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**Hydrogen Cyanide, Hydrogen Fluoride  
and Diatomic Chlorine  
from  
a Portland Cement Plant**

**Holcim Inc.  
Ada Facility  
1100 West 18th Street  
Ada, Oklahoma 74820**

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## 1. INTRODUCTION

The United States Environmental Protection Agency (US EPA) has directed the portland cement industry (SIC 3241) to conduct emissions testing as part of the US EPA Risk and Technology Review (RTR). This document provides the emission test results and supporting quality assurance/quality control (QA/QC) measures used to produce standardized data having known precision and accuracy. Collection of accurate, representative, and standardized data for facilities with low emissions is necessary especially in view of MACT standard setting procedures.

The process tested at the Ada facility is a semi-dry kiln, equipped with a single stage preheater and flash calciner in front, producing portland cement. There is no inline raw mill.

The modern semi-dry technology utilizes a single stage preheater and flash calciner arrangement in front of the kiln. This additional step helps to promote self-combustion of organics that evolve from the raw materials. The air pollution control equipment at the Ada facility consists of multiple control devices; inherent dry scrubbing for SO<sub>2</sub>; inline low NO<sub>x</sub> calciner, low NO<sub>x</sub> burner and selective non-catalytic reduction (SNCR) for NO<sub>x</sub>. The selective non-catalytic reduction (SNCR) post combustion emissions control technology reduces NO<sub>x</sub> by injecting ammonia into the process at a properly determined location. (**NOTE:** The add-on SNCR control technology is included in the design due to Holcim's corporate policy, not for regulatory compliance.) A fabric filter baghouse controls PM emissions from the clinker cooler and alkali bypass as well as the kiln.

The emissions from the clinker cooler, alkali bypass, and kiln are co-mingled in to a common stack.

A more detailed description of the processes is provided in Section 2 of the RTR Sampling and Analytical Protocol reproduced in Appendix G.

The Holcim Inc. retained DEECO Inc. (DEECO) to conduct emission tests for for hydrogen cyanide (HCN), hydrogen fluoride (HF), and diatomic chlorine (Cl<sub>2</sub>). All sampling runs were be one hour long. Concurrent measurements to determine volumetric flow rate were made.

A summary of the test results is shown in Table 1.1.

**TABLE 1.1 SUMMARY OF HYDROGEN CYANIDE, HYDROGEN FLUORIDE, AND DIATOMIC CHLORINE EMISSIONS; HOLCIM INC., ADA, OK FACILITY; KILN 3; JANUARY 8, 2024**

Test Parameters	Main Stack
Hydrogen Cyanide (FTIR) parts-per-million, dry basis corrected to 7% O <sub>2</sub> pounds-per-hour pounds-per-ton of clinker	2.9 1.69 0.023
Hydrogen Fluoride (FTIR) parts-per-million, dry basis corrected to 7% O <sub>2</sub> pounds-per-hour pounds-per-ton of clinker	<0.08 <0.032 <0.0004
Hydrogen Fluoride (Method 26A) parts-per-million, dry basis corrected to 7% O <sub>2</sub> pounds-per-hour pounds-per-ton of clinker	<0.62 <0.266 <0.0036
Diatomic Chlorine (Method 26A) parts-per-million, dry basis corrected to 7% O <sub>2</sub> pounds-per-hour pounds-per-ton of clinker	<0.18 <0.269 <0.0036

The sampling and analytical procedures followed are summarized in Table 1.2 and discussed in detail in Section 3.

Testing was performed on the Kiln 3 under one conditions on January 8, 2024.

Sampling was conducted by personnel from DEECO, Inc. of Raleigh, North Carolina. All questions regarding sampling and analytical data should be directed to Dr. Scott Steinsberger of DEECO at (800) 733-3261. The field sampling was completed by Dustin Carpenter, Lee Harris, and Scott Steinsberger of DEECO.

The remainder of this document summarizes the results, procedures and quality control measures followed for this program. Section 2 contains tabulated air emission results for each parameter of interest. Section 3 summarizes the air emission sampling and analytical procedures performed by DEECO, with a brief description and/or reference to the applicable methodologies. Section 4 discusses the basic quality control elements in place for this program to assure the collection of representative, accurate air emission data.

The appendices provided in this document contain all of the necessary information to verify the reported results. Included as Appendices are: Appendix A - Emission Summary Tables; Appendix B - Field Data and CEM/FTIR Data; Appendix C - Ion Chromatography Analytical Report Data; Appendix D - Plant Process Data; Appendix E - Calibration Documents; Appendix F - Test Participants; Appendix G - RTR Sampling and Analytical Protocol

**TABLE 1.2 SUMMARY OF SAMPLING AND ANALYTICAL PROTOCOLS FOR HOLCIM INC, ADA, OKLAHOMA FACILITY**

Location and Frequency	Test Parameter	Sampling Method	Sampling Procedure	Analysis Method	Analysis Procedure
Kiln 3 Main Stack	Volumetric Flow Rate and cyclonic check	EPA Methods 1 and 2	Velocity and temperature traverses	EPA Methods 1 and 2	Manometer for differential pressure and thermocouple for temperature
	Oxygen and Carbon Dioxide and Stratification Check	EPA Method 3A	Continuous; extractive sample	EPA Method 3A	Paramagnetic for O <sub>2</sub> and NDIR for CO <sub>2</sub>
	Moisture	EPA Method 4	Condensation	EPA Method 4	Gravimetric
	Hydrogen Fluoride and Diatomic Chlorine (Cl <sub>2</sub> )	EPA Method 26A	Isokinetic integrated sample	EPA Method 26A	Ion chromatography
	Hydrogen Fluoride and Hydrogen Cyanide	EPA Method 320	Continuous; extractive sample	EPA Method 320	Fourier Transform Infrared (FTIR) Spectroscopy

## **2. SUMMARY OF RESULTS**

Emissions sampling was conducted at the Holcim Ada OK facility. Sampling was conducted for stack gas flow rate (EPA Methods 1 and 2), stack gas oxygen and carbon dioxide (EPA Method 3A), stack gas moisture (EPA Method 4), stack gas hydrogen fluoride and diatomic chlorine (EPA Method 26A) and stack gas hydrogen cyanide and hydrogen fluoride (EPA Method 320).

Testing was conducted on the Kiln 3 main stack under one condition and the results are summarized in Table 2.1.

**TABLE 2.1 HOLCIM INC., ADA, OK FACILITY; KILN 3 MAIN STACK HYDROGEN CYANIDE, HYDROGEN FLUORIDE, AND DIATOMIC CHLORINE EMISSIONS; JANUARY 8, 2024**

Test Parameter	Kiln 3 Main Stack Run 1	Kiln 3 Main Stack Run 2	Kiln 3 Main Stack Run 3	Kiln 3 Main Stack Average
Time	15:21-16:27	16:44-17:50	18:05-19:11	January 8, 2024
Flow Rate (dscfm)	243,450	239,100	244,000	242,200
Oxygen	13.0%	13.0%	13.0%	13.0%
Carbon Dioxide	9.0%	9.7%	9.9%	9.5%
Moisture	24.9%	24.6%	24.9%	24.8%
Hydrogen Cyanide (FTIR)				
ppm <sub>dry</sub> at 7% O <sub>2</sub>	2.9	2.8	2.9	2.9
pounds-per-hour	1.71	1.63	1.72	1.69
pounds-per-ton of clinker	0.024	0.022	0.022	0.023
Hydrogen Fluoride (FTIR)				
ppm <sub>dry</sub> at 7% O <sub>2</sub>	<0.08	<0.08	<0.08	<0.08
pounds-per-hour	<0.032	<0.032	<0.032	<0.032
pounds-per-ton of clinker	<0.0005	<0.0004	<0.0004	<0.0004
Hydrogen Fluoride (Method 26A)				
ppm <sub>dry</sub> at 7% O <sub>2</sub>	<0.60	<0.63	<0.63	<0.62
pounds-per-hour	<0.260	<0.267	<0.274	<0.266
pounds-per-ton of clinker	<0.0037	<0.0035	<0.0035	<0.0036
Diatomic Chlorine (Method 26A)				
ppm <sub>dry</sub> at 7% O <sub>2</sub>	<0.17	<0.19	<0.17	<0.18
pounds-per-hour	<0.260	<0.282	<0.265	<0.269
pounds-per-ton of clinker	<0.0037	<0.0037	<0.0034	<0.0036

### 3. SAMPLING AND ANALYTICAL PROCEDURES

Table 1.2 presents a summary of the overall sampling and analytical protocols used for the test program for the Kiln 3 main stack at Holcim's Ada, OK facility. All sampling and analytical methods employed for this test program were performed in accordance with the procedures outlined in the Reference Test Methods contained in the Code of Federal Regulations, Title 40, Part 60, Appendix A (40 CFR 60, Appendix A) and 40 CFR 63, Appendix A.

#### 3.1 Sampling Point Determination - EPA Method 1

The Kiln 3 common stack is vertically oriented, round stack with an inside diameter of 139". The test location is approximately 1571" downstream (11.3 diameters) of the duct breaching and 1218" (8.8 Diameters) upstream of the stack outlet. The sampling locations met the minimum specifications for selection of a measurement site as outlined in EPA Method 1. Cyclonic flow checks, as described in EPA Method 1 Section 2.4, using the Type-S pitot null procedure and angle measurements was conducted at the Kiln 3 stack test location.

The number and location of the sampling or traverse points were determined according to the procedures outlined in EPA Method 1. All points were at least 1.0 inches from the stack wall, per Method 1.

A twelve (12) point sampling traverse was made using three (3) point traverses in each of 4 sampling ports) at the stack. Each traverse were made at each sampling location using a type-S pitot tube in accordance with EPA Methods 2 procedures. Gas temperatures were measured using calibrated Type K thermocouples and digital readout devices. All measurements were performed in accordance with the procedures in EPA Methods 2, and 26A.

A schematic of the Kiln 3 main stack is provided in Figure 3.1.

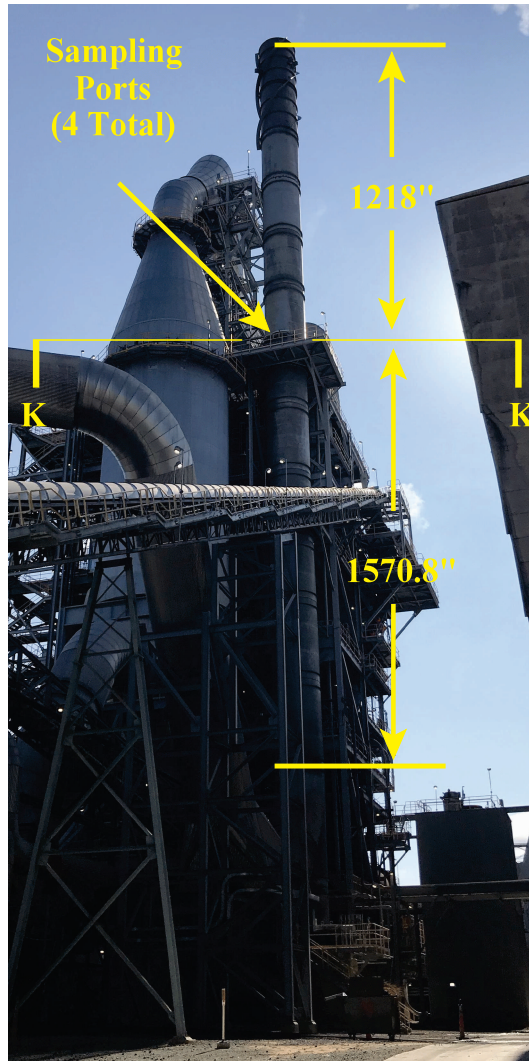
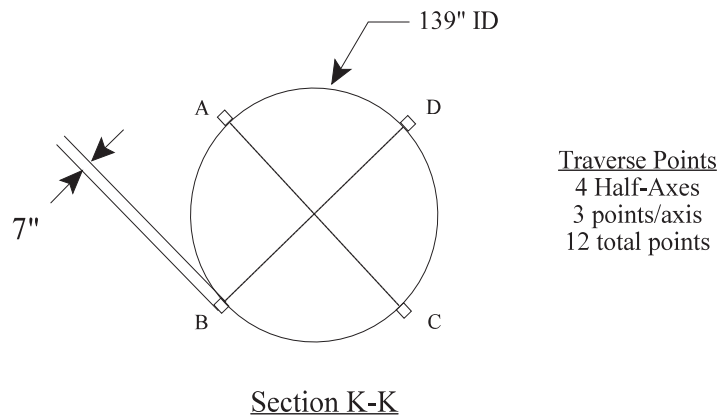


Figure 3.1 Schematic of the Kiln 3 Stack Sampling Location

### **3.2 Flue Gas Velocity and Volumetric Flow Rate - EPA Method 2**

The flue gas velocity and volumetric flow rate were determined according to the procedures outlined in EPA Method 2. Velocity measurements were using type S pitot tubes conforming to the calibration specifications outlined in EPA Method 2, Section 10.1. Each Type-S pitot tube, calibrated according to these standards, had an assigned coefficient. Differential pressures were measured with fluid manometers. Effluent gas temperatures were measured with chromel-alumel thermocouples equipped with digital readouts.

### **3.3 Outlet Flue Gas Composition - EPA Method 3A**

Outlet flue gas analysis for oxygen (O<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>) concentrations, and the calculation of percent excess air and flue gas dry molecular weight was performed in accordance with EPA Method 3A.

To evaluate the sampling location and points for FTIR and O<sub>2</sub> sampling, a three-point O<sub>2</sub> concentration stratification test on a line passing through the centroidal area at (for stacks is greater than 2.4 meters) at 0.4, 1.2 and 2.0 meters from the stack or duct wall. The procedures in Section 8.1.2 of Method 7E were followed, using oxygen as allowed by fourth sentence in Section 8.1.2. The plant O<sub>2</sub> CEMS was used as a control. A criteria of <5% variation from combined mean for each point was used as indication of non-stratification to allow single point sampling at the point closest to the mean.

Per EPA Method 3A for determining molecular weight, continuous extractive sampling was obtained using the same Method 320 sampling system described in Section 3.6.

A portion of the hot, wet gas sample was sent through a condensing system to remove the stack moisture. A portion of the moisture-free gas sample was sent to an O<sub>2</sub>/CO<sub>2</sub> analyzer.

Calibration procedures were performed in accordance with EPA methodology. Analyzers were calibrated before and after each test and a calibration check between each test run.

The pretest calibrations consisted of the following steps:

- Internal (direct) calibration of each analyzer to adjust calibration and check linearity.
- External (through the entire sampling system) calibration to check the system bias on zero and span gases.

The post test calibration consisted of an external system bias calibration check.

The analyzer calibrated using a certified zero and span (mid or high range) gas. Zero and span gases were directed to each analyzer through the appropriate plumbing, the calibration gas flow rates were adjusted to the correct flow rate and the analyzer was adjusted with the appropriate span pot.

After the analyzer was properly adjusted the linearity was checked using a low and high range

calibration gas. The maximum allowable limit for linearity is 2% of the analyzer range and all analyzers were shown to be linear within these limits before proceeding.

The external calibration bias check were performed by placing the CEM system in sampling mode and injecting a zero and span gas into the sample line at the probe exit. This check showed if there is any sampling system related bias, and also checks the integrity of the sample line.

### **3.3.1 Calibration Gases**

DEECO used EPA Protocol and/or  $\pm 2\%$  NIST Traceable gases for calibration as required by the various reference methods employed in this test program. Calibration gases were selected from previous experience with similar sources and/or from information obtained from the facility engineer prior to sampling. In some cases if the gases that are selected are out of the optimum range of operation then no significant impact of data quality is expected due to the linear nature of the analyzers that were used.

Specific HCN gases were manufactured for this test program in the range of 50-100 ppm to provide spikes in the 5-10 ppm range, or lower; with an SF<sub>6</sub> or appropriate tracer used to calculate the exact spike gas dilution ratio of 10% or less.

No audit gases from a federal or a state agency were provided.

### **3.3.2 Sampling Procedures**

At the completion of the pretest calibration routine, the CEM system was ready for operation. No further adjustments of sample flow rates, analyzer zero or span adjustments, or other critical CEM operating parameters were made until testing and post test calibration were complete.

Each sampling run was one hour. At the completion for each test run, calibration gases were used to check between test runs. A zero and the upscale calibration gas closest to the actual emission concentrations were used for the pretest and post test calibrations.

## **3.4 Flue Gas Moisture Content - EPA Method 4**

The flue gas moisture content was determined in conjunction with the EPA Method 26A trains according to the sampling and analytical procedures outlined in EPA Method 4. (**NOTE:** In order to maintain isokinetic sampling, the sampling rate used may have been required to temporarily exceed the EPA Method 4-specified maximum sampling rate of 0.75 CFM, based on observed stack gas pitot readings.) The impingers were connected in series and contained reagents as described below. The impingers were contained in an ice bath in order to assure condensation of the moisture in the flue gas stream. Any moisture that is not condensed in the impingers was captured in the silica gel, therefore all moisture was weighed and entered into moisture content calculations.

### 3.5 Hydrogen Fluoride and Diatomic Chlorine - EPA Method 26A

Sampling and analytical procedures were those outlined in EPA Method 26A to determine primarily diatomic chlorine (Cl<sub>2</sub>) emissions and hydrogen fluoride (HF) emissions at main stack outlet sampling locations. Duplicate simultaneous trains (a.k.a “paired trains”) for each test run were used to determine precision.

Sample was collected through a heated glass probe, followed by a heated Teflon filter, where stack gas HF and Cl<sub>2</sub> were collected in a series of chilled impingers. The sampling train impingers contained 100 ml of 0.1N sulfuric acid in the first and second, an empty third impinger, 100 ml of 0.1N NaOH in the fourth and fifth and 200 grams of silica gel in the last impinger

Sampling was conducted isokinetically (±10%) with readings of flue gas parameters recorded at traverse points selected according to EPA Method 1. Leak-checks on the Method 26A sampling train were performed before and after each sampling run and optionally for any port change. The sampling train leak-checks and leakage rate (where applicable) were documented on the field test data sheet for each respective run. All leak checks were acceptable.

The glass button hook nozzle and probe liner was constructed of borosilicate glass. The filter holder was constructed of borosilicate glass with a Teflon frit filter support and a sealing gasket. A PTFE-bonded glass fiber filter was used. The probe and filter housing were heated to above 248 °F and not exceed an upper boundary of 273 °F. Probe liners and filter holders were cleaned thoroughly prior to testing.

The Method 26A trains was operated isokinetically for a minimum of 60 minutes and collected a minimum of 1 dry, standard cubic meter (DSCM). Pretest preparations, preliminary determinations, and leak check procedures were those outlined in EPA Method 5.

After completion of sampling the train was leak checked and transferred to the sample recovery trailer. All leak checks were acceptable. The impingers were weighed to determine moisture gain in accordance with EPA Method 4.

Sample recovery involved quantitative recovery of the sulfuric acid impinger contents and the NaOH impinger contents into separate tare-weighed, precleaned polyethylene sample containers.

The nozzle, probe, filter and filter housing were not recovered.

The contents of sulfuric acid impingers, including the contents if any of the empty (2<sup>nd</sup> knockout or third) impinger were quantitatively transferred to the tare-weighed, precleaned polyethylene sample container, followed by three rinses with deionized (DI) water of the impingers and all connecting glassware (including the connecting glassware to the first impinger) placed in the same H<sub>2</sub>SO<sub>4</sub> container. The container was labeled and weighed to determine the final sample volume.

The contents NaOH impingers were quantitatively transferred to a second tare-weighed, precleaned polyethylene sample container, followed by three rinses with DI water of the impingers

and all connecting glassware placed in the same NaOH container. The container was labeled and weighed to determine the final sample volume.

Sample recovery from each train included:

1. Container No. 1 - Contents of H<sub>2</sub>SO<sub>4</sub> impingers and knockout impinger and, and DI rinse of impingers and connecting glassware; and
2. Container No. 2 - Contents NaOH impingers, and DI rinse of impingers and connecting glassware.

Additional quality control consisted of collecting and analyzing a field blank train for every three test runs. The blank train was assembled from a used train, leak checked and sat for a period equal to the sampling time (i.e, 1-hr). The blank train data was to be used to determine the method detection limit for the test program target analytes (ie. The lowest number that could be detected), and compared to stack emissions.

Reagent blanks of 0.1 N H<sub>2</sub>SO<sub>4</sub>, 0.1N NaOH, and DI water were collected and archived for later analysis should there be any issues with the field blank train samples

The H<sub>2</sub>SO<sub>4</sub> impinger solutions were analyzed using ion chromatography techniques for fluoride ions (F<sup>-</sup>) (EPA SW-9057). Duplicate analyses performed on the samples and field blanks. Precision was demonstrated by duplicate injection of each sample, the results of each individual analysis being within 5% of their mean to be acceptable.

The NaOH impinger solutions was treated with sodium thiosulfate to ensure complete conversion of hypochlorous acid (HClO) to chloride ions (Cl<sup>-</sup>). The resulting solution was analyzed using ion chromatography techniques for chloride ions (EPA SW-9057). Duplicate analyses was performed on the samples and field blanks. Precision was demonstrated by duplicate injection of each sample, the results of each individual analysis being within 5% of their mean to be acceptable

All EPA Method 26A HF/Cl<sub>2</sub> samples were analyzed by Element One of Wilmington NC. Refer to Section 1, Figure 1.1 of the RTR Sampling and Analytical Protocol for contact information.

For this test program, the relative deviation (RD) was to be calculated as described in EPA Method 30B between the Cl<sub>2</sub> concentrations measured with the paired trains. A criteria of a less than 10% relative deviation or 0.2 ppm absolute difference was required.

The absolute differences between the Cl<sub>2</sub> concentrations measured with the paired trains is summarized in Table 3.1. For each paired run, Cl<sub>2</sub> concentrations met the 0.2 ppm absolute difference criteria.

**TABLE 3.1 PAIRED METHOD 26A SAMPLING TRAIN DIATOMIC CHLORINE CONCENTRATION COMPARISON RESULTS FOR THE KILN 3 MAIN STACK; JANUARY 8, 2024**

<b>Run</b>	<b>Time</b>	<b>Train A Diatomic Chlorine Concentration (ppm,dry)</b>	<b>Train B Diatomic Chlorine Concentration (ppm,dry)</b>	<b>Absolute Difference (ppm,dry)</b>
Run 1	15:21-16:27	<0.09	<0.10	0.01
Run 2	16:44-17:50	<0.11	<0.11	0.00
Run 3	18:05-19:11	<0.10	<0.10	0.00

### 3.6 Hydrogen Cyanide and Hydrogen Fluoride - EPA Method 320

EPA Method 320 was performed to determine emissions of concentrations of HCN and HF. Three, 1-hour sampling runs were conducted under each representative process and control system operating conditions.

The gas sample was extracted from the stack through a glass-lined probe and filter heated to 375° F. For external calibration checks and analyte spikes, the gases were introduced in front of the heated filter. Any excess calibration gas was diverted through the sample probes into the source. Outflow of gas from the heated filter enclosure was transported through a Teflon sample line heated to 375° F. For these sources approximately 300' of sample line was required. The heated sample line was connected directly to the FTIR sample cell. Using heat-traced Teflon tubing the exit of the FTIR cell was connected to a sample pump with a heated stainless steel pump head. The pump discharge was directed to a proprietary chiller-type gas conditioner to remove moisture prior to delivery sample gas to the O<sub>2</sub>/CO<sub>2</sub> monitor.

The distribution of the gas sample to the monitors was accomplished using a panel equipped with valves and rotometers. The gas sample was then divided and directed to the O<sub>2</sub>/CO<sub>2</sub> analyzer.

FTIR sample cell was maintained at 191° C and connected to a MKS Instruments Multigas 2030 Fourier Transform Infrared Spectrometer and Detector.

The FTIR spectrometer measured vapor phase organic or inorganic compounds which absorb energy in the mid-infrared spectral region, about 400 to 4000 cm<sup>-1</sup> (25 to 2.5 μm). Continuous measurement were made by matching sample absorbance bands with bands in reference spectra, and comparing sample band intensities with reference band intensities.

The principle limitation to FTIR spectroscopy are the presence of interfering compounds that also absorb energy in the mid-infrared spectral region. In a cement kiln stack gas matrix, water vapor (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are the primary interferents that must be incorporated into the identification and quantitation method.

The FTIR software performs the computation for a single compound by subtracting all the other compounds (interferants and target) from the absorbance spectra and quantifies the single compound based on the remain absorbance. The FTIR software provides a Standard Error Calculation (SEC) value that is an indication of how well the identification and quantitation has been performed. A high SEC indicates that other interferants have not been accounted for in the analysis method, and a low SEC is indicative of greater confidence measurement.

The instrument is operated with a resolution of 0.5 cm<sup>-1</sup> with 4x zero filling. Beer-Norton Medium apodization is used with amplitude phase correction.

For this RTR test program, following specific QA/QC activities for EPA Method 320 were performed and are summarized in Table 3.2

### **3.6.1 Laboratory QA/QC Activities Before Field Test Program**

Before field testing occurs, the following QA/QC activities were conducted;

- 1) Seven consecutive samples of dry nitrogen through the sampling system was acquired and used to calculate the standard deviation for each of the test program target analytes multiplied by a factor of 3. These data were considered representative of detection limits (DL) for this test program and were below the 0.5 ppm required DL for both HCN and HF;
- 2) From these seven dry nitrogen samples, the results for the Signal-to-Noise Ratio (SNR) @ 2500  $\text{cm}^{-1}$  was >2500, at 64 scans and the results for single beam intensity @ 2500  $\text{cm}^{-1}$  was >0.9; and
- 3) The HCN calibration gases was analyzed directly and the FTIR responses agreed with tag value within 5%

### **3.6.2 QA/QC Activities During Field Test Program**

During the field test program, following QA/QC activities were be performed and criterium met;

- 1) On each test day prior to any testing, an instrument background was collected using dry nitrogen directed to the gas cell. The background was collected with at least 128 scans;
- 2) The probe, filter, sample line and all sample system components in contact with effluent were be maintained at or above 375°F or 191°C (consistent with FTIR calibration temperature) to avoid any possible “cold spots;”
- 3) A system zero with all sampling system components at operating temperature was performed by injecting nitrogen at the sample probe and through sample filter and entire measurement system. After zero equilibration was achieved, all measurement components were quantified for at least 128 scans;
- 4) The sample probe was position at effluent measurement point and sampling was continue until equilibration of the measurement system has been achieved. At this point, the effluent concentrations was quantified with two consecutive 64-scan samples as the initial native concentration for the dynamic spike;
- 5) Analyte spiking was conducted for HCN before the first test run, and after each successive test run for a minimum of 4 spikes per test condition. These results were used to determine accuracy and are summarized in Table 3.3;
- 6) The spike gas injections was maintained at 10% or less of total sample volume. The spike gas concentration and flow rate was be selected to approximately double the native effluent concentration. Spike recovery results were within  $\pm 20\%$  of the expected value. An  $\text{SF}_6$  tracer was used to calculate the exact spike gas dilution ratio of 10% or less;

**TABLE 3.2 FTIR PRETEST AND FIELD TEST QA/QC SUMMARY**

Spectrum	HCN	SF6	HF	SNR 2500	sBeam @2500
<b>Seven consecutive samples of dry nitrogen for detection limit</b>					
SPC__000837.LAB	-0.051		-0.002	6223.51	1.42
SPC__000838.LAB	-0.032		-0.000	5809.30	1.42
SPC__000839.LAB	0.046		-0.017	3759.60	1.42
SPC__000840.LAB	-0.011		0.016	4373.66	1.42
SPC__000841.LAB	0.080		0.002	5347.95	1.42
SPC__000842.LAB	0.059		-0.012	5012.46	1.42
SPC__000843.LAB	-0.029		-0.006	4706.13	1.42
<b>Standard Deviation X 3</b>	<b>0.156</b>		<b>0.032</b>		
<b>Averages</b>				<b>5033.23</b>	<b>1.42</b>
<b>HCN Standard (CC768222; 49.9 ppm HCN/5.0 ppm SF6)</b>					
SPC__006995.LAB	49.49	4.88			
SPC__006996.LAB	49.58	4.87			
SPC__006997.LAB	49.45	4.86			
<b>Averages</b>	<b>49.50</b>	<b>4.87</b>			
<b>Residuals for Post HCN analyte spike native scans</b>					
SPC_007092.LAB					
Concentration	0.98		-0.12		
MDC3	0.16		0.40		
MDC3%	NA		NA		
SPC_007093.LAB					
Concentration	1.33		-0.16		
MDC3	0.13		0.39		
MDC3%	NA		NA		
<b>Final SNR @ 2500 cm<sup>-1</sup> and single beam intensity @ 2500 cm<sup>-1</sup></b>					
SPC__007306.LAB				6836.5	1.32

- 7) After the dynamic spike, nitrogen was sent through the sampling system until all traces of spike gas removed and lines proven below DL for target analytes;
- 8) The nitrogen purge was discontinued and the sampling system was allowed to equilibrate with stack gas before starting a test run. The first two consecutive 64-scan samples of a sample run was used for the final native concentration. Residual results for HCN and HF were verified to be less than 0.2-0.3 ppm for data acceptance, or less than 5% of the measured value, whichever was least restrictive.
- 9) The final SNR @ 2500  $\text{cm}^{-1}$ , at 64 scans, and the results for single beam intensity @ 2500  $\text{cm}^{-1}$  were verified to met the >2500 and >0.9 criterium; respectively.

**TABLE 3.3 ETHYLENE CALIBRATION TRANSFER STANDARD (CTS) AND HYDROGEN CYANIDE ANALYTE SPIKING TEST RESULTS FOR THE KILN 3 MAIN STACK; JANUARY 8, 2024**

<b>Run</b>	<b>Time</b>	<b>Average Native Hydrogen Cyanide Concentration (ppm,wet)</b>	<b>Spike plus Average Hydrogen Cyanide Native Concentration (ppm,wet)</b>	<b>Hydrogen Cyanide Spike Recovery</b>	<b>CTS Error</b>
Pre Run 1	15:02-15:15	1.08	3.99	99.1%	-1.3%
Post Run 1	16:20-16:34	1.41	4.11	96.5%	
Post Run 2	17:43-17:56	1.09	3.92	94.1%	
Post Run 3	19:03-19:15	1.22	4.29	102.9%	-1.8%

## 4. QA/QC PROCEDURES AND RESULTS

The objective of a quality assurance/quality control (QA/QC) program is to assure that the precision and accuracy of all environmental data generated by DEECO for clients are commensurate with data quality objectives (DQO's). DQO's are based on a common understanding of the intended end use(s) of the data, the measurement process, and the availability of resources. Once DQO's are established, formally or informally, QC protocol can be defined for the measurements.

In this project, the final data user is Holcim. The data quality objectives in this project are to generate scientifically sound data to be used for compliance purposes.

### 4.1 Sampling Equipment

All of the sampling equipment used was calibrated according to the procedures outlined in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, EPA-600/4-77-027b.

#### 4.1.1 Manual Sampling Equipment Calibrations

For sampling Methods 1, 2, and 4 the procedures and equipment used to measure stack gas velocity and temperature measurements and the metering system used to maintain constant rate sampling conditions and to determine the sample gas volume were subjected to pretest and posttest calibrations and/or inspections as required by the appropriate EPA methods.

**Barometer** - Barometric pressure values were obtained from a calibrated barometer, verified by phone call to a local airport, and corrected for elevation to sample port level (0.01 inches Hg per 10 ft. elevation).

**Pitot Tubes** - Each pitot tube used in sampling meets the design specifications for type-S pitot tubes in EPA Method 2. Therefore, a maximum value baseline coefficient ( $C_p$ ) of 0.84 is assigned to each pitot tube. Calibration by the manufacturer for pitot face-opening alignment included measuring the external tubing diameter (dimension  $D_t$ ), the base-to-opening plane distance (dimensions  $P_a$  and  $P_b$ ), and the face opening misalignment angles, with all terms as described in EPA Method 2. Pitot tubes were visually inspected for structural integrity at the completion of each test. Inspection sheets for pitot tubes are included in Appendix E.

**Calibration Meter and Metering System** - The secondary reference meter equipment arrangement for calibration is shown in Figure 5.7 of EPA Method 5. The prescribed procedures were followed. A wet test meter with a 1 ft<sup>3</sup>/rev capacity and  $\pm 1$  percent accuracy is used as the primary calibrant. The dry gas meter's pump is run for a minimum of 5 minutes at a flow rate of 0.35 cfm to condition the interior surface of the wet test meter. Leak checks are performed and if satisfactory, triplicate runs at each of no less than five different flow rates are done. A calibration curve is prepared and the meter is recalibrated after 200 hours of operation or annually, whichever comes first.

The calibration set-up for the dry gas metering system using the secondary reference meter in lieu of the wet test meter is given in Figure 5.5 of EPA Method 5. A leak check of the metering system before calibration was performed as shown in Figure 5.4 of EPA Method 5. The metering system's pump is operated for 5 minutes at an orifice manometer setting of 0.5 inches H<sub>2</sub>O to heat up the pump and system to stabilize the meter inlet and outlet temperatures. Values for the orifice setting ( $\Delta H$ ), wet test meter volume ( $V_w$ ), corresponding dry test meter volume ( $V_d$ ), dry test meter inlet and outlet gas temperatures ( $t_{di}$  and  $t_{dn}$ ), and time are recorded for the initial calibration. Then the ratio of the wet test meter to the dry test meter ( $\gamma$ ) and the orifice pressure differential that equates to 0.75 cfm at standard conditions ( $\Delta H@$ ) are calculated.

A post-test meter calibration was made on the dry gas meter used during the test to check its accuracy against the pre-test calibration. This post-test calibration check was made using the average orifice setting obtained during each test run and setting the vacuum at the maximum value obtained during each test run. These test runs were made against DEECO's secondary reference dry gas meter which was calibrated against a wet test meter. The calibration data sheets for the dry gas meters are included in Appendix E.

**Thermocouples and Digital Indicators** - Thermocouples were calibrated by comparing them against an ASTM-3F mercury-in-glass thermometer at approximately 32 °F (ice water), ambient temperature, and at approximately 220 °F. Each thermocouple was calibrated against temperature ranges to which it is typically exposed during test conditions, and they agreed within 1.5 percent (expressed in °R) of the reference thermometer throughout the entire calibration range. Also, thermocouples were checked at ambient temperature at the test site to verify calibration. The calibration data sheets for the thermocouples are included in Appendix E.

