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# Comprehensive Emissions Test Report

Port Inland Plant PM, PM-10, Opacity Compliance Testing

Testing Date(s): August 16 and 17, 2022 Report Date: September 27, 2022 Revision Date: No revision to date

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Pace® Project No. 22-06155A



#### Subject Facility:

Graymont Western Lime Port Inland Plant 181 W County Road 432 Gulliver, MI 49840

#### Regulatory Permit No .:

ROP: MI-ROP-N7362-2020

SRN: N7362

#### Subject Emission Sources:

Lime Kiln

Kiln 1

Processed Stone Handling

#### **Test Locations:**

Baghouse Exhaust

SV-2

N7362\_test\_20220816

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### **Regulatory Summary**

Subject Facility:

**Graymont Western Lime** 

Port Inland Plant

Plant Address:

181 W County Road 432

Gulliver, MI 49840

Air Permit No.:

ROP: MI-ROP-N7362-2020

Facility ID No.:

SRN: N7362

Emission Unit Name	Regulated Constituent	Regulatory Citations	Regulatory Limit	Average Test Result	
		Particulate	40 CFR 63.7090(a)	≤0.10 LB/Ton Stone Feed (TSF)	0.0475 LB/TSF
Lime Kiln	watter		≤0.05 grams/dscm	0.0175 grams/dscm	
	PM-10 <sup>2</sup>	R 336 1205	≤7.5 LB/HR	7.85 LB/HR	
		PM-10 <sup>2</sup> 40 CFR 52.21(j)	≤0.10 LB/TSF	0.125 LB/TSF	
Screen Enclosure – South Wall Screen Enclosure – East Wall Screen Enclosure – West Wall Screen Enclosure – North Wall C118 Conveyor C119 Bin	Opacity	40 CFR 63.7090(a)	There must be no visible emissions from the building, except from the vent. Emissions from the vent must have a sixminute average of ≤10%.	0%	
	Screen Enclosure – South Wall Screen Enclosure – East Wall Screen Enclosure – West Wall Screen Enclosure – West Wall Conveyor	Unit Name  Constituent  Particulate Matter¹  Lime Kiln  PM-10²  Screen Enclosure – South Wall Screen Enclosure – Heast Wall Screen Enclosure – West Wall Screen Enclosure – West Wall Conveyor C119 Bin  Constituent  Particulate Matter¹  PM-10²  PM-10²  Opacity	Unit Name  Particulate Matter¹  PM-10²  R 336.1205 40 CFR 52.21(j)  Screen Enclosure – South Wall Screen Enclosure – East Wall Screen Enclosure – West Wall Screen Enclosure – North Wall C118 Conveyor C119 Bin  Citations  40 CFR 63.7090(a)  40 CFR 63.7090(a)	Unit Name     Constituent     Citations     Limit       Lime Kiln     Particulate Matter¹     40 CFR 63.7090(a)     ≤0.10 LB/Ton Stone Feed (TSF)       Storeen Enclosure – South Wall Screen Enclosure – East Wall Screen Enclosure – West Wall Screen Enclosure – West Wall Screen Enclosure – North Wall C118 Conveyor C119 Bin     A0 CFR 63.7090(a)     ≤7.5 LB/HR       Streen Enclosure – North Wall C118 Conveyor C119 Bin     A0 CFR 63.7090(a)     There must be no visible emissions from the building, except from the vent. Emissions from the vent must have a sixminute average of ≤10%.	

<sup>&</sup>lt;sup>1</sup>Filterable non-condensable particulate matter (EPA Method 5).

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<sup>&</sup>lt;sup>2</sup>Filterable + condensable particulate matter (EPA Method 201A/202).

### Introduction

Pace<sup>®</sup> Analytical Services personnel conducted particulate, PM-10, and visible emission compliance testing on the Lime Kiln (Kiln 1) Baghouse Exhaust at the Graymont Western Lime facility located in Gulliver, Michigan. Matt McDermott, Andrew Radabaugh, and Lucas Ruhland performed on-site testing activities on August 16 and 17, 2022. Tom Rehling provided administrative project management. Hal Lee and Steve White with Graymont Western Lime coordinated plant activities during testing. Pace<sup>®</sup> Analytical Services prepared a comprehensive test protocol that was submitted to the Michigan Department of Environment, Great Lakes & Energy (EGLE) and approved prior to testing. Joseph Scanlan and Dan Droste with EGLE were on-site to witness testing. On-site activities consisted of the following measurements:

- Particulate (EPA Method 5/202), three independent 90-minute samplings.
- PM-10 (EPA Method 201A/202), three independent 90 to 93-minute samplings.
- Gas composition (O<sub>2</sub>/CO<sub>2</sub>), integrated bags collected concurrent with above.
- Volumetric airflow, measurements collected in conjunction with isokinetic testing.
- Visible emissions, one independent one-hour monitoring period.

