06/18/19	06/18/19	06/18/19	Average	
Time	Start	12:42	15:40	18:25		
	Stop	14:54	17:46	20:31		
<b>Sample &amp; Stack Conditions (M5/26A data)</b>						
Flow Rate	dscfm <sup>a</sup>	16,692	16,270	17,322	16,761	
Temperature	°F	72	79	78	76	
Moisture	%	2.6	3.6	3.4	3.2	
<b>Continuous Emissions Monitoring Systems</b>						
Oxygen	%	9.2	10.5	10.9	10.2	
Carbon Dioxide	%	9.7	8.4	7.7	8.6	
Oxides of Nitrogen	PPM	183.3	144.2	148.6	158.7	
	PPM@7% O <sub>2</sub>	217.77	192.73	206.55	205.68	220
	lb/mmBtu	0.341	0.301	0.323	0.322	
Carbon Monoxide	lb/hr	21.93	16.82	18.45	19.07	
	PPM	2126.7	494.1	564.0	1061.6	
	PPM@7% O <sub>2</sub>	2526.6	660.4	784.0	1323.6	3,800
Sulfur Dioxide	lb/mmBtu	2.405	0.629	0.746	1.260	
	lb/hr	154.9	35.1	42.6	77.5	
	PPM	0.1	1.8	1.9	1.3	
Sulfur Dioxide	PPM@7% O <sub>2</sub>	0.07	2.41	2.64	1.71	26
	lb/mmBtu	0.000	0.005	0.006	0.004	
	lb/hr	0.01	0.29	0.33	0.21	
<b>Particulate Matter &amp; Hydrogen Chloride Emissions</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	48.923	46.781	49.726	48.477	
Volume	(dscm) <sup>b</sup>	1.39	1.32	1.41	1.37	
Isokinetics	(%)	99	100	97		
Filterable PM Catch	Front Half (mg)	38.9	42.0	117.1	66.0	
Filterable PM Emission Rate	(mg/dscf)	0.8	0.9	2.4	1.3	
	(mg/dscm@7% O <sub>2</sub> )	26.7	27.0	88.2	47.3	80
	(lb/hr)	1.8	1.9	5.4	3.0	
Hydrogen Chloride Emission Rate	(PPM)	<0.19	<0.20	<0.19	<0.19	
	(PPM @ 7% O <sub>2</sub> )	<0.18	<0.17	<0.20	<0.18	1.2
	(lb/hr)	<0.018	<0.018	<0.018	<0.018	
<b>Trace Metals</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	67.528	67.737	71.261	68.842	
	(dscm) <sup>b</sup>	1.91	1.92	2.02	1.95	
Isokinetics	(%)	97	111	105		
Cadmium (Cd) Catch	(mg)	0.026	0.033	0.033	0.031	
Cd Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.014	0.025	0.020	0.020	0.095
Cd Emission Rate	(lb/hr)	8.90E-04	1.00E-03	1.03E-03	9.74E-04	
Lead (Pb) Catch	(mg)	0.084	0.131	0.166	0.13	
Pb Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.047	0.099	0.101	0.08	0.30
Pb Emission Rate	(lb/hr)	2.92E-03	4.00E-03	5.21E-03	4.04E-03	
Mercury (Hg) Catch	(mg)	0.010	0.108	0.103	0.07	
Hg Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.006	0.081	0.062	0.05	0.28
Hg Emission Rate	(lb/hr)	3.63E-04	3.27E-03	3.23E-03	2.29E-03	
<b>Total Tetra through Octa Dioxins &amp; Furans Emissions</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	85.542	61.665	69.291	72.166	
	(dscm) <sup>b</sup>	2.4	1.7	2.0	2.0	
Isokinetics	(%)	101	101	97		
Total PCDD/PCDF Catch (TMB)	(pg)	11472.9	6104.9	5994.8	7857.5	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm)	4.74	3.50	3.05	3.76	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm@7%O <sub>2</sub> )	5.03	4.19	3.51	4.2	5.0
Total PCDD/PCDF Emission Rate (TMB)	(lb/hr)	3.83E-07	2.03E-07	2.09E-07	2.65E-07	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(pg)	495.1	271.1	284.1	350.1	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(ng/dscm@7%O <sub>2</sub> )	0.217	0.186	0.166	0.19	0.32
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(lb/hr)	1.65E-08	9.01E-09	9.89E-09	1.18E-08	

