

VALIDATION TESTING OF THE DRAFT ASTM IMPINGER METHOD FOR MEASURING GASEOUS CHLORIDE EMISSIONS FROM MINERAL CALCINERS

Test Report

Prepared for: National Lime Association 200 North Glebe Road Arlington, VA

VALIDATION TESTING OF THE DRAFT ASTM IMPINGER METHOD FOR MEASURING GASEOUS CHLORIDE EMISSIONS FROM MINERAL CALCINERS

Mississippi Lime Company Draft Test Report

November 2000

Prepared for:

National Lime Association 200 North Glebe Road Arlington, VA

Prepared by: L. L. Kinner Emission Monitoring Incorporated 8901 Glenwood Avenue Raleigh, NC 27617

Executive Summary

Emission Monitoring Incorporated was hired by the National Lime Association (NLA) to conduct validation testing of a draft method for measuring gaseous chlorides in lime kiln emissions. Dr. L. Kinner of Emission Monitoring in conjunction with Mr. G. Cobb and Mr. F. Shaw of AirSource Technologies Inc. performed the field-testing.

The draft method (attached in Volume II Appendix A) is entitled "Measurement of Gaseous Chlorides and Fluorides from Mineral Calcining Exhaust Sources – Impinger Method -Z8662Z " and its development is under the auspices of the American Society for Testing and Materials (ASTM). The gaseous chlorides collected by this method are all attributed to hydrogen chloride gas.

A test plan and quality assurance project plan were prepared and reviewed by members of the NLA. A copy of the plan is included in Volume II Appendix B.

Method validation testing was conducted at two facilities. One facility had a scrubber controlled kiln system and the other had a baghouse controlled kiln system. This report describes the test program performed at Mississippi Lime Company (scrubber) - Ste. Genevieve, Missouri, from September 6-9, 2000. Testing at this facility provided a low concentration sample matrix (<10 ppm) from which to conduct the validation.

Data acquired by the Draft ASTM Method was evaluated using the statistical analysis procedures contained in EPA Method 301 (40 CFR Part 63, Appendix A). In this capacity two pairs (quad trains) of lime kiln effluent samples were acquired simultaneously using the Draft ASTM method. Two of the four samples were spiked with a small amount of gaseous hydrogen chloride at the end of each run. Six sets of the "quad" samples were acquired to provide 12 spiked and 12 unspiked pairs of data sets. The combination of 12 spiked samples and 12 unspiked samples provided a means to determine the accuracy and precision of the draft method using the EPA Method 301 statistics.

Based on the Method 301 statistical analyses, the method has a relative standard deviation of 14% for the unspiked samples and 17% for the spiked samples. This standard deviation also factors in the natural variation in the underlying concentration of the effluent. The bias of the method using Method 301 calculations is -0.12, with a t-value of 0.60 indicating a statistically insignificant bias when compared to the critical t value of 2.201. The concentration level evaluated during this validation test ranged from about 2.5 ppm to 8 ppm.

The percent recovery for each spiked sample was also calculated. The results ranged from a low of 83% recovery to a high of 113% recovery with an average of 98% for the 12 runs. These results are well within the generally accepted range of 70%-130% that is used when performing spiking in instrumental test methods such as Fourier transform infrared (FTIR) spectrometry.

The results from the Mississippi Lime testing indicate that the method is reliable at concentrations below 10 ppm for measuring gaseous chlorides. According to EPA method 301, this method passes the precision criteria and does not have a statistically significant bias at concentration levels below 10 ppm.

1.0 BACKGROUND AND INTRODUCTION

Existing HCl measurement methods such as USEPA Method 26/26A (40 CFR Part 60 Appendix A) have been reported to have both high and low biases in many measurement applications. In 1994, the US EPA recognized the inaccuracies of Method 26/26A, and a directive was issued to solve the problems associated with the method, or to find a better measurement method for measuring HCl at low concentration levels¹.

Since that time, Fourier transform infrared (FTIR) methods such as Methods 320 and 321 (Part 63) have been developed for measuring HCl. Although, FTIR instrumentation is highly qualified to measure HCl in the gaseous form, the technique is constrained at low HCl concentration levels. Low concentration level measurements of HCl are difficult by FTIR because; 1) reference spectra and calibration gases can be inaccurate, 2) measurement systems can have highly variable detection limits, 3) many FTIR instruments can have HCl non-linearity problems that may go uncorrected by the operator, and 4) sampling systems often require in excess of 100 feet of heated transfer line to direct the effluent to the instrumentation which can scrub HCl.