The project objectives were to quantify particulate, PM-10, and visible emission constituents and compare them to applicable air emissions regulations stipulated by EGLE, the facility permit, and 40 CFR Part 63 Subpart AAAAA. Process conditions during testing were recorded by Graymont Western Lime personnel and are included in Appendix E. Quality protocols comply with regulatory compliance testing requirements.

Subsequent sections summarize the test results and provide descriptions of the process and test methods. Supporting information and raw data are in the appendices.

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Results Summary

Results of particulate determinations are summarized in Table 1. The filterable non-

condensable particulate emission rate averaged 2.99 LB/HR and 0.0475 LB/TSF at

0.0175 grams/dscm. The filterable non-condensable particulate emission limits for this

source are 0.10 LB/TSF and 0.05 grams/dscm.

Results of PM-10 determinations are summarized in Table 2. The PM-10 emission rate

averaged 7.85 LB/HR and 0.125 LB/TSF at 0.0481 grams/dscm. The PM-10 emission

limits for this source are 7.5 LB/HR and 0.10 LB/TSF. Subsequent tables provide

expanded detail of the testing results.

The particulate dry catch (EPA Method 5) was used to report filterable non-condensable

particulate matter. PM-10 was collected from an EPA Method 201A/202 train. The <10

µm filterable catch (EPA Method 201A) was combined with the organic wet catch and

inorganic wet catch to report PM-10.

The data in this report are indicative of emission characteristics of the measured

sources for process conditions at the time of the test. Representations to other sources

and test conditions are beyond the scope of this report.

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## **Summary Tables**

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Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 1
Results Summary (M5/202)
Lime Kiln (Kiln 1) Baghouse Exhaust
Test 1

Parameter Date of Run Time of Run	Run 1 8/17/22 0750-0928	Run 2 8/17/22 1105-1211	Run 3 8/17/22 1405-1542	Average
Limestone Feed Rate, TPH	60.9	63.3	64.1	62.8
Volumetric Flow Rate ACFM DSCFM	77,600 44,500	79,300 45,600	79,600 46,300	78,800 45,500
Gas Temperature, °F Gas Moisture Content, %v/v	382 7.1	382 6.9	368 7.3	377 7.1
Particulate Mass Rate, LB/HR Filterable Particulate	2.57	3.38	3.01	2.99
Total Particulate (PM-10 Eq.)	7.23	8.26	8.28	7.92
Particulate Concentration, GR/DSCF Filterable Particulate	0.00674	0.00866	0.00757	0.00766
Total Particulate (PM-10 Eq.)	0.0189	0.0211	0.0209	0.0203
Regulatory Units, LB/Ton Stone Feed Filterable Particulate	0.0422	0.0534	0.0469	0.0475
Total Particulate (PM-10 Eq.)	0.119	0.130	0.129	0.126
Particulate Concentration, grams/dscm Filterable Particulate	0.0154	0.0198	0.0173	0.0175
Total Particulate (PM-10 Eq.)	0.0433	0.0483	0.0478	0.0465

Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 2
Results Summary (M201A/202)
Lime Kiln (Kiln 1) Baghouse Exhaust
Test 1

Parameter Date of Run Time of Run	<b>Run 1</b> 8/17/22 0750-0928	<b>Run 2</b> 8/17/22 1105-1246	Run 3 8/17/22 1405-1543	Average
Limestone Feed Rate, TPH	60.9	63.3	64.1	62.8
Volumetric Flow Rate (Rounded to 100 CFM) ACFM DSCFM	75,700 43,200	75,700 43,800	75,400 43,800	75,600 43,600
Gas Temperature, °F Gas Moisture Content, %v/v	383 7.4	382 6.3	371 7.2	379 7.0
Gas Composition, %v/v, dry Carbon Dioxide, CO <sub>2</sub> Oxygen, O <sub>2</sub> Nitrogen, N <sub>2</sub> (by difference)	25.8 7.4 66.8	25.8 7.6 66.6	26.0 7.9 66.1	25.9 7.6 66.5
Particulate Concentration, GR/DSCF < 10 μm Filterable PM < 10 μm Particulate Matter	0.00570 0.0227	0.00817 0.0187	0.00846 0.0216	0.00744 0.0210
Particulate Mass Rate, LB/HR < 10 μm Filterable PM < 10 μm Particulate Matter	2.11 8.40	3.07 7.04	3.18 8.10	2.78 7.85
Regulatory Units, LB/Ton Stone Feed < 10 μm Filterable PM < 10 μm Particulate Matter	0.0346 0.138	0.0485 0.111	0.0495 0.126	0.0442 0.125
Particulate Concentration, grams/dscm < 10 μm Filterable PM < 10 μm Particulate Matter	0.0130 0.0519	0.0187 0.0429	0.0194 0.0494	0.0170 0.0481

### **Detail Tables**

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Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 3
Major Gases and Moisture Results (M5/202)
Lime Kiln (Kiln 1) Baghouse Exhaust
Test 1

