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**Air**

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# Lime Manufacturing Industry Fourier Transform Infrared Spectroscopy

Chemical Lime  
Marble Falls, Texas



# **Lime Kiln Source Characterization**

## **Final Report**

**Contract No. 68-D7-0007**  
**Work Assignment 1-08**

Chemical Lime Company  
Marble Falls, Texas

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## Table of Contents

### Page

1.0	INTRODUCTION .....	1-1
1.1	Objectives .....	1-2
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 .....	2-2
2.2.1	Overview .....	2-2
2.2.2	FTIR Emission Results .....	2-3
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 .....	3-11
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

Figure 2-1.	HCl Inlet Run - Chemical Lime Company .....	2-5
Figure 2-2.	HCl Outlet Run - Chemical Lime Company .....	2-6
Figure 3-1.	FTIR Sampling and Measurement System .....	3-3

## List of Tables

### Page

Table 2-1.	Emissions Test Log .....	2-1
Table 2-2.	Baghouse FTIR HCl Results, ppmv .....	2-4
Table 2-3.	Other Species Detected by FTIR - Baghouse Inlet .....	2-7
Table 2-4.	Other Species Detected by FTIR - Baghouse Outlet .....	2-8
Table 3-1.	Typical FTIR Operating Parameters .....	3-7
Table 3-2.	Compounds for Which Reference FTIR Spectra Are Available in the ERG Spectral Library .....	3-10
Table 4-1.	HCl QC Pre-test Spike Results - Chemical Lime Company .....	4-3
Table 4-2.	HCl QC Post-test Spike Results - Chemical Lime Company .....	4-4
Table 4-3.	Gas Standard Analysis Results .....	4-5

## 1.0 INTRODUCTION

The purpose of this testing program is to obtain uncontrolled and controlled hydrogen chloride (HCl) and speciated hydrocarbon Hazardous Air Pollutants (HAPs) emissions data 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. 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.

FTIR source testing was conducted for the following purposes:

- Quantify HCl emission levels; and
- Gather screening (i.e., qualitative) data on other HAP emissions.

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

## **1.1 Objectives**

The objective of the FTIR testing of the lime facility was to perform quantitative measurement of hydrogen chloride (HCl) and screening of other HAPs detectable by FTIR, using EPA Draft Method 320.

## **1.2 Brief Site Discussion**

Testing was conducted at the Chemical Lime Company located in Marble Falls, Texas. Testing was performed on the inlet and outlet of a fabric-filter baghouse on Kiln #1, a double-shaft vertical 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 Chemical Lime Company, located in Marble Falls, Texas. Included in this section are summaries of the test matrix, test schedule, and authorized deviations from the test plan. Additional detail on these topics are provided in the sections that follow.

### **1.3.1 Test Matrix**

The complete sampling and analytical matrix that was performed is 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 also, in a screening capacity, to measure other HAPs that can be detected by FTIR.

FTIR measurements were conducted in two sets:

- Unconditioned; and
- Conditioned.

Unconditioned sampling was conducted during the extent 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 one-hour period to screen for aromatic species such as benzene, toluene, etc. At this particular site, the conditioned run was conducted prior to the unconditioned run.

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 90 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 data truly representing the location being tested in that set.

### **1.3.2 Test Schedule**

The test schedule for EPA Method 23 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 24 minute intervals at the outlet and inlet, in order to synchronize with the process cycle time and GFC-IR measurements performed by APCC.
- The EPA Work Assignment Manager authorized one hour total sample collection of the conditioned samples, ½ hour each on inlet and outlet. If detection of other HAPs was determined, then the run would extend to the full two hours, as originally planned. In this case, no additional HAPs were detected in the conditioned samples.

## 1.4 Test Report

This final report, presenting all data collected and the results of the analyses, has been prepared in four sections, and six appendices 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 measurements 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 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 raw FTIR concentration data are contained in the appendices.

Seven 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 pre-test 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 Chemical Lime Company in Marble Falls, Texas on June 25, 1998. Results for the extractive FTIR test conducted for HCl and screening for selected HAPs are provided in this section. Other (non-HAP) species which were detected are also reported. Testing was performed at the inlet and outlet of the baghouse.

### 2.1 Emissions Test Log

ERG performed extractive FTIR measurements for HCl and other HAPs. Table 2-1 presents the emissions test log which 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
6/25/98	Baghouse (inlet/outlet)	Spike 1	FTIR HCl Spike (inlet/outlet)	09:53 - 11:53
6/25/98	Baghouse (inlet/outlet)	Run 1	FTIR (Unconditioned) FTIR (Conditioned)	15:18 - 18:54 12:25 - 13:45
6/25/98	Baghouse (inlet/outlet)	Spike 2	FTIR HCl Spike (inlet/outlet)	19:32 - 20:19

## **2.2 FTIR Results**

### **2.2.1 Overview**

FTIR data for HCl and other species were collected at the inlet and outlet of the baghouse. 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 24 minutes. Inlet and outlet samples were drawn on a continuous basis; only the FTIR sample analysis was alternated between inlet and outlet. The first five data points from each 24 minute inlet/outlet measurement period were discarded to eliminate data for samples containing both inlet and outlet sample gas. Five data points correspond to the measured 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 76 and 95 1-minute average data points for both inlet and outlet measurements, after discarding the first five data points per set. 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 Chemical Lime Company in Marble Falls, Texas. Both unconditioned samples and conditioned samples were analyzed. Conditioned samples were generated by passing the raw sample gas through a sample conditioning system (See Section 3.1.1 for details). Conditioned samples extracted from the baghouse were measured prior to unconditioned sample extraction for one hour. Five minute average data points were generated by analysis of the composite spectrum consisting of an average of 215 FTIR spectra collected over the 5 minute time period. These results are reported in Section 2.2.2.2.

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

No attempt was made at correcting measurement data to a standard level of oxygen (e.g., 15%). Oxygen data were not collected by ERG.

## **2.2.2 FTIR Emission Results**

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

### **2.2.2.1 FTIR HCl Test Results**

The estimated FTIR HCl detection limit for this study was in the range of 0.18 to 0.19 ppmv. Half of 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. Appendix C contains the raw 1-minute FTIR concentration data for all monitored species. All HCl emission tests were collected during the unconditioned runs.

**Baghouse Outlet/Inlet HCl Results**—Table 2-2 gives a summary of the baghouse outlet/inlet FTIR HCl results. The measured HCl removal efficiency due to the baghouse was 39.98 percent. Figures 2-1 and 2-2 show a real-time graph for the inlet and outlet runs, respectively. Due to the splitting of FTIR analysis time between inlet and outlet, the time axis in the graphs is not continuous.

