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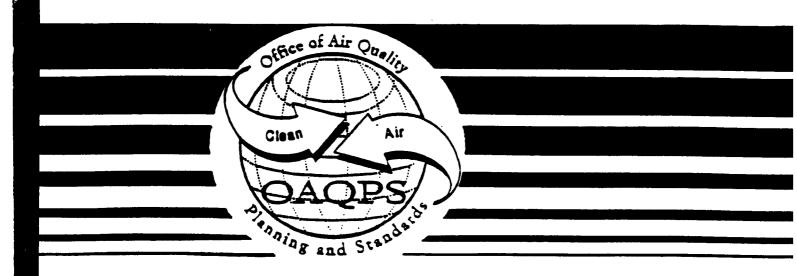
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Final Report of Lime Manufacturing Industry Fourier Transform Infrared Spectroscopy

Redland Stone Products Company San Antonio, Texas



Lime Kiln Source Characterization Final Report

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

Redland Stone Products San Antonio, Texas

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Table of Contents

		Page
1.0	INTRO 1.1 1.2 1.3	DDUCTION 1-1 Objectives 1-2 Brief Site Discussion 1-2 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 Test Report 1-4
2.0	SUMN 2.1 2.2	MARY OF RESULTS 2-1 Emissions Test Log 2-1 FTIR Results 2-1 2.2.1 Overview 2-1 2.2.2 FTIR Emission Results 2-3
3.0	SAMP 3.1	Determination of Gaseous Organic HAPs, HCl, and Criteria Pollutants by FTIR 3-1 3.1.1 FTIR Sampling Equipment 3.1.2 Preparation for Sampling 3.1.3 Sampling and Analysis 3.1.4 FTIR Method Data Review Procedures 3.1.5 FTIR QA/QC Procedures 3-1
4.0	QUAL 4.1	ITY ASSURANCE/QUALITY CONTROL
		List of Appendices
Appen Appen Appen Appen Appen	dix B dix C dix D dix E	FTIR Data Spreadsheet Calculation QA/QC Sheets Gas Cylinder Certification Sheets Raw FTIR Data FTIR Field Data Sheets Pre-test Calculations Post-test Calculations

List of Figures

	Page
2-1 2-2 3-1	HCl Inlet Run - Redland Stone Products2-5HCl Outlet Run - Redland Stone Products2-6FTIR Sampling and Measurement System3-3
	List of Tables
	Page
2-1 2-2 2-3	Emissions Test Log
2-4 3-1 3-2	Other Species Detected by FTIR - Wet Scrubber Outlet
4-1 4-2 4-3	Library

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. 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 Redland Stone Products in San Antonio, Texas. Testing was performed on the inlet and outlet of a wet scrubber. 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 Redland Stone Products in San Antonio, Texas. 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 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 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 at least a total of 25 minutes. Some data points 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 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 30 minute intervals alternating from the inlet and the outlet, 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 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.
- 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 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 OC 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 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 Redland Stone Products in San Antonio, Texas on June 28, 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
6/28/98	Wet Scrubber (inlet/outlet)	Spike 1	FTIR HCI Spike (inlet/outlet)	08:13 - 10:13
6/28/98	Wet Scrubber (inlet/outlet)	Run 1	FTIR (Unconditioned) FTIR (Conditioned)	10:34 - 13:54 16:20 - 17:03
6/28/98	Wet Scrubber (inlet/outlet)	Spike 2	FTIR HCl Spike (inlet/outlet)	13:57 - 14:32

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 30 minutes. Inlet and outlet samples were drawn on a continuous basis; only the FTIR sample analysis was alternated between inlet and outlet. The first (23 outlet and 18 inlet) data points from each 30 minute measurement period were discarded to eliminate data for samples containing both inlet and outlet sample gas. These discarded 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). This response time is somewhat greater than usual, due to the presence of very fine lime dust in the 2 micron FTIR cell filter. The measurement run contained a total of 36 and 28 1-minute average data points for both inlet and outlet measurements, after discarding the transfer 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 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 HCI Test Results. The estimated FTIR HCl detection limit for this study was between 0.14 and 0.17 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 the 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 78.5 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 HCI Results, ppmv

	Run 1				
Date	6/2	8/98			
Time	10:34 - 13:54				
Location	Inlet	Outlet			
Average	21.05	4.69			
SD	0.52	0.28			
Maximum	22.07	5.21			
Minimum	19.89	4.28			
NDP	36 28				
RE	78	3.5			

SD - Standard Deviation

NDP - Number of data points measured RE- Removal Efficiency in percent: 100 X (Avg. inlet-Avg. outlet)/Avg. inlet

NOTE: Raw data presented in Appendix C.

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.

