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Air



Final Report of Lime Manufacturing Industry Fourier Transform Infrared Spectroscopy

National Lime and Stone Company
Carey, Ohio



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Lime Kiln Source Characterization

Final Report

**Contract No. 68-D7-0001
Work Assignment 2-03**

**National Lime and Stone Company
Carey, Ohio**

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Table of Contents

	Page
1.0 INTRODUCTION	1-1
1.1 Objectives	1-1
1.2 Brief Site Discussion	1-2
1.3 Emissions Measurements Program	1-2
1.3.1 Test Matrix	1-2
1.3.2 Test Schedule	1-3
1.3.3 Deviations from Test Plan/Schedule	1-3
1.4 Test Report	1-4
2.0 SUMMARY OF RESULTS	2-1
2.1 Emissions Test Log	2-1
2.2 FTIR Results	2-1
2.2.1 Overview	2-1
2.2.2 FTIR Emission Results	2-2
3.0 SAMPLING AND ANALYTICAL PROCEDURE	3-1
3.1 Determination of Gaseous Organic HAPs, HCl, and Criteria Pollutants by Fourier Transform Infrared Spectroscopy (FTIR)	3-1
3.1.1 FTIR Sampling Equipment	3-1
3.1.2 Preparation for Sampling	3-4
3.1.3 Sampling and Analysis	3-6
3.1.4 FTIR Method Data Review Procedures	3-9
3.1.5 FTIR QA/QC Procedures	3-12
4.0 QUALITY ASSURANCE/QUALITY CONTROL	4-1
4.1 FTIR Analytical Quality Control	4-1
Appendix A FTIR Data Spreadsheet Calculation QA/QC Sheets	
Appendix B Gas Cylinder Certification Sheets	
Appendix C Raw FTIR Data	
Appendix D FTIR Field Data Sheets	
Appendix E Pre-test Calculations	
Appendix F Post-test Calculations	

List of Figures

	Page
2-1 HCl Inlet Run - Wet Scrubber	2-4
2-2 HCl Outlet Run - Wet Scrubber	2-5
3-1 FTIR Sampling and Measurement System	3-3

List of Tables

	Page
2-1. Emissions Test Log	2-1
2-2. Wet Scrubber FTIR HCl Results, ppmv	2-3
2-3. Other Species Detected by FTIR - Wet Scrubber Inlet	2-6
2-4. Other Species Detected by FTIR - Wet Scrubber Outlet	2-7
3-1. Typical FTIR Operating Parameters	3-6
3-2. Compounds for Which Reference FTIR Spectra Are Available in the ERG Spectral Library	3-10
4-1. HCl QA Spike Run 1 Results	4-3
4-2. HCl QA Spike Run 2 Results	4-4
4-3. Gas Standard Analysis Results	4-5

1.0 INTRODUCTION

The purpose of this testing program is to: (1) quantify hydrogen chloride (HCl) emission levels; and (2) gather screening data on other hazardous air pollutants (HAP) emissions from lime production plants to support a national emission standard for hazardous air pollutants (NESHAP).

Three measurement methods were conducted at this facility:

- Fourier Transform Infrared Spectroscopy (FTIR) (EPA Draft Method 320);
- Gas Filter Correlation - Infrared (GFC-IR) (EPA Method 322); and
- Dioxin/furan manual trains (EPA Method 23).

This report presents data from the FTIR measurements performed by Eastern Research Group. The EPA Method 23, 25A, and 322 measurements were conducted by Pacific Environmental Services, Inc. (PES), and Air Pollution Characterization and Control, Ltd. (APCC), under subcontract to PES, respectively. Process data was collected by Research Triangle Institute, Inc. (RTI), under contract to EPA. Please refer to the report prepared by PES for information and results of the Method 23, 25A, and 322 testing. For this test, screening means a measurement to determine approximate levels of species other than HCl.

The lime kiln facility and sampling locations tested in this program are detailed in the report prepared by PES.

1.1 Objectives

The objective of the FTIR testing of the lime facility was to quantify HCl and perform screening of other HAPs detectable by FTIR, using EPA Draft Method 320.

1.2 Brief Site Discussion

Testing was conducted at the National Line and Stone Company in Carey, Ohio. Testing was performed on the inlet and outlet of a wet-scrubbing device on Kiln #1, a natural-gas fired "doughnut" kiln. Detailed site information can be found in the report prepared by PES.

1.3 Emissions Measurements Program

This section provides an overview of the emissions measurement program conducted at the National Line and Stone Company, located in Carey, Ohio. Included in this section are summaries of the test matrix, test schedule, and authorized deviations from the test plan. Additional details on these topics are provided in the sections that follow.

1.3.1 Test Matrix

The complete sampling and analytical matrix performed are presented in the report prepared by PES. In this report, only FTIR-related test matrix will be provided. FTIR spectroscopy was used, in accordance with EPA Draft Method 320, to quantify HCl and, in a screening capacity, to measure other HAPs that FTIR can detect.

FTIR measurements were conducted in two sets:

- Unconditioned; and
- Conditioned.

Unconditioned sampling was conducted for the duration of the EPA Method 23 dioxin manual train runs. These runs were approximately 3 hours in duration. After completion of a dioxin run, the FTIR measured conditioned sample gas for a 1-hour period to screen for aromatic species such as benzene, toluene, etc.

During each run (i.e., unconditioned or conditioned) the FTIR analysis time was divided equally between inlet and outlet samples. Each location was monitored for no less than a total of 50 minutes. Some data points (typically, 5 minutes) were discarded for each set due to inlet/outlet sample mixing in the FTIR analysis cell. The actual amount of data points discarded is given later in this report. This procedure ensures the remaining data points were truly representative of the location being tested in that set.

1.3.2 Test Schedule

The test schedule for EPA Methods 23, 25A, and 322 measurements is given by the report prepared by PES. Section 2.1 gives the test log for the FTIR testing at this site.

1.3.3 Deviations from Test Plan/Schedule

Deviations from the original FTIR Site-Specific Test Plan (SSTP) are listed below:

- Testing was originally planned for 15 minute intervals between the inlet and outlet. The measurements consisted of collecting for 35 minute intervals at the outlet and inlet, in order to synchronize with the GFC-IR measurements performed by APCC.
- The EPA Work Assignment Manager authorized 1 hour total sample collection of the conditioned samples, ½ hour each on inlet and outlet. If other HAPs were detected, then the run would extend to the full 2 hours, as originally planned. In this case, no additional HAPs were detected in the conditioned samples.
- Some indicated sampling system temperatures were below the 350°F target that was stated in the test plan. These temperatures are the highest attainable with these sampling system components. It was determined after completion of the test program that the measured temperature of some of the sampling system components was a sensitive function of thermocouple location. When test thermocouples were inserted in the sample-wetted regions of the sampling system, they indicated temperatures above 350°F in all cases

1.4 Test Report

This final report, presenting all data collected and the results of the analyses, has been prepared in four sections, and an appendix as described below:

- Section 1 provides an introduction to the testing effort and includes a brief description of the test site and an overview of the emissions measurement program;
- Section 2 gives a summary of the test results for the FTIR results for HCl and other detected species;
- Section 3 presents detailed descriptions of the sampling and analysis procedures; and;
- Section 4 provides details of the quality assurance/quality control (QA/QC) procedures used on this program and the QC results.

A detailed description of the site, sampling locations, process and plant operation during the field test is provided in the PES-prepared report. Copies of the field data sheets and FTIR concentration data are contained in the appendices.

Six appendices are found in this report. They are organized as follows:

- Appendix A contains spreadsheet QA/QC review sheets;
- Appendix B contains QC gas cylinder certification sheets;
- Appendix C contains raw FTIR data;
- Appendix D contains FTIR field data sheets;
- Appendix E contains pretest calculations; and
- Appendix F contains post-test calculations.

2.0 SUMMARY OF RESULTS

This section provides the FTIR results of the emissions test program conducted at the National Lime and Stone Company in Carey, Ohio on September 2, 1998. Results for the extractive FTIR test conducted for HCl and screening for selected HAPs are provided in this section. Other (non-HAP) species detected are also reported. Testing was performed at the inlet and outlet of the wet scrubber.

2.1 Emissions Test Log

ERG performed extractive FTIR measurements for HCl and other HAPs. Table 2-1 presents the emissions test log that shows the test date, location, run number, test type, and run times for each method.

Table 2-1. Emissions Test Log

Date	Location	Run Number	Test Type	Run Time
9/02/98	Wet Scrubber (inlet/outlet)	Spike 1	FTIR HCl Spike (inlet)/ System QC (outlet)	08:06 - 09:21
9/02/98	Wet Scrubber (inlet/outlet)	Run 1	FTIR (Unconditioned) FTIR (Conditioned)	12:50 - 17:10 17:45 - 18:45
9/02/98	Wet Scrubber (inlet/outlet)	Spike 2	FTIR HCl Spike (inlet)/ System QC (outlet)	17:06 - 17:30

2.2 FTIR Results

2.2.1 Overview

FTIR data for HCl and other species in unconditioned sample gas were collected at the inlet and outlet of the wet scrubber. FTIR data collection of unconditioned samples was synchronized with EPA Method 23 manual dioxin/furan testing and EPA Method 322 GFC-IR HCl measurements. Conditioned samples were measured by FTIR for other HAP species.

FTIR data were collected by alternating sample analysis between inlet and outlet every 35 minutes. Inlet and outlet samples were drawn on a continuous basis; only the FTIR sample analysis was alternated between inlet and outlet. The first 10 (or 25) data points from each 35 minute inlet (or outlet, respectively) measurement period were discarded to eliminate data for samples containing both inlet and outlet sample gas. These data points correspond to the apparent response time of the complete FTIR sampling and analysis system (details on measurement of system response time are given below). The measurement run contained a total of 50 and 47 1-minute average data points for both inlet and outlet measurements, after discarding the transient data points. A 1-minute average data point is generated by analysis of a composite spectrum consisting of an average of 43 FTIR spectra collected over the 1 minute time period.

Section 2.1 gives the schedule of the tests performed at the Redland Stone Products in San Antonio, Texas. Both unconditioned and conditioned samples were analyzed. Conditioned samples were generated by passing the raw sample gas through a water vapor/carbon dioxide scrubbing system (see Section 3.1.1 for details). Conditioned samples extracted from the wet scrubber were measured after unconditioned sample extraction for the next hour. One minute average data points were generated by analysis of the composite spectrum consisting of an average of 43 FTIR spectra collected over the 1 minute period. These results are reported in Section 2.2.2.2.

The wet scrubber removal efficiency for HCl was measured from the inlet/outlet data from each location and is reported in Section 2.2.2.1.

2.2.2 FTIR Emission Results

This section contains the FTIR HCl test results for the wet scrubber inlet and outlet.

2.2.2.1 FTIR HCl Test Results. The estimated FTIR HCl detection limit for this study was between 0.11 and 0.18 parts per million by volume (ppmv). Approximately half the FTIR instrument analysis time was split equally between inlet and outlet. Results given below are organized by location. HCl removal efficiency was also calculated for each run. Raw data are presented in Appendix C listing each measured species 1-minute average concentration. All HCl emission runs were collected during the unconditioned tests.

Wet Scrubber Outlet/Inlet HCl Results—Table 2-2 gives a summary of the wet scrubber outlet/inlet FTIR HCl results. Appendix C provides 1-minute averages for all target species. The measured HCl removal efficiency due to the wet scrubber was 92.1 percent, assuming that the sample gas composition to the inlet of the scrubber did not change significantly during the outlet testing. Figures 2-1 and 2-2 show a real-time graph for the inlet and outlet runs, respectively.

Table 2-2. Wet Scrubber FTIR HCl Results, ppmv

	Run 1	
Date	9/02/98	
Time	12:50 - 17:10	
Location	Inlet	Outlet
Average	56.19	4.43
SD	7.13	0.22
Maximum	65.54	5.16
Minimum	45.43	4.03
NDP	50	47
RE	92.1	

SD - Standard Deviation

NDP - Number of data points measured

RE- Removal Efficiency in percent: $100 \times (\text{Avg. inlet} - \text{Avg. outlet}) / \text{Avg. inlet}$

Note: Raw data presented in Appendix C.