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**Table 3-6 Summary of Results EUINC09**

Test Run No.		Run 5	Run 2	Run 3	Average	Facility Permit Limits
Date		06/19/19	06/19/19	06/19/19		
Time	Start	8:55	12:30	16:00		
	Stop	11:50	14:58	18:11		
<b>Sample &amp; Stack Conditions (M5/26A data)</b>						
Flow Rate	dscfm <sup>a</sup>	21,108	25,242	17,339	21,230	
Temperature	°F	71	72	72	72	
Moisture	%	2.6	1.2	3.3	2.4	
<b>Continuous Emissions Monitoring Systems</b>						
Oxygen	%	6.8	7.8	7.8	7.5	
Carbon Dioxide	%	12.4	11.7	11.6	11.9	
Oxides of Nitrogen	PPM	182.8	160.6	169.9	171.1	
	PPM@7% O <sub>2</sub>	180.2	170.4	180.3	177.0	220
	lb/mmBtu	0.282	0.266	0.282	0.277	
Carbon Monoxide	lb/hr	27.7	29.1	21.1	25.9	
	PPM	1806.1	428.4	535.7	923.4	
	PPM@7% O <sub>2</sub>	1780.5	454.6	568.4	934.5	3,800
Sulfur Dioxide	lb/mmBtu	1.695	0.433	0.541	0.890	
	lb/hr	166.3	47.2	40.5	84.7	
	PPM	3.3	1.9	1.8	2.3	
	PPM@7% O <sub>2</sub>	3.30	2.02	1.91	2.41	26
	lb/mmBtu	0.007	0.004	0.004	0.005	
	lb/hr	0.70	0.48	0.31	0.50	
<b>Particulate Matter &amp; Hydrogen Chloride Emissions</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	43.237	51.179	37.610	44.009	
Volume	(dscm) <sup>b</sup>	1.22	1.45	1.07	1.25	
Isokinetics	(%)	104	103	110		
Filterable PM Catch	Front Half (mg)	23.0	109.4	127.7	86.7	
Filterable PM Emission Rate	(mg/dscf)	0.5	2.1	3.4	2.0	
	(mg/dscm@7% O <sub>2</sub> )	22.1	77.2	108.2	69.2	80
	(lb/hr)	1.5	7.1	7.8	5.5	
Hydrogen Chloride Emission Rate	(PPM)	0.30	0.59	0.25	0.38	
	(PPM @ 7% O <sub>2</sub> )	0.36	0.61	0.22	0.40	1.2
	(lb/hr)	0.04	0.08	0.02	0.05	
<b>Trace Metals</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	90.458	85.818	77.596	84.624	
	(dscm) <sup>b</sup>	2.56	2.43	2.20	2.40	
Isokinetics	(%)	97	106	107		
Cadmium (Cd) Catch	(mg)	0.019	0.019	0.014	0.017	
Cd Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.008	0.007	0.007	0.007	0.095
Cd Emission Rate	(lb/hr)	6.45E-04	5.96E-04	4.20E-04	5.54E-04	
Lead (Pb) Catch	(mg)	0.062	0.090	0.052	0.068	
Pb Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.025	0.034	0.025	0.028	0.30
Pb Emission Rate	(lb/hr)	2.10E-03	2.85E-03	1.60E-03	2.18E-03	
Mercury (Hg) Catch	(mg)	0.14	0.16	0.14	0.15	
Hg Concentration	(mg/dscm @ 7%O <sub>2</sub> )	0.06	0.06	0.07	0.06	0.28
Hg Emission Rate	(lb/hr)	4.90E-03	4.96E-03	4.18E-03	4.68E-03	
<b>Total Tetra through Octa Dioxins &amp; Furans Emissions</b>						
<i>Sample Conditions</i>						
Volume	(dscf) <sup>a</sup>	84.711	77.142	77.729	79.861	
	(dscm) <sup>b</sup>	2.40	2.18	2.20	2.26	
Isokinetics	(%)	108	103	106		
Total PCDD/PCDF Catch (TMB)	(pg)	1153.3	579.3	564.0	765.5	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm)	0.48	0.27	0.26	0.33	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm@7%O <sub>2</sub> )	0.49	0.24	0.27	0.33	5.0
Total PCDD/PCDF Emission Rate (TMB)	(lb/hr)	3.61E-08	1.89E-08	1.80E-08	2.43E-08	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(pg)	49.0	34.3	32.2	38.5	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(ng/dscm@7%O <sub>2</sub> )	0.021	0.014	0.015	0.017	0.32
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(lb/hr)	1.53E-09	1.12E-09	1.03E-09	1.23E-09	