Figure 2-1. HCl Inlet Run - Red Land Stone Products

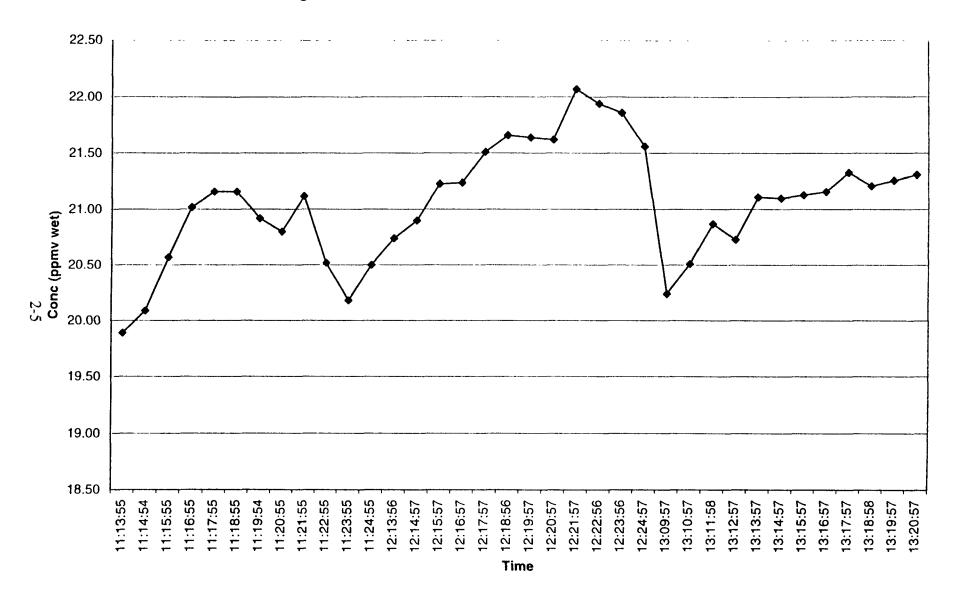
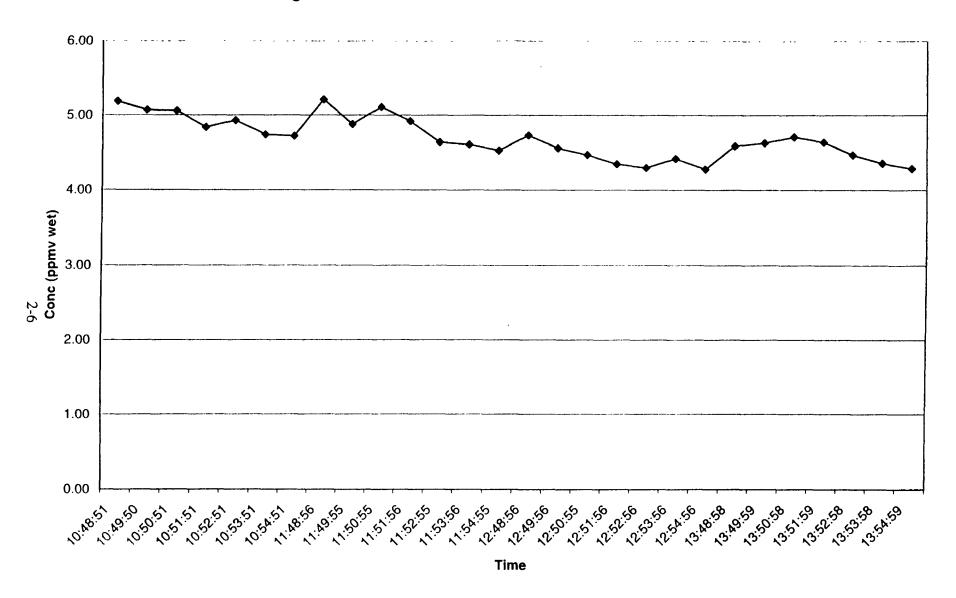


Figure 2-2. HCI Outlet Run - Red Land Stone Products



Wet Scrubber - Outlet/Inlet for Other Species Results—Table 2-3 and 2-4 gives the summary of the wet scrubber for the inlet and outlet FTIR results for other species found during the standard Draft Method 320 extractive analysis, respectively.

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

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

Parameter	CO ₂	NO ₂	SO ₂	СО	NO	C ₄ +	H ₂ O
U/C	U	U	U	U	U	U	U
Average	15.3	2.91	177	23.0	408	0.80	6.22
Std. Dev.	0.557	1.11	83.4	12.2	27.0	0.04	0.176
Max.	16.0	4.63	306	48.1	448	0.87	6.44
Min.	14.5	1.59	59	7.39	365	0.73	5.95
NDP	36	36	36	36	36	36	36
EDL	0.062	1.8	9.7	0.36	8.0	0.33	0.13

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

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

Std. Dev. = Standard Deviation

Max. = Maximum

Min. = Minimum

NOTE: Raw data presented in Appendix C.

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

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

Parameter	CO ₂	NO ₂	SO ₂	co ·	NO	C ₄ +	H ₂ O
U/C	U	U	U	U	U	U	U
Average	12.3	2.82	16.0	22.1	328	1.08	17.3
Std. Dev.	0.423	1.41	37.8	22.1	30.3	0.05	0.141
Max.	13.0	4.89	129	73.8	363	1.16	17.5
Min.	11.7	< 2.31	< 7.6	6.45	265	0.95	17.1
NDP	28	28	28	28	28	28	28
EDL	0.062	2.31	7.6	0.49	7.5	0.53	0.12

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

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.

Std. Dev. = Standard Deviation

Max. = Maximum

Min. = Minimum

NOTE: Raw data presented 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 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 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 sample cell pressure was monitored in real-time 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

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 120 points due to elimination of 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	18 minute cell purge and 12 minute sample collection	23 minute cell purge 7 minute sample collection
Conditioned	l 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 \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 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 nonlinear 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 nonlinear corrections. An experienced spectroscopist can determine whether nonlinear 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.

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

^a Spectra were collected at a cell temperature of 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 nonlinear corrections) for the species in question and compare the result with 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 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 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 three pairs of spiked/unspiked samples. The 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. The 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 Draft 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;
- Direct measurement of methane (CH₄), nitrous oxide (NO₂), and carbon dioxide (CO₂) standard; and
- Pathlength calibration using H22.

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, H22, 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 nitrogen oxides (NO₃) is measured as NO + NO₂. Examination of Table 4-3 shows that all QC checks met the above criteria.

Table 4-1. HCI QC Pre-Test Spike Results - Redland Stone Products

	Outlet									
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio			
1	5.32	12.67	7.59	11.43		0.23	0.045			
2	4.61	12.54	8.14	11.43		0.23	0.045			
3	4.02	12.54	8.69	10.94		0.22	0.043			
4	3.79	12.25	8.62	10.94		0.22	0.043			
5	4.06	12.13	8.25	10.94		0.22	0.043			
6	4.49	12.08	7.78	10.94		0.22	0.043			
Average	4.38	12.37	8.18	11.10	73.68	0.223	0.044			
			Inlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio			
l	7.29	21.02	14.35	21.42		0.43	0.085			
2	6.46	21.62	15.71	21.42		0.43	0.085			
3	6.82	22.37	16.13	21.42		0.43	0.085			
4	7.84	23.36	16.17	20.92		0.42	0.083			
5	7.02	23.41	16.97	20.92		0.42	0.083			
6	6.13	22.45	16.84	21.42		0.43	0.085			
Average	6.93	22.37	16.03	21.25	75.42	0.427	0.084			

NOTE: The spike runs were conducted before and after the test runs, therefore the minimum and maximum values listed here may be different from 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).