Figure 2-1. HCl Inlet Run - National Lime and Stone Company - Wet Scrubber

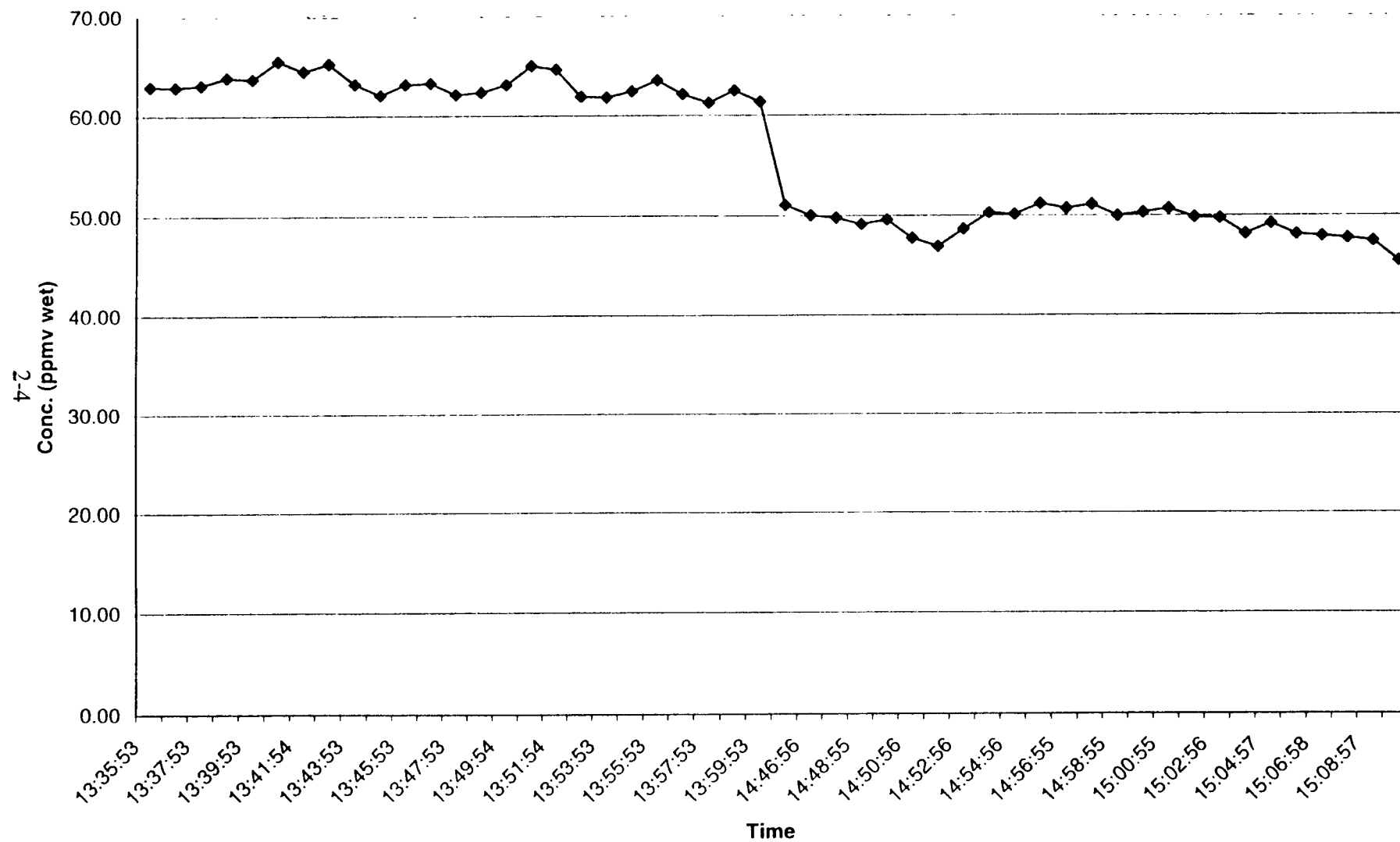
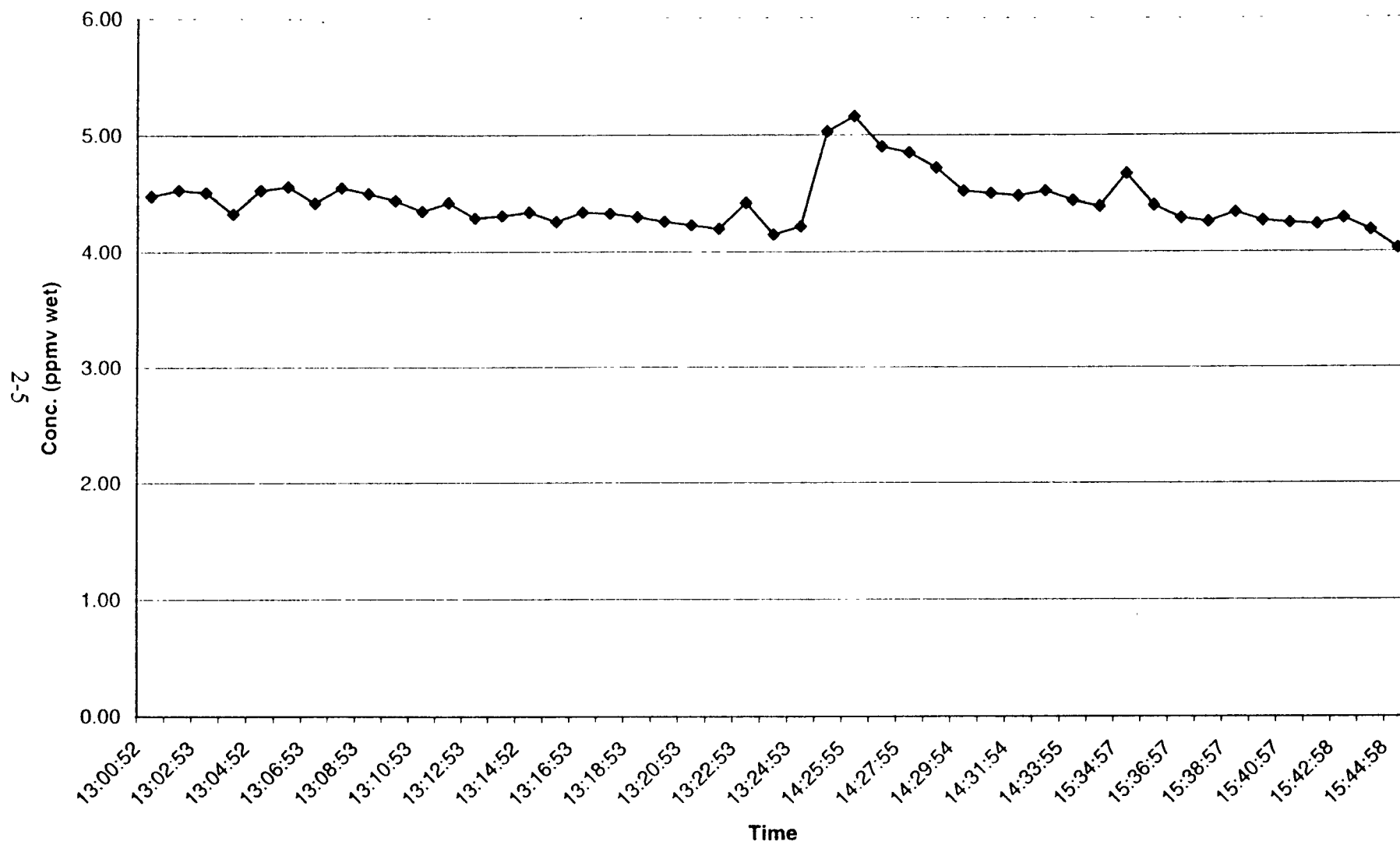


Figure 2-2. HCl Outlet Run - National Lime and Stone Company - Wet Scrubber



2.2.2.2 Other Species Detected by FTIR. Other species were detected during the unconditioned and conditioned FTIR test runs. These species were measured concurrently with HCl. Results given below are organized by location. No additional HAPs were detected in the conditioned samples.

Wet Scrubber Outlet/Inlet for Other Species Results—Tables 2-3 and 2-4 are a summary of the FTIR results for other species detected.

Table 2-3. Other Species Detected by FTIR - Wet Scrubber Inlet

(All values are ppmv, except CO₂ and H₂O in percent)

Parameter	CO ₂	CO	NO	H ₂ CO	C ₄ ⁺	CH ₄	H ₂ O
U/C	U	U	U	U	U	U	U
Average	9.59	28.7	37.9	1.00	2.50	5.66	9.27
Std. Dev.	1.42	18.9	7.22	0.35	0.81	1.29	1.11
Max.	11.5	113	48.9	1.65	3.55	12.1	10.9
Min.	7.63	13.7	27.9	0.45	1.61	3.65	7.74
NDP	50	50	50	50	50	50	50
EDL	0.060	0.24	6.9	0.15	0.14	2.2	0.10

U/C - Unconditioned (U) or Conditioned (C) Source Matrix

C₄⁺ - Total aliphatic hydrocarbons larger than 3 carbons (ppmv hexane equivalent)

NDP - Number of data points

EDL - Estimated detection limit for spectral region used for analysis

Note: Raw data reported in Appendix C.

Table 2-4. Other Species Detected by FTIR - Wet Scrubber Outlet

(All values are ppmv, except for CO₂ and H₂O in percent)

Parameter	CH ₄	C ₄ ⁺	CO ₂	CO	NO	H ₂ O
U/C	U	U	U	U	U	U
Average	3.95	0.85	9.28	17.7	38.4	13.1
Std. Dev.	0.68	0.08	0.175	12.0	2.62	0.115
Max.	6.39	1.02	9.62	66.4	44.3	13.3
Min.	2.51	0.73	8.96	7.60	33.6	12.8
NDP	47	47	47	47	47	47
EDL	2.1	0.14	0.061	0.27	6.6	0.11

U/C - Unconditioned (U) or Conditioned (C) Source Matrix

C₄⁺ - Total aliphatic hydrocarbons larger than 3 carbons (ppmv hexane equivalent)

NDP - Number of data points

EDL - Estimated detection limit for spectral region used for analysis

Note Raw data reported in Appendix C.

3.0 SAMPLING AND ANALYTICAL PROCEDURE

The sampling and analytical procedure used by ERG for the lime plant test program is extractive FTIR spectroscopy, conducted in accordance with EPA Draft Method 320. In this section, description of the FTIR method used is provided.

3.1 Determination of Gaseous Organic HAPs, HCl, and Criteria Pollutants by FTIR

The extractive FTIR measurement method is based on continuous extraction of sample gas from the stack, transporting the sample to the FTIR spectrometer and performing real-time spectral measurement of the sample gas. The sample gas spectra are analyzed in real time for target analytes, archived and possibly re-analyzed at a later date for other target analytes. This section provides details on the FTIR sampling and measurement system.

3.1.1 FTIR Sampling Equipment

The FTIR measurement system meets the sampling and analysis requirements set forth in EPA Draft Method 320, "Measurement of Vapor Phase Organic and Inorganic Emissions By Extractive Fourier Transform Infrared Spectroscopy." This system has been used with complete success with many source categories, and can also be adapted to switch quickly between two sources (i.e., inlet and outlet) with a single FTIR spectrometer.

The sampling and measurement system consists of the following components:

- Heated probe;
- Heated filter;
- Heat-traced Teflon® sample line;
- Teflon® diaphragm, heated-head sample pump;

- FTIR spectrometer;
- FTIR sample conditioning system; and
- QA/QC apparatus.

Figure 3-1 illustrates the extractive unconditioned FTIR sampling and measurement system. In operation at a stationary source, the sample is continuously extracted from the stack through the heated probe. Sample gas is then sent into a heated filter assembly that will remove any particulate matter from the sample stream to protect the remainder of the sampling and analysis system. The probe liner and filter body consist of glass, and the filter element is polytetrafluoroethylene (PTFE or Teflon®). In addition to providing an inert surface, the glass filter holder allows the operator to observe the filter loading during sampling operations. The probe and filter are contained in a heated box mounted on the stack and maintained at a nominal temperature of 177° C (350° F). A second probe/filter, heat-traced sample line, and heated head pump used are not shown in Figure 3-1.

After passing through the filter assembly, a primary heat-traced PTFE sample line transports the sample gas to the FTIR spectrometer maintained at approximately 177° C (350° F) driven by a heated-head PTFE diaphragm sample pump maintained at approximately 204° C (400° F). The sampling flow rate through the probe, filter, and sampling line is a nominal 20 standard liters per minute (LPM). Sample gas then enters an atmospheric pressure heated PTFE distribution manifold where it is sent to the FTIR spectrometer via a slipstream flowing at 9 LPM. Other slipstreams can be sent to other instruments, if necessary. Excess sample gas not used by instruments is vented to atmosphere.

A secondary heated-head PTFE diaphragm sample pump takes FTIR spectrometer sample gas from the distribution manifold maintained at approximately 204° C (400° F) and directed into the FTIR sample cell maintained at 185° C (365° F) for real-time analysis. The cell consists of nickel-plated aluminum, with gold-plated glass substrate mirrors and potassium chloride windows. Exhaust gas from the cell is vented to the atmosphere.