### **2.2.2.2 Other Species Detected by FTIR**

Other species were detected during the unconditioned FTIR test runs. The compounds were collected concurrently during the collection of the HCl test results. Results given below are organized by location. No additional HAP species were detected during the conditioned run. Appendix C contains a listing of 1-minute average values for all monitored analytes.

**Table 2-2. Baghouse FTIR HCl Results, ppmv**

	Run 1	
Date	6/25/98	
Time	15:18 - 18:54	
Location	Inlet	Outlet
Average	38.72	23.24
SD	13.13	8.46
Maximum	62.53	51.19
Minimum	16.79	13.24
NDP	76	95
RE	39.98	

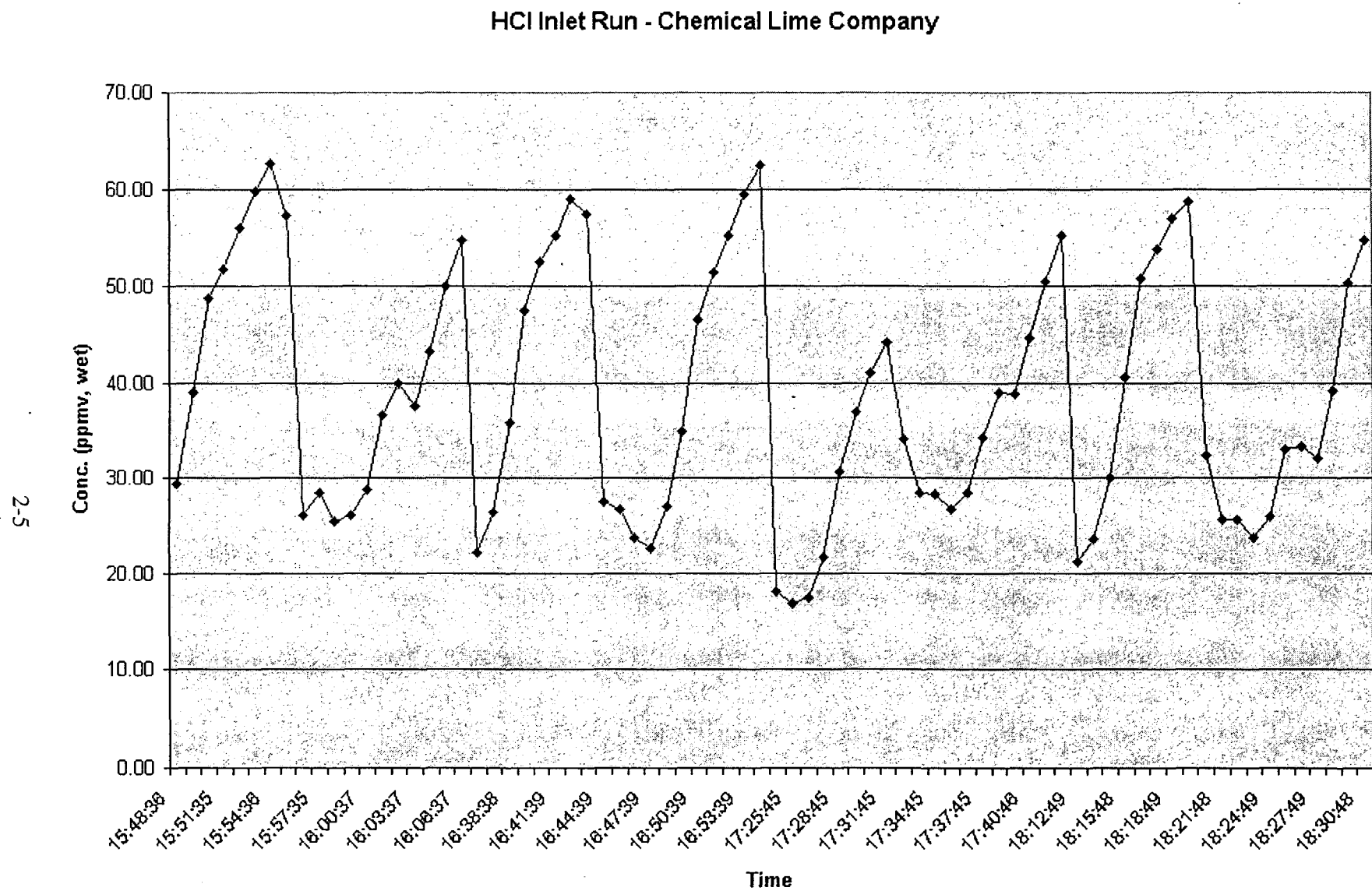
**SD** - Standard Deviation

**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 are presented in Appendix C.

**Baghouse Outlet/Inlet for Other Species Results**—Table 2-3 and 2-4 respectively gives the summary of the baghouse for the inlet and outlet FTIR results for other species found during the standard Draft Method 320 extractive sampling and analysis.