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

Corrected Difference = Spiked - (1 - Dilution Ratio) X Unspiked

Table 4-2. HCI QC Post-Test Spike Results - Redland Stone Products

	Outlet									
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio			
1	4.24	17.34	13.37	15.94		0.320	0.063			
2	4.27	17.97	13.97	15.94		0.320	0.063			
3	4.23	18.06	14.10	15.94		0.320	0.063			
4	4.39	18.25	14.14	15.44		0.310	0.061			
5	4.03	18.31	14.53	15.44		0.310	0.061			
6	4.73	18.59	14.16	15.44		0.310	0.061			
Average	4.32	18.09	14.04	15.69	89.48	0.315	0.062			
			Inlet							
Spike Run Number	Lowest Unspiked Value (ppmv)	Spiked (ppmv)	Corrected Difference (ppmv)	Spike Level (ppmv)	% Recovery	SF6 Conc. (ppmv)	Dilution Ratio			
I	17.50	40.32	24.58	25.40		0.510	0.100			
2	18.79	43.54	26.64	25.40		0.510	0.100			
3	19.81	45.06	27.24	25.90		0.520	0.102			
4	20.59	46.50	27.98	25.90		0.520	0.102			
5	21.24	46.98	27.87	25.90		0.520	0.102			
6	21.12	48.13	29.13	25.90		0.520	0.102			
Average	19.84	45.09	27.24	25.73	105.90	0.517	0.101			

NOTE: The spike runs were conducted before and after the test runs, therefore the minimum and maximum values listed here may be different from 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).

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

Corrected Difference = Spiked - (1 - Dilution Ratio) X Unspiked

Table 4-3. Gas Standard Analysis Results

Date	Time	Compound	True (ppm)*	Result (ppm)*	% Recovery
6/28/98	08:00 AM	HCI CO CH₄ NO CO₂ H22	253 102.3 491 503 4.99 %	253.4 102.8 489.9 506.3 5.06 % 3.45 m	100.2 100.5 99.8 100.7 101.4
6/28/98	02:45 PM	HCI CO CH₄ NO CO₂ H22	253 102.3 491 503 4.99 %	248.6 102.5 490.5 503.1 5.08% 3.41 m	98.3 100.2 99.9 100 101.8

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; ± 1 percent; Acceptance Criteria: ± 6 percent of target. * All compounds are recorded in ppm except 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:

* Not able to determine

- 1. Excel QA/QC workbook
- 2. Inlet and Outlet QA/QC information

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3. Number of data points match.	V						
4. Column statistics match (i.e., Average, Standard Deviation, Maximum, Minimum)	V						
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FTIR QA/QC REVIEW Calculation and Methodology QA/QC Checklist

• For each facility tested, the reviewer will have:

* Not able to determine

- 1. Excel QA/QC workbook
- 2. Inlet and Outlet QA/QC information

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

• For each facility tested, the reviewer will have:

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- 1. Excel QA/QC workbook
- 2. Inlet and Outlet QA/QC information

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

• For each facility tested, the reviewer will have:

* Not able to determine

- 1. Excel QA/QC workbook
- 2. Inlet and Outlet QA/QC information

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Below are the results of the Draft 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 -REDLANDS

Spectral File Name	Inlet/Outlet	Error (ppm)	Concentration (ppm)	FMU
RN010032.spa	Outlet	0.15	6.62	0.023
RN010062.spa	Inlet	0.19	17.4	0.011

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.

APPENDIX F POST-TEST CALCULATIONS

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 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:

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%	-

Below are the results of the Draft 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
H ₂ O (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	
CH ₄ (potential interferant)	20 ppmv	0.0105	19.3	0.00006213
TO THE STATE OF TH	THE PROPERTY OF SALE		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 E PRE-TEST CALCULATIONS

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FTIR Temperature Readout Sheet

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3	Inlet Probe	339	338	338	778	329		317	31	4							
4	Outlet Probe	341	327	336	727	305		147	10	7							· · · · · · · · · · · · · · · · · · ·
5	Inlet Filter	340	338	338	334	339		335	33								
6	Outlet Filter	324	301	718	303	289		205	16	,y							
7	Inlet HT	308	307	307	307	306		295	29	7							
8	Outlet HT	329	331	331		330		284	26	8							
9	Inlet Pump	278	270	252	279	263		246	21	9							
10	Outlet Pump	336	319	329	332	310		211	318	7							
11	FTIR Pump	309	302	296	203	266		786	24	0							
12	Pump Box	133	132	135	134	122		119	119								
13	Extra HT	269	268	268	270	268		239	22								
14	FTIR Jumper	316	321	323	324	324		325	22								
15	Pump Jumper	324	325	324	325	325		324	32	2			<u> </u>				
16	Hot Box	351	351	355	356	352		353	35	4						L	
17	Hot Box	248	248	248	240	749	<u> </u>	249	24								
18	Extra HT	292	292	295	297	296		237	12	9							
19	Electronics Box	90	90	91	91	92		91	9								
20									T	1		<u> </u>					

Facility	Radlands
Stack ID	outlet linket
Date	6/28/98
Run Number	

FTIR Temperature Readout Sheet

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1	Inlet Stack	460	458	455	458	456	460	453	452	456	459	456	450	456	453	
2	Outlet Stack	114	115	114	114	114	114	114	1/3	113	1/2	112	1/2	1/3	113	
3	Inlet Probe	334	336	335	337	335	339	335	338	336	341	337	337	338	337	
4	Outlet Probe	323	339	335	279	329	324	339	337	336	747	336	338	323	339	
5	Inlet Filter	336	338	338	329	374	336	340	338	337	340	336	242	340	342	
6	Outlet Filter	294	325	320	202	203	301	307	318	317	325	313	319	295	326	
7	Inlet HT	295	29	300	302	299	304	308	310	310	310	312	313	312	310	
88	Outlet HT	321	321	321		321	320		320	320	319	320	320	721	322	
9	Inlet Pump	258	253	264	323	265	250	255	270	749	265	262	245	270	249	
10	Outlet Pump	342	312	330	329	322	313	319	313	327	308	326	329	318	329	
11	FTIR Pump	293	274	272	290	281	273	286	298	283	276	27/	2.78	299	294	
12	Pump Box	123	111	117	117	119	120	122	122	122	121	123	128	126	127	
13	Extra HT	259	26	260	259	258	260	261	262	263	263	262	264	264	264	
14	FTIR Jumper	313	312	312	311	311	210	310	311	312	3/2	3/2	3/3	315	3/6	
15	Pump Jumper	318	318	319	318	318	317	317	318	319	320	320	322	323	323	
16	Hot Box	351	351	354		352	354	353	352	353	353	353	351	349	352	
17	Hot Box	246	246	247		247	<u> 247</u>	247	247	246	247	247	247	247	248	ļ
18	Extra HT	265	265	262	266	264	262	269	270	271	271	273	275	279	278	
19	Electronics Box	77	79	30	92	32	43	84	84	84	85	86	87	87	87	
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APPENDIX D FTIR FIELD DATA SHEETS