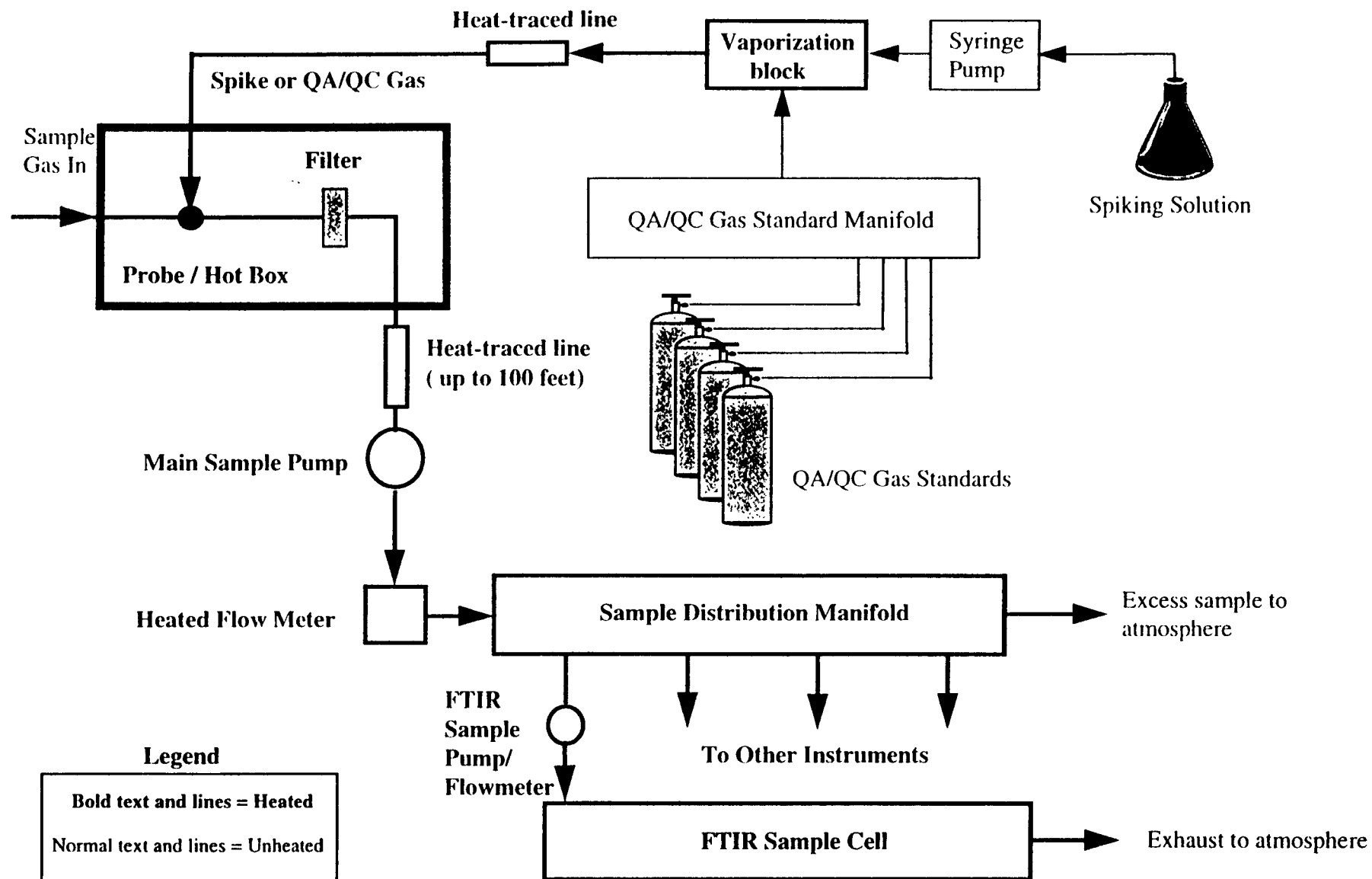


Figure 3-1. FTIR Sampling and Measurement System

Sample conditioning (when required) is achieved by passing raw matrix gas through a PermaPure® dryer and a series of impingers filled with sodium (or lithium) hydroxide pellets. The PermaPure® drier selectively removes water vapor and the sodium hydroxide pellets remove CO₂ and other acid gases. The sample conditioning apparatus is switched into the FTIR sample path by a valving system. Lower detection limits for some compounds can be achieved with a conditioned sample.

3.1.2 Preparation for Sampling

Before commencement of daily sampling operations, the following tasks were carried out:

- System leak check;
- Measurement of FTIR background spectrum;
- Instrumental QC; and
- Sampling and measurement system QC spike run.

Detailed descriptions of these tasks are described in the paragraphs below.

The heated sampling lines, probes, and a heated filter were positioned at the inlet and outlet locations. All heated components were brought to operating temperature, and a leak check of both inlet and outlet sampling systems were performed. The leak check was performed by plugging the end of the probe and watching the main sample rotameter to observe the reading. Positive leak check was confirmed when the rotameter reading was zero.

A background spectrum was measured using zero nitrogen through the cell. Next the QC gases were measured. They agreed to within ± 6 percent (± 10 percent for HCl) of target value. The QC gases used for this program include:

- Halocarbon 22 (H22), used to calibrate the pathlength. Halocarbon 22 is used for its highly linear response due to the lack of sharp spectral features, and is an extremely stable compound.
- Carbon monoxide (CO) used for a frequency calibration. Carbon monoxide is directly injected into the sample cell to measure photometric accuracy, validity of the nonlinear correction algorithm and serve as a frequency (i.e., wavelength) calibration. Acceptable limits for CO standard analysis are ± 6 percent of certified concentration;
- Methane/nitric oxide/carbon dioxide mixture, used for overall system performance check (calibration transfer standard) (acceptance limits are ± 6 percent of the certified concentration); and
- Hydrogen chloride standard, analyzed to verify the instrumental response of HCl, a key target analyte (acceptance limits are ± 10 percent of certified concentration).

The sampling and measurement system spike test was done to perform validation and directly challenge the complete system and provide information on system accuracy and bias. This test is conducted to satisfy the requirements set in EPA Draft Method 320 entitled "Measurement of Vapor Phase Organic and Inorganic Emissions By Extractive Fourier Transform Infrared Spectroscopy." Section B.1.C of Draft Method 320 gives a description of the dynamic spiking apparatus.

The following FTIR spiking procedure was used:

- Measured native stack gas until system equilibrates - took two measurements (i.e., two 1-minute samples);
- Started spike gas flow into sample stream, upstream of the heated filter;
- Let system equilibrate;
- Measured spiked sample stream for 2 minutes (i.e., two 1-minute samples);
- Turned off spike gas flow;
- Let system equilibrate with native stack gas; and

- Repeated cycle, two more times.

The above procedure produced six spiked/unspiked sample pairs. Spike recovery for six spiked/unspiked sample pairs were computed from the procedure given in Section 8.6.2 of EPA Draft Method 320. The recovery was between 70-130 percent and allowed the system to be considered acceptable for testing.

3.1.3 Sampling and Analysis

FTIR unconditioned sampling was performed simultaneously with the manual testing. The start and stop times of the manual methods were coordinated with the FTIR operator, so that FTIR data files can be coordinated with manual method start and stop times. FTIR inlet/outlet sampling was accomplished using two heated transfer lines, and a valving system to switch from inlet to outlet and vice versa.

Table 3-1 gives typical FTIR operating conditions. These parameters provide detection limits of 0.1-1 ppm for typical FTIR analytes, while providing adequate dynamic range (nominally 1-1,000 ppm). Some of these parameters are sample matrix dependent.

Table 3-1. Typical FTIR Operating Parameters

Parameter	Value
Spectral Range (cm ⁻¹)	400 - 4,000
Spectral Resolution (cm ⁻¹)	0.5
Optical Cell Pathlength (m)	3.4
Optical Cell Temperature (° C)	185
Sample Flow Rate (liters/minute)	9 (3.0 optical cell volumes/minute)
Integration Time (minutes)	1 (Average of 43 spectra)

Sample flow rate was determined by the data averaging interval and FTIR spectrometer sample cell volume. A minimum of three sample cell volumes of gas must flow through the

system to provide a representative sample during a single integration period. Typically, a 1 minute averaging period with a 3 liter volume sample cell gives a minimum flow rate of 9 LPM. Typically a flow rate of 20 standard LPM is used to accommodate the FTIR and other instrumentation on-site, and to minimize sample residence time in the sampling system.

The temperature of all sampling system components were at a minimum of 177°C (350 °F) to prevent condensation of water vapor or other analytes in the sampling system. Actual sampling system operating temperatures were determined before the start of testing. The FTIR sample cell temperature was maintained at 365° F (185° C) to minimize condensation of high-boiling point analytes on the cell optics.

FTIR cell pressure was monitored in real-time in order to calculate analyte concentration in parts-per-million. The cell was normally operated near atmospheric pressure with the cell pressure continuously monitored.

Sampling probe location was determined by the requirements set in EPA Method 1 in terms of duct diameters upstream and downstream of disturbances. Concurrent EPA Method 2 velocity measurements were not carried out at the same process stream location as the FTIR sampling point to provide mass emission rate determination. The stack gas velocity and flow rate were determined by the applicable manual test methods performed by PES. Velocity information can be found in the report prepared by PES.

Sampling and analysis procedures are straightforward for a single-source measurement. Once QA/QC procedures were completed at the beginning of the test day, the sample was allowed to flow continuously through the FTIR spectrometer cell and the software was instructed to start spectral data collection. The spectrometer collected one interferogram per second and averaged a number of interferograms to form a time-integrated interferogram. The typical averaging times range was approximately 1 minute. The interferogram was converted into a spectrum and analyzed for the target analytes. After spectral analysis, the spectrum was stored on the computer and later permanently archived. Spectral data collection was stopped after a

predetermined time, corresponding to a "run." Typical runs were approximately 3 hours long, giving approximately 180 1-minute average data points for each target analyte. The figure of 180 points were reduced by approximately 30 points due to elimination of five data points per switch between inlet/outlet samples and vice versa. At the end of the test day, the end-of-day QA/QC procedures were conducted.

Before any testing was started at a given site, an initial "snapshot" of the stack gas was taken with the FTIR measurement and analysis system to determine the true sample matrix. Because sample conditioning was required for certain analytes, the FTIR spectrometer analyzed these compounds after the unconditioned analysis. The order used during this program is shown in the table below.

Sampling Conditions	Sampling Time	Inlet	Outlet
Unconditioned	Synchronized with dioxin sampling	5 minute cell purge and 30 minute sample collection	5 minute cell purge and 30 minute sample collection
Conditioned	1 hour (after completion of dioxin run)	2 minute cell purge and 28 minute sample collection	2 minute cell purge and 28 minute sample collection

The sample being delivered to the FTIR cell alternated between the inlet and the outlet. The switching valve, located just upstream of the common manifold, was manually activated periodically to provide alternating inlet and outlet sample collections during each 3-hour period (the estimated dioxin run duration). This procedure resulted in a set of data points collected for the inlet and outlet, respectively. Five data points per set are discarded to eliminate analysis results with combined inlet and outlet samples.

FTIR method performance was gauged from the results of the QA/QC procedures given in Section B5 of EPA Draft Method 320. Acceptable spiking tests met acceptance for accuracy within ± 30 percent. The acceptable instrument diagnostic and system response checked

accuracy to be within ± 6 percent of target for all gas standards and ± 10 percent for the HCl standards. Acceptable system response check precision was 6 percent RSD.

Quantitative analysis was performed by a mathematical method called multi-variate least squares (commonly known as Classical Least Squares or CLS). CLS constructs an optimized linear combination (or 'fit') of the reference spectra to duplicate the sample spectrum, utilizing the Beer-Lambert Law. The Beer-Lambert Law states that the absorbance of a particular spectral feature due to a single analyte is proportional to its concentration. This relationship is the basis of FTIR quantitative analysis. The coefficients of each compound in the linear fit yield the concentration of that compound. If it is found that the quantitative analysis of a given compound responds non-linearly to concentration, a calibration curve is developed by measuring a series of reference spectra with differing optical depths (concentration times pathlength) and using them in the linear fit. Low molecular weight species such as water vapor and carbon monoxide require non-linear correction, possibly even at levels as low as 100 ppm-meters (concentration times pathlength). Analytes greater than 50-60 amu molecular weight usually does not require non-linear corrections. An experienced spectroscopist can determine whether non-linear corrections are necessary for an analyte in a given source testing scenario.