**Figure 2-1. HCl Inlet Run - Chemical Lime Company**

# HCI Outlet Run - Chemical Lime Company

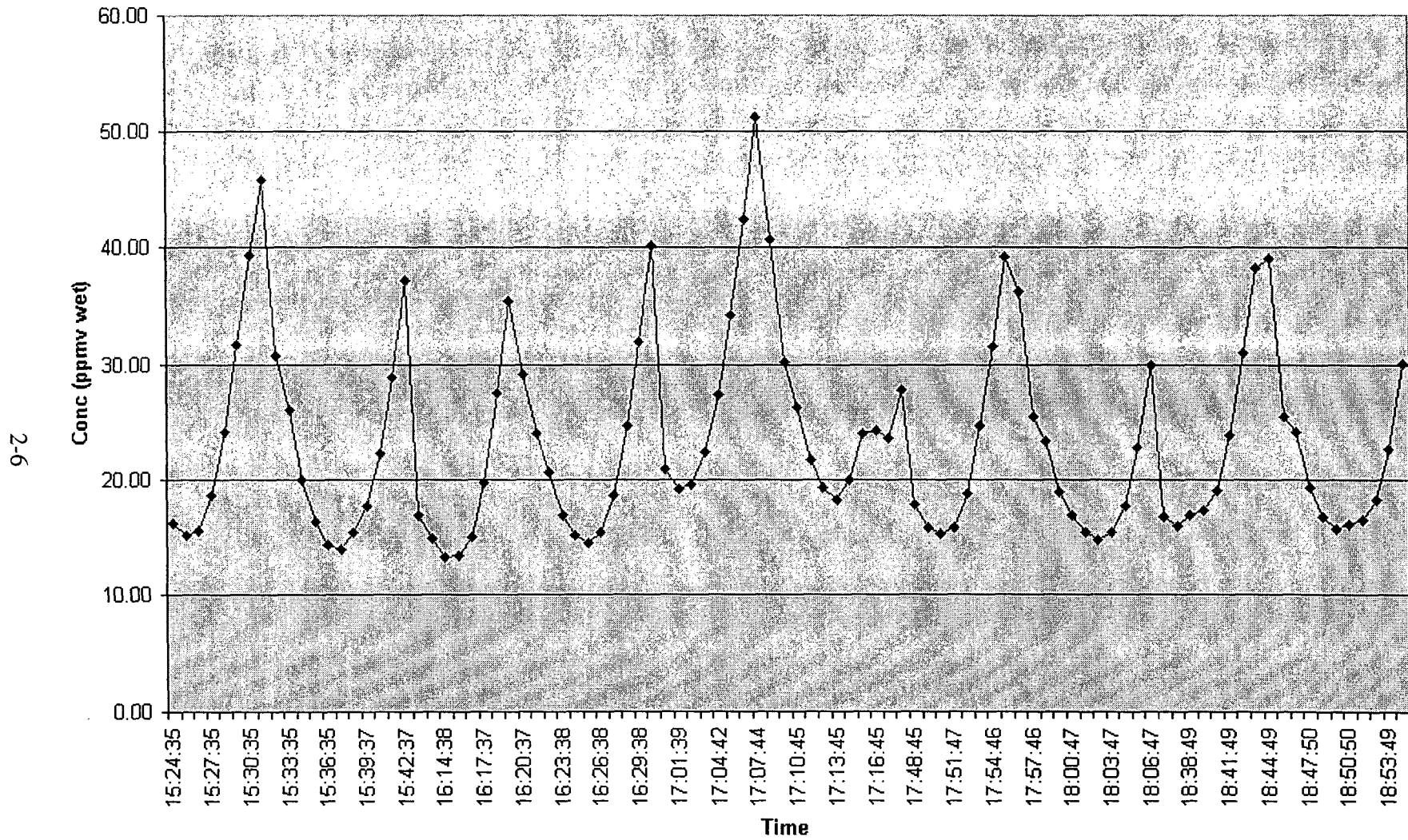


Figure 2-2. HCl Outlet Run - Chemical Lime Company

**Table 2-3. Other Species Detected by FTIR - Baghouse Inlet**

All values are ppmv, except CO<sub>2</sub> and H<sub>2</sub>O in percent

<b>Parameter</b>	<b>CO<sub>2</sub></b>	<b>CO</b>	<b>NO</b>	<b>CH<sub>4</sub></b>	<b>H<sub>2</sub>O</b>
<b>U/C</b>	<b>U</b>	<b>U</b>	<b>U</b>	<b>U</b>	<b>U</b>
<b>Average</b>	17.8	15.1	26.4	24.7	16.2
<b>Std. Dev.</b>	1.75	4.55	3.82	52.9	2.71
<b>Max.</b>	20.1	37.3	31.1	237.3	18.1
<b>Min.</b>	9.96	11.5	10.3	< 3.5	5.63
<b>NDP</b>	76	76	76	76	76
<b>EDL</b>	0.035	0.28	6.5	3.5	0.072

U/C - Unconditioned (U) or Conditioned (C) Sample

NDP - Number of data points.

EDL - Estimated detection limit for spectral region used for analysis

Std. Dev. = Standard Deviation

Max. = Maximum

Min. = Minimum

**Table 2-4. Other Species Detected by FTIR - Baghouse Outlet**

All values are ppmv, except CO<sub>2</sub> and H<sub>2</sub>O in percent

<b>Parameter</b>	<b>CO<sub>2</sub></b>	<b>CO</b>	<b>NO</b>	<b>CH<sub>4</sub></b>	<b>H<sub>2</sub>O</b>
<b>U/C</b>	<b>U</b>	<b>U</b>	<b>U</b>	<b>U</b>	<b>U</b>
<b>Average</b>	17.5	15.7	25.7	26.6	15.9
<b>Std. Dev.</b>	1.34	4.89	2.76	57.6	2.49
<b>Max.</b>	18.4	39.7	28.6	256	17.6
<b>Min.</b>	10.9	12.2	12.8	< 3.9	6.81
<b>NDP</b>	95	95	95	95	95
<b>EDL</b>	0.038	0.30	7.0	3.9	0.081

U/C - Unconditioned (U) or Conditioned (C) Sample

NDP - Number of data points

EDL - Estimated detection limit for spectral region used for analysis

Std. Dev. = Standard Deviation

Max. = Maximum

Min. = Minimum



### **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 Draft EPA 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 Fourier Transform Infrared Spectroscopy (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<sup>®</sup> sample line;
- Teflon<sup>®</sup> 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 which 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 are made 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 which is mounted on the stack and maintained at a 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, the sample gas is transported to the FTIR spectrometer by a primary heat-traced PTFE sample line 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 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.

FTIR spectrometer sample gas is taken from the distribution manifold by a secondary heated-head PTFE diaphragm sample pump 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 is made of nickel-plated aluminum, with gold-plated glass substrate mirrors and potassium chloride windows. Exhaust gas from the cell is vented to the atmosphere.

