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Air

FTIR EMISSIONS TEST AT AN IRON FOUNDRY

Waupaca Foundry, Inc.
Plant No. 5, Tel City, Indiana

FTIR EMISSIONS TEST AT AN IRON FOUNDRY

Waupaca Foundry, Inc.
Plant No. 5, Tell City, Indiana

Prepared for

Office of Air Quality Planning and Standards
Emissions, Monitoring and Analysis Division
Emission Measurement Center (MD-19)
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PREFACE

This draft report was prepared by Midwest Research Institute (MRI) for the U. S. Environmental Protection Agency (EPA) under EPA Contract No. 68-D-98-027, Work Assignment No. 2-13. Mr. Michael Ciolek is the EPA Work Assignment Manager (WAM). Dr. Thomas Geyer is the MRI Work Assignment Leader (WAL). The field test was performed under EPA Contract No. 68-D2-0165, Work Assignment No. 4-25 and a draft report was submitted under EPA Contract No. 68-W6-0048, Work Assignment No. 2-08. Mr. Michael Ciolek was the EPA WAM for the Emission Measurement Center (EMC) under Work Assignment 4-25 and Mr. Michael Toney was the WAM under Work Assignment No. 2-08. Mr. John Hosenfeld was the MRI WAL under Work Assignment 2-08 and Dr. Thomas Geyer was the MRI task leader for Work Assignment 2-08, task 08.

This report presents the procedures, schedule, and test results for an emissions test performed at Waupaca Foundry in Tel City, Indiana. The emissions test used Fourier transform infrared (FTIR) sampling procedures to measure hazardous air pollutants (HAP's) and other pollutants.

This report consists of one volume (354 pages) with seven sections and four appendices.

Midwest Research Institute

For John Hosenfeld
Program Manager

Approved:

Jeff Shular
Director, Environmental Engineering Division

September 30, 1999

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1.0 INTRODUCTION

1.1 BACKGROUND

The Emission Measurement Center (EMC) of the U. S. EPA received a request from the Metals Group of the Emission Standards Division (ESD) and Source Characterization Group of the Emission Monitoring and Analysis Division (EMAD), both in the Office of Air Quality Planning and Standards (OAQPS), U. S. EPA, to perform emissions testing at iron foundries, specifically on cupola emission control devices, as well as pouring, cooling, and shake-out operations. The test program was performed in September, 1997 under Work Assignment 4-25, under EPA Contract No. 68-D2-0165. This draft report was prepared under Work Assignment 2-08, under Contract No. 68-W6-0048.

1.2 PROJECT SUMMARY

The cupola melting process is used to melt iron for casting into automotive and machine parts. It is potentially a significant source of HAP emissions, including metal and organic compounds. Emissions from the mold pouring, cooling, and shake-out are also potential sources of HAP emissions.

The principal emission point at a cupola furnace is the exhaust from the furnace itself. Emission controls for the Waupaca Plant No. 5 include a movable cap on the cupola, that seals the charge, coarse grain separator, afterburner, drop out chamber, heat exchangers (recuperators), dry calcium hydroxide injection system, pulse-jet baghouse, and stack. Cupola emissions testing was conducted at the stack (outlet) and an inlet location to the baghouse to determine the measurable emissions released during the melting process. Testing was also conducted at the cooling line and shake-out housing ducts to determine the measurable emissions released during the cooling and shake-out of the castings. Pouring operations had no emission capture or control system; thus, no testing was conducted at the mold pouring location.

Three test runs were conducted at the cupola inlet and outlet locations over a three day period simultaneously with manual method testing conducted by Pacific Environmental Services (PES). One test run over a 4 hour (hr) period was conducted by FTIR only at both the cooling line and shake-out housing locations. A summary of the FTIR results at the cooling and shake-out housing locations is presented in Table 1-1. Emissions from the mold cooling and shakeout housing included CO, methane, and ethylene. The emissions also contained a mixture of heavier aliphatic hydrocarbon compounds. In the draft report the mixture of heavier

hydrocarbons was represented by "hexane" because hexane and isooctane are the only aliphatic hydrocarbons in the EPA library of HAP reference spectra. There are many hydrocarbon compounds that are structurally similar to hexane and also have similar spectral features. After the draft report was submitted, EPA directed MRI to measure quantitative spectra of some additional hydrocarbon compounds. MRI selected candidate compounds that, based on their infrared spectra in the region of the analyses, near 2900 cm^{-1}, were likely to be components of the sample mixture. MRI obtained commercially-prepared cylinder standards of butane, n-heptane, pentane, 1-pentene, 2-methyl-1-pentene, 2-methyl-2butene, 2-methyl-2-pentene, and 3-methylpentane. MRI then measured FTIR reference spectra of these compounds in the laboratory. MRI also measured new high-temperature spectra of the HAPs hexane and isooctane. Documentation of the new reference spectra and a brief description of the laboratory procedures is presented in Appendix B.

The new spectra were used in revised analyses that gave the results presented in Table 1-1 and in Tables B-1 and B-2. The new spectra made it possible to better represent the sample mixture spectrum. Consequently hexane was not detected in mold cooling emissions, and was only detected in one sample in the shakeout housing emissions. The reported hexane concentrations are lower in the revised results because the spectrum of the sample hydrocarbon mixture, which was represented by "hexane" in the draft results, is better represented by some of the new spectra of other non-HAP hydrocarbons. In particular, 3-methylpentane and 1-pentene were detected in cooling and shakeout housing process emissions. Butane and 2-methyl-2-butene were also measured at the shakeout housing. The revised results give a more accurate representation of the process emissions, but it's possible that other hydrocarbon compounds could be measured in the emissions if their reference spectra were available.

The FTIR results from the cupola baghouse inlet and outlet locations are presented in Table 1-2. Toluene was included in the analysis because this compound was spiked at the inlet and outlet. Additional description of the results is in Section 4.

EPA Method 320 uses an extractive sampling procedure. A probe, pump, and heated line are used to transport samples from the test port to a gas manifold in a trailer that contains the FTIR equipment. Infrared spectra of a series of samples are recorded. Quantitative analysis of the spectra was performed after the FTIR data collection was completed. All spectral data and

results were saved on computer media for review after the test is completed. A compact disk containing all of the FTIR data was provided with the draft report.

TABLE 1-1. SUMMARY OF FTIR RESULTS FROM THE MOLD COOLING AND SHAKE-OUT HOUSING DUCTS

Compound		Cooling	Uncertainty	Shakeout	Uncertainty
Toluene	ppm[a]	17.5	3.9	0.81	3.5
	lb/hr	5.01		0.33	
	kg/hr	2.27		0.15	
Hexane	ppm	ND	30.6	0.16	17.3
	lb/hr			0.060	
	kg/hr			0.027	
Ethylene	ppm	13.3	0.8	3.4	0.8
	lb/hr	1.158		0.42	
	kg/hr	0.525		0.19	
Methane	ppm	178.5	2.6	26.0	1.6
	lb/hr	8.859		1.82	
	kg/hr	4.017		0.826	
Carbon Monoxide	ppm	402.3	28.1	106.7	19.1
	lb/hr	34.95		13.06	
	kg/hr	15.85		5.92	
Formaldehyde	ppm	ND	2.97	ND	1.74
	lb/hr				
	kg/hr				
3-Methylpentane	ppm	5.42	1.68	3.35	1.05
	lb/hr	1.45		1.263	
	kg/hr	0.656		0.573	
Butane	ppm	ND	34.90	3.21	5.83
	lb/hr			0.816	
	kg/hr			0.370	
1-Pentene	ppm	17.9	3.89	0.92	8.82
	lb/hr	3.88		0.28	
	kg/hr	1.76		0.13	
2-Methyl-2butene	ppm	ND	8.81	7.02	1.53
	lb/hr			2.19	
	kg/hr			0.991	

[a]Average ppm concentration for the Run.

TABLE 1-2. SUMMARY OF FTIR RESULTS (ppm) AT THE CUPOLA BAGHOUSE INLET AND OUTLET[a]

Compound		9/8/97 (12:02 - 17:07)				9/9/97 (7:49 - 14:19)				9/10/97 (7:53 - 14:19)			
		Inlet	Unc[b]	Outlet	Unc	Inlet	Unc	Outlet	Unc	Inlet	Unc	Outlet	Unc
HCl	ppm[c]	33.5	3.3	23.3	3.3	27.7	3.6	16.3	3.4	29.7	3.3	22.6	3.1
	lb/hr	5.2		4.7		6.2		4.6		6.7		6.4	
	kg/hr	2.4		0.4		2.8		0.6		3.0		0.8	
Toluene	ppm	ND	2.9	ND	2.9	ND	3.2	0.4	3.0	ND	2.9	ND	2.8
	lb/hr							0.3					
	kg/hr							0.1					
Methane	ppm	5.2	1.2	4.7	1.2	5.3	1.3	4.9	1.2	4.8	1.2	4.7	1.1
	lb/hr	0.6		0.7		0.6		1.1		0.6		1.0	
	kg/hr	0.3		0.1		0.3		0.1		0.3		0.1	
Formaldehyde	ppm	0.3	1.7	ND	1.6	ND	1.8	ND	1.7	ND	1.7	ND	1.6
	lb/hr	0.04											
	kg/hr	0.02											

[a] PES did not complete a run on 9/8, but completed a manual run on 9/9 and two manual runs on 9/10. The PES flow data from 9/9 were used to calculate mass emission rates for the MRI runs on 9/8 and 9/9. The PES flow data from their first manual run on 9/10 were used to calculate emission rates for the MRI run on 9/10.

[b] Estimated uncertainty in ppm in the reported concentration.

[c] Average ppm concentration for the Run.

1.3 PROJECT PERSONNEL

The EPA test program was administered by the EMC. The Test Request was initiated by the Metals Group of the ESD and the Source Characterization Group of the EMAD, both in OAQPS. Some key project personnel are listed in Table 1-3.

TABLE 1-3. PROJECT PERSONNEL

Organization and Title	Name	Phone Number
Waupaca Foundry, Inc. P.O. Box 249 311 S. Tower Road Waupaca, WI 54981	Jeff Loeffler	(715) 258-6629
Waupaca Foundry, Inc. P.O. Box 189 9856 State Highway 66 Tell City, IN 47586	Keith Tremblay	(812) 547-0700
U. S. EPA, EMC Work Assignment Manager Work Assignment 4-25	Michael K. Ciolek	(919) 541-4921
U. S. EPA, EMC Work Assignment Manager Work Assignment 2-08	Michael L. Toney	(919) 541-5247
MRI Work Assignment Leader Work Assignment 4-25 Work Assignment 2-13	Thomas J. Geyer	(919) 851-8181 Ext 3120
MRI Work Assignment Leader Work Assignment 2-08	John Hosenfeld	(816) 753-7600 Ext 1336

2.0 PROCESS AND CONTROL EQUIPMENT OPERATION

The material in Section 2 was prepared by Research Triangle Institute and provided to MRI by the EMC. It was included in the report without MRI review.