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Time	H2O	(±)H2O	COZ	(±)CO2	CO	(±)CO	NO	(±)NO	NO2	(±)NO2	N20	(±)N2O	NH3	(±)NH3	CH4	(±)CH4	CZH6	(±)C2H8	C2H4	(±)C2H4	H2CO	(±)H2CO	ACTLD	(t)ACTLD	C4+	(±)C4+	CH3OH	(±)CH3OH	HCL	(±)HCL	SF8	(±)SF8	802	(2)502	NOX	(2)NOX
11 13 55	102051 90	1380 97	156204 20	637 20	22 02	0 38	391 66	8 11	2 26	1 68	0 00	1 03	0 00	0 36	1 36	5 19	1 60	1 42	0 07	0 58	0 12	0 14	0.71	0 74	0 80	0 35	0.36	0 98	19 89	0 20	0 01	0 D1	173 61	9 91	393 92	9 99
11 14 54	102373 16	1378 09	156844 34	834 74	25 31	0 40	384 97	8 19	2 04	1 69	0 00	1 08	0 00	0.36	0 16	5 30	1 39	1 45	0 07	0 58	0 00	0 15	0 94	0 77	0 80	0.36	0 17	0 97	20 09	0 21	0.01	0 01	195 16	10 04	387 00	10 08
11 15 55	103282 16	1430 48	158195 75	659 22	33 39	0 42	373 43	8 3c	187	1 76	0 00	1 06	0.00	0 37	0 66	5 23	1 53	1 43	0 11	0 60	0 14	0 15	1 16	0 77	0 76	0 35	0 00	1 01	20 57	0 21	0 01	0 01	246 84	10 19	375 30	10 08
11 16 55	102868 91	1428 47	158458 31	658 57	37 18	0 41	368 27	8 35	1 59	1 87	0 00	1 10	0 00	0 37	0 66	5 30	1 50	1 45	0 15	0 60	0 04	0 15	0 95	0 80	0 75]	0.36	0.00	1 01	21 02	0 22	0 01	0.01	252 58	10 12	389 86	10 22
11 17 55	103277 23	1420 90	158862 63	654 80	38 63	0 42	368 62	8 31	1 75	1 83	0 00	1 12	0 00	0 37	0 78	5 34	_ 1 49	1 46	0 12	0.60	0 05	0 15	0 33	0.78	0 80	0 36	0 00	1 03	21 16	0 21	0 01	0 01	264 15	10 12	370 38	10 14
11 18 55	103226 53	1451 05	159855 87	668 73	42 57	0 43	365 10	8 32	171	1 86	0 00	1 07	0 00	0 37	1 32	5 32	1 61	1 45	0 16	0.61	0.08	0 15	1 12	0.79	0 79	0.38	0.00	1 04	21 16	0 21	0.01	0 01	288 84	10 27	386 81	10 19
11 19 54	102377 07	1440 66	159434 94	064 52	38 81	0 42	380 93	8 27	1 92	1 87	0 00	1 09	0 00	0 37	0 52	5 24	1 42	1 43	0 00	0 60	0.08	0 15	0 85	0.78	0 78	0 35	0 02	1 03	20 92	0 21	0 01	0 01	241 58	10 28	382 84	10 14
11 20 55	103117 60	1451 42	159660 95	868 98	39 47	0 41	373 19	8 32	1 88	1 95	0 00	1 04	0 00	0 37	0 42		1 40	141	0 31	0.61	0 05	0 14	0 84	0.76	0 83	0 35	0.00	1 05	20 80	0.21	0 01	0 01	269 58	10 30	375 08	10 27
11 21 55	103023 47	1479 79	160187 50	882 12	48 07	0 43	369 90	8 37	1 95	191	0 00	1 05	0 00	0 38	0 82		1 63	1 39	0 17	0 62	0 11	0 14	1 24	0.75	0 80	0 34	0 00	1 07	21 12	0 20	0 01	0 01	306 37	10 40	371 80	10 20
11.22 55	101209 87		158208 88	856 78	34 46	0 40	397 23	8 14	2 16	1 94	0 00	1 05	0 00	0 37	0 49	L	1 65	1 40	0 14	0 60	0.00	0 15	0.79	077	0 75	0 34	0 30	1 01	20 52	0.21	1001	0 01	191 19	10 18	399 39	10 08
11 23 55	100773 51	1369 32	156323 25	632 66	24 21	0 38	408 48	8 00	2 56	1 87	0 00	1 08	0 00	0 35	0.66	5 15	1 85	141	0 00	0 58	0 00	0 14	0 54	0.74	0 73	0 34	0 54	0 90	20 18	0 20	0 01	0 01	135 89	10 21	411 04	9 96
11 24 55	101773 52	1354 97	156754 64	625 38	24 50	0 37	397 17	8 18	221	1 78	000	1 02	0 00	0 35	0 22	5 18	1 60	1.42	0 25	0.57	0 07	0 15	0.96	0 77	073	0 35	0.36	0.98	20 50	021	0 01	0 01	159 40	10 00	309 38	0 96
12 13 56	100061 91	1398 20	155695 06	646 08	26 25	0 37	400 74	8 08	2 20	1 60	0 00	0 99	0 00	0.36	1 21	5 04	1 44	1 38	0 03	0.59	0 01	0 14	0.83	0.76	0 80	0 34	0.00	0 00	20 74	0 21	0 01	0 01	222 80	0 70	402 95	9 66
12 14 57	100204 08	1388 42	155131 78	631 69	24 91	0.36	402 54	8 01	2 30	1 82	0 00	1 01	0 00	0 35	0 69	4 99	1 34	1 36	0 16	0.58	0 07	0 15	1 00	0.79	0 78	0 33	0.31	0.98	20 90	0 21	0 01	0 01	200 66	9 63	404 84	0 83
12:15:57	101210 00	1359 63	155738 50	627 99	27 25	0 37	363 33	6 16	2 20	1 67	0 00	1 05	0 00	0 35	1 59		1 32	1 37	0 00	0.57	0.06	0 14	1 32	0.76	1001	0 34	0.08	0 99	21 23	0 20	0 01	0.01	240 38	0 89	395 53	10 03