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**Table 3-8 Summary of Results EUINC12**

Test Run No.		D/F - Run 11	D/F - Run 12	D/F - Run 13		
Date		06/21/19	06/21/19	06/21/19	Average	Facility Permit Limits
Time	Start	10:01	12:20	14:40		
	Stop	12:06	14:23	16:45		
<b>Sample Conditions</b>						
Volume	(dscf) <sup>a</sup>	54.742	46.882	45.558	49.061	
	(dscm) <sup>b</sup>	1.550	1.328	1.290	1.389	
Isokinetics	(%)	109.7	100.9	101.6		
<b>Stack Conditions</b>						
Flow Rate	(dscfm) <sup>c</sup>	12,449	12,296	11,047	11,931	
Temperature	(°F)	66.1	77.0	66.6	69.9	
Moisture	(%)	1.8	0.5	2.0	1.4	
Oxygen	(%)	10.4	8.7	9.1	9.4	
Carbon Dioxide	(%)	9.0	10.5	10.4	10.0	
<b>Total Tetra through Octa Dioxins &amp; Furans Emissions</b>						
Total PCDD/PCDF Catch (TMB)	(pg)	1298.3	1110.1	274.9	894.4	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm)	0.837	0.836	0.213	0.629	
Total PCDD/PCDF Concentration (TMB)	(ng/dscm@7%O <sub>2</sub> )	1.109	0.953	0.251	0.771	5.0
Total PCDD/PCDF Emission Rate (TMB)	(lb/hr)	3.90E-08	3.85E-08	8.81E-09	2.88E-08	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(pg)	78.2	54.4	26.6	53.1	
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(ng/dscm@7%O <sub>2</sub> )	0.0668	0.0467	0.0243	0.0459	0.32
Total PCDD/PCDF Concentration TEQ (EPA TEF)	(lb/hr)	2.35E-09	1.89E-09	8.53E-10	1.70E-09	

- a) dry standard cubic feet
- b) dry standard cubic meters
- c) dry standard cubic feet per minute

**Table 3-9 Summary of Results EUINC13**

Test Run No.		Run 1	Run 2	Run 3	Averages	Facility Permit Limits
Date		06/22/19	06/22/19	06/22/19		
Time		15:04 - 16:04	16:04 - 17:04	17:04 - 18:04		
<b>Continuous Emissions Monitoring Systems</b>						
Oxygen	%	4.7	3.7	4.4	4.3	
Carbon Dioxide	%	13.6	14.8	14.2	14.2	
Oxides of Nitrogen	PPM	142.9	109.0	101.0	117.6	
	PPM@7% O <sub>2</sub>	122.61	88.09	85.08	98.59	220
	lb/mmBtu	0.1917	0.1377	0.1330	0.1542	