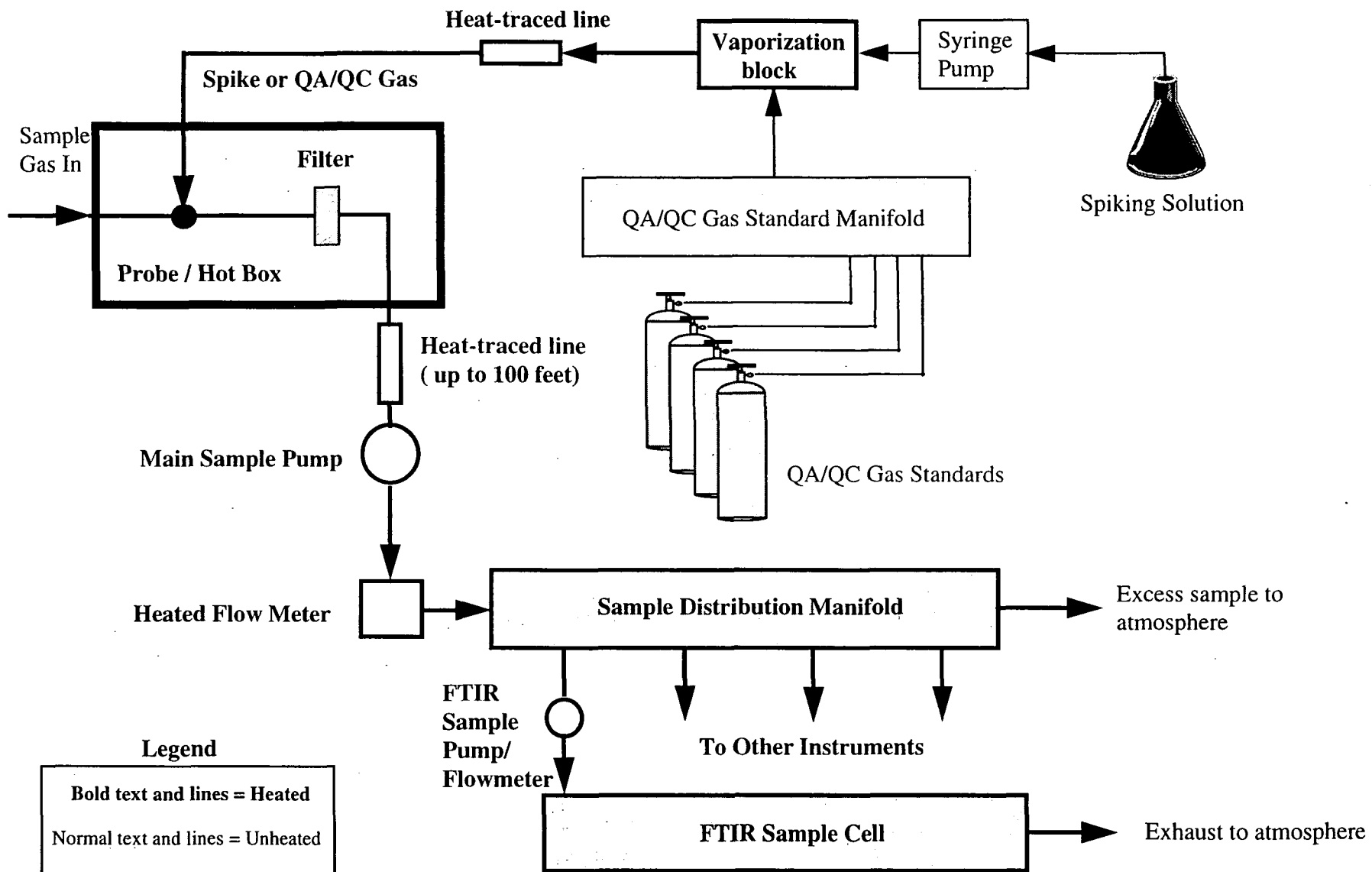


Figure 3-1. FTIR Sampling and Measurement System

Sample conditioning (when required) is achieved by passing raw sample 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 CO<sub>2</sub> 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  $\pm 6\%$  ( $\pm 10\%$  for HCl) of target value. The QC gases used for this program include:

- Halocarbon 22, 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 frequency calibration. Carbon monoxide is directly injected into the sample cell to measure photometric accuracy, validity of the non-linear correction algorithm and serve as a frequency (i.e., wavelength) calibration. Acceptable limits for CO standard analysis are  $\pm 6$  percent of certified concentration;
- Methane/nitric oxide/carbon dioxide mixture, used for overall system performance check (calibration transfer standard) (acceptance limits are  $\pm 6\%$  of the certified concentration); and
- Hydrogen chloride standard, analyzed to verify the instrumental response of HCl, a key target analyte (acceptance limits are  $\pm 10\%$  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 Method 320 gives a description of the dynamic spiking apparatus.

The FTIR spiking procedure used the following:

- Measured native stack gas until system equilibrates - took 2 measurements (i.e., 2-1 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., 2- 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 6 spiked/unspiked sample pairs. Spike recovery for 6 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% 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 1-1000 ppm). Some of these parameters are sample matrix dependent.

Sample flow rate was determined by the data averaging interval and FTIR spectrometer sample cell volume. A minimum of 3 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.

**Table 3-1. Typical FTIR Operating Parameters**

Parameter	Value
Spectral Range (cm <sup>-1</sup> )	400 - 4000
Spectral Resolution (cm <sup>-1</sup> )	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)

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 ensure that condensation of high-boiling point analytes on the cell optics was minimized.

FTIR sample 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 from 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 pre-determined time, corresponding to a "run". Typical runs were approximately 3 hours long, giving a minimum of 180 one-minute averaged points for each target analyte. The figure of 180 points were reduced by 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.

<b>Sampling Conditions</b>	<b>Sampling Time</b>	<b>Inlet</b>	<b>Outlet</b>
Unconditioned	Synchronized with dioxin sampling	5 minute cell purge and 19 minute sample collection	5 minute cell purge 19 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 three-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 Draft EPA Method 320. Acceptable spiking tests met acceptance for accuracy of within  $\pm 30$  percent. The acceptable instrument diagnostic and system response checked accuracy to be within  $\pm 6$  percent of target for all gas standards, and  $\pm 10\%$  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 do 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 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.

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

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

<sup>a</sup> Spectra were collected at a cell temperature of 185° C.

### **3.1.4 FTIR Method Data Review Procedures**

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

#### **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 which was 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 which were not detected or detected at low levels:
  - a) estimate detection limits from validated data;
  - b) 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 will be 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 sample 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 Method 320 criteria (i.e., accuracy of within  $\pm 30$  percent) or a statistical equivalent when less than 12 spiked/unspiked pairs are collected. Draft EPA Method 320 instructs the user to determine the percent spike recovery of 3 pairs of spiked/unspiked samples. The Draft EPA 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  $\pm 6$  percent of target ( $\pm 10$  percent for HCl standards). Acceptable system response check precision was 6 percent RSD.

## **4.0 QUALITY ASSURANCE/QUALITY CONTROL**

Specific Quality Assurance/Quality Control (QA/QC) procedures were strictly followed during this test program to ensure the production of useful and valid data throughout the course of 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 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 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  $\pm 6$  percent of target for all gas standards except HCl. The accuracy for the HCl standard should be within  $\pm 10$  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; and
- Direct measurement of a CH<sub>4</sub>, NO, and CO<sub>2</sub> standard.

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. Tables 4-1 and 4-2 summarize the dynamic spiking results. 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, H<sub>2</sub>, and a CH<sub>4</sub>, NO<sub>2</sub> and CO<sub>2</sub> mixture was conducted before and after daily testing activities. Acceptance criteria are normally  $\pm 6$  percent of target, using EPA protocol gases. However, since the HCl standard was obtained at a  $\pm 5$  percent analytical tolerance, the acceptance criteria was set at  $\pm 10$  percent. FTIR NO<sub>x</sub> is measured as NO + NO<sub>2</sub>. Examination of Table 4-3 shows that all QC checks met the above criteria.