12 16 57	100839 64	1354 75	155168 64	625 68			396 45	8 11	2 13	1 91	0 00	0 98	0 00	0.35	0 62	4 96	1 57	1 36	0 17	0.57	0 00	0 14	0 47	0.74	0.62	0 33	0.22	0 98	21 24		1001	0 01	216 62	9 85	396 58	10 02
12 17 57	101046 04		155134 38				392 80	8 19	2 22	1 81	0 00	1 01	0 00	0 36	0 95	4 92	1 41	1 35	0 11	0 58	0.08	0 14	0.98	0 76	0 63	0 33	0 08	0 99	21 51		001	0 01	238 61	9 99	395 02	10 00
12 18 56	100913 55						396 96	8 15	2 38	1 78	0 00	0 95	0 00	0 36	0.94	4 66	1 43	1 33	0 22	0.58	0 17	0 15	0 80	077	0 84	0 33	0 01	0 99	21 66		001	0 01	237 40	9 87	399 36	9 93
12.19 57	100883 04	+					399 10	6 06	2 30	1 78	0 00	0 95	0 00	0.36	10 70	4 62	1 45	1 32	0 17	0.59	0 17	0 15	0.89	0 79	0.83	0 32	0.04	0 96	21 64	0 21	1001	0 01	223 70	9 89	401 39	
	101336 66		155113 61				395 31		2 06	1 76	0 00	0.98	0 00	0 36	1 18	4 81	1 25	1 31	0 00	0 58	0 09	0 15	0 95	0.78	0.63	0 32	0.00	101	21 62	+	0 01	0 01	263 49	9 84	397 37	9 90
12 21 57	101374 98						397 97		2 34	1 82	0 00	1 03	0 00	0 35	0.57	4 1111	1 45	1 36	0 00	0 57	0.08	0 15	0.99	0 81	0.86	0 33	0.00	1 00	22 07	0 22	0 01	0 01	271 48	9 86	400 30	8 97
12 22 58	100501 85		155335 33				409 07	8 05	2 48	1 85	0 00	0 97	0 00	0 35	1 63		1 33	1 36	0.06	0 58	0 13	0 15	0.70	0.79	0.83	0 33	0.00	0 99	21 94	0 21	0.01	0 01	236 66	0 82	411 55	9 90
12 23 56	100522 20						411 58	8 07	2 66	1 80	0 00	0 97	0 00	0.35	051		1 29	1 35	0.01	0.58	0 11	0 16	1 17	0.81	0 85	0 33	0.00	0.90	21 86		0 01	001	223 63	10 12	414 22	9 67
12 24 57	100353 60		155400 88			0 36	411 02		2 57	1.87	0 00	0.99	0 00	0 35	1 03		1 50	1 35	0.08	0 57	0 10	0 16	0.79	0.83	0 87	0 33	0 13	0 98	21 56		0 01	001 -	220 77	9 67	413 59	
13 09 57	96318 57	1228 12			-	0 33	442 10	7 53	4 34	1 79	0 00	0 89	0 00	0.32	0 63		1 39	1 27	0 10	0 52	0 00	0 15	1 57	0.78	274	031	0 64	0 88	20 24		001	0 01	87 69	9 39	448 43	9 32
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13 11 50	96815 96	1254 37				0 33	441 08		4 18	1 90	0 00	0 93	0 00	0 33	D 76	4 68	1 68	1 28	0.00	0.53	0 02	0 15	1 03	077	0 82	0 31	0.65	0 88	20 73		0 01	001	59 17	9 06	452 39	9 28
	95933 13	1218 97				0 33	447 82		4 34	107	0 00	0 92	0 00	0 32	1083	4 81	1 67	1 32	0 00	0 51	0 08	0 15	1 25	0 77	0 78	0 32	0 78	0 07	21 11		0 01	001	89 11	9 03	449 31	9 33
13 13 67		1237 68	148062 52			0 33	444 93		4 47	- 170 -	0 00	0 89	0 00	032	1 46		+ 1 16	1 24	0 11	0 63	1 8 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	0 15	1 14	077	0 00	0 30	0 72	0.87	21 10		0 01	0 01	70 00	8 60	445 79	9 25
13 14 67	95846 08	1236 12					442 78		4 47	185	0 00	0.03	0 00	0 32	0.42		1 32	1 24	0 00	0 52	0 00	0 15	114	079	0.81	0 30	0 73	0 96	21 13		0 01	001	63 95	8 90	447 25	9 20
13 15 57	96148 41						440 28		4 29	1 70	15 8	0 89	0 00		1 3	1-33	1 26	1 22	000	0 54	0 05	0 15	1 38	0 78	0 83	0 30	0.08	0.87	21 16		0 01	0 01	75 99	9 07	444 57	9 17
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13 18 58	95809 18		145033 66				440 33		4 63	174	0 00	0 86	0 00	0 32	0.56	4 47	1 34	1 22	0 23	0 52	0 00	0 15	1 18	0 77	0 85	0 30	0.58	0.86	21 21		0 01	0 01	89 31	8 98	444 95	9 26
	95205 48	120/ /4	144663 38				441 78		4 62	1 66	0 00	0 86	1000	0 32	1121	4 44	1 24	121	0 26	0 53	0 18	0 15	072	0.77	0 80	0 30	D74	0.87	21 26		0.01	0 01	63 72	9 02	446 40	9 19
	95858 49	1238 99				0 31	439 39		4 36	174	0 00	0 90	000	0 32	10 02	4 53	1 33	1 24	0.05	0 52	0.06	0 15	1 36	0.80	0 84	0 30	071	0.87	21 31		0 01	0.01	61 05	8 90	443 75	9 32
Average	99755 20	1348 57		822 28		0.36	407 86		291	1 61	0 00	D 98	0 00	0 35	0 00	491	1 44	134	0 10	0 57	0 07	0 15	0.99	0.78	0 80	0 33	0 30	0.98	21 06		0 01	0.01	178 51	9 67	410 87	9 76