2.1 INTRODUCTION

The Waupaca foundry in Tell City, Indiana, is a completely new grey iron foundry that started operation in February 1997. The foundry casts a diverse group of products, including brake drums, shoes, rotors, calipers, and other parts. The plant operates one large cupola that melts at a rate of about 60 tons/hr (tph), and operates four pouring lines. This section of the test report provides a description of the cupola operation for iron melting, and the casting operation, including pouring, cooling, and shake out.

2.2 PROCESS DESCRIPTION

2.2.1 Iron Melting in Cupolas

The Waupaca foundry in Tell City operates a large, water-cooled cupola that melts at a rate of approximately 60 tph, with a blast rate of 10,000 to 15,000 standard cubic feet per minute (scfm), which makes it a large cupola by U.S. industry standards. Figure 2-1 is a simplified schematic of the cupola gas handling system and emission control equipment.

The cupola is charged with metal scrap, re-melt, coke, and limestone at the top of the cupola, using one of two automated skip buckets. The level of metal within the cupola is monitored, and the charge material in the skip bucket is dumped into the cupola when the level of charge falls below a set level. The seal from the charge material and a draft on the cupola prevent gases from escaping. If, for any reason, the charge material cannot be added to the cupola within 5 minutes (min) of the level falling below the set point, the cupola will automatically go "off blast" until the appropriate charge level in the cupola can be achieved.

The blast air is preheated to about 1,000°F in the blast air recuperator and is introduced into the bottom of the cupola through 8 tuyeres. The blast is also enriched with oxygen under certain melting conditions. The off gas from the cupola is removed at 250-300°F. The off-take duct is lined with refractory material and leads to a coarse grain separator where heavy particles are removed. The separator is cooled with non-contact water.

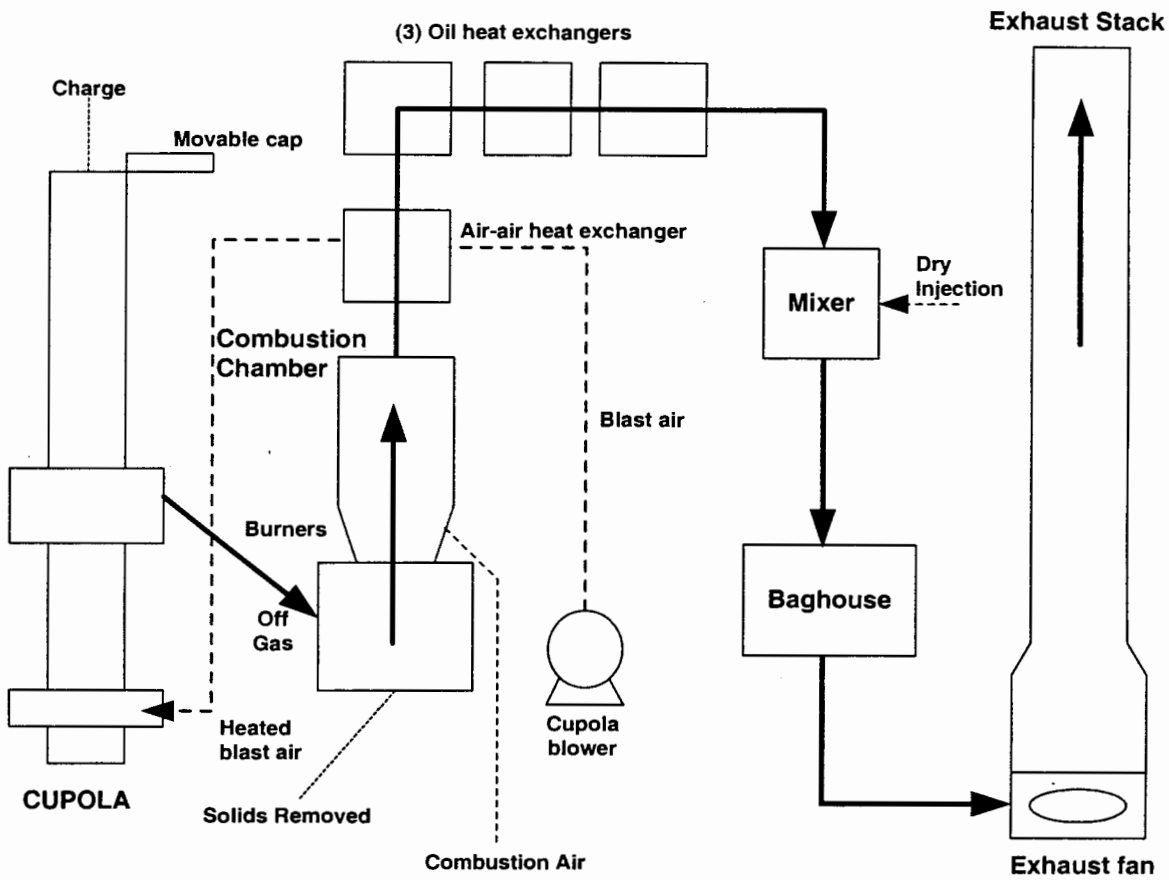


Figure 2-1. Simplified schematic of cupola gas handling system.

After removal of the heavy particles, the gas enters a large combustion chamber where combustion air is introduced and the CO is burned. Two burners are used when necessary to maintain the combustion temperature. The gas leaves the combustion chamber at approximately 1650°F, and enters a dropout chamber where additional heavy particles are removed. The hot gas then passes through an air-to-air heat exchanger (blast air recuperator), followed by a series of three oil heat exchangers that are used to cool the air. These oil heat exchangers are not currently used for heat recovery. (Modifications may be made in the future to recover and use the heat, such as for heating the building.)

The gas from the heat exchangers is injected with a dry mixture (mostly calcium carbonate and magnesium oxide) in a venturi mixer that increases the gas velocity and suspends the injected particles. Dry injection is used to improve pollutant removal in the baghouse.

During the source test, the dry powder injection was turned off to afford better characterization of uncontrolled emissions and baghouse efficiency for HAP constituents. The gas is then sent to a 10 module, negative pressure baghouse that uses a high temperature fabric designed to withstand temperatures of up to 320°F. The temperature of the gas at this point is typically 280 to 290°F. A fan pulls the gas through the system and discharges the cleaned gases through a stack.

The pressure drop across the baghouse is monitored, and when the pressure drop increases to 6 inches (in.) of water, individual bag house compartments are cycled off-line, the bags are cleaned with a pulses of air, then the compartments are brought back on-line. The baghouse uses plenum pulsing. During testing, each compartment was off line for approximately 8 min for cleaning, with cleaning pulses occurring approximately every 30 seconds (sec) during this interval.

The plant routinely monitors several parameters associated with the cupola, including blast air and oxygen rate, and afterburner air addition rates, as well as temperature at various points in the process. The combined air flow rate through the recuperator and the baghouse system is not directly monitored, but can be estimated from the blast air, oxygen and afterburner air addition rates. During testing, the combined flow rate of offgas was also measured by the test crew at the final stack sampling location. The plant also records the amount of each type of material added to the cupola by the automatic skip buckets for each charge load. The composition of a typical charge is given in Table 2-1 and contains approximately 4 tons of iron. The iron includes remelt from the foundry, steel scrap, and pig iron.

TABLE 2-1. TYPICAL CUPOLA CHARGE MATERIALS

Material	Typical range (lbs/charge)[a]
Remelt from foundry[b]	3,500 to 4,500
Steel scrap[b]	3,200 to 4,000
Pig iron[b]	600 to 1,400
Silicon bricks	70 to 105
Blend bricks (Si, Mn, Cr)	260 to 300
Silicon carbide	210 to 250
Coke	500 to 900
Limestone	280 to 300

[a] Typical range observed during the test days.
[b] Remelt, steel scrap, and gray iron bricks are the sources of iron and total 8,700 lbs (4.35 tons) per charge.

2.2.2 Pouring, Cooling and Shakeout

The plant has four lines for pouring, cooling, and shakeout. Silica sand, bentonite, and seacoal constitute the molding sand, which is recycled about 50 times prior to disposal in a monofill. Resins and a catalyst are used to produce warmbox cores. Some of the company's cast products use cores, and others do not. During the source test, cores were not being used on any of the lines. The lines are all similar except that Line 4, which is designed to handle larger molds, has an automatic pouring station and the other lines do not. Line 4 is typically used for casting the larger size parts.

Pouring emissions are not captured at any of the four pouring stations. Cooling emissions are captured by hoods that cover the entire cooling line prior to shakeout. The shakeout operation is totally enclosed and evacuated to capture the emissions. After shakeout, the parts are transferred to a casting cooling house where they are placed on a metal "tree." The parts then proceed to a "spinner house" and are shot blasted to remove residual sand. The spinner house is also evacuated to the duct that removes emissions from shakeout. The captured emissions from shakeout and cooling are sent to a baghouse for gas cleaning. There are three baghouse systems; each system predominantly receives emissions vented from a single line, but a few of the vents from a given line are routed to another line's baghouse system. Consequently, controlled emissions represent contributions from multiple lines and multiple processes.

The ductwork for the cooling lines are interconnected with either other cooling lines or shakeout enclosure ductwork. Therefore, it is impossible to get a representative sample for cooling emissions that could be attributed to an entire cooling line. The least amount of interconnection was on Line 4, so it was selected for emissions testing. The first third of the cooling section of Line 4 was ducted to a single vent that had a long, straight vertical section before connecting with other ductwork. Ports were installed in this straight section of the vent, so that the uncontrolled emissions from the first third (approximately 20-25 min) of the cooling line could be measured. The shakeout enclosure ductwork has a short vertical rise, then elbows to a horizontal section where it is tied to the vent from the spinner housing and the last hood from the cooling line. Ports were installed in the short vertical duct from the shakeout enclosure approximately one foot (ft) above the roof of the enclosure prior to the point where the ducts from the spinner house and the end of the cooling line join the shakeout enclosure duct. This point represents uncontrolled emissions from the shakeout operations.