**Pretest and Posttest Leak Checks of Sampling Trains** - Each Method 4 sampling train was subjected to pretest leak checks and posttest leak checks. For all sampling runs the posttest leak checks were acceptable (less than 4% of the sampling rate at the highest vacuum recorded during the test run).

## **4.2 Analytical QA/QC Results**

Analytical measurements of precision and accuracy were made on stack gas samples, and are summarized in a separate report.

**Appendix A**  
**Emission Summary Tables**

**Company: Holcim; Ada OK**  
**Source: Kiln 3 Main Stack**  
**Job ID: 24-3326**  
**Train Type: EPA Method 26A**

	1A	1B	2A	2B	3A	3B	Average
	01/08/24	01/08/24	01/08/24	01/08/24	01/08/24	01/08/24	
	1521-1627	1521-1627	1644-1750	1644-1750	1805-1911	1805-1911	
Initial Meter Volume, ft <sup>3</sup>	436.416	618.448	484.263	665.311	531.261	711.853	
Final Meter Volume, ft <sup>3</sup>	483.688	664.783	530.969	711.549	578.636	759.327	
Intra-Port Volume and/or Leak Check Correction, ft <sup>3</sup>	0.000	0.000	0.000	0.000	0.000	0.000	
<b>Total Sample Volume, cf</b>	<b>47.272</b>	<b>46.335</b>	<b>46.706</b>	<b>46.238</b>	<b>47.375</b>	<b>47.474</b>	<b>46.900</b>
DGM Calibration Factor	0.973	0.967	0.973	0.967	0.973	0.967	0.970
Average DGM Temp, F	58.9	41.5	68.8	47.9	79.8	47.2	57.4
Average DGM delta H, "H2O	1.67	1.96	1.62	1.91	1.69	2.00	1.81
Barometric Pressure, "Hg	28.15	28.15	28.15	28.15	28.15	28.15	28.15
<b>Corrected Sample Vol, dscf</b>	<b>44.198</b>	<b>44.582</b>	<b>42.846</b>	<b>43.923</b>	<b>42.581</b>	<b>45.169</b>	<b>43.883</b>
<b>Corrected Sample Vol, dscm</b>	<b>1.252</b>	<b>1.262</b>	<b>1.213</b>	<b>1.244</b>	<b>1.206</b>	<b>1.279</b>	<b>1.243</b>
<b>Sample Volume (at Stack Conditions), acf</b>	<b>90.099</b>	<b>89.441</b>	<b>87.426</b>	<b>88.913</b>	<b>87.505</b>	<b>91.352</b>	<b>89.123</b>
<b>Sample Volume (at Stack Conditions), acm</b>	<b>2.551</b>	<b>2.533</b>	<b>2.476</b>	<b>2.518</b>	<b>2.478</b>	<b>2.587</b>	<b>2.524</b>
Oxygen, %	13.0	13.0	13.0	13.0	13.0	13.0	13.0
Carbon Dioxide, %	9.0	9.0	9.7	9.7	9.9	9.9	9.5
Nitrogen, %	78.0	78.0	77.3	77.3	77.1	77.1	77.5
Stack Gas Excess Air, %	171.2	171.2	175.5	175.5	176.8	176.8	174.5
Total Moisture Catch Weight, grams	320.0	303.8	301.6	298.9	308.4	306.7	304.7
<b>Stack Gas Moisture, %</b>	<b>25.5</b>	<b>24.3</b>	<b>24.9</b>	<b>24.3</b>	<b>25.5</b>	<b>24.3</b>	<b>24.8</b>
Stack Gas Dry Molecular Weight, lb/lbmole	29.96	29.96	30.07	30.07	30.10	30.10	30.05
Stack Gas Wet Molecular Weight, lb/lbmole	26.91	27.05	27.07	27.14	27.02	27.16	27.06
Average Stack Temp, F	293.9	293.9	300.7	300.7	300.0	300.0	298.2
Stack Static (Gauge) Pressure, "H2O	-0.25	-0.25	-0.25	-0.25	-0.25	-0.25	-0.25
Stack Gas Actual Pressure, "Hg	28.13	28.13	28.13	28.13	28.13	28.13	28.13
Average Sqrt delta P *	1.075	1.075	1.059	1.059	1.084	1.084	1.073
Pitot Tube Coefficient	0.85	0.85	0.85	0.85	0.85	0.85	0.85
<b>Stack Gas Velocity, ft/second</b>	<b>77.96</b>	<b>77.76</b>	<b>76.91</b>	<b>76.82</b>	<b>78.77</b>	<b>78.56</b>	<b>77.80</b>
Nozzle Inside Diameter, inches	0.250	0.250	0.250	0.250	0.250	0.250	
Total Sample Time, min	60	60	60	60	60	60	60
<b>Isokinetic Rate, %</b>	<b>94.2</b>	<b>93.8</b>	<b>92.7</b>	<b>94.4</b>	<b>90.6</b>	<b>94.8</b>	<b>93.4</b>
Stack Dimensions	139 in. ID	139 in. ID	139 in. ID	139 in. ID	139 in. ID	139 in. ID	
Stack Area, sq ft	105.38	105.38	105.38	105.38	105.38	105.38	105.38
<b>Actual Stack Gas Flow Rate, acfm</b>	<b>492,900</b>	<b>491,700</b>	<b>486,300</b>	<b>485,700</b>	<b>498,000</b>	<b>496,700</b>	<b>491,900</b>
Actual Stack Gas Flow Rate, acmm	13,957	13,923	13,771	13,754	14,102	14,065	13,929
<b>Stack Gas Flow Rate, dscfm</b>	<b>241,800</b>	<b>245,100</b>	<b>238,300</b>	<b>239,900</b>	<b>242,400</b>	<b>245,600</b>	<b>242,200</b>
Stack Gas Flow Rate, dscmm	6,847	6,940	6,748	6,793	6,864	6,955	6,858

Company: Holcim; Ada OK  
Source: Kiln 3 Main Stack  
Job ID: 24-3326  
Train Type: EPA Method 26A

NOTE: Average INCLUDES Non-detect runs' results

"ND()" denotes values below detection limits

		1A 01/08/24 1521-1627		1B 01/08/24 1521-1627		2A 01/08/24 1644-1750		2B 01/08/24 1644-1750		3A 01/08/24 1805-1911		3B 01/08/24 1805-1911		Average
Hydrogen Fluoride	Catch Wt, mg	ND( 0.348 )	ND( 0.367 )	ND( 0.365 )	ND( 0.365 )	ND( 0.361 )	ND( 0.383 )	ND( 0.365 )	ND( 0.365 )	ND( 0.361 )	ND( 0.383 )	ND( 0.365 )	ND( 0.365 )	ND( 0.365 )
	Conc., mg/dscm	ND( 0.278 )	ND( 0.291 )	ND( 0.301 )	ND( 0.293 )	ND( 0.299 )	ND( 0.299 )	ND( 0.294 )	ND( 0.294 )	ND( 0.299 )	ND( 0.299 )	ND( 0.294 )	ND( 0.294 )	ND( 0.294 )
	Conc., mg/dscm @ 7% O2	ND( 0.489 )	ND( 0.512 )	ND( 0.529 )	ND( 0.516 )	ND( 0.527 )	ND( 0.527 )	ND( 0.517 )	ND( 0.517 )	ND( 0.527 )	ND( 0.527 )	ND( 0.517 )	ND( 0.517 )	ND( 0.517 )
	Conc., mg/dscm @ 12% CO2	ND( 0.371 )	ND( 0.388 )	ND( 0.372 )	ND( 0.363 )	ND( 0.363 )	ND( 0.363 )	ND( 0.370 )	ND( 0.370 )	ND( 0.363 )	ND( 0.363 )	ND( 0.370 )	ND( 0.370 )	ND( 0.370 )
	Conc., ppmvd	ND( 0.334 )	ND( 0.350 )	ND( 0.362 )	ND( 0.353 )	ND( 0.360 )	ND( 0.360 )	ND( 0.353 )	ND( 0.353 )	ND( 0.360 )	ND( 0.360 )	ND( 0.353 )	ND( 0.353 )	ND( 0.353 )
	Conc., ppmvd @ 7% O2	ND( 0.588 )	ND( 0.615 )	ND( 0.637 )	ND( 0.621 )	ND( 0.633 )	ND( 0.634 )	ND( 0.621 )	ND( 0.621 )	ND( 0.633 )	ND( 0.634 )	ND( 0.621 )	ND( 0.621 )	ND( 0.621 )
	Conc., ppmvd @ 12% CO2	ND( 0.446 )	ND( 0.466 )	ND( 0.448 )	ND( 0.436 )	ND( 0.436 )	ND( 0.436 )	ND( 0.445 )	ND( 0.445 )	ND( 0.436 )	ND( 0.436 )	ND( 0.445 )	ND( 0.445 )	ND( 0.445 )
	Emission Rate, lb/hr	ND( 0.252 )	ND( 0.267 )	ND( 0.269 )	ND( 0.264 )	ND( 0.272 )	ND( 0.275 )	ND( 0.266 )	ND( 0.266 )	ND( 0.272 )	ND( 0.275 )	ND( 0.266 )	ND( 0.266 )	ND( 0.266 )
	Clinker Rates (mtph and lbs/ton)	64.20	ND( 0.0037 )	68.40	ND( 0.0035 )	70.10	ND( 0.0035 )	ND( 0.0036 )	ND( 0.0036 )	70.10	ND( 0.0035 )	ND( 0.0036 )	ND( 0.0036 )	ND( 0.0036 )
Chlorine	Catch Wt, mg	ND( 0.335 )	ND( 0.381 )	ND( 0.384 )	ND( 0.388 )	ND( 0.359 )	ND( 0.362 )	ND( 0.368 )	ND( 0.368 )	ND( 0.359 )	ND( 0.362 )	ND( 0.368 )	ND( 0.368 )	ND( 0.368 )
	Conc., mg/dscm	ND( 0.268 )	ND( 0.302 )	ND( 0.317 )	ND( 0.312 )	ND( 0.298 )	ND( 0.283 )	ND( 0.296 )	ND( 0.296 )	ND( 0.298 )	ND( 0.283 )	ND( 0.296 )	ND( 0.296 )	ND( 0.296 )
	Conc., mg/dscm @ 7% O2	ND( 0.471 )	ND( 0.531 )	ND( 0.557 )	ND( 0.549 )	ND( 0.524 )	ND( 0.498 )	ND( 0.522 )	ND( 0.522 )	ND( 0.524 )	ND( 0.498 )	ND( 0.522 )	ND( 0.522 )	ND( 0.522 )
	Conc., mg/dscm @ 12% CO2	ND( 0.357 )	ND( 0.403 )	ND( 0.392 )	ND( 0.386 )	ND( 0.361 )	ND( 0.343 )	ND( 0.373 )	ND( 0.373 )	ND( 0.361 )	ND( 0.343 )	ND( 0.373 )	ND( 0.373 )	ND( 0.373 )
	Conc., ppmvd	ND( 0.091 )	ND( 0.102 )	ND( 0.107 )	ND( 0.106 )	ND( 0.101 )	ND( 0.096 )	ND( 0.101 )	ND( 0.101 )	ND( 0.101 )	ND( 0.096 )	ND( 0.101 )	ND( 0.101 )	ND( 0.101 )
	Absolute Difference, ppmvd (<0.2 required)		0.01		0.00		0.00					0.00		
	Conc., ppmvd @ 7% O2	ND( 0.160 )	ND( 0.180 )	ND( 0.189 )	ND( 0.186 )	ND( 0.178 )	ND( 0.169 )	ND( 0.177 )	ND( 0.177 )	ND( 0.178 )	ND( 0.169 )	ND( 0.177 )	ND( 0.177 )	ND( 0.177 )
	Conc., ppmvd @ 12% CO2	ND( 0.121 )	ND( 0.137 )	ND( 0.133 )	ND( 0.131 )	ND( 0.122 )	ND( 0.116 )	ND( 0.127 )	ND( 0.127 )	ND( 0.122 )	ND( 0.116 )	ND( 0.127 )	ND( 0.127 )	ND( 0.127 )
	Emission Rate, lb/hr	ND( 0.242 )	ND( 0.277 )	ND( 0.283 )	ND( 0.280 )	ND( 0.270 )	ND( 0.260 )	ND( 0.269 )	ND( 0.269 )	ND( 0.270 )	ND( 0.260 )	ND( 0.269 )	ND( 0.269 )	ND( 0.269 )
Clinker Rates (mtph and lbs/ton)	64.20	ND( 0.0037 )	68.40	ND( 0.0037 )	70.10	ND( 0.0034 )	ND( 0.0036 )	ND( 0.0036 )	70.10	ND( 0.0034 )	ND( 0.0036 )	ND( 0.0036 )	ND( 0.0036 )	

Holcim; Ada OK  
 Kiln 3 Main Stack  
 Run 1

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C	Ethylene (100,3000) 191C	H2O% (40) 191C	CO2% (40) 191C	
SPC__007098.LAB	01/08/24	15:21:30.639	1.210	-0.192	0.004	0.898	24.740	6.499	
SPC__007099.LAB	01/08/24	15:22:34.171	1.441	-0.157	0.005	0.773	24.305	6.706	
SPC__007100.LAB	01/08/24	15:23:38.038	0.843	-0.189	0.001	0.923	24.791	6.293	
SPC__007101.LAB	01/08/24	15:24:41.937	0.858	-0.184	-0.002	0.849	24.981	6.503	
SPC__007102.LAB	01/08/24	15:25:45.840	1.434	-0.189	0.003	0.807	24.605	6.619	
SPC__007103.LAB	01/08/24	15:26:49.785	0.997	-0.198	0.002	0.851	24.462	6.417	
SPC__007104.LAB	01/08/24	15:27:53.674	1.081	-0.198	0.003	0.830	24.793	6.267	
SPC__007105.LAB	01/08/24	15:28:57.611	0.979	-0.169	0.005	0.788	24.585	6.464	
SPC__007106.LAB	01/08/24	15:30:01.475	0.940	-0.186	0.002	0.812	24.619	6.322	
SPC__007107.LAB	01/08/24	15:31:05.400	0.944	-0.185	0.003	0.917	24.903	6.355	
SPC__007108.LAB	01/08/24	15:32:09.250	1.413	-0.178	0.001	0.846	24.685	6.683	
SPC__007109.LAB	01/08/24	15:33:13.127	1.464	-0.169	0.005	0.908	24.768	6.534	
SPC__007110.LAB	01/08/24	15:34:17.031	2.161	-0.194	-0.010	17.682	25.369	6.885	
SPC__007111.LAB	01/08/24	15:35:20.937	1.386	-0.195	0.002	1.058	24.785	6.544	
SPC__007112.LAB	01/08/24	15:36:24.942	0.944	-0.214	0.002	0.914	24.623	6.426	
SPC__007113.LAB	01/08/24	15:37:28.766	1.168	-0.204	0.001	1.028	24.997	6.570	
SPC__007114.LAB	01/08/24	15:38:32.726	1.008	-0.200	0.001	1.040	25.437	6.803	
SPC__007115.LAB	01/08/24	15:39:36.515	1.166	-0.181	-0.002	1.110	24.189	6.772	
SPC__007116.LAB	01/08/24	15:40:40.555	1.072	-0.176	-0.002	1.075	26.378	6.322	
SPC__007117.LAB	01/08/24	15:41:44.324	0.632	-0.176	-0.001	0.948	25.831	6.502	
SPC__007118.LAB	01/08/24	15:42:48.272	1.247	-0.179	-0.003	1.080	25.399	6.795	
SPC__007119.LAB	01/08/24	15:43:52.140	1.207	-0.198	0.005	0.940	25.263	7.870	
SPC__007120.LAB	01/08/24	15:44:56.276	1.287	-0.192	0.000	0.988	24.965	7.315	
SPC__007121.LAB	01/08/24	15:45:59.924	1.236	-0.216	0.001	0.950	24.999	6.883	
SPC__007122.LAB	01/08/24	15:47:03.870	1.448	-0.199	0.005	1.002	24.989	6.854	
SPC__007123.LAB	01/08/24	15:48:08.019	1.215	-0.205	0.002	0.934	24.925	6.813	
SPC__007124.LAB	01/08/24	15:49:11.761	1.161	-0.212	0.003	0.951	25.195	6.599	
SPC__007125.LAB	01/08/24	15:50:15.551	1.068	-0.174	-0.003	1.059	24.788	6.860	
SPC__007126.LAB	01/08/24	15:51:19.840	1.986	-0.180	-0.001	0.904	24.823	6.730	
SPC__007127.LAB	01/08/24	15:52:23.304	1.092	-0.206	0.001	0.949	24.909	6.726	
SPC__007128.LAB	01/08/24	15:53:27.208	0.949	-0.167	0.000	1.102	25.047	6.652	
SPC__007129.LAB	01/08/24	15:54:31.113	1.228	-0.190	0.002	0.934	24.938	6.798	
SPC__007130.LAB	01/08/24	15:55:35.152	1.645	-0.199	0.003	1.042	25.054	6.781	
SPC__007131.LAB	01/08/24	15:56:38.906	1.116	-0.200	0.003	1.021	25.402	6.567	
SPC__007132.LAB	01/08/24	15:57:43.195	1.014	-0.180	-0.001	1.158	24.431	6.831	
SPC__007133.LAB	01/08/24	15:58:46.822	1.589	-0.189	0.002	0.894	24.828	6.685	
SPC__007134.LAB	01/08/24	15:59:50.649	1.756	-0.212	0.003	0.911	25.005	6.581	
SPC__007135.LAB	01/08/24	16:00:54.509	1.329	-0.203	0.002	0.860	24.754	6.592	
SPC__007136.LAB	01/08/24	16:01:58.510	1.640	-0.190	0.003	0.797	24.629	6.591	
SPC__007137.LAB	01/08/24	16:03:02.346	1.823	-0.174	0.003	0.963	25.006	6.683	
SPC__007138.LAB	01/08/24	16:04:06.208	1.155	-0.188	0.002	0.882	24.933	6.679	
SPC__007139.LAB	01/08/24	16:05:10.106	1.539	-0.193	0.003	0.991	24.829	6.828	
SPC__007140.LAB	01/08/24	16:06:14.010	1.136	-0.188	0.002	1.069	25.057	6.745	
SPC__007141.LAB	01/08/24	16:07:17.996	1.659	-0.191	0.000	0.985	24.975	6.714	
SPC__007142.LAB	01/08/24	16:08:22.188	1.098	-0.177	0.003	1.026	25.166	6.788	
SPC__007143.LAB	01/08/24	16:09:25.794	1.618	-0.203	0.003	0.944	25.277	6.829	
SPC__007144.LAB	01/08/24	16:10:29.993	1.344	-0.198	0.002	0.840	25.148	6.861	
SPC__007145.LAB	01/08/24	16:11:33.546	0.907	-0.165	-0.003	1.014	24.859	6.871	
SPC__007146.LAB	01/08/24	16:12:37.397	1.370	-0.194	0.003	0.850	25.250	7.021	
SPC__007147.LAB	01/08/24	16:13:41.302	1.237	-0.182	-0.001	1.097	24.975	7.006	
SPC__007148.LAB	01/08/24	16:14:45.549	0.985	-0.185	0.002	1.040	25.507	7.047	
SPC__007149.LAB	01/08/24	16:15:49.218	0.834	-0.165	-0.001	1.132	25.097	7.063	
SPC__007150.LAB	01/08/24	16:16:53.003	1.088	-0.160	-0.001	1.148	24.830	7.218	
SPC__007151.LAB	01/08/24	16:17:57.170	1.527	-0.183	0.003	1.014	25.406	7.021	
SPC__007152.LAB	01/08/24	16:19:00.795	1.169	-0.171	-0.005	1.164	25.224	7.127	
SPC__007153.LAB	01/08/24	16:20:04.697	1.476	-0.183	0.002	0.974	25.112	7.136	
SPC__007154.LAB	01/08/24	16:21:08.598	1.027	-0.181	-0.004	1.125	25.035	7.029	
Run 1		(actual)	1.252	<	0.032	0.001	1.260	24.980	6.749
Oxygen	13.0%	(ppm,dry @7% O2)	2.933	<	0.075	0.003	2.951	24.9	M26A Moisture
DSCFM	243,450	(lbs/hr)	1.707	<	0.032	0.008	1.783		
Clinker (mtons/hr)	64.2	(lbs/ton clinker)	0.024	<	0.0005				

Holcim; Ada OK  
 Kiln 3 Main Stack  
 Run 2

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm(10) 191C	SF6 (10) 191C	Ethylene (100,3000) 191C	H2O% (40) 191C	CO2% (40) 191C
SPC__007176.LAB	01/08/24	16:45:25.334	1.653	-0.183	0.002	1.088	25.021	7.095
SPC__007177.LAB	01/08/24	16:46:29.184	1.556	-0.192	0.001	1.125	25.142	7.294
SPC__007178.LAB	01/08/24	16:47:33.464	1.192	-0.150	-0.003	1.272	24.966	7.150
SPC__007179.LAB	01/08/24	16:48:36.984	1.106	-0.171	-0.002	1.234	24.809	7.547
SPC__007180.LAB	01/08/24	16:49:40.878	0.981	-0.188	0.001	1.023	25.024	7.386
SPC__007181.LAB	01/08/24	16:50:44.777	1.238	-0.179	0.003	1.104	24.951	7.209
SPC__007182.LAB	01/08/24	16:51:48.759	1.200	-0.174	0.003	1.042	24.968	7.142
SPC__007183.LAB	01/08/24	16:52:52.616	1.208	-0.183	0.002	1.097	25.117	6.997
SPC__007184.LAB	01/08/24	16:53:56.577	0.724	-0.146	-0.002	1.180	24.585	7.051
SPC__007185.LAB	01/08/24	16:55:00.377	1.064	-0.189	-0.001	0.893	24.913	7.179
SPC__007186.LAB	01/08/24	16:56:04.277	1.047	-0.185	-0.001	0.935	25.088	7.237
SPC__007187.LAB	01/08/24	16:57:08.319	1.226	-0.178	-0.000	0.889	24.891	7.325
SPC__007188.LAB	01/08/24	16:58:12.121	1.302	-0.184	0.002	1.020	25.116	7.141
SPC__007189.LAB	01/08/24	16:59:16.016	1.224	-0.191	0.002	0.973	24.943	7.251
SPC__007190.LAB	01/08/24	17:00:19.917	1.327	-0.178	0.002	1.023	25.023	7.231
SPC__007191.LAB	01/08/24	17:01:23.812	1.377	-0.182	0.002	1.028	25.205	7.368
SPC__007192.LAB	01/08/24	17:02:28.051	1.420	-0.149	-0.005	1.290	24.343	7.516
SPC__007193.LAB	01/08/24	17:03:31.598	1.087	-0.181	-0.004	1.365	24.716	7.451
SPC__007194.LAB	01/08/24	17:04:35.473	0.990	-0.195	0.000	1.169	25.071	7.290
SPC__007195.LAB	01/08/24	17:05:39.641	0.926	-0.177	0.002	1.094	25.053	7.285
SPC__007196.LAB	01/08/24	17:06:43.262	1.254	-0.199	0.002	1.026	24.882	7.179
SPC__007197.LAB	01/08/24	17:07:47.207	1.461	-0.185	-0.001	0.958	24.875	7.060
SPC__007198.LAB	01/08/24	17:08:51.056	1.415	-0.191	0.001	0.932	24.593	7.112
SPC__007199.LAB	01/08/24	17:09:55.042	1.489	-0.174	0.004	0.892	24.945	7.075
SPC__007200.LAB	01/08/24	17:10:59.253	1.149	-0.182	0.001	0.926	24.689	7.293
SPC__007201.LAB	01/08/24	17:12:02.798	1.687	-0.202	0.002	0.925	24.522	7.220
SPC__007202.LAB	01/08/24	17:13:06.781	1.425	-0.198	0.002	0.953	24.734	7.183
SPC__007203.LAB	01/08/24	17:14:10.558	1.223	-0.193	0.002	0.995	24.760	7.327
SPC__007204.LAB	01/08/24	17:15:14.449	1.408	-0.197	0.006	1.003	24.978	7.241
SPC__007205.LAB	01/08/24	17:16:18.345	1.293	-0.166	0.000	0.976	24.924	7.298
SPC__007206.LAB	01/08/24	17:17:22.256	1.316	-0.177	0.000	1.035	24.800	7.446
SPC__007207.LAB	01/08/24	17:18:26.135	1.227	-0.199	0.001	1.048	25.080	7.296
SPC__007208.LAB	01/08/24	17:19:30.087	1.022	-0.203	-0.001	0.995	24.984	7.355
SPC__007209.LAB	01/08/24	17:20:33.978	1.087	-0.198	-0.001	1.128	24.877	7.415
SPC__007210.LAB	01/08/24	17:21:37.833	1.050	-0.203	-0.000	1.082	25.106	7.387
SPC__007211.LAB	01/08/24	17:22:41.727	0.544	-0.200	0.001	1.129	24.988	7.472
SPC__007212.LAB	01/08/24	17:23:46.008	1.291	-0.217	0.000	1.142	24.932	7.376
SPC__007213.LAB	01/08/24	17:24:49.875	0.973	-0.194	0.002	1.018	25.366	7.216
SPC__007214.LAB	01/08/24	17:25:53.415	1.368	-0.150	-0.001	1.208	24.219	7.508
SPC__007215.LAB	01/08/24	17:26:57.324	1.015	-0.200	0.001	0.994	24.669	7.333
SPC__007216.LAB	01/08/24	17:28:01.211	1.458	-0.191	0.002	0.940	24.971	7.233
SPC__007217.LAB	01/08/24	17:29:05.228	1.486	-0.201	0.001	0.984	24.950	7.259
SPC__007218.LAB	01/08/24	17:30:09.053	1.344	-0.199	0.001	1.006	24.980	7.319
SPC__007219.LAB	01/08/24	17:31:12.902	1.154	-0.198	0.000	1.048	24.639	7.508
SPC__007220.LAB	01/08/24	17:32:17.151	1.302	-0.194	0.001	1.104	25.181	7.285
SPC__007221.LAB	01/08/24	17:33:20.811	1.063	-0.167	-0.002	1.258	24.430	7.529
SPC__007222.LAB	01/08/24	17:34:24.639	1.458	-0.202	-0.000	1.184	25.182	7.253
SPC__007223.LAB	01/08/24	17:35:28.611	1.035	-0.168	-0.003	1.258	24.396	7.365
SPC__007224.LAB	01/08/24	17:36:32.396	1.307	-0.190	0.002	1.021	24.928	7.303
SPC__007225.LAB	01/08/24	17:37:36.352	0.795	-0.189	-0.001	0.914	24.875	7.292
SPC__007226.LAB	01/08/24	17:38:40.187	1.442	-0.208	-0.000	1.049	24.898	7.351
SPC__007227.LAB	01/08/24	17:39:44.090	0.862	-0.198	0.002	0.973	24.913	7.189
SPC__007228.LAB	01/08/24	17:40:48.030	1.344	-0.188	-0.000	1.017	24.667	7.403
SPC__007229.LAB	01/08/24	17:41:51.883	1.304	-0.212	0.002	1.020	24.637	7.246
SPC__007230.LAB	01/08/24	17:42:56.171	1.123	-0.209	-0.001	1.052	24.948	7.283
SPC__007231.LAB	01/08/24	17:43:59.725	1.190	-0.186	-0.002	1.034	24.724	7.397
<b>Run 2</b>		<b>(actual)</b>	<b>1.218</b>	<b>&lt;</b>	<b>0.032</b>	<b>0.000</b>	<b>1.055</b>	<b>24.878</b>
<b>Oxygen</b>	<b>13.0%</b>	<b>(ppm,dry @7% O2)</b>	<b>2.842</b>	<b>&lt;</b>	<b>0.075</b>	<b>0.001</b>	<b>2.461</b>	<b>M26A Moisture</b>
<b>DSCFM</b>	<b>239,100</b>	<b>(lbs/hr)</b>	<b>1.625</b>	<b>&lt;</b>	<b>0.032</b>	<b>0.003</b>	<b>1.461</b>	
<b>Clinker (mtons/hr)</b>	<b>68.4</b>	<b>(lbs/ton clinker)</b>	<b>0.022</b>	<b>&lt;</b>	<b>0.0004</b>			

Holcim; Ada OK  
 Kiln 3 Main Stack  
 Run 3

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C	Ethylene (100,3000) 191C	H2O% (40) 191C	CO2% (40) 191C
SPC__007251.LAB	01/08/24	18:05:56.094	1.203	-0.202	0.002	1.066	24.875	7.298
SPC__007252.LAB	01/08/24	18:07:00.023	1.415	-0.212	0.001	0.989	24.720	7.427
SPC__007253.LAB	01/08/24	18:08:03.941	1.331	-0.204	0.001	0.923	24.990	7.199
SPC__007254.LAB	01/08/24	18:09:08.029	1.732	-0.207	-0.000	1.101	24.642	7.470
SPC__007255.LAB	01/08/24	18:10:11.728	1.478	-0.216	-0.000	1.015	24.613	7.313
SPC__007256.LAB	01/08/24	18:11:15.626	1.080	-0.216	0.002	1.015	24.923	7.355
SPC__007257.LAB	01/08/24	18:12:19.486	1.410	-0.217	0.001	1.051	24.952	7.411
SPC__007258.LAB	01/08/24	18:13:23.431	1.366	-0.200	0.001	1.051	24.696	7.419
SPC__007259.LAB	01/08/24	18:14:27.279	1.369	-0.192	0.001	1.063	24.550	7.369
SPC__007260.LAB	01/08/24	18:15:31.179	1.530	-0.193	0.001	0.987	24.494	7.195
SPC__007261.LAB	01/08/24	18:16:35.118	1.324	-0.197	0.001	1.051	24.723	7.117
SPC__007262.LAB	01/08/24	18:17:39.005	1.072	-0.203	0.001	0.972	24.583	7.217
SPC__007263.LAB	01/08/24	18:18:42.875	1.276	-0.187	0.000	1.040	24.889	7.233
SPC__007264.LAB	01/08/24	18:19:46.769	1.522	-0.192	0.002	0.923	24.488	7.276
SPC__007265.LAB	01/08/24	18:20:50.697	1.252	-0.192	0.001	0.962	24.880	7.298
SPC__007266.LAB	01/08/24	18:21:54.614	1.460	-0.197	0.001	0.976	24.769	7.313
SPC__007267.LAB	01/08/24	18:22:58.855	1.088	-0.212	0.002	0.964	24.776	7.233
SPC__007268.LAB	01/08/24	18:24:02.612	1.150	-0.198	0.005	0.972	24.769	7.332
SPC__007269.LAB	01/08/24	18:25:06.360	1.228	-0.217	-0.000	0.960	24.931	7.426
SPC__007270.LAB	01/08/24	18:26:10.187	1.487	-0.199	0.004	0.984	24.740	7.316
SPC__007271.LAB	01/08/24	18:27:14.061	1.383	-0.193	0.002	1.037	24.989	7.306
SPC__007272.LAB	01/08/24	18:28:18.094	1.452	-0.196	0.001	0.949	24.775	7.595
SPC__007273.LAB	01/08/24	18:29:22.141	1.280	-0.205	0.002	1.007	24.766	7.408
SPC__007274.LAB	01/08/24	18:30:25.753	1.300	-0.192	0.001	1.027	24.973	7.368
SPC__007275.LAB	01/08/24	18:31:30.012	1.006	-0.206	0.004	1.061	25.021	7.438
SPC__007276.LAB	01/08/24	18:32:33.671	1.321	-0.183	0.002	1.065	25.104	7.500
SPC__007277.LAB	01/08/24	18:33:37.451	1.356	-0.198	0.002	1.147	25.209	7.433
SPC__007278.LAB	01/08/24	18:34:41.719	1.462	-0.207	0.003	1.014	24.826	7.458
SPC__007279.LAB	01/08/24	18:35:45.249	1.414	-0.154	-0.001	1.231	24.756	7.285
SPC__007280.LAB	01/08/24	18:36:49.157	1.355	-0.203	0.001	1.048	25.296	7.376
SPC__007281.LAB	01/08/24	18:37:53.049	1.193	-0.162	-0.004	1.290	24.730	7.561
SPC__007282.LAB	01/08/24	18:38:56.945	1.359	-0.207	0.001	1.015	24.802	7.438
SPC__007283.LAB	01/08/24	18:40:01.127	1.306	-0.219	0.000	1.076	25.036	7.418
SPC__007284.LAB	01/08/24	18:41:05.114	1.220	-0.173	-0.001	1.236	24.602	7.313
SPC__007285.LAB	01/08/24	18:42:08.675	1.508	-0.199	0.001	0.988	25.058	7.532
SPC__007286.LAB	01/08/24	18:43:12.881	1.184	-0.204	0.002	1.073	25.397	7.446
SPC__007287.LAB	01/08/24	18:44:16.826	0.553	-0.148	-0.001	1.177	24.519	7.486
SPC__007288.LAB	01/08/24	18:45:20.490	1.353	-0.203	0.001	1.036	25.322	7.417
SPC__007289.LAB	01/08/24	18:46:24.236	0.770	-0.159	-0.003	1.139	24.571	7.626
SPC__007290.LAB	01/08/24	18:47:28.511	1.058	-0.189	-0.000	1.102	25.351	7.381
SPC__007291.LAB	01/08/24	18:48:32.076	1.286	-0.202	-0.000	1.093	25.021	7.723
SPC__007292.LAB	01/08/24	18:49:36.270	0.969	-0.203	0.002	1.136	25.082	7.406
SPC__007293.LAB	01/08/24	18:50:39.835	1.243	-0.204	0.001	1.061	25.327	7.404
SPC__007294.LAB	01/08/24	18:51:43.729	1.057	-0.180	-0.005	1.294	24.332	7.704
SPC__007295.LAB	01/08/24	18:52:47.628	1.320	-0.195	0.001	1.132	25.430	7.264
SPC__007296.LAB	01/08/24	18:53:51.571	1.174	-0.166	0.000	0.996	25.125	7.607
SPC__007297.LAB	01/08/24	18:54:55.572	1.002	-0.178	0.002	1.114	25.270	7.537
SPC__007298.LAB	01/08/24	18:55:59.318	0.883	-0.210	0.004	1.171	25.092	7.419
SPC__007299.LAB	01/08/24	18:57:03.465	1.092	-0.205	-0.003	1.010	25.104	7.336
SPC__007300.LAB	01/08/24	18:58:07.149	1.474	-0.204	0.002	0.988	24.923	7.339
SPC__007301.LAB	01/08/24	18:59:11.011	0.941	-0.214	-0.001	1.018	25.028	7.209
SPC__007302.LAB	01/08/24	19:00:15.200	0.954	-0.214	-0.000	0.936	24.651	7.383
SPC__007303.LAB	01/08/24	19:01:18.905	1.535	-0.208	0.002	0.963	24.953	7.308
SPC__007304.LAB	01/08/24	19:02:22.750	1.608	-0.181	-0.001	0.973	24.654	7.428
SPC__007305.LAB	01/08/24	19:03:26.718	1.065	-0.204	0.002	1.066	24.990	7.393
SPC__007306.LAB	01/08/24	19:04:30.648	1.070	-0.206	0.002	1.041	25.027	7.628
Run 3		(actual)	1.255	<	0.032	1.050	24.895	7.395
Oxygen	13.0%	(ppm,dry @7% O2)	2.940	<	0.075	2.459		M26A Moisture
DSCFM	244,000	(lbs/hr)	1.716	<	0.032	1.489		
Clinker (mtons/hr)	70.1	(lbs/ton clinker)	0.022	<	0.0004			

**Appendix B**

**Field Data  
and  
CEM/FTIR Data**



### Cyclonic Flow Check Data Sheet

PLANT AND CITY		DATE	SAMPLING LOCATION		SAMPLE TYPE	RUN NUMBER			
Molten Ala OFE		1/8/24	KIT-3 Main Stack		Cyclonic Flow Check				
OPERATOR	Barometric Pressure (Pb) (In. Hg)	STATIC PRESS (in. H2O)	AMBIENT TEMP (deg. F)	STACK ID (in.)	PITOT Cp	DGM BOX No.	DGM delta H@	DGM CAL FACTOR (gamma)	PROBE ID NO
W/DGC	28.15	-0.25	48	139	0.84	M5-16	NA	NA	5B

#### EPA Method 2 Data

Run Time (24 hr)	Traverse Port ID	Pitot Delta P READING " H2O	STACK TEMP deg F	Absolute Angle at null (0) Delta P READING " H2O
1430	A 1	1.4	294	3
	2	1.2	295	2
	3	1.0	295	5
	B 1	1.4	294	1
	2	1.3	295	3
	7	1.0	295	1
	C 1	1.3	296	5
	2	1.1	295	7
	3	1.0	296	2
D	1	1.3	295	0
	2	1.1	296	2
	7	1.0	296	3
Pitot Leak Check		OK		
Averages				

Average of Absolute Angle Readings must be < 20 degrees

A

# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION			SAMPLE TYPE		RUN NUMBER		
Holcim; AQA		1/3/24		Method 26A			- ON OFF -M26A- 1A-				
OPERATOR	AMBIENT PRESS (in. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (In.)	PITOT Cp	PROBE LENGTH AND LINER TYPE	NOZZLE NUMBER	DIAMETER		
	28.15	-22	45	NA	139"	.875	5' effective glass	250	.250		
ASSUMED MOISTURE (%)	DGM H@	DGM CAL FACTOR (Y)	STACK THERM NO.	STACK PITOT NO.	ORSAT NO.	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	CO2 CONTENT %	K FACTOR		
25	1.65	.973	5A	5A	CEM	.001 CU.FT @ 10 "Hg	.001 CU.FT @ 10 "Hg	17	1.44		
TRaverse ELAPSED TIME (MIN)	CLOCK TIME (24-HR)	DGM READING Vm (cu. ft.)	VELOCITY HEAD (in. H2O)	delta H ORIFICE (in. H2O)	PROBE TEMP (deg. F)	STACK TEMP (deg. F)	DGM IN/OUT TEMP (deg. F)	OVEN TEMP (deg. F)	SIL GEL EXIT TEMP (deg. F)	SAMPLE TRAIN VAC (in. Hg)	
0	1521	436.416	1.24	2.0	250	293	57	247	65	9	
5		440.64	21.21.3	1.9	256	292	58	255	63	9	
10		444.78	.99	490	256	279	58	257	62	8	
15	1524	448.499	End of Port	1.47	End of Port						
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg					
B-1	15	1538	448.499	1.3	1.9	254	295	58	255	60	9
2	20		452.56	1.3	1.9	254	293	59	259	59	9
3	25		456.67	1.0	1.4	254	296	59	256	59	8
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg					
C-1	30	1555	460.792	1.3	1.9	255	297	59	254	55	9
2	35		464.93	1.2	1.7	253	297	59	254	54	9
3	40		468.88	.97	1.4	253	296	60	256	50	8
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg					
D-1	45	1612	472.537	1.2	1.7	254	296	60	254	47	9
2	50		476.43	1.0	1.4	258	294	60	256	47	8
3	55		480.07	.97	1.4	255	297	60	257	49	8
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg					
TOTAL TIME		60		End of Port		AVE SQRT delta P		AVE. TEMP.			
60 Min.		DGM VOLUME		AVE SQRT delta P		AVE. TEMP.		AVE. TEMP.			

