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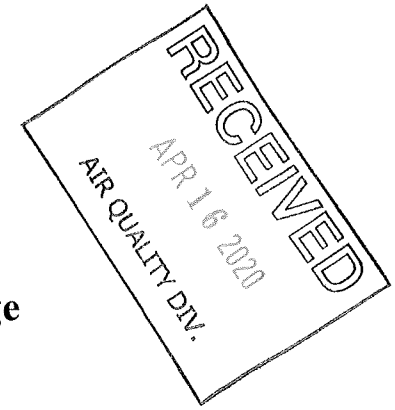

Holcim (US) Inc. d/b/a Lafarge
Alpena, MI Plant
US EPA Method 321
Hydrogen Chloride Testing
March 5, 2020
Project No. 046AS-715253

Prepared For

Holcim (US) Inc. d/b/a Lafarge Alpena Plant

By

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March 12, 2020

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Scope

Prism Analytical Technologies (Mt. Pleasant, MI) was contracted to conduct air testing for Holcim (US) Inc. d/b/a Lafarge Alpena Plant (Lafarge), Alpena, MI. Effluent emissions from the scrubber stack outlet (FG KG 6) were tested for gaseous hydrogen chloride (HCl), oxygen (O_2), and moisture (H_2O) content. Testing was performed March 5, 2020.

This test has been conducted with respect to the air emission testing requirements specified in section §63.1349 of 40 CFR Part 63, Subpart LLL, "National Emission Standards for Hazardous Air Pollutants for the Portland Cement Manufacturing Industry (NESHAP)" promulgated on February 12, 2013, and effective September 9, 2015. All testing will be performed following accepted EPA methodology. Lafarge intends to utilize sulfur dioxide (SO2) as a surrogate measurement in lieu of HCl CEMS for this emission unit as this is an acceptable means of demonstrating compliance per the aforementioned regulation.

Extractive Fourier transform infrared (FTIR) spectrometry following US EPA Method 321 was used to quantify gaseous hydrogen chloride and moisture from the effluent stream of the scrubber stack outlet. Moisture concentrations were determined from the FTIR data to correct hydrochloric acid to a dry value.

US EPA Method 3A was performed to quantify the oxygen concentrations to be used to calculate results corrected to 7% oxygen.

Phillip Kauppi (Prism) performed data collection. Blake Ericson (Prism) performed data validation and report generation.

Procedures

FTIR Instrumental Configuration

FTIR data were collected using an MKS MultiGas 2030 FTIR spectrometer. See Table 1 below for sampling system details.

The FTIR was equipped with a temperature-controlled, 5.11-meter multipass gas cell maintained at 191°C. Gas flows and sampling system pressures were monitored using rotameters and

pressure transducers. All data were collected at 0.5 cm^{-1} resolution. Each spectrum was derived from the coaddition of 64 scans, with a new data point generated approximately every 60 seconds.

FTIR diagnostics were performed daily, prior to beginning testing. Peak shape (FWHH), laser frequency, signal intensity, and linearizer checks were made to ensure all FTIR data generated will spectrally match all reference spectra, resulting in an accurate analysis. See the FTIR Diagnostics appendix for all results.

Table 1 – FTIR Sampling System

Source	MKS Serial #	Sampling Line	Probe Assembly	Particulate Filter Media	Operating Temperatures
Scrubber Stack Outlet	016630515	50' 3/8" dia. Teflon	8', 3/8" dia. Stainless steel + heated filter element	0.01µ borosilicate glass fiber	191°C FTIR + system

FTIR QA/QC Methodology

QA/QC procedures followed US EPA Method 321. See Tables 2 and 3 below for QA/QC procedure details and list of calibration gas standards. All calibration gases were introduced to the analyzers and the sampling systems using instrument grade stainless steel rotameters. All QA/QC procedures were within the acceptance criteria allowance of the EPA methodology. QA/QC procedures were run at each sampling location. QA/QC calculations are presented in detail below.