2.3 SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING SOURCE TEST

2.3.1 Process Operating Data for Cupola Melting Operations

Testing of the melting operations was conducted over a three-day period. Single test runs were performed on September 8th and 9th, and two runs were performed on September 10th. During testing, process information was collected from the operating room's computer control panel. Process information collected included cupola charging data, process chemistry, gas flow rates, temperatures, baghouse pressure drop, and cupola stack opacity.

Table 2-2 and Figure 2-2 present metal charging rates for the cupola during the three days of testing. Table 2-2 and Figure 2-2 show that the average metal production rate for September 8th was higher than on the 9th or 10th. Table 2-2 and Figure 2-2 also show that hourly production rates varied significantly within a given day.

TABLE 2-2. SUMMARY OF CUPOLA CHARGING DURING THE TEST DAYS

9/08/97		9/09/97		9/10/97	
Time period	Tons charged	Time period	Tons charged	Time period	Tons charged
				7:01-7:56	48
		8:01-8:56	47	8:03-8:54	35
		9:01-9:56	52	9:01-9:59	49
		10:02-10:46	42	10:04-10:55	47
11:02-11:57	45	11:03-11:50	35	11:01-11:57	54
12:00-12:54	56	12:00-12:51	26	12:03-12:59	59
13:00-13:58	59	13:00-13:56	58	13:03-13:41	40
14:03-14:55	51	14:06-14:55	30	14:02-14:58	35
15:00-15:57	55			15:01-15:28	32
				16:13-16:56	45
				17:00-17:56	51
				18:01-18:57	56
				19:02-19:58	55
Average rate (tons/hr)	53.2	Average rate (tons/hr)	41.4	Average rate (tons/hr)	46.6

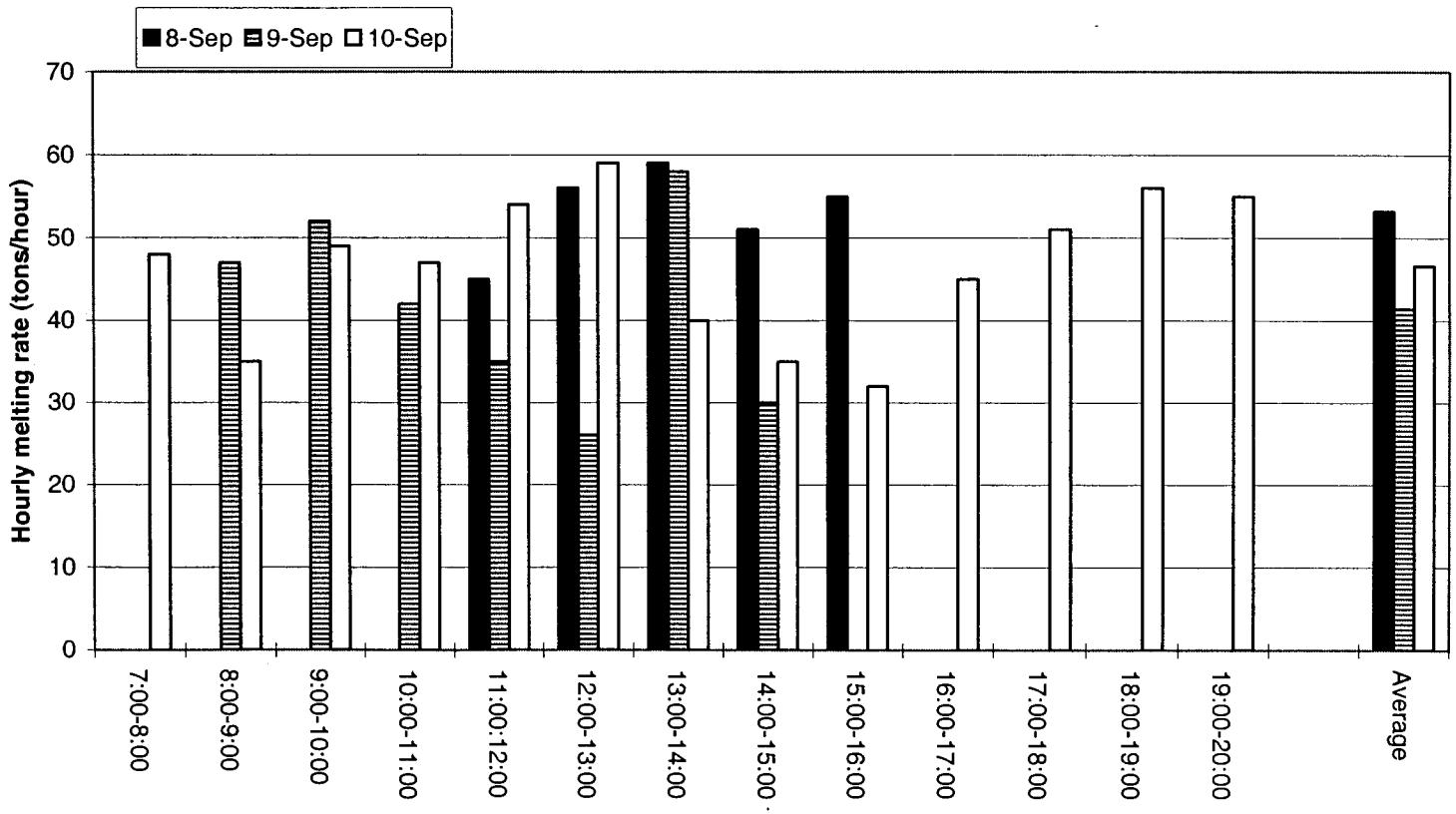


Figure 2-2. Hourly melting rates during cupola testing.

Table 2-3 presents average gas flow rates and temperatures for several locations in the cupola flue gas system on September 8th, 9th, and 10th. As can be seen from Table 2-3, the average blast rate was lowest on September 9th, and the cupola typically operated with oxygen addition on that day. On September 8th and 10th, the average blast rate was higher, and oxygen addition was not used. The average baghouse pressure drop was lower on September 9th than on September 8th or 10th, (2.8 in. of water versus 4.4 in. of water), and the average opacity was higher on September 9th than September 10th (5.7 percent versus 2.8 percent).

TABLE 2-3. PROCESS DATA DURING THE DAYS OF CUPOLA TESTING

Process Parameter	Average Value on Testing Date		
	September 8	September 9	September 10
Cupola process air flow information			
Blast rate (scfm)	14,794	9,131	13,665
Oxygen addition (on/off)	off	on	off
Temperature in (F)	1,156	1,067	1,101
Temperature out (F)	330	232	297
Afterburner air flow information			
Primary air (scfm)	5,271	2,764	4,969
Secondary air (scfm)	5,893	2,823	5,476
Cooling air (scfm)	2,794	2,300	2,792
Temperature out (F)	1,717	1,639	1,668
Baghouse information			
Temperature in (F)	297	281	299
Pressure drop (inches H2O)	4.4	2.8	4.4
Opacity (%)	Not Recorded	5.7†	2.8†

†Average of opacity readouts recorded every 15 minutes; opacity readouts are 6-minute averages from KVB EPA-2 stack mounted opacity monitor.

Continuous records of blast air flow rates are presented in Figure 2-3. Blast air rates were significantly reduced (i.e., the cupola was placed "on relief") for varying lengths of time on September 9th and 10th. The specific times when the cupola was on relief are listed in Table 2-4.

TABLE 2-4. PERIODS WHEN CUPOLA WAS "ON RELIEF" DURING TESTING

September 8	September 9	September 10
Continuous blast.	10:54 - 11:09	13:51 - 14:04
		14:11 - 14:28
		15:27 - 16:03
		16:06 - 16:08

Table 2-5 presents average process iron chemistry values (from cupola) for September 9th and 10th. Although process chemistry values were not recorded on September 8th, average trace metal impurity levels were typical on all three days.

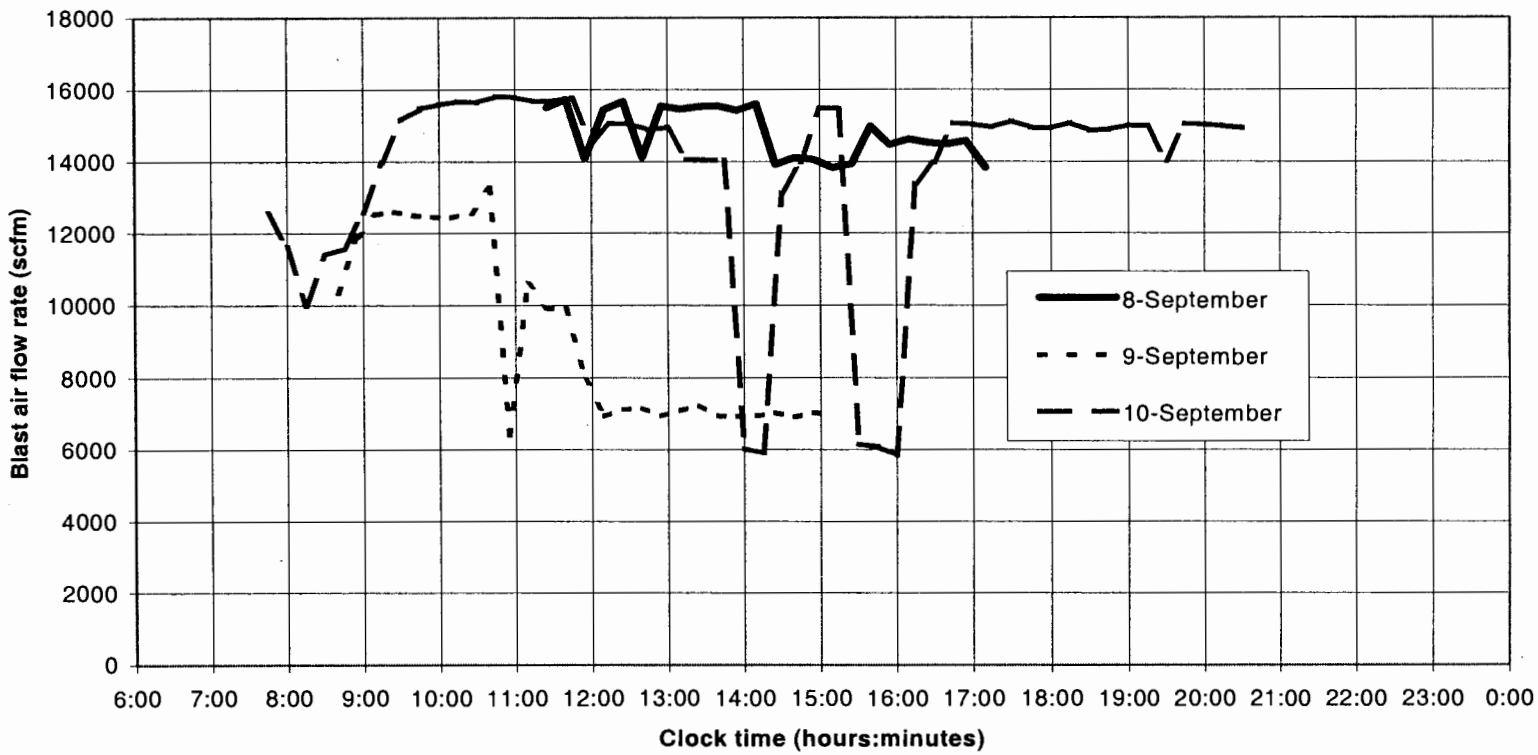


FIGURE 3-3. BLAST AIR FLOW RATE THROUGH CUPOLA.