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Belcimj Adg OK  
 Sample Location Kiln 3 Main Stack  
 Run No. ADA -M26A- 1A  
 Filter Number(s) Not Applicable

Sample Date 11/8/24  
 Recovery Date 11/8/24  
 Recovered by SCD

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight		914.7	880.9	633.2	768.0	747.2	997.8	g
Initial weight		777.5	772.7	598.7	753.7	744.0	974.6	g
Net weight		137.2	108.2	34.5	14.3	3.2	23.2	g

Description of impinger water clear 25 % spent  
B/W Sil gel color  
 Total moisture = 320.6 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA -M26A-H2SO4- 1A

Liquid level marked/sealed  661.8

NaOH Impingers contents and water rinse  
 container no. ADA -M26A-NaOH 1A

Liquid level marked/sealed  335.1

Samples stored and locked  
 Remarks Decal 100g 499.9g

# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION		SAMPLE TYPE		RUN NUMBER			
Holcim, Ada, Oklahoma		1 / 08 / 24		Method 26A		Method 26A		1 - ON OFF -M26A-1B			
OPERATOR	AMBIENT PRESS (In. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (In.)	PITOT Cp	PROBE LENGTH AND LINER TYPE		NOZZLE NUMBER	DIAMETER	
							5 ft glass effective				.250 .249 .250
Lee Harris		-1.25		N/A		0.845				.250	
ASSUMED MOISTURE (%)	DGM BOX No.	DGM H@	DGM CAL FACTOR (Y)	STACK THERM NO.	STACK PITOT NO.	ORSAT NO.	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	O2 CONTENT %	CO2 CONTENT %	K FACTOR
	MS-25	1A5	0.967	5A	5A	CEM FAILED	.001 CU.FT @ 10 "Hg	.001 CU.FT @ 9 "Hg	13	8	1-70
TRAVERSE PORT/ POINT NO.	ELAPSED TEST TIME (MIN)	CLOCK TIME (24-HR)	DGM READING Vm (cu. ft.)	VELOCITY HEAD (In. H2O)	delta H ORIFICE (In. H2O)	PROBE TEMP (deg. F)	STACK TEMP (deg. F)	DGM IN/OUT TEMP (deg. F)	FILTER OVEN TEMP (deg. F)	Sil Gel EXIT TEMP (deg. F)	SAMPLE TRAIN VAC (in. Hg)
	A-1	1624	0	1521	1.4	2.4	250	85	247	60	5
	2		5	622.67	1.3	2.2	250	84	255	55	5
	3		10	626.70	0.99	1.7	249	219	257	53	4
INTRA-PORT LEAK CHECK?											
DGM VOLUME (CU. FT)	INITIAL	FINAL	LEAK RATE: / CU.FT @ / CU.FT @		INCHES Hg / INCHES Hg						
B-1	15	1538	1.3	2.2	250	295	37	255	55	5	
2	20	634.43	1.3	2.2	250	293	39	259	53	5	
3	25	638.45	1.0	1.7	248	296	40	256	62	4	
INTRA-PORT LEAK CHECK?											
DGM VOLUME (CU. FT)	INITIAL	FINAL	LEAK RATE: / CU.FT @ / CU.FT @		INCHES Hg / INCHES Hg						
C-1	30	1553	1.3	2.2	252	290	412	254	56	6	
2	35	646.24	1.2	2.0	250	297	413	254	55	6	
3	40	650.19	0.97	1.6	250	296	415	256	57	5	
INTRA-PORT LEAK CHECK?											
DGM VOLUME (CU. FT)	INITIAL	FINAL	LEAK RATE: / CU.FT @ / CU.FT @		INCHES Hg / INCHES Hg						
D-1	45	1610	1.2	2.0	254	296	47	254	56	6	
2	50	653.695	1.0	1.7	251	296	49	250	52	6	
3	55	661.24	0.97	1.6	252	297	49	257	53	5	
INTRA-PORT LEAK CHECK?											
DGM VOLUME (CU. FT)	INITIAL	FINAL	LEAK RATE: / CU.FT @ / CU.FT @		INCHES Hg / INCHES Hg						
D-1	60	1627	1.2	2.0	254	296	47	254	56	6	
2	50	657.57	1.0	1.7	251	296	49	250	52	6	
3	55	661.24	0.97	1.6	252	297	49	257	53	5	
INTRA-PORT LEAK CHECK?											
DGM VOLUME (CU. FT)	INITIAL	FINAL	LEAK RATE: / CU.FT @ / CU.FT @		INCHES Hg / INCHES Hg						
TOTAL TIME			AVE SQRT delta P		AVE delta H		AVE TEMP.				
60 Min.											

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Holein; Ada OK  
 Sample Location Kih 3' Main stack  
 Run No. ADA -M26A- 1B  
 Filter Number(s) Not Applicable

Sample Date 11/8/24  
 Recovery Date 11/8/24  
 Recovered by SCS

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight	/	865.5	849.6	691.9	773.1	753.8	960.4	g
Initial weight	/	736.7	754.2	612.4	753.2	748.4	945.5	g
Net weight	/	128.8	95.4	39.7	11.9	5.4	114.9	g

Description of impinger water clean 75 % spent  
13/14 Sil gel color  
 Total moisture = 303.8 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA -M26A-H2SO4- 1B

Liquid level marked/sealed  696.5

NaOH Impingers contents and water rinse  
 container no. ADA -M26A-NaOH- 1B

Liquid level marked/sealed  380.5

Samples stored and locked \_\_\_\_\_  
 Remarks \_\_\_\_\_

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# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION			SAMPLE TYPE		RUN NUMBER	
Holcim;		1 / 6 / 24		Method 26A			- ON OFF -M26A-2A			
OPERATOR	AMBIENT PRESS (In. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (In.)	PITOT Cp	PROBE LENGTH AND LINER TYPE	NOZZLE NUMBER	DIAMETER	
D66	24.15	-1.25	45	NA	139	.89	5 effective/gles	250	.250	
ASSUMED MOISTURE (%)	DGM H@	DGM CAL FACTOR (Y)	STACK THERM NO.	STACK PITOT NO.	ORSAT NO.	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	CO2 CONTENT %	K FACTOR	
25	M5-16 1.65	.973	5A	5A	CEM	.001 CU.FT @ 10 "Hg	.001 CU.FT @ 10 "Hg	6	1.74	
TRaverse ELAPSED TEST TIME (MIN)	CLOCK TIME (24-HR)	DGM READING Vm (cu. ft.)	delta P VELOCITY HEAD (In. H2O)	delta H ORIFICE (In. H2O)	PROBE TEMP (deg. F)	STACK TEMP (deg. F)	DGM IN/OUT TEMP (deg. F)	FILTER OVEN TEMP (deg. F)	SAMPLE TRAIN VAC (in. Hg)	
A-1	0 1644	484.263	1.3	1.9	253	300	65	261	55 7	
2	5	488.36	1.1	1.6	252	302	65	256	54 6	
3	10	492.25	.95	1.4	251	302	66	254	51 6	
15	1659	495.902	End of Port							
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)	INITIAL FINAL	LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg						
B-1	15 1701	495.902	1.4	2.0	254	300	67	255	53 7	
2	20	500.16	1.3	1.9	254	306	67	257	53 7	
3	25	504.51	1.0	1.4	254	306	67	255	52 6	
30	1716	508.064	End of Port							
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)	INITIAL FINAL	LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg						
C-1	30 1718	508.064	1.3	1.9	252	301	68	254	50 7	
2	35	512.22	1.0	1.4	251	306	70	255	50 6	
3	40	515.96	1.0	1.4	250	297	71	257	50 6	
45	1733	519.677	End of Port							
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)	INITIAL FINAL	LEAK RATE: CU.FT @ CU.FT @		INCHES Hg INCHES Hg						
D-1	45 1735	519.677	1.2	1.7	256	303	73	254	51 7	
2	50	523.100	1.0	1.4	259	303	73	255	51 6	
3	55	527.31	.97	1.4	258	300	74	255	53 6	
60	1756	530.969	End of Port							
TOTAL TIME	DGM VOLUME	AVE SQRT delta P	AVE delta H	AVE TEMP.						
60 Min.										

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Molcim; Adg OK  
 Sample Location Kilm 3 Main Stack  
 Run No. ADA-M26A-2A  
 Filter Number(s) Not Applicable

Sample Date 11/8/24  
 Recovery Date 11/8/24  
 Recovered by GCJ

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight	/	933.0	862.2	691.7	782.2	762.8	955.8	g
Initial weight	/	774.3	761.9	669.4	776.6	761.4	942.5	g
Net weight	/	158.7	100.3	22.3	5.6	1.4	13.3	g

Description of impinger water clear 15 % spent  
B/w Sil gel color  
 Total moisture = 30.6 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA-M26A-H2SO4-2A

Liquid level marked/sealed  693.2

NaOH Impingers contents and water rinse  
 container no. ADA-M26A-NaOH-2A

Liquid level marked/sealed  783.8

Samples stored and locked \_\_\_\_\_  
 Remarks \_\_\_\_\_

# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION		SAMPLE TYPE		RUN NUMBER		
Holcim; <i>Adelphi, Oklahoma</i>		1 / 08 / 24		<i>Main stack</i>		Method 26A		2 - ON OFF -M26A-23		
OPERATOR	AMBIENT PRESS (in. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (in.)	PITOT Cp	PROBE LENGTH AND LINER TYPE	NOZZLE NUMBER	DIAMETER	
	28.15	-0.25	43	NA	139"	0.44	5 ft glass active	250	.250	
ASSUMED MOISTURE (%)	DGM H@	DGM CAL FACTOR (Y)	STACK THERM NO.	STACK PITOT NO.	ORSAT NO.	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	O2 CONTENT %	CO2 CONTENT %	K FACTOR
23	1.95	0.967	5A	5A	CEM	0.001 CU.FT @ 10 "Hg	0.001 CU.FT @ 13 "Hg	13	8	1.70
TRAVERSE ELAPSED TEST TIME (MIN)	CLOCK TIME (24-HR)	DGM READING Vm (cu. ft.)	delta P VELOCITY HEAD (in. H2O)	delta H ORIFICE (in. H2O)	PROBE TEMP (deg. F)	STACK TEMP (deg. F)	DGM IN / OUT TEMP (deg. F)	FILTER OVEN TEMP (deg. F)	Sil Gel EXIT TEMP (deg. F)	SAMPLE TRAIN VAC (in. Hg)
A-1	0 1644	665.311	1.3	2.2	250	300	39	261	53	8
2	5 669.07	669.07	1.1	1.9	253	302	42	256	52	7
3	10 672.99	672.99	0.95	1.0	251	302	44	254	53	6
15	1669	676.641	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL	FINAL	LEAK RATE: CU.FT @ CU.FT @	INCHES Hg	INCHES Hg				
B-1	15	1701	676.641	1.4	2.4	250	48	255	57	9
2	20	680.93	680.93	1.3	2.2	253	49	257	57	8
3	25	685.03	685.03	1.0	1.7	252	50	255	58	7
30	1716	688.695	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL	FINAL	LEAK RATE: CU.FT @ CU.FT @	INCHES Hg	INCHES Hg				
C-1	30	1716	688.695	1.3	2.2	251	48	254	57	9
2	35	692.85	692.85	1.0	1.7	255	50	255	59	7
3	40	696.57	696.57	1.0	1.7	258	52	257	60	7
45	1733	700.294	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port
INTRA-PORT LEAK CHECK? DGM VOLUME (CU. FT)		INITIAL	FINAL	LEAK RATE: CU.FT @ CU.FT @	INCHES Hg	INCHES Hg				
D-1	45	1735	700.294	1.2	2.0	252	50	254	60	8
2	50	704.24	704.24	1.0	1.7	254	51	255	62	7
3	55	707.91	707.91	0.97	1.0	251	52	255	63	7
60	1750	711.549	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port	End of Port
TOTAL TIME		DGM VOLUME		AVE SQRT delta P	AVE delta H	AVE TEMP.	AVE TEMP.			
60 Min.										

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Holcim ; Ada OK  
 Sample Location Kita 13 Main Stack  
 Run No. ADA -M26A- 2B  
 Filter Number(s) Not Applicable

Sample Date 11/8/24  
 Recovery Date 11/8/24  
 Recovered by SCS

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight		910.9	865.8	639.0	765.8	754.4	973.9	g
Initial weight		764.0	759.7	617.7	756.4	751.8	961.3	g
Net weight		146.9	106.1	21.3	9.4	2.6	12.6	g

Description of impinger water clear 10 % spent  
B/W Sil gel color  
 Total moisture = 298.9 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA -M26A-H2SO4- 2B

Liquid level marked/sealed  692.8

NaOH Impingers contents and water rinse  
 container no. ADA -M26A-NaOH 2B

Liquid level marked/sealed  387.8

Samples stored and locked \_\_\_\_\_  
 Remarks \_\_\_\_\_

# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION			SAMPLE TYPE		RUN NUMBER			
		1/20/24					Method 26A		- ON OFF - M26A-3A			
OPERATOR	AMBIENT PRESS (in. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (In.)	PITOT Cp	PROBE LENGTH AND LINER TYPE		NUMBER	DIAMETER		
Doo	28.15	- 25	45	NA	139	0.85	5' effective / glass		250	250		
75	1.065	1.973	5A	FA	CEM	2001	1.0	1.0	8	1.44		
PITOT LEAK CHECK				PASSED		FAILED						
TRaverse ELAPSED TEST TIME (MIN)	CLOCK TIME (24-HR)	DGM H@	DGM CAL FACTOR (Y)	delta P VELOCITY HEAD (in. H2O)	delta H ORIFICE (in. H2O)	DGM IN/OUT TEMP (deg. F)	STACK TEMP (deg. F)	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	FILTER OVEN TEMP (deg. F)	CO2 CONTENT %	K FACTOR
A-1	0	1605	531.201	1.3	1.9	75	298	2001	2001	260	64	5
2	5		535.49	1.2	1.7	76	300			262	63	4
3	10		539.47	.99	1.4	76	300			257	60	4
15	1820		543.135	End of Port								
INTRA-PORT LEAK CHECK?		INITIAL		LEAK RATE:	CU.FT @	INCHES Hg						
DGM VOLUME (CU. FT)		FINAL			CU.FT @	INCHES Hg						
B-1	15	1622	543.135	1.5	2.2	80	301			255	58	5
2	20		547.62	1.2	1.7	81	300			259	55	4
3	25		551.01	.99	1.4	82	300			256	53	4
30	1837		555.208	End of Port								
INTRA-PORT LEAK CHECK?		INITIAL		LEAK RATE:	CU.FT @	INCHES Hg						
DGM VOLUME (CU. FT)		FINAL			CU.FT @	INCHES Hg						
C-1	30	1839	555.208	1.4	2.0	81	300			256	53	5
2	35		559.43	1.2	1.7	81	302			252	52	4
3	40		563.34	1.1	1.6	81	299			255	52	4
45	1854		567.145	End of Port								
INTRA-PORT LEAK CHECK?		INITIAL		LEAK RATE:	CU.FT @	INCHES Hg						
DGM VOLUME (CU. FT)		FINAL			CU.FT @	INCHES Hg						
D-1	45	1854	567.145	1.3	1.9	80	300			255	50	5
2	50		571.31	1.0	1.4	82	300			256	49	4
3	55		574.98	.98	1.4	82	300			252	50	4
60	1911		578.634	End of Port								
TOTAL TIME		DGM VOLUME		AVE SQRT delta P	AVE delta H	AVE. TEMP.		AVE. TEMP.				
60 Min.												

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Medicin; ADA OK  
 Sample Location Kila 3 Main 5 tech  
 Run No. ADA -M26A- 3A  
 Filter Number(s) Not Applicable

Sample Date 11 8 124  
 Recovery Date 11 8 124  
 Recovered by SCJ

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight	/	970.4	827.7	679.5	761.2	748.1	972.9	g
Initial weight	/	759.0	755.1	670.4	758.3	748.6	961.0	g
Net weight	/	211.4	72.6	9.1	2.9	0.5	11.9	g

Description of impinger water clear 30 % spent  
BW Sil gel color  
 Total moisture = 308.4 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA -M26A-H2SO4- 3A

Liquid level marked/sealed  685.3

NaOH Impingers contents and water rinse  
 container no. ADA -M26A-NaOH 3A

Liquid level marked/sealed  358.6

Samples stored and locked \_\_\_\_\_  
 Remarks \_\_\_\_\_

# METHOD 26A FIELD DATA SHEET

PLANT AND CITY		DATE		SAMPLING LOCATION		SAMPLE TYPE		RUN NUMBER		
Holicim: Ada, Oklahoma		1 / 06 / 24		Mansfield		Method 26A		3 - ON OFF -M26A-3B		
OPERATOR	AMBIENT PRESS (in. Hg)	STATIC PRESSURE (in. Water)	AMBIENT TEMP (deg. F)	FILTER NUMBERS	STACK ID (in.)	PITOT Cp	PROBE LENGTH AND LINER TYPE	NOZZLE NUMBER	DIAMETER	
	28.15	-0.25	45	N/A	139"	0.845	Set glass effective	.250	.250	
ASSUMED MOISTURE (%)	DGM H@	DGM CAL FACTOR (Y)	STACK THERM NO.	STACK PITOT NO.	ORSAT NO.	LEAK CHECK (INITIAL)	LEAK CHECK (FINAL)	O2 CONTENT %	CO2 CONTENT %	
15	1.05	0.967	5A	5A	FAILED	.001 CU. FT @ 9 "Hg	.001 CU. FT @ 12 "Hg	13	8	
PITOT LEAK CHECK ----> PASSED										
TRaverse PORT / POINT NO.	ELAPSED TEST TIME (MIN)	CLOCK TIME (24-HR)	DGM READING Vm (cu. ft.)	delta P VELOCITY HEAD (in. H2O)	delta H ORIFICE (in. H2O)	PROBE TEMP (deg. F)	STACK TEMP (deg. F)	DGM IN / OUT TEMP (deg. F)	FILTER OVEN TEMP (deg. F)	SAMPLE TRAIN VAC (in. Hg)
A-1	0	1807	711.853	1.3	2.2	250	298	410	260	60
2	5		715.90	1.2	2.0	251	300	41	262	60
3	10		719.95	0.99	1.7	250	300	44	267	61
15	15	1920	723.594	End of Port						
INTRA-PORT LEAK CHECK?										
DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU. FT @ CU. FT @		INCHES Hg INCHES Hg				
B-1	15	1822	723.594	1.5	2.6	250	301	45	255	60
2	20		726.06	1.2	2.0	250	300	48	259	59
3	25		732.07	0.99	1.7	253	300	49	250	57
30	30	1937	735.728	End of Port						
INTRA-PORT LEAK CHECK?										
DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU. FT @ CU. FT @		INCHES Hg INCHES Hg				
C-1	30	1839	735.728	1.4	2.1	252	300	50	256	59
2	35		740.02	1.2	2.0	251	302	50	252	58
3	40		744.04	1.1	1.9	255	299	51	255	58
45	45	1954	747.939	End of Port						
INTRA-PORT LEAK CHECK?										
DGM VOLUME (CU. FT)		INITIAL FINAL		LEAK RATE: CU. FT @ CU. FT @		INCHES Hg INCHES Hg				
D-1	45	1856	747.939	1.3	2.2	250	300	50	255	59
2	50		752.04	1.0	1.7	250	300	50	250	60
3	55		755.71	0.96	1.7	250	300	48	252	60
60	60	1911	759.227	End of Port						
TOTAL TIME										
60 Min.		DGM VOLUME		AVE SQRT delta P		AVE. TEMP.		AVE. TEMP.		

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Holcim ; ADA OK  
 Sample Location Kita 3 Main Stack  
 Run No. ADA -M26A-3B  
 Filter Number(s) Not Applicable

Sample Date 11/8/24  
 Recovery Date 11/8/24  
 Recovered by LCG

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight		972.0	826.3	654.6	752.1	753.0	988.5	g
Initial weight		759.8	754.4	647.8	749.1	753.0	975.7	g
Net weight		212.2	71.9	6.8	3.0	0.0	12.8	g

Description of impinger water clear 100 % spent  
13/w Sil gel color  
 Total moisture = 306.4 grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. ADA -M26A-H2SO4- 3B

Liquid level marked/sealed  727.1

NaOH Impingers contents and water rinse  
 container no. ADA -M26A-NaOH 3B

Liquid level marked/sealed  361.5

Samples stored and locked \_\_\_\_\_  
 Remarks \_\_\_\_\_

**EPA METHOD 26A RECOVERY  
AND INTEGRITY DATA SHEET**

Plant Adf  
 Sample Location \_\_\_\_\_  
 Run No. ADA -M26A- FB  
 Filter Number(s) Not Applicable

Sample Date 11 8 124  
 Recovery Date 11 8 124  
 Recovered by SCS

**MOISTURE**

Impingers	1 50 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (knockout)	2 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	3 100 ml 0.1N H <sub>2</sub> SO <sub>4</sub> (tipped)	4 Optional Knockout (untipped)	5 100 ml 0.1N NaOH (untipped)	6 100 ml 0.1N NaOH (untipped)	Silica gel (untipped)	
Final weight	/	772.1	771.1	598.0	767.8	773.4	997.9	g
Initial weight	/	772.2	771.1	598.0	768.1	773.5	998.0	g
Net weight	/							g

Description of impinger water \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 Total moisture = 15/30 % spent  
30 Sil gel color  
 \_\_\_\_\_ grams

**RECOVERED SAMPLE**

H<sub>2</sub>SO<sub>4</sub> Impingers and knockout contents and water rinse  
 container no. -M26A-H2SO4-

Liquid level marked/sealed 421.8

NaOH Impingers contents and water rinse  
 container no. -M26A-NaOH

Liquid level marked/sealed 374.4

Samples stored and locked \_\_\_\_\_  
 Remarks From train LA

FV

Client: Holcim Ada Ok  
 Test Location: Kiln 3 Main Stack  
 Date: Jan 08 24 Start Time: 15:21:04  
 Run number 1  
 One Minute Averages

	O2 %,dry	CO2 %,dry
3:22:02 PM	13.2	8.5
3:23:02 PM	13.2	8.3
3:24:02 PM	13.2	8.2
3:25:02 PM	13.2	8.4
3:26:02 PM	13.2	8.5
3:27:02 PM	13.3	8.2
3:28:02 PM	13.4	8.1
3:29:02 PM	13.3	8.4
3:30:02 PM	13.4	8.2
3:31:02 PM	13.3	8.2
3:32:02 PM	13.2	8.5
3:33:02 PM	13.2	8.4
3:34:02 PM	12.7	8.9
3:35:02 PM	13.0	8.5
3:36:02 PM	13.3	8.3
3:37:02 PM	13.2	8.4
3:38:02 PM	13.0	8.6
3:39:02 PM	13.0	8.9
3:40:02 PM	13.1	8.5
3:41:02 PM	13.2	8.5
3:42:02 PM	13.4	8.4
3:43:02 PM	13.1	9.1
3:44:02 PM	12.8	10.2
3:45:02 PM	13.0	9.3
3:46:02 PM	13.1	8.8
3:47:02 PM	13.1	8.8
3:48:02 PM	13.1	8.8
3:49:02 PM	13.3	8.5
3:50:02 PM	13.2	8.7
3:51:02 PM	13.2	8.8
3:52:02 PM	13.3	8.5
3:53:02 PM	13.2	8.7
3:54:02 PM	13.3	8.7
3:55:02 PM	13.2	8.7
3:56:02 PM	13.2	8.7
3:57:02 PM	13.3	8.5
3:58:02 PM	13.3	8.8
3:59:02 PM	13.4	8.6
4:00:02 PM	13.5	8.5
4:01:02 PM	13.4	8.4
4:02:02 PM	13.5	8.4
4:03:02 PM	13.4	8.6
4:04:02 PM	13.4	8.6
4:05:02 PM	13.3	8.7
4:06:02 PM	13.2	8.7
4:07:02 PM	13.3	8.6
4:08:02 PM	13.3	8.7
4:09:02 PM	13.3	8.8
4:10:02 PM	13.3	8.9
4:11:02 PM	13.3	8.8
4:12:02 PM	13.2	8.9
4:13:02 PM	13.1	9.1
4:14:02 PM	13.0	9.1
4:15:02 PM	13.0	9.1
4:16:02 PM	13.0	9.2
4:17:02 PM	13.0	9.3
4:18:02 PM	13.0	9.1
4:19:02 PM	13.0	9.2
4:20:02 PM	13.0	9.2
4:21:02 PM	13.1	9.0
Run Avgs	13.2	8.7
Cal Gas	11.8	10.2
Initial Zero	0.3	0.1
Final Zero	0.2	0.1
Initial cal.	12.0	9.8
Final Cal.	12.0	9.8
Corrected Average	13.0	9.0

Client: Holcim Ada Ok  
 Test Location: Kiln 3 Main Stack  
 Date: Jan 08 24 Start Time: 16:44:16  
 Run number 2  
 One Minute Averages

	O2 %,dry	CO2 %,dry
4:45:14 PM	13.1	9.1
4:46:14 PM	13.0	9.3
4:47:14 PM	12.9	9.4
4:48:14 PM	13.0	9.3
4:49:14 PM	12.9	9.7
4:50:14 PM	13.0	9.4
4:51:14 PM	13.1	9.2
4:52:14 PM	13.2	9.1
4:53:14 PM	13.3	9.1
4:54:14 PM	13.4	9.0
4:55:14 PM	13.3	9.3
4:56:14 PM	13.2	9.3
4:57:14 PM	13.2	9.4
4:58:14 PM	13.2	9.2
4:59:14 PM	13.1	9.3
5:00:14 PM	13.1	9.3
5:01:14 PM	13.0	9.5
5:02:14 PM	12.9	9.6
5:03:14 PM	12.9	9.5
5:04:14 PM	12.9	9.6
5:05:14 PM	13.1	9.3
5:06:14 PM	13.2	9.3
5:07:14 PM	13.3	9.2
5:08:14 PM	13.5	9.0
5:09:14 PM	13.4	9.1
5:10:14 PM	13.4	9.2
5:11:14 PM	13.3	9.2
5:12:14 PM	13.3	9.2
5:13:14 PM	13.3	9.2
5:14:14 PM	13.1	9.4
5:15:14 PM	13.2	9.3
5:16:14 PM	13.1	9.4
5:17:14 PM	13.0	9.5
5:18:14 PM	13.0	9.4
5:19:14 PM	13.1	9.4
5:20:14 PM	13.0	9.5
5:21:14 PM	13.0	9.4
5:22:14 PM	12.9	9.6
5:23:14 PM	13.0	9.5
5:24:14 PM	13.0	9.4
5:25:14 PM	13.2	9.4
5:26:14 PM	13.0	9.6
5:27:14 PM	13.1	9.3
5:28:14 PM	13.2	9.3
5:29:14 PM	13.2	9.3
5:30:14 PM	13.1	9.4
5:31:14 PM	12.9	9.6
5:32:14 PM	12.9	9.4
5:33:14 PM	12.9	9.6
5:34:14 PM	13.0	9.4
5:35:14 PM	13.1	9.4
5:36:14 PM	13.1	9.4
5:37:14 PM	13.2	9.3
5:38:14 PM	13.1	9.5
5:39:14 PM	13.2	9.2
5:40:14 PM	13.2	9.5
5:41:14 PM	13.2	9.3
5:42:14 PM	13.2	9.3
5:43:14 PM	13.1	9.4
5:44:14 PM	13.1	9.4
Run Avgs	13.1	9.4
Cal Gas	11.8	10.2
Initial Zero	0.2	0.1
Final Zero	0.2	0.1
Initial cal.	12.0	9.8
Final Cal.	11.9	9.8
Corrected Average	13.0	9.7

Client: Holcim Ada Ok  
 Test Location: Kiln 3 Main Stack  
 Date: Jan 08 24 Start Time: 18:05:20  
 Run number 3  
 One Minute Averages

	O2 %,dry	CO2 %,dry
6:06:18 PM	13.2	9.3
6:07:18 PM	13.2	9.5
6:08:18 PM	13.3	9.3
6:09:18 PM	13.1	9.5
6:10:18 PM	13.2	9.4
6:11:18 PM	13.1	9.4
6:12:18 PM	13.1	9.5
6:13:18 PM	13.1	9.5
6:14:18 PM	13.1	9.4
6:15:18 PM	13.2	9.3
6:16:18 PM	13.3	9.1
6:17:18 PM	13.3	9.2
6:18:18 PM	13.3	9.3
6:19:18 PM	13.3	9.3
6:20:18 PM	13.3	9.3
6:21:18 PM	13.2	9.4
6:22:18 PM	13.3	9.3
6:23:18 PM	13.3	9.3
6:24:18 PM	13.2	9.5
6:25:18 PM	13.1	9.5
6:26:18 PM	13.2	9.4
6:27:18 PM	13.2	9.4
6:28:18 PM	13.0	9.7
6:29:18 PM	13.1	9.5
6:30:18 PM	13.0	9.5
6:31:18 PM	12.9	9.6
6:32:18 PM	12.9	9.6
6:33:18 PM	12.9	9.7
6:34:18 PM	13.0	9.5
6:35:18 PM	12.9	9.4
6:36:18 PM	13.0	9.5
6:37:18 PM	13.0	9.6
6:38:18 PM	12.9	9.6
6:39:18 PM	13.0	9.5
6:40:18 PM	13.0	9.5
6:41:18 PM	13.1	9.4
6:42:18 PM	13.0	9.7
6:43:18 PM	13.0	9.6
6:44:18 PM	13.1	9.6
6:45:18 PM	13.0	9.6
6:46:18 PM	12.9	9.7
6:47:18 PM	13.0	9.6
6:48:18 PM	13.0	9.8
6:49:18 PM	13.0	9.6
6:50:18 PM	13.0	9.6
6:51:18 PM	13.0	9.7
6:52:18 PM	13.0	9.7
6:53:18 PM	13.1	9.4
6:54:18 PM	12.9	9.9
6:55:18 PM	13.0	9.7
6:56:18 PM	13.1	9.4
6:57:18 PM	13.2	9.5
6:58:18 PM	13.3	9.4
6:59:18 PM	13.3	9.3
7:00:18 PM	13.2	9.4
7:01:18 PM	13.2	9.4
7:02:18 PM	13.2	9.5
7:03:18 PM	13.2	9.5
7:04:18 PM	13.0	9.7
7:05:18 PM	12.9	10.0
Run Avgs	13.1	9.5
Cal Gas	11.8	10.2
Initial Zero	0.2	0.1
Final Zero	0.2	0.1
Initial cal.	11.9	9.8
Final Cal.	11.9	9.8
Corrected Average	13.0	9.9