Table 2 – FTIR QA/QC Procedures

QAQC Specification	Purpose	Calibration Gas Analyte	Delivery	Frequency	Acceptance Criteria	Result
M321: Zero	Verify that the FTIR is free of contaminants & zero the FTIR	Nitrogen (zero)	Direct to FTIR	pre/post test	< MDL or Noise	Pass
M321: Calibration Transfer Standard (CTS) Direct	Verify FTIR stability, confirm optical path length	Ethylene	Direct to FTIR	pre/post test	+/- 5% cert. value	Pass
M321: Analyte Direct	Verify FTIR calibration	HCl/SF6	Direct to FTIR	pretest	+/- 5% cert. value	Pass
M321: CTS Response	Verify system stability, recovery, RT	Ethylene	Sampling System	pre/post each run	+/- 5% of Direct Measurement	Pass
M321: Zero Response	Verify system is free of contaminants, system bias	Nitrogen (zero)	Sampling System	pre/post test	Bias correct data	Pass
M321: Analyte Spike	Verify system ability to deliver and quantify analyte of interest in the presence of other effluent gases	HCl/SF6	Dynamic Addition to Sampling System, 1:10 effluent	pre/post each run	+/- 30% theoretical recovery	Pass

Table 3 – Calibration Gas Standards

Components	Concentration	Vendor	Cylinder #	Standard Type
Ethylene	100.5 ppm	Airgas	CC512555	Primary +/- 1%
HCl/SF6	9.90/5.05 ppm	Airgas	CC482234	HCl GMACS +/- 2.03%
Nitrogen	Zero gas	Airgas	CC151109	CEMS Grade
Oxygen (Span)	20.26%	Airgas	CC108555	EPA Protocol +/- 1%
Oxygen (Mid)	10.07%	Airgas	SG9173662BAL	EPA Protocol +/- 0.4%

FTIR QA/QC Calculations

Method 321: Analyte Spiking

Hydrogen chloride spiking was performed at the source prior to and after each test run to verify the ability of the sampling systems to quantitatively deliver a sample containing hydrogen chloride from the base of the probe to the FTIR. The spike target dilution ratio was 1:10 or less. Analyte spiking assures the ability of the FTIR to quantify hydrogen chloride in the presence of effluent gas.

As part of the spiking procedure, samples from the source were measured to determine hydrogen chloride concentrations to be used in the spike recovery calculations. The analyte spiking gas contained a low concentration of sulfur hexafluoride (SF_6). The determined SF_6 concentration in the spiked sample was used to calculate the dilution factor of the spike and thus used to calculate the concentration of the spiked hydrogen chloride. The following equation illustrates the percent recovery calculation.

$$\%R = 100\,x\,\frac{Sm-Su(1-DF)}{DF\,x\,Cs} \qquad \text{(Sec. 9.3.1 (1) USEPA Method 321)}$$

$$Ce = DF\,x\,Cs + Su(1-DF) \qquad \text{(Sec. 9.3.1 (2) USEPA Method 321)}$$

Sm = Mean concentration of the analyte spiked effluent samples (observed).
Ce = Expected concentration of the spiked samples (theoretical).
Df = dilution Factor (Total flow/Spike flow)
Cs = cylinder concentration of spike gas.
Su = native concentration of analytes in unspiked samples.

FTIR Post Collection Data Validation

As part of the data validation procedure, reference spectra are manually fit to that of the sample spectra and a concentration is determined. The reference spectra are scaled to match the peak amplitude of the sample, thus providing a scale factor. The scale factor multiplied by the reference spectra concentration is used to determine the concentration value for the sample spectra. Sample pressure and temperature corrections are then applied to compute the final

sample concentration. The manually calculated results are then compared with the software-generated results. The data is then validated if the two concentrations are within ± 20% agreement. If there is a difference greater than ± 20% the spectra are reviewed for possible spectra interferences or any other possible causes leading to misquantified data.

Oxygen Determination

Oxygen concentrations were determined using a Brand Gaus, Model 4710 Oxygen Analyzer, which utilizes Linear Output Zirconium Oxide Technology. The O2 analyzer was installed at the exhaust of the FTIR, with all flow passing through the O2 analyzer. The O2 analyzer continually measures oxygen as it flows through the system.