Figure 2-3. Blast air flow rate through cupola.

TABLE 2-5. PROCESS IRON CHEMISTRY AT CUPOLA[a]

Element	Concentration (%)	
	September 9	September 10
ElCe (Elemental Carbon Equivalents)	3.665	4.045
Calc C (Calculated Carbon)	2.470	3.450
Si	1.050	4.490
Mn	0.350	0.595
P	0.055	0.042
S	0.070	0.090
Ni	0.045	0.090
Mo	0.105	0.025
Cr	0.205	0.240
Cu	0.114	0.230
Al	0.008	0.009
Ti	0.009	0.011
Sn	0.005	0.009
Mg	0.006	0.001
V	0.006	0.011
Pb	0.001	0.002

[a] The process chemistry values reported here are considered typical; the process chemistry values were not specifically recorded on September 8, but were also considered typical.

2.3.2 Process Operating Data for Pouring, Cooling and Shakeout Operations

Emissions from cooling (first third section) and shakeout were measured on September 5, 1997. Line 4 employs an automated molding machine. Testing was conducted only when the entire mold line was filled with recently poured molds. During the day of the source test of the cooling and shakeout operations, Line 4 was used to cast brake drums. Each mold produced two brake drums, and used 189 pounds (lb) of poured metal. For the test day, 249 molds per operating hour were produced. Each mold contains 1393 lb of green sand (lake sand, sea coal, and bentonite), so the molds had a sand to metal ratio of 7.35:1.

The properties of the molding sand measured during the test day are given in Table 2-6. A bonding agent was added to the sand in the amount of 38.1 lb of bond per ton of sand mulled.

The bonding agent is a dry mixture of coal, brittle asphalt, cellulose, bentonite, starch, and cereal. The material safety data sheet for the product indicates no volatile components, and no hazardous ingredients other than coal dust and crystalline quartz.

TABLE 2-6. TYPICAL RESULTS FROM GREEN SAND ANALYSIS

Property	Value
Moisture (%)	3.5
Clay (%)	8.7
Loss on ignition (%, at 1800°F)	7.8
Volatile content (%, at 900°F)	4.0

3.0 TEST LOCATIONS AND GAS COMPOSITION

Figure 3-1 is a schematic showing an overview of the cupola gas handling system that presents the locations of both cupola test points. The baghouse inlet (location "A") and the outlet stack (location "B") were sampled concurrently.

Figure 3-2 is a schematic showing a closer view of the cupola test locations. Location "A" was at the duct leading to the baghouse, and location "B" was at the exhaust stack following the baghouse.

Figure 3-3 is a schematic with a view of test locations at the mold cooling and mold shake-out housing. The sample location of the mold cooling line was the collector duct for the first several cooling line vents. The shake-out housing was sampled at the duct drawing from the enclosure.

3.1 BAGHOUSE OUTLET - STACK

The test ports on the stack are located at about 70 ft above ground level. Access to the stack ports is at roof level, which can be reached by a ladder on the baghouse. Test ports on the 7 ft 9 in.-diameter stack allowed for concurrent FTIR and manual sampling.

3.2 BAGHOUSE INLET DUCT

The test ports on the inlet duct are located at roof level about 70 ft above the ground. Ports allowed simultaneous testing by both FTIR and manual methods. The baghouse inlet and outlet were sampled by FTIR concurrently using a dual line extractive sampling system.

3.3 MOLD COOLING LINE

Two ports were utilized on the vertical duct that collects the emissions from the first seven take-off vents over the cooling line conveyer following the pouring station. That vertically-oriented duct is inside the main facility. Sampling was conducted at a height of approximately 30 ft above the facility floor to obtain volumetric flow, diluent, moisture, and FTIR data across the diameter of the duct interior. A dual-line system was used to conduct concurrent testing at the mold cooling line and shake-out housing.

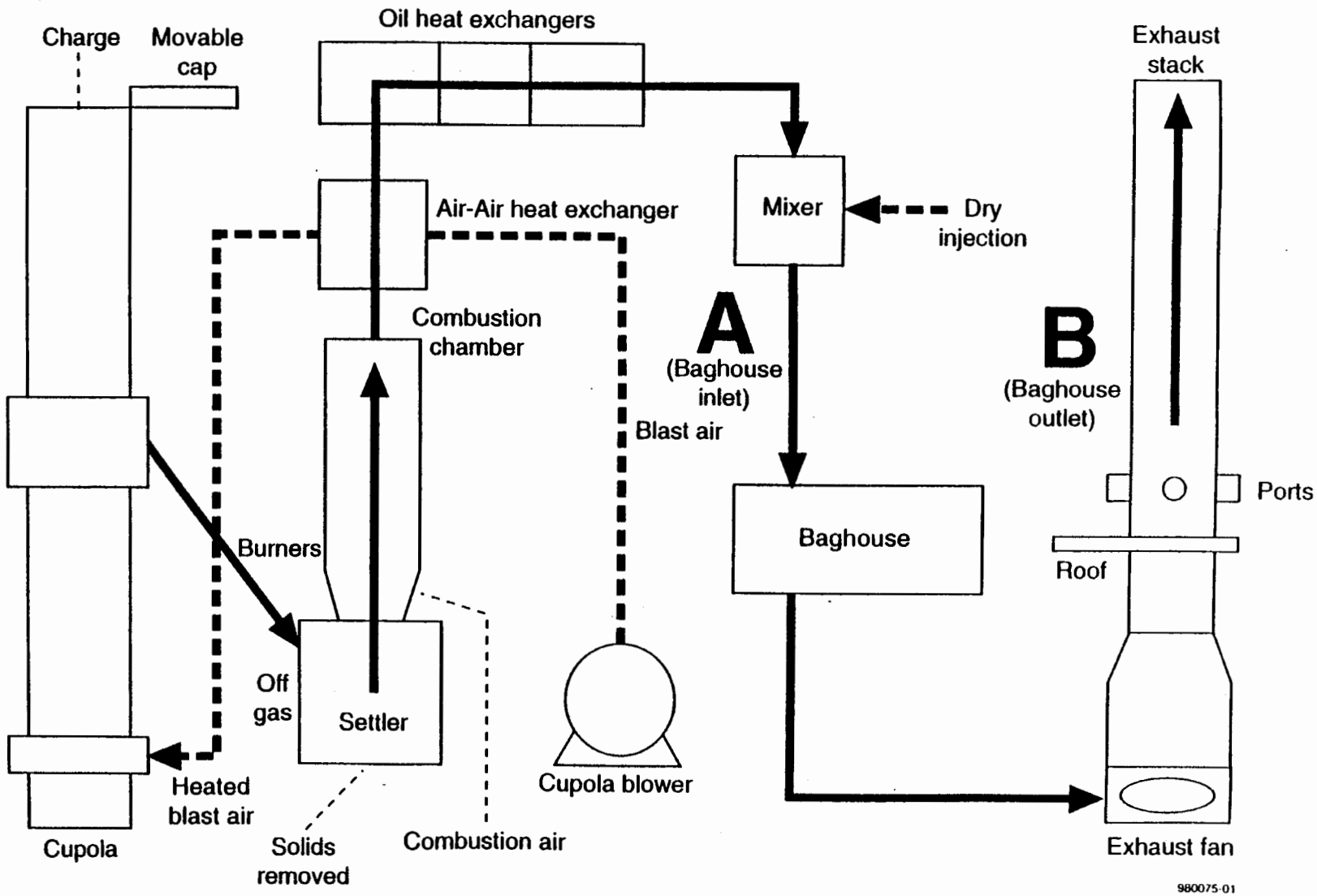


Figure 3-1. Schematic of cupola gas handling system, sampling points A and B.