Parameter	Run 1	Run 2	Run 3
Date of Run	8/17/22	8/17/22	8/17/22
Time of Run	0750-0928	1105-1211	1405-1542
Major Gas Constituents - Instrumental, % v/v Dry Basis (as measured) Carbon Dioxide Oxygen Nitrogen (by difference)	25.80 7.40 66.80	25.80 7.60 66.60	26.00 7.90 66.10
Wet Basis (calculated)			
Carbon Dioxide	23.96	24.02	24.10
Oxygen	6.87	7.07	7.32
Nitrogen	62.03	62.00	61.28
Maragan	02.00	02.00	01.20
Portable Oxygen Monitor Result			
Time Weighted Average, %O <sub>2</sub>	6.8	6.6	6.7
Moisture Collected, ml	96.4	98.6	106.7
Moisture Content, %v/v	7.15	6.91	7.29
Moisture Content if Saturated, %v/v	NA (>RP)	NA (>BP)	NA (>BP)
Relative Humidity, % rH	NA (>BP)	, ,	, ,
relative Furnicity, 70 HT	IVA (-DI)	INA (-DI)	IVA (FDI)
Molecular Weight of Flue Gas, lb/lb-mole			
Dry	32.42	32.43	32.48
Wet	31.39	31.43	31.42

Table 4

Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Major Gases and Moisture Results (M201A/202) Lime Kiln (Kiln 1) Baghouse Exhaust Test 1

Parameter Date of Run Time of Run	Run 1 8/17/22 0750-0928	Run 2 8/17/22 1105-1246	Run 3 8/17/22 1405-1543
Major Gas Constituents - Instrumental, % v/v Dry Basis (as measured)	05.00	25.00	20.00
Carbon Dioxide Oxygen Nitrogen (by difference)	25.80 7.40 66.80		26.00 7.90 66.10
Wet Basis (calculated) Carbon Dioxide Oxygen Nitrogen	23.88 6.85 61.84	7.12	24.13 7.33 61.35
Portable Oxygen Monitor Result Time Weighted Average, %O <sub>2</sub>	6.5	6.6	6.7
Moisture Collected, ml	59.5	48.9	55.8
Moisture Content, %v/v	7.43	6.35	7.19
Moisture Content if Saturated, %v/v Relative Humidity, % rH	NA (T>BP) NA (T>BP)	'	, ,
Molecular Weight of Flue Gas, lb/lb-mole Dry Wet	32.42 31.35	32.43 31.52	32.48 31.44

NA (T>BP) = Not applicable, gas temperature is greater than boiling point of water (supersaturation is possible).

Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 5
Particulate Results (M5/202)
Lime Kiln (Kiln 1) Baghouse Exhaust
Test 1

		-	-
Parameter	Run 1	Run 2	Run 3
Date of Run	8/17/22 0750-0928	8/17/22	8/17/22
Time of Run	90	90	90
Sample Duration, Minutes	90	90	90
Average Flue Gas Temperature, °F	381.8	381.8	368.4
Moisture Content of Flue Gas, %v/v	7.1	6.9	7.3
Particulate Collected, mg	25.7	35.1	24.2
Dry Catch	25.7 45.9	35.1 49.2	31.3
Inorganic Wet Catch	45.9	49.2 1.4	54.3 0.7
Organic Wet Catch	0.7	1.4	0.7
Volumetric Flow Rate (Rounded to 100 CFM)			
ACFM	77,600	79,300	79,600
SCFM	48,000	49,000	50,000
DSCFM	44,500	45,600	46,300
Sample Volume, Meter Conditions, Ft <sup>3</sup>	60.70	64.83	66.94
Sample Volume, Dry Standard, Ft <sup>3</sup>	58.95	62.51	63.83
Particulate Concentration, GR/DSCF			
Filterable Particulate	0.00674	0.00866	0.00757
Inorganic Condensables	0.0120	0.0121	0.0131
Organic Condensables	0.000181	0.000338	0.000164
Total Particulate (PM-10 Eq.) (F+I+O)	0.0189	0.0211	0.0209
rotal rantediate (rivi-ro Eq.)	0.0100	0.02.11	0.0200
Particulate Emission Rate, LB/HR			
Filterable Particulate	2.57	3.38	3.01
Inorganic Condensables	4.59	4.74	5.21
Organic Condensables	0.0690	0.132	0.0653
Total Particulate (PM-10 Eq.) (F+I+O)	7.23	8.26	8.28

NR=Not required or not requested.

Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 6
Particulate Results (M201A/202)
Lime Kiln (Kiln 1) Baghouse Exhaust
Test 1