**Table 4-1. HCl QC Pre-test Spike Results - Chemical Lime Company**

Outlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	10.16	41.26	32.00	22.41		0.450	0.089
2	16.26	49.40	34.55	21.91		0.440	0.087
3	10.44	33.68	24.08	20.42		0.410	0.081
4	14.28	41.83	28.70	20.42		0.410	0.081
5	7.68	25.21	18.10	18.93		0.380	0.075
6	9.30	29.35	20.75	18.93		0.380	0.075
Average	11.35	36.79	26.36	20.50	128.55	0.412	0.081
Inlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio
1	44.92	67.72	26.69	21.91		0.440	0.087
2	48.20	68.28	24.25	21.91		0.440	0.087
3	47.41	60.27	16.78	20.92		0.420	0.083
4	50.27	69.44	23.23	20.42		0.410	0.081
5	40.32	59.97	22.90	20.42		0.410	0.081
6	46.91	58.64	15.61	20.92		0.420	0.083
Average	46.34	64.05	21.58	21.08	102.34	0.423	0.083

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 SF<sub>6</sub> values are 253 ppmv and 5.08 ppmv, respectively).



**Table 4-2. HCl QC Post-test Spike Results - Chemical Lime Company**

<b>Outlet</b>							
<b>Spike Run Number</b>	<b>Lowest Unspiked Value (ppmv)</b>	<b>Spiked (ppmv)</b>	<b>Corrected Difference (ppmv)</b>	<b>Spike Level (ppmv)</b>	<b>% Recovery</b>	<b>SF6 Conc. (ppmv)</b>	<b>Dilution Ratio</b>
1	18.01	39.43	22.12	23.91		0.480	0.094
2	16.48	40.37	25.45	23.91		0.480	0.094
3	16.42	43.68	28.81	23.91		0.480	0.094
4	18.58	49.17	32.35	23.91		0.480	0.094
5	22.71	56.48	35.92	23.91		0.480	0.094
6	29.98	63.10	35.95	23.91		0.480	0.094
<b>Average</b>	<b>20.36</b>	<b>48.54</b>	<b>30.10</b>	<b>23.91</b>	<b>125.91</b>	<b>0.482</b>	<b>0.094</b>
<b>Inlet</b>							
<b>Spike Run Number</b>	<b>Lowest Unspiked Value (ppmv)</b>	<b>Spiked (ppmv)</b>	<b>Corrected Difference (ppmv)</b>	<b>Spike Level (ppmv)</b>	<b>% Recovery</b>	<b>SF6 Conc. (ppmv)</b>	<b>Dilution Ratio</b>
1	41.09	51.31	13.70	21.42		0.430	0.085
2	49.65	56.36	10.91	21.42		0.430	0.085
3	53.18	66.38	17.70	21.42		0.430	0.085
4	56.64	74.24	22.39	21.42		0.430	0.085
5	60.27	76.06	20.89	21.42		0.430	0.085
6	63.10	78.46	20.70	21.42		0.430	0.085
<b>Average</b>	<b>53.99</b>	<b>67.14</b>	<b>17.72</b>	<b>21.42</b>	<b>82.73</b>	<b>0.430</b>	<b>0.085</b>

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 SF<sub>6</sub> values are 253 ppmv and 5.08 ppmv, respectively).

**Table 4-3. Gas Standard Analysis Results**

Date	Time	Compound	True (ppm)*	Result (ppm)*	% Recovery	Comments
6/25/98	08:31 AM	HCl	253	249.8	98.7	
		CO	102.3	102.1	99.8	
		CH <sub>4</sub>	491	487.9	99.4	
		NO	503	502.7	99.9	
		CO <sub>2</sub>	4.99 %	4.95 %	99.2	
		H22		3.35 m		
6/25/98	08:40 PM	HCl	253	249.9	98.8	
		CO	102.3	101.7	99.4	
		CH <sub>4</sub>	491	489.6	99.7	
		NO	503	500.6	99.5	
		CO <sub>2</sub>	4.99 %	4.96 %	99.4	
		H22		3.36 m		

HCl Gas Standard Accuracy:  $\pm 5$  percent; Acceptance Criteria:  $\pm 10$  percent of target.

CO Gas Standard Accuracy:  $\pm 1$  percent; Acceptance Criteria:  $\pm 6$  percent of target.

CH<sub>4</sub>, NO<sub>2</sub>, and CO<sub>2</sub> Gas Standard Accuracy;  $\pm 1$  percent; Acceptance Criteria:  $\pm 6$  percent of target.

\* All compounds are recorded in ppm except for CO<sub>2</sub> 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

<b>Facility Name:</b> Chemline Lime Kilns	<b>DATE:</b> JUNE-25-1998
<b>Source Location (INLET or OUTLET)</b> INLET	<b>TIME:</b> 12:25:45
<b>Run Description</b> Run 01 Conditioned INLET	
<b>Reviewer:</b> MICHAEL BRYANT	<b>Date:</b> Dec-2-98

Checklist	Yes	No	N/A	Not det.*	Resolution
<b>A. QA/QC entries match references values</b>					
<i>(Check the following by comparing the printout of the QA/QC run to the original run information:</i>					
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. 7/7	✓				
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>B. Check that calculations are correct</b>					
1. No mathematical errors	✓				
2. No errors in the data macro	✓				
<b>C. Comments</b>					
1. All data is identical	✓				M. Bryant 12-2-98
2.					

\* Not able to determine

**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

<b>Facility Name:</b> Chemline Line Kiln	<b>DATE:</b> JUNE 25 - 98
<b>Source Location (INLET or OUTLET):</b> OUTLET	<b>TIME:</b> 12:25:49
<b>Run Description:</b> Run 01 Conditioned OUTLET	
<b>Reviewer:</b> Michael Bryant	<b>Date:</b> Dec 2-98

Checklist	Yes	No	N/A	Not det. *	Resolution
<b>A. QA/QC entries match references values</b>					
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. 7/7	✓				
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>B. Check that calculations are correct</b>					
1. No mathematical errors	✓				
2. No errors in the data macro	✓				
<b>C. Comments</b>					
1. All data is identical	✓				M. Bryant 12-2-98
2.					