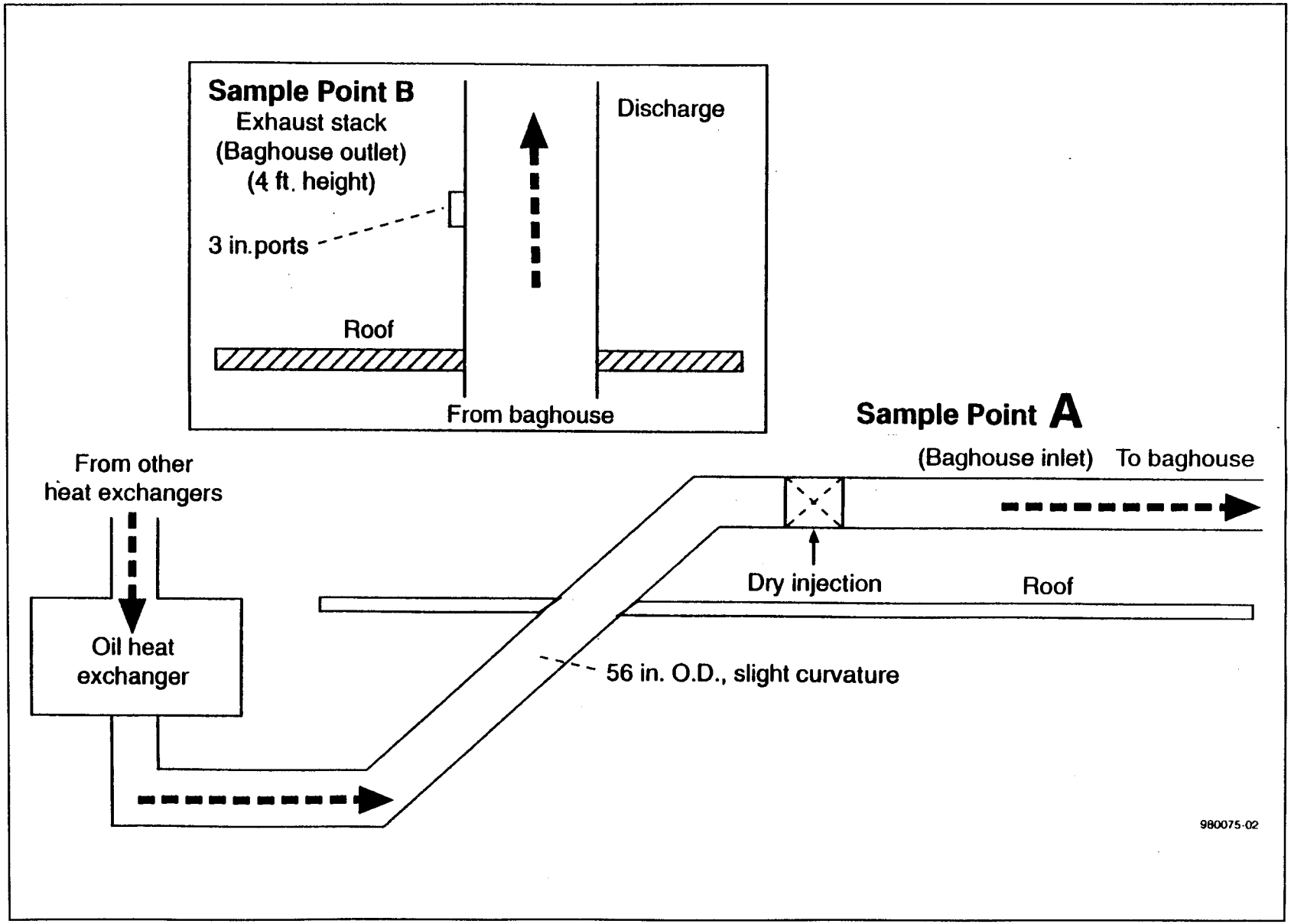


Figure 3-2. Schematic of baghouse inlet and baghouse outlet. Sampling points A and B, respectively.

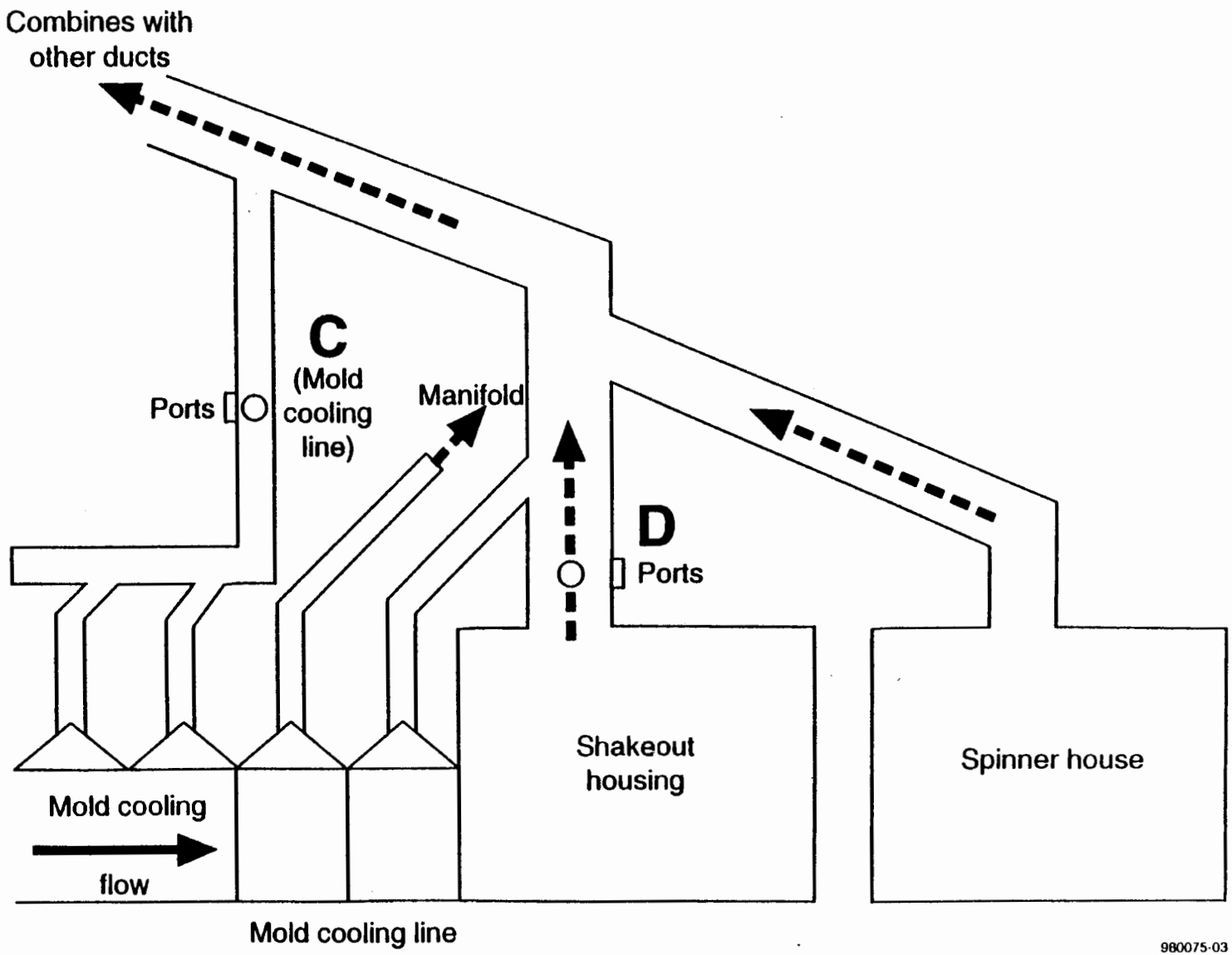


Figure 3-3. Schematic of mold cooling and mold shake-out gas handling system; sampling points C and D.

3.4 MOLD SHAKE-OUT HOUSING

The mold shake-out housing and its ducting system are located inside the main facility. Two 3-in. test ports were installed and utilized on the vertical portion of the duct. At a height of approximately 25 ft above the facility floor, sampling was conducted to obtain volumetric flow, diluent, moisture, and FTIR data across the diameter of the duct interior.

3.5 VOLUMETRIC FLOW

Table 3-1 summarizes the gas composition and flow data for the mold cooling and mold shake-out housing. Measurements for velocity, flow, and oxygen and carbon dioxide concentrations were conducted and calculated following EPA Test Methods 1, 2, and 3B referenced in *40 CFR Part 60, Appendix A*. Moisture content of the stack gas was calculated using wet bulb/dry bulb measurements. Records of volumetric flow data are located in Appendix A.

Table 3-2 summarizes the gas composition and flow data provided by PES for the cupola test locations. As part of their manual testing, PES provided volumetric flow rates, moisture content, gas molecular weight, etc.; therefore, MRI did not conduct these tests.

TABLE 3-1. FLOW DATA AT WAPAUCA MOLD COOLING AND SHAKE-OUT HOUSING PROCESSES[a]

Location Date	Mold Cooling Line 05-Sep-97	Shake-out Housing 05-Sep-97
Carbon Dioxide, %	0.0	0.0
Oxygen, %	20.9	20.9
Moisture Content, %	2.6	5.4
Gas Stream Velocity, fps	57.6	81.1
Volumetric Flow Rate, dscfm	19,399	26,576
Volumetric Flow Rate, dscmm	549.0	753
Stack diameter, in.	34.25	34.25
Stack area, ft^2	6.4	6.4

[a] Flow data uncorrected for Absolute Pressure - This permits a variance of +/-1% in volumetric flow.

TABLE 3-2. CUPOLA BAGHOUSE INLET AND OUTLET GAS COMPOSITION AND FLOW SUMMARIES

Cupola Test Data[a]			
Run Number	1	2	3
Date	09-Sep-97	10-Sep-97	10-Sep-97
Baghouse Inlet			
Oxygen, %	10.9	9.5	8.8
Carbon Dioxide, %	10.8	11.6	12.4
Moisture Content, %	2.5	2.8	2.4
Volumetric Flow Rate, dscfm	26,800	38,200	38,500
Volumetric Flow Rate, dscmm	759	1,080	1,090
Baghouse Outlet (Stack)			
Oxygen, %	12.7	11.0	11.0
Carbon Dioxide, %	8.8	10.1	10.0
Moisture Content, %	4.1	2.6	2.6
Volumetric Flow Rate, dscfm	33,967	48,700	48,933
Volumetric Flow Rate, dscmm	962	1,380	1,383

[a] Data provided by PES.

4.0 RESULTS

4.1 TEST SCHEDULE

The testing at Waupaca Foundry, Plant No. 5 was completed from September 5 to September 10, 1997. Table 4-1 summarizes the sampling schedule. A complete record of all FTIR sampling is in Appendix B. The FTIR sampling at the cupola locations was coordinated with the manual sampling conducted by PES. The FTIR sampling at the mold cooling line and the mold shake-out housing were conducted independently.

TABLE 4-1. TEST SCHEDULE AT WAUPACA FOUNDRY

Date	Task	Location [a]
9/4/97	Arrive on site and set up at mold cooling and shake-out.	Mold cooling ("C") and Shake-out ("D")
9/5/97	Mold cooling and shake-out test run w/ FTIR. 15:23 - 19:11	
9/6/97	Relocation to cupola testing area	
9/8/97	Complete setup at cupola. Test Run 1 w/ FTIR. 12:56 - 17:05	Baghouse inlet ("A") and outlet ("B") at Cupola
9/9/97	Test Run 2. FTIR in conjunction with manual methods by PES. 9:25 - 13:56	
9/10/97	Test Run 3. FTIR in conjunction with manual methods by PES. 8:15 - 12:33 Pack equipment and depart site	

[a] Location descriptions are in Section 3.

4.2 FIELD TEST PROBLEMS AND CHANGES

The cupola gas at Waupaca contained high concentrations of both water vapor and (carbon dioxide) CO_2 with respect to other compounds. Analyte spiking for quality assurance was conducted using toluene and formaldehyde vapor. The CO_2 spectrum interfered with the strongest toluene infrared band near 730 cm^{-1} so the weaker toluene absorbance, in the analytical region 2,850-3,100 cm^{-1} range, was used for the analysis. The presence of other aliphatic hydrocarbon species also contributed to the total infrared absorbance in this 2,850-3,100 cm^{-1} region.