The ERG validated spectral database includes the compounds shown in Table 3-2. These spectra were validated in the laboratory at a cell temperature of 365° F (185° C) against certified gaseous standards. Any compounds identified in the stack gas and not included in the ERG database can be quantified if necessary after subsequent laboratory reference spectrum generation.

3.1.4 FTIR Method Data Review Procedures

The following procedure was conducted to review and validate the FTIR data.

**Table 3-2 . Compounds for Which Reference FTIR Spectra Are Available
in the ERG Spectral Library^a**

1-butene	chlorobenzene	<i>n</i> -butanol
1,3-butadiene	<i>cis</i> -2-butene	<i>n</i> -butane
2-methylpropane	cyclohexane	<i>n</i> -pentane
2-propanol	cyclopentane	nitric oxide
2-methoxyethanol	cyclopropane	nitrogen dioxide
2-methyl-2-propanol	ethane	nitrous oxide
2-methylbutane	ethylbenzene	<i>o</i> -cresol
4-vinylcyclohexane	ethylene	<i>o</i> -xylene
acetaldehyde	formaldehyde	<i>p</i> -cresol
acetic acid	hydrogen fluoride	<i>p</i> -xylene
acetone	hydrogen chloride	phenol
acetylene	isobutylene	propane
acrolein	<i>m</i> -xylene	propylene
ammonia	<i>m</i> -cresol	styrene
benzene	methane	sulfur dioxide
carbon monoxide	methanol	toluene
carbon dioxide	methyl ethyl ketone	<i>trans</i> -2-butene
carbonyl sulfide	methylene chloride	water vapor

^a Spectra were collected at a cell temperature of 365° F (185° C).

A. Post-test Data Review procedure (on-site)

1. Examine the concentration vs. time series plot for each compound of interest, and identify regions with the following characteristics:
 - sudden change in concentration;
 - unrealistic concentration values;
 - significant changes in 95 percent confidence intervals reported by software; and
 - sudden increase of noise in data.
2. Select representative spectra from the time periods indicated from Step 1.
3. Subtract from each representative spectrum chosen in Step 2 a spectrum taken immediately prior in time to the indicated time region.
4. Manually quantitate (including any non-linear corrections) for the species in question and compare the result to the difference in software-computed concentrations for respective spectra.
5. If concentration values in Step 4 do not agree to within 5 percent, determine whether the difference is due to a recoverable or non-recoverable error.
- 6 (i). If the error is non-recoverable, the spectra in the indicated time region are declared invalid.
- 7 (ii). If the error is recoverable, and time permits, determine possible source(s) of error and attempt to correct. If time is critical, proceed with measurement. If correction is achieved, conduct QA/QC checks before continuing.
8. Determine the peak-to-peak scatter or the root mean square (RMS) noise-equivalent-absorbance (NEA) for the representative spectra.
9. If the NEA exceeds the limits required for acceptable detection limits, the spectra in the time region are declared invalid (due to non-recoverable error).
10. Data found invalid are subject to re-measurement.

B. Final Data Review (off-site)

The procedures for final data review include those given above. However, if a non-recoverable error was found during this phase, the data are considered invalid. In addition, the following procedures are carried out by the spectroscopist to perform a final data validation:

1. If any recoverable data errors are detected from the procedure, determine the cause and perform any necessary corrections.
2. For analytes that were not detected or detected at low levels:
 - estimate detection limits from validated data;
 - check for measurement bias.
3. Verify spreadsheet calculations by independent calculation (results in Appendix A).

3.1.5 FTIR QA/QC Procedures

The FTIR QA/QC apparatus was used to perform two functions:

- Dynamic analyte spiking; and
- Instrumental performance checks.

Dynamic analyte spiking was used for quality control/quality assurance of the complete sampling and analysis system. Dynamic spiking is continuous spiking of the matrix gas to provide information on system response, sample matrix effects, and potential sampling system biases. Spiking is accomplished by either:

- Direct introduction of a certified gas standard; or
- Volatilization of a spiking solution.

Certified gas standards are preferred due to simplicity of use, but many target analytes cannot be obtained as certified gas standards, and must be spiked using standards generated by volatilized solutions.

Gaseous spiking is carried out by metering the spike gas into the sample stream at a known rate. Spike levels are calculated from mass balance principles. When certified gas standards are used, a dilution tracer, such as sulfur hexafluoride, is used to directly measure the fraction of spike gas spiked into the sample. This technique can be used instead of mass balance calculations.

FTIR method performance is gauged from the results of the QA/QC. Acceptable spiking tests will meet Draft Method 320 criteria (i.e., accuracy of within ± 30 percent) or a statistical equivalent when less than 12 spiked/unspiked pairs are collected. The EPA Draft Method 320 instructs the user to determine the percent spike recovery of 3 pairs of spiked/unspiked samples. The Draft EPA Draft Method 320 acceptance criterion is 70 to 130 percent recovery for the three pairs of samples. The acceptable instrument diagnostic and system response check accuracy were within ± 6 percent of target (± 10 percent for HCl standards). Acceptable system response check precision was 6 percent RSD.

4.0 QUALITY ASSURANCE/QUALITY CONTROL

Specific QA/QC procedures were strictly followed during this test program to ensure the production of useful and valid data throughout the project. A detailed presentation of QC procedures for all sampling and analysis activities can be found in the Site Specific Test Plan and Quality Assurance Project Plan for this project. This section reports all QC results so that the data quality can be ascertained.

In summary, a high degree of data quality was maintained throughout the project. All sampling system leak checks met the QC criteria as specified in Draft Method 320. Acceptable spike recoveries and close agreement between duplicate analyses were shown for the sample analyses. The data completeness was 100 percent, based on changes authorized by the Work Assignment Manager.

4.1 FTIR Analytical Quality Control

Dynamic analyte spiking was used for quality control/quality assurance of the complete sampling and analysis system. Dynamic spiking is continuous spiking of the sample gas to provide information on system response, sample matrix effects, and potential sampling system biases. Spiking was accomplished by direct introduction of a certified gas standard.

Gaseous spiking was carried out by metering the spike gas into the sample stream at a known rate. A sulfur hexafluoride dilution tracer was used to directly measure the fraction of spike gas spiked into the sample. EPA Draft Method 320 limits the dilution of the sample gas to 10 percent.

Before any testing was started at a given site, an initial “snapshot” of the stack gas is taken with the FTIR measurement and analysis system to determine the true sample matrix. If any target analytes are present at significantly higher levels than expected, adjustments were

made to the cell pathlength and/or the spectral analysis regions used for quantitative analysis. These adjustments minimized interferences due to unexpectedly high levels of detected analytes.

FTIR method performance is gauged from the results of the QA/QC. All spiking tests met Method 320 criteria. The acceptable instrument diagnostic and system response check accuracy should be within ± 6 percent of target for all gas standards except HCl. The accuracy for the HCl standard should be within ± 10 percent.

Analytical QC checks for the FTIR system consisted of the following:

- Dynamic spiking of HCl;
- Direct measurement of a HCl gas standard;
- Direct measurement of a CO gas standard;
- Direct measurement of a CH₄, NO₂, and CO₂ standard; and
- Pathlength calibration using H₂O.

Dynamic spiking runs were conducted twice daily: before and after testing. Six spiked/unspiked data points were collected. Statistical calculations consistent with EPA Method 301 were performed on the data. Recovery of 70-130 percent was the acceptance criteria. Table 4-1 summarizes the dynamic spiking results. On the inlet, all dynamic spiking tests met the above acceptance criteria. In all runs, sample gas was diluted 10 percent or less.

Direct instrumental measurement of HCl, CO, H₂O, and a CH₄, NO₂ and CO₂ mixture was conducted before and after daily testing activities. Acceptance criteria are normally ± 6 percent of target, using EPA protocol gases. However, since the HCl standard was obtained at a ± 5 percent analytical tolerance, the acceptance criteria was set at ± 10 percent. FTIR NO_x is measured as NO + NO₂. Examination of Table 4-2 shows that all QC checks met the above criteria.

Table 4-1. HCl QA Spike Run 1 Results - National Lime and Stone Company

Outlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	71.55	141.40	75.06	74.96		0.147	0.073
2	69.49	141.70	77.58	79.54		0.156	0.077
3	67.71	139.60	76.88	75.98		0.149	0.074
4	66.54	139.90	78.27	75.98		0.149	0.074
5	67.00	141.20	79.01	73.94		0.145	0.072
6	65.93	142.80	81.64	74.45		0.146	0.072
Average	68.04	141.10	78.07	75.81	102.99	0.149	0.074
Inlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	8.61	22.97	14.86	15.21		0.117	0.059
2	8.18	22.18	14.49	15.60		0.120	0.060
3	7.75	21.51	14.22	15.47		0.119	0.060
4	7.52	21.13	14.06	15.60		0.120	0.060
5	7.26	21.35	14.55	16.51		0.127	0.064
6	7.32	20.93	14.05	15.60		0.120	0.060
Average	7.77	21.68	14.37	15.67	91.75	0.121	0.060

NOTE: The spike runs were conducted before and after the test runs, therefore the minimum and maximum values listed here may be different than those listed in the test runs, Section 2. Sample gas dilution was held to 10 percent or less in all runs. Percent recovery is defined in Draft Method 320.

(Stock spike gas values for HCl and SF6 values are 253 ppmv and 5.08 ppmv, respectively).

$$\% \text{ Recovery} = 100 \times \frac{\text{Corrected Difference}}{\text{Spike level}}$$

$$\text{Corrected Difference} = \text{Spiked} - (1 - \text{Dilution Ratio}) \times \text{Unspiked}$$

Table 4-2. HCl QA Spike Run 2 Results - National Lime and Stone Company

Outlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	59.98	116.80	62.19	92.29		0.181	0.090
2	59.23	132.40	78.48	92.29		0.181	0.090
3	58.41	134.90	81.72	92.29		0.181	0.090
4	57.52	129.30	76.36	82.09		0.161	0.080
5	56.44	128.30	76.22	79.54		0.156	0.077
6	57.51	131.40	78.27	78.52		0.154	0.076
Average	58.18	128.85	75.54	86.17	87.66	0.169	0.084

Inlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	6.11	23.36	17.72	20.15		0.155	0.078
2	5.67	16.60	11.17	11.05		0.085	0.043
3	5.39	14.92	9.77	11.44		0.088	0.044
4	5.12	14.43	9.53	11.18		0.086	0.043
5	5.04	14.22	9.41	11.70		0.090	0.045
6	4.94	14.02	9.30	11.44		0.088	0.044
Average	5.38	16.26	11.15	12.83	86.89	0.099	0.049

NOTE: The spike runs were conducted before and after the test runs, therefore the minimum and maximum values listed here may be different than those listed in the test runs, Section 2. Sample gas dilution was held to 10 percent or less in all runs. Percent recovery is defined in Draft Method 320.

(Stock spike gas values for HCl and SF6 values are 253 ppmv and 5.08 ppmv, respectively).

$$\% \text{ Recovery} = 100 \times \frac{\text{Corrected Difference}}{\text{Spike level}}$$

$$\text{Corrected Difference} = \text{Spiked} - (1 - \text{Dilution Ratio}) \times \text{Unspiked}$$

Table 4-3. Gas Standard Analysis Results

Date	Time	Compound	True (ppm)*	Result (ppm)*	% Recovery
8/31/98	07:56 AM	HCl	54.3	52.0	95.8
		CO	102.3	101.4	99.1
		CH ₄	491	492.2	100.2
		NO	503	494.0	98.2
		CO ₂	4.99 %	5.15 %	103.2
		H22		3.30 m	
9/01/98	02:02 AM	HCl	54.3	53.6	98.7
		CO	102.3	100.8	98.5
		CH ₄	491	489.0	99.6
		NO	503	491.0	97.6
		CO ₂	4.99 %	5.07 %	101.6
		H22		3.29 m	

HCl Gas Standard Accuracy: ± 5 percent; Acceptance Criteria: ± 10 percent of target.

CO Gas Standard Accuracy: ± 1 percent; Acceptance Criteria: ± 6 percent of target.

CH₄, NO₂, and CO₂ Gas Standard Accuracy: $\pm 1\%$; Acceptance Criteria: ± 6 percent of target.

* All compounds are recorded in ppm except for CO₂ in percent (%), and H22 in meters (m).
The Halocarbon 22 (H22) is used to calibrate the pathlength.