Holcim, Ada OK Kiln 3 Main Stack January 8, 2024 Operator *KS*

Cylinder ID	Gas Type	Value	Internal Response	Cal Error	Run No. 15:21-16:21			Run No. 2 17:44-18:44			Run No. 3 18:05-19:05		
					Pre Run Bias	Post Run Bias	Percent Bias	Pre Run Bias	Post Run Bias	Percent Bias	Pre Run Bias	Post Run Bias	Percent Bias
	O2 Zero	Zero N2	0.0%	0.00%	0.3%	0.2%	0.91%	0.2%	0.91%	0.2%	0.96%	0.2%	0.96%
XC025341	O2 Mid	11.8%	12.1%	1.28%	12.0%	12.0%	-0.46%	11.9%	-0.46%	11.9%	-0.91%	11.9%	-0.91%
ALM056015	O2 Span	21.9%	21.9%	0.00%									
	CO2 Zero	Zero N2	0.0%	0.00%	0.1%	0.1%	0.55%	0.1%	0.55%	0.1%	0.55%	0.1%	0.55%
XC025341	CO2 Mid	10.2%	9.9%	-1.65%	9.8%	9.8%	-0.55%	9.8%	-0.55%	9.8%	-0.55%	9.8%	-0.55%
ALM056015	CO2 Span	18.2%	18.2%	0.17%				zzzz					



**Holcim  
Ada OK  
Kiln 3 Main Stack  
HCN Analyte Spikes**

		01/08/24		01/08/24		01/08/24		01/08/24	
		15:02-15:15		16:20-16:34		17:43-17:56		19:03-19:15	
Date Time		Main Pre 1	Main Post 1	Main Post 2	Main Post 3				
		HCN	HCN	HCN	HCN				
<b>Cs</b>	Spike Direct, ppm	49.50	49.50	49.50	49.50				
	SF6 Tracer Direct, ppm	4.87	4.87	4.87	4.87				
<b>SF6</b>	Diluted SF6 Tracer, ppm	0.302	0.296	0.308	0.306				
	Diluted SF6 Tracer, ppm	0.289	0.272	0.298	0.296				
	Average Diluted SF6 Tracer, ppm	0.296	0.284	0.303	0.301				
<b>DF</b>	Dilution Ratio	16.49	17.15	16.08	16.18				
<b>Ct</b>	Total, ppm	4.023	4.440	4.100	4.354				
	Total, ppm	3.962	3.775	3.739	4.229				
	Average Total, ppm	3.993	4.108	3.920	4.292				
<b>Cn</b>	Pre Spike Native , ppm	0.855	1.476	1.123	1.070				
	Pre Spike Native , ppm	0.981	1.027	1.190	1.468				
	Post Spike Native , ppm	1.161	1.526	1.041	1.102				
	Post Spike Native , ppm	1.327	1.591	1.010	1.241				
	Average Native , ppm	1.081	1.405	1.091	1.220				
<b>Spike Recovery</b>		<b>99.1%</b>	<b>96.5%</b>	<b>94.1%</b>	<b>102.9%</b>				
<b>CTS Direct (CC426155)</b>									
Ethylene Expected (ppm)		75.47				75.48			
Ethylene Measured (ppm)		74.49				74.11			
<b>CTS Error</b>		<b>-1.3%</b>				<b>-1.8%</b>			

Holcim; Ada OK  
Kiln 3 Main Stack  
Pre Run 1 HCN Analyte Spike

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C
SPC__007080.LAB	01/08/24	15:00:48.214	0.649	-0.171	0.005
SPC__007081.LAB	01/08/24	15:01:52.365	0.855	-0.150	0.007
SPC__007082.LAB	01/08/24	15:02:56.012	0.981	-0.170	0.005
SPC__007083.LAB	01/08/24	15:03:59.911	2.049	-0.176	0.158
SPC__007084.LAB	01/08/24	15:05:03.971	4.023	-0.185	0.302
SPC__007085.LAB	01/08/24	15:06:07.711	3.962	-0.166	0.289
SPC__007086.LAB	01/08/24	15:07:11.637	4.389	-0.122	0.362
SPC__007087.LAB	01/08/24	15:08:15.496	0.566	-0.016	0.003
SPC__007088.LAB	01/08/24	15:09:19.730	0.621	-0.005	0.001
SPC__007089.LAB	01/08/24	15:10:23.658	1.403	-0.095	-0.004
SPC__007090.LAB	01/08/24	15:11:27.216	1.059	-0.145	0.003
SPC__007091.LAB	01/08/24	15:12:31.108	0.719	-0.166	0.001
SPC__007092.LAB	01/08/24	15:13:35.049	1.161	-0.154	0.004
SPC__007093.LAB	01/08/24	15:14:38.949	1.327	-0.161	0.004
SPC__007094.LAB	01/08/24	15:15:42.837	0.491	-0.130	-0.004

Holcim; Ada OK  
Kiln 3 Main Stack  
Post Run 1 HCN Analyte Spike

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C
SPC__007152.LAB	01/08/24	16:19:00.795	1.169	-0.171	-0.005
SPC__007153.LAB	01/08/24	16:20:04.697	1.476	-0.183	0.002
SPC__007154.LAB	01/08/24	16:21:08.598	1.027	-0.181	-0.004
SPC__007155.LAB	01/08/24	16:22:12.535	1.455	-0.188	0.003
SPC__007156.LAB	01/08/24	16:23:16.510	3.343	-0.191	0.213
SPC__007157.LAB	01/08/24	16:24:20.330	3.490	-0.199	0.306
SPC__007158.LAB	01/08/24	16:25:24.228	4.440	-0.193	0.296
SPC__007159.LAB	01/08/24	16:26:28.267	3.775	-0.009	0.272
SPC__007160.LAB	01/08/24	16:27:32.261	0.635	-0.012	0.002
SPC__007161.LAB	01/08/24	16:28:35.911	0.429	-0.010	0.000
SPC__007162.LAB	01/08/24	16:29:39.766	0.465	-0.037	-0.010
SPC__007163.LAB	01/08/24	16:30:43.682	0.858	-0.135	-0.014
SPC__007164.LAB	01/08/24	16:31:47.587	1.152	-0.144	-0.006
SPC__007165.LAB	01/08/24	16:32:51.527	1.526	-0.178	0.002
SPC__007166.LAB	01/08/24	16:33:55.767	1.591	-0.170	0.001

Holcim; Ada OK  
Kiln 3 Main Stack

Post Run 2 HCN Analyte Spike

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C
SPC__007229.LAB	01/08/24	17:41:51.883	1.304	-0.212	0.002
SPC__007230.LAB	01/08/24	17:42:56.171	1.123	-0.209	-0.001
SPC__007231.LAB	01/08/24	17:43:59.725	1.190	-0.186	-0.002
SPC__007232.LAB	01/08/24	17:45:03.578	1.208	-0.199	0.001
SPC__007233.LAB	01/08/24	17:46:07.560	2.036	-0.219	0.069
SPC__007234.LAB	01/08/24	17:47:11.373	4.100	-0.196	0.308
SPC__007235.LAB	01/08/24	17:48:15.316	3.739	-0.205	0.298
SPC__007236.LAB	01/08/24	17:49:19.274	5.517	-0.128	0.350
SPC__007237.LAB	01/08/24	17:50:23.198	0.471	-0.002	0.002
SPC__007238.LAB	01/08/24	17:51:27.070	0.158	-0.014	-0.003
SPC__007239.LAB	01/08/24	17:52:30.882	0.483	-0.048	-0.008
SPC__007240.LAB	01/08/24	17:53:35.143	1.566	-0.002	-0.011
SPC__007241.LAB	01/08/24	17:54:38.704	1.041	-0.187	0.000
SPC__007242.LAB	01/08/24	17:55:42.565	1.010	-0.194	0.002

Holcim; Ada OK  
Kiln 3 Main Stack  
Post Run 3HCN Analyte Spike

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C
SPC__007304.LAB	01/08/24	19:02:22.750	1.608	-0.181	-0.001
SPC__007305.LAB	01/08/24	19:03:26.718	1.065	-0.204	0.002
SPC__007306.LAB	01/08/24	19:04:30.648	1.070	-0.206	0.002
SPC__007307.LAB	01/08/24	19:05:34.394	1.468	-0.210	0.002
SPC__007308.LAB	01/08/24	19:06:38.293	1.170	-0.195	0.001
SPC__007309.LAB	01/08/24	19:07:42.454	2.709	-0.207	0.166
SPC__007310.LAB	01/08/24	19:08:46.087	4.354	-0.216	0.306
SPC__007311.LAB	01/08/24	19:09:49.993	4.229	-0.200	0.296
SPC__007312.LAB	01/08/24	19:10:53.870	5.223	-0.122	0.363
SPC__007313.LAB	01/08/24	19:11:57.761	0.273	0.005	0.003
SPC__007314.LAB	01/08/24	19:13:01.681	0.284	-0.041	-0.005
SPC__007315.LAB	01/08/24	19:14:05.616	1.102	-0.177	0.001
SPC__007316.LAB	01/08/24	19:15:09.479	1.241	-0.184	-0.004

Holcim; Ada OK  
Kiln 3 Main Stack  
Post Test CTS

Spectrum	Date	Time	Ada 1 HCN (200) PCA 191C 191c	HF ppm (10) 191C	SF6 (10) 191C	Ethylene (100,3000) 191C
SPC__007522.LAB	01/08/24	23:56:20.367	0.083	-0.004	-0.012	74.124
SPC__007523.LAB	01/08/24	23:57:24.309	0.280	-0.022	-0.012	74.097

Holcim; Ada OK  
 Kiln 3 Main Stack  
 CTS and HCN Direct

Spectrum	Date	Time	HCN PCA 191c R1 191c	HF ppm (10) 191C	SF6 (10) 191C	Ethylene (100,3000) 191C
SPC__006978BKG.LAB	01/08/24	13:03:27.740	0.000	0.000	0.000	0.000
SPC__006979.LAB	01/08/24	13:04:38.114	0.209	-0.006	0.000	-0.047
SPC__006980.LAB	01/08/24	13:05:42.014	-0.041	-0.004	0.001	-0.030
SPC__006981.LAB	01/08/24	13:06:45.963	0.098	0.010	0.001	-0.035
SPC__006982.LAB	01/08/24	13:07:49.933	-0.097	0.017	-0.017	33.540
SPC__006983.LAB	01/08/24	13:08:53.709	-0.086	0.020	-0.012	74.245
SPC__006984.LAB	01/08/24	13:10:01.011	0.144	-0.012	-0.011	74.550
SPC__006985.LAB	01/08/24	13:11:27.070	0.068	-0.017	-0.013	74.420
<b>Ethylene CTS (CC426155)</b>						<b>74.485</b>
SPC__006986.LAB	01/08/24	13:12:29.002	2.036	-0.005	0.098	69.297
SPC__006987.LAB	01/08/24	13:13:33.271	174.725	0.009	8.725	3.404
SPC__006988.LAB	01/08/24	13:14:36.760	195.377	-0.010	9.634	-0.741
SPC__006989.LAB	01/08/24	13:15:40.724	197.037	-0.023	9.622	-0.837
SPC__006990.LAB	01/08/24	13:16:44.828	196.782	0.000	9.611	-0.831
SPC__006991.LAB	01/08/24	13:17:47.379	195.568	0.000	9.584	-0.911
SPC__006992.LAB	01/08/24	13:18:48.780	185.321	0.011	9.526	-0.903
SPC__006993.LAB	01/08/24	13:19:52.818	182.620	0.009	9.482	-0.982
SPC__006994.LAB	01/08/24	13:20:56.671	106.647	-0.004	6.682	0.215
SPC__006995.LAB	01/08/24	13:22:00.754	49.487	0.012	4.881	-0.577
SPC__006996.LAB	01/08/24	13:23:04.470	49.579	0.005	4.871	-0.693
SPC__006997.LAB	01/08/24	13:24:08.340	49.447	-0.009	4.862	-0.613
<b>HCN Analyte Spike (CC768222)</b>			<b>49.504</b>		<b>4.871</b>	

## **Appendix C**

### **Ion Chromatography Analytical Report Data**

**Deeco, Inc.**  
3404 Lake Woodard Drive  
Raleigh, NC 27604

Project ID: 24-3326  
Holcim Ada OK

Hydrogen Fluoride & Chlorine

EPA Method 26A Analysis

Analytical Report  
41891



Element One, Inc.  
6319-D Carolina Beach Rd., Wilmington, NC 28412  
910-793-0128 FAX: 910-792-6853 [e1lab@e1lab.com](mailto:e1lab@e1lab.com)

The following data for Analytical Report 41891  
has been reviewed for completeness, accuracy,  
adherence to method protocol,  
and compliance with quality assurance guidelines.

Review by:

A handwritten signature in black ink, appearing to read 'Katie Gattis', with a large, stylized flourish at the end.

Katie Gattis, Quality Assurance Officer  
January 29, 2024

Report Reviewed and Finalized by:

A handwritten signature in black ink, appearing to read 'Ken Smith', with a long, sweeping horizontal flourish extending to the right.

Ken Smith, Laboratory Director  
January 29, 2024

**elementOne**

# **SUMMARY OF RESULTS**

## Summary of Analysis

### Summary of Method 26A Analysis

Element -----	ADA- M26A-R1A e41891-1 Total mg -----	ADA- M26A-R2A e41891-2 Total mg -----	ADA- M26A-R3A e41891-3 Total mg -----
HF	< 0.348	< 0.365	< 0.361
Cl <sub>2</sub>	< 0.335	< 0.384	< 0.359

Element -----	ADA- M26A-R1B e41891-4 Total mg -----	ADA- M26A-R2B e41891-5 Total mg -----	ADA- M26A-R3B e41891-6 Total mg -----
HF	< 0.367	< 0.365	< 0.383
Cl <sub>2</sub>	< 0.381	< 0.388	< 0.362

Element -----	ADA- M26A-FB e41891-7 Total mg -----
HF	< 0.222
Cl <sub>2</sub>	< 0.187

# **ANALYTICAL NARRATIVE**

elementOne

Certification: NJ NELAP NC009  
41891 Deeco M26A Report Packet  
Page 5 of 26

## Element One Analytical Narrative

Client:	Deeco, Inc.	Element One #:	41891
Client ID:	24-3326 Holcim Ada OK	Analyst:	LAW, MNB
Method:	M26A	Dates Received:	01.15.24
Analytes:	HF, Cl <sub>2</sub>	Dates Analyzed:	01.23-26.24

### Summary of Analysis

The samples were prepared and analyzed according to Method 26A protocol. The samples were analyzed for fluoride and chloride on a Metrohm 861/788 ion chromatograph system.

### Detection Limits

The Metrohm reporting limit was 0.1 µg/mL for fluoride and chloride.

### Analysis QA/QC

Duplicate analyses relative percent difference (RPD), spike recovery and second source verification data are summarized in the Quality Control section. All QA/QC data was within the criteria of the method.

### Additional Comments

The reported results have not been corrected for any blank values or spike recovery values. Due to the sample matrix, it was necessary to analyze all samples at a minimum five-fold dilution to reduce interferences and to preserve the anion column. Due to matrix interference, it was necessary to analyze the field samples for Cl<sub>2</sub> at a minimum ten-fold dilution. The reported results relate only to the items tested or calibrated.

# **QUALITY CONTROL SUMMARY**

## Summary of Quality Control Data

### Summary of Method 26A Duplicate Analysis RPD

*(Method 26A QC limits: <5% for RPD)*

Element	ADA- M26A-R1A RPD	ADA- M26A-R2A RPD	ADA- M26A-R3A RPD
HF	NA	NA	NA
Cl <sub>2</sub>	NA	NA	NA

Element	ADA- M26A-R1B RPD	ADA- M26A-R2B RPD	ADA- M26A-R3B RPD
HF	NA	NA	NA
Cl <sub>2</sub>	NA	NA	NA

Element	ADA- M26A-FB RPD
HF	NA
Cl <sub>2</sub>	NA

### Summary of Method 26A Spike Recoveries

*(Method 26A QC limits: 90-110% for Spike Recoveries)*

Element	ADA- M26A-R3A Recovery	ADA- M26A-R3B Recovery
HF	109%	103%
Cl <sub>2</sub>	98%	106%

### Second Source Calibration Verification

*(\*Laboratory QC limits: 90-110%)*

Element	DL 0.1mg/L Recovery	*QC 5.0mg/L Recovery
HF	104%	102%
Cl <sub>2</sub>	110%	101%

# **SAMPLE CUSTODY**

elementOne

Certification: NJ NELAP NC009  
41891 Deeco M26A Report Packet  
Page 9 of 26

286  
 1.16.24  
 41891  
 41873  
 Date: 1/12/24  
 Lab: Element One  
 Train: EPA Method 26A

**DEECO, Inc**  
 3404 Lake Woodard Dr.  
 Raleigh, NC 27604  
 919-250-0285

Plant Name: Holcim		Plant Location: Ada OK		Project Name: 24-3326	
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Analysis Required	Sampling Train	Date/Time
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Analysis Required	Sampling Train	Date/Time
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Analysis Required	Sampling Train	Date/Time
Field Sample No.	Date	Composite or Grab	Analysis Required	Sampling Train	Sample Description
ADA-1A-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses Final Volume 661.8 mL
ADA-1A-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses Final Volume 335.1 mL Sodium thiosulfate not added
ADA-1B-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses Final Volume 696.5 mL
ADA-1B-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses Final Volume 380.5 mL Sodium thiosulfate not added
ADA-2A-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses Final Volume 693.2 mL
ADA-2A-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses Final Volume 383.8 mL Sodium thiosulfate not added

Samples received in good condition in Env. Express containers. No empty containers.

**DEECO, Inc**  
 3404 Lake Woodard Dr.  
 Raleigh, NC 27604  
 919-250-0285

250 1-10-24 41891 41893  
 Date: 1/12/24  
 Lab: Element One  
 Train: EPA Method 26A

Plant Name: Holcim		Plant Location: Ada OK		Project Name: 24-3326			
Relinquished by: (Signature)	Date	Composite or Grab	Analysis Required	Sampling Train	Sample Description		
Received by: (Signature)							
Received by: (Signature)							
Received by: (Signature)							
Field Sample No.	Date	Composite or Grab	Analysis Required	Sampling Train	Sample Description	Special Notes	Lab
ADA-2B-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses	Final Volume 692.8 mL	Element One
ADA-2B-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses	Final Volume 387.8 mL Sodium thiosulfate not added	Element One
ADA-3A-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses	Final Volume 685.3 mL	Element One
ADA-3A-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses	Final Volume 358.6 mL Sodium thiosulfate not added	Element One
ADA-3B-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses	Final Volume 727.1 mL	Element One
ADA-3B-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses	Final Volume 361.5 mL Sodium thiosulfate not added	Element One

**DEECO, Inc**  
 3404 Lake Woodard Dr.  
 Raleigh, NC 27604  
 919-250-0285

228 11824 41891 41875  
 Date: 1/12/24  
 Lab: Element One  
 Train: EPA Method 26A

Plant Name: Holcim  
 Plant Location: Ada OK  
 Project Name: 24-3326

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Comments			
		<i>Loa B...</i>	1/15/24				
Field Sample No.	Date	Composite or Grab	Analysis Required	Sampling Train	Sample Description	Special Notes	Lab
ADA-FB-H <sub>2</sub> SO <sub>4</sub>	01/08/24	Comp.	Fluoride ion as Hydrogen Fluoride	EPA Method 26A	0.1N H <sub>2</sub> SO <sub>4</sub> and DI Rinses	Final Volume 421.8 mL	Element One
ADA-FB-NaOH	01/08/24	Comp.	Chloride ion as Diatomic Chlorine	EPA Method 26A	0.1N NaOH and DI Rinses	Final Volume 374.4 mL Sodium thiosulfate not added	Element One

# **ANALYTICAL DATA**

## Analytical Calculations

**HF -**

$$\text{Total HX (mg)} = \frac{[\text{X Results } (\mu\text{g/mL}) * \text{Dilution} * \text{Beginning Vol (mL)}] * \text{Correction Factor}}{1000}$$

**Where-**

X Results= Raw sample concentration (ppm) — *IC Data Sheet*

Dilution=  $\frac{\text{Diluted Volume}}{\text{Aliquot}}$  — *IC Run Sheet*

Beginning Volume--*Sample Submission*

1.053= Correction factor for hydrogen fluoride

**Cl<sub>2</sub> -**

$$\text{Total X}_2 \text{ (mg)} = \frac{\text{X Results } (\mu\text{g/mL}) * \text{Dilution} * \text{Beginning Volume (mL)}}{1000}$$

**Where-**

X Results= Raw sample concentration (ppm)—*Cl<sub>2</sub> IC Data Sheet*

Dilution=  $\frac{\text{Diluted Volume}}{\text{Aliquot}}$  — *IC Run Sheet*

Beginning Volume--*Sample Submission*

## Analytical Calculations

### Spike Recovery-

$$\text{Spike (\%)} = \frac{(\text{Spiked Result } (\mu\text{g/mL}) - \text{Sample Result } (\mu\text{g/mL}))}{\text{Spike Amount } (\mu\text{g/mL})} \times 100$$

### Where-

Spike Result = Raw sample concentration (ppm)--*IC-Data Sheet*

Sample Result = Raw sample concentration (ppm)--*IC-Data Sheet*

Spike Amount—*IC-Data Sheet*

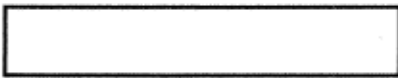
### Duplicate Analysis RPD-

$$\text{RPD (\%)} = \frac{(\text{Duplicate Result } (\mu\text{g/mL}) - \text{Sample Result } (\mu\text{g/mL}))}{\text{Average } (\mu\text{g/mL})} \times 100$$

### Where-

Sample Result and Duplicate Results=Raw sample concentration (ppm)--*IC-Data Sheet*

$$\text{Average} = \frac{(\text{Duplicate} + \text{Sample Results})}{2}$$



Analysis Due Date 01.23.24  
QA/QC/Report Due Date 01.25.24

Client: Deeco, Inc.  
Project No 24-3326

Date Rec 01.15.24  
Time Rec 1100

Volume Marked	Volume Loss	FH pH < 2	BH pH > 8	Ref. Method: 26A
Y N	Y N ?	Y N	Y N	

**Sample Identification**

1	ADA-M26A-R1A	4	ADA-M26A-R1B	7	ADA-M26A-FB
2	ADA-M26A-R2A	5	ADA-M26A-R2B		
3	ADA-M26A-R3A	6	ADA-M26A-R3B		
	ADA-M26A-R3A Spike		ADA-M26A-R3B Spike		

Analyses Requested	Samples 1-7	HF
	Samples 1-7	Cl <sub>2</sub>

**Runs/FB**

Lab ID	FH Impinger 1 (or Combined Imp)		FH Impinger 2		FH Impinger 3		BH Impinger 4 (or Combined Imp)		BH Impinger 5	
	BV, ml	FV, ml	BV, ml	FV, ml	BV, ml	FV, ml	BV, ml	FV, ml	BV, ml	FV, ml
1	661.8						335.1			
2	693.2						383.8			
3.S	685.3						358.6			
4	696.5						380.5			
5	692.8						387.8			
6.S	727.1						361.5			
7	421.8						374.4			

**Reagent Blanks**

Lab ID	Fractions	BV, ml	FV, ml	Notes
	0.1 N H <sub>2</sub> SO <sub>4</sub>			
	0.1 N NaOH			
	DI H <sub>2</sub> O			

**Lab Communications**

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Rec Runs/FB: H2SO4; NaOH; No RB received--01.15.24 LLB

SS Page 1 of 1  
SS by RLB  
1/18/2024 4:22:46 PM

Imp 1, 2, & 3 Prep By / Date LAB 01-23-24  
Imp 4 & 5 Prep By / Date LAB 1-25-24  
Labeled By/Date RLB 1-18-24  
ID Verification By/Date RLB 1-18-24

elementOne

M26A-HF IC Data Sheet

Lab ID #: 41891

Client: Deeco

Column: IonPac AS14A

Date: 01.24.24

Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0 mM NaHCO<sub>3</sub>

Analyst: LAW

Flow Rate: 1.0 mL/min.

Detection Limit, (µg/ml): 0.10

F to HF factor: 1.053

Sample ID	F µg/ml	Dilution	Final Vol, ml	HF, Total mg	Spike, µg/ml	% Recovery/ RPD	File Name	Date Time
LRB	0.00	1	10	< 0.001			_2024-01-23_	1/23/2024 20:14
LRB	0.00	1	10	< 0.001		NA	_2024-01-23_	1/23/2024 20:33
LRB SPK	4.91	1	10	0.052	5.00	98%	_2024-01-23_	1/23/2024 20:51
LRB SPK	4.92	1	10	0.052	5.00	98%	_2024-01-23_	1/23/2024 21:10
41891-1	0.00	5	661.8	< 0.348			_2024-01-23_	1/23/2024 21:29
41891-1 DUP	0.00	5	661.8	< 0.348		NA	_2024-01-23_	1/23/2024 21:48
41891-2	0.035	5	693.2	< 0.365			_2024-01-24_	1/24/2024 0:36
41891-2 DUP	0.033	5	693.2	< 0.365		NA	_2024-01-24_	1/24/2024 0:55
41891-3	0.00	5	685.3	< 0.361			_2024-01-23_	1/23/2024 22:06
41891-3 DUP	0.00	5	685.3	< 0.361		NA	_2024-01-23_	1/23/2024 22:06
41891-3 SPK	5.47	5	685.3	19.7	5.00	109%	_2024-01-23_	1/23/2024 22:25
41891-3 SPK DUP	5.46	5	685.3	19.7	5.00	109%	_2024-01-23_	1/23/2024 22:44
41891-4	0.00	5	696.5	< 0.367			_2024-01-23_	1/23/2024 23:03
41891-4 DUP	0.00	5	696.5	< 0.367		NA	_2024-01-24_	1/24/2024 1:14
41891-5	0.00	5	692.8	< 0.365			_2024-01-24_	1/24/2024 1:32
41891-5 DUP	0.00	5	692.8	< 0.365		NA	_2024-01-24_	1/24/2024 1:51
41891-6	0.00	5	727.1	< 0.383			_2024-01-24_	1/24/2024 2:10
41891-6 DUP	0.00	5	727.1	< 0.383		NA	_2024-01-24_	1/24/2024 2:29
41891-6 SPK	10.1	5	727.1	38.6	10.0	101%	_2024-01-24_	1/24/2024 2:47
41891-6 SPK DUP	10.5	5	727.1	40.1	10.0	105%	_2024-01-24_	1/24/2024 3:06
41891-7 FB	0.00	5	421.8	< 0.222			_2024-01-24_	1/24/2024 3:25
41891-7 FB DUP	0.00	5	421.8	< 0.222		NA	_2024-01-24_	1/24/2024 4:59



elementOne

M26A-HF IC Data Sheet

Lab ID #: 41891

Client: Deeco

Column: IonPac AS14A

Date: 01.24.24

Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0 mM NaHCO<sub>3</sub>

Analyst: LAW

Flow Rate: 1.0 mL/min.

Detection Limit, (µg/ml): 0.10

F<sup>-</sup> to HF factor: 1.053

Standards	F <sup>-</sup> µg/ml	Dilution	QC, µg/ml	% Relative Error	% Recovery	File Name	Date Time
0.0	0.00					_2024-01-23_	1/23/2024 16:29
0.1	0.100			0.0%	100%	_2024-01-23_	1/23/2024 16:48
1.0	1.01			1.2%	101%	_2024-01-23_	1/23/2024 17:07
3.0	2.92			-2.6%	97%	_2024-01-23_	1/23/2024 17:25
5.0	5.09			1.8%	102%	_2024-01-23_	1/23/2024 17:44
10.0	9.98			-0.3%	100%	_2024-01-23_	1/23/2024 18:03
0.1	0.110			10.0%	110%	_2024-01-24_	1/24/2024 7:10
1.0	1.03			2.5%	103%	_2024-01-24_	1/24/2024 7:28
3.0	2.99			-0.5%	100%	_2024-01-24_	1/24/2024 7:47
5.0	5.15			3.0%	103%	_2024-01-24_	1/24/2024 8:06
10.0	10.3			3.2%	103%	_2024-01-24_	1/24/2024 8:25
<b>Correlation-</b>	0.9998						
QC	5.09		5.00		102%	_2024-01-23_	1/23/2024 18:22
QC	4.73		5.00		95%	_2024-01-23_	1/23/2024 18:40
QC	5.24		5.00		105%	_2024-01-23_	1/23/2024 23:21
QC	4.89		5.00		98%	_2024-01-23_	1/23/2024 23:40
QC	5.23		5.00		105%	_2024-01-24_	1/24/2024 3:44
QC	5.03		5.00		101%	_2024-01-24_	1/24/2024 4:02
QC	4.53		5.00		91%	_2024-01-24_	1/24/2024 6:13
QC	5.14		5.00		103%	_2024-01-24_	1/24/2024 8:43
DL	0.104		0.10		104%	_2024-01-23_	1/23/2024 19:37
DL	0.110		0.10		110%	_2024-01-23_	1/23/2024 19:55
DL	0.108		0.10		108%	_2024-01-24_	1/24/2024 6:32
DL	0.109		0.10		109%	_2024-01-24_	1/24/2024 9:02
36738-5 QC	6.73	1	6.96		102%	_2024-01-24_	1/24/2024 5:36
36728-5 QC DUP	6.34	1	6.96		96%	_2024-01-24_	1/24/2024 5:55
BLK	0.00					_2024-01-23_	1/23/2024 18:59
BLK	0.00					_2024-01-23_	1/23/2024 19:18
BLK	0.00					_2024-01-24_	1/24/2024 0:18
BLK	0.00					_2024-01-24_	1/24/2024 4:40
BLK	0.00					_2024-01-24_	1/24/2024 6:51
BLK	0.00					_2024-01-24_	1/24/2024 9:21

elementOne

IC Sample Sheet/Digestion Worksheet

Lab ID #: 41891

Date: 01.23.24  
 Analyst: WJ  
 Batch name: 012324-41891

Column: IonPac AS14A  
 Conc. Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0mM NaHCO<sub>3</sub>  
 10mL Conc. Eluent Diluted to FV=1L with filtered UPDI  
 Regenerant: 100 mM H<sub>3</sub>PO<sub>4</sub>  
 Flow Rate: 1.0 mL/min.

Instrument: 861788  
 Lot# 1C11706-1  
 Lot # 1C11-117-2  
 Method: 300/26A

AS LOC.	Sample ID	Client	Analyte	Results (ug/mL)	Results (ug/mL)	Dilution	Wt (g) / FV (mL)
1	00			GC	max	0.2	
2	6.1		F	BBC70524	Supp. Alkal.	0.9998	
3	1.0						
4	30						
5	50						
6	10.0						
7	GC						
8	GC						
9	BK						
10	BK						
11	DL						
12	DL						
13	LES						
14	LES						
15	LEBT						
16	LEBT						
17	41891-1	Deeco	HF		—	5X	
18	-1D	↓	↓		—	↓	
19	-3	↓	↓		—	↓	
20	-3D	↓	↓		—	↓	
21	-3+	↓	↓		5.465	↓	
22	-3+D	↓	↓		5.456	↓	
23	GC						
24	GC						
25	BK Backflow						

Manual integrations noted by M

Curve IC Lot # 1C11-118-1 Comments: pg 10F3  
 Spike 50 uL from 1000 ug/mL Std. to 10mL sample Lot #'s: IC ME Solution 2308024-250115 IC NO2 Solution 2308942-250115  
 QC: Spike 50 uL from 1000 ug/mL F, Cl, Br, and SO<sub>2</sub> Std. to 10mL sample; lot #'s listed above.  
 QC: Spike 20 uL from 1000 ug/mL NO<sub>2</sub>, NO<sub>3</sub>, and PO<sub>4</sub> Std. to 10mL sample; lot #'s listed above.  
 Submitted for QC- Date: 01.24.23 Time: 09:46 By: WJ QC Review- Date: \_\_\_\_\_ Time: \_\_\_\_\_ By: \_\_\_\_\_

elementOne

IC Sample Sheet/Digestion Worksheet

Lab ID #: 41891

Date: 01-23-24  
 Analyst: LWS  
 Batch name: 02324-41891

Column: IonPac AS14A  
 Conc. Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0mM NaHCO<sub>3</sub>  
 10mL Conc. Eluent Diluted to FV=1L with filtered UPDI  
 Regenerant: 100 mM H<sub>3</sub>PO<sub>4</sub>  
 Flow Rate: 1.0 mL/min.

Instrument: 861/788  
 Lot# 1C11-1007  
 Lot # 1C11-172  
 Method: 300/26A

AS LOC.	Sample ID	Client	Analyte	Results (ug/mL)	Results (ug/mL)	Dilution	Wt (g) / FV (mL)
26	BLK						
27	41891-2	Deeco	HF		---	5X	
28	-2D	↓	↓		---	↓	
29	-4				---		
30	-4D				---		
31	-5				---		
32	-5D				---		
33	-6				---		
34	-6D				---		
35	-6+				10ppm STX		10.082
36	-6+D						10161
37	GC						
38	GC						
39	BLK						
40	BLK						
41	41891-7FB	Deeco	HF		---	5X	
42	-7FBD	↓	↓		---	↓	
43	36738-5 -GC		↓		6.730	1X	TV= 696
44	-5-GC		↓		6.339	↓	
45	GC						
46	D						
47	BLK						
48	0.1						
49	1.0						
50	30						

Manual integrations noted by M

Curve IC Lot #

Comments: pg 20F3

Spike 50 uL from 1000 ug/mL Std. to 10mL sample Lot #'s: IC ME Solution \_\_\_\_\_ IC NO2 Solution \_\_\_\_\_

QC: Spike 50 uL from 1000 ug/mL F, Cl, Br, and SO<sub>4</sub> Std. to 10mL sample; lot #'s listed above.