\* Not able to determine

**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

<b>Facility Name:</b> Chemline Lime Kiln	<b>DATE:</b> JUNE 25-98
<b>Source Location (INLET or OUTLET)</b> INLET	<b>TIME:</b> 15:18:38 (START)
<b>Run Description</b> Run 2 Outlet/Inlet unconditioned	
<b>Reviewer:</b> Michael Bryant	<b>Date:</b> Dec-2-98

Checklist	Yes	No	N/A	Not det. *	Resolution
<b>A. QA/QC entries match references values</b>					
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. 76/76	✓				
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>B. Check that calculations are correct</b>					
1. No mathematical errors	✓				
2. No errors in the data macro	✓				
<b>C. Comments</b>					
1. Files Match QA/QC OK	✓				M. Bryant 12-2-98
2.					

\* Not able to determine

**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: <u>Chemline Line Kiln</u>	DATE: <u>JUNE 25, 1998</u>
Source Location (INLET or OUTLET) <u>OUTLET</u>	TIME: <u>15:18:38 (START)</u>
Run Description <u>Run 2 outlet/inlet</u> <u>unconditioned</u>	
Reviewer: <u>Michael Bryant</u>	Date: <u>Dec-2-98</u>

Checklist	Yes	No	N/A	Not det. *	Resolution
<b>A. QA/QC entries match references values</b>					
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.	95/95 ✓				
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>B. Check that calculations are correct</b>					
1. No mathematical errors	✓				
2. No errors in the data macro	✓				
<b>E. Comments</b>					
1. Files match QA/QC OK	✓				M.D. Bryant 12-2-98
2.					

\* Not able to determine

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

ANALYTICAL ACCURACY: + / - 5%

<u>COMPONENT</u>	<u>REQUESTED GAS CONC</u>	<u>ANALYSIS</u>
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

USA • United Kingdom • Germany • Japan

ISO 9002

**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 # :	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

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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 NeemeDATE: 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:   
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#: 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

**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 # :	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	CYLINDER # :	982153Y
ITEM# :	3	CYLINDER PRES:	2000 psig
CERTIFICATION DATE:	8/10/98	CYLINDER VALVE:	CGA 330
P.O.# :	9101008011-R132		
BLEND TYPE:	CERTIFIED		

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

DATE: \_\_\_\_\_

8/10/98

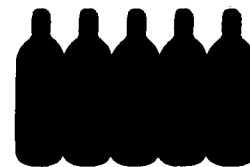
USA • United Kingdom • Germany • Japan

ISO 9002



# 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	102.1 ppm	102.3 ppm	+/- 1%
	8/26/97	102.6 ppm		

**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 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 # :	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

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## 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**