#### 4.0 SAMPLING LOCATION

All MHI sampling locations are identical. Outlet flue gas sampling was performed at a location that is between the scrubber exhaust and induced draft fan. The inside diameter of the exhaust duct is 54 inches. Two test ports, spaced 90° apart, are located 120 inches (2.2 duct diameters) to the nearest upstream disturbance and 108 inches (2.0 duct diameters) to the nearest downstream disturbance. In accordance with EPA Method 1, twenty-four (24) traverse points (12 per port) were used for isokinetic sampling and volumetric flowrate determinations. Continuous emissions monitoring (CEM) was conducted through a single port that is located adjacent to the GLWA total hydrocarbons (THC) sampling probe (same elevation). Prior to the start of the continuous emissions monitoring (CEM) a three-point stratification check was performed at the following traverse points (9", 27", and 45"). All measurements were verified on-site.



## US EPA Method 29

## Determination of Metals Emissions from Stationary Sources

The following sections describe the sampling and analytical methodologies utilized during the emissions compliance testing. Reduced data is contained in Appendix A, field data sheets are included in Appendix B. Supporting documentation for the results presented are contained in C, D, E and F.

### 5.1 Test Methods

#### 5.1.1 Volumetric Flow Rate and Moisture – EPA Method 1, 2 & 4

The exhaust gas flow rate and moisture content were measured using EPA Methods 1 through 4. These measurements included the determination of the proper number of traverse points and their locations in the stack (RM1), stack velocity and volumetric flow rate (RM2), stack gas molecular weight (RM3) and stack gas moisture content (RM4).

A S-type Pitot tube, inclined manometer and K-type thermocouple were used for the velocity pressure and temperature measurements. The Pitot tube meets the criteria of EPA Method 2 and was assigned a coefficient of 0.84. Velocity pressure and temperature readings were taken and recorded at each of the traverse points in the exhaust stack.

The moisture content was determined in conjunction with the Method 5 and 202 sampling trains. The trains consisted of a series of impingers and applicable sampling reagents. The impingers were housed in an impinger bucket filled with water and ice to assure that the moisture in the stack gas condenses out. The impingers and their contents were weighed before and after testing. The last impinger contained a known quantity of silica gel to capture the remaining moisture from the gas stream. The resultant net weight gain of the impinger train was used to calculate the moisture content of the stack gas. A calibration check with certified weight was performed on the field balance and was noted on the first moisture run.

#### 5.1.2 Oxygen/Carbon Dioxide – EPA Method 3A

Oxygen and carbon dioxide were measured in accordance with EPA Method 3A. This Method utilizes continuous emissions monitoring instrumentation. CK Environmental uses a Teledyne Model 326A oxygen analyzer with a range of 0-25% oxygen and a California Analytical Instruments Model ZRH non-dispersive infrared carbon dioxide analyzer with a range of 0-20% carbon dioxide. The instruments meet all of the performance specifications of the Method. They were calibrated before and after each test period using low, mid or high calibration gases prepared according to EPA Protocol. Sampling occurred simultaneously with flow measurements in order to obtain volumetric flow data for mass emission calculations.



### 5.1.7 Multiple Metals – EPA Methods 29

Metals emissions were determined according to procedures outlined in the EPA Multi-Metals Procedure - 40 CFR 60, EPA Method 29. Emissions of mercury, cadmium, and lead were quantified in accordance with the method. Sampling was conducted isokinetically for a period of 120 minutes per run, collecting a minimum of 60 dry standard cubic feet. The following is a description of the sampling train and the procedures used to quantify multi-metals during the emissions compliance testing.

The multi-metals sampling train consists of a glass button hook nozzle, a heated glass lined sample probe, a quartz fiber filter in a holder in an oven box, a set of seven tared glass impingers connected in series in an ice bath, and a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter. A Teflon® lined fitting connects the nozzle to the probe liner. A glass cyclone bypass connects the sampling probe to the filter holder. All of the sampling train glassware undergoes the cleaning and nitric acid soaking procedure described in US EPA Method 29 prior to testing. Silicone grease is not used as a sealant on the ground glass fittings, to prevent potential sample contamination.