Parameter	Run 1	Run 2	Run 3
Date of Run	8/17/22	8/17/22	8/17/22
Time of Run	0750-0928	1105-1246	1405-1543
Sample Duration, Minutes	93.0	90.5	90.0
Average Flue Gas Temperature, °F	383.4	381.6	370.9
Moisture Content of Flue Gas, %v/v	7.4	6.3	7.2
Volumetric Flow Rate (Rounded to 100 CFM)			
ACFM	75,700	75,700	75,400
SCFM	46,700	46,800	47,200
DSCFM	43,200	43,800	43,800
Particulate Collected, mg Blank Corrected			
PM <sub>10</sub> Cyclone - >10 μm Filterable	NTP	NTP	NTP
PM <sub>2.5</sub> Cyclone - 2.5 - 10 µm Filterable	10.0	14.5	15.2
Filter Catch - <2.5 µm Filterable	2.9	3.4	3.4
CPM <sub>ORG</sub> - Organic Condensible	1.60	0.883	0.943
CPM <sub>INORG</sub> - Inorganic Condensible	36.8	22.4	27.9
mond 3			
Actual PM10 Cut Diameter, μm	10.2	10.3	10.2
Actual PM2.5 Cut Diameter, µm	2.33	2.36	2.31
·			
Particulate Concentration, GR/DSCF			
< 10 μm Filterable PM	0.00570	0.00817	0.00846
Organic Condensible PM	0.000709	0.000401	0.000429
Inorganic Condensible PM	0.0163	0.0102	0.0127
Combined Condensible PM	0.0170	0.0106	0.0131
Particulate Emission Rate, LB/HR			
< 10 µm Filterable PM	2.11	3.07	3.18
Organic Condensible PM	0.263	0.151	0.161
Inorganic Condensible PM	6.03	3.82	4.76
Combined Condensible PM	6.29	3.97	4.92
	J.m. J		

NTP = Non-Target Parameter, intentionally excluded from the test protocol.

Port Inland Plant Gulliver, MI Pace Project No. 22-06155A Table 7

Opacity Observations Lime Kiln (Kiln 1) Baghouse Exhaust

Test 1

Percei	nt Opacity	Optical Dens	ity Rela	tive Frequency
	0	0.000		100.00
	5	0.022		0.00
	10	0.046		0.00
	15	0.071		0.00
	20	0.097		0.00
	25	0.125		0.00
	30	0.155		0.00
	35	0.187		0.00
	40	0.222		0.00
	45	0.260		0.00
	50	0.301		0.00
	55	0.347		0.00
	60	0.398		0.00
	65	0.456		0.00
	70	0.523		0.00
	75	0.602		0.00
	80	0.699		0.00
	85	0.824		0.00
	90	1.000		0.00
	95	1.301		0.00
	99	2.000	<u> </u>	0.00
Average >	0.0	0.000	Total >	100

Average Opa	icity Per Sequ	uential Six M	inute Period:	High Six Minute Average	e: 0.0
<u>Period</u>	<b>Opacity</b>	<u>Period</u>	<b>Opacity</b>	Maximum reading:	0.0
1	0.0	6	0.0	Minumum reading:	0.0
2	0.0	7	0.0		
3	0.0	8	0.0	Observer: Matt McDe	rmott
4	0.0	9		Date of test: 8/16/2022	
5	0.0	10	0.0	Time of test: 1315-1414	

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NOTE: The high six-minute average opacity is the maximum value for any consecu**ng to the part of the p** 

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**Process Description** 

Graymont Western Lime operates a rotary lime kiln near Gulliver, Michigan. The operations at this facility are subject to the requirements of air quality operating permit MI-ROP-N7362-2020, issued November 19, 2020. The plant has a maximum lime production rate of 870 tons per day (TPD) and 292,000 tons of lime production per year.

A rotary kiln is a long, cylindrical, refractory-lined furnace that is slightly inclined. The limestone and hot gases pass counter-currently through the kiln. The lime plant consists of a single 235-foot long rotary kiln with a pre-heater and lime cooler. The kiln is fired with coal or a mixture of coal and petroleum coke. Coal and/or petroleum coke is burned near the discharge end of the kiln to provide the necessary heat for the process. The kiln rotates continuously to prevent the drum from sagging, to improve the product contact with the hot gases, and to move the product through the kiln. To maximize fuel efficiency, a product cooler and limestone pre-heater are used to recover heat from the product and the hot gasses. The lime product is discharged from the kiln and then conveyed to various storage silos, where it is screened to size and then shipped to the end user. Lime is used in the metallurgical, pulp and paper, construction, and waste treatment industries.

Emissions from the process consist primarily of particulate matter (PM), carbon monoxide (CO), nitrogen oxides (NO<sub>x</sub>), and sulfur dioxide (SO<sub>2</sub>) from fuel combustion. Emission controls for the kiln consist of a fabric filter baghouse for PM control, a fuel sulfur content limit and combustion optimization to reduce CO and NO<sub>x</sub> emissions. The majority of the SO<sub>2</sub> is collected within the process, owing to reactions with calcium oxide in the kiln.

Test related process and operational details were collected by Graymont Western Lime personnel and included in Appendix E.

#### **Test Procedures**

EPA Method 1 specifies test location acceptability criteria and defines the minimum number of traverse points for representative sampling. Linear measurements from upstream and downstream flow disturbances and the duct equivalent diameter are compared and the distances related to number of diameters. A flow disturbance can be defined as anything that changes or upsets the direction of flow within the duct including bends, dampers, fans, shape or size transitions, and open flames. Method 1 stipulates that test ports should be located at least eight diameters downstream and two diameters upstream of any flow disturbance. The minimum acceptable criteria are two diameters downstream and 0.5 diameters upstream of flow disturbances. The test location must also be free of cyclonic or multidirectional flow. Once the distances have been determined, the values are used to select the minimum number of traverse points for representative sampling. Shorter distances require a greater number of traverse points. The test site configuration and measurement details are documented on EPA Method 1 Field Data Sheet.