Outlet

ppmv wet  
outlet/min

Description: Chemtime time kin

Method title: Line Uncond

Starting Date/Time: Thu Jun 25 15:38:39 1998

Time	FName	H2O	(+)-H2O	CO2	(+)-CO2	CO	(+)-CO	NO	(+)-NO	NO2	(+)-NO2	N2O	(+)-N2O	NH3	(+)-NH3	CH4	(+)-CH4	C2H6	(+)-C2H6	C2H4	(+)-C2H4	H2CO	(+)-H2CO	ACTLD	(+)-ACTLD	C4+	(+)-C4+	CH3OH	(+)-CH3OH	HCL	(+)-HCL
15:24:35	r020007	183619.84	2032.88	175801.75	883.32	13.07	0.48	26.22	10.52	0.00	2.33	0.00	1.08	0.00	0.49	5.42	7.78	3.30	2.13	0.36	0.80	0.22	0.13	0.77	0.71	0.80	0.52	1.69	1.33	18.16	0.18
15:25:35	r020008	164553.98	2064.17	177294.84	896.16	13.37	0.50	25.90	10.70	0.21	2.18	0.00	1.17	0.00	0.50	3.44	7.96	3.18	2.18	0.36	0.82	0.15	0.13	1.34	0.70	0.79	0.53	1.73	1.34	15.12	0.19
15:26:35	r020009	168371.27	2099.06	179074.91	908.15	14.14	0.49	26.29	10.86	0.00	2.29	0.00	1.17	0.00	0.51	3.83	7.88	3.41	2.15	0.33	0.83	0.23	0.14	0.84	0.71	0.75	0.53	1.84	1.39	15.44	0.19
15:27:35	r020010	170600.28	2150.65	180183.00	928.60	14.69	0.51	26.83	11.04	0.00	2.14	0.00	1.12	0.00	0.52	3.91	7.92	3.14	2.16	0.41	0.85	0.24	0.15	1.02	0.78	0.84	0.53	1.94	1.41	18.56	0.21
15:28:38	r020011	173343.91	2161.15	181739.73	930.83	15.13	0.53	27.49	11.12	0.00	2.18	0.00	1.12	0.00	0.52	4.14	8.01	3.14	2.19	0.53	0.85	0.26	0.17	1.40	0.89	0.84	0.54	2.04	1.44	24.12	0.24
15:29:35	r020012	174200.65	2205.69	182510.23	949.29	15.53	0.53	26.06	11.17	0.18	2.36	0.00	1.06	0.00	0.53	4.21	7.96	2.98	2.18	0.27	0.86	0.18	0.20	1.12	1.06	0.84	0.53	2.07	1.46	31.65	0.26
15:30:35	r020013	174840.13	2230.75	183456.13	959.52	16.38	0.50	26.60	11.12	0.07	2.17	0.00	1.08	0.00	0.54	2.28	8.10	2.53	2.21	0.80	0.87	0.17	0.24	1.02	1.25	0.93	0.54	2.15	1.48	39.35	0.34
15:31:35	r020014	155678.38	1942.72	172398.63	850.34	21.07	0.50	25.73	10.35	0.00	2.24	0.00	1.11	0.00	0.48	38.36	7.87	4.45	2.15	1.08	0.77	0.33	0.26	1.77	1.38	0.96	0.53	2.02	1.30	45.89	0.37
15:32:35	r020015	168734.70	2004.00	182519.12	874.80	31.13	0.30	15.50	7.84	0.00	1.08	0.00	0.88	0.00	0.27	240.77	4.05	12.06	1.11	3.64	0.44	0.42	0.19	1.05	0.69	1.87	0.27	1.33	0.72	30.75	0.27
15:33:35	r020016	124502.04	2037.63	181022.47	918.82	14.67	0.44	26.91	10.54	0.00	2.00	0.00	1.03	0.00	0.51	102.65	5.95	6.75	1.63	0.49	0.84	0.13	0.17	1.05	0.68	1.31	0.40	2.15	1.43	26.00	0.24
15:34:35	r020017	157047.25	1925.68	174025.95	841.11	13.11	0.49	27.46	10.22	0.00	2.19	0.00	1.12	0.00	0.47	38.26	7.58	4.54	2.07	0.48	0.77	0.02	0.15	0.99	0.79	1.04	0.51	1.73	1.30	19.93	0.21
15:35:36	r020018	159901.83	1968.96	174647.63	857.60	12.76	0.52	26.89	10.40	0.00	2.33	0.00	1.23	0.00	0.48	15.51	7.87	3.79	2.15	0.37	0.78	0.14	0.13	1.17	0.68	0.84	0.53	1.81	1.31	16.34	0.18
15:36:35	r020019	161612.03	2031.42	176281.67	884.31	13.02	0.52	27.14	10.65	0.11	2.12	0.00	1.14	0.00	0.49	7.39	7.44	3.07	2.04	0.41	0.81	0.18	0.13	0.65	0.70	0.73	0.50	1.88	1.36	14.29	0.19
15:37:35	r020020	162998.38	2036.04	177547.25	885.20	13.41	0.50	27.56	10.78	0.00	2.32	0.00	1.20	0.00	0.50	4.22	7.71	3.06	2.11	0.31	0.81	0.22	0.13	1.19	0.68	0.70	0.52	1.92	1.36	13.93	0.18
15:38:36	r020021	166125.84	2060.70	176378.16	893.37	14.06	0.50	27.95	10.70	0.00	2.25	0.00	1.20	0.00	0.50	3.11	8.10	3.23	2.21	0.25	0.81	0.27	0.13	0.73	0.70	0.80	0.54	1.88	1.36	15.43	0.19
15:39:37	r020022	169618.59	2055.66	179334.68	888.37	14.70	0.52	28.00	10.99	0.09	2.21	0.00	1.10	0.00	0.50	3.68	7.81	3.00	2.14	0.52	0.81	0.19	0.14	0.83	0.73	0.84	0.52	1.91	1.39	17.66	0.20
15:40:36	r020023	172168.23	2148.41	180163.28	928.30	15.64	0.53	27.16	10.66	0.00	2.15	0.00	1.12	0.00	0.52	3.78	7.69	2.81	2.10	0.50	0.84	0.18	0.16	1.00	0.84	0.91	0.52	1.96	1.42	22.26	0.23
15:41:38	r020024	174335.75	2165.56	181021.30	931.90	16.30	0.54	27.48	11.11	0.00	2.10	0.00	1.09	0.00	0.52	2.43	7.97	2.93	2.18	0.61	0.85	0.24	0.18	0.70	0.96	0.83	0.53	1.97	1.44	28.92	0.26
15:42:37	r020025	174774.98	2145.30	181555.23	922.82	16.88	0.54	28.62	11.21	0.00	2.34	0.00	1.13	0.00	0.52	3.29	8.53	3.30	2.33	0.46	0.84	0.26	0.22	0.60	1.17	0.79	0.57	1.87	1.43	37.23	0.32
16:12:38	r020055	162360.31	2060.01	175498.97	870.04	13.01	0.50	25.23	10.55	0.06	2.17	0.00	1.13	0.00	0.49	8.91	7.64	3.11	2.09	0.32	0.79	0.22	0.14	1.06	0.72	0.85	0.51	1.78	1.32	16.92	0.20
16:13:37	r020056	165298.31	2006.88	177022.66	896.77	13.38	0.50	25.56	10.59	0.06	2.15	0.00	1.08	0.00	0.50	5.76	7.65	2.76	2.09	0.43	0.82	0.18	0.14	0.57	0.71	0.83	0.51	1.87	1.35	14.85	0.19
16:14:38	r020057	167620.13	2118.14	177858.67	917.03	13.99	0.50	25.74	10.77	0.10	2.23	0.00	1.07	0.00	0.51	4.05	7.71	2.86	2.11	0.41	0.83	0.21	0.12	1.04	0.65	0.86	0.52	1.83	1.38	13.24	0.18
16:15:37	r020058	170726.16	2151.76	179908.47	928.98	14.50	0.49	26.27	11.03	0.17	2.24	0.00	1.15	0.00	0.52	4.02	7.92	3.32	2.17	0.35	0.85	0.32	0.12	1.70	0.65	0.82	0.53	1.95	1.41	13.38	0.18
16:16:38	r020059	172724.53	2193.74	181231.28	945.39	15.14	0.53	26.35	11.12	0.11	2.28	0.00	1.13	0.00	0.53	4.11	8.15	3.34	2.23	0.46	0.88	0.25	0.13	1.01	0.69	0.91	0.55	1.93	1.43	15.01	0.19
16:17:37	r020060	174344.33	2202.50	182290.42	947.79	15.67	0.54	26.60	11.27	0.06	2.21	0.00	1.15	0.00	0.53	3.51	8.09	3.03	2.21	0.48	0.88	0.24	0.15	1.17	0.69	0.89	0.54	2.00	1.44	19.66	0.21
16:18:37	r020061	174256.94	2207.63	182694.61	950.16	16.42	0.51	26.55	11.17	0.00	2.22	0.00	1.11	0.00	0.53	4.21	8.26	3.13	2.28	0.32	0.88	0.18	0.18	1.49	0.95	0.92	0.55	2.10	1.43	27.57	0.26
16:19:38	r020062	167172.97	2130.78	179353.80	922.87	13.77	0.53	25.58	10.91	0.00	2.26	0.00	1.13	0.00	0.52	4.79	7.99	2.73	2.18	0.48	0.84	0.36	0.22	1.58	1.16	0.86	0.54	2.15	1.40	35.40	0.31
16:20:37	r020063	175471.93	2088.99	180330.31	933.76	13.93	0.52	26.80	11.00	0.00	2.22	0.00	1.09	0.00	0.51	265.60	4.39	13.34	1.20	0.56	0.35	0.73	0.18	1.44	0.96	2.01	0.29	0.87	0.53	29.10	0.26
16:21:38	r020064	180426.76	2133.61	183858.23	925.98	15.77	0.41	26.12	10.55	0.00	1.64	0.00	0.97	0.00	0.52	123.60	5.29	7.20	1.45	0.71	0.84	0.10	0.16	1.42	0.86	1.48	0.35	2.18	1.45	24.02	0.23
16:22:37	r020065	154868.09	1957.35	173918.94	857.38	13.10	0.45	26.24	10.33	0.00	2.16	0.00	1.02	0.00	0.48	46.96	7.27	4.72	1.99	0.53	0.78	0.05	0.15	1.50	0.79	1.06	0.49	1.72	1.32	20.60	0.21
16:23:38	r020066	159389.44	1970.17	174531.09	858.40	12.89	0.49	25.51	10.39	0.08	2.24	0.00	1.09	0.00	0.46	17.25	7.32	3.42	2.00	0.41	0.78	0.10	0.14	1.58	0.75	0.91	0.49	1.79	1.33	16.81	0.20
16:24:37	r020067	161431.27	2062.13	178168.92	897.83	12.99	0.51	25.60	10.55	0.00	2.20	0.00	1.12	0.00	0.50	7.84	7.45	2.99	2.04	0.33	0.82	0.13	0.13	1.11	0.68	0.81	0.50	1.71	1.34	15.14	0.18
16:25:37	r020068	164482.42	2054.68	178849.83	892.09	13.52	0.50	26.20	10.65	0.00	2.20	0.00	1.14	0.00	0.50	5.04	7.76	3.21	2.12	0.38	0.81	0.09	0.12	0.99	0.65	0.86	0.52	1.88	1.36	14.38	0.18
16:26:38	r020069	167722.56	2104.97	179061.83	911.24	14.07	0.53	26.37	10.87	0.04	2.28	0.00	1.18	0.00	0.51	3.41	7.90	2.98	2.16	0.38	0.83	0.08	0.13	1.07	0.70	0.83	0.53	1.72	1.38	15.41	0.19
16:27:37	r020070	170229.89	2120.77	179894.33	916.00	14.85	0.52	26.74	10.91	0.00	2.35	0.00	1.15	0.00	0.51	3.25	8.08	3.01	2.21	0.43	0.83	0.25	0.14	1.19	0.75	0.89	0.54	2.06	1.39	18.61	0.20
16:28:38	r020071	172423.41	2171.95	180678.56	938.25	15.42	0.51	26.67	10.96	0.13	2.36	0.00	1.11	0.00	0.52	2.76	8.05	2.84	2.20	0.51	0.85	0.24	0.17	1.21	0.89	0.98	0.54	2.00	1.42	24.66	0.24
16:29:38	r020072	173806.52	2208.90	181266.13	951.00	15.96	0.55	26.99	11.07	0.00	2.18	0.00	1.07	0.00	0.53	3.59	8.04	2.69	2.20	0.45	0.87	0.22	0.21	1.31	1.09	0.99	0.54	2.05	1.42	31.95	0.29
16:30:38	r020073	174079.52	2184.38	181898.13	940.22	16.40	0.51	27.51	11.16	0.17	2.29	0.00	1.10	0.00	0.53	2.67	8.36	3.04	2.28	0.48	0.88	0.18	0.24	1.56	1.25	0.92	0.56	2.06	1.44	40.18	0