The sample probe and oven box are maintained at a temperature of 248±25°F during sampling to prevent moisture condensation. The first impinger is initially empty. The second and third impingers each contain 100 ml of 5% nitric acid / 10% hydrogen peroxide (5% $\text{HNO}_3$ /10% $\text{H}_2\text{O}_2$ ). The fourth impinger will be initially empty. The fifth and sixth impingers will each contain 100 ml of 4% potassium permanganate / 10% sulfuric acid (4% $\text{KMnO}_4$ /10% $\text{H}_2\text{SO}_4$ ). The acidic permanganate solution will be prepared fresh on-site daily, in accordance with US EPA Method 29. The seventh impinger contains a known quantity of indicating silica gel. The third impinger will be a Greenburg-Smith impinger with a standard tip, while the other impingers have modified tips. The temperature at the outlet of the seventh impinger is maintained below 68°F during sampling by adding ice to the ice bath. A vacuum line connects the outlet of the seventh impinger to the control module.

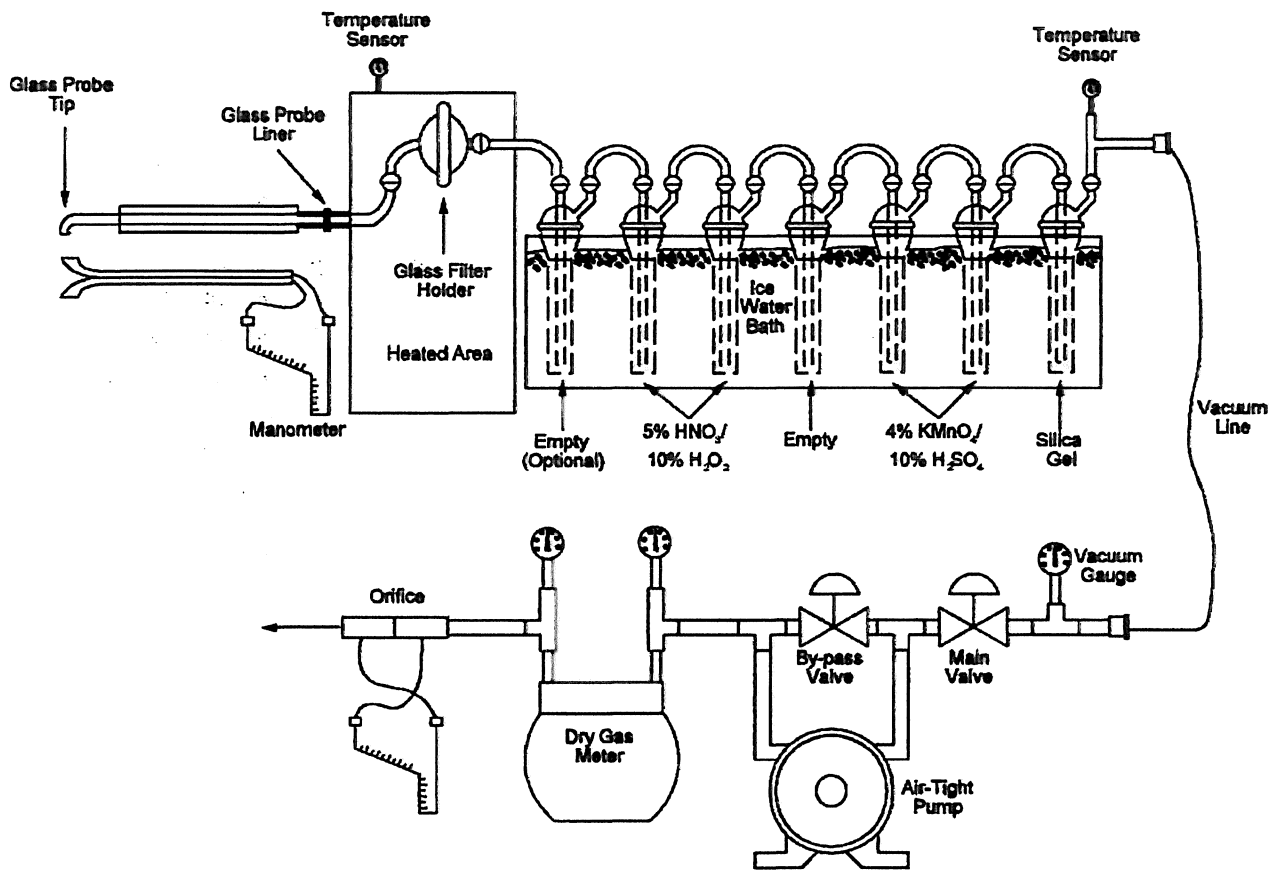
Before each test, the sampling train is leak checked to ensure a leakage rate no greater than 0.02 cfm at 15 in. Hg sample vacuum. The probe is then placed in the stack and stack gas is withdrawn isokinetically for an equal period of time at each traverse point. The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter temperature, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum are recorded at five-minute intervals during sampling. Before port changes and at the completion of each test, the sampling train is leak checked to ensure a leakage rate no greater than 0.02 cfm at the highest recorded test vacuum.