The analyte spiking for the inlet sample system was introduced into the sample line at the junction 50 ft downstream of the probe before and after Run 1 at the cupola. The plumbing was modified for Run 2 and Run 3 to allow introduction of the spiking analyte at the inlet probe.

4.3 FTIR RESULTS

The FTIR results and the mass emissions are summarized in Tables 1-1 and 1-2. The complete FTIR concentration results are presented in Appendix B in Tables B-1 to B-4.

4.3.1 Mold Cooling Line and Shake-out Housing

The FTIR results at the cooling and shake-out housing ducts are summarized in Table 1-1. The complete results for all of the samples from these locations are presented in Tables B-1 and B-2. The compounds detected consisted primarily of light hydrocarbon species methane and ethylene. Some higher molecular weight hydrocarbon species were also detected. In the draft report, the heavier hydrocarbons were reported as hexane. The revised analysis of the cooling and shakeout spectra included reference spectra of additional hydrocarbon compounds. The additional reference spectra were measured in the laboratory by MRI. Additional explanation of these spectra is provided in Section 1.2. Reference spectrum documentation is provided in Appendix B.

Both toluene and formaldehyde were included in the analysis because some samples were spiked with each of these compounds. Formaldehyde was not detected in the unspiked samples. Toluene was detected in unspiked samples at the cooling and shake-out locations, but the uncertainties were relatively high (Table 1-1).

4.3.2 Baghouse Inlet and Outlet

The emissions were similar at both locations and are summarized in Table 1-2. The complete concentration results are in Tables B-3 and B-4. The samples contained moisture, CO_2, hydrogen chloride (HCl), and methane. Some samples were spiked with either toluene or formaldehyde, but neither toluene nor formaldehyde was detected in any of the unspiked samples.

4.4 ANALYTE SPIKE RESULTS

The revised cooling and shakeout spike results are slightly different from the draft report results due to the effect of using the additional hydrocarbon reference spectra. A permeation tube saturated with paraformaldehyde was heated to produce a vapor of the formaldehyde monomer. A steady state concentration of formaldehyde vapor was maintained with a temperature controller

set at 100°C and with a controlled flow of carrier gas. During spiking the carrier gas was 4.01 ppm SF_6 in nitrogen.

The inlet and outlet locations were also spiked with toluene from a cylinder standard of 60 ppm toluene in nitrogen (Scott Specialty Gases, ± 2 percent). The toluene spike flow passed through a mass flow meter and into the spike line where it was preheated before injection into the sample at the back of the sample probe. Section 5.3 gives additional description of the analyte spike QA procedure.

The formaldehyde spike results for the cooling and shake-out locations are presented in Tables 4-2 and 4-3. The toluene spike results for the cooling and shake-out are summarized in Tables 4-4 and 4-5. The formaldehyde spike results at the inlet and outlet are summarized in Tables 4-6 and 4-7. The toluene spike results at the baghouse inlet and outlet are summarized in Tables 4-8 and 4-9. The toluene and formaldehyde spike standards were quantitatively mixed before the spike mixture was introduced to the sample stream. The analytical results for each spiked analyte are presented separately. Section 5.3 gives a discussion of the procedure for determining the analyte standard concentrations in the spike mixtures. The spike standard concentrations are presented in Table 5-2. The spiked sample spectrum file names are identified in Tables 4-2 to 4-9. These correspond to the sample file names in Section 5.3, where the formaldehyde and toluene spike standard concentrations are given for each spike mixture.

Table 4-10 compares measured band areas of the EPA toluene reference spectra (deresolved to 2.0 cm^{-3}) and spectra of samples taken directly from the 60 ppm toluene cylinder standard. The cylinder standard spectrum was measured at the Waupaca test site. The band area comparison differs from the comparison of the certified concentrations by about 35 percent. For a given concentration, (ppm-M)/K, the infrared absorbance in the cylinder standard spectra is about 35 percent greater than the absorbance in the EPA library spectra. Therefore, the library spectra calculate a toluene concentration that is 35 percent lower than that calculated using the cylinder standard spectra. Tables 4-4, 4-5, 4-8, and 4-9 present the toluene spike recoveries using both the library spectra and the cylinder standard spectra.

A similar effect was observed in some other field tests using another toluene cylinder standard. One possibility is that there was a systematic error in the original toluene library reference spectra. This could be assessed by evaluating several toluene gas standards from different sources and doing a comparison similar to that shown in Table 4-10.

The above observation is compound specific, and the information in Table 4-10 does not apply to the measurements of other analytes. The deresolved calibration transfer standard (CTS) spectra give a path length result that is consistent with the observed number of laser passes and the instrument resolution. The disagreement is also not related to the deresolution of the toluene spectra because the band areas in the original 0.25 cm^{-1} toluene spectra are nearly equal to the band areas in the deresolved 2.0 cm^{1} versions of these spectra.

TABLE 4-2. FORMALDEHYDE SPIKE RESULTS FROM THE MOLD COOLING PROCESS

Files	Average Formaldehyde Concentration			Average SF_6 Concentration			DF	Cexp	Δ	% Recovery
	spike	unspike	(calc)	spike	unspike	(calc)				
Coosp101, 102,	12.5	0.0	12.5	0.442	0.000	0.442	4.7	13.2	-0.6	95.0
Coosp107	12.8	0.0	12.8	0.545	0.000	0.544	3.8	16.2	-3.5	79.0

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated formaldehyde concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to Cexp – formaldehyde(calc).

TABLE 4-3. FORMALDEHYDE SPIKE RESULTS FROM THE WAUPACA SHAKE-OUT HOUSING PROCESS

Files	Average Formaldehyde Concentration			Average SF_6 Concentration			DF	Cexp	Δ	% Recovery
	spike	unspike	(calc)	spike	unspike	SF_6 (calc)				
Shksp101, 102	14.7	0.0	14.7	0.472	0.000	0.472	4.4	14.0	0.6	104.6
Shksp107	18.7	0.0	18.7	0.605	0.000	0.605	3.5	18.0	0.7	103.8

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated formaldehyde concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to Cexp – formaldehyde(calc).

TABLE 4-4. TOLUENE SPIKE RESULTS FROM THE MOLD COOLING PROCESS

Files	Average Toluene Concentration			Average SF_6 Concentration			DF	Cexp	Δ	% Recovery	% R[a]
	spike	unspike	(calc)	spike	unspike	(calc)					
coosp101, 102	20.4	18.9	1.5	0.442	0.000	0.442	4.7	8.4	-6.9	17.6	11.3
coosp107	16.5	16.4	0.1	0.545	0.000	0.545	3.0	10.4	-10.3	0.7	0.45

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated toluene concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to Cexp – toluene(calc). The toluene % recoveries were obtained using EPA reference spectra of toluene.

[a] %R is the calculated percent recovery obtained if the spectra of the 60 ppm toluene cylinder standard are used in the analysis (see Table 4-10).

TABLE 4-5. TOLUENE SPIKE RESULTS FROM THE SHAKE-OUT HOUSING PROCESS

Files	Average Toluene Concentration			Average SF_6 Concentration			DF	Cexp	Δ	% Recovery	% R[a]
	spike	unspike	(calc)	spike	unspike	(calc)					
shksp101, 102	11.3	0	11.3	0.472	0.000	0.472	4.4	9.0	2.3	125.7	81.0
shksp107	9.3	0	9.3	0.605	0.000	0.605	3.5	11.5	-2.2	80.7	52.0

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated toluene concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to the difference Cexp – toluene(calc). The toluene % recoveries were obtained using EPA reference spectra of toluene.

[a] %R is the calculated percent recovery obtained if the spectra of the 60 ppm toluene cylinder standard are used in the analysis (see Table 4-10).

TABLE 4-6. SUMMARY OF FORMALDEHYDE SPIKE RESULTS AT THE CUPOLA BAGHOUSE INLET

Files	Average Formaldehyde Concentration				Average SF_6 Concentration						
	Date	spike	unspike	(calc)	spike	unspike	(calc)	DF	Cexp	Δ	% Recovery
insp101, 102	9/8/97	17.5	0.0	17.5	0.561	0.000	0.561	3.9	12.8	4.7	137
insp117		14.6	0.0	14.6	0.484	0.000	0.484	3.0	11.4	3.2	128
insp201	9/9/97	16.4	0.0	16.4	1.175	0.000	1.175	3.3	13.2	3.2	124
insp205		17.3	0.0	17.3	0.619	0.000	0.619	3.3	13.2	4.1	131
insp301	9/10/9	16.2	0.0	16.2	0.595	0.000	0.595	3.5	12.4	3.9	131
insp312		31.9	0.0	31.9	1.141	0.000	1.141	2.8	26.7	5.2	120

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated formaldehyde concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to Cexp – formaldehyde(calc).

TABLE 4-7. SUMMARY OF FORMALDEHYDE SPIKE RESULTS AT THE CUPOLA BAGHOUSE OUTLET

Files	Average Formaldehyde Concentration				Average SF_6 Concentration						
	Date	spike	unspike	(calc)	spike	unspike	(calc)	DF	Cexp	Δ	% Recovery
outsp101	9/8/97	15.0	0.0	15.0	0.450	0.000	0.450	4.9	10.3	4.7	145
outsp111		12.9	0.0	12.9	0.465	0.000	0.465	3.2	11.0	2.0	118
outsp201	9/9/97	13.4	0.0	13.4	0.923	0.000	0.923	4.2	10.3	3.0	129
outsp208		14.1	0.0	14.1	0.497	0.000	0.497	4.0	10.6	3.5	133
outsp301	9/10/9	14.0	0.0	14.0	0.504	0.000	0.504	4.3	9.9	4.0	141
outsp316	9/10/9	25.7	0.0	25.7	0.904	0.000	0.904	2.4	31.2	-5.4	83

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated formaldehyde concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to Cexp – formaldehyde(calc).

TABLE 4-8. SUMMARY OF TOLUENE SPIKE RESULTS AT THE CUPOLA BAGHOUSE INLET

Files	Date	Average Toluene Concentration spike	unspike	(calc)	Average SF_6 Concentration spike	unspike	(calc)	DF	Cexp	Δ	%Recovery	% R[a]
insp101,	9/8/97	10.5	0.0	10.5	0.561	0.000	0.561	3.9	7.6	2.9	138.3	88.8
Insp117		24.4	0.0	24.4	0.484	0.000	0.484	3.0	17.4	7.0	140.3	90.0
insp205	9/9/97	14.2	0.0	14.2	0.619	0.000	0.619	3.3	9.8	4.4	144.8	92.9
insp301	9/10/9	12.5	0.0	12.5	0.595	0.000	0.595	3.5	7.9	4.6	157.9	101.3

Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated toluene concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to the difference Cexp – toluene(calc). The toluene % recoveries were obtained using EPA reference spectra of toluene.