		Outlet																													
18:50:50	m020213	164089.89	2078.72	177529.67	902.86	13.20	0.49	26.74	10.75	0.00	2.04	0.00	1.08	0.00	0.51	2.92	7.67	3.03	2.10	0.49	0.82	0.13	0.14	1.51	0.71	0.76	0.51	1.82	1.36	16.05	0.19
18:51:49	m020214	169748.16	2152.40	179019.36	930.07	13.80	0.48	26.55	10.80	0.11	1.99	0.00	1.07	0.00	0.52	2.81	7.69	3.10	2.10	0.52	0.85	0.14	0.14	1.14	0.74	0.88	0.52	2.02	1.40	16.44	0.20
18:52:50	m020215	171664.14	2199.44	180146.08	948.75	14.40	0.48	26.67	10.96	0.03	2.08	0.00	1.06	0.00	0.53	2.78	7.77	3.12	2.13	0.52	0.86	0.15	0.14	1.56	0.73	0.88	0.52	2.13	1.42	18.17	0.20
18:53:49	m020216	173560.81	2200.95	180898.59	947.79	15.02	0.51	26.97	11.01	0.11	2.09	0.00	1.07	0.00	0.53	3.96	8.03	3.11	2.20	0.47	0.86	0.23	0.18	1.18	0.84	0.88	0.54	2.08	1.42	22.60	0.23
18:54:50	m020217	174348.27	2238.57	181658.83	963.31	15.47	0.50	27.57	11.13	0.00	2.13	0.00	1.10	0.00	0.54	2.54	8.26	3.10	2.26	0.32	0.88	0.18	0.20	1.60	1.03	0.90	0.55	1.93	1.43	30.09	0.28
	Average	159439.20	2047.50	175532.26	891.32	15.66	0.49	25.72	10.61	0.05	2.12	0.00	1.07	0.00	0.50	26.64	7.51	3.86	2.05	0.65	0.81	0.18	0.16	1.23	0.86	0.95	0.50	1.87	1.35	23.24	0.23
	Std. dev.	24925.55	268.46	13424.49	105.67	4.89	0.05	2.78	0.80	0.07	0.18	0.00	0.08	0.00	0.06	57.61	0.97	2.26	0.27	0.87	0.10	0.12	0.04	0.29	0.19	0.25	0.07	0.24	0.17	6.46	0.05
	Maximum	175888.27	2254.75	183658.23	970.17	39.71	0.55	28.62	11.33	0.33	2.36	0.00	1.23	0.00	0.54	265.80	8.53	13.73	2.33	5.17	0.88	0.73	0.29	2.09	1.50	2.01	0.57	2.29	1.46	51.19	0.41
	Minimum	68060.90	806.99	109330.31	383.76	12.25	0.30	12.80	7.02	0.00	1.42	0.00	0.75	0.00	0.21	2.28	3.89	2.45	1.06	0.13	0.35	0.00	0.12	0.57	0.65	0.70	0.26	0.83	0.53	13.24	0.18

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**APPENDIX D**

**FTIR FIELD DATA SHEETS**

# FTIR Temperature Readout Sheet

# FTIR Temperature Readout Sheet







**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
<b>H<sub>2</sub>O (potential interferant)</b>	<b>20%</b>	<b>0.2213</b>	<b>407</b>	0.00131
CO <sub>2</sub> (potential interferant)	20%	0.000002	0.0036	
H <sub>2</sub> CO (potential interferant)	1 ppmv	0.0002100	0.386	
<b>CH<sub>4</sub> (potential interferant)</b>	<b>20 ppmv</b>	<b>0.0105</b>	<b>19.3</b>	0.00006213
			<b>AVT</b>	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: H<sub>2</sub>O and CH<sub>4</sub>.

## 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 <sub>2</sub> O)	ISC (HCl)	ISC (CH <sub>4</sub> )	FCU	AU
H <sub>2</sub> O	113000	115000	0.000	0.000	-1.7%	-
HCl	253	-22.5	254	0.000	-0.4%	30%
CH <sub>4</sub>	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 -Chemlime**

Spectral File Name	Inlet/Outlet	Error (ppm)	Concentration (ppm)	FMU
RN020005.spa	Outlet	0.23	22.6	0.010
RN020029.spa	Inlet	0.23	22.7	0.010

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.