The silica gel impinger is weighed for moisture gain. The silica gel is then returned to its original storage container to be dried for reuse.

The multi-metals samples are submitted to the CK sub-contract laboratory, Maxxam Analytics Inc., for analysis. Containers 1 through 4 are digested in concentrated acid solutions before being analyzed for the target metals by inductively coupled argon plasma emission spectroscopy (ICAP) or graphite furnace atomic absorption spectroscopy (GFAAS) if lower detection limits are required. All samples are labeled, logged and stored in a cool, dark area until delivery to the laboratory. A set of reagent blanks are also taken for analysis along with the samples.

**Figure 5-1**  
Method 29 Sampling Train





Prior to sampling the train is leak checked at a vacuum of -15" Hg to ensure that there is a leak rate of less than 0.02 cfm. The train is operated in the same manner as an EPA Method 5 train for a period of 2 hours per run.

Following sampling, the train is disassembled and sealed with hexane-rinsed foil. Once in the field lab, the train components are recovered in four separate fractions: 1) front half rinse, 2) filter, 3) filter holder back half and condenser coil rinse and 4) sorbent trap. Fractions 1 and 3 components are rinsed three times with HPLC-grade acetone and methylene chloride (Container 2). The connecting line between the filter and the condenser is rinsed three times with acetone. Additionally, the condenser is soaked with three separate portions of methylene chloride for 5 minutes each. These soakings are added to Container 2. Fractions 1 and 3 components are rinsed again three times with HPLC-grade toluene (QA/QC rinse, Container 3). Additionally, the condenser is soaked with three separate portions of toluene for 5 minutes each. These soakings are added to Container 3. The QA/QC toluene rinses are kept separate until final analysis when they are combined with other fractions.

Following recovery, the samples are sealed, labeled and stored in a cooler or refrigerator until shipment to the analytical laboratory. The samples are overnight shipped to the laboratory in coolers containing a sufficient number of freezer packs to insure that the sample temperatures do not exceed 4°C.

All samples are extracted, combined and analyzed for Polychlorinated Dibenzo-p-Dioxins and Dibenzofurans and their congeners by High-Resolution Chromatography / High Resolution Mass Spectrometry. Maxxam Analytics, Mississauga, Ontario, Canada, perform the analysis in accordance with the Method. The samples are analyzed with a gas chromatograph coupled to a mass spectrometer (GC/MS) using the instrumental parameters specified in the Method. Immediately prior to analysis a 20 µl aliquot of the Recovery Standard solution is added to each sample. A 2 µl aliquot of the extract is injected into the GC. Sample extracts are first analyzed using a DB-5 capillary column to determine the concentration of each isomer of PCDD's and PCDF's (tetra-through octa-). If tetra-chlorinated dibenzofurans are detected in this analysis, another aliquot of the sample is analyzed in a separate run, using the DB-225 column to measure the 2,3,7,8 tetra-chloro dibenzofuran isomer.