The fact that the FTIR is problematic for measuring HCl at low concentration levels was demonstrated numerous times during FTIR testing at lime producing facilities. During these tests, the analyte spike quality assurance procedures did not meet the criterion defined by the Method 320 or 321 at concentration levels below 10 ppm as demonstrated by low analyte spike recoveries ².

This issue is significant because accurate low concentration level measurements are needed by many industrial facilities. A laboratory study was conducted during the summer of 1999 that investigated many of these phenomena. A report was prepared that details these issues and presents the basis for developing a new method for measuring HCl (g) and conducting this field validation ³.

The purpose of the test program was to evaluate the method entitled "Measurement of Gaseous Chlorides and Fluorides from Mineral Calcining Exhaust Sources – Impinger Method." This method is currently in draft form and its development is under the auspices of the American Society for Testing and Materials (ASTM). The evaluation approach used EPA Method 301 to perform a method "validation". The gaseous chlorides collected by this method are attributed to HCl (g).

Two lime kiln systems provided the sample matrix for this test program. This test report describes the testing conducted at Mississippi Lime Company - Ste. Genevieve Missouri facility. The calciner system consisted of a rotary kiln (with an extended section referred to as a pre-heater) in combination with a wet venturi scrubber for the purpose of dust collection. The effluent matrix contained concentration levels of HCl that ranged from about 2.5 ppm to 8 ppm.

¹ Memorandum dated March 14, 1994 from James Crowder Industrial Studies Branch Chief United States EPA to Gilbert Wood Emission Measurement Branch Chief requesting a new or revised test method to measure HCl from Portland Cement and Secondary Aluminum Facilities. Docket to the Portland Cement NESHAP – Item number II-B-45.

² Reference #1 – Testing at Austin White Lime Company and Test Report Prepared for US EPA "Lime Kiln Source Characterization – Draft Final Report – Huron Lime" Prepared by Eastern Research Group, EPA Contract 68-D70007

³ Kinner, Peeler, and Willis "Development of an Improved Impinger-Based Method for Measuring Gaseous Chloride Emissions from Mineral Calciners" Air and Waste Management Association Annual Meeting – Salt Lake City, Utah, June 19, 2000.

2.0 DESCRPTION OF METHOD VALIDATION AND APPARATUS

This test program was performed at Mississippi Lime Company's scrubber equipped kiln designated at MRK8. This rotary kiln system is fired with a blend of coal and coke, and uses a venturi scrubber to control air emissions. The kiln was operated under conditions considered to be representative and in accordance with the facility operating permit.

Testing was conducted at the stack location. Six sets of quadruplet sampling trains were operated in succession to total 24 samples for the method validation testing. Figure 2-1 presents a schematic of the paired sample train and spike apparatus.

Before starting Run 1, a conditioning run was performed to passivate any active sites within the probe and filter box. The impinger contents from this run were discarded.

Two pairs of pre-test runs were also performed. The pre-test sampling experiment was designed to evaluate whether the results provided by this method are different when the sampling components are held at 250?F versus 350?F. The results provided by this method at these two different temperatures were virtually identical, and similar to the results provided by the validation testing. This was not a surprise because the temperature of the effluent was about 180?F, and sampling system temperatures that prevented condensation within the sampling components are all that is necessary to transport the gaseous chlorides to the impinger solutions.

The draft ASTM method prescribes collecting about 120 liters of gas into four midget impingers containing a solution of dilute sulfuric acid (0.1 N). The gas sample is collected for one-hour at a rate of 2 liters per minute. Sample gas is withdrawn from a single point in the stack or duct. Analysis of the impinger train sample is performed using ion chromatography, a method that analyzes total chloride ions in the solution. Concentration data only were collected during this validation test program.

After the end of each one- hour validation sample run, one sample train of the pair was spiked with a concentration of HCl calibration gas, equivalent to about 2 ppm. For this validation test, the amount of HCl spiked into the impinger trains was about one half of the concentration found in the unspiked samples.

The spiking procedure is detailed in section 11.2.6 of the draft method and consists of adding (about 36 liters for this application) HCl calibration gas into the sampling train upstream of the particulate matter filter. The spike gas flow rate was adjusted to be greater than the total sample flow so that the excess spike gas was vented out the probe. This enabled the meter box and pump to function identically to conditions encountered during sample collection.