QC: Spike 20 uL from 1000 ug/mL NO<sub>2</sub>, NO<sub>3</sub>, and PO<sub>4</sub> Std. to 10mL sample; lot #'s listed above.

Submitted for QC- Date: \_\_\_\_\_ Time: \_\_\_\_\_ By: \_\_\_\_\_ QC Review- Date: \_\_\_\_\_ Time: \_\_\_\_\_ By: \_\_\_\_\_

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M26A-Cl<sub>2</sub> IC Data Sheet

Lab ID #: 41891

Client: Deeco

Date: 01.29.24

Analyst: LAW/MNB

Detection Limit, (µg/ml): 0.10

Column: IonPac AS14A

Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0 mM NaHCO<sub>3</sub>

Flow Rate: 1.0 mL/min.

Sample ID	Cl <sup>-</sup> µg/ml	Dilution	Final Vol, ml	Cl <sub>2</sub> , Total mg	Spike, µg/ml	% RPD/ Recovery	File Name	Date Time
LRB	0.015	1	10	< 0.001			_2024-01-25_	1/25/2024 23:18
LRB	0.009	1	10	< 0.001		NA	_2024-01-25_	1/25/2024 23:46
LRB SPK	5.115	1	10	0.051	5.00	102%	_2024-01-26_	1/26/2024 0:13
LRB SPK	5.174	1	10	0.052	5.00	103%	_2024-01-26_	1/26/2024 0:41
41891-1	-0.008	10	335.1	< 0.335			_2024-01-26_	1/26/2024 1:09
41891-1 DUP	-0.015	10	335.1	< 0.335		NA	_2024-01-26_	1/26/2024 1:36
41891-2	-0.015	10	383.8	< 0.384			_2024-01-26_	1/26/2024 2:04
41891-2 DUP	-0.013	10	383.8	< 0.384		NA	_2024-01-26_	1/26/2024 2:32
41891-3	-0.017	10	358.6	< 0.359			_2024-01-26_	1/26/2024 5:46
41891-3 DUP	-0.019	10	358.6	< 0.359		NA	_2024-01-26_	1/26/2024 6:13
41891-3 SPK	5.126	10	358.6	18.4	5.00	103%	_2024-01-26_	1/26/2024 6:41
41891-3 SPK DUP	4.595	10	358.6	16.5	5.00	92%	_2024-01-26_	1/26/2024 7:09
41891-4	0.067	10	380.5	< 0.381			_2024-01-26_	1/26/2024 2:59
41891-4 DUP	-0.022	10	380.5	< 0.381		NA	_2024-01-26_	1/26/2024 3:27
41891-5	-0.015	10	387.8	< 0.388			_2024-01-26_	1/26/2024 7:36
41891-5 DUP	0.012	10	387.8	< 0.388		NA	_2024-01-26_	1/26/2024 8:04
41891-6	-0.002	10	361.5	< 0.362			_2024-01-26_	1/26/2024 14:37
41891-6 DUP	-0.017	10	361.5	< 0.362		NA	_2024-01-26_	1/26/2024 15:05
41891-6 SPK	5.217	10	361.5	18.9	5.00	104%	_2024-01-26_	1/26/2024 15:33
41891-6 SPK DUP	5.314	10	361.5	19.2	5.00	107%	_2024-01-26_	1/26/2024 16:00
41891-7 FB	0.058	5	374.4	< 0.187			_2024-01-26_	1/26/2024 12:47
41891-7 FB DUP	0.058	5	374.4	< 0.187		NA	_2024-01-26_	1/26/2024 13:14



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M26A-Cl<sub>2</sub> IC Data Sheet

Lab ID #: 41891

Client: Deeco

Column: IonPac AS14A

Date: 01.29.24

Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0 mM NaHCO<sub>3</sub>

Analyst: LAW/MNB

Flow Rate: 1.0 mL/min.

Detection Limit, (µg/ml): 0.10

Standards	Cl <sup>-</sup> µg/ml	Dilution	QC µg/ml	%Relative Error	% Recovery	File Name	Date Time
0	0.000					_2024-01-25_	1/25/2024 17:45
0.1	0.104			4.0%	104%	_2024-01-25_	1/25/2024 18:13
1	0.958			-4.2%	96%	_2024-01-25_	1/25/2024 18:41
3	3.007			0.2%	100%	_2024-01-25_	1/25/2024 19:09
5	5.058			1.2%	101%	_2024-01-25_	1/25/2024 19:36
10	9.973			-0.3%	100%	_2024-01-25_	1/25/2024 20:04
0.1	0.118			18.0%	118%	_2024-01-26_	1/26/2024 17:51
1	1.045			4.5%	105%	_2024-01-26_	1/26/2024 18:19
3	3.125			4.2%	104%	_2024-01-26_	1/26/2024 18:47
5	5.416			8.3%	108%	_2024-01-26_	1/26/2024 19:14
10	10.824			8.2%	108%	_2024-01-26_	1/26/2024 19:42
Correlation-	1.0000						
QC	5.055		5.00		101%	_2024-01-25_	1/25/2024 20:32
QC	5.158		5.00		103%	_2024-01-25_	1/25/2024 20:59
QC	5.226		5.00		105%	_2024-01-26_	1/26/2024 3:55
QC	5.089		5.00		102%	_2024-01-26_	1/26/2024 4:23
QC	5.312		5.00		106%	_2024-01-26_	1/26/2024 9:57
QC	5.420		5.00		108%	_2024-01-26_	1/26/2024 16:28
QC	5.201		5.00		104%	_2024-01-26_	1/26/2024 20:10
DL	0.110		0.10		110%	_2024-01-25_	1/25/2024 22:22
DL	0.102		0.10		102%	_2024-01-25_	1/25/24 22:50
DL	0.100		0.10		100%	_2024-01-26_	1/26/2024 16:56
DL	0.102		0.10		102%	_2024-01-26_	1/26/2024 20:37
40034-7 QC	7.998	10	78.0		103%	_2024-01-26_	1/26/2024 13:42
40034-7 QC	8.306	10	78.0		106%	_2024-01-26_	1/26/2024 14:10
BLK	-0.006					_2024-01-25_	1/25/2024 21:27
BLK	-0.001					_2024-01-25_	1/25/2024 21:55
BLK	0.033					_2024-01-26_	1/26/2024 4:50
BLK	0.000					_2024-01-26_	1/26/2024 5:18
BLK	0.001					_2024-01-26_	1/26/2024 11:51
BLK	0.003					_2024-01-26_	1/26/2024 12:19
BLK	0.000					_2024-01-26_	1/26/2024 17:24
BLK	0.000					_2024-01-26_	1/26/2024 21:05

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IC Sample Sheet/Digestion Worksheet

Lab ID #: 41891

Date: 1-25-24  
 Analyst: LFW/MMW  
 Batch name: 012524-41891

Column: IonPac AS14A  
 Conc. Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0mM NaHCO<sub>3</sub>  
 10mL Conc. Eluent Diluted to FV=1L with filtered UPDI  
 Regenerant: 100mM H<sub>3</sub>PO<sub>4</sub>  
 Flow Rate: 1.0 mL/min.

Instrument: 8611788  
 Lot# 1C11-102-1  
 Lot # 1C11-117-2  
 Method: 26A NaOH

AS LOC.	Sample ID	Client	Analyte	Results (ug/mL)	Results (ug/mL)	Dilution	Wt (g) / FV (mL)
1	0.0			QC	MANE	R <sup>2</sup>	
2	0.1		Cl <sub>2</sub>	4308/59	RICA	1.0700	
3	1.0						
4	3.0						
5	5.0						
6	10.0						
7	QC						
8	BLK QC						
9	BLK						
10	BLK						
11	DL						
12	DL						
13	LRB						
14	LRB						
15	LRB+						
16	LRB+						
17	41891-1	DEECO	Cl <sub>2</sub>		-	10x	
18	-1 dup				-		
19	-2				-		
20	-2 dup				-		
21	-4				-		
22	-4 dup				-		
23	QC						
24	QC						
25	BLK						

Manual Integr:

Curve IC Lot # 1C11-121-3 Sodium Thiosulfate Lot # 1C11-73-4 Comments: pg 1 of 3

Spike 50 uL from 1000 ug/mL Std. to 10mL sample Lot #'s: IC ME Solution 23030291-250 HPS

QC: Spike 50 uL from 1000 ug/mL Br Std. to 10mL sample; lot #'s listed above.

Submitted for QC- Date: 1-24-24 Time: 9:28 By: MMW QC Review- Date:  Time: \_\_\_\_\_ By: \_\_\_\_\_

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IC Sample Sheet/Digestion Worksheet

Lab ID #: 41891

Date: 1-25-24  
Analyst: LAW/mmw

Column: IonPac AS14A  
Conc. Eluent: 8.0 mM Na<sub>2</sub>CO<sub>3</sub>/ 1.0mM NaHCO<sub>3</sub>  
10mL Conc. Eluent Diluted to FV=1L with filtered UPDI  
Regenerant: 100mM H<sub>3</sub>PO<sub>4</sub>  
Flow Rate: 1.0 mL/min.

Instrument: Bell 788  
Lot# 1011-100-1  
Lot # 1011-1172  
Method: 26A NaOH

Batch name: 012524-41891

AS LOC.	Sample ID	Client	Analyte	Results (ug/mL)	Results (ug/mL)	Dilution	Wt (g) / FV (mL)
26	BK						
27	41891-3	Deeco	Cl <sub>2</sub>		—	10x	
28	-3 dup				—		
29	-3t				5.126		
30	-3t dup				4.595		
31	-5				—		
32	-5 dup				—		
33 SB	-6	Sent Back Software deleted			—		
34 SB	-6 dup				—		
35 SB	-6t				5.217		
36 SB	-6t dup				5.814		
37	QC						
38	QC						
39	BK						
40	BK						
41	41891-7 PB	deeco	Cl <sub>2</sub>		—	5x	
42	-7 PB dup				—		
43	40034-7 QC		Cl <sub>2</sub>		7.998	10x	TV=78.0
44	-7 QC dup				8.300		
45	QC						
46	DL						
47	BK						
48	0.1						
49	1.0						
50	3.0						

Manual integr:

Curve IC Lot # Sodium Thiosulfate Lot # Comments: pg 2 of 3

Spike 50 uL from 1000 ug/mL Std. to 10mL sample Lot #'s: IC ME Solution

QC: Spike 50 uL from 1000 ug/mL Br Std. to 10mL sample; lot #'s listed above.

Submitted for QC- Date: Time: By: QC Review- Date: Time: By:

elementOne



**Appendix D**  
**Plant Process Data**

Holcim; Ada OK  
Kiln 3 RTR - Run 1

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
<b>Average</b>	<b>64.2</b>
01/08/2024 15:21	60.71
01/08/2024 15:22	61.43
01/08/2024 15:23	60.27
01/08/2024 15:24	61.56
01/08/2024 15:25	60.61
01/08/2024 15:26	61.42
01/08/2024 15:27	60.78
01/08/2024 15:28	61.10
01/08/2024 15:29	61.05
01/08/2024 15:30	60.50
01/08/2024 15:31	61.40
01/08/2024 15:32	60.52
01/08/2024 15:33	61.19
01/08/2024 15:34	60.56
01/08/2024 15:35	61.51
01/08/2024 15:36	60.98
01/08/2024 15:37	60.96
01/08/2024 15:38	62.65
01/08/2024 15:39	62.82
01/08/2024 15:40	67.60
01/08/2024 15:41	67.43
01/08/2024 15:42	65.43
01/08/2024 15:43	64.75
01/08/2024 15:44	63.88
01/08/2024 15:45	64.14
01/08/2024 15:46	63.44
01/08/2024 15:47	64.04
01/08/2024 15:48	64.03
01/08/2024 15:49	63.63
01/08/2024 15:50	64.14
01/08/2024 15:51	63.50
01/08/2024 15:52	64.17
01/08/2024 15:53	64.00
01/08/2024 15:54	63.79
01/08/2024 15:55	64.06
01/08/2024 15:56	63.50
01/08/2024 15:57	63.99
01/08/2024 15:58	63.32

Holcim; Ada OK  
Kiln 3 RTR - Run 1

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
01/08/2024 15:59	64.34
01/08/2024 16:00	63.74
01/08/2024 16:01	63.94
01/08/2024 16:02	64.12
01/08/2024 16:03	63.62
01/08/2024 16:04	64.13
01/08/2024 16:05	63.55
01/08/2024 16:06	64.56
01/08/2024 16:07	64.70
01/08/2024 16:08	65.82
01/08/2024 16:09	65.61
01/08/2024 16:10	66.44
01/08/2024 16:11	66.42
01/08/2024 16:12	66.85
01/08/2024 16:13	67.03
01/08/2024 16:14	66.55
01/08/2024 16:15	67.29
01/08/2024 16:16	66.37
01/08/2024 16:17	66.72
01/08/2024 16:18	67.06
01/08/2024 16:19	66.70
01/08/2024 16:20	66.67
01/08/2024 16:21	66.75
01/08/2024 16:22	66.94
01/08/2024 16:23	66.60
01/08/2024 16:24	66.38
01/08/2024 16:25	67.18
01/08/2024 16:26	66.53
01/08/2024 16:27	66.77
01/08/2024 16:28	66.99
01/08/2024 16:29	66.75

Holcim; Ada OK  
Kiln 3 RTR - Run 2

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
<b>Average</b>	<b>68.4</b>
01/08/2024 16:44	66.94
01/08/2024 16:45	66.46
01/08/2024 16:46	67.06
01/08/2024 16:47	66.98
01/08/2024 16:48	66.93
01/08/2024 16:49	68.33
01/08/2024 16:50	67.61
01/08/2024 16:51	68.72
01/08/2024 16:52	68.30
01/08/2024 16:53	68.59
01/08/2024 16:54	68.71
01/08/2024 16:55	68.08
01/08/2024 16:56	68.80
01/08/2024 16:57	68.21
01/08/2024 16:58	68.93
01/08/2024 16:59	68.18
01/08/2024 17:00	68.47
01/08/2024 17:01	68.85
01/08/2024 17:02	68.32
01/08/2024 17:03	68.74
01/08/2024 17:04	68.59
01/08/2024 17:05	68.38
01/08/2024 17:06	68.71
01/08/2024 17:07	68.24
01/08/2024 17:08	68.94
01/08/2024 17:09	68.04
01/08/2024 17:10	68.92
01/08/2024 17:11	68.55
01/08/2024 17:12	68.22
01/08/2024 17:13	68.76
01/08/2024 17:14	68.41
01/08/2024 17:15	68.57
01/08/2024 17:16	68.80
01/08/2024 17:17	68.05
01/08/2024 17:18	68.70
01/08/2024 17:19	68.84
01/08/2024 17:20	68.06
01/08/2024 17:21	68.87

Holcim; Ada OK  
Kiln 3 RTR - Run 2

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
01/08/2024 17:22	68.13
01/08/2024 17:23	68.75
01/08/2024 17:24	68.56
01/08/2024 17:25	68.56
01/08/2024 17:26	68.46
01/08/2024 17:27	68.40
01/08/2024 17:28	68.83
01/08/2024 17:29	68.05
01/08/2024 17:30	69.00
01/08/2024 17:31	68.41
01/08/2024 17:32	68.24
01/08/2024 17:33	68.95
01/08/2024 17:34	68.05
01/08/2024 17:35	68.78
01/08/2024 17:36	68.31
01/08/2024 17:37	68.68
01/08/2024 17:38	68.18
01/08/2024 17:39	68.54
01/08/2024 17:40	68.66
01/08/2024 17:41	68.11
01/08/2024 17:42	68.75
01/08/2024 17:43	68.43
01/08/2024 17:44	68.39
01/08/2024 17:45	68.79
01/08/2024 17:46	68.26
01/08/2024 17:47	68.60
01/08/2024 17:48	68.36
01/08/2024 17:49	68.94
01/08/2024 17:50	68.09

Holcim; Ada OK  
Kiln 3 RTR - Run 3

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
<b>Average</b>	<b>70.1</b>
01/08/2024 18:05	68.22
01/08/2024 18:06	68.86
01/08/2024 18:07	68.35
01/08/2024 18:08	68.62
01/08/2024 18:09	68.45
01/08/2024 18:10	68.26
01/08/2024 18:11	68.74
01/08/2024 18:12	68.06
01/08/2024 18:13	68.82
01/08/2024 18:14	68.50
01/08/2024 18:15	68.45
01/08/2024 18:16	68.29
01/08/2024 18:17	68.90
01/08/2024 18:18	69.48
01/08/2024 18:19	69.11
01/08/2024 18:20	69.77
01/08/2024 18:21	69.31
01/08/2024 18:22	69.42
01/08/2024 18:23	69.95
01/08/2024 18:24	69.40
01/08/2024 18:25	70.10
01/08/2024 18:26	69.60
01/08/2024 18:27	69.42
01/08/2024 18:28	69.80
01/08/2024 18:29	69.43
01/08/2024 18:30	69.52
01/08/2024 18:31	70.03
01/08/2024 18:32	69.31
01/08/2024 18:33	69.91
01/08/2024 18:34	69.38
01/08/2024 18:35	69.76
01/08/2024 18:36	69.90
01/08/2024 18:37	69.52
01/08/2024 18:38	69.79
01/08/2024 18:39	69.31
01/08/2024 18:40	69.93
01/08/2024 18:41	70.66
01/08/2024 18:42	70.32

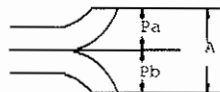
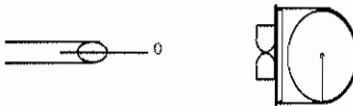
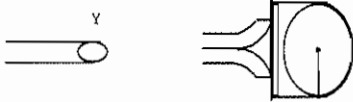
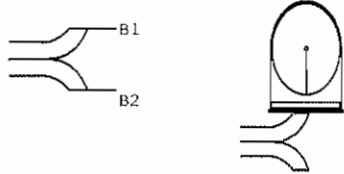
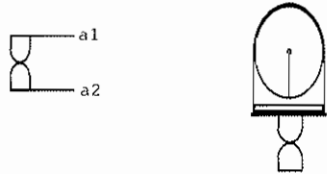
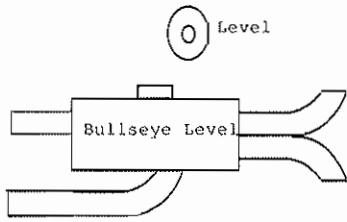
Holcim; Ada OK

Kiln 3 RTR - Run 3

Date/Time	Kiln: CLINKER_PROD (MTHR) Raw Value
01/08/2024 18:43	71.06
01/08/2024 18:44	70.51
01/08/2024 18:45	70.81
01/08/2024 18:46	71.17
01/08/2024 18:47	70.50
01/08/2024 18:48	71.44
01/08/2024 18:49	71.17
01/08/2024 18:50	71.35
01/08/2024 18:51	71.72
01/08/2024 18:52	71.23
01/08/2024 18:53	71.04
01/08/2024 18:54	71.63
01/08/2024 18:55	71.13
01/08/2024 18:56	71.42
01/08/2024 18:57	71.46
01/08/2024 18:58	71.32
01/08/2024 18:59	71.77
01/08/2024 19:00	71.05
01/08/2024 19:01	71.36
01/08/2024 19:02	71.24
01/08/2024 19:03	71.24
01/08/2024 19:04	71.92
01/08/2024 19:05	71.78
01/08/2024 19:06	71.62
01/08/2024 19:07	70.88
01/08/2024 19:08	71.61
01/08/2024 19:09	70.99
01/08/2024 19:10	71.55
01/08/2024 19:11	71.12

**Appendix E**  
**Calibration Documents**

**Pitot Tube Inspection Sheet**



Date	01/03/23
Tube Assembly Level?	Yes
Ports Damaged?	No
-10 deg < a1 < +10 deg	2
-10 deg < a2 < +10 deg	1
-5 deg < B1 < +5 deg	1
-5 deg < B2 < +5 deg	1
Y (gamma)	1
θ (theta)	1
A (alpha)	0.951
Z = A (sin γ) < 0.125"?	yes
W = A (sin θ) < 0.031"?	yes
Pa =	0.475
Pb =	0.476
Tube Diameter (Dt) =	0.376
Pa = Pb +/- 0.063"?	yes
(1.05 x Dt)?	0.3948
(1.50 x Dt)?	0.564
(1.05 x Dt) < P < (1.50 x Dt)?	yes

<b>Eligible for Default Pitot Calibration Factor (Cp = 0.84)?</b>	<b>Yes</b>
---	------------

**Thermocouple Calibration**

Type of Reference Thermometer?	Mercury	Date	01/03/23
Barometric Pressure?	29.52	Ambient Temperature?	68

Source	Reference Temp, F	Thermocouple Temp, F	Absolute Temp Difference
cold air	37	38	-0.20%
medium air	215	215	0.00%
hot air	325	325	0.00%

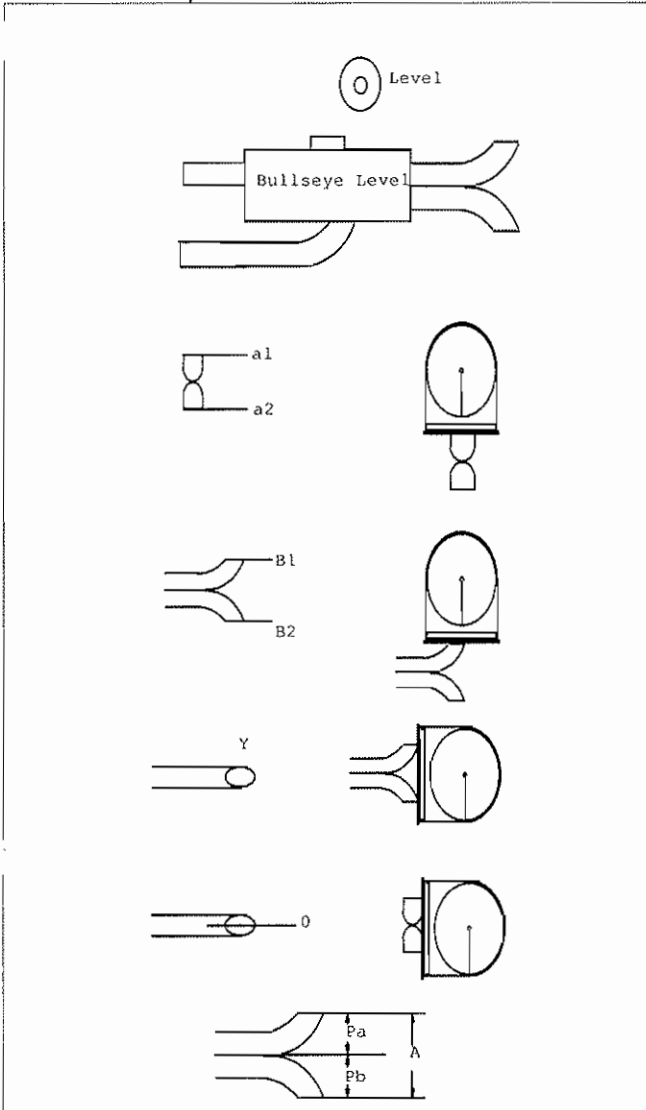
**Windtunnel Calibration**

Pitot Reading	Reference (0.99)	5A S-Type Pitot	Cp
ΔP <sub>1</sub>	0.31	0.44	0.84
ΔP <sub>2</sub>	0.31	0.43	0.85
ΔP <sub>3</sub>	0.31	0.43	0.85
<b>Average Pitot Tube Calibration Factor--&gt;</b>			<b>0.85</b>

**Thermocouple Calibration Check (EPA ALT-011 Procedure), performed on 1/3/23**

Source	Ref. Temp. F	Thermocouple Temp. F	± 2 deg F?
Ambient	68	66.9	Yes

Pitot Tube Inspection Sheet



Date	01/03/23
Tube Assembly Level?	Yes
Ports Damaged?	No
-10 deg < a1 < +10 deg	1
-10 deg < a2 < +10 deg	2
-5 deg < B1 < +5 deg	1
-5 deg < B2 < +5 deg	2
Y (gamma)	1
theta (theta)	1
A (alpha)	0.94
Z = A (sin y) < 0.125"?	yes
W = A (sin theta) < 0.031"?	yes
Pa =	0.47
Pb =	0.47
Tube Diameter (Dt) =	0.376
Pa = Pb +/- 0.063"?	yes
(1.05 x Dt)?	0.3948
(1.50 x Dt)?	0.564
(1.05 x Dt) < P < (1.50 x Dt)?	yes

<b>Eligible for Default Pitot Calibration Factor (Cp = 0.84)?</b>	<b>Yes</b>
---	------------

Thermocouple Calibration

Type of Reference Thermometer?	Mercury	Date	01/03/23
Barometric Pressure?	29.52	Ambient Temperature?	69.9

Source	Reference Temp, F	Thermocouple Temp, F	Absolute Temp Difference
cold air	37	38	-0.20%
medium air	215	215	0.00%
hot air	325	324	0.13%

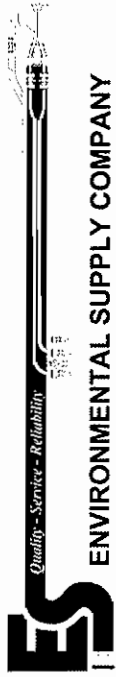
Windtunnel Calibration

Pitot Reading	Reference (0.99)	5B S-Type Pitot	Cp
ΔP <sub>1</sub>	0.31	0.44	0.84
ΔP <sub>2</sub>	0.31	0.43	0.85
ΔP <sub>3</sub>	0.31	0.44	0.84
<b>Average Pitot Tube Calibration Factor----&gt;</b>			<b>0.84</b>

Thermocouple Calibration Check (EPA ALT-011 Procedure), performed on 1/3/23

Source	Ref. Temp. F	Thermocouple Temp. F	± 2 deg F?
Ambient	69.9	69.4	Yes

# METHOD 5 DRY GAS METER CALIBRATION USING CRITICAL ORIFICES



- 1) Select three critical orifices to calibrate the dry gas meter which bracket the expected operating range.
- 2) Record barometric pressure before and after calibration procedure.
- 3) Run at tested vacuum (from Orifice Calibration Report), for a period of time necessary to achieve a minimum total volume of 5 cubic feet.
- 4) Record data and information in the GREEN cells, YELLOW cells are calculated.

DATE: **12/23/23** METER SERIAL #: **M5-18** INITIAL: **30.15** FINAL: **30.18** AVG (P<sub>bar</sub>): **30.165**

METER PART #: **M5-18** CRITICAL ORIFICE SET SERIAL #: **14315** BAROMETRIC PRESSURE (m Hg): **30.15**

IF Y VARIATION EXCEEDS 2.00%,  
ORIFICE SHOULD BE RECALIBRATED

ORIFICE #	RUN #	K' FACTOR (AVG)	TESTED VACUUM (in Hg)	DGM READINGS (FT <sup>3</sup> )		TEMPERATURES °F		ELAPSED TIME (MIN)	DGM ΔH (in H <sub>2</sub> O)	V <sub>m</sub> (STD)	V <sub>cr</sub> (STD)	(3)	Y	VARIATION (%)	ΔH@			
				INITIAL	FINAL	NET (V <sub>m</sub> )	AMBIENT									DGM INLET	DGM INLET	DGM OUTLET
<b>12</b>	1	0.3283																
	2			0.000														
	3			0.000														
<b>15</b>	1	0.4094	18	5.561	361.456	367.017	69	59	67	58	61	61.25	10.00	0.81	5.6915	5.3709	0.944	1.62
	2	0.4094	18	5.447	367.017	372.464	69	64	71	61	64	65	10.00	0.81	5.5349	5.3709	0.970	1.60
	3			0.000								0						
<b>19</b>	1	0.5047	18	6.708	372.917	379.625	69	71	76	65	68	70	10.00	1.3	6.7600	6.6212	0.979	1.68
	2	0.5047	18	6.738	379.625	386.363	69	73	77	68	70	72	10.00	1.3	6.7647	6.6212	0.979	1.67
	3			0.000								0						
<b>23</b>	1	0.6426	18	8.553	387.121	395.674	68	75	81	70	73	74.75	10.00	2.1	8.5593	8.4303	0.985	1.66
	2	0.6426	18	8.620	395.674	404.294	68	79	80	73	74	76.5	10.00	2.1	8.5982	8.4383	0.981	1.65
	3			0.000								0						
<b>32</b>	1	0.8587																
	2			0.000														
	3			0.000														

USING THE CRITICAL ORIFICES AS CALIBRATION STANDARDS:  
 The following equations are used to calculate the standard volumes of air passed through the DGM, V<sub>m</sub> (std), and the critical orifice, V<sub>cr</sub> (std), and the DGM calibration factor, Y. These equations are automatically calculated in the

$$(1) V_{m, std} = K_1 * V_m * \frac{P_{bar} + (\Delta H / 13.6)}{T_m} = \text{Net volume of gas sample passed through DGM, corrected to standard conditions}$$

K<sub>1</sub> = 17.64 °R/in. Hg (English); 0.3858 °K/(mm Hg (Metric))

$$(2) V_{cr, std} = K_2 * \frac{P_{bar} * \Theta}{\sqrt{T_{amb}}} = \text{Volume of gas sample passed through the critical orifice, corrected to standard conditions}$$

T<sub>amb</sub> = Absolute ambient temperature (°R - English, °K - Metric)

$$(3) Y = \frac{V_{cr, std}}{V_{m, std}} = \text{DGM calibration factor}$$

K' = Average K' factor from Critical Orifice Calibration

**AVERAGE DRY GAS METER CALIBRATION FACTOR, Y = 0.973**

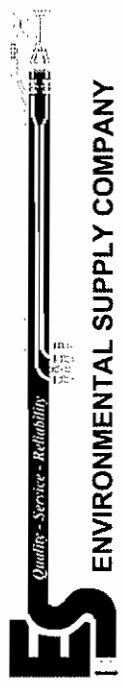
**AVERAGE ΔH@ = 1.65**

Potentiometer Check, °F

@ 0 F	0
@ 500 F	496
@ 1000 F	1000

Avg Absolute Difference = **0.1%**

# METHOD 5 DRY GAS METER CALIBRATION USING CRITICAL ORIFICES



- 1) Select three critical orifices to calibrate the dry gas meter which bracket the expected operating range.
- 2) Record barometric pressure before and after calibration procedure.
- 3) Run at tested vacuum (from Orifice Calibration Report), for a period of time necessary to achieve a minimum total volume of 5 cubic feet.
- 4) Record data and information in the GREEN cells, YELLOW cells are calculated.

DATE: 12/21/23 METER SERIAL #: m5-25  
 METER PART #: m5-25 CRITICAL ORIFICE SET SERIAL #: 1431S  
 INITIAL BAROMETRIC PRESSURE (in Hg): 30.18 FINAL AVG (P<sub>bar</sub>): 30.165  
 IF Y VARIATION EXCEEDS 2.00%, ORIFICE SHOULD BE RECALIBRATED

ORIFICE #	RUN #	K' FACTOR (AVG)	TESTED VACUUM (in Hg)	DGM READINGS (FT <sup>3</sup> )		TEMPERATURES °F			ELAPSED TIME (MIN)	DGM ΔH (in H <sub>2</sub> O)	V <sub>m</sub> (STD)	V <sub>cr</sub> (STD)	Y VARIATION (%)	ΔH <sub>g</sub>
				INITIAL	FINAL	NET (V <sub>m</sub> )	AMBIENT	DGM INLET						
12	1	0.3283												
	2													
	3													
15	1	0.4094	18	576.201	581.661	5.460	55	53	55	53	54	54	53.75	0.97
	2	0.4094	18	581.661	587.119	5.458	55	55	58	54	55	55	55.5	0.97
	3					0.000							0	
19	1	0.5047	18	587.424	594.121	6.697	55	57	61	55	56	56	57.25	1.5
	2	0.5047	18	594.121	600.831	6.710	57	59	63	56	57	57	58.75	1.5
	3					0.000							0	
23	1	0.6426	18	601.129	609.648	8.519	57	62	65	58	59	59	61	2.5
	2	0.6426	18	609.648	618.182	8.534	58	63	62	58	55	55	59.5	2.5
	3					0.000							0	
32	1	0.8587												
	2													
	3													

USING THE CRITICAL ORIFICES AS CALIBRATION STANDARDS:  
 The following equations are used to calculate the standard volumes of air passed through the DGM, V<sub>m</sub> (std), and the critical orifice, V<sub>cr</sub> (std), and the DGM calibration factor, Y. These equations are automatically calculated in the

(1)  $V_{m, std} = K_1 * Y * V_m * \frac{P_{bar} + (\Delta H / 13.6)}{T_m}$   
 = Net volume of gas sample passed through DGM, corrected to standard conditions  
 K<sub>1</sub> = 17.64 °R/in. Hg (English), 0.3858 °K/(mm Hg (Metric))  
 T<sub>m</sub> = Absolute DGM avg. temperature (°R - English, °K - Metric)

(2)  $V_{cr, std} = K' * \frac{P_{bar} * \Theta}{\sqrt{T_{amb}}}$   
 = Volume of gas sample passed through the critical orifice, corrected to standard conditions  
 T<sub>amb</sub> = Absolute ambient temperature (°R - English, °K - Metric)  
 K' = Average K' factor from Critical Orifice Calibration

(3)  $Y = \frac{V_{cr, std}}{V_{m, std}}$   
 = DGM calibration factor

AVERAGE DRY GAS METER CALIBRATION FACTOR, Y = **0.967**  
 AVERAGE ΔH<sub>g</sub> = **1.95**  
 Potentiometer Check, °F: **0 @ 0 F**, **498 @ 500 F**, **1004 @ 1000 F**  
 Avg Absolute Difference = **0.0%**

Company: Holcim; Ada OK  
 Source: Kiln 3 Main Stack  
 Job ID: 24-3326  
 Train Type: EPA Method 26A

Alt-009 Alternate Post Test Calibration Data						
	M5-18 1A 01/08/24 1521-1627	M5-25 1B 01/08/24 1521-1627	M5-18 2A 01/08/24 1644-1750	M5-25 2B 01/08/24 1644-1750	M5-18 3A 01/08/24 1805-1911	M5-25 3B 01/08/24 1805-1911
	Averages		Averages		Averages	
Vm	47.272	46.335	46.706	46.238	47.375	47.474
Tm	518.9	501.5	528.8	507.9	539.8	507.2
Pb	28.15	28.15	28.15	28.15	28.15	28.15
Havg	1.67	1.96	1.62	1.91	1.69	2
H@	1.65	1.95	1.65	1.95	1.65	1.95
Md	29.96	29.96	30.072	30.072	30.104	30.104
(Havg)*0.5	1.28763282	1.39599357	1.26812296	1.37805958	1.29696328	1.41110159
Run Time, Min	60	60	60	60	60	60
Calculated Gamma (Yqa)	0.958	0.958	0.962	0.952	0.979	0.948
Meter Gamma	0.973	0.967	0.973	0.967	0.973	0.967
% difference from Actual Y	1.56%	0.96%	1.12%	1.59%	0.66%	1.98%
					1.12%	1.51%

# CERTIFICATE OF ANALYSIS

## Grade of Product: EPA Protocol

Part Number: E04NI77E15A3796 Reference Number: 122-402248430-1  
 Cylinder Number: XC025341B Cylinder Volume: 151.1 CF  
 Laboratory: 124 - Durham (SAP) - NC Cylinder Pressure: 2015 PSIG  
 PGVP Number: B22021 Valve Outlet: 590  
 Gas Code: CO,CO2,O2,BALN Certification Date: Oct 13, 2021

**Expiration Date: Oct 13, 2029**

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a mole/mole basis unless otherwise noted.

Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.

ANALYTICAL RESULTS					
Component	Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty	Assay Dates
CARBON MONOXIDE	65.00 PPM	64.10 PPM	G1	+/- 0.8% NIST Traceable	10/13/2021
CARBON DIOXIDE	10.00 %	10.22 %	G1	+/- 0.6% NIST Traceable	10/12/2021
OXYGEN	12.00 %	11.79 %	G1	+/- 0.4% NIST Traceable	10/12/2021
NITROGEN	Balance				

CALIBRATION STANDARDS					
Type	Lot ID	Cylinder No	Concentration	Uncertainty	Expiration Date
NTRM	09010213	KAL004779	98.48 PPM CARBON MONOXIDE/NITROGEN	+/- 0.5%	Oct 16, 2024
NTRM	19060402	6162642Y	11.105 % CARBON DIOXIDE/NITROGEN	+/- 0.6%	Dec 04, 2025
NTRM	10010616	K014963	9.967 % OXYGEN/NITROGEN	+/- 0.3%	Apr 19, 2022

ANALYTICAL EQUIPMENT		
Instrument/Make/Model	Analytical Principle	Last Multipoint Calibration
Horiba VA-5001 CO2 BF89GV17	Nondispersive infrared (NDIR)	Sep 15, 2021
Horiba VIA510 CO 1G46EA07	Nondispersive infrared (NDIR)	Sep 22, 2021
Siemens Oxymat 61 M3299 O2	Paramagnetic	Sep 14, 2021

Triad Data Available Upon Request



Approved for Release

## CERTIFICATE OF ANALYSIS

### Grade of Product: EPA PROTOCOL STANDARD

Part Number:	E04NI59E15A38X3	Reference Number:	122-402389885-1A
Cylinder Number:	ALM-056015	Cylinder Volume:	143.7 CF
Laboratory:	124 - Durham (SAP) - NC	Cylinder Pressure:	2016 PSIG
PGVP Number:	B22022	Valve Outlet:	590
Gas Code:	CO,CO2,O2,BALN	Certification Date:	Mar 28, 2022

**Expiration Date: Mar 28, 2030**

Certification performed in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (May 2012)" document EPA 600/R-12/531, using the assay procedures listed. Analytical Methodology does not require correction for analytical interference. This cylinder has a total analytical uncertainty as stated below with a confidence level of 95%. There are no significant impurities which affect the use of this calibration mixture. All concentrations are on a mole/mole basis unless otherwise noted.

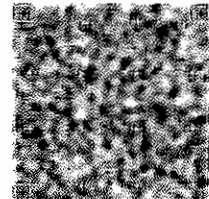
Do Not Use This Cylinder below 100 psig, i.e. 0.7 megapascals.


ANALYTICAL RESULTS					
Component	Requested Concentration	Actual Concentration	Protocol Method	Total Relative Uncertainty	Assay Dates
CARBON MONOXIDE	120.0 PPM	116.5 PPM	G1	+/- 0.3% NIST Traceable	03/28/2022
CARBON DIOXIDE	18.00 %	18.17 %	G1	+/- 0.7% NIST Traceable	03/28/2022
OXYGEN	22.00 %	21.90 %	G1	+/- 0.5% NIST Traceable	03/28/2022
NITROGEN	Balance				

CALIBRATION STANDARDS					
Type	Lot ID	Cylinder No	Concentration	Uncertainty	Expiration Date
NTRM	13010207	KAL003102	246.9 PPM CARBON MONOXIDE/NITROGEN	+/- 0.2%	Oct 16, 2024
NTRM	12061508	CC354696	19.87 % CARBON DIOXIDE/NITROGEN	+/- 0.6%	Jan 11, 2024
NTRM	08010220	K013155	23.20 % OXYGEN/NITROGEN	+/- 0.4%	Jun 01, 2024

ANALYTICAL EQUIPMENT		
Instrument/Make/Model	Analytical Principle	Last Multipoint Calibration
Horiba VA-5001 CO2 BF89GV17	Nondispersive Infrared (NDIR)	Mar 01, 2022
Horiba VIA510 CO RS2EGL6K	Nondispersive Infrared (NDIR)	Mar 01, 2022
Siemens Oxymat 61 M3299 O2	Paramagnetic	Mar 01, 2022

Triad Data Available Upon Request



  
 Approved for Release



Air Liquide company

ANALYSIS LABORATORY

CERTIFICATE OF ACCURACY: GMACS-c Calibration Standard

CUSTOMER INFORMATION

AIRGAS SPECIALTY GASES
Exploratory Products Group
6141 Easton Road
Plumsteadville, PA 18949

Work Order #: 160-402845897-1
Sales Order #: 1123601913
PO #: 7100179560

Customer: DEECO Inc.
Address 1: 3404 Lake Woodard Road
Address 2:
City / State / Zip: Raleigh, NC 27604

PRODUCT INFORMATION

Table with 4 columns: COMPOSITION, CONCENTRATION, UNCERTAINTY (Abs), UNCERTAINTY (Rel). Rows include Hydrogen Cyanide, Sulfur Hexafluoride, and Nitrogen.

CYLINDER #: CC768222
CYLINDER TYPE: 150A Aluminum
CGA: 350 SS
CYLINDER PRESSURE: 2000 psig
AIRGAS PART #: X03NI99C15AC0W8
CERTIFICATION DATE: 7-Sep-2023
EXPIRATION DATE: 7-Mar-2024
MIXTURE DEW POINT: N/A

CERTIFICATION DATA

Table with 4 columns: COMPONENT, CONCENTRATION, UNCERTAINTY (Abs), UNCERTAINTY (Rel). Includes BLENDED PROCESS: GravStat™ Gravimetry. Rows for Hydrogen Cyanide and Sulfur Hexafluoride.

CONFIRMING ANALYSIS: FTIR Spectroscopy
INSTRUMENT / MODEL: CAI Model 700 FTIR

Table with 4 columns: COMPONENT, CONCENTRATION, UNCERTAINTY (Abs), UNCERTAINTY (Rel). Row for Hydrogen Cyanide.

REFERENCE STANDARD: GMPS-c 50 PPM Hydrogen Cyanide
CYLINDER NUMBER: CC768196
EXPIRATION DATE: 2/29/2024

Table with 4 columns: COMPONENT, CONCENTRATION, UNCERTAINTY (Abs), UNCERTAINTY (Rel). Row for Hydrogen Cyanide.

Table with 6 columns: CALIBRATION CURVE DATA, Curve Order, Correlation, Slope (X2), Slope (X), Intercept. Row for Point-to-Point Matching Std.

INTERLOCK STATISTICS

Table with 4 columns: CONCENTRATION, UNCERTAINTY (Abs), UNCERTAINTY (Rel). Rows for BLEND RESULT, ANALYSIS RESULT, INTERLOCK RESULT.

COMMENTS / SPECIAL INSTRUCTIONS

- 1. A GMACS-c ("Candidate GMACS") is made and certified according to the EPA GMACS Procedure (Alt-114) found at: https://cfpub.epa.gov
2. Do not use this standard if pressure is less than 200 psig.
3. Do not use or store this product at or below the stated dew point.

APPROVED BY: \_\_\_\_\_

BOB GRASMEDER

## CERTIFICATE OF ANALYSIS

### Grade of Product: CERTIFIED STANDARD-SPEC

Part Number:	X02NI99C15A54F5	Reference Number:	122-402705571-1
Cylinder Number:	CC426155	Cylinder Volume:	144.0 CF
Laboratory:	124 - Durham (SAP) - NC	Cylinder Pressure:	2015 PSIG
Analysis Date:	Mar 28, 2023	Valve Outlet:	350
Lot Number:	122-402705571-1		

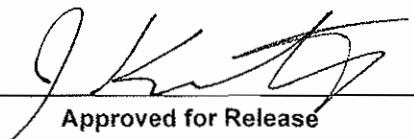
Expiration Date: Mar 28, 2031

Product composition verified by direct comparison to calibration standards traceable to N.I.S.T. weights and/or N.I.S.T. Gas Mixture reference materials.

### ANALYTICAL RESULTS

Component	Req Conc	Actual Concentration (Mole %)	Analytical Uncertainty
ETHYLENE	75.00 PPM	75.47 PPM	+/- 2%
NITROGEN	Balance		



  
Approved for Release

Client: Holcim Ada Ok  
Test Location: Kiln 3 Main Stack  
Date: Jan 08 24 Start Time: 15:21:04  
Run number Stratification Check  
One Minute Averages

	Reference O2 %,dry	Plant O2 %,dry
3:22:02 PM	13.2	13.8
3:23:02 PM	13.2	13.8
3:24:02 PM	13.2	13.6
3:25:02 PM	13.2	14.0
3:26:02 PM	13.2	14.0
Point A	13.2	13.8
3:27:02 PM	13.3	14.0
3:28:02 PM	13.4	14.0
3:29:02 PM	13.3	14.0
3:30:02 PM	13.4	14.1
3:31:02 PM	13.3	14.1
Point B	13.4	14.0
3:32:02 PM	13.2	14.1
3:33:02 PM	13.2	13.9
3:34:02 PM	12.7	13.8
3:35:02 PM	13.0	12.9
3:36:02 PM	13.3	13.8
Point C	13.1	13.7

Holcim; Ada OK  
Kiln 3 RTR - Run 1

Date/Time	Kiln: O2_DRY (PCT) Expression Value
01/08/2024 15:21	13.83
01/08/2024 15:22	13.77
01/08/2024 15:23	13.60
01/08/2024 15:24	14.00
01/08/2024 15:25	13.97
01/08/2024 15:26	13.95
01/08/2024 15:27	14.04
01/08/2024 15:28	13.97
01/08/2024 15:29	14.10
01/08/2024 15:30	14.08
01/08/2024 15:31	14.05
01/08/2024 15:32	13.88
01/08/2024 15:33	13.70
01/08/2024 15:34	12.87
01/08/2024 15:35	13.77

Analysis Validation Report

Sample Filename: F:\Ada\January 8\SPC\_007092.LAB

Filename for noise: F:\Midlothian on Rental\November 14\SPC\_000837.LAB  
 Interferences Filenames: C:\Midlothian on Rental\November 15\SPC\_001463.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001464.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001465.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001466.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001467.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001468.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001469.LAB  
 C:\Midlothian on Rental\November 15\SPC\_001470.LAB

Recipe path: C:\OL\T\recipes\Cement Testing R3.MGRCP

Gas calibration Name	Conc	MDC3	MDC2	MDC1	MAU	FMU*R	OCU	~DL	~CL	~Bias	Sigma	Range	Span	Comment
ADA 1 HCN (200) PCA 191C 191C	0.98	0.16	-	0.29	0.35	0.19	0.35	-	0.09	-	-	0-200	-	Good
HF PPM (10) 191C	-0.12	0.4	0.05	0.04	0.07	0.72	0.72	0.07	0.36	0.02	0.02	0-10	-	Close to DL
SF6 (10) 191C	0	0.02	-	0	0	0.02	0.02	-	0.01	-	-	0-10	-	Close to DL
ETHYLENE (100,3000) 191C	1.02	1.56	0.33	0.17	0.29	2.71	2.71	1.08	1.37	0.75	0.11	0-3000	-	Close to DL
H2O% (40) 191C	24.56	0.4	-	0.01	0.02	0.76	0.76	-	0.49	-	-	0-40	-	Good
CO2% (40) 191C	6.44	0.19	-	0.01	0.02	0.26	0.26	-	0.13	-	-	0-40	-	Good
HCL PPM (100) 191C	0.54	0.5	0.05	0.15	0.28	0.91	0.91	0.06	0.36	0.01	0.02	0-100	-	Check it!
SO2 (1000) 191C	0.21	2.07	0.37	0.14	0.27	3.99	3.99	0.79	1.95	0.42	0.12	0-1000	-	Close to DL
NO (350,3000) 191C	73.66	10.74	0.22	0.26	0.35	14.61	14.61	0.45	8.45	0.23	0.07	0-3000	-	Good
NH3 (300) 191C (1OF2)	4.13	1.25	0.03	0.12	0.2	2.12	2.12	0.15	1.02	0.11	0.01	0-300	-	Check it!
CO (500) 191C (1OF2)	2.93	2.22	-	0.2	0.43	4.79	4.79	-	1.95	-	-	0-500	-	Check it!
CO% (1) 191C (2OF2)	0	0	0	0	0	0.01	0.01	0	0	0	0	0-1	-	Close to DL
FORMALDEHYDE (70) 191C	0.53	0.96	0.28	0.2	0.24	1.16	1.16	0.33	0.76	0.04	0.09	0-70	-	Close to DL
ACETALDEHYDE (1000) 191C	1.03	2.11	0.23	0.35	0.38	2.29	2.29	0.59	1.73	0.35	0.08	0-1000	-	Close to DL
CH4 (250) 191C (1OF2)	0.94	1.88	0.05	0.26	0.7	4.97	4.97	0.06	1.6	0.01	0.02	0-250	-	Check it!
PROPANE (100) 191C	1.86	1.6	0.42	0.12	0.14	1.91	1.91	0.72	1.43	0.3	0.14	0-100	-	Close to DL
HBR (100) 180C	-0.44	5.46	0.17	0.68	1.39	11.1	11.1	0.19	4.8	0.02	0.06	0-100	-	Close to DL
NO2 (150) 191C (1OF2)	-0.9	1.03	0.04	0.02	0.03	1.08	1.08	0.05	0.98	0.01	0.01	0-150	-	Check it!
NO2 (2000) 191C (2OF2)	2.32	7.75	2.03	0.88	1.12	9.87	9.87	3.81	6.84	1.78	0.68	0-2000	-	Close to DL
N2O (100,200,300) 191C	0.32	0.36	0.05	0.04	0.05	0.42	0.42	0.07	0.32	0.02	0.02	0-300	-	Close to DL
NH3 (3000) 191C (2OF2)	9.2	25.47	0.35	1.19	2.25	48.04	48.04	0.44	24.23	0.09	0.12	0-3000	-	Check it!
CH4 (3000) 191C (2OF2)	2.66	17.65	0.34	1.1	1.81	29.08	29.08	0.61	16.52	0.27	0.11	0-3000	-	Close to DL
ACETYLENE (1000) 191C	-0.45	3.27	0.15	0.31	0.37	3.97	3.97	0.21	2.97	0.06	0.05	0-1000	-	Close to DL
PROPYLENE (200,1000) 191C	1.07	4.06	0.19	0.43	0.5	4.76	4.76	0.49	3.62	0.3	0.06	0-1000	-	Close to DL
COS (100) 150C	-0.03	0.25	0.02	0.02	0.02	0.34	0.34	0.02	0.24	0	0.01	0-100	-	Close to DL
ETHANE (500) 191C	0.91	2.74	0.32	0.2	0.22	3.06	3.06	0.54	2.53	0.22	0.11	0-500	-	Close to DL
H2SO4 (50) 150C	-0.12	0.22	0.04	0.05	0.05	0.24	0.24	0.29	0.29	0.25	0.01	0-50	-	Close to DL
MEOH (10) 191C	0.19	0.6	0.14	0.14	0.16	0.69	0.69	0.17	0.47	0.03	0.05	0-10	-	Close to DL
SO3 (150) 191C	-13.54	0.85	0.7	0.03	0.04	0.92	0.92	1.14	1.17	0.44	0.23	0-150	-	Check it!

Analysis Validation Report

Sample Filename: F:\Ada\January 8\SPC\_007093.LAB

Filename for noise: F:\Midlothian on Rental\November 14\SPC\_000837.LAB

Interferences Filenames: C:\Midlothian on Rental\November 15\SPC\_001463.LAB

C:\Midlothian on Rental\November 15\SPC\_001464.LAB

C:\Midlothian on Rental\November 15\SPC\_001465.LAB

C:\Midlothian on Rental\November 15\SPC\_001466.LAB

C:\Midlothian on Rental\November 15\SPC\_001467.LAB

C:\Midlothian on Rental\November 15\SPC\_001468.LAB

C:\Midlothian on Rental\November 15\SPC\_001469.LAB

C:\Midlothian on Rental\November 15\SPC\_001470.LAB

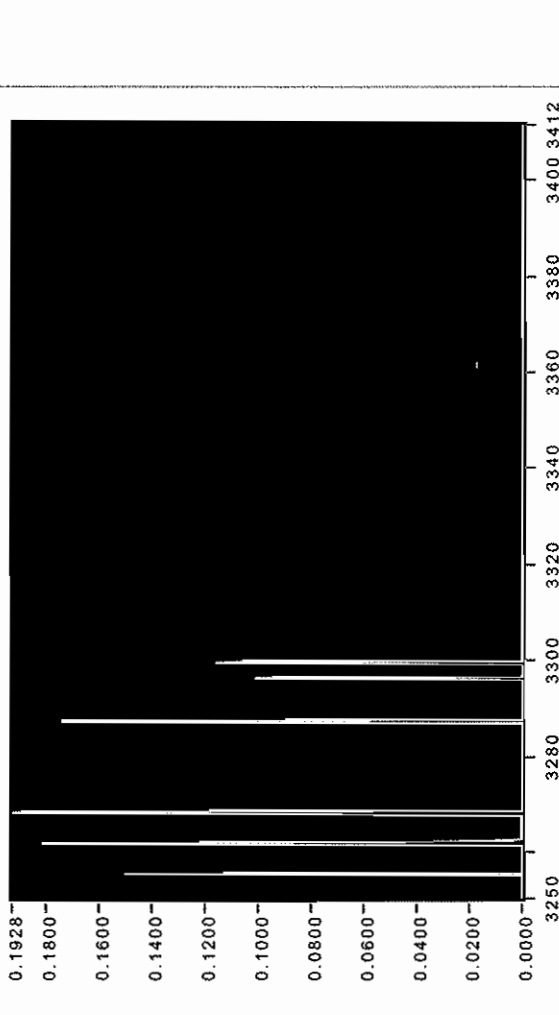
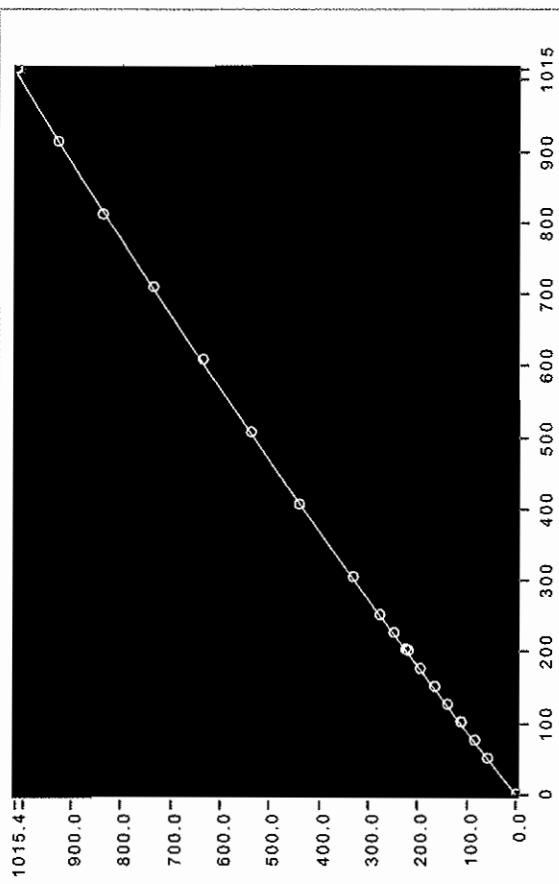
Recipe path: C:\OLT\recipes\Cement Testing R3.MGRCP

Gas calibration Name	Conc	MDC3	MDC2	MDC1	MAU	FMU*R	OCU	~DL	~CL	~Bias	Sigma	Range	Span	Comment
ADA 1 HCN (200) PCA 191C 191C	1.33	0.13	-	0.29	0.35	0.15	0.35	-	0.05	-	-	0-200	-	Good
HF PPM (10) 191C	-0.16	0.39	0.05	0.04	0.07	0.7	0.7	0.07	0.35	0.02	0.02	0-10	-	Close to DL
SF6 (10) 191C	0	0.02	-	0	0	0.02	0.02	-	0.01	-	-	0-10	-	Close to DL
ETHYLENE (100,3000) 191C	0.8	1.52	0.33	0.17	0.29	2.65	2.65	1.08	1.34	0.75	0.11	0-3000	-	Close to DL
H2O% (40) 191C	24.25	0.42	-	0.01	0.02	0.8	0.8	-	0.49	-	-	0-40	-	Good
CO2% (40) 191C	6.69	0.15	-	0.01	0.02	0.21	0.21	-	0.13	-	-	0-40	-	Good
HCL PPM (100) 191C	0.43	0.5	0.05	0.15	0.28	0.91	0.91	0.06	0.36	0.01	0.02	0-100	-	Close to DL
SO2 (1000) 191C	-0.54	1.88	0.37	0.14	0.27	3.62	3.62	0.79	1.74	0.42	0.12	0-1000	-	Close to DL
NO (350,3000) 191C	75.89	7.91	0.22	0.26	0.35	10.76	10.76	0.45	5.63	0.23	0.07	0-3000	-	Good
NH3 (300) 191C (1OF2)	3.96	1.23	0.03	0.12	0.2	2.08	2.08	0.15	1	0.11	0.01	0-300	-	Check it!
CO (500) 191C (1OF2)	2.9	2.31	-	0.2	0.43	4.98	4.98	-	2.04	-	-	0-500	-	Check it!
CO% (1) 191C (2OF2)	0	0	0	0	0	0	0	0	0	0	0	0-1	-	Close to DL
FORMALDEHYDE (70) 191C	0.54	0.97	0.28	0.2	0.24	1.16	1.16	0.33	0.76	0.04	0.09	0-70	-	Close to DL
ACETALDEHYDE (1000) 191C	1	2.07	0.23	0.35	0.38	2.25	2.25	0.59	1.69	0.35	0.08	0-1000	-	Close to DL
CH4 (250) 191C (1OF2)	0.9	1.55	0.05	0.26	0.7	4.09	4.09	0.06	1.27	0.01	0.02	0-250	-	Check it!
PROPANE (100) 191C	2.23	1.47	0.42	0.12	0.14	1.75	1.75	0.72	1.29	0.3	0.14	0-100	-	Close to DL
HBR (100) 180C	-0.22	5.36	0.17	0.68	1.39	10.89	10.89	0.19	4.7	0.02	0.06	0-100	-	Close to DL
NO2 (150) 191C (1OF2)	-0.51	0.78	0.04	0.02	0.03	0.82	0.82	0.05	0.75	0.01	0.01	0-150	-	Check it!
NO2 (2000) 191C (2OF2)	2.2	7.21	2.03	0.88	1.12	9.19	9.19	3.81	6.3	1.78	0.68	0-2000	-	Close to DL
N2O (100,200,300) 191C	0.23	0.28	0.05	0.04	0.05	0.33	0.33	0.07	0.24	0.02	0.02	0-300	-	Close to DL
NH3 (3000) 191C (2OF2)	8.6	21.46	0.35	1.19	2.25	40.48	40.48	0.44	20.24	0.09	0.12	0-3000	-	Check it!
CH4 (3000) 191C (2OF2)	1.58	16.3	0.34	1.1	1.81	26.85	26.85	0.61	15.2	0.27	0.11	0-3000	-	Close to DL
ACETYLENE (1000) 191C	-0.03	2.75	0.15	0.31	0.37	3.33	3.33	0.21	2.46	0.06	0.05	0-1000	-	Close to DL
PROPYLENE (200,1000) 191C	1.11	3.92	0.19	0.43	0.5	4.6	4.6	0.49	3.48	0.3	0.06	0-1000	-	Close to DL
COS (100) 150C	-0.03	0.21	0.02	0.02	0.02	0.28	0.28	0.02	0.19	0	0.01	0-100	-	Close to DL
ETHANE (500) 191C	0.84	2.49	0.32	0.2	0.22	2.78	2.78	0.54	2.28	0.22	0.11	0-500	-	Close to DL
H2SO4 (50) 150C	-0.09	0.19	0.04	0.05	0.05	0.21	0.21	0.29	0.29	0.25	0.01	0-50	-	Close to DL
MEOH (10) 191C	-0.08	0.48	0.14	0.14	0.16	0.56	0.56	0.17	0.36	0.03	0.05	0-10	-	Close to DL
SO3 (150) 191C	-8.32	0.78	0.7	0.03	0.04	0.84	0.84	1.14	1.15	0.44	0.23	0-150	-	Check it!

Holcim; Ada OK  
Kiln 3 Main Stack  
Run 3

Spectrum	Date	Time	SNR 2500	sBeam @ 2500
SPC__007306.LAB	01/08/24	19:04:30.648	6836.45752	1.315389

Calibration Generated 02/13/2024 02:43 PM Gas ID Ada 1 HCN (200) PCA 191C 191c LRF Path C:\OLT\Methods\R3\Cement-coal-EGU method 191C\Ada 1 HCN (200) PCA 191C 191c\_18.lrf



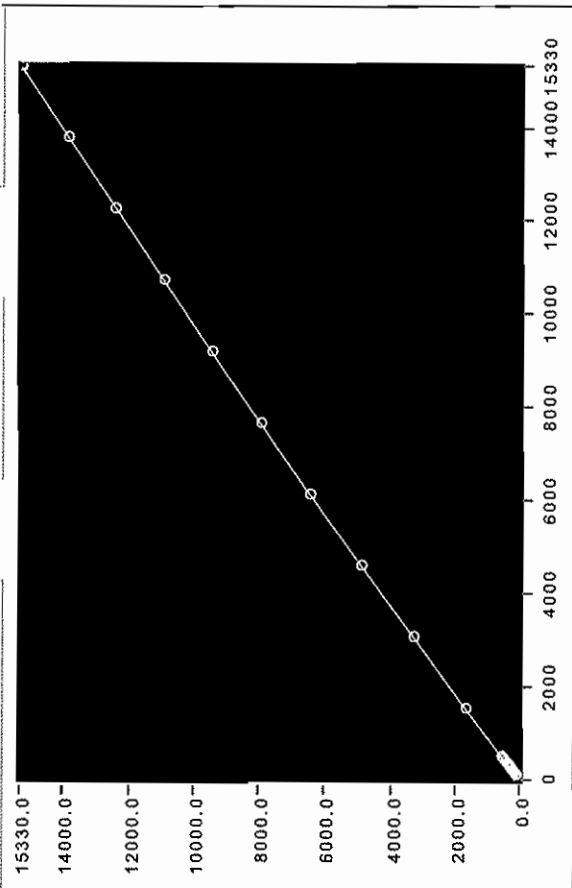
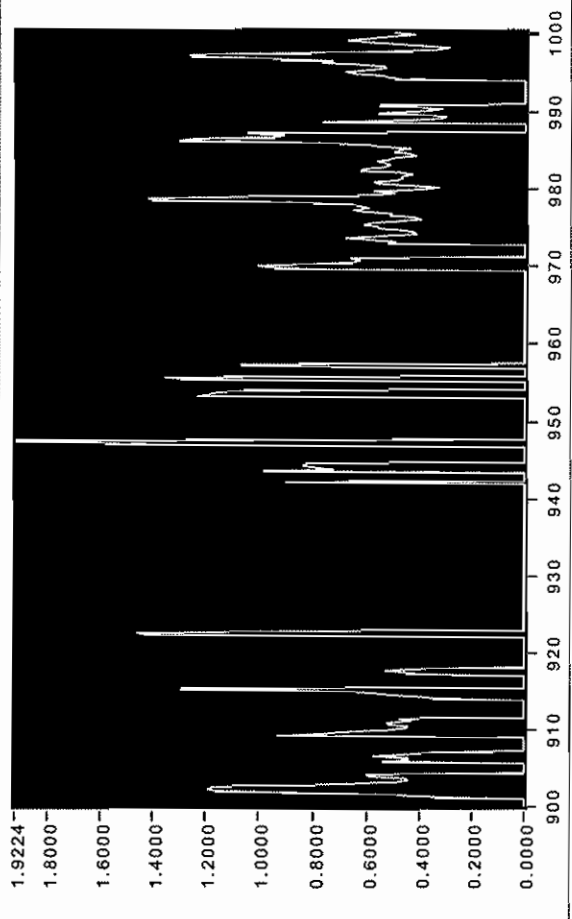
Cal Spectra	Temp (C)	Pres (atm)	Conc (ppm-m)	Actual	Calc'd
HCN (200) PCA 191C ( 9.86ppm, 5.11)	191.209	0.977	50.385	50.338	58.529
HCN (200) PCA 191C ( 14.79ppm, 5.11)	191.194	0.977	75.577	75.505	84.369
HCN (200) PCA 191C ( 19.72ppm, 5.11)	191.202	0.977	100.769	100.670	111.322
HCN (200) PCA 191C ( 19.87ppm, 5.11)	191.196	0.978	101.536	101.566	113.223
HCN (200) PCA 191C ( 24.65ppm, 5.11)	191.204	0.977	125.962	125.832	140.433
HCN (200) PCA 191C ( 29.58ppm, 5.11)	191.209	0.977	151.154	150.999	166.993
HCN (200) PCA 191C ( 34.51ppm, 5.11)	191.225	0.977	176.346	176.168	196.595
HCN (200) PCA 191C ( 39.44ppm, 5.11)	191.217	0.977	201.538	201.349	221.224
HCN (200) PCA 191C ( 39.74ppm, 5.11)	191.194	0.978	203.071	203.055	225.759
HCN (200) PCA 191C ( 44.37ppm, 5.11)	191.203	0.977	226.731	226.500	249.527
HCN (200) PCA 191C ( 49.30ppm, 5.11)	191.199	0.977	251.923	251.691	277.705
HCN (200) PCA 191C ( 59.61ppm, 5.11)	191.221	0.978	304.607	304.577	332.001
HCN (200) PCA 191C ( 79.48ppm, 5.11)	191.195	0.977	406.143	405.954	441.406
HCN (200) PCA 191C ( 99.35ppm, 5.11)	191.258	0.977	507.679	507.181	539.668
HCN (200) PCA 191C ( 119.22ppm, 5.11)	191.209	0.977	609.214	608.643	636.593
HCN (200) PCA 191C ( 139.09ppm, 5.11)	191.246	0.977	710.750	709.953	736.674
HCN (200) PCA 191C ( 158.96ppm, 5.11)	191.263	0.977	812.286	811.364	839.368
HCN (200) PCA 191C ( 178.83ppm, 5.11)	191.221	0.977	913.821	913.339	930.091
HCN (200) PCA 191C ( 198.70ppm, 5.11)	191.202	0.978	1015.357	1015.357	1015.357

Interpolation Quadratic  Force Through Zero   
 Min % Residuals  Excl. Zero  Quant w/ Highest

Spectral Regions

- Region 00 - 3250.25 to 3411.52 cm-1
- Region 01 - 593.01 to 840.34 cm-1
- Region 02 - 1295.95 to 1518.21 cm-1
- Region 03 - 2496.93 to 2901.43 cm-1
- Region 04 - 3172.38 to 3250.01 cm-1
- Region 05 - 3881.35 to 4113.97 cm-1

Calibration Generated: 11/30/2012 11:50 AM  
 Gas ID: Ethylene (100,3000) 191C  
 LRF Path: C:\OLTM\Methods\R3\Cement-coal-EGU method 191C\Ethylene (100,3000) 191C\_19.lrf  
 Lo Alarm: NaN  
 Hi Alarm: NaN  
 Span: Mutations  
 Offset: .J0000  
 Mutations: FALSE