Pace® FSD conducts the method as written with no routine deviations.

**EPA Method 2** defines procedures used to measure linear velocity and volumetric flow rate of a confined gas stream. Using traverse points determined by EPA Method 1, multiple differential pressure measurements (pitot impact opening versus static pressure) are made using a pitot tube and differential pressure gauge. The individual measurements are averaged and combined with the gas density to calculate the average gas velocity. The velocity and duct cross-sectional area are used to calculate the volumetric flow rate. The volumetric flow rate is expressed as actual cubic feet per minute (ACFM), standard cubic feet per minute (SCFM), and dry standard cubic feet per minute (DSCFM). The technician maintains comprehensive test records on EPA Method 2 Field Data Sheet. Details of the equipment used to measure gas velocity include:

Pitot Tube: S-Type

Differential Pressure Gauge: Oil or Electronic Digital Manometer

Temperature Device: Type K Thermocouple

Barometer Type: Electronic Digital Barometer

Gas Density Determination: EPA Method 3
Gas Moisture Determination: EPA Method 4

#### Method Defined Quality Control:

- Pitot tubes are verified on an annual basis.
- Temperature device operation is confirmed for single point temperature and polarity for each test. Temperature devices undergo a full multipoint verification on an annual basis.
- Electronic barometers are verified for accuracy and calibrated on a semi-annual basis. Aneroid barometers are not used.

- Electronic Digital Manometers (EDMs) are verified for accuracy and calibrated on a semi-annual basis. EDMs are operationally confirmed and leak checked for each run.
- Sampling system leak-checks are performed before and after each run and prior to any component change during a run.

Pace® FSD conducts the method as written with no routine deviations.

**Modified EPA Method 3/3A** defines procedures to quantify carbon dioxide (CO<sub>2</sub>) and oxygen (O<sub>2</sub>) concentrations from stationary combustion sources. An integrated gas sample is collected simultaneously with other emissions testing. Sample gases are extracted from an emission stream at a constant rate over the course of a test period equal to other test constituents. A Tedlar<sup>TM</sup>, aluminized Mylar<sup>TM</sup>, or other inert material bag contains the collected gas sample prior to sample analyses. Instrumental gas analyzers compliant to EPA Method 3A quantify the CO<sub>2</sub> and O<sub>2</sub> concentrations. Three point instrument calibrations (zero, mid, and high span) are performed to certify the instruments for gas analyses. The technician maintains comprehensive test records on EPA Method 3 and Gas Analysis Field Data Sheets. Equipment used for measuring gas composition includes:

Filter Material:

Glass-fiber Filter or equivalent

Moisture removal:

Condenser and/or sorbent

Bag Material:

Tedlar<sup>™</sup> or Aluminized Mylar<sup>™</sup> or equivalent

Gas Analyzer:

Non-dispersive Infrared Detector (CO<sub>2</sub>)

Paramagnetic Detector (O<sub>2</sub>)

Calibration Gases:

EPA Protocol 1

Method Defined Quality Control:

Sampling bag leak check.

Pace® FSD conducts the method as written with the following routine sampling deviation:

In the field, the gas sample is analyzed within two hours of collection using a portable  $O_2$  detector. At a later time, potentially outside of the eight hour hold period, the gas sample is re-analyzed using an EPA Method 3A instrumental gas analyzer to quantify  $CO_2$  and  $O_2$  concentrations.

The preliminary analysis result from the portable  $O_2$  detector is used to validate the Method 3A results. The results are acceptable when the  $O_2$  result from the field and the  $O_2$  result from the lab differ by  $\leq 0.3\%$ .

**EPA Method 4 - Isokinetic** defines procedures to measure the moisture content of emission gas streams from stationary sources. The moisture content of the gas stream is determined in conjunction with an isokinetic sampling train. Collected water

condensate is measured from the back half of the isokinetic train. Method 4 equations convert the condensed liquid volume to a gas volume. The water vapor volume compared with the dry standard gas volume collected through the isokinetic train determines the moisture content of the emissions gas stream and is reported in percent by volume. Test records are included on the associated isokinetic method data sheet. Equipment used for measuring moisture content includes:

Probe Material: Borosilicate glass or Stainless Steel

Filter Media: Glass or Quartz fiber Impinger Train Material: Borosilicate Glass

Desiccant: Drierite

Condensate Measure: Graduated Cylinder or Electronic Scale

Desiccant Measure: Electronic Scale

#### Method Defined Quality Control:

- Dry gas meters are verified by wet test meter comparison for a three-point "as found" determination and a full five-point calibration every 500 CF, or 90 days (first occurring). The Pace® standard "as left" calibration factor is within ± 1% (the method standard is ± 2%).