Conducting a gaseous spike into an impinger train is very ambitious. Spiking with a gaseous standard adds much in the evaluation of this method because the spike had to pass through the filter, and thus it evaluated the scrubbing potential of the lime dust on the particulate filter cake.

Figures 2-2 and 2-3 present digital images of the method under actual application.





Figure 2-2 Sampling Apparatus in Testing Application



Figure 2-3 Sampling Apparatus with Spike Assembly Attached

3.0 DATA QUALITY OBJECTIVES

The test program data quality objectives and achieved data quality are summarized in the following tables.

Table 3-1 Data	Quality	Objectives	Method	301
----------------	---------	------------	--------	-----

Target Analyte	Bias	Bias Precision	
			Target
HCl (g)	t value <2.201 for 12 pairs	? 50% Relative Standard Deviation	100%

These data quality objectives were chosen to reflect the acceptable bias and precision detailed in Section 6.3 of EPA Method 301 4 .

The following table shows the actual data quality achieved during this field test.

Table 3-2 Achieved Data Quality Method 301

Target Analyte	Bias	Precision	Data Completeness
HCl (g)	Bias = -0.12 mg	14% RSD for unspiked samples	100%
		17% RSD for spiked samples	
	t value = 0.60 for 12 pairs		
	t<2.201		

These results are presented in Tables 4-1.

An additional data quality objective for calculating the spike recovery from each impinger train pair was set at 70% to 130% of the expected value. Table 3-3 summarizes the findings from this analysis.

Table 3-3 Spike Recovery Data Quality Objectives and Data Qu
--

Target Analyte	Accuracy Expected	Accuracy Achieved
HCl (g)	Spike Recovery 70% to 130% of	83% - 113%
	Expected Value	

These results are presented in Table 4-2.

⁴ EPA Method 301 CFR Part 63, Appendix A, Section 6.3. December 1992.

4.0 RESULTS

The target data completeness of 100% was achieved during this test program.

4.1 Method 301 - Precision

Based on EPA Method 301 statistical analyses of the data, the method has a relative standard deviation of 14% for the unspiked samples and 17% for the spiked samples. This standard deviation also factors in the natural variation in the underlying concentration of the effluent.

The Mississippi Lime kiln MRK8 effluent had appreciably variable HCl concentration levels during this testing, however, using the quad train sampling approach minimized the apparent imprecision because the pairs of samples were acquired simultaneously.

4.2 Method 301 - Bias

The bias of the method using the Method 301 calculations is -0.12, with a t-value of 0.60 indicating a statistically insignificant bias when compared to the critical t value of 2.201.

According to EPA Method 301, a method provides valid data if the bias is statistically insignificant and the precision is less than 50% RSD.

Table 4-1 presents a spreadsheet that contains the EPA Method 301 statistical analyses of the data.

4.3 Spike Recovery Results

Although not a requirement of Method 301, the percent recovery for each spiked sample was calculated to present the results in terms of expected versus observed values. This was done by determining the value of the amount of spike collected relative to the amount expected.

The percent recovery of the 12 spiked samples ranged from a low of 83% to a high of 113% with an average of 98%. This is well within the range of 70-130% for valid analyte spike recovery. The 30% criterion is generally accepted as the basis for "valid" data produced by instrumental test methods using Fourier transform infrared spectrometry ⁵.

Table 4-2 presents all of the pertinent sample collection data and the corresponding percent recoveries for each of the spiked sample trains.

The data file prefixes were named as follows;

CGA10A and 10B – Calibration gas audits of the cylinder used for spiking – duplicate analysis MPRE – Mississippi Lime pre runs MVAL – Mississippi Lime validation runs

The analytical laboratory report prepared by Resolution Analytics Inc. is attached in Volume II Appendix C, and raw data sheets, calculations, and gas meter calibrations are contained in Volume II Appendix D of this report.