APPENDIX A

FTIR DATA SPREADSHEET CALCULATION
QA/QC SHEETS

FTIR QA/QC REVIEW
Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 1. Excel QA/QC workbook
 2. Inlet and Outlet QA/QC information

Facility Name: CAREY LIME KLIN	DATE: 09/02/98
Source Location (INLET or OUTLET) OUTLET	TIME: 1745-1845
Run Description Run of Outlet Conditioned	
Reviewer: M. D. [Signature]	Date: Dec-10-98

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match reference values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments:					
1.					
2.					
3.					

FTIR QA/QC REVIEW **Calculation and Methodology QA/QC Checklist**

- For each facility tested, the reviewer will have:
 - Excel QA/QC workbook
 - Inlet and Outlet QA/QC information

Facility Name:	CAREY Lime kiln	DATE:	09/02/98
Source Location (INLET or OUTLET)	Inlet	TIME:	1745-1845
Run Description	Run 01 Inlet Conducted		
Reviewer:	M. D. [Signature]	Date:	Dec-10-98

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match references values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments					
1.					
2.					
3.					

FTIR QA/QC REVIEW

Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 1. Excel QA/QC workbook
 2. Inlet and Outlet QA/QC information

Facility Name: CAREY LIME MILN	DATE: 09/02/08
Source Location (INLET or OUTLET): INLET	TIME: 12507710
Run Description: Run 01 Inlet Unconditional	
Reviewer: M.D. Stewart	Date: 09-10-08

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match reference values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments:					
1.					
2.					
3.					

FTIR QA/QC REVIEW

Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 1. Excel QA/QC workbook
 2. Inlet and Outlet QA/QC information

Facility Name: CAREY LIME MILN	DATE: 09/02/08
Source Location (INLET or OUTLET): INLET	TIME: 12507710
Run Description: Run 01 Inlet Unconditional	
Reviewer: M.D. BLANT	Date: DE-10-08

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match reference values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments					
1.					
2.					
3.					

FTIR QA/QC REVIEW
Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 1. Excel QA/QC workbook
 2. Inlet and Outlet QA/QC information

Facility Name: CAREY LIME MILN	DATE: 09/02/08
Source Location (INLET or OUTLET): INLET	TIME: 12507710
Run Description: Run 01 Inlet Unconditioned	
Reviewer: M.D. Stewart	Date: 09-10-08

Checklist	Yes	No	N/A	Not done	Resolution
A: QA/QC entries match reference values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B: Check that calculations are correct					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C: Comments					
1.					
2.					
3.					

FTIR QA/QC REVIEW **Calculation and Methodology QA/QC Checklist**

- For each facility tested, the reviewer will have:
 - Excel QA/QC workbook
 - Inlet and Outlet QA/QC information

Facility Name:	CAREY LINE KUN	DATE:	09/02/98
Source Location (INLET or OUTLET)	OUTLET	TIME:	1250-1710
Run Description	Run 01 OUTLET unconditional		
Reviewer:	M. D. [Signature]	Date:	02-10-98

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match references values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments					
1.					
2.					
3.					

FTIR QA/QC REVIEW

Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 - Excel QA/QC workbook
 - Inlet and Outlet QA/QC information

Facility Name:	CAREY LIME KILN	DATE:	09/02/08
Source Location (INLET or OUTLET)	INLET	TIME:	1250-7710
Run Description	Run 01 Inlet Unconditioned		
Reviewer:	M.D. BLANT	Date:	09-10-08

Checklist	Yes	No	N/A	Not	Resolution
A. QA/QC entries match reference values					
Check the following by comparing the printouts of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments					
1.					
2.					
3.					

FTIR QA/QC REVIEW

Calculation and Methodology QA/QC Checklist

- For each facility tested, the reviewer will have:
 - Excel QA/QC workbook
 - Inlet and Outlet QA/QC information

Facility Name:	CAREY LIME MILN	DATE:	09/02/98
Source Location (INLET or OUTLET)	INLET	TIME:	12507710
Run Description	Run 01 Inlet Uncondensed		
Reviewer:	M.D. BLANK	Date:	09-10-98

Checklist	Yes	No	N/A	Not det.	Resolution
A. QA/QC entries match reference values					
Check the following by comparing the printout of the QA/QC run to the original run information:					
1. Pollutants matches pollutants in both the original and QA/QC data	✓				
2. Times for Inlet/Outlet samples match.	✓				
3. Number of data points match.	✓				
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	✓				
5. Verify that the QA/QC value is zero. This indicated that both the original and the QA/QC values are identical.	✓				
B. Check that calculations are correct:					
1. No mathematical errors	✓				
1. No errors in the data macro	✓				
2.					
C. Comments					
1.					
2.					
3.					

APPENDIX B

GAS CYLINDER CERTIFICATION SHEETS

REC'D AUG 14 1998



SPECTRA GASES

3434 Route 22 West • Branchburg, NJ 08876 USA Tel: (908) 252-9300 • (800) 932-0624 • Fax: (908) 252-0811

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

**CERTIFICATE
OF
ANALYSIS**

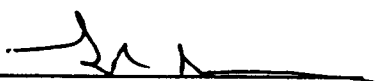
SGI ORDER # :	134942	
ITEM# :	1	CYLINDER # : 1689487Y
CERTIFICATION DATE:	8/10/98	CYLINDER PRES: 2000 psig
P.O.# :	9101008011-R132	CYLINDER VALVE: CGA 330
BLEND TYPE:	CERTIFIED	

ANALYTICAL ACCURACY: + / - 5%

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	50.0 ppm	54.3 ppm
Sulfur Hexafluoride	2.00 ppm	2.01 ppm
Nitrogen	Balance	Balance

Sulfur Hexafluoride is +/- 2%

ANALYST:


Ted Neeme

DATE: 8/10/98

**SPECTRA GASES**

277 Cort Street • Irvington, NJ 07111 USA Tel: (973) 372-2060 • (800) 929-2427 • Fax: (973) 372-8551

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # :	126876	CYLINDER # :	1852209Y
ITEM# :	1	CYLINDER PRES:	2000 PSIG
CERTIFICATION DATE:	8/29/97	P.O.# :	7904004005-R562
BLEND TYPE:	CERTIFIED		

ANALYTICAL ACCURACY: +/- 5 %

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	200 ppm	210 ppm
Sulfur Hexafluoride	20.0 ppm	20.2 ppm
Nitrogen	Balance	Balance

ANALYST: _____


Ted Neeme

DATE: 8/29/97

**SPECTRA GASES**

277 Coit Street • Irvington, NJ 07111 USA Tel: (973) 372-2060 • (800) 929-2427 • Fax: (973) 372-8551

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # :	128118	CYLINDER # :	1757972Y
ITEM# :	1	CYLINDER PRES:	2000 psig
CERTIFICATION DATE:	10/16/97	P.O.# :	7904004005-R690
BLEND TYPE:	CERTIFIED		

ANALYTICAL ACCURACY: +/- 2%

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride**	200 ppm	220 ppm
Sulfur Hexafluoride	20.0 ppm	20.0 ppm
Nitrogen	Balance	Balance

** Analytical Accuracy of Hydrogen Chloride is +/- 5%

ANALYST: _____

Ted Neeme

DATE: 10/16/97

**SPECTRA GASES**

REC'D MAY 15 1998

277 Coit Street • Irvington, NJ 07111 USA Tel: (973) 372-2060 • (800) 929-2427 • Fax: (973) 372-8551

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # :	132874	CYLINDER # :	1370597Y
ITEM# :	2	CYLINDER PRES:	2000 psig
CERTIFICATION DATE:	5/11/98	P.O.# :	9101008004-R986
BLEND TYPE:	CERTIFIED		

ANALYTICAL ACCURACY: +/- 2%*

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	250 ppm	253 ppm
Sulfur Hexafluoride	5.00 ppm	5.08 ppm
Nitrogen	Balance	Balance

* Analytical Accuracy of Hydrogen Chloride is +/- 5%

ANALYST: Michael Doyle

Mike Doyle

DATE: 5/11/98

USA • United Kingdom • Germany • Japan

ISO 9002

**SPECTRA GASES**

3434 Route 22 West • Branchburg, NJ 08876 USA Tel: (908) 252-9300 • (800) 932-0624 • Fax: (908) 252-0811

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville , NC 27560

**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # : 134942
ITEM# : 2
CERTIFICATION DATE: 8/10/98
P.O.# : 9101008011-R132
BLEND TYPE: CERTIFIED


CYLINDER # : 1015632Y
CYLINDER PRES: 2000 psig
CYLINDER VALVE: CGA 330

ANALYTICAL ACCURACY: + / - 5%

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	250 ppm	260 ppm
Sulfur Hexafluoride	2.00 ppm	2.00 ppm
Nitrogen	Balance	Balance

Sulfur Hexafluoride is +/- 2%

ANALYST: _____


Ted Neeme

DATE: 8/10/98

USA • United Kingdom • Germany • Japan



277 Coit Street • Irvington, NJ 07111 USA Tel: (973) 372-2060 • (800) 929-2427 • Fax: (973) 372-8551

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # :	132874	CYLINDER # :	1757934Y
ITEM# :	1	CYLINDER PRES:	2000 psig
CERTIFICATION DATE:	5/11/98	P.O.# :	9101008004-R986
BLEND TYPE:	CERTIFIED		

ANALYTICAL ACCURACY: +/- 2%*

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	500 ppm	516 ppm
Sulfur Hexafluoride	5.00 ppm	5.09 ppm
Nitrogen	Balance	Balance

* Analytical Accuracy of Hydrogen Chloride is +/- 5%

ANALYST: Mike Doyle

Mike Doyle

DATE: 5/11/98

**SPECTRA GASES**

3434 Route 22 West • Branchburg, NJ 08876 USA Tel: (908) 252-9300 • (800) 932-0624 • Fax: (908) 252-0811

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

**CERTIFICATE
OF
ANALYSIS**

SGI ORDER # : 134942
ITEM# : 3
CERTIFICATION DATE: 8/10/98
P.O.# : 9101008011-R132
BLEND TYPE: CERTIFIED

CYLINDER # : 982153Y
CYLINDER PRES: 2000 psig
CYLINDER VALVE: CGA 330

ANALYTICAL ACCURACY: + / - 5%

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Hydrogen Chloride	1,000 ppm	1,030 ppm
Sulfur Hexafluoride	2.00 ppm	2.02 ppm
Nitrogen	Balance	Balance

Sulfur Hexafluoride is +/- 2%

ANALYST: Ted Neeme
Ted Neeme

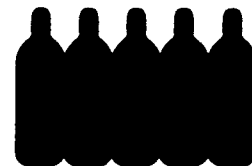
DATE: 8/10/98

USA • United Kingdom • Germany • Japan



SPECTRA GASES

277 Coit St. • Irvington, NJ 07111 USA Tel.: (201) 372-2060 • (800) 932-0624 • Fax: (201) 372-8551
Shipped From: 80 Industrial Drive • Alpha, N.J. 08865



CERTIFICATE OF ANALYSIS

EPA PROTOCOL MIXTURE PROCEDURE #: G1

CUSTOMER: Eastern Research Group Inc.
SGI ORDER # : 126876
ITEM# : 3
P.O.# : 7904004005-R562

CYLINDER # : CC80890
CYLINDER PRES: 2000 PSIG
CGA OUTLET: 350

CERTIFICATION DATE: 8/26/97
EXPIRATION DATE: 8/26/2000

CERTIFICATION HISTORY

COMPONENT	DATE OF ASSAY	MEAN CONCENTRATION	CERTIFIED CONCENTRATION	ANALYTICAL ACCURACY
Carbon Monoxide	8/19/97 8/26/97	102.1 ppm 102.6 ppm	102.3 ppm	+/- 1%

BALANCE Nitrogen

REFERENCE STANDARDS

COMPONENT	SRM/NTRM#	CYLINDER#	CONCENTRATION
Carbon Monoxide	SRM-1680b	CLM010013	490.4 ppm

INSTRUMENTATION

COMPONENT	MAKE/MODEL	SERIAL #	DETECTOR	CALIBRATION DATE(S)
Carbon Monoxide	Horiba-VIA-510	570423011	NDIR	8/26/97

THIS STANDARD WAS CERTIFIED ACCORDING TO THE EPA PROTOCOL PROCEDURES.
DO NOT USE THIS STANDARD IF THE CYLINDER PRESSURE IS LESS THAN 150 PSIG.