- Gas meter volumes are verified at each traverse point by calculating the expected gas volume for each interval and comparing the gas volume metered during the interval.
- Sample rate orifices are calibrated every 500 CF, or 90 days (first occurring).
- Temperature device operation is confirmed for single point temperature and polarity for each test. Temperature devices undergo a full multipoint verification on an annual basis.
- Electronic barometers are verified for accuracy and calibrated on a semi-annual basis. Aneroid barometers are not used.
- Sampling system leak-checks are performed before and after each run and prior to any component change during a run.
- Field scales are verified for accuracy over the entire range of use on an annual basis and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.

The metering system verification cited above is a method QC alternative but considered more rigorous. Pace® FSD conducts the method as written with no routine sampling deviations.

**EPA Method 5** defines procedures to measure particulate emissions from stationary sources. Using traverse points determined from EPA Method 1 and incorporating procedures from EPA Methods 2, 3, and 4, a sample gas stream is isokinetically drawn from the emission stream. The particulate dry fraction collects in the sampling probe and on a quartz or glass-fiber filter. The probe and filter components of the sampling train are heated to 248°F (±25°F) to prevent moisture condensation and preserve

sample integrity. The filtered sample gas stream passes through a series of impingers to condense water vapor and collect gaseous constituents. The first two impingers initially contain deionized water, and the third impinger is empty. A desiccant packed drying column follows the impingers to quantitatively collect the remaining moisture. An ice bath maintains the impinger train temperature (outlet) at 68°F or less. The impinger contents can be discarded or saved for additional analyses. Sample recovery and train clean up are performed after each run using procedures to ensure sample integrity and quantitative recovery. The train operator maintains comprehensive test records on EPA Method 5 Field Data Sheet, Isokinetic Particulate Sampling. Details of particulate testing are outlined below:

Nozzle/Probe Material:

Stainless Steel and Borosilicate Glass

Filter Holder Material:

Borosilicate Glass with glass or Teflon support

Filter Media:

Quartz or Glass-fiber, >99.95% efficient at

 $0.3 \mu m$ 

Impinger Train Material:

Borosilicate Glass

Impinger Reagents:

Deionized Water

Recovery Reagents: Acetone

Control Train:

Deionized water

Gas meter, orifice, differential pressure

gauges, pump, valves, temperature monitors

and controllers

Analytical Techniques:

Gravimetric

#### Method Defined Quality Control:

- Dry gas meters are verified by wet test meter comparison for a three-point "as found" determination and a full five-point calibration every 500 CF, or 90 days (first occurring). The Pace® standard "as left" calibration factor is within ± 1% (the method standard is ± 2%).
- Sample rate orifices are calibrated every 500 CF, or 90 days (first occurring).
- Gas meter volumes are verified at each traverse point by calculating the expected gas volume for each interval and comparing the gas volume metered during the interval.
- Pitot tubes are verified on an annual basis.
- Temperature device operation is confirmed for single point temperature and polarity for each test. Temperature devices undergo a full multipoint verification on an annual basis.
- Electronic barometers are verified for accuracy and calibrated on a semi-annual basis. Aneroid barometers are not used.
- Electronic Digital Manometers (EDMs) are verified for accuracy and calibrated on a semi-annual basis. EDMs are operationally confirmed and leak checked for each run.
- Sampling system leak-checks are performed before and after each run and prior to any component change during a run.

- Sampling is performed at an isokinetic rate between 90 and 110%.
- A field blank is collected to verify site conditions to be non-contaminating.
- Sampling and recovery reagents are reagent grade or better.
- Analytical balances are calibrated and certified on an annual basis by an external service provider and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.
- Field scales are verified for accuracy over the entire range of use on an annual basis and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.

The metering system verification cited above is a method QC alternative but considered more rigorous. Pace® FSD conducts the method as written with no routine sampling deviations

**EPA Method 9** defines procedures to evaluate the opacity of the plume emitted from a source stack. An independently certified visible emissions observer visually estimates the opacity of the non-moisture plume from the source. The observer positions themselves with the sun (or other light source) at their back and perpendicular to the plume when directly facing the emission point. The observer must also ensure a clear and contrasting background behind the plume. The certified observer then estimates (based on certification trials) the percentage of the background blocked by the source plume (plume opacity) in increments of 5%. Observed opacity readings are recorded at 15-second intervals throughout the run. Tabulated results include run average and successive six-minute averages. The spreadsheet software also searches the data set for any group of 24 consecutive readings that yield the highest possible six-minute average. The train operator maintains comprehensive test records on the Visible Emission Observation Form. Details of the opacity evaluation are outlined below:

Evaluation Period: One hour Observation Frequency: 15 Seconds

No. of Observations: 240
No. of Six-minutes Averages: 10

Observer Certifications: Semi-annual

Pace® FSD conducts the method as written with no routine deviations.

