EMISSIONS TESTING IN THE LIME INDUSTRY

A GENERIC TEST PLAN FOR
MEASURING HCl USING ASTM D6735-01

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(supplementary information is highlighted)

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1.0 INTRODUCTION and SCOPE

The purpose of this document is to provide a “generic” test plan for conducting hydrochloric acid gas testing from the outlet of air pollution control devices installed on lime kiln systems. This document details the use of ASTM D6735-01 “Determination of Gaseous Chlorides and Fluorides from Mineral Calciners” for measuring in-stack concentrations of HCl (g).

It is expected that the Lime Industry National Emission Standard for Hazardous Air Pollutants (NESHAP) will include an exemption for “area sources”. Area sources are those that emit less than 10 tons/yr of any single hazardous air pollutant (HAP) or 25 tons/yr of combined HAPs. The HCl testing will be required in order to demonstrate that a lime plant emits less than 10 tpy of HCl.

In the ASTM Method, kiln effluent is drawn through a particulate matter filter held at 350ºF and collected in dilute sulfuric acid solutions. All chlorides passing through the filter are quantified from the acidic impinger solutions using ion chromatography. The chloride “catch” is all attributed to HCl (g). Gaseous HCl concentrations are calculated using the total impinger catch weight divided by the total sample volume and expressed as mg/DSCM or in PPM (v) dry.

Emission rates are calculated from the concentration results using stack gas volumetric flow rates determined by performing EPA Methods 1-4.

1.1 Test Program Organization

Facility Name and Address:
Facility Contact

Testing Firm Name and Address
Test Firm Contact

Analytical Laboratory Name and Address
Lab Contact

2.0 DATA QUALITY OBJECTIVES

The data quality objectives are specific to each source. Method precision is determined using a paired train configuration. If the difference between the two paired results is greater than 2 ppm or 20% of the mean, the results should be considered suspect.

Accuracy is determined using the spiking procedure detailed in Section 11.2.7 of the method. This procedure consists of quantitatively adding HCl into one of the sample trains upstream of the particulate matter filter. Calculating the recovery of the spike gives an estimate of the method accuracy. Acceptable recoveries are those within 30% of the expected value at a 5 ppm equivalent in-stack spike value.

Before the testing starts, a conditioning run must be performed, with the impinger solutions discarded at the end of the run. Then, four test runs should be performed. Three paired runs (which generates 6 samples) are used to determine whether the method's precision criteria (described above) are satisfied. The fourth run serves as the spike run, and is used to determine if the above-described accuracy criterion is met. Only one train of the fourth pair is spiked at the completion of the fourth run.

The HCl spike into the impinger train should approximate an in-stack equivalent concentration of 5 ppm (v) dry or less. The spike volume should not exceed 30 liters.

Example:

Spike cyl – 15 ppm or 22.8 mg/DSCM
Spike Volume - 30 L or 0.03 DSCM
22.6.03= 0.678 mg spike added*
Stack sample volume – 120 L or 0.12DSCM
0.678 mg/0.12 DSCM = 5.65 mg/DSCM or 3.7 ppm (v) dry

In addition to the paired trains and spiking, the method places strict criteria on the heated train components. The temperature of the probe sections and heated filter box must be maintained within strict tolerances and the temperature of these zones must be recorded on field data sheets every 5 minutes.

For the Methods 1-4, quality assurance consists of using calibrated pitot tubes, dry gas meters, barometers, and analytical balances. Cyclonic flow checks should also be performed and these results included in the final report.

For the analytical laboratory, the QA measures detailed in the ASTM Method should be used.

3.0 SOURCE DESCRIPTION

A general process description should be provided.

3.1 Fuels and Feed

The source should be operated at the maximum routine production rate 90^{+}% of capacity. The source should attempt to use the fuels combination that they use the majority of the time or they should attempt to determine which fuel combination has the greatest potential to emit chlorides.

A description of the fuels and feed source should be provided.

3.2 Process Samples

A description of the process samples that will be acquired and the time interval of collection should be included. It is recommended that samples of feed and fuel should be acquired. Samples of LKD and lime product may also be useful. Samples should be collected starting 1-2 hours before the first run and should continue until 1-2 hours after the last run is completed. Samples should be taken every 60 to 90 minutes. Note: for preheater kilns, stone samples should be taken starting 6-7 hours before the first sampling run to account for the additional residence time in the preheater.

These samples should be archived until the test results are known. Analysis of these process samples for chlorides and sulfur should be performed if the test results are not consistent with anticipated results. Recommended analytical test methods include:

Analytical Methods for Chlorides and Sulfur in LIMESTONE

Constituent	*Method*	*ASTM Reference*	*Comments*
chlorides			
	X-Ray Flouresence (XRF)	C 1271	Typical detection limit = 1-10 ppm
	Inductively Coupled Plasma (ICP)	C 1301	
	Graphite Furnace Atomic Absorption (AA)	C 1301	
sulfur			
	X-Ray Flouresence (XRF)	C 1271	
	Inductively Coupled Plasma (ICP)	C 1301	
	Combustion/Infrared	C 25	

Analytical Methods for Chlorides and Sulfur in COAL

Constituent	*Method*	*ASTM Reference*	*Comments*
chlorides			
	Oxygen Bomb Combustion/ Titration	D 2361	Typical detection limit = 50 ppm; ASTM considering improvements to the method
	Oxygen Bomb Combustion/ Ion Selective Electrode	D 4208	
	Oxidative Hydrolysis Microcoulometry	D 6721	
sulfur			
	High Temperature Tube Furnace Combustion	D 4239	

3.3 Sampling Location Description

A description of the sampling location including height above grade, port configuration, and approximate stack gas parameters such as flow rate, and % moisture should be provided.

4.0 TEST PROGRAM

Place a test schedule in this section

4.1 Safety Considerations

Place safety considerations in this section.

5.0 REPORTING

The report should include the following;

- Introduction and Approach
- Concentration results in ppm (v) dry for both trains
- A calculation of method precision from the three paired runs
- Results from the spikes in ppm (v) dry
- A calculation of the spike recovery
- Stack gas volumetric flowrate and supporting data
- Results in lb/hr and ton/yr and lb/ton of lime
- Process data
- Deviations from testing
- Appendices
    - Copy of Test Plan
    - Calibration results of pitot tubes, dry gas meters etc.
    - Raw field data sheets (cyclonic flow checks, ASTM Method data sheets (use those similar to M26 but for paired trains), and M1-4)
    - If instrumental test methods are used for O_2 and CO_2 then the results from these instrumental test methods should be included.
    - Manufacturers certificate of analysis for HCl spike gas (and O_2 and CO_2 if used)
    - Analytical lab data, calibrations, and results
    - Process Data including fuel feed rate, stone feed rate and/or production rate