ANALYST: _____

TED NEEME

DATE: 8/26/97



SPECTRA GASES

277 Cort Street • Irvington, NJ 07111 USA Tel: (973) 372-2060 • (800) 929-2427 • Fax: (973) 372-8551

SHIPPED FROM: 80 INDUSTRIAL DRIVE ALPHA, NJ. 08865 TEL: (908) 454-7455

SHIPPED TO: Eastern Research Group Inc.
900 Perimeter Park
Morrisville, NC 27560

CERTIFICATE OF ANALYSIS

SGI ORDER # :	128118	CYLINDER # :	CC82244
ITEM# :	2	CYLINDER PRES:	2000 psig
CERTIFICATION DATE:	10/16/97	P.O.# :	7904004005-R690
BLEND TYPE:	CERTIFIED		

ANALYTICAL ACCURACY: +/- 2%

COMPONENT	REQUESTED GAS CONC	ANALYSIS
Chlorodifluoromethane	40.0 ppm	40.3 ppm
Nitrogen	Balance	Balance

ANALYST: 
Ted Neeme

DATE: 10/16/97



SPECTRA GASES

277 Coit St. • Irvington, NJ 07111 USA Tel.: (201) 372-2060 • (800) 932-0624 • Fax: (201) 372-8551
Shipped From: 80 Industrial Drive • Alpha, N.J. 08865



CERTIFICATE OF ANALYSIS

EPA PROTOCOL MIXTURE

PROCEDURE #: G1

CUSTOMER: Eastern Research Group Inc.
SGI ORDER #: 126876
ITEM#: 5
P.O.#: 7904004005-R562

CYLINDER #: CC79878
CYLINDER PRES: 2000 PSIG
CGA OUTLET: 660

CERTIFICATION DATE: 8/27/97

EXPIRATION DATE: 8/19/99

CERTIFICATION HISTORY

COMPONENT	DATE OF ASSAY	MEAN CONCENTRATION	CERTIFIED CONCENTRATION	ANALYTICAL ACCURACY
Methane	8/21/97	491 ppm	491 ppm	+/- 1%
Nitric Oxide	8/20/97 8/27/97	502.1 ppm 504.6 ppm	503 ppm	+/- 1%
NOx			503 ppm	Reference Value Only
Carbon Dioxide	8/19/97	4.99 %	4.99 %	+/- 1%

BALANCE

Nitrogen

REFERENCE STANDARDS

COMPONENT	SRM/NTRM#	CYLINDER#	CONCENTRATION
Methane	SRM-2751	CAL013479	98.6 ppm
Nitric Oxide	NTRM-81687	CC57165	1009 ppm
Carbon Dioxide	SRM-1674b	CLM007273	6.98 %

INSTRUMENTATION

COMPONENT	MAKE/MODEL	SERIAL #	DETECTOR	CALIBRATION DATE(S)
Methane	H. Packard-6890	US00001434	GC - FID	8/21/97
Nitric Oxide	Nicolet-760	ADM9600121	FTIR	8/27/97
Carbon Dioxide	Horiba-VIA-510	571417045	NDIR	7/25/97

THIS STANDARD WAS CERTIFIED ACCORDING TO THE EPA PROTOCOL PROCEDURES.

DO NOT USE THIS STANDARD IF THE CYLINDER PRESSURE IS LESS THAN 150 PSIG.

ANALYST:

TED NEEME

DATE: 8/27/97

APPENDIX C
RAW FTIR DATA

All data in ppmv wet basis																																								
Title: Outlet then inlet run 1																																								
Description: Carey line run 1 outlet then inlet																																								
Method title: Ohio Line																																								
Starting Date/Time: Wed Sep 02 12 49 56 1998																																								
Time	H2O	(%H2O)	CO2	(%CO2)	CO	(%CO)	NO	(%NO)	NC2	(%NC2)	N2O	(%N2O)	NH3	(%NH3)	CH4	(%CH4)	C2H6	(%C2H6)	C2H4	(%C2H4)	H2CO	(%H2CO)	ACTLD	(%ACTLD)	C4+	(%C4+)	CH3OH	(%CH3OH)	HCL	(%HCL)	SF6	(%SF6)	SO2	(%SO2)	COS	(%COS)	COHI	(%COHI)	NOX	(%NOX)
13 35 53	105307.71	1199.34	113635.02	736.56	23.23	0.33	45.20	7.59	0.00	1.37	0.00	0.63	0.00	0.31	5.26	2.35	0.00	0.64	0.00	0.50	0.67	0.19	0.00	0.74	1.71	0.16	0.00	0.53	82.96	0.25	0.00	0.01	0.00	0.84	0.14	0.11	82.31	7.49	45.20	8.96
13 36 54	108116.92	1205.00	114983.09	739.25	23.05	0.38	41.29	7.62	0.00	1.42	0.00	0.55	0.00	0.31	6.98	2.35	0.00	0.64	0.00	0.50	0.63	0.19	0.00	0.71	1.75	0.16	0.00	0.53	82.90	0.24	0.00	0.01	0.00	0.71	0.12	0.12	120.40	7.79	41.29	9.05
13 37 53	108593.72	1209.90	113624.71	740.55	21.05	0.32	44.75	7.56	0.00	1.53	0.00	0.53	0.00	0.31	5.02	2.34	0.00	0.64	0.00	0.50	0.45	0.19	0.00	0.71	1.76	0.16	0.00	0.52	83.09	0.24	0.00	0.01	0.00	0.84	0.12	0.11	82.87	7.51	44.75	9.09
13 38 54	107591.38	1188.37	112711.47	727.22	23.21	0.35	43.68	7.55	0.00	1.48	0.00	0.58	0.00	0.31	5.45	2.36	0.00	0.65	0.00	0.50	0.85	0.19	0.00	0.72	1.72	0.18	0.00	0.52	83.89	0.25	0.00	0.01	0.00	0.84	0.12	0.11	81.12	7.58	42.68	9.04
13 39 53	107751.01	1179.14	112414.18	720.30	20.30	0.35	43.42	7.70	0.00	1.62	0.00	0.62	0.00	0.30	5.77	2.38	0.00	0.65	0.00	0.49	0.66	0.19	0.00	0.74	1.87	0.16	0.00	0.52	83.73	0.25	0.00	0.01	0.00	0.70	0.14	0.11	80.25	7.59	43.42	9.32
13 40 53	108445.56	1193.62	113019.89	730.10	22.37	0.35	41.97	7.66	0.00	1.68	0.00	0.63	0.00	0.31	5.77	2.39	0.00	0.65	0.00	0.50	0.70	0.19	0.00	0.74	1.69	0.18	0.00	0.55	85.54	0.25	0.00	0.01	0.00	0.74	0.00	0.12	130.70	8.07	41.97	9.34
13 41 54	106260.45	1171.14	112022.68	716.99	20.35	0.35	44.26	7.55	0.00	1.65	0.00	0.60	0.00	0.30	5.42	2.30	0.00	0.61	0.00	0.49	0.68	0.19	0.00	0.71	1.65	0.15	0.00	0.52	84.53	0.24	0.00	0.01	0.00	0.75	0.13	0.11	83.40	7.32	44.26	9.20
13 42 53	105487.27	1137.86	111432.27	696.62	18.61	0.35	43.81	7.47	0.00	1.47	0.00	0.54	0.00	0.29	5.43	2.24	0.00	0.61	0.00	0.48	0.67	0.19	0.00	0.72	1.71	0.15	0.00	0.51	82.09	0.24	0.00	0.01	0.00	0.81	0.14	0.11	85.91	7.13	43.81	9.44
13 43 53	103261.57	1168.60	110333.03	728.25	15.59	0.32	44.64	7.48	0.00	1.65	0.00	0.56	0.00	0.31	4.58	2.26	0.00	0.62	0.00	0.50	0.59	0.19	0.00	0.74	1.62	0.15	0.00	0.52	83.24	0.25	0.00	0.01	0.00	0.70	0.12	0.10	81.38	6.89	44.64	9.13
13 44 53	105765.41	1174.09	111021.15	718.19	17.68	0.30	45.36	7.54	0.00	1.46	0.00	0.60	0.00	0.30	6.39	2.26	0.00	0.62	0.00	0.49	0.49	0.19	0.00	0.72	1.64	0.15	0.00	0.51	82.09	0.24	0.00	0.01	0.00	0.87	0.12	0.10	89.05	7.12	45.36	9.01
13 45 53	104511.59	1140.46	110406.86	697.93	18.17	0.32	46.19	7.48	0.00	1.73	0.00	0.53	0.00	0.29	5.78	2.28	0.00	0.62	0.00	0.48	0.69	0.19	0.00	0.73	1.69	0.15	0.00	0.51	83.17	0.25	0.00	0.01	0.00	0.82	0.12	0.10	83.20	7.05	46.19	9.21
13 46 54	102642.34	1124.01	109419.19	686.27	15.00	0.32	44.46	7.38	0.00	1.52	0.00	0.51	0.00	0.29	5.41	2.20	0.00	0.60	0.00	0.47	0.65	0.19	0.00	0.73	1.66	0.15	0.00	0.52	83.31	0.25	0.00	0.01	0.00	0.93	0.13	0.10	59.40	6.67	44.46	9.00
13 47 53	103026.83	1181.87	108790.13	724.81	14.84	0.30	45.92	7.56	0.00	1.50	0.00	0.51	0.00	0.31	5.50	2.26	0.00	0.62	0.00	0.50	0.74	0.19	0.00	0.71	1.61	0.15	0.00	0.50	82.15	0.24	0.00	0.01	0.00	0.92	0.10	0.10	57.51	6.78	45.92	9.06
13 48 53	102050.38	1157.54	109899.92	708.80	16.06	0.32	45.19	7.42	0.00	1.55	0.00	0.51	0.00	0.30	4.60	2.26	0.00	0.62	0.00	0.49	0.69	0.19	0.00	0.73	1.71	0.15	0.00	0.52	82.37	0.25	0.00	0.01	0.00	0.84	0.12	0.10	63.14	6.90	45.19	8.97
13 49 54	101833.36	1138.23	108848.60	697.02	15.54	0.33	45.02	7.48	0.00	1.46	0.00	0.52	0.00	0.29	5.29	2.30	0.00	0.63	0.00	0.48	0.69	0.19	0.00	0.73	1.69	0.15	0.00	0.50	83.12	0.25	0.00	0.01	0.00	0.78	0.13	0.10	61.90	6.80	45.02	8.95
13 50 53	102692.41	1144.39	109521.91	700.81	16.24	0.34	44.76	7.43	0.00	1.50	0.00	0.56	0.00	0.30	4.29	2.26	0.00	0.62	0.00	0.48	0.56	0.20	0.00	0.75	1.78	0.15	0.00	0.51	85.02	0.25	0.00	0.01	0.00	0.75	0.12	0.10	63.35	6.84	44.76	8.92
13 51 54	100813.04	1130.43	107706.16	691.89	16.43	0.31	44.61	7.24	0.00	1.36	0.00	0.49	0.00	0.29	4.90	2.25	0.00	0.61	0.00	0.48	0.50	0.22	0.00	0.83	1.78	0.15	0.00	0.52	84.68	0.26	0.00	0.01	0.00	0.83	0.13	0.10	63.22	6.54	44.61	8.80
13 52 53	97483.53	1134.77	106126.47	695.44	13.69	0.32	44.59	7.36	0.00	1.53	0.00	0.50	0.00	0.29	5.08	2.20	0.00	0.60	0.00	0.48	0.69	0.20	0.00	0.75	1.71	0.15	0.00	0.50	81.92	0.25	0.00	0.01	0.00	0.80	0.12	0.10	52.77	6.44	44.59	8.89
13 53 53	99490.43	1113.20	107174.16	681.78	14.68	0.33	48.88	7.38	0.00	1.46	0.00	0.54	0.00	0.29	5.14	2.15	0.00	0.59	0.00	0.47	0.62	0.18	0.00	0.69	1.71	0.14	0.00	0.49	81.84	0.23	0.00	0.01	0.00	0.98	0.12	0.10	56.95	6.68	48.88	8.84
13 54 54	101336.23	1121.05	107767.72	685.85	16.01	0.32	46.34	7.41	0.00	1.50	0.00	0.59	0.00	0.29	4.99	2.18	0.00	0.60	0.00	0.47	0.78	0.19	0.00	0.72	1.69	0.15	0.00	0.51	82.45	0.24	0.00	0.01	0.00	0.92	0.12	0.10	62.82	6.66	46.34	8.91
13 55 53	104572.32	1187.54	110675.77	726.96	18.21	0.34	47.72	7.26	0.00	1.56	0.00	0.56	0.00	0.31	3.65	2.30	0.00	0.63	0.00	0.50	0.70	0.19	0.00	0.74	1.82	0.15	0.00	0.51	83.54	0.25	0.00	0.01	0.00	0.93	0.14	0.10	71.99	6.80	47.72	8.82
13 56 54	99268.61	1091.72	106301.95	667.96	15.09	0.33	43.04	7.25	0.00	1.34	0.00	0.49	0.00	0.28	4.57	2.18	0.00	0.60	0.00	0.46	0.68	0.18	0.00	0.69	1.78	0.15	0.00	0.50	82.14	0.23	0.00	0.01	0.00	0.80	0.13	0.10	59.36	6.56	43.04	8.60
13 57 53	97870.89	1110.71	105609.36	679.94	14.52	0.35	46.41	7.40	0.00	1.65	0.00	0.58	0.00	0.29	5.58	2.22	0.00	0.61	0.00	0.47	0.80	0.18	0.00	0.70	1.73	0.15	0.00	0.50	81.25	0.24	0.00	0.01	0.00	0.81	0.12	0.10	55.41	6.67	46.41	9.05
13 58 54	99780.46	1135.48	104648.94	692.62	18.56	0.32	46.90	7.31	0.00	1.55	0.00	0.54	0.00	0.29	5.39	2.21	0.00	0.60	0.00	0.48	0.79	0.18	0.00	0.69	1.81	0.15	0.00	0.50	82.50	0.23	0.00	0.01	0.00	0.85	0.12	0.10	64.33	6.53	46.90	8.86
13 59 53	99106.58	1094.03	102554.32	666.56	15.37	0.30	43.02	7.32	0.00	1.37	0.00	0.51	0.00	0.28	5.16	2.30	0.00	0.63	0.00	0.43	0.02	0.18	0.00	0.69	1.79	0.15	0.00	0.49	81.38	0.24	0.00	0.01	0.00	0.85	0.12	0.10	59.21	6.51	43.02	8.69
14 00 56	85635.75	1129.52	107854.20	683.18	39.11	0.31	32.62	7.25	0.00	1.44	0.00	0.54	0.00	0.30	5.71	2.40	0.00	0.66	0.00	0.48	1.16	0.17	0.00	0.64	2.88	0.16	0.00	0.58	51.10	0.21	0.00	0.01	0.00	0.86	0.00	0.11	156.47	7.38	32.62	8.69
14 01 56	83818.21	1059.70	85978.82	640.45	44.00	0.31	32.78	7.20	0.00	1.38	0.00	0.55	0.00	0.28	5.48	2.38	0.00	0.65	0.00	0.45	1.09	0.16	0.00	0.61	2.90	0.16	0.00	0.59	50.05	0.20	0.00	0.01	0.00	0.83	0.00	0.12	180.10	7.77	32.78	8.58
14 02 56	83031.56	1030.00	84118.35	621.34	26.48	0.30	34.33	7.19	0.00	1.40	0.00	0.53	0.00	0.27	5.05	2.31	0.00	0.63	0.00	0.44	1.09	0.17	0.00	0.63	3.00	0.15	0.00	0.58	48.81	0.21	0.00	0.01	0.00	0.85	0.00	0.11	99.54	6.59	34.33	8.59
14 03 56	83136.87	1070.82	84234.64	645.96	35.26	0.29	33.26	7.08	0.00	1.36	0.00	0.46	0.00	0.28	6.42	2.38	0.00	0.65	0.00	0.46	1.17	0.16	0.00	0.62	3.00	0.16	0.00	0.67	49.18	0.20	0.00	0.01	0.00	0.89	0.00	0.11	160.67	7.20	33.26	8.41
14 04 56	84504.27	1026.26	84187.30	618.18	39.23	0.29	33.25	7.09	0.00	1.47	0.00	0.54	0.00	0.27	5.56	2.41	0.00	0.66	0.00	0.44	1.23	0.16	0.00	0.61	3.10	0.16	0.00	0.57	49.61	0.20	0.00	0.01	0.00	0.86	0.00	0.11	165.29	7.27	33.25	8.50
14 05 56	79263.09	1030.96	79441.																																					