QA/QC procedures followed US EPA Methods 3A. See Tables 3 and 4 for list of calibration gas standards and QA/QC procedure details. All calibration gases were introduced to the analyzer and the sampling system using an instrument grade stainless steel rotameter. All QA/QC procedures were within the acceptance criteria allowance of the applicable US EPA methodology. See the Oxygen Analyzer Data Appendices for numerical results.

Table 4 – Oxygen QA/QC Procedures

QAQC Specification	Purpose	Calibration Gas Analyte	Delivery	Frequency	Acceptance Criteria	Result
M3A: Zero	Zero the analyzer	Nitrogen (zero)	Direct to O2 Analyzer	pretest	< MDL or Noise	Pass
M3A: Span	Establish the upper range of the analyzer	20.26 % O2	Direct to O2 Analyzer	pretest	+/- 2% cert. value	Pass
M3A: MidPoint	Confirm linear response	10.07 % O2	Direct to O2 Analyzer	pretest	+/- 2% cert. value	Pass
M3A: System Zero	Verify system Bias & Drift / leak check	Nitrogen / Ethylene (zero)	Sampling System	Daily, pre/post test	< 5% Bias < 3% Drift	Pass
M3A: System MidPoint	Verify system Bias & Drift / leak check	10.07 % O2	Sampling System	Daily, pre/post test	< 5% Bias < 3% Drift	Pass

Results and Discussion

Detection Limit

The detection limit of each analyte was calculated following Annex A2 of ASTM D6348-12 procedure using spectra that contained similar amounts of moisture.

Table 5 - FTIR Detection Limits

Analyte	Detection Limit (ppmv wet)	Detection Limit (%v)
HCl	0.2	-
H_2O	-	0.1

Scrubber Stack Outlet

Testing was performed on March 5, 2020. Three, one-hour test runs were performed on the scrubber stack outlet. Hydrogen chloride, oxygen, and moisture content were detected during the test runs. Analyte spiking was performed prior to and after each test run to confirm the ability of the measurement system to deliver and quantify HCl.

The determined moisture concentrations were used to correct HCl concentrations to dry values. Oxygen concentrations were used to correct results to 7% oxygen.

See the FTIR QA/QC Data Appendix for results. See Table 6 below for a summary of the analyte averages during the separate run periods. See the FTIR Test Run Data and Oxygen Analyzer Data Appendices for detailed run time results.

Lafarge supplied SO2 CEMS QAQC data and CEMS data corresponding to the reference method test run times. Larfarge calculated the Site Specific Operating Limit (SSOL) using HCl results from the reference method. See the Lafarge SO2 References Appendix for all material supplied from Lafarge.

Table 6 – Scrubber Stack Outlet Summary – Run Averages

Condition	Calculation	H2O (%v)	HCl (ppmv wet)	HCl (ppmv dry)	O2 (%v)	O2 (%v dry - Using 3A Corrections)	HCl (ppmv dry corrected)
03/05/2020 Scrubber Stack - Run 1 8:29 - 9:29	Minimum	12.86	< 0.2	< 0.2	7.66	9.08	< 0.2
	Maximum	13.33	0.29	0.33	8.16		0.39
	Average	13.08	< 0.2	< 0.2	7.90		< 0.2
03/05/2020 Scrubber Stack - Run 2 9:54 - 10:54	Minimum	12.85	< 0.2	< 0.2	7.41	8.94	< 0.2
	Maximum	13.70	< 0.2	< 0.2	7.96		0.20
	Average	13.27	< 0.2	< 0.2	7.69		< 0.2
03/05/2020 Scrubber Stack - Run 3 11:25 - 12:25	Minimum	13.05	< 0.2	< 0.2	7.54	9.10	< 0.2
	Maximum	14.02	< 0.2	< 0.2	8.06		< 0.2
	Average	13.47	< 0.2	< 0.2	7.79		< 0.2

The following Appendices include FTIR data and QA/QC procedures:

- FTIR Test Run Data
- FTIR QA/QC Data
- Oxygen Analyzer Data
- FTIR Diagnostics
- Gas Certificates
- Lafarge SO2 References

Date: March 12, 2020

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