[a] %R is the calculated percent recovery obtained if the spectra of the 60 ppm toluene cylinder standard are used in the analysis (see Table 4-10).

TABLE 4-9. SUMMARY OF TOLUENE SPIKE RESULTS AT THE CUPOLA BAGHOUSE OUTLET

Files	Date	Average Toluene Concentration spike	unspike	(calc)	Average SF_6 Concentration spike	unspike	(calc)	DF	Cexp	Δ	% Recovery	% R[a]
outsp101	9/8/97	7.6	0.0	7.6	0.450	0.000	0.450	4.9	6.1	1.5	124.5	79.9
outsp111	9/9/97	21.2	0.0	21.2	0.465	0.000	0.465	3.2	16.7	4.5	126.9	81.4
outsp301	9/10/9	10.0	0.0	10.0	0.504	0.000	0.504	4.3	6.3	3.6	157.4	101.0

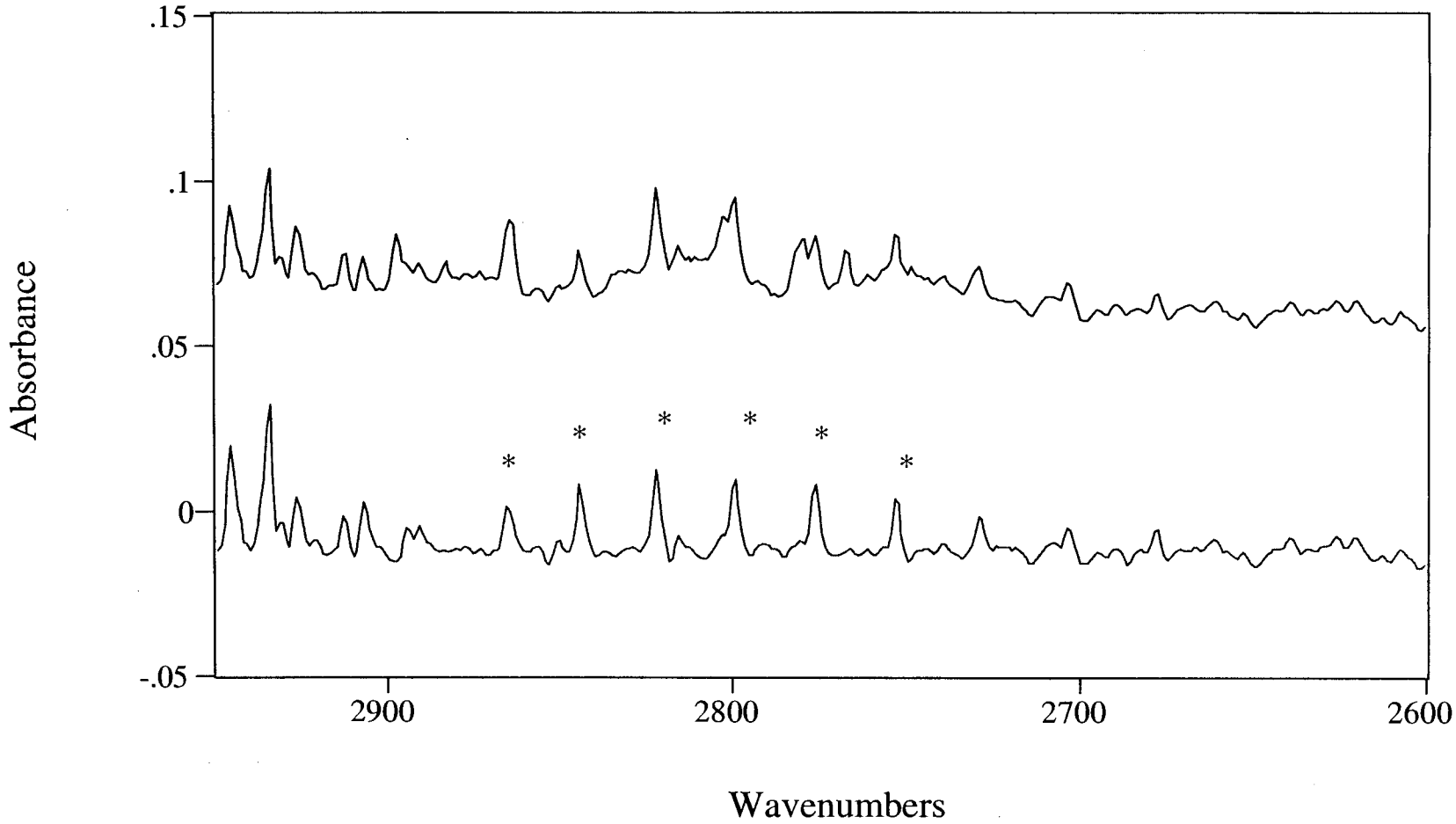
Calc is equal to the difference, spike – unspike for the analyte or for SF_6. Cexp is the calculated toluene concentration at 100 percent recovery in the spiked samples. DF is the dilution factor calculated from the SF_6 concentration. Δ is equal to the difference Cexp – toluene(calc). The toluene % recoveries were obtained using EPA reference spectra of toluene.

[a] %R is the calculated percent recovery obtained if the spectra of the 60 ppm toluene cylinder standard are used in the analysis (see Table 4-10).

TABLE 4-10. COMPARISON OF FTIR SPECTRA OF SAMPLES FROM TOLUENE (60 ppm) CYLINDER TO EPA TOLUENE REFERENCE SPECTRA[a]

Toluene Spectra	Source	Band Area	Region (cm^{-1})	Spectra comparison based on band areas		Comparison of spectra based on standard concentrations		
				Ratio (Ra)[b]	1/Ra	(ppm-m)/K	Ratio (Rc)[c]	1/Rc
153a4ara (2cm-1)	EPA library	23.4	3160.8 - 2650.1	5.4	0.184	4.94	4.8	0.210
153a4arc (2cm-1)	EPA library	4.3		1.0	1.000	1.04	1.0	1.000
tol0905a	Waupaca	10.2	3160.8 - 2650.1	2.4	0.423	1.58	1.5	0.655
tol0905b	Waupaca	10.1		2.3	0.427	1.58	1.5	0.655
tol0909ab	Waupaca	10.5		2.4	0.411	1.58	1.5	0.655

[a]The relevant comparison is Rc/Ra for tol0905a,b and tol0909ab, which is about 65 percent.
[b]Ratio of band area to band area of 153a4arc.
[c]Ratio of concentration to concentration of 153a4arc.



The top trace is a spectrum of an outlet sample spiked with formaldehyde, "outsp208." The bottom trace is a spectrum of an unspiked outlet sample in the same run, "19080006." The asterisk (*) indicates features from HCl in the unspiked sample.

Figure 4-1. Example spectra of spiked and unspiked baghouse outlet samples.

5.0 TEST PROCEDURES

The procedures followed in this field test are described in the EPA Method 320 for using FTIR spectroscopy to measure HAP's and the EPA Protocol for extractive FTIR testing at industrial point sources. The objectives of the field test were to use the FTIR method to measure emissions from the processes, screen for HAP's in the EPA FTIR reference spectrum library, and analyze the spectra for compounds not in the EPA library. Concentrations are reported for compounds that could be measured with FTIR reference spectra. Additionally, manual measurements of gas temperature, gas velocities, moisture, CO_2, and O_2 were used to calculate the mass emissions rates. MRI collected data for the mold cooling and the shake-out lines, and PES collected the data at the baghouse inlet and outlet.

5.1 SAMPLING SYSTEM DESCRIPTION

A schematic of the extractive sampling and spiking system is shown in Figure 5-1.

5.1.1 Sample System Components

The sampling system consists of three separate components:

- two sample probe assemblies
- two sample lines and pumps
- a gas distribution manifold cart.

All wetted surfaces of the system are made of unreactive materials, Teflon®, stainless steel, or glass and are maintained at temperatures at or above 300° F to prevent condensation.

The sample probe assembly consists of the sample probe, a pre-filter, a primary particulate filter, and an electronically actuated spike valve. The sample probe is a standard heated probe assembly with a pitot tube and thermocouple. The pre-filter is a threaded piece of tubing loaded with glass wool attached to the end of sample probe. The primary filter is a Balston particulate filter with a 99 percent removal efficiency at 0.1 μm. The actuated spike valve is controlled by a radio transmitter connected to a switch on the sample manifold cart. All sample probe assembly components are attached to or enclosed in an insulated metal box.

The sample lines are standard heated sample lines with three ⅜ in. Teflon tubes in 10, 25, 50, and 100 ft lengths. The pumps are heated, single-headed diaphragm pumps manufactured by either KNF Neuberger or Air Dimensions. These pumps can sample at rates up to 20 liters per minute (Lpm) depending on the pressure drop created by the components installed upstream.