**EPA Method 201A** defines procedures to measure particulate matter equal to or less than 10 microns (PM-10) and 2.5 microns (PM-2.5) from stationary sources. Using traverse points determined from EPA Method 1 and incorporating procedures from EPA Methods 2, 3, 4, and 5, a sample gas stream is drawn from the emission stream at a constant rate through an in-stack sizing devices: a PM-10 cyclone followed by a PM-2.5 cyclone. The cyclones separator classifies particulate matter at 10-micron ( $\mu$ m) and 2.5-micron ( $\mu$ m) aerodynamic cut diameters (nominal). Cyclones collect particulate matter at the cut size and larger. The omission of either cyclone excludes the

measurement of that particle cut size from the method. The cyclones are followed by an in-stack glass fiber filter to collect remaining filterable particulate (less than the cut diameter). The sample gas moves through a heated sampling probe to the back half of the sampling train. This method is used in conjunction with Method 202 when the gas stream temperature exceeds 85°F to collect condensable particulate which is included as PM-2.5. See separate summary for Method 202. The back half of the train consists of glass impingers and a desiccant packed drying column to quantitatively collect water vapor. An ice bath maintains the impinger train temperature (outlet) at 68°F or less. Sample recovery and train clean-up are performed after each run using procedures to ensure sample integrity and quantitative recovery. Sample fractions are processed from the cyclone heads into separate sample containers using a brush and acetone. Gravimetric analysis is applied to determine the particulate mass for each size fraction. The train operator maintains comprehensive test records on EPA Method 201A Field Data Sheet. Details of PM-10 and PM-2.5 particulate testing include:

Nozzle/Probe Material:

Stainless Steel

PM-2.5 Separator:

Stainless Steel Cyclone

PM-10 Separator:

Stainless Steel Cyclone

Filter Holder Material:

Stainless Steel

Filter Media:

Glass-fiber, >99.95% efficient at 0.3 um

Impinger Train Material:

Borosilicate Glass

Recovery Reagents:

Acetone

Control Train:

Deionized Water EPA Method 17

Analytical Technique:

Gravimetric

#### Method Defined Quality Control:

- Dry gas meters are verified by wet test meter comparison for a three-point "as found" determination and a full five-point calibration every 500 CF, or 90 days (first occurring). The Pace® standard "as left" calibration factor is within ± 1% (the method standard is ± 2%).
- Sample rate orifices are calibrated every 500 CF, or 90 days (first occurring).
- Gas meter volumes are verified at each traverse point by calculating the expected gas volume for each interval and comparing the gas volume metered during the interval.
- Pitot tubes are verified on an annual basis.
- Temperature device operation is confirmed for single point temperature and polarity for each test. Temperature devices undergo a full multipoint verification on an annual basis.
- Electronic barometers are verified for accuracy and calibrated on a semi-annual basis. Aneroid barometers are not used.
- Electronic Digital Manometers (EDMs) are verified for accuracy and calibrated on a semi-annual basis. EDMs are operationally confirmed and leak checked for each run.

- Sampling system leak-checks are performed before and after each run and prior to any component change during a run.
- Sampling is performed at an isokinetic rate between 90 and 110%.
- A field blank is collected to verify site conditions to be non-contaminating.
- Sampling and recovery reagents are reagent grade or better.
- Analytical balances are calibrated and certified on an annual basis by an external service provider and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.
- Field scales are verified for accuracy over the entire range of use on an annual basis and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.

The metering system verification cited above is a method QC alternative but considered more rigorous. Pace® FSD conducts the method as written with no routine sampling deviations.

EPA Method 202 defines procedures to determine organic and inorganic condensable particulate matter (CPM) emissions from stationary sources. The CPM is collected in a condensate knock-out impinger and Teflon filter after filterable PM has been collected by either Method 5 or Method 201A. The gas stream is sample isokinetically following EPA Method 5 or Method 201A procedures. The gas stream is initially cooled with a spiral condenser using recirculated cool water to maintain a sample gas temperature of 85°F or less. Condensate from the spiral condenser collects in glass, stemless, dropout impingers. The intent of the condenser and dropout impinger is to minimize gas/water contact to reduce collection of unintended artifacts. The dropout impinger is followed by a second impinger to provide overflow capacity. A Teflon<sup>TM</sup> filter, also maintained at 85°F or less is used to collect any remaining organic CPM. The filter is followed by an iced, water prepared impinger and desiccant packed drying column to quantitatively Immediately after sampling, the Method 202 CPM collect remaining moisture. condensate is purged with nitrogen (N<sub>2</sub>) to liberate dissolved sulfur dioxide (SO<sub>2</sub>) gases. The contents of the dropout and backup impingers prior to the CPM filter are measured, weighed, and transferred to an appropriate sample bottle. CPM is quantitatively recovered with water, acetone, and hexane rinses. The CPM filter and water are extracted with hexane and combined with solvent rinses to determine the organic CPM. Following extraction, the water is dried and the residue measured as the inorganic CPM. The combination of both fractions represents the total condensable particulate matter (CPM). The train operator maintains comprehensive test records on appropriate Field Data Sheets.