⁵ EPA Method 321 40 CFR Part 63, Appendix A – Section 9.3.2

Table 4-1 Mississ	ippi Lime - Met	hod 301 Statistica	I Analysis of Quad 1	Train Samples					
		ma difforonco		Difference		Difforence			
Sample Name	mg catch	between pairs	ma spike added	Unspiked Pairs	Squared	Spiked Pairs	Squared		
MVAL11A	0.87	betheen pane	ing opino addou	enopineu i une	oquaiou	opinou i uno	oquaioa		
MVAL11B	1 16	0.29	0.41	0.17	0.03	0.38	0.14		
MVAL 12A	1.04	0.20	0	0	0.00	0.00	0		
MVAL12R	1 54	0.50	0.42						
	1.04	0.00	0.12						
MVAL21A	0.47								
MVAL21B	0.82	0.35	0.42	0.07	0.00	0.12	0.01		
MVAL22A	0.54	0.00	0.12	0.07	0.00	0.12	0.01		
MVAL22B	0.94	0.40	0.41						
	0.04	0.40	0.41						
MVAL31A	0.60								
MVAL31B	0.97	0.37	0.42	0.01	0.00	0.13	0.02		
MVAL32A	0.61								
MVAL32B	1.10	0.49	0.42						
-									
MVAL41A	0.66								
MVAL41B	0.97	0.31	0.42	0.09	0.01	0.23	0.05		
MVAL42A	0.75								
MVAL42B	1.20	0.45	0.41						
MVAL51A	0.58								
MVAL51B	1.11	0.53	0.41	0.26	0.07	0.14	0.02		
MVAL52A	0.84								
MVAL52B	1.25	0.41	0.42						
MVAL61A	0.81								
MVAL61B	0.99	0.18	0.41	0.11	0.01	0.33	0.11		
MVAL62A	0.92								
MVAL62B	1.32	0.40	0.41						
Mean Unspiked	0.72		Sum	0.71	0.12	1.33	0.36		
Mean Spiked	1.02								
Mean mg Added	0.42		Sdu	0.11		Standard Deviation	on of the un	spiked "A" sample	es
			Sds	0.18		Standard Deviation	on of the spi	ked "B" samples	
			%RSDu	14.52		% Relative Stand	lard Deviatio	on of unspiked sa	mples
			%RSDs	17.62		% Relative Stand	lard Deviatio	on of spiked samp	oles
			Bias	-0.12		Mean of Spiked -	Mean unspi	iked - Mean mg a	dded
			SDM	0.21	0.21 Square root of the sum of the squares of			squares of spike	d and unspiked
			t	0.58		Absolute value of	Bias/SDM		
			t < 2.201	bias not statistically	significant				