Standard Deviation	2834 05	77 21	5565 70	34 43		0 04	26 97	0 32	111	0.09	0 00	0.08	0 00		10.30	0 28	0 15	0 00	0 09	0.03	0.05	0 01	0 28	0 02	0.04	0 02	0.30	0 07	0 52	0 01	0 00	0 00	83 41	0 52	28 04	0 38
Maximum value	103282 16		160187 50			0 43	447 82		4 63	1 95	0 00	1 12	0 00		1 63		1 85	1 46	0 31	0.62	0 17	0 16	1 58	0 83	087	0.38	0.78	1 07	22 07		0 01	0.01	306 37	10 40	452 39	10 29
Minimum value	95205 48			4			365 10	+	1 59	1 60	0 00	0.86	0 00	0 32	0 16	4 44	1 15	121	0 00	051	0 00	0 14	0 33	0.73	0 73	0 30	0.00	0.86	19 89		0 01	0.01	59 17	8 60	368 81	9 17
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Method title Lane Uncond	-									1		1 - 1		1				1	-	+ -								ļ		 					
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	20 (±1H)	4	(±)CO	2 CO	(±)CO	NO	(±1NO	MOS	(±)NO2	N20	(±)N2O	MILE !	(±)NH3	CH4	(±)CH4	C2H6	(±)C2H6	C2H4	(±)C2H4	HZCO	(±)H2CO	ACTLD	(±)ACTLD	C4+	(±)C4+	СНЗОН	(±)CH3OH	HCL	(±)HCL	SF6	(±)SF6	802	(±)502	NOX	(± NNOX
		62 121623 34			+-3-5	328 75	J	2 30	12,700	0.00	1 43	0 00	0.28	č ša	127~114	3 60	2 32	0.00	0.46	0 13	0.11	0 53	0.57	1 08		0 55		1	0 15	0.00	0.01		7 88		E SS
10 49 50 1739						331 79	4	2 24	2 32	2 00	-124	0 00	0 28	1 5 54	4.64	3 83	2 36	0.00	046	0 00	0 11	1 17	0.56	1 05	0 57	0 40	0 69	5 19	0 15	0.00	0 01	0 00		331 05	9 97
10 50 51 1733					0 49	328 10		218	2 30	0.00		0 00	0 28	0.50	R 40	3 69	2 32	0 00	0.45	000	0 10	0 29	0.55	1 11	0 57	0 49	0 69	5 08	0 15	001	001	000		330 28	10 01
10 51 51 1732						325 70	746	2 25	-2 33	0.00	1 30	0 00	0 29	6 27	- 11	3 62	2 22	0 00	0 47	0 07	0 10	011	0 54	1 00	0 54	0 60	- 070	4 84	0 15	0 00	0.01	000		327 95	9 79
10 52 51 1724					0 49	326 22	7 48	208	2 18	0 00	134	0 00	0 29	1 23	8 00	3 54	2 19	0 00	0 47	0 10	0 10	0.61	0.52	100	0 54	0 52	068	4 93	0 14	0 00	001	000		328 28	9.64
10 53 51 1728					0 49	320 92	7 58	2 03	234	0 00	1 40	0 00	0 29	0.05	6 23	3.58	2 25	0 00	0.47	0 10	0.10	0 13	0.54	1 08	0 55	0 66	0.69	474	0 15	0 00	0 01	0.00		322 95	9 92
10 54 51 1725		55 123112 12	+		0.51	318 78	7 59	2 02	241	0 00	1 48	0 00	0.26	0.76	A 39	3 73	2 29	0 00	0.46	0 00	0 10	0.45	0.54	1 08	0.56	0.58	070	4 72	0 15	0 00	0 01	000		320 80	10 01
11 48 56 1742						264 89	7 80	0.56	5 14	0 00	1 50	0 00	031	0.66	R 29	3 78	2 27	0 00	0 50	0 07	0 10	0.54	0.54	0 00	0.56	0 00	077	1 2 2	0 15	1881	001	107 09	7 90	265 45	10 25
		58 130258 14	547 4		4 - -	267 42	7 02	0.50	241	0 00	1 49	0.00	0.31	0.47	8 37	3 70	2 29	0 00	0.50	0 13	0 10	0.50	0 52	0 95	0 56	000	077	1 4 86	0 14	1001	0.01	106 09	6 32	267 91	10 33
11 50 55 1751		63 130215 69				268 11	7 88	0 96	2 40	0.00	147	0 00	031	0.80	B 32	3 30	2 27	0.00	0.50	0 12	011	0 92	0.55	1 03	0 56	0 03	0.79	3 11	0 15	0 00	0.01	126 74	7 97	269 07	10 29
11 51 56 1751					0 55	278 66	7 86	0 84	2 36	0 00	1 43	0 00	0 30	0.45	8 11	3 31	2 22	0 00	0 49	0 03	0 10	0 73	0.54	1 05	0 54	0 13	0.76	4 92	0 15	0 00	0 01	72 72	8 13	279 49	10 21
11 52 55 1751				8 49 22		293 58	7 80	111	2 19	ōã		0 00	0.30	0 00	7 99	3 43	2 18	0 00	0 49	0 07	0 11	0.58	0.56	1 02	0 53	0.36	0.75	4 84	0 15	0 00	0 01	32 36		294 69	10 00
11 53 56 1745	36 92 1254	90 127541 84	540 2	7 38 91	0.50	305 69	767	1 37	2 21	0 00	1 28	0 00	0.30	0 71	7 85	3 49	2 15	0.00	0.49	0 11	0 10	0 26	0 53	1 00	0 53	0 47	0.75	4 61	0 14	0 00	0 01	0.00	8 05	307 08	9.67
11 54 55 1737	96 34 1230	03 127238 62			0 52	301 22	7 65	1 49	2 22	0 00	1 30	0 00	0 30	0.70	7 90	3 34	2 16	0 00	0 48	0 10	0 10	0 44	0.54	104	0 53	0 62	0 73	4 53		0.00	0.01	0.83	7 69	302 72	0.87
12 48 56 1715	47 06 1135	79 119806 83	490 3	0 82	0 44	350 54	7 38	3 99	2 46	0 00	1 28	0 00	0 27	0 55	7 67	3 42	2 10	0 00	0 45	0 10	0 10	1 20	0.54	1 13	0 51	0 57	0.69	4 73	0 14	0.00	0.01	0.00	7 37	354 53	9.84