Filter Holder Material: Glass, Stainless Steel (316 or equivalent), or

Fluoropolymer-coated Stainless Steel

Filter Media: Teflon, >99.95% efficient at 0.3 um

Impinger Train Material: Borosilicate Glass Impinger Reagents: Deionized Water

Recovery Reagents:

Acetone

Hexane

Control Train: Analytical Technique: Deionized Water EPA Method 5

Gravimetric

#### Method Defined Quality Control:

- Dry gas meters are verified by wet test meter comparison for a three-point "as found" determination and a full five-point calibration every 500 CF, or 90 days (first occurring). The Pace® standard "as left" calibration factor is within ± 1% (the method standard is ± 2%).
- Sample rate orifices are calibrated every 500 CF, or 90 days (first occurring).
- Gas meter volumes are verified at each traverse point by calculating the expected gas volume for each interval and comparing the gas volume metered during the interval.
- Pitot tubes are verified on an annual basis.
- Temperature device operation is confirmed for single point temperature and polarity for each test. Temperature devices undergo a full multipoint verification on an annual basis.
- Electronic barometers are verified for accuracy and calibrated on a semi-annual basis. Aneroid barometers are not used.
- Electronic Digital Manometers (EDMs) are verified for accuracy and calibrated on a semi-annual basis. EDMs are operationally confirmed and leak checked for each run.
- Sampling system leak-checks are performed before and after each run and prior to any component change during a run.
- Sampling is performed at an isokinetic rate between 90 and 110%.
- A field blank is collected to verify site conditions to be non-contaminating.
- Sampling and recovery reagents are reagent grade or better.
- Analytical balances are calibrated and certified on an annual basis by an external service provider and verified before each use using stainless steel reference weights traceable to national standards maintained by NIST.

The metering system verification cited above is a method QC alternative but considered more rigorous. Pace® FSD conducts the method as written with no routine sampling deviations.

Reference Standards. Pace® implements a comprehensive program to verify and validate reference standards to further enhance and support method standards. Primary reference standards are directly comparable to a reference base. The National Institute of Standards and Technology (NIST) maintains primary reference base that the last or very closely traceable secondary standards. These materials are then used to certify

AIR QUALITY DIVISION

Graymont Western Lime

secondary or transfer standards for use in quality management programs. Secondary reference standards are calibrated with primary standards using a high precision comparator. Materials that have a documented path to the primary standard are often referred to as traceable to NIST or NIST traceable. Where commercially and feasibly available, Pace® uses primary reference standards to perform calibrations and verifications. In other cases, Pace® maintains traceable secondary reference standards. Primary and secondary reference standards are used to calibrate and verify equipment and materials. Pace® reference standards are calibrated by external vendors that have a formal, registered quality system. Calibrations are performed with equipment and materials that are traceable to NIST.

Quality Controls (not defined in test methods):

- Sampling/Recovery Reagents are Reagent Grade or better.
- Reference Temperature Simulator is calibrated annually.
- Reference Pressure Transducer is calibrated annually.
- Reference DryCal airflow meter is calibrated annually.
- Mercury Barometer is a primary reference standard.
- Liquid Manometers are primary reference standards.
- Angle Blocks, Gauge Blocks, and Measuring Rods are verified every five years.
- Angle Gauges are verified each day of use.
- Calipers are verified annually.
- Stainless steel reference weights are verified every five years.
- Analytical balances are calibrated annually and verified at each use.
- Field balances are calibrated annually and verified at each use.

Quality Management System. To produce data that is complete, representative, and of known precision and accuracy, Pace® Analytical Field Services Division has designed and implemented a rigorous and innovative quality management system. The system was initially based on the USEPA Quality Assurance Handbook for Air Pollution Measurement Systems and continually developed as procedural complexities and standards progressed. The Field Services Division Quality Management System (Pace® FSD QMS) is now accredited by the American Association of Laboratory Accreditation (A2LA) to comply with three national accreditation standards:

- ASTM D7036 Standard Practice for Competence of Air Emission Testing Bodies (AETB).
- ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories
- The NELAC Institute General Requirements for Field Sampling and Measurement Organizations (FSMO)

#### The Pace® FSD QMS includes:

- Quality Programs
  - Ethics policy and training.
  - Corrective Action and Preventative Action (CAPA).

- Continuous Process Improvement.
- Documented Demonstrations of Capability.
- Internal and third party proficiency testing.
- Qualified Individual program (QI)
- Internal and external audits.
- Annual management reviews.
- Documentation and Traceability
  - High quality traceable standards and reagents.
  - Reagent tracking and management system.
  - Use of matrix spikes, duplicate analysis, internal standards, and blanks.
  - Validated workbooks for data collection and results reporting.
  - Electronic quality, training, and safety documents available in-field.
  - Sample security and preservation procedures.
  - Chain of custody maintained from sample collection through laboratory analysis.
- Equipment Calibration
  - Full time staff dedicated to equipment maintenance and calibration.

All equipment and instruments are calibrated by trained personnel on a frequency that meets or exceeds method requirements.



## Figure 1 Graymont Western Lime Gulliver, MI Lime Kiln Exhaust (Kiln 1)