A group of nine carbon labeled PCDD's and PCDF's representing, the tetra-through octa chlorinated homologues, is added to every sample prior to extraction. The role of the internal standards is to quantify the native PCDD's and PCDF's present in the sample as well as to determine the overall method efficiency. Recoveries of the internal standards must be between 40 to 130 percent for the tetra-through hexa- chlorinated compounds while the range is 25 to 130 percent for the higher hepta- and octa- chlorinated homologues.

Five surrogate compounds are added to the resin in the adsorbent sampling cartridge before the sample is collected. The surrogate recoveries are measured relative to the internal standards and are a measure of collection efficiency. They are not used to measure native PCDD's and PCDF's. All

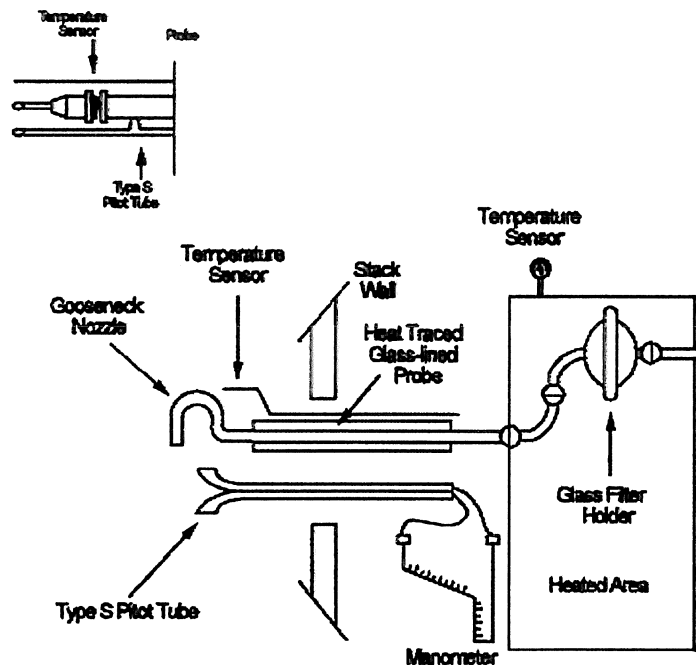


Prior to sampling, the isokinetic correlation is established, the train is carefully assembled and leak checked. After the probe and filter compartment reach the desired operating temperature ( $248^{\circ}\text{F} \pm 25^{\circ}$ ), the probe is placed in the stack and isokinetic sampling takes place.

At the completion of isokinetic sampling, the train is leak checked for leaks, disassembled, and sealed. All train recovery procedures are conducted in accordance with EPA Method 5. The filter is carefully removed from the filter holder and placed in a sample label identified petri dish. The nozzle, probe and the front portion of the filter holder are thoroughly brushed and rinsed with acetone and collected in a container labeled for sample identification. Sample volumes are noted and liquid levels marked. An acetone field blank is also taken for analysis along with the samples.

The samples are analyzed gravimetrically by Maxxam Analytical in accordance with the method. The acetone rinses are evaporated to dryness in tared beakers. All filters and beakers are desiccated before and after sampling for 24 hours, and weighed at 6-hour intervals until two consecutive weights are within  $\pm 0.5$  mg.

**Figure 5-3**  
Method 5 Front Half Filterable PM Set-up





**Preliminary Traverse and Cyclonic Flow Check-** A preliminary traverse is conducted. Readings include the velocity pressure, angle of flow, gas temperature and static pressure. The average angle of flow is used to determine whether the exhaust gas is considered “cyclonic” ( $\geq 20^\circ$ ).

**Stratification Check-** Before any gaseous reference method test runs are performed, a stratification check is conducted to ensure that there is no stratification at the sampling location. Stratification is defined as a difference in excess of 10 percent between the average concentration of the stack and the concentration at any other point. Once the traverse is completed, each point is checked to see if it is less than or equal to 5% of the average of all the points, or 0.5ppm NOx.