Table 4-2 N	lississippi L	ime Calcu	lations for Spike	e Recoveries													
	1																
Calibration	Gas Audit S	ample Du	plicate Analysis								25.40)					
Run #	Mg HCI	Vdry (L)	Meter Temp C	Meter Temp K	Std Temp	Pressure " Hq	Pressure torr	Std Pressure	DSCM Collected	mg/dscm	ppm	Avg					
CGA10A	0.37	31.84	4 28	3 30	1 293	30	764	760	0.031	1 11.75	7.74						
CGA10B	0.38	30.57	7 30	303	3 293	30	764	760	0.030	0 12.93	8.52	8.13					
Pre-Runs	Mg HCI	Vdry (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Hq	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	mg/dscm	ppm				
MPRE11A	0.74	130.09	9 no spike	3	4 307	293	29.98	761	760	0.12	2 NA	5.97	3.94	250F			
MPRE11B	0.76	127.13	3 no spike	4	1 314	293	29.98	761	760	0.12	2 NA	6.43	4.24	250F			
MPRE12A	0.60	114.70) no spike	3	5 308	293	29.98	761	760	0.11	NA	5.46	3.60	350F			
MPRE12B	0.83	116.21	1 no spike	3	5 308	293	29.98	761	760	0.11	NA	7.49	4.94	350F			
Mississippi	Lime Valida	tion Runs	- Spike Cylinde	r 8.13 ppm								In Stack Conce	ntration Valu	es			
Run # 1	Ma HCI	Vdrv (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	ma/dscm	ppm	mg spike added	% Recoverv		
MVAL11A	0.87	121.1	1 no spike	2	7 300	293	29.24	743	760	0.12	NA	7.52	4.95	5			
MVAL11B	1.16	121.1	1 36.06	3	6 309	293	29.24	743	760	0.11	0.03	10.34	6.81	0.4	1 92%		
MVAL12A	1.04	120.34	1 no spike	2	9 302	293	29.28	744	760	0.11	NA	9.10	6.00	0.1			
MVAL12B	1.54	120.9	36.00) 3	0 303	293	29.28	744	760	0.11	0.03	13.46	8.87	0.42	2 105%		
intractice		12010	00.00		000	200	20.20			0	0.00	In Stack Conce	ntration Valu	es			
Run # 2	Ma HCI	Vdrv (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	ma/dscm	ppm	ma spike added	% Recovery		
MVAL21A	0.47	121.05	5 no spike	2	8 301	293	29.24	743	760	0.12	NA	4.08	2.69))	,,		
MVAL21B	0.82	123.94	4 36.06	3	3 306	293	29.24	743	760	0.12	0.03	7 10	4.68	0.43	92%		
MVAL22A	0.54	120.95	no spike	3	0 303	293	29.24	743	760	0.11	0.00	4.68	3.08	3			
MVAL22B	0.94	122.38	3 35.3	3 3	2 305	293	29.24	743	760	0.11	0.03	8.15	5.37	0.4	1 99%		
				-								In Stack Conce	ntration Valu	es			
Run # 3	Ma HCI	Vdrv (L)	V spike drv (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	ma/dscm	ppm	mg spike added	% Recoverv		
MVAL31A	0.60	120.2	2 no spike	2	8 301	293	29.21	742	760	0.11		5.28	3.48	3			
MVAL 31B	0.97	124.14	4 36.46	3	4 307	293	29.21	742	760	0.12	0.03	8.35	5.50	0.42	2 94%		
MVAL32A	0.61	120.34	1 no spike	3	0 303	293	29.31	744	760	0.11		5.37	3.54	4			
MVAL32B	1.06	121.44	4 36.14	3	2 305	293	29.31	744	760	0.11	0.03	9.28	6.11	0.42	2 103%		
				-								In Stack Concentration Values					
Run # 4	Ma HCI	Vdrv (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	ma/dscm	ppm	mg spike added	% Recovery		
MVAL41A	0.66	120.85	5 no spike	2	7 300	293	29.21	742	760	0.12	2	5.76	3.80)	,,		
MVAL41B	0.97	124.09	35.99	3	3 306	293	29.21	742	760	0 12	0.03	8.38	5.52	0.43	90%		
MVAL42A	0.75	121.92	2 no spike	2	9 302	293	29.21	742	760	0.12	2 0.00	6.46	4.26	0.11			
MVAL42B	1.19	120.91	1 35.47	3	1 304	293	29.21	742	760	0.11	0.03	10.46	6.89	0.4	1 104%		
				-								In Stack Conce	ntration Valu	es			
Run # 5	Ma HCI	Vdrv (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure torr	Std Pressure	DSCM Collected	DSCM Spike added	ma/dscm	ppm	mg spike added	% Recovery		
MVAL51A	0.58	121.445	5 no spike	2	9 302	293	29.21	742	760	0.12	2	5.07	3.34	l g d			
MVAL51B	1.11	121.335	36.18	3	5 308	293	29.21	742	760	0.11	0.03	9.85	6.49	0.4	1 113%	high value	
MVAL52A	0.84	117.38	3 no spike	2	8 301	293	29.21	742	760	0.11	0.00	7.49	4.94			riigit value	
MVAL52B	1.25	119.41	1 36.31	3	0 303	293	29.21	742	760	0.11	0.03	11.09	7.31	0.42	2 99%		
IIII IIII	1.20				000	200	20.21			0	0.00	In Stack Conce	ntration Valu	es			
Run # 6	Ma HCI	Vdry (L)	V spike dry (L)	Meter Temp C	Meter Temp	Std Temp	Pressure " Ha	Pressure forr	Std Pressure	DSCM Collected	DSCM Spike added	mg/dscm	nom	ma spike added	% Recovery		
MVAL61A	0.81	121 223	3 no spike	3	1 304	293	29.21	742	760	0 11		7.08	4 67	7	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,		
MVAL61B	1.00	121 150	36.084	1 3	6 309	203	29.21	742	760	0.11	0.03	8.89	5.86	0.4	1 83%	low value	
MVAL62A	0.92	123.45	5 no spike	3	1 304	293	29.21	742	760	0.12	0.00	7.93	5.22	0.1			
MVAL62B	1.32	121.12	35.94	1 3	3 306	293	29.21	742	760	0.11	0.03	11.66	7.68	0.4	1 101%		
					- 000	200	20.21	142	100	0.11	0.00		7.00	0.4			
						1							4 1F	Ava. Unspiked	98%	Ava. % Recov	erv
MVAL = Mis	sissippi Lime	validation	runs		-								6.42	Ava. Spiked	5076		
"A" trains up	sniked					1							5.42				
"B" trains sp	iked				-									1			
b			1	1		1	1	1	1	1	1	1	1	1	1		