All data in ppmv wet basis

Title Outlet then inlet run 1

Description Carey line run 1 outlet then inlet

Method title Ohio Line

Starting Date/Time Wed Sep 02 12 49 56 1998

Time	H2O	(s)H2O	CO2	(s)CO2	CO	(s)CO	NO	(s)NO	NO2	(s)NO2	N2O	(s)N2O	NH3	(s)NH3	CH4	(s)CH4	C2H6	(s)C2H6	C2H4	(s)C2H4	H2CO	(s)H2CO	ACTL	(s)ACTL	C4+	(s)C4+	CH3OH	(s)CH3OH	HCL	(s)HCL	SF6	(s)SF6	SO2	(s)SO2	COS	(s)COS	COH	(s)COH	NOX	(s)NOX
13:00:52	130875.59	1104.11	92455.08	642.00	12.98	0.32	36.33	6.81	0.00	1.68	0.00	0.57	0.66	0.28	3.46	2.18	0.00	0.60	0.00	0.45	0.00	0.11	0.00	0.42	0.83	0.15	0.00	0.46	4.48	0.12	0.00	0.01	0.00	5.52	0.13	0.10	49.49	6.79	36.33	8.49
13:01:52	129100.23	1109.26	90966.22	644.74	9.63	0.31	37.47	6.77	0.00	1.54	0.00	0.57	0.64	0.28	3.60	2.20	0.00	0.60	0.00	0.45	0.26	0.11	0.00	0.42	0.73	0.15	0.00	0.48	4.53	0.12	0.00	0.01	0.00	6.06	0.13	0.10	36.55	6.59	37.47	8.30
13:02:53	129962.77	1053.21	92251.33	612.77	14.18	0.29	39.45	6.72	0.00	1.65	0.00	0.55	0.56	0.26	4.77	2.17	0.87	0.59	0.00	0.43	0.16	0.11	0.00	0.44	0.81	0.15	0.00	0.48	4.51	0.13	0.00	0.01	0.00	5.84	0.13	0.10	54.37	6.65	39.45	8.37
13:03:53	129282.70	1083.23	91960.23	630.39	14.11	0.31	36.15	6.78	0.00	1.48	0.00	0.57	0.52	0.27	4.08	2.14	0.00	0.58	0.00	0.44	0.16	0.11	0.00	0.43	0.85	0.14	0.00	0.48	4.33	0.12	0.00	0.01	0.00	5.67	0.13	0.10	54.02	6.77	36.15	8.26
13:04:52	130696.07	1105.83	92278.27	642.95	11.76	0.32	38.06	6.64	0.00	1.48	0.00	0.54	0.61	0.28	4.94	2.18	0.63	0.60	0.00	0.45	0.19	0.10	0.00	0.40	0.80	0.15	0.00	0.47	4.53	0.11	0.00	0.01	0.00	5.72	0.12	0.10	43.28	6.57	38.06	8.13
13:05:53	131520.06	1145.26	92636.85	665.68	13.51	0.31	36.49	6.88	0.00	1.74	0.00	0.61	0.67	0.29	3.95	2.18	0.00	0.60	0.00	0.47	0.34	0.11	0.00	0.43	0.79	0.15	0.00	0.47	4.56	0.12	0.00	0.01	0.00	6.26	0.11	0.10	50.73	6.90	36.49	8.61
13:06:53	130102.95	1059.84	91742.34	616.09	9.35	0.30	37.66	6.77	0.00	1.75	0.00	0.61	0.56	0.27	3.89	2.22	0.68	0.61	0.00	0.43	0.14	0.11	0.00	0.44	0.77	0.15	0.00	0.48	4.42	0.13	0.00	0.01	0.00	5.82	0.11	0.10	33.74	6.62	37.66	8.52
13:07:53	131421.66	1100.62	93738.16	640.80	15.68	0.32	37.52	6.74	0.00	1.62	0.00	0.63	0.56	0.28	4.08	2.18	0.00	0.60	0.00	0.45	0.13	0.12	0.00	0.45	0.81	0.15	0.00	0.48	4.55	0.13	0.00	0.01	0.00	5.86	0.11	0.10	60.05	6.95	37.52	8.35
13:08:53	131014.59	1116.67	92823.23	649.55	14.97	0.28	35.63	6.85	0.00	1.82	0.00	0.65	0.49	0.28	3.58	2.19	0.00	0.60	0.00	0.46	0.00	0.11	0.00	0.43	0.85	0.15	0.00	0.48	4.50	0.12	0.00	0.01	0.00	5.72	0.13	0.10	57.79	6.89	35.63	8.68
13:09:53	130500.02	1139.70	93014.93	663.46	18.79	0.32	37.37	6.87	0.00	1.62	0.00	0.59	0.53	0.29	3.54	2.20	0.00	0.60	0.00	0.46	0.00	0.11	0.00	0.44	0.73	0.15	0.00	0.48	4.44	0.13	0.00	0.01	0.00	5.79	0.11	0.10	72.61	6.89	37.37	8.49
13:10:53	131141.75	1140.74	92715.39	663.37	16.62	0.32	35.90	7.33	0.00	1.92	0.00	0.67	0.63	0.29	4.22	2.57	0.00	0.70	0.00	0.47	0.23	0.12	0.00	0.44	0.77	0.17	0.00	0.48	4.35	0.13	0.00	0.01	0.00	5.79	0.11	0.11	62.37	7.60	35.90	9.24
13:11:54	130555.82	1072.81	92674.89	624.19	16.32	0.33	35.18	7.00	0.00	1.78	0.00	0.62	0.53	0.27	3.49	2.23	0.00	0.61	0.00	0.44	0.00	0.12	0.00	0.46	0.80	0.15	0.00	0.46	4.42	0.13	0.00	0.01	0.00	5.86	0.12	0.11	62.01	7.09	35.18	8.78
13:12:53	129490.95	1117.48	91577.73	649.84	12.64	0.28	35.79	6.87	0.00	1.56	0.00	0.58	0.52	0.28	3.41	2.14	0.00	0.59	0.00	0.46	0.25	0.11	0.00	0.42	0.76	0.14	0.00	0.48	4.29	0.12	0.00	0.01	0.00	5.90	0.10	0.10	48.43	6.71	35.79	8.52
13:13:53	130851.01	1081.38	92863.52	629.16	14.30	0.33	40.07	6.77	0.00	1.73	0.00	0.59	0.57	0.27	4.73	2.21	0.00	0.60	0.00	0.44	0.00	0.11	0.00	0.43	0.83	0.15	0.00	0.50	4.31	0.12	0.00	0.01	0.00	6.08	0.11	0.10	53.86	6.75	40.07	8.40
13:14:52	132445.33	1142.34	95281.23	665.87	32.09	0.38	36.72	6.90	0.00	1.67	0.00	0.57	0.42	0.29	4.29	2.19	0.00	0.60	0.00	0.47	0.25	0.11	0.00	0.43	0.78	0.15	0.00	0.50	4.34	0.12	0.00	0.01	0.00	6.11	0.00	0.12	127.86	7.84	36.72	8.57
13:15:53	132681.88	1131.36	95261.02	659.31	29.14	0.36	35.77	6.74	0.00	1.60	0.00	0.52	0.55	0.28	4.09	2.17	0.65	0.59	0.00	0.46	0.19	0.11	0.00	0.42	0.76	0.15	0.00	0.49	4.26	0.12	0.00	0.01	0.00	6.05	0.00	0.11	114.48	7.55	35.77	8.34
13:16:53	131435.03	1135.62	94335.68	661.72	21.77	0.35	36.99	6.77	0.00	1.52	0.00	0.58	0.40	0.28	3.45	2.14	0.00	0.58	0.00	0.46	0.00	0.11	0.00	0.43	0.81	0.14	0.49	0.49	4.34	0.12	0.00	0.01	0.00	5.99	0.11	0.11	82.22	7.17	36.99	8.29
13:17:53	131053.18	1130.54	94388.24	659.06	23.49	0.33	38.49	6.74	0.00	1.50	0.00	0.47	0.48	0.28	4.35	2.13	0.70	0.58	0.00	0.46	0.19	0.11	0.00	0.43	0.78	0.14	0.00	0.49	4.33	0.12	0.00	0.01	0.00	6.16	0.00	0.11	93.03	7.19	38.49	8.24
13:18:53	131866.23	1147.42	94914.50	668.87	44.16	0.36	35.03	6.67	0.00	1.61	0.00	0.49	0.48	0.29	4.92	2.16	0.00	0.59	0.00	0.47	0.24	0.12	0.00	0.46	0.79	0.14	0.00	0.50	4.30	0.13	0.00	0.01	0.00	6.13	0.00	0.12	176.66	8.21	35.03	8.28
13:19:52	131254.11	1148.87	95335.83	669.34	34.84	0.34	38.28	6.78	0.00	1.62	0.00	0.52	0.38	0.29	3.49	2.14	0.00	0.59	0.00	0.47	0.15	0.11	0.00	0.44	0.83	0.14	0.00	0.48	4.28	0.13	0.00	0.01	0.00	5.99	0.00	0.12	139.86	7.78	38.28	8.39
13:20:53	132879.25	1180.40	95700.75	676.85	54.15	0.37	33.85	6.74	0.00	1.71	0.00	0.56	0.38	0.29	4.88	2.18	0.00	0.60	0.00	0.47	0.17	0.11	0.00	0.43	0.76	0.15	0.00	0.49	4.23	0.12	0.00	0.01	0.00	6.18	0.00	0.13	221.79	8.97	33.85	8.44
13:21:53	131933.17	1144.43	94993.73	667.15	20.51	0.31	37.13	6.74	0.00	1.44	0.00	0.55	0.42	0.29	4.21	2.17	0.71	0.59	0.00	0.47	0.18	0.12	0.00	0.46	0.78	0.15	0.00	0.50	4.20	0.13	0.00	0.01	0.00	5.99	0.11	0.11	79.25	7.15	37.13	8.18
13:22:53	133186.19	1161.67	95988.60	677.33	33.91	0.35	35.66	6.76	0.00	1.64	0.00	0.56	0.39	0.29	4.07	2.16	0.76	0.59	0.00	0.47	0.17	0.12	0.00	0.45	0.76	0.14	0.00	0.50	4.42	0.13	0.00	0.01	0.00	6.16	0.00	0.12	135.31	7.83	35.66	8.40
13:23:53	133328.52	1133.04	96250.37	660.79	66.35	0.41	34.24	7.00	0.00	1.79	0.00	0.65	0.41	0.28	6.39	2.34	0.00	0.64	0.00	0.46	0.22	0.11	0.00	0.41	0.85	0.16	0.00	0.50	4.15	0.12	0.00	0.01	0.00	6.01	0.00	0.15	272.42	10.17	34.24	8.79
13:24:53	131029.32	1137.93	94167.29	663.17	23.72	0.32	33.63	6.77	0.00	1.68	0.00	0.61	0.49	0.29	3.87	2.19	0.00	0.60	0.00	0.46	0.22	0.12	0.00	0.44	0.78	0.15	0.54	0.49	4.22	0.13	0.00	0.01	0.00	5.76	0.00	0.11	92.32	7.22	33.63	8.45
14:24:54	129418.99	1169.10	93229.38	681.50	13.73	0.31	38.64	7.13	0.00	1.81	0.00	0.66	0.00	0.29	4.10	2.46	0.00	0.67	0.00	0.48	0.21	0.12	0.00	0.44	0.94	0.16	0.00	0.49	5.03	0.13	0.00	0.01	0.00	5.82	0.00	0.11	51.80	7.25	38.64	8.94
14:25:55	130393.56	1148.77	93534.68	669.30	14.19	0.34	39.50	7.02	0.00	1.73	0.00	0.68	0.00	0.29	3.27	2.44	0.00	0.67	0.00	0.47	0.00	0.12	0.00	0.47	0.91	0.16	0.00	0.49	5.16	0.14	0.00	0.01	0.00	5.77	0.12	0.11	53.41	7.09	39.50	8.74
14:26:54	130026.68	1156.04	93622.12	673.86	13.44	0.33	37.81	6.95	0.00	1.57	0.00	0.62	0.00	0.29	3.26	2.47	0.00	0.68	0.00	0.47	0.17	0.11	0.00	0.43	0.91	0.17	0.00	0.50	4.90	0.13	0.00	0.01	0.00	5.70	0.12	0.10	49.00	6.97	37.81	8.53
14:27:55	128283.81	1134.68	91595.63	660.63	9.39	0.30	39.87	7.00	0.00	1.89	0.00	0.61	0.00	0.29	3.15	2.40	0.00	0.65	0.00	0.46	0.23	0.12	0.00	0.46	0.89	0.16	0.00	0.48	4.85	0.13	0.00	0.01	0.00	5.79	0.12	0.10	33.20	6.95	39.87	8.89
14:28:55	129512.75	1110.22	93756.54	647.60	12.78	0.31	42.73	6.85	0.00	1.64	0.00	0.72	0.32	0.28	3.08	2.28	0.00	0.62	0.00	0.45	0.13	0.11	0.00	0.43	0.88	0.15	0.00	0.50	4.72	0.12	0.0									