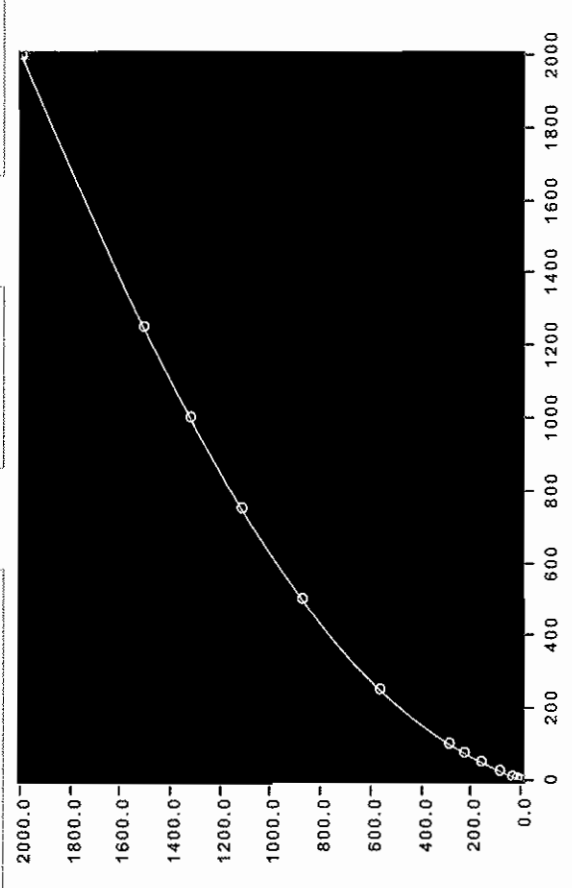
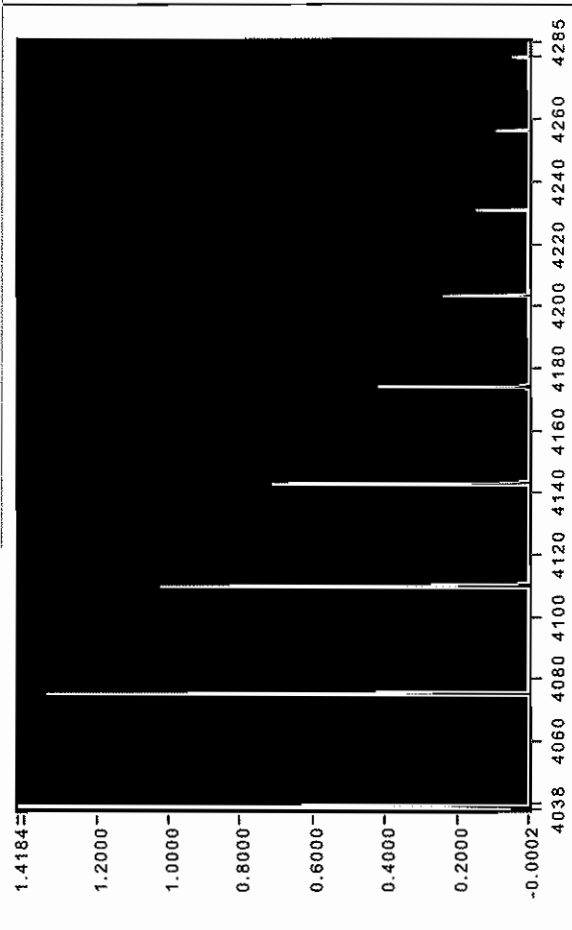
Cal Spectra	Temp (C)	Pres (atm)	Conc (ppm-m)	Actual	Calc'd
Ethylene ( 9.74ppm, 5.11m, 190C).lab	190.014	0.994	49.792	49.542	54.522
Ethylene ( 19.49ppm, 5.11m, 190C).lab	189.989	0.994	99.594	99.135	107.070
Ethylene ( 29.23ppm, 5.11m, 190C).lab	189.985	0.994	149.365	148.729	162.298
Ethylene ( 38.98ppm, 5.11m, 190C).lab	189.988	0.995	199.188	198.392	217.388
Ethylene ( 48.72ppm, 5.11m, 190C).lab	189.971	0.995	248.959	247.990	272.190
Ethylene ( 58.46ppm, 5.11m, 190C).lab	189.964	0.995	298.731	297.587	327.537
Ethylene ( 68.21ppm, 5.11m, 190C).lab	189.977	0.995	348.553	347.251	383.508
Ethylene ( 77.95ppm, 5.11m, 190C).lab	189.933	0.995	398.325	396.868	439.409
Ethylene ( 87.70ppm, 5.11m, 190C).lab	189.964	0.995	448.127	446.506	495.696
Ethylene ( 97.44ppm, 5.11m, 190C).lab	189.934	0.995	497.918	496.089	551.021
Ethylene ( 300.00ppm, 5.11m, 190C).lab	189.942	0.997	1533.000	1531.357	1654.977
Ethylene ( 600.00ppm, 5.11m, 190C).lab	189.991	0.998	3066.000	3063.066	3290.420
Ethylene ( 900.00ppm, 5.11m, 190C).lab	189.934	0.998	4599.000	4597.015	4885.213
Ethylene ( 1200.00ppm, 5.11m, 190C).lab	189.933	0.998	6132.000	6130.754	6446.341
Ethylene ( 1500.00ppm, 5.11m, 190C).lab	189.960	0.998	7664.999	7658.046	7940.995
Ethylene ( 1800.00ppm, 5.11m, 190C).lab	189.922	0.998	9197.999	9195.039	9445.204
Ethylene ( 2100.00ppm, 5.11m, 190C).lab	189.971	0.998	10730.999	10728.462	10942.161
Ethylene ( 2400.00ppm, 5.11m, 190C).lab	189.936	0.998	12263.999	12262.913	12444.229
Ethylene ( 2700.00ppm, 5.11m, 190C).lab	189.914	0.998	13796.999	13797.629	13888.468

Interpolation:  Quartic  
 Min % Residuals:  Excl. Zero  Quant w/ Highest   
 Force Through Zero

Spectral Regions

- Region 00 - 900.12 to 1000.16 cm-1
- Region 01 - 615.91 to 899.88 cm-1
- Region 02 - 1000.40 to 1211.33 cm-1
- Region 03 - 1341.75 to 2117.96 cm-1
- Region 04 - 2901.41 to 3286.87 cm-1

Calibration Generated: 06/26/2012 10:22 AM  
 Gas ID: HF ppm (10) 191C  
 LRF Path: C:\OLT\Methods R3\Cement-coal-EGU method 191C\HF ppm (10) 191C\_12.lrf  
 Lo Alarm: NaN  
 Hi Alarm: NaN  
 Span: Mutations  
 Offset: J0000  
 Mutations: FALSE



Cal Spectra	Temp (C)	Pres (atm)	Conc (ppm-m)	Actual	Calc'd
HF 96 MNB-fz 1ppm-m (1ppm, 1.000m, 1	191.000	1.000	1.000	1.000	3.620
HF 96 MNB-fz 5ppm-m ( 5ppm, 1.000m, 1	191.000	1.000	5.000	5.000	17.914
HF 96 MNB-fz 10ppm-m (10ppm, 1.000m, 1	191.000	1.000	10.000	10.000	35.375
HF 96 MNB-fz 25ppm-m ( 25ppm, 1.000m, 1	191.000	1.000	25.000	25.000	85.208
HF 96 MNB-fz 50ppm-m ( 50ppm, 1.000m, 1	191.000	1.000	50.000	50.000	160.659
HF 96 MNB-fz 75ppm-m ( 75ppm, 1.000m, 1	191.000	1.000	75.000	75.000	228.048
HF 96 MNB-fz 100ppm-m (100ppm, 1.000m, 1	191.000	1.000	100.000	100.000	288.789
HF 96 MNB-fz 250ppm-m ( 250ppm, 1.000m, 1	191.000	1.000	250.000	250.000	565.147
HF 96 MNB-fz 500ppm-m ( 500ppm, 1.000m, 1	191.000	1.000	500.000	500.000	875.753
HF 96 MNB-fz 750ppm-m ( 750ppm, 1.000m, 1	191.000	1.000	750.000	750.000	1115.993
HF 96 MNB-fz 1000ppm-m (1000ppm, 1.000m, 1	191.000	1.000	1000.000	1000.000	1322.828
HF 96 MNB-fz 1250ppm-m ( 1250ppm, 1.000m, 1	191.000	1.000	1250.000	1250.000	1509.378
HF 96 MNB-fz 2000ppm-m ( 2000ppm, 1.000m, 1	191.000	1.000	2000.000	2000.000	2000.000

Interpolation:  Spline  Force Through Zero  
 Min % Residuals:  Excl. Zero  Quant w/ Highest   
 Spectral Regions:  
 Region 00 - 4037.90 to 4284.87 cm-1  
 Region 01 - 3405.41 to 4037.84 cm-1  
 Region 02 - 4284.93 to 4363.63 cm-1



## **Appendix F**

### **Test Participants**

Scott Steinsberger

Project Manager and FTIR Operator

Dustin Carpenter

Sampling Technician

Lee Harris

Sampling Technician

Shelby Hudgens

Holcim Plant Contact

## **Appendix G**

### **RTR Sampling and Analytical Protocol**



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(919) 250-0285 (ph); (919) 250-1835 (Fax)

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**PROTOCOL TO PERFORM A SAMPLING  
AND ANALYTICAL TESTING PROGRAM  
AS PART OF THE US EPA RISK AND TECHNOLOGY REVIEW  
at  
Holcim Inc.  
Ada Facility  
1100 West 18th Street  
Ada, Oklahoma 74820**

**Submitted By:  
DEECO, INC.  
3404 Lake Woodard Road  
Raleigh, NC 27604**

**September 29, 2023**

**Copy # 1**

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## APPENDICES

Appendix A - Sampling and Analytical Methods

## 1.0 INTRODUCTION

### 1.1 SUMMARY OF TEST PROGRAM

The United States Environmental Protection Agency (US EPA) has directed the portland cement industry (SIC 3241) to conduct emissions testing as part of the US EPA Risk and Technology Review (RTR). This document provides the overall test program approach and specifies minimum sample collection procedures, data quality objectives, and quality assurance/quality control measures to be used by the source testing firms selected by the cement companies performing tests. The test program is designed to be a comprehensive and robust test of each facility. The quality assurance and quality control (QA/QC) measures are designed to produce standardized data having known precision and accuracy. Collection of accurate, representative, and standardized data for facilities with low emissions is necessary especially in view of MACT standard setting procedures.

Cement kiln pyro-processing systems located throughout the US will be included in this request. Individual facilities have a wide range of kiln system configurations and air pollution control (APC) trains. Site-specific considerations will be required to capture emissions profiles for the target analytes that represent the extent of control or possible emissions increases from these controls.

### 1.2 PLANT NAME, ADDRESS, AND CONTACT

Holcim (US) Inc. - Ada Facility  
1100 West 18 th Street  
Ada, Oklahoma 74820

Mr. Shelby Hudgens  
Telephone: (580) 618-1232  
Fax: (580) 421-8971  
E-Mail [shelby.hudgens@holcim.com](mailto:shelby.hudgens@holcim.com)

### 1.3 PROCESS OF INTEREST

The process to be tested at the Ada facility is a semi-dry kiln, equipped with a single stage preheater and flash calciner in front, producing portland cement. There is no inline raw mill.

### 1.4 AIR POLLUTION CONTROL EQUIPMENT

The modern semi-dry technology utilizes a single stage preheater and flash calciner arrangement in front of the kiln. This additional step helps to promote self-combustion of organics that evolve from the raw materials. The air pollution control equipment at the Ada facility consists of multiple control devices; inherent dry scrubbing for SO<sub>2</sub>; inline low NO<sub>x</sub> calciner, low NO<sub>x</sub> burner and selective non-catalytic reduction (SNCR) for NO<sub>x</sub>. The selective non-catalytic reduction (SNCR) post combustion emissions control technology reduces NO<sub>x</sub> by injecting ammonia into the process at a properly determined location. (**NOTE:** The add-on SNCR control technology is included in the design due to Holcim's corporate policy, not for regulatory compliance.) A fabric filter baghouse controls PM emissions from the clinker cooler and alkali bypass as well as the kiln.

## **1.5 EMISSION POINTS AND SAMPLING LOCATIONS**

The emissions from the clinker cooler, alkali bypass, and kiln are co-mingled in to a common stack.

## **1.6 POLLUTANTS TO BE MEASURED**

Emission testing will be conducted for hydrogen cyanide (HCN), hydrogen fluoride (HF), and diatomic chlorine (Cl<sub>2</sub>). Concurrent measurements to determine volumetric flow rate will be made. The sampling and analytical procedures to be followed are discussed in detail in Section 4.

## **1.7 EXPECTED TEST DATES**

Test dates are to be determined.

## **1.8 TEST PROGRAM ORGANIZATION**

The test program organizational chart is presented in Figure 1.1.

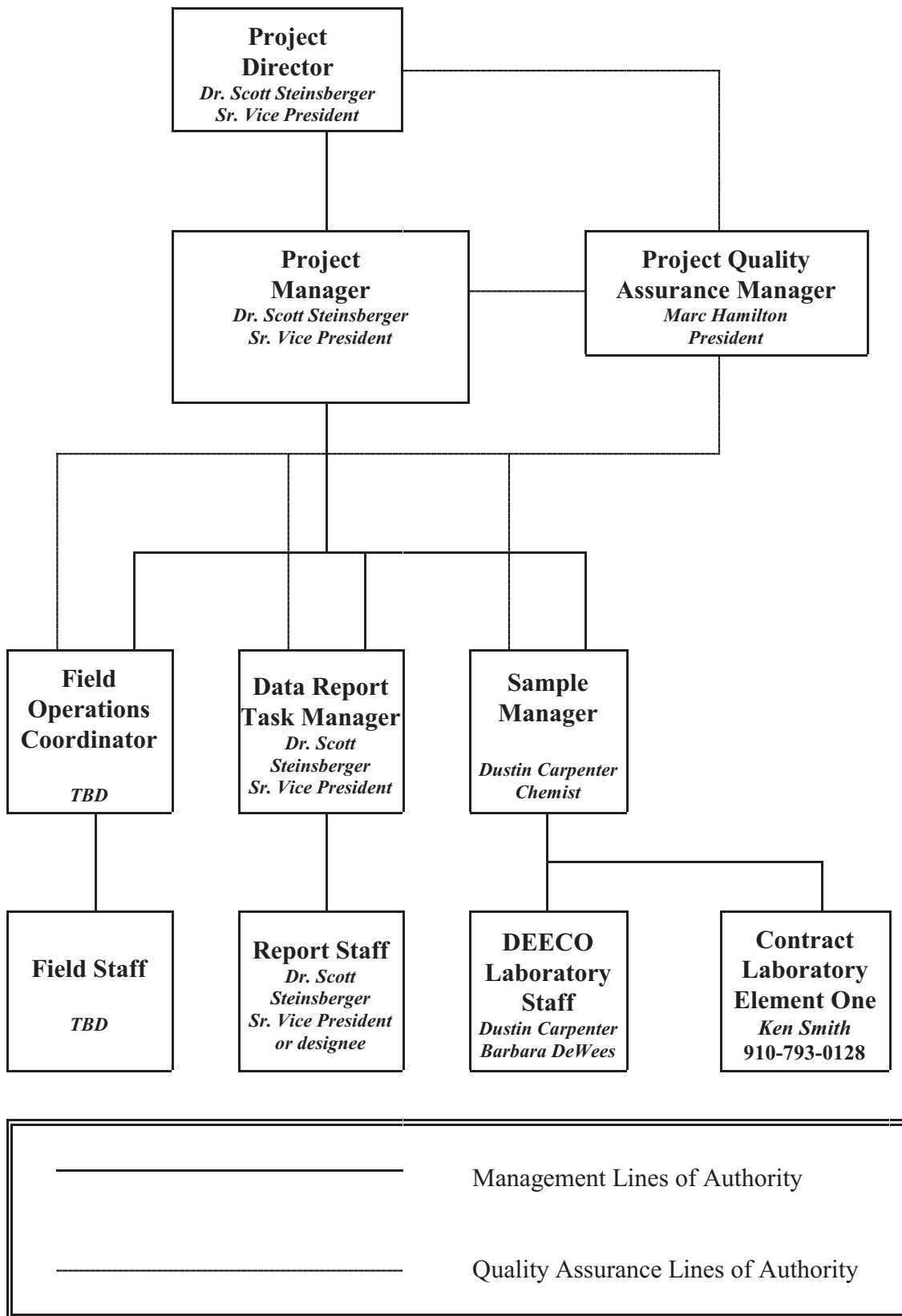


Figure 1.1 Organizational Chart

## 2.0 SOURCE DESCRIPTION

### 2.1 PROCESS DESCRIPTION

The Holcim Ada facility operates one (1) semi-dry single-stage preheater/precalciner kiln line, 12 feet in diameter by 177 feet in length, with a nominal clinker production capacity of 2,200 TPD and a true capacity of 764,453 TPY. The facility also operates process units which conduct the following operations:

- Mining and transport of raw materials (limestone and shale);
- Raw material crushing, screening, and transport operations;
- Coal crushing, transport, and storage;
- Raw material preparation, transport, and storage;
- Clinker cooling, transport, and storage;
- Finish milling resulting in production of cement;
- Product transport, storage, loading, and shipping.

Holcim Ada's cement manufacturing process begins with mining (i.e., drilling and blasting) of raw materials: calcium carbonate (limestone) and argillaceous materials (shale) from the quarry. The raw materials are then loaded into trucks which haul the materials to be unloaded at the primary crusher. The materials then travel by conveyor to the secondary crusher where they are discharged onto an overland conveyor belt system.

The conveying system transports the raw material to the raw material storage silos. It is then fed into the raw mills. Water is added to the materials, which are then ground into slurry. Additional raw materials, such as sand, spent catalyst, mill scale, etc. may be added to the mix to adjust the chemistry of the slurry to meet quality standards.

The slurry is pumped from the raw mills to the kiln feed storage tanks. The kiln feed is pumped to the kiln system, where it undergoes physical and chemical changes during the heating process to form hydraulic calcium silicates found in clinker. Heat for the kiln is provided by natural gas, coal, tire-derived fuel (TDF), liquid fuels (i.e., non-hazardous used oil and bio-diesel) and/or other approved alternative non-hazardous fuels. The kiln exhaust is vented through a baghouse. Cement kiln dust (CKD) collected by the baghouse is transported to a waste dust bin, insufflation bin, or shipping silos. The kiln is also equipped with selective non-catalytic reduction (SNCR) for NO<sub>x</sub> control.

The clinker is discharged from the kiln into the clinker cooler and then transported to clinker storage silos. The clinker cooler exhausts to the same baghouse as the kiln. From the clinker storage silos, the clinker is conveyed to the finish mills to be finely ground. Gypsum and additional materials are ground with the clinker. The cement is transported from the finish mill to the shipping silos or packing silos for distribution by truck, rail, or bagged product. Dust collectors are in place to collect and recover product and also control emissions from the bagging. Dust collectors in the shipping area are used to convey product to and between silos and to the truck and rail loading areas.

Coal, TDF, whole tires and other solid fuels are received by truck or rail. Rail cars are unloaded with a shaker apparatus, if necessary, to remove the materials from the cars. The coal is stored outside and transferred to the coal silo and then conveyed to coal mills for grinding into a fine powder prior to injection into the kiln or precalciner.

## 2.2 CONTROL EQUIPMENT DESCRIPTION

The air pollution control equipment at the Ada facility consists of multiple control devices; inherent dry scrubbing for SO<sub>2</sub>; inline low NO<sub>x</sub> calciner, low NO<sub>x</sub> burner and selective non-catalytic reduction (SNCR) for NO<sub>x</sub>. The selective non-catalytic reduction (SNCR) post combustion emissions control technology reduces NO<sub>x</sub> by injecting ammonia into the process at a properly determined location. (NOTE: The add-on SNCR control technology is included in the design due to Holcim's corporate policy, not for regulatory compliance) A fabric filter baghouse controls particulate emissions from the clinker cooler and alkali bypass as well as the kiln.

A schematic of the Ada process, including control equipment is shown below in Figure 2.1.

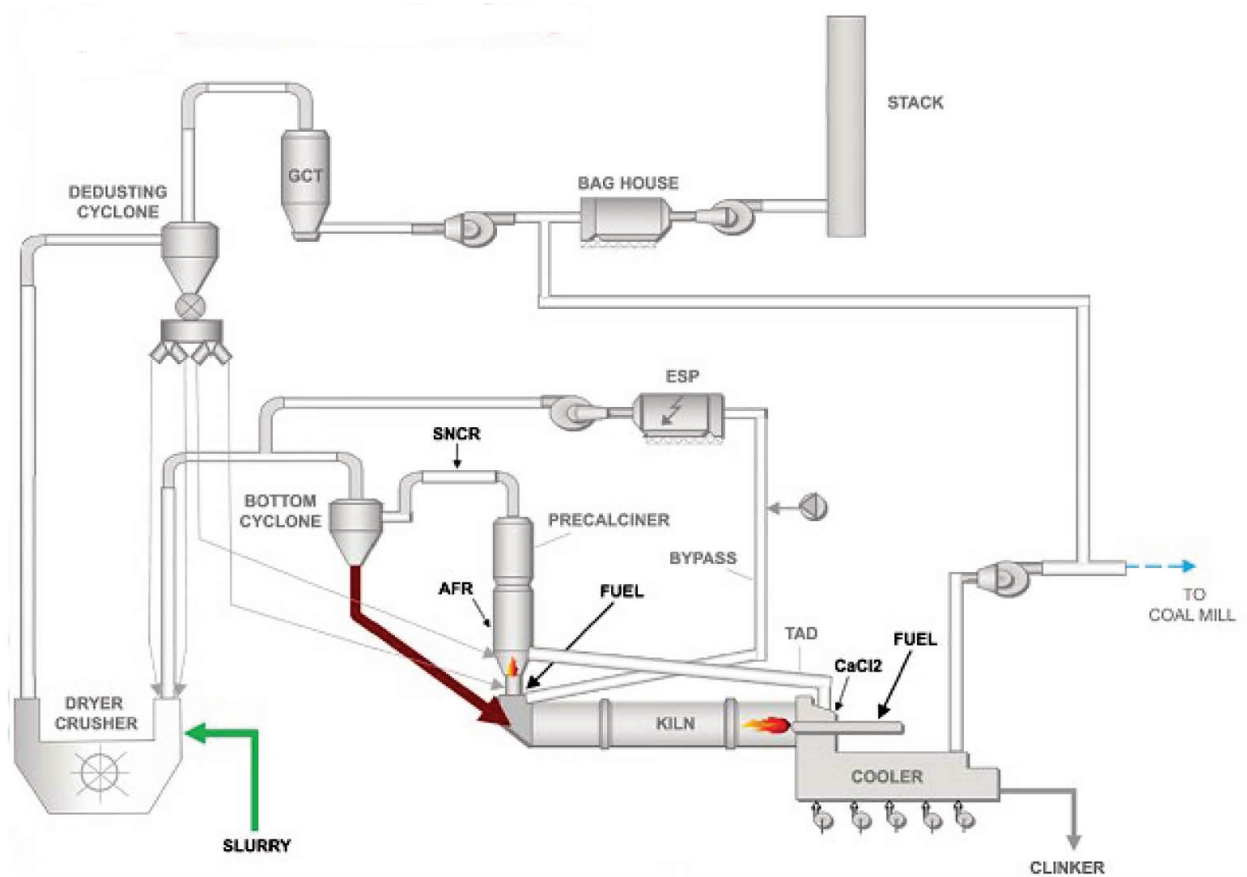


Figure 2.1 Ada Detailed Process Schematic

### 3.0 TEST PROGRAM

#### 3.1 OBJECTIVES

An air emissions sampling and analytical program will be conducted on the Common stack at the Ada cement facility located in Ada, Oklahoma. All testing will be performed following accepted EPA methodology. The test program is to provide a standardized data set to the EPA and the cement industry so that reliable facility inter-comparisons of emissions can be made.

All testing will be performed in strict accordance with "DRAFT GENERAL TEST PLAN Testing To Determine HCN, HF, and Cl<sub>2</sub> Emissions From Cement Kilns" dated March 2, 2023" and the specifications stipulated in 40 CFR 60, Appendix A for flow rate following EPA Method 1, 2, 3A, and 4) and hydrogen fluoride (HF) and diatomic chlorine (Cl<sub>2</sub>) following EPA Method 26A and in 40 CFR 63, Appendix A for hydrogen cyanide (HCN) and (HF) following EPA Method 320. All sampling runs will be one hour long.

The source emission test will be performed on a date to be determined. Testing will be conducted under representative process and control system operating conditions. For facilities with inline raw mills, testing will be performed while operating in the "Mill On" and "Mill Off" conditions. Ada has no inline raw mill, therefore only a single operating condition will be tested.

#### 3.2 TEST MATRIX

Table 3-1 presents the sampling and analytical matrix and proposed test schedule.

**TABLE 3-1 PROGRAM OUTLINE AND TENTATIVE TEST SCHEDULE**

Sampling Location	No. of runs	Sample/Type Pollutant	Sampling Method	Sample Run Times (min)	Analytical Method	Analytical Laboratory
<b>Day 1</b>						
Stack	Arrive on-site and set up test equipment on the Kiln 3 common stack					
<b>Day 2</b>						
Kiln 3 Stack	3	O <sub>2</sub> /CO <sub>2</sub>	EPA Method 3A	60	Paramagnetic (O <sub>2</sub> ) NDIR (CO <sub>2</sub> )	DEECO
	3	HF and Cl <sub>2</sub>	EPA Method 26A <sup>1</sup>	60	Ion Chromatograph	Element One
	3	HCN and HF	EPA Method 320	60	FTIR (Method 320)	DEECO

<sup>1</sup> Stack gas flow rate and moisture measurement may be taken from concurrent Method 26A sampling trains.

### **3.3 TEST COORDINATION**

Mr. Shelby Hudgens, the Ada facility contact, will serve as the test coordinator and will be responsible for:

1. Scheduling the start of all testing
2. Principal contact with the agency officials concerning the tests
3. Principal contact with DEECO concerning the tests
4. Recording the process data during the testing
5. Providing copies of any field test data to the agency

If there is a temporary equipment malfunction in the middle of a test, radio contact will be made with the test crew in order to delay the test. When problems have been corrected, the test will continue from the point where it was delayed. If the malfunction or upset condition results in an extended test delay, then the affected test run(s) may be aborted and a new run(s) conducted when the malfunction has been corrected or process upset cleared. Any samples or field data from aborted runs may be discarded.

## **4.0 SAMPLING LOCATION DESCRIPTIONS**

### **4.1 SAMPLING LOCATION DESCRIPTION**

The Kiln 3 common stack is vertically oriented, round stack with an inside diameter of 139". The test location is approximately 1571" downstream (11.3 diameters) of the duct breaching and 1218" (8.8 Diameters) upstream of the stack outlet. The sampling locations meet the minimum specifications for selection of a measurement site as outlined in EPA Method 1. Cyclonic flow checks, as described in EPA Method 1 Section 2.4, using the Type-S pitot null procedure and angle measurements will be conducted at the Common stack test location.

A twelve (12) point sampling traverse will be made using six (6) point traverses in each of two perpendicular directions (or 3 points in each of 4 sampling ports) at the Common stack. Each traverse will be made at each sampling location using a type-S pitot tube in accordance with EPA Methods 2 procedures. Gas temperatures are to be measured using calibrated Type K thermocouples and digital readout devices. All measurements are to be performed in accordance with the procedures in EPA Methods 2, and 26A.

A schematic of the common stack is provided in Figure 4.1.

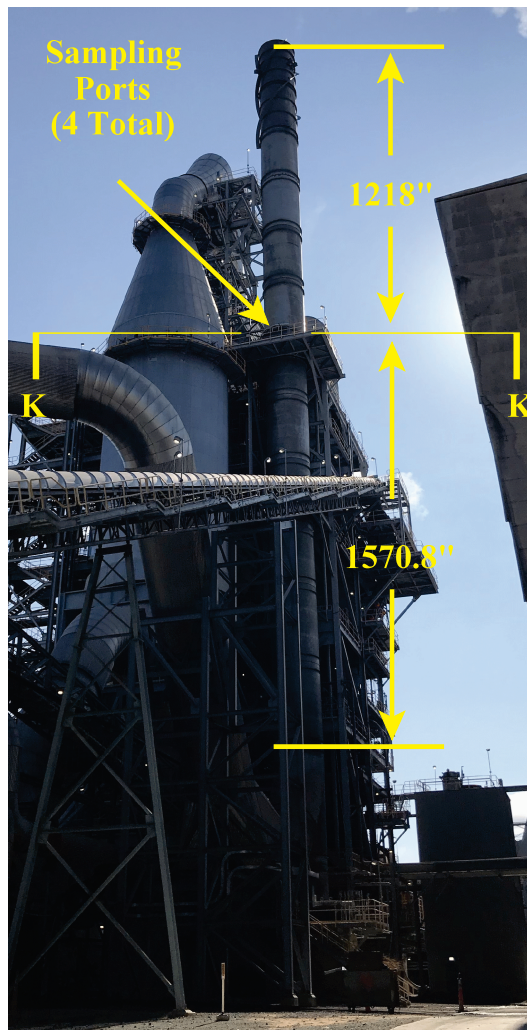
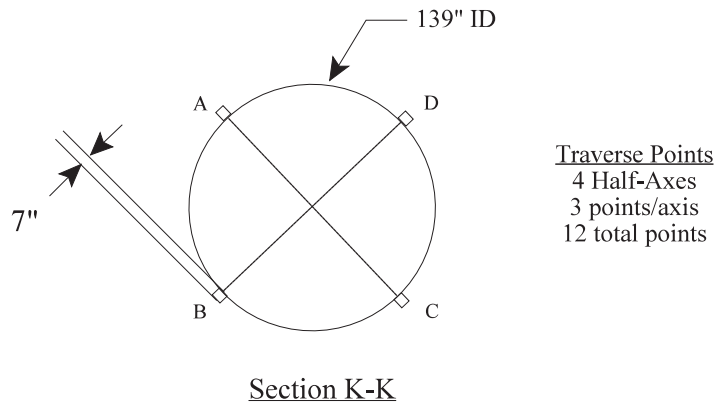


Figure 4.1 Schematic of Stack Sampling Location

## **5.0 SAMPLING AND ANALYTICAL PROCEDURES**

This section contains a brief description of the sampling and analytical procedures for each method that will be employed during the test program. All equipment, procedures, and quality assurance measures necessary for completion of the test program will meet or exceed the specifications of the appropriate methods. Any deviations from the methods to ensure quality representativeness of the results are also discussed.

### **5.1 TEST METHODS**

The methods for the test program are described below, and apply to all process operating conditions (e.g. where there is an inline raw mill, testing will be performed while operating in the "Mill On" and "Mill Off" conditions). Table 3-1 outlines expected operating conditions for this test.

#### **5.1.1 SAMPLING POINT DETERMINATION - EPA METHOD 1**

The number and location of the sampling or traverse points will be determined according to the procedures outlined in EPA Method 1. The sample location will be inspected to insure EPA Method 1 criteria is met. All points will be at least 1.0 inches from the stack wall, per Method 1.

#### **5.1.2 FLUE GAS VELOCITY AND VOLUMETRIC FLOW RATE - EPA METHOD 2**

The flue gas velocity and volumetric flow rate will be determined according to the procedures outlined in EPA Method 2. Velocity measurements will be made using type S pitot tubes conforming to the calibration specifications outlined in EPA Method 2, Section 10.1. Each Type-S pitot tube, calibrated according to these standards, will have an assigned coefficient. Differential pressures will be measured with Magnehelic gauges of appropriate range or with fluid manometers. Effluent gas temperatures will be measured with chromel-alumel thermocouples equipped with digital readouts.

#### **5.1.3 OUTLET FLUE GAS COMPOSITION - EPA METHOD 3A**

Outlet flue gas analysis for oxygen (O<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>) concentrations, and the calculation of percent excess air and flue gas dry molecular weight will be performed in accordance with EPA Method 3A.

To evaluate the sampling location and points for FTIR and O<sub>2</sub> sampling, a three-point O<sub>2</sub> concentration stratification test on a line passing through the centroidal area at 16.7, 50.0 and 83.3 percent of the measurement line (or for stacks is greater than 2.4 meters (7.8 ft) at 0.4, 1.2 and 2.0 meters from the stack or duct wall). The procedures in Section 8.1.2 of Method 7E will be followed, but oxygen will be used as parameter as allowed by fourth sentence in Section 8.1.2. The plant O<sub>2</sub> CEMS as a control. A criteria of <5% variation from combined mean for each point will be used as indication of non-stratification and allowing single point sampling at the point closest to the mean. Otherwise, sampling for equal periods at the three test points during test run will be conducted.

Per EPA Method 3A for determining molecular weight, integrated sampling will be obtain using the Method 320 sampling system described in Section 5.1.6.

A portion of the hot, wet gas sample will be sent through a condensing system to remove the stack moisture, A portion of the moisture-free gas sample will be sent to a CAI Model 200 O<sub>2</sub> (or

equivalent) analyzer measures using the paramagnetic technique. An oxygen molecule, because of its sp<sup>3</sup> electron orbital distribution, has an unpaired electron and hence displays a magnetic orientation. Since other elements that display this magnetic phenomenon are not common gasses at normal temperatures, the paramagnetic measurement technique is virtually specific for oxygen. The sample gas flows through a detection cell located in a very strong magnetic field. The concentration of O<sub>2</sub> gas present induces a pressure differential in the detector cell. The amount of differential pressure is proportional to the concentration of O<sub>2</sub> gas present.

Calibration procedures will be performed in accordance with EPA methodology. Analyzers will be calibrated before and after each test and a calibration check between each test run.

The pretest calibrations will consist of the following steps:

- Internal (direct) calibration of each analyzer to adjust calibration and check linearity.
- External (through the entire sampling system) calibration to check the system bias on zero and span gases.

The post test calibration will consist of an external system bias calibration check.

The analyzer will be as calibrated using a certified zero and span (mid or high range) gas. Zero and span gases were directed to each analyzer through the appropriate plumbing, the calibration gas flow rates will be adjusted to the correct flow rate and the analyzer will be adjusted with the appropriate span pot.

After the analyzer is properly adjusted the linearity will be checked using a low and high range calibration gas. The maximum allowable limit for linearity is 2% of the analyzer range. All analyzers will be shown to be linear within these limits before proceeding.

The external calibration bias check will be performed by placing the CEM system in sampling mode and injecting a zero and span gas into the sample line at the probe exit. This check shows if there is any sampling system related bias, and also checks the integrity of the sample line.

**5.1.3.1 Calibration Gases**-DEECO will use EPA Protocol and/or ±2% NIST Traceable gases for calibration as required by the various reference methods employed in this test program. Calibration gases will be selected from previous experience with similar sources and/or from information obtained from the facility engineer prior to sampling. In some cases if the gases that are selected are out of the optimum range of operation then no significant impact of data quality is expected due to the linear nature of the analyzers that were used.

Audit gases, if available from a federal or a state agency, will be analyzed.

**5.1.3.2 Sampling Procedures**-At the completion of the pretest calibration routine, the CEM system will be ready for operation. No further adjustments of sample flow rates, analyzer zero or span adjustments, or other critical CEM operating parameters will be made until testing and post test calibration were complete.

Each sampling run will be one hour. At the completion for each test run, calibration gases will be used to check between test runs. A zero and the upscale calibration gas closest to the actual emission concentrations will be used for the pretest and post test calibrations.