12 49 56 1712	95 45 1128	87 118566 68	487 3	3 8 36	0 43	357 07	7 27	4 40	2 35	0 00	1 24	0 00	0 27	0 32	7 69	3 36	2 10	0 00	0 44	0.08	011	0.60	0.56	1 10	0 51	0.78	0.68	4 56	0 15	0 00	0 01	0.00		361 47	9.62
12 50 55 1710	85 00 1147	29 117541 93	495 4	8 7 27	0 49	382 13	7 26	4 69	2 54	0 00	1 26	0 00	0 28	0 70	7 66	3 56	2 09	0 00	0 45	0 15	0 10	0 43	0 53	1 09	0 51	0.54	0.68	4 47	0 14	0 00	0.01	0.00	7 17	367 02	9 80
12 51 56 1711	33 70 1146	95 118038 22	495 3	7 05	0 47	360 62	7 23	4 54	2 42	0 00	1 18	0 00	0 28	1 13	7 55	3 35	2 06	0 00	0 45	0 16	0 10	0 15	0 52	1 18	0 51	0.65	0 66	4 35	0 14	0 00	0 01	0 00	7 23	365 16	9 65
12 52 56 1712	89 63 1150.	08 117431.30	496 5	6 96	0 48	362 89	7 37	4 84	2 30	0 00	1 24	0 00	0 28	1 10	7 80	3 54	2 13	0 00	0 45	0.08	0 11	0 52	0 55	1 10	0 52	0 57	0 87	4 30	0 15	0 00	0 01	0.00	7 37	367 73	9 08
12 53 56 1710	41 92 1131	10 110007 12	440 4	0 45	0 45	362 78	7 18	4 76	2.34	0 00	1 23	0 00	0 27	1 06	7 66	3 76	2 00	0 00	0 44	0 11	0 10	0.64	0 54	1 11	0 51	0 40	0 67	4 42	0 15	000	0 01	0 00	7 08	367 53	9 52
12 54 56 1717	03 38 1156	06 117805 13	496 5	8 762	C 46	355 18	7 36	4 30	2 31	0 00	1 23	0 00	0.28	1 43	7 68	3 73	2 10	0 00	0.46	011	0 10	0 11	0 63	1 07	0 61	064	0 00	4 28	0 14	0 00	0 01	0 00	7 63	360 87	0 06
13 48 58 1712	97 02 1171	08 120036 26	506 6	4 9 76	0 48	348 75	7 41	4 07	2 33	0 00	1 23	0 00	0 28	1 40	7 73	3 32	211	0.00	0 46	0 03	0 10	0 24	0 54	1 14	0 52	0 64	0 69	4 59	0 14	000	0 01	0 00	7 07	350 83	974
13 49 59 1710	32 58 1191	23 120746 97	614 4	7 10 76	0 47	340 37	7 33	3 70	2 19	0 00	1 24	0 00	0 29	0 93	7 62	3 31	2 08	0 00	0 47	0.09	0 10	0 57	0 54	1 10	0 51	0 58	0.66	4 83	0 15	0 00	0.01	0.00	7 20	344 07	9 51
13 50 56 1717	08 98 1173	05 121025 48	508 3	1 11 27	0 48	343 67	7 44	3 45	2 27	0 00	1 29	0 00	0 28	0 44	7 87	3 51	2 15	0 00	0 46	0.00	0 11	0 73	0.56	1 08	0 63	0 63	0.89	471	0 15	0 00	0 01	0 00	7 28	347 12	971
13 51 59 1716	06 42 1170	57 120391 80	505 2	10 31	0 46	348 52	7 28	3 61	2 23	0 00	1 17	0 00	0.28	0 68	7 67	3 30	2 10	0 00	0 46	0 00	0 10	0 35	0 53	1 15	0 51	0 57	0 70	4 64	0 14	0 00	0.01	0 00	6 63	352 33	951
13 52 58 1716	60 89 1212	32 121692 01	523 2	8 11 40	0 46	342 78	7 48	3 44	2 16	D 00	1 22	0 00	0 29	0 40	7 75	3 67	2 12	0 00	0 48	0 08	0 11	0 63	0 55	1 11	0 52	0 52	0 69	4 47	0 15	0 00	0 01	0 00	7 70	348 22	9 62
	53 33 1206					344 04	7 44	3 40	2 15	0 00	1 23	0 00	0 29	0 00	7 73	3 48	2 11	0 00	0 47	0 11	0 10	0 88	0 52	1 12	0 52	0 63	0.71	4 36	0 14	0 00	0 01	0 00	7 40	347 44	9 50
13 54 59 1720	87 27 1175					340 64		3 26	2 22	0 00	1 21	0 00	0 28	1 54	7 79	3 45	2 13	0 00	0 46	0.00	0 10	0 57	0 54	1 14	0 52	0.58	0 70	4 20	0 14	0 00	0 01	0 00	7 53	343 90	9 65
Average 1726	84 47 1191	82 122575 61	513 9	4 22 10	0 49	327 78	7 51	2 82	2 31	0 00	1 32	0 00	0 29	0 73	7 97	3 52	2 18	0 00	0.47	0.08	0 10	0.53	0 54	1 08	0 53	0 49	0 71	4 69	0 15	0 00	0 01	15 99	7 56	330 59	9 83
Standard Deviation 140	9 62 44 3	7 4234 34	18 58	22 09	0.04	30 30	0 21	141	0 10	0 00	0 11	0 00	0 01	0 40	0 32	0 17	0.08	0.00	0 02	0 05	000	0 28	0.01	0 05	0 02	0 20	0 03	0 28	0.01	0 00	0 00	37 75	0 36	31 65	0 24
Maximum value 1751	47 92 1271	58 130258 14	547 4	73 80	0 58	362 89	7 92	4 89	2 54	0 00	1 54	000	0 31	1 54	8 64	3 83	2 36	0.00	0 50	0 16	0 11	1 20	057	1 16	0 58	0.76	0.79	5 21	0 15	001	0 01	128 74	8 32	367 73	10 33
Minimum value 1710:	32 58 1128	116997 12	487 3	3 6 45	0.43	264 89	7 18	0 50	2 15	0 00	1 17	0 00	0 27	0 00	7 55	3 30	2 06	0 00	0 44	0 00	0 10	0 11	0 52	0 95	0 51	0 00	0 67	4 28	0 14	0 00	0 01	0.00	6 83	265 45	9 51