APPENDIX D
FTIR FIELD DATA SHEETS

Switch
@ Reading 35 ✓
70 ✓
105 ✓
140 ✓
175 ✓
210 ✓
245 ✓

FTIR Temperature Readout Sheet

Facility	Carey National Uno
Stack ID	Scrubber
Date	Sept 2, 1998
Run Number	1

Recorded By		Temperature (deg. F)														
Channel	Description	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time	Time
1	Inlet Stack	360	368	368	365	360	349	363	376	371	365	369	376	369	370	371
2	Outlet Stack	104	105	106	106	106	106	106	108	107	107	108	108	108	108	109
3	Inlet Probe	253	263	347	343	348	257	344	347	345	342	345	344	253	344	347
4	Outlet Probe	280	338	334	325	334	336	337	339	336	338	335	334	340	341	338
5	Inlet Filter	169	173	337	345	347	170	328	347	345	316	345	186	159	345	347
6	Outlet Filter	248	320	332	332	335	336	338	340	337	341	340	334	168340	341	339
7	Inlet HT	264	264	264	263	262	264	266	271	275	278	281	279	340	275	277
8	Outlet HT	322	322	323	323	323	324	323	324	325	324	325	326	326	329	331
9	Inlet Pump	263	256	258	248	240	240	246	239	262	255	266	262	262	265	246
10	Outlet Pump	296	291	269	268	278	275	291	292	270	272	295	279	277	293	286
11	FTIR Pump	276	260	276	260	267	277	261	262	267	263	275	264	275	270	278
12	Pump Box	101	101	103	102	102	104	105	105	105	108	111	111	108	107	108
13																
14	FTIR Jumper	344	344	344	344	347	345	345	345	345	345	346	345	345	345	346
15	Pump Jumper	307	308	313	315	317	314	312	317	318	313	314	317	311	313	312
16	Hot Box	364	364	364	364	364	363	364	365	364	363	364	364	364	364	364
17	Hot Box	340	340	341	341	340	340	340	341	340	340	340	340	340	340	340
18																
19	Electronics Box	87	88	87	88	90	88	89	89	88	88	89	89	88	87	87
20																

Started outlet again
because inlet flow
was too low. Started
last inlet run after
35 outlet readings on Run 2 - (Still run 1)

FTIR Temperature Readout Sheet

Facility	Carey National Mine
Stack ID	Scrubber
Date	9-2-98
Run Number	Conditioned Run 1

[illegible]

APPENDIX E
PRE-TEST CALCULATIONS

Below are the results of the Method 320 pre-test calculations for this test program. The calculations are organized by appendix as found in the FTIR Protocol. These calculations were originally taken from the Secondary Aluminum HCl program from late 1997.

Appendix B

Potential Interferant Calculations:

These calculations determine potential **spectral** interferants for the analytes of interest (i.e., HCl). The results for HCl are given in the table below. The analysis region for HCl is not given since it is considered proprietary information.

TABLE 1. INTERFERANT CALCULATIONS

Analyte	Concentration	Band area	IAI/AAI	Average absorbance
HCl (target)	0.1 ppmv	0.0005436	-	0.00000322
H₂O (potential interferant)	20%	0.2213	407	0.00131
CO ₂ (potential interferant)	20%	0.000002	0.0036	
H ₂ CO (potential interferant)	1 ppmv	0.0002100	0.386	
CH₄ (potential interferant)	20 ppmv	0.0105	19.3	0.00006213
			AVT	0.00137

Note: compounds in bold are known interferants. AVT is computed from target and known interferants.

Known interferant criteria is IAI/AAI > 0.5

From the Table, two potential interferants are identified: H₂O and CH₄.

Appendix C

Noise Level

This calculation determines instrumental noise level in the spectral analysis region for HCl. For a 1 minute integration time, the RMS noise is found to be 0.00022 (absorbance units) in the HCl spectral analysis region by the procedure given in Appendix G.

Appendix D

Estimating Minimum Concentration Measurement Uncertainties (MAU)

The result for HCl is:

MAU (HCl) = 0.4 ppmv.

This value is computed using the formula given in Appendix D. However, this value is derived using band area calculations. The FTIR spectral data in this field study are analyzed by classical least squares (CLS), not band areas. CLS derived minimum measurement uncertainties for HCl are on the order of 0.1-0.2 ppmv for this test program.

Appendix E

Determining Fractional Reproducibility Uncertainties (FRU)

This calculation estimates the uncertainty in analysis, using band areas, of two sequentially measured CTS spectra collected immediately before and after the HCl reference spectrum. The calculation is performed in the analysis region used for HCl. The result is:

$$\text{FRU (HCl region)} = 0.093.$$

The corresponding value using CLS is somewhat lower. For most analytes of interest, FRU usually falls between 0.001 and 0.04 using CLS.

Appendix F

Determining Fractional Calibration Uncertainties (FCU)

This section determines the fractional calibration uncertainties when analyzing each reference spectrum. These results will be applied to the compounds analyzed in the HCl analysis region. The table below gives the results.

TABLE 2. FCU DETERMINATION

Analyte	ASC (ppm)	ISC (H ₂ O)	ISC (HCl)	ISC (CH ₄)	FCU	AU
H ₂ O	113000	115000	0.000	0.000	-1.7%	-
HCl	253	-22.5	254	0.000	-0.4%	30%
CH ₄	491	-23.0	0.000	493	-0.2%	-

Appendix G

Measuring Noise Levels

The result of this calculation is given under the Appendix C heading.

Appendix H

Determining Sample Absorption Pathlength (Ls) and Fractional Analytical Uncertainty

Since the HCl reference spectrum used in this program were measured at the same pathlength to be used during testing, these calculations are not required.

APPENDIX F

POST-TEST CALCULATIONS

Below are the results of the Method 320 post-test calculations for this test program. The calculations are organized by appendix as found in the FTIR Protocol. Since classical-least-squares (CLS) is used for analysis, the CLS-equivalent calculations are used, since in some cases, the FMU values using band-areas can differ as much as an order of magnitude compared to CLS-derived results.

Appendix I

Determining Fractional Model Uncertainties:

These calculations determine the fractional error in the analysis for the analytes of interest (i.e., HCl). The results for HCl are given in the table below for 1 spectrum selected from the inlet and outlet test. In order to achieve results that are consistent with the CLS analysis approach, the CLS equivalent of the calculation was performed. This is simply the reported analysis error divided by the HCl concentration.

TABLE 1. FMU CALCULATION FOR HCL - CAREY

Spectral File Name	Inlet/Outlet	Error (ppm)	Concentration (ppm)	FMU
RN010011.spa	Outlet	0.12	4.5	0.027
RN010046.spa	Inlet	0.25	63.0	0.0039

Error is 95% confidence interval reported by CLS software

Appendix J

Overall Concentration Uncertainty

The CLS equivalent of overall concentration uncertainty is simply the error reported by the CLS software. The results for this test program are found in Table 1, above.

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WASHINGTON, D.C. 20460

TECHNICAL REPORT DATA*(Please read Instructions on reverse before completing)*

1. REPORT NO. EPA- 454/R-00-009	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Final Report of Lime Manufacturing Industry Fourier Transform Infrared Spectroscopy National Lime and Stone Company, Carey Ohio		5. REPORT DATE May 2000
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) EMAD		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Environmental Protection Agency Office of Air Quality Planning and Standards Research Triangle Park, NC 27711		10. PROGRAM ELEMENT NO.
		11. CONTRACT/GRANT NO. 68-D7-0001
12. SPONSORING AGENCY NAME AND ADDRESS Director Office of Air Quality Planning and Standards Office of Air and Radiation U.S. Environmental Protection Agency Research Triangle Park, NC 27711		13. TYPE OF REPORT AND PERIOD COVERED Final Report
		14. SPONSORING AGENCY CODE EPA/200/04
15. SUPPLEMENTARY NOTES		
16. ABSTRACT <p>The United States Environmental protection Agency is investigating the lime manufacturing industry source category to identify and quantify emissions of hazardous air pollutants (HAPs) from rotary kilns. The primary objective of this test program was to obtain data on controlled and uncontrolled emissions of hydrogen chloride (HCL) and gather screening data on other hazardous air pollutants from lime production plants. EPA test Method 320 was used to collect the emission data.</p>		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Hydrogen Chloride (HCL) Hazardous Air Pollutants	Air Pollution control Wet Scrubber	
18. DISTRIBUTION STATEMENT Release Unlimited	19. SECURITY CLASS (Report) Unclassified	21. NO. OF PAGES 104
	20. SECURITY CLASS (Page) Unclassified	22. PRICE