#### **5.1.4 FLUE GAS MOISTURE CONTENT - EPA METHOD 4**

The flue gas moisture content will be determined in conjunction with the EPA Method 26A trains according to the sampling and analytical procedures outlined in EPA Method 4. (**NOTE:** In order to maintain isokinetic sampling, the sampling rate used may be required to temporarily exceed the EPA Method 4-specified maximum sampling rate of 0.75 CFM, based on observed stack gas pitot readings.) The impingers will be connected in series and will contain reagents as described below. The impingers will be contained in an ice bath in order to assure condensation of the moisture in the flue gas stream. Any moisture that is not condensed in the impingers is captured in the silica gel, therefore all moisture can be weighed and entered into moisture content calculations.

#### **5.1.5 HYDROGEN FLUORIDE AND DIATOMIC CHLORINE - EPA METHOD 26A**

Sampling and analytical procedures will be similar to those outlined in EPA Method 26A to determine primarily diatomic chlorine ( $\text{Cl}_2$ ) emissions and hydrogen fluoride (HF) emissions at main stack outlet sampling locations. Duplicate simultaneous trains (a.k.a “paired trains”) for each test run will be used to determine precision.

Sample is collected through a heated glass probe, followed by a heated quartz fiber filter, where stack gas HF and  $\text{Cl}_2$  are collected in a series of chilled impingers. The sampling train impingers will contain 50 ml of 0.1N sulfuric acid in the first impinger (optional should high moisture warrant a modified short stem), 100 ml of 0.1N sulfuric acid in the second and third, an empty fourth impinger, 100 ml of 0.1N NaOH in the fifth and sixth and 200 grams of silica gel in the last impinger. (**NOTE:** For plants with scrubbers, the optional cyclone may be used since the gas stream may be saturated with moisture.)

Sampling will be conducted isokinetically ( $\pm 10\%$ ) with readings of flue gas parameters recorded at traverse points selected according to EPA Method 1. Leak-checks on the Method 26A sampling train will be performed before and after each sampling run and optionally for any port change. In the event that any portion of the train needed to be disassembled and reassembled (i.e., due to filter or resin changes), leak-checks are performed. The sampling train leak-checks and leakage rate (where applicable) are documented on the field test data sheet for each respective run. All leak checks will be acceptable.

The glass button hook nozzle and probe liner will be constructed of borosilicate glass or quartz. The filter holder will be constructed of borosilicate glass with a Teflon frit filter support and a sealing gasket. A heated quartz fiber filter, for sources above  $210^\circ\text{C}$ , or PTFE-bonded glass fiber filter will be used. The probe and filter housing will be heated to above  $248^\circ\text{F}$  and not exceed an upper boundary of  $273^\circ\text{F}$ . Probe liners and filter holders will be cleaned thoroughly prior to testing.

The Method 26A trains will be operated isokinetically for a minimum of 60 minutes and collect a minimum of 1 dry, standard cubic meter (DSCM). Pretest preparations, preliminary determinations, and leak check procedures will be those outlined in EPA Method 5.

After completion of sampling the train will be leak checked and transferred to the sample recovery trailer. All leak checks will be acceptable. The impingers will be weighed to determine moisture gain in accordance with EPA Method 4.

Sample recovery will involve quantitative recovery of the sulfuric acid impinger contents and the NaOH impinger contents into separate tare-weighed, precleaned polyethylene sample containers.

The nozzle, probe, filter and filter housing will not be recovered.

The contents of sulfuric acid impingers, including the contents if any of the empty (2<sup>nd</sup> knockout or fourth) impinger will be quantitatively transferred to the tare-weighed, precleaned polyethylene sample container, followed by three rinses with deionized (DI) water of the impingers and all connecting glassware (including the connecting glassware to the first impinger) placed in the same H<sub>2</sub>SO<sub>4</sub> container. The container will be labeled and weighed to determine the final sample volume. The liquid level will be marked on the sample container.

The contents NaOH impingers will be quantitatively transferred to a second tare-weighed, precleaned polyethylene sample container, followed by three rinses with DI water of the impingers and all connecting glassware placed in the same NaOH container. The container will be labeled and weighed to determine the final sample volume. The liquid level will be marked on the sample container

Sample recovery from each train will include:

1. Container No. 1 - Contents 1<sup>st</sup> knockout, H<sub>2</sub>SO<sub>4</sub> impingers, and 2<sup>nd</sup> knockout and, and DI rinse of impingers and connecting glassware; and
2. Container No. 2 - Contents NaOH impingers, and DI rinse of impingers and connecting glassware.

Additional quality control consists of collecting and analyzing a field blank train for every three test runs. The blank train is to be assembled from a used train, leak checked and sit for a period equal to the sampling time (i.e, 1-hr). The blank train data will be used to determine the method detection limit for the test program target analytes (ie. The lowest number that could be detected), and compared to stack emissions.

Reagent blanks of 0.1 N H<sub>2</sub>SO<sub>4</sub>, 0.1N NaOH, and DI water will be collected and archived for later analysis should there be any issues with the field blank train samples

The H<sub>2</sub>SO<sub>4</sub> impinger solutions will be analyzed using ion chromatography techniques for fluoride ions (F<sup>-</sup>) (EPA SW-9057). Duplicate analyses will be performed on the samples and a reagent blank. Precision will be demonstrated by duplicate injection of each sample, the results of each individual analysis must be within 5% of their mean to be acceptable. If the precision criteria is not met, analysis of the sample is repeated until consecutive injections meet the criteria.

The NaOH impinger solutions will be treated with sodium thiosulfate to ensure complete conversion of hypochlorous acid (HClO) to chloride ions (Cl<sup>-</sup>). The resulting solution will be analyzed using ion chromatography techniques for chloride ions (EPA SW-9057). Duplicate analyses will be performed on the samples and a reagent blank. Precision will be demonstrated by duplicate injection of each sample, the results of each individual analysis must be within 5% of their mean to be acceptable. If the precision criteria is not met, analysis of the sample is repeated until consecutive injections meet the criteria.

All EPA Method 26A HF/Cl<sub>2</sub> samples will be analyzed by Element One of Wilmington NC. Refer to Section 1, Figure 1.1 for contact information.

The relative deviation (RD) will be calculated as described in EPA Method 30B between the Cl<sub>2</sub> concentrations measured with the paired trains.

### 5.1.6 HYDROGEN CYANIDE AND HYDROGEN FLUORIDE - EPA METHOD 320

EPA Method 320 will be performed to determine emissions of concentrations of HCN and HF. Three, 1-hour sampling runs will be conducted under representative process and control system operating conditions.

The gas sample will be extracted from the stack through a glass-lined probe and filter heated to 375° F. For external calibration checks and analyte spikes, the gases will be introduced in front of the heated filter. Any excess calibration gas will be diverted through the sample probes into the source. Outflow of gas from the heated filter enclosure was transported through a Teflon sample line heated to 375° F. For this source approximately 100' of sample line will be required. The heated sample line will be connected directly to the FTIR sample cell. Using heat-traced Teflon tubing the exit of the FTIR cell will be connected to a sample pump with a heated stainless steel pump head. The pump discharge will be directed to a proprietary chiller-type gas conditioner to remove moisture prior to delivery sample gas to the O<sub>2</sub>/CO<sub>2</sub> monitors.

The distribution of the gas sample to the monitors will be accomplished using a panel equipped with valves and rotometers. The gas sample was then divided and directed to the analyzers.

FTIR sample cell will be maintained at 191° C and connected to a MKS Instruments Multigas 2030 Fourier Transform Infrared Spectrometer and Detector.

The FTIR spectrometer will measure vapor phase organic or inorganic compounds which absorb energy in the mid-infrared spectral region, about 400 to 4000 cm<sup>-1</sup> (25 to 2.5 μm). Continuous measurement will be made by matching sample absorbance bands with bands in reference spectra, and comparing sample band intensities with reference band intensities.

The principle limitation to FTIR spectroscopy are the presence of interfering compounds that also absorb energy in the mid-infrared spectral region. In a cement kiln stack gas matrix, water vapor (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are the primary interferents that must be incorporated into the identification and quantitation method.

The FTIR software performs the computation for a single compound by subtracting all the other compounds (interferants and target) from the absorbance spectra and quantifies the single compound based on the remain absorbance. The FTIR software provides a Standard Error Calculation (SEC) value that is an indication of how well the identification and quantitation has been performed. A high SEC indicates that other interferants have not been accounted for in the analysis method, and a low SEC is indicative of greater confidence measurement.

The instrument is operated with a resolution of 0.5 cm<sup>-1</sup> with 4x zero filling. Beer-Norton Medium apodization is used with amplitude phase correction.

For this RTR test program, following specific QA/QC activities for EPA Method 320 will be performed and criterium met.

**5.1.6.1 Laboratory QA/QC Activities Before Field Test Program-** Before field testing occurs, the following QA/QC activities will be conducted;

- 1) Seven consecutive samples of dry nitrogen through the sampling system will be acquired and used to calculate the standard deviation for each of the test program target analytes multiplied

- by a factor of 3. These data will be considered representative of detection limits for this test program and are to be compared to the 0.5 ppm required DL;
- 2) From these seven dry nitrogen samples, the results for the Signal-to-Noise Ratio (SNR) @ 2500  $\text{cm}^{-1}$  should be  $>2500$ , at 64 scans and the results for single beam intensity @ 2500  $\text{cm}^{-1}$  should be  $>0.9$ ; and
  - 3) Upon receipt of HCN calibration gases a direct analysis will be performed to verify FTIR response agrees with tag value within 5%. Analysis results will be reported to PCA to assess need for modified reference spectra and/or change to direct analysis criterion:

**5.1.6.2 QA/QC Activities During Field Test Program-** During the field test program, following QA/QC activities will be performed and criterium met;

- 1) On each test day prior to any testing , an instrument background will be collected using dry nitrogen directed to the gas cell. The background will be collected with at least 128 scans;
- 2) The probe, filter, sample line and all sample system components in contact with effluent will be maintained at or above 375°F or 191°C (consistent with FTIR calibration temperature) to avoid any possible “cold spots;”
- 3) Heated sample lines will be  $\leq 100$  feet wherever possible, and not longer than 200 feet, without prior approval for unusual test circumstances;
- 4) A system zero with all sampling system components at operating temperature will be performed by injecting nitrogen at the sample probe and through sample filter and entire measurement system. After zero equilibration has been achieved, all measurement components will be quantified for at least 128 scans;
- 5) Ambient air will be sampled until equilibration of the measurement system has been achieved and all measurement components will be quantify for at least 128 scans;
- 6) The sample probe will be position at effluent measurement point and sampling will continue until equilibration of the measurement system has been achieved. At this point, the effluent concentrations will be quantified with two consecutive 64-scan samples as the initial native concentration for the dynamic spike;
- 7) Analyte spiking will be conducted for HCN before the first test run, and after each successive test run for a minimum of 4 spikes per test condition. (Additional spikes would be required before and after corrective action for the sampling or analysis system and/or before and after removing the sampling system from the stack.) These results will determine accuracy;
- 8) The spike gas injections will be maintained at 10% or less of total sample volume. The spike gas concentration and flow rate will be selected to approximately double the native effluent concentration, or the spike will be conducted to add 3-4 ppm to native concentration, whichever results in greater spiked concentration. Spike recovery results will be within  $\pm 20\%$  of the expected value or  $\pm 0.5$  ppm, whichever is least restrictive. (Specific HCN gases will be manufactured for this test program in the range of 50-100 ppm to provide spikes in the 5-10 ppm range, or lower. An  $\text{SF}_6$  or appropriate tracer will be used to calculate the exact spike gas dilution ratio of 10% or less;)
- 9) After the dynamic spike, nitrogen will be sent through the sampling system until all traces of spike gas are removed and lines are proven below DL for target analytes;
- 10) The nitrogen purge will be discontinued and the sampling system will be allowed to equilibrate with stack gas before starting a test run. The first two consecutive 64-scan samples of a sample run will be used for the final native concentration. Residual results for HCN and HF will be verified to be less than 0.2-0.3 ppm for data acceptance, or less than 5% of the measured value, whichever is least restrictive. Calculate the standard deviation for

- each of the test program target analytes for seven consecutive sample spectra from Run 1, multiplied by a factor of 3. These data will be compared to the pre-test system nitrogen standard deviation results and also included in the facility test report;
- 11) The SNR @ 2500  $\text{cm}^{-1}$ , at 64 scans, and the results for single beam intensity @ 2500  $\text{cm}^{-1}$  will be verified to meet the >2500 and >0.9 criterium; respectively. The analyte spiking for HCN and subsequent system nitrogen injection will be conducted after each test run. Continue sequence until at least three valid runs per test condition are completed.

## 6.0 QUALITY ASSURANCE/QUALITY CONTROL ACTIVITIES

### 6.1 QA/QC PROCEDURES

The QA/QC procedures for this RTR test program are summarized in Table 6-1.

**TABLE 6-1 QA/QC PROCEDURES AND REQUIREMENTS**

Target Analyte	Test Method	Detection Limit	QA/QC
HCN	EPA Method 320	0.5 ppm	<p>Increase scans if needed to achieve detection limits. Increasing to 400 from relative 64 (gives a 2.5 S/N advantage).</p> <p>HCN spiking before and after each run by adding 10% or less spike to approximately double the native effluent concentration, or conduct spike to add 3-4 ppm to native concentration, whichever results in greater spiked concentration.</p> <p>Spike recovery results shall be within <math>\pm 20\%</math> of the expected value or <math>\pm 0.5</math> ppm, whichever is least restrictive</p> <p>5% pre-to-post run calibration transfer standard (CTS) requirement</p>
HF		0.2-0.3 ppm	Rely on CTS (5%), HCN and tracer gas responses to validate HF FTIR data
Cl <sub>2</sub>	EPA Method 26A	~ 0.07 mg/m <sup>3</sup> (~0.2 ppm)	<p>Duplicate Simultaneous Trains;                      Collect minimum of 1 dscm for each sample train.                      Acceptance criteria for paired samples: 10% Relative Deviation or 0.2 ppm absolute difference, whichever is least restrictive.                      Insert dry impinger between acid and base impingers</p>
Effluent Flow Rate	EPA Methods 1-4	Not Applicable	<p>As per M26A isokinetic testing or separately by Methods 1-3. FTIR measurements for H<sub>2</sub>O.  <b>Wind Tunnel calibrated pitot tube having a Cp of 0.84 or less is required for all flow measurements.</b>                      Compare preliminary velocity traverse measurements and sample run flow rate measurements to installed certified flow rate monitor. Investigate and resolve differences greater than 10% of average flow rate.</p>
O <sub>2</sub>	EPA Method 3A	Not Applicable	<p>Analyte concentrations corrected @ 7% O<sub>2</sub>                      Span is 10%, 15%, or 20% (for co-mingled stacks only)                      Acceptance criteria are 0.2% O<sub>2</sub> difference for analyzer calibration error, and 0.3% O<sub>2</sub> for system bias checks, and zero and upscale drift checks.</p>

## 6.2 SAMPLE IDENTIFICATION AND CUSTODY

Sample custody procedures for this program are based on EPA recommended procedures. Since samples will be analyzed by one or more laboratories as well as in the field, the custody procedures emphasize careful documentation of sample collection and field analytical data and the use of chain of custody records for samples being transported. The procedures which will be used are discussed below.

The project manager will be responsible for ensuring that proper custody and documentation procedures are followed for the field sampling and field analytical efforts. He will be assisted in this effort by key sampling personnel involved in sampling recovery.

Samples will be collected, transported, and stored in clean containers which are constructed of materials inert to the analytical matrix such as glass jars. Only containers which allow air tight seals will be used. Amber glass jars will be employed when containers are needed to inhibit photochemical reactions.

All sampling data, including information regarding sampling times, locations, and any specific considerations associated with sample acquisition will be recorded on preformatted data sheets. All samples will be given unique, identifying alphanumeric sample codes which will serve to track samples from the field to the laboratory.

Samples will be stored for transport from the lab to the field to the lab in storage boxes constructed in a fashion which minimizes movement and thus prevents breakage of containers. For example, boxes used for transporting glass containers will have foam inserts with form-fitting cutouts. Sample transport boxes will be locked except when in use. Vans containing equipment and samples will be locked whenever they are left unattended.

A daily activity log will be maintained by the project supervisor. This will be an informal log used to record various types of information, such as minor problems which arise, sketches of sampling locations, names and phone numbers of plant contacts, daily activity summaries, etc.

This section provides information regarding the organization of the sampling and analytical program. The following details the key positions and their responsibilities. Once personnel have been assigned to these positions, their qualifications will be provided as an addendum.

The organization of the project team, including QA functions, is shown in the project organization chart (see Figure 1.1).

## **7.0 SAMPLE CUSTODY**

Sample custody procedures for this program are based on EPA recommended procedures. Since samples will be analyzed by one or more laboratories as well as in the field, the custody procedures emphasize careful documentation of sample collection and field analytical data and the use of chain of custody records for samples being transported. The procedures which will be used are discussed below.

### **7.1 FIELD SAMPLING OPERATIONS**

The project manager will be responsible for ensuring that proper custody and documentation procedures are followed for the field sampling and field analytical efforts. He will be assisted in this effort by key sampling personnel involved in sampling recovery.

Samples will be collected, transported, and stored in clean containers which are constructed of materials inert to the analytical matrix such as glass jars. Only containers which allow air tight seals will be used. Amber glass jars will be employed when containers are needed to inhibit photochemical reactions.

All sampling data, including information regarding sampling times, locations, and any specific considerations associated with sample acquisition will be recorded on preformatted data sheets. All samples will be given unique, identifying alphanumeric sample codes which will serve to track samples from the field to the laboratory.

Samples will be stored for transport from the lab to the field to the lab in storage boxes constructed in a fashion which minimizes movement and thus prevents breakage of containers. For example, boxes used for transporting glass containers will have foam inserts with form-fitting cutouts. Sample transport boxes will be locked except when in use. Vans containing equipment and samples will be locked whenever they are left unattended.

A daily activity log will be maintained by the project supervisor. This will be an informal log used to record various types of information, such as minor problems which arise, sketches of sampling locations, names and phone numbers of plant contacts, daily activity summaries, etc.

### **7.2 ANALYTICAL OPERATIONS**

Analytical operations will be performed on-site in the laboratory as well as in the remote laboratories. Samples analyzed by outside laboratories are transported with a Change of Custody form. This form will list sample identifications, analytical parameters, sample matrices, anticipated date of results, and other relevant information necessary to ensure the appropriate analyses are performed and to document the progress of the samples.

## 8.0 INTERNAL QUALITY CONTROL CHECKS

Specific quality control (QC) procedures will be followed to ensure the continuous production of useful and valid data throughout the course of this test program. The QC checks and procedures described in this section represent an integral part of the overall sampling and analytical scheme. Strict adherence to prescribed procedures is quite often the most applicable QC check. A discussion of both the sampling and analytical QC checks that will be utilized during this program is presented below.

### 8.1 EQUIPMENT INSPECTION AND MAINTENANCE

Each item of field test equipment will be assigned a unique, permanent identification number. An effective preventative maintenance program is necessary to ensure data quality. Each item of equipment returning from the field will be inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned, and recalibrated where necessary.

Each item of equipment transported to the field for this test program will be inspected again before being packed to detect equipment problems which may originate during periods of storage. This minimizes lost time on the job site due to equipment failure.

Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, replacement equipment for all critical sampling train components will be transported to the job site.

### 8.2 EQUIPMENT CALIBRATION

New items for which calibration is required will be calibrated before initial field use. Equipment whose calibration status may change with use or time will be inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it will be repaired and recalibrated or retired from service. All equipment will be periodically recalibrated in full, regardless of the outcome of these regular inspections.

Calibrations will be conducted in a manner, and at a frequency, which meets or exceeds U.S. EPA specifications. The calibration procedures outlined in the EPA Methods will be followed. When these methods are inapplicable, methods such as those prescribed by the American Society for Testing Materials (ASTM) will be used.

Data obtained during calibrations will be recorded on standardized forms, which will be checked for completeness and accuracy by the quality assurance manager. Data reduction and subsequent calculations will be performed using computer facilities. Calculations will be checked at least twice for accuracy. Copies of calibration forms will be included in the test or projects reports.

Emissions sampling equipment requiring calibration includes pitot tubes, pressure gauges, thermometers, dry gas meters and barometers. The following sections elaborate on the calibration procedures to be followed for these items of equipment.

**A: Pitot Tubes.** All Type S pitot tubes used, whether separate or attached to a sampling probe, will be constructed in-house or by a third-party vendor. Each new pitot will

be calibrated in accordance with Section 10.1 of EPA Method 2. Each Type-S pitot tube, calibrated according to these standards, will have an assigned coefficient. This coefficient should not change as long as the pitot tube is not damaged.

Each pitot tube will be inspected visually upon return from the field. If a cursory inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tube will be refurbished as needed and recalibrated.

- B: Differential Pressure Gauge.** All meter consoles used are equipped with 10-inch water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak checks. Manometers will be leak checked in the field prior to each test series, and again upon return from the field.
- C: Impinger Thermometer.** Prior to the start of testing, the thermometer used to monitor the temperature of the gas leaving the last impinger will be compared with a mercury-in-glass thermometer which meets ASTM E-1 No. 63F specifications. The impinger thermometer is adjusted if necessary until it agrees within 2°F of the reference thermometer. If the thermometer is not adjustable, it is labeled with a correction factor.
- D: Dry Gas Meter Thermometer.** The thermometer used to measure the temperature of the metered gas sample will be checked prior to each field trip against an ASTM mercury-in-glass thermometer. The dry gas meter thermometer is acceptable if the values agree within  $\pm 5.4^\circ\text{F}$ . Thermometers not meeting this requirements will be adjusted or labeled with a correction factor.
- E: Flue Gas Temperature Sensor.** All thermocouples employed for the measurement of flue gas temperature are calibrated upon receipt. Initial calibrations will be performed at three points (ice bath, boiling water, and hot oil). An ASTM mercury-in-glass thermometer will be used as a reference. The thermocouple is acceptable if the agreement is within 1.5 percent (absolute) at each of the three calibration points.

Before and after each field use, the reading from the flue gas thermocouple-potentiometer combination will be compared with an ASTM mercury-in-glass reference thermometer at ambient conditions. If the two agree within  $\pm 1.5$  percent (absolute), the thermocouple and potentiometer are considered to be in proper working order.

- F: Dry Gas Meter and Orifice.** Two procedures will be used to calibrate the dry gas meter and orifice simultaneously. The full calibration will be a complete laboratory procedure used to obtain the calibration factor of the dry gas meter. Full calibrations will be performed over a wide range of orifice settings. A simpler procedure, the post-test calibration, will be designed to check whether the calibration factor has changed.

A dry gas meter that is calibrated annually against a spirometer or a set of calibrated critical orifices will be used as a transfer standard. During the annual calibration, triplicate calibration runs will be performed at seven flow rates ranging from 0.25 to 1.40 cfm.

**G: Dry Gas Meter.** Each metering system receives a full calibration at the time of purchase and a post-test calibration after each field use. If the calibration factor,  $\gamma$ , deviates by less than five percent from the initial value, the test data are acceptable. If  $\gamma$  deviates by more than 5 percent, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the post-test calibration check indicates that  $\gamma$  changed by more than 5 percent. Standard practice is to adjust and recalibrate the dry gas meter anytime  $\gamma$  is found to be outside the range of 0.96 to 1.04. Post-test calibrations will be performed after each field test series per EPA Method 5, section 16.3 procedures.

**H: Orifice.** An orifice calibration factor will be calculated for each flow setting during a full calibration. If the range of values does not vary by more than 0.20 in  $H_2O$  over a range of 0.4 to 4.0 in  $H_2O$ , the arithmetic average of the values obtained during the calibration is used.

**I: Barometer.** Each field barometer will be adjusted before each test series to agree within  $\pm 0.1$  inches of a reference aneroid barometer. The reference barometer will be checked against the station pressure value (corrected for elevation difference) reported by the National Weather Service.

### 8.3 SAMPLING QUALITY CONTROL PROCEDURES

The following pretest QC checks will be conducted:

- All sampling equipment will be thoroughly checked to ensure clean and operable components.
- Equipment will be inspected for possible damage from shipment.
- The oil manometer or Magnehelic gauge used to measure pressure across the Type S pitot tube will be leveled and zeroed.
- The number and location of the sampling traverse points will be checked before taking measurements.
- The temperature measurement system will be visually checked for damage and operability by measuring the ambient temperature prior to each traverse.

In addition to the general QC procedures listed above, QC procedures specific to each sampling method will also be incorporated into the sampling scheme. These methods and specific procedures are discussed below.

**A:** Sampling Train QC checks. The following QC procedures will be emphasized:

***Prior to Start of Tests***

- Keep all cleaned glassware and sample train components sealed until train assembly.
- Assemble the sampling trains in an environment free from uncontrolled dust.
- Visually inspect each sampling train for proper assembly.
- Perform pretest calculations to determine the proper sampling nozzle size.

***Prior to Each Test Run***

- Visually inspect the sampling nozzle.
- Visually inspect the Type S pitot tube.
- Leak check each leg of the Type S pitot tube.
- Leak check the entire sampling train.

***During Each Test Run***

- Readings of temperature and differential pressure will be taken at each transverse point.
- All sampling data and calculations will be recorded on preformatted data sheets.
- All calibration data forms will be reviewed for completeness and accuracy.
- Any unusual occurrences will be noted during each run on the appropriate data form.
- The project supervisor will review sampling data sheets daily during testing.
- Properly maintain the roll and pitch axis of the Type S pitot tube and the sampling nozzle.
- Leak check the train before and after any move from one sampling port to another during a run (at DEECO's option) or if a filter change takes place.
- Conduct additional leak checks if the sampling time exceeds 4 hours.
- Maintain the probe, filter, and impingers at the proper temperatures.
- Maintain ice in the ice bath at all times.
- Make proper readings of the dry gas meter, delta P and delta H, temperature, and pump vacuum during sampling at each traverse point.
- Maintain isokinetic sampling within  $\pm 10\%$  of 100%.

***After Each Test Run***

- Visually inspect the sampling nozzle.
- Visually inspect the Type S pitot tube.
- Leak check each leg of the Type S pitot tube.
- Leak check the entire sampling train.

## **B: QC for Volumetric air flow rate determinations**

**Flue Gas Velocity.** Data required to determine the flue gas velocity will be collected using the methodology specified in EPA Method 2. Quality control procedures are as follows.

- Visually inspect the Type S pitot tube before and after sampling.
- Leak check both legs of the pitot tube before and after sampling.
- Check the number and location of the sampling traverse points before taking measurements.

**Flue Gas Molecular Weight.** In the event that that integrated bag samples are to be used for determination of flue gas molecular weight, EPA Method 3 will be the sampling technique specified. Quality control will focus on the following procedures:

- The sampling train will be leak checked before and after each run.
- A constant sampling rate will be used in withdrawing a sample.
- The sampling train will be purged prior to sample collection.
- The sampling port will be properly sealed to prevent air in-leakage.

**Moisture Content.** The moisture content of the gas stream will be determined using the technique specified in EPA Method 4. The following QC checks will be performed:

- The sampling train will be leak checked before and after each run.
- Ice will be maintained in the ice bath throughout each run to insure an exit temperature (after the silica gel impinger) of  $\leq 67^{\circ}\text{F}$ .

## **8.4 ANALYTICAL QUALITY CONTROL PROCEDURES**

All analyses for this program will be performed using accepted laboratory procedures in accordance with the specified analytical protocols. Adherence to prescribed QC procedures will ensure data of consistent and measurable quality. Analytical QC will focus upon the use of control standards to provide a measure of analytical precision and accuracy. Also, specific acceptance criteria are defined for various analytical operations including calibrations, control standard analyses, drift checks, blanks, etc. The following general QC procedures will be incorporated into the analytical effort:

- The on-site project manager will review all analytical data and QC data on a daily basis for completeness and acceptability.
- Analytical QC data will be tabulated using the appropriate charts and forms on a daily basis
- Copies of the QC data tabulation will be submitted to the quality assurance manager following the completion of the test program.
- All hard copy raw data (i.e., chromatograms, computer printouts, etc.) will be maintained in organized files.

Specific analytical QC procedures for the Orsat analyzer (if used) are listed below.

- The analyzer will be leveled and the fluid levels zeroed prior to use.
- The analyzer will be leak checked prior to use.
- The analyzer will be thoroughly purged with sample prior to use.
- The analyzer will be checked by analyzing an ambient air sample.

EPA Method 26A Sample Analysis QC Checks are listed below.

- Calibration curve consisting of 4 calibration levels that bracket the expected sample range. Dilute samples as necessary to reach the calibration range;
- Duplicate analysis of calibration standards, before and after sample analysis, with duplicate injections being within 5% of their mean;
- Duplicate analysis of reagent blanks, quality control samples and field samples with duplicate injections being within 5% of their mean;
- Matrix spike samples may be prepared and analyzed. Matrix spike recoveries should be 90-110%
- A field blank will be carried through the procedure and analyzed with the field samples.
- An audit sample will be analyzed for if available from two or more independent, Approved Audit Sample Providers no less than 60 days prior to the test effort.

## **9.0 REPORTING AND DATA REDUCTION REQUIREMENTS**

### **9.1 DATA REPORTING**

The reporting units for HCN, HF, and Cl<sub>2</sub> will be in parts-per-million by volume, wet basis (ppm<sub>v,d</sub>), parts-per-million by volume, dry corrected to 7% oxygen (ppm<sub>v,d</sub>@7%O<sub>2</sub>), pounds-per-hour (lbs/hr), and pounds-per-ton of clinker (lbs/ton). Additional supporting data for CO<sub>2</sub>, O<sub>2</sub>, and H<sub>2</sub>O concentrations and volumetric flow rates (actual cubic feet-per-minute, wet, standard cubic feet-per-minute, and dry, standard cubic feet-per-minute) will be reported. The clinker production, in short tons-per-hour (TPH) will be reported.

Any data that is not acceptable because of technical difficulties will be indicated, and an explanation of the technical problem will be given. All related QC and calibration data will be in the final report.

### **9.2 REPORT CONTENTS**

Copies of the test report will be submitted after the test series has been completed. Results reported will include, but not be limited to emission rates and concentrations of gaseous pollutants, and process sample determinations, any liquid stream constituents determinations, and any other type of data requested. This report will also include a list of all personnel present during testing, summary results, descriptions of test procedures used, a description of the source and its operation during testing, test locations drawings, example calculations, raw field data, and equipment calibrations.

### **9.3 DATA REDUCTION**

Care will be exercised to ensure hand recorded data is written accurately and legibly. Additionally, the use of prepared data recording forms, conveniently formatted, is an important aid to verify that all necessary data items are recorded. The collected field and laboratory data will be reviewed by the analyst and the Project Manager.

The Project Manager will reduce and validate all of the sampling and analytical data that is collected. The sampling data will include flow measurements, calibrations, etc. Each laboratory will reduce all analytical results prior to their submission to the Project Manager. The analytical data will be used to determine concentrations and emission rates of the compounds of interest.

Data reduction follows guidelines published in EPA Reference Methods, where applicable, and by guideline documents where EPA Reference Methods are not available. Validated computer programs will be used to calculate all reported values.

### **9.4 DATA VALIDATION**

A second technical review of the data will be performed and documented by a qualified scientist other than the one who performed the actual analyses. The second reviewer will include evidence (e.g., check marks, recalculations, etc.) that show which data points were checked. Finally, the second reviewer will sign and date the cover page of the data packet or the record that was reviewed.

In-situ measurements will be validated by demonstrated acceptable post-test leak checks and calibration verifications according to the referenced method used.

Analysis data may be validated according to defined criteria by a secondary reviewer or by the analyst. At a minimum and if applicable, analysis data will be validated according to the following criteria (additional method-specific criteria or project requirements may apply):

- Sampling records complete and traceable
- All appropriate QC samples included with the analytical batch and reported with the sample results
- Routine tuning, calibration and inspection of analytical instrumentation documented and performed prior to analyses
- Initial and continuing calibration criteria met
- Method/reagent blanks confirm no background contamination
- Surrogate recoveries within criteria
- Qualitative sample results (e.g., retention times, mass spectra, isotopic ratios) consistent with standard data
- Sample data within the calibrated range of the instrument
- Chromatograms or other raw data consistent with computer-generated quantitation reports
- Accuracy of intermediate data manipulations, transcribed numbers and/or final reported results verified
- Reference standards, instrumentation, sample identification, analysts, methodology, and sequence of processing clearly identified and traceable in the project records
- Lost data or corrective actions documented (e.g., loss of sample, reanalysis, redilutions, additional cleanup steps, alternative calculations etc.)
- Data that does not meet the validation requirements flagged accordingly
- Data reported in the correct units (e.g., "ppm" should not be used without specifying volume or mass units; "ug/g" are preferred units for data reporting)

## **10.0 PLANT ENTRY AND SAFETY**

### **10.1 SAFETY RESPONSIBILITIES**

The Project Manager is responsible for ensuring compliance with plant entry, health, and safety requirements. The Facility Contact (refer to Section 1.2) as the authority to impose or waive facility restrictions. The Project Manager has the authority to negotiate with facility person any deviations from the facility restrictions.

### **10.2 SAFETY PROGRAM**

DEECO has a comprehensive health and safety program that satisfies Federal OSHA and MSHA requirements. The basic elements include: (1) written policies and procedures, (2) routine training of employees and supervisors, (3) medical monitoring, (4) use of personal protection equipment, (5) hazard communication, (6) pre-mobilization meetings with Holcim personnel and DEECO test team personnel, and (7) routine surveillance of the on-going test work.

### **10.3 SAFETY REQUIREMENTS**

All test personnel will adhere to the following standard safety and precautionary measures as follows:

- 1) Confine activities to test area only;
- 2) Wear hard hats at all times on-site, except inside sample recovery trailers and mobile CEM laboratory;
- 3) Wear protective shoes or boots in test area;
- 4) Wear protective glasses or goggles at the outlet test sites, and other areas as designated;
- 5) Have readily available first aid equipment and fire extinguishers.

Before or on the first day on-site, the Project Manager will fill out the Emergency Response Procedure form and provide copies to be posted at each test site.

## **Appendix A**

### **Sampling and Analytical Methods**

**The sampling and analytical methods for this sampling effort can be found at the following website:**

*Promulgated EPA test methods, 40 CFR 60 (Methods 1-4, 26A, and 320)*

<https://www.epa.gov/emc/emc-promulgated-test-methods>