APPENDIX C RAW FTIR DATA

APPENDIX B GAS CYLINDER CERTIFICATION SHEETS



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.#: **BLEND TYPE:** 9101008011-R132

CERTIFIED

CYLINDER #: 982153Y

CYLINDER PRES: 2000 psig **CYLINDER VALVE: CGA 330**

ANALYTICAL ACCURACY: + / - 5%

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

Sulfur Hexafluoride is +/- 2%

8

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 #:

132874

ITEM#:

CERTIFICATION DATE: 5/11/98

BLEND TYPE:

CERTIFIED

CYLINDER #:

1757934Y CYLINDER PRES: 2000 psig

P.O.#:

9101008004-R986

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

DATE:

5/11/98

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



3434 Houte 22 West • Branchburg, No 08676 USA

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

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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 Sulfur Hexafluoride	250 ppm 2.00 ppm	260 ppm 2.00 ppm
Nitrogen	Balance	Balance

Sulfur Hexafluoride is +/- 2%

ANALYST:	DATE:	8/10/98
Ted Neeme		



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SHIPPED TO:

Eastern Research Group Inc.

900 Perimeter Park Morrisville , NC 27560

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OF

ANALYSIS

SGI ORDER #:

BLEND TYPE:

132874

CERTIFIED

ITEM#:

CERTIFICATION DATE: 5/11/98

CYLINDER #:

1370597Y

CYLINDER PRES: 2000 psig

P.O.#:

9101008004-R986

ANALYTICAL ACCURACY:

+/- 2%*

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

DATE:

5/11/98

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



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> CERTIFICATE OF

> > **ANALYSIS**

SGI ORDER #:

128118

ITEM#:

Nitrogen

CERTIFICATION DATE: 10/16/97

BLEND TYPE:

CERTIFIED

CYLINDER #:

1757972Y

CYLINDER PRES: 2000 psig

P.O.#:

7904004005-R690

ANALYTICAL ACCURACY:

+/- 2%

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

Balance

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

ANALYST:

DATE:

10/16/97

Balance



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		CERTIFICATE		- <u></u>
		OF ANALYSIS	·	
SGI ORDER #:	126876	ANALTOIS		
ITEM#:	1		CYLINDER #:	1852209Y
CERTIFICATION DATE:	8/29/97		CYLINDER PRES:	
BLEND TYPE:	CERTIFIED		P.O.#:	7904004005-R562
		ANALYTICAL	ACCURACY:	+/- 5 %
COMPONENT		REQUESTED GAS		ANALYSIS
Hydrogen Chloride		200 ppm		210 ppm
Sulfur Hexafluoride		20.0 ppm		20.2 ppm
Nitrogen		Balance		Balance
NALYST: Ted	Neeme		DATE:	8/29/97

USA • United Kingdom • Germany • Japan



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SHIPPED TO:

Eastern Research Group Inc.

900 Perimeter Park Morrisville, NC 27560

CERTIFICATE

OF

ANALYSIS

SGI ORDER #:

BLEND TYPE:

134942

ITEM#:

CERTIFICATION DATE: 8/10/98

P.O.#:

9101008011-R132

CERTIFIED

CYLINDER #: 1689487Y

CYLINDER PRES: 2000 psig

CYLINDER VALVE: CGA 330

ANALYTICAL ACCURACY: + / - 5%

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

Sulfur Hexafluoride is +/- 2%

ANALYST: Ted Neeme

DATE: 8/10/98

USA • United Kingdom • Germany • Japan



SPECTRA GASES

MECT SEP 1 8 1907



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CERTIFICATE OF ANALYSIS

EPA PROTOCOL MIXTURE

PROCEDURE #: G1

CUSTOMER:

Eastern Research Group Inc.

CYLINDER #:

CC79878

SG! ORDER #:

126876

CYLINDER PRES: 2000 PSIG

ITEM#:

660

P.O.#:

7904004005-R562

CGA OUTLET:

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%
				
ALANCE	Nitrogen			L

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
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SPECTRA GASES

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EPA PROTOCOL MIXTURE

CYLINDER PRES: 2000 PSIG

CC80890

350

PROCEDURE #: G1

CYLINDER #:

CGA OUTLET:

CUSTOMER:

Eastern Research Group Inc.

SGI ORDER #: 126876

ITEM#:

P.O.#:

7904004005-R562

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		
				
		1		
		_1i	ļ	

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:

DATE: 8/26/97



SHIPPED TO:	Eastern Research (900 Perimeter Pari Morrisville , NC 27	(•
7-		CERTIFICATE		
		OF		
SGI ORDER #:	126876	ANALYSIS		
ITEM# :	2		CYLINDER #:	CC80877
CERTIFICATION DAT			CYLINDER PRES:	
BLEND TYPE:	CERTIFIED		P.O.#:	7904004005-R562
		ANALYTICAL	ACCURACY:	+/- 2 %
COMPONENT		REQUESTED GAS	į	ANALYSIS
Halocarbon 22		40.0 ppm	•	40.3 ppm
Nitrogen		Balance		Balance
ANALYST:Te	ed Neeme	MOREONE PER RELIES TO MARKET SANSTAN Fill I To 1 KANNET MONEY TO MAKET SANSTAN		8/ 29/97

USA • United Kingdom • Germanv • Janan

TECHNICAL REPORT DATA (Please read Instructions on reverse before completing)					
1. REPORT NO. EPA- 454/R-00-012	2.	3. RECIPIENT'S ACCESSION NO.			
4. TITLE AND SUBTITLE Final Report of Lime Manufacturing Industry Fourier Transform Infrared Spectroscopy		5. REPORT DATE May 2000			
Redland Stone Products, San Antonio Texas		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		13. TYPE OF REPORT AND PERIOD COVERED			
Director Office of Air Quality Planning and Standards Office of Air and Radiation U.S. Environmental Protection Agency Research Triangle Park, NC 27711		Final Report			
		14. SPONSORING AGENCY CODE EPA/200/04			

15. SUPPLEMENTARY NOTES

16. ABSTRACT

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.

17. KEY WORDS AND DOCUMENT ANALYSIS					
a. DESCRIPTORS Hydrogen Chloride (HCL)	b. IDENTIFIERS/OPEN ENDED TERMS Air Pollution control	c. COSATI Field/Group			
Hazardous Air Pollutants	Wet Scrubber				
18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (Report) Unclassified	21. NO. OF PAGES 96			
Release Unlimited	20. SECURITY CLASS (Page) Unclassified	22; PRICE			