5.0 DATA CONTROL OBJECTIVES

HCl Calibration Gas

The spike gas used for this testing program was a certified compressed HCl calibration gas prepared in a dry nitrogen balance. Because the accuracy certification for HCl standards is limited to ? 5%, the certified value of this cylinder was verified by two independent means. Independent verification of the gas value was first conducted using FTIR spectroscopy, and then an impinger train analysis of the gas was conducted in the field. (See Table 4-2 for CGA10A/B analysis.)

This cross checking mechanism provided for analysis using two separate analytic techniques (infrared and ion chromatography), and enabled more accurate representation of the true value of the cylinder gas.

A copy of the cylinder gas manufacturer certification is included in Appendix E.

Table 5-1 summarizes the certified cylinder gas value, the results from the independent FTIR analysis of the gas and the results of the impinger analysis of the gas.

Table 5-1 Analysis of the field canonation standard						
HCl Calibration Gas	Analysis Values					
Certified Value	9.8 ppm					
FTIR Analysis	7.3 ppm					
Impinger Analysis	8.1 ppm					

 Table 5-1 Analysis of the HCl Calibration Standard

A conversation on October 5, 2000 with Mr. Ted Neeme of Spectra Gas (the individual who signed the gas manufacturer certificate of analysis) revealed that FTIR spectrometry is used to analyze the HCl gas after it is placed into the cylinder An independent FTIR analysis of this gas by Prism Analytical Technologies Inc. (Mt. Pleasant Michigan) revealed that the cylinder value was about 25% less (2.5) ppm than that indicated by the certified value. Furthermore, the independent FTIR analysis was within 10% (1.2 ppm) of the impinger based analysis, which used a completely different analytical technique to quantify the HCl concentration. The disagreement between the certified value and the two separate independent analyses is problematic, and continues to cause concern for industries that must rely on accurate calibration gases for measuring HCl^6 .

For the purpose of calculating spike recoveries, the average value from the direct analysis of the cylinder gas by the duplicate impinger trains was used (8.1 ppm). This approach is consistent with the procedures and calculations prescribed by EPA FTIR Methods 320 and 321 (equations 3 &4) where direct analysis of the cylinder gas is used as the basis for calculating spike recovery.

Special Apparatus

The draft ASTM Method places strict requirements on the operating temperature of the front half (probe and filter) of the sampling train. This is necessary in order to prevent condensation and minimize reactions of the HCl with the materials of fabrication. In order to meet the temperature requirements of the method, the front half of the sample train was designed and fabricated to be specific for this method and validation approach.

The front half of the sampling train has: a single sample probe with two separate heated sections, two sets of filters, proportionally controlled heaters to maintain the probe and hot box temperatures within the

⁶ Personal conversations with Dr. Marty Spartz On-Line Technologies (November 2000), Mr. Les Keepper Prism Analytical Technologies (October 2000), Dr. Robert Spellicy IMACC (June 2000), and Dr. Jeff LaCoss Eastern Research Group (August 1995 and August 1998)

prescribed range, and provisions for delivering a gaseous spike into the sampling system at a point immediately upstream of the particulate filter.

Because it has been demonstrated that lime kiln dust adsorbs HCl (g) ³, the front half of the sampling train has a unique feature that is designed to reject as much particulate matter as possible. This design feature consists of a large pore 25-micron stainless steel frit with a particulate shield welded on one side. The assembly is fitted on the end of the probe and remains in-stack (with the shield positioned in the flow stream to deflect particles). This arrangement allows small particles and gases to pass into the sampling system and primary particulate filters (0.3 micron), and is similar to designs used by instrumental test methods such as gas filter correlation.

6.0 CONCLUSIONS

The Method 301 validation of the draft ASTM HCl Method met the data quality objectives of the test program. Method 301 statistical analyses of the data indicate the method has an insignificant bias at concentration levels less than 10 ppm as indicated by the very low bias and critical t values.

The percent recovery range of the spiked samples (within 30% of the expected values) further supports the method accuracy at this low concentration level.

The precision of the method in this concentration range is less than 20% RSD.