**Static Pressure** - The static pressure of the stack is checked and recorded.

**Nomograph** - Once the above information is obtained, the nomograph for the actual test is set up to correlate the isokinetic relationships.

**Barometric Pressure** - Barometric pressure is obtained from the Weather Channel application

**Sampling Train Set-Up -**

- (a) The filter is placed in the filter holder and visually checked. Filter number and tare weights are recorded on the field data sheets.
- (b) The impingers are loaded with the appropriate solution and volumes are recorded on the field data sheets.
- (c) Approximately 200 grams of silica gel are placed in the final impinger. Exact weights are logged on the field data sheets.
- (d) Crushed ice is placed around the impingers.
- (e) Once the entire train is assembled, the probe and filter compartment heaters are turned on.

**Pre-Test Leak Check** - Once the filter compartment heater is at the desired temperature for testing, the system is leak checked at fifteen inches of vacuum (15"Hg). A leak rate of less than 0.02 CFM must be achieved prior to the start of sampling.

**Final Check** – When sampling is ready to commence, facility operations are checked to confirm that the process is operating at the desired capacity.

**Sampling** - Isokinetic sampling, per the Reference Method takes place. Sample gas is extracted isokinetically at each traverse point. The sample rate is established according to the velocity pressure and temperature of measured at the sample point. Traverse points are sampled for equal periods over the course of the required test run time.



**Calibration Valve** - A t-valve, located at the base of the probe allows the operator to select either the sample stream or introduce calibration gas to the system.

**Sample Distribution System** - A series of flow meters, valves and backpressure regulators allows the operator to maintain constant flow and pressure conditions during sampling and calibration.

**Gas Analyzers** - capable of the continuous determination of O<sub>2</sub>, CO<sub>2</sub>, SO<sub>2</sub>, CO, and NO<sub>x</sub> concentrations in a sample gas stream. They each meet or exceed the following specifications:

**Calibration Error** - Less than +2% of span for the zero, mid-and hi-range calibration gases

**System Bias** - Less than  $\pm 5\%$  of span for the zero, mid- or hi-range calibration gases.

**Zero Drift** - Less than +3% of span over the period of each test run.

**Calibration Drift** - Less than +3% of span over the period of each test run.

**Data Acquisition System** - A Monarch Model 4600, or equivalent, data logger system is used to record analyzer response to the sample and calibration gas streams. The data logger records at 15-second intervals and the data used to report test interval averages. The Monarch saves data to a compact flash drive that is downloaded to a computer. Separate files for each test run and associated calibrations are generated and saved. Data is loaded into a Microsoft Excel® spreadsheet for calculation of test interval average concentrations and emission rates.





**Determination of Sampling System Bias** - Sampling system bias is determined immediately prior to testing in accordance with EPA Methods 3A, 6C, 7E, 10.

**Determination of Zero and Calibration Drift** - Before and after each test run, each analyzer's response to zero and mid- or hi-range calibration gases are determined. The pre-and post-test analyzer responses are compared to determine drift. The results are evaluated based upon specifications defined in EPA Methods 3A, 6C, 7E, 10.

**NO<sub>2</sub> to NO Converter Check**- A NO<sub>2</sub> to NO converter check is conducted on the NO<sub>x</sub> analyzer in accordance with Section 8.2.4 of Method 7E. A calibration gas of ≈50 ppm NO<sub>2</sub> is introduced into the analyzer in direct calibration mode. The NO<sub>x</sub> concentration measured by the analyzer is recorded and the conversion efficiency calculated using equation 7E-7 in Method 7E. The converter check is acceptable if the calculated converter efficiency is between 90 and 110%.

**Data Reduction** - An average pollutant/diluent concentration for each test time interval is determined from the data acquisition system. This data is then reduced to determine relative pollutant concentrations in units of ppm and mass, lb/hr.