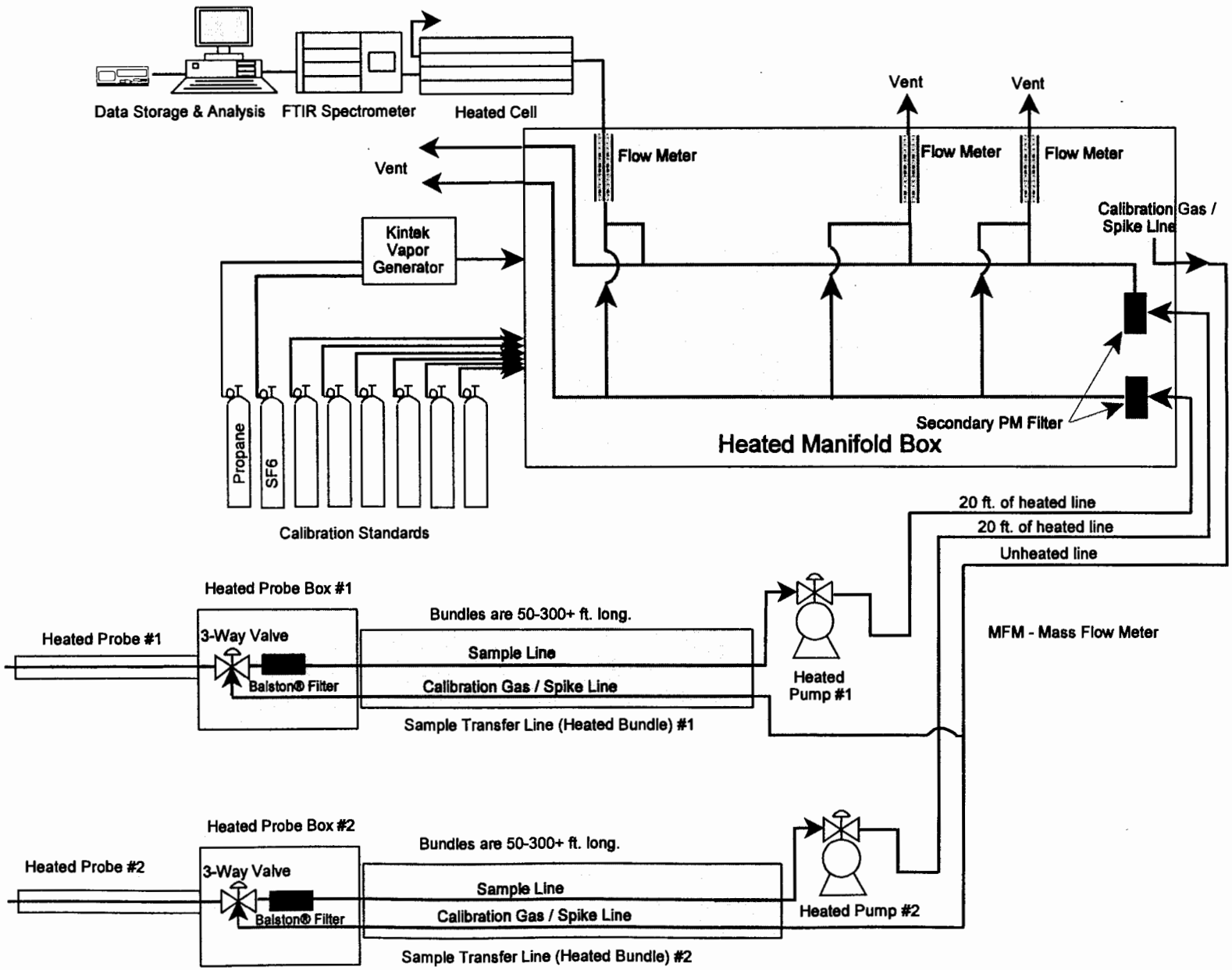


Figure 5-1. Sampling system schematic.

The gas distribution manifold was constructed for FTIR sampling by MRI. It is built onto a cart that can be operated inside the MRI mobile lab or in an alternate location, if necessary. The manifold consists of a secondary particulate filter, control valves, rotameters, back pressure regulators and gauges, and a mass flow controller. The manifold can control two sample gas stream inputs, and eight calibration gases; it has three individual outputs for analyzers. The cart also contains a computer work station and controls for the spike valves and mass flow controller.

5.1.2 Sample Gas Stream Flow

Exhaust gas was withdrawn through the sample probe and transported to the gas distribution manifold. The mold cooling and shake-out processes were each sampled alternately with the two gas handling systems during a single run. The baghouse inlet and outlet were sampled alternately over three runs. Inside the manifold the gas passed through separate secondary particulate filters. Downstream of the secondary filters, a portion of either gas stream could be directed to the FTIR gas cell. The remainder of each gas stream was exhausted through a manifold vent. The baghouse inlet and outlet were sampled alternately (i.e., inlet sample was analyzed for an interval while the outlet sample was exhausted, then outlet sample was analyzed while the inlet sample was vented). A location was selected for analysis using the four-way gas selection valve on the manifold outlet to the instrument. Gas flow to the instrument was regulated with a needle valve on a rotameter at the manifold outlet.

5.2 FTIR SAMPLING PROCEDURES

For each run, two locations were sampled using two separate sample systems that were both connected to the main manifold (Figure 5-1). In the first run, the mold cooling and shake-out housing were sampled together and for three runs the baghouse inlet and baghouse outlet were sampled together. A single FTIR instrument was used to analyze samples from both locations during a test run. The manifold's four-way valves allowed the sample from either of two locations to be directed alternately to the FTIR cell. Sample flow was controlled by a needle valve and measured with a rotameter.

FTIR sampling was conducted using either the batch or the continuous sampling procedures. All data were collected according to the Method 320 sampling procedure, which are described below.

5.2.1 Batch Samples

In this procedure, the 4-way valve on the manifold outlet was turned to divert a portion of the sample flow to the FTIR cell. A positive flow to the main manifold outlet vent was maintained as the cell was filled to just above ambient pressure. The cell inlet valve was then closed to isolate the sample, the cell outlet valve was opened to vent the cell to ambient pressure, the spectrum of the static sample was recorded, and the cell was evacuated for the next sample. This procedure was repeated to collect a desired number of discreet samples.

Batch sampling has the advantage that every sample is independent from the other samples. The time resolution of the measurements is limited by the interval required to evacuate a sample, pressurize the cell, and record a spectrum. All of the calibration transfer standards, and spiked samples were collected using this procedure. Several spectra in each run were also collected in this manner.

5.2.2 Continuous Sampling

The cell was filled as in the batch sampling procedure, but the cell inlet and outlet valves were kept open to allow gas to continuously flow through the cell. The inlet and outlet flows were regulated to keep the sample in the cell at ambient pressure. The flow through the cell was maintained at about 5 Lpm. The cell volume was about 7 liters (L).

The FTIR instrument was automated to record spectra of the flowing sample about every 2 min and the quantitative analysis was automated to measure pollutant concentrations as each spectrum was recorded. The analytical program was revised after the test was completed and all of the spectra were reanalyzed.

This procedure with automated data collection was used during each of the test runs. Because spectra were collected continuously as the sample flowed through the cell, there was mixing between consecutive samples. The interval between independent measurements (and the time resolution) depends on the sample flow rate (through the cell) and the cell volume.

The Time Constant (TC) defined by Performance Specification 15 for FTIR continuous emissions monitoring systems (CEMS), is the period for one cell volume to flow through the cell. The TC determines the minimum interval for complete removal of an analyte from the cell volume. It depends on the sampling rate (R_s in Lpm), the cell volume (V_{cell} in L) and the analyte's chemical and physical properties. Performance Specification 15 defines 5 * TC as the minimum interval between independent samples.

$$TC = \frac{V_{cell}}{R_S} \quad (1)$$

A stainless steel tube ran from the cell inlet connection point to the front interior of the cell. The outlet vent was at the back of the cell so that the flowing sample passed through the greatest portion of the cell volume and minimized the likelihood of a short circuiting flow.

5.3 ANALYTE SPIKING

Since there was little information available about HAP emissions from this source, there was no plan for validating specific HAP's at this test. MRI conducted limited spiking for quality assurance (QA) purposes using a toluene in nitrogen standard and a vapor-generated formaldehyde standard.

5.3.1 Analyte Spiking Procedures

The infrared spectrum is ideally suited for analyzing and evaluating spiked samples because many compounds have distinct infrared spectra.

The reason for analyte spiking is to provide a QA check that the sampling system can transport the spiked analytes to the instrument and that the quantitative analysis program can measure the analyte in the sample gas matrix. If at least 12 (independent) spiked and 12 (independent) unspiked samples are measured, then this procedure can be used to perform a Method 301 validation.[3]

The spike procedure follows Sections 9.2 and 13 of EPA Method 320 in Appendix D. In this procedure a gas standard is measured directly in the cell. This direct measurement is then compared to measurements of the analyte in spiked samples. Ideally, the spike will comprise about 1/10 or less of the spiked sample. The actual dilution depends on the ratio of the sample and spike flow rates. The expected concentration of the spiked component is determined using a tracer gas, in this test SF_6. The SF_6 concentration in the direct sample divided by the SF_6 concentration in the spiked sample(s) is used as the spike dilution factor (DF). The analyte standard concentration divided by DF gives the expected value of the spiked analyte concentration.

5.3.2 Analysis of Spiked Results

5.3.2.1 Determination of Formaldehyde Standard

Formaldehyde vapor was produced by heating a permeation tube filled with solid paraformaldehyde. The tube was placed in a vapor generation oven (Kintek) equipped with a temperature controller and mass flow meter to regulate the carrier gas. The oven was raised to 100°C, and the vapor of the formaldehyde monomer was purged with a continuous flow of a carrier gas. For spiking the carrier gas was a constant flow from the SF_6 cylinder standard (4.01 ppm in nitrogen at ± 2 percent, Scott Specialty Gases). The SF_6 cylinder certification had expired before the test. The SF_6 concentration was confirmed by comparison to spectra of SF_6 from another cylinder. The SF_6 concentration was confirmed to be within 1.5 percent of the certified concentration of 4.01 ppm. When spiking was not performed the formaldehyde vapor was continuously vented using a low flow of nitrogen as the carrier gas. Using this device it was practical to generate a very stable concentration output of formaldehyde. The concentration of this formaldehyde standard was determined with respect to formaldehyde reference spectra in the EPA FTIR spectral library (Table 5-1).

TABLE 5-1. DETERMINATION OF FORMALDEHYDE STANDARD CONCENTRATION

Date	File name of Direct Measurement	Formaldehyde ppm [a]	Uncertainty
9/5/97	FORMAL01	77.3	1.1
9/9/97	FRM0909A	80.0	1.0
	Average –>	78.7	1.1

[a] Measured between 3160.8 and 2650.1 cm^{-1} using EPA reference spectrum 087b4anb, deresolved to 2.0 cm^{-1}. The vapor generation oven was kept at 100°C and the carrier gas flow rate was 1.00 Lpm. Nitrogen was the carrier gas for the direct-to-cell measurements of formaldehyde.

5.3.2.2 Determination of Concentrations in Spike Mixtures

Frequently the output formaldehyde from the vapor generation oven was mixed quantitatively with the toluene standard so that sample stream could be spiked with toluene, SF_6 and formaldehyde simultaneously. Mixing the two spike streams together introduced another dilution factor that had to be accounted for to determine the concentrations of each component of the spike mixture. The concentration of each component in the spike mixtures was determined independently by preparing a separate analytical computer program. The input for the computer

program consisted of reference spectra of each analyte in the mixtures. For SF_6 and toluene spectra the program used spectra of samples taken directly from the cylinder standards and measured in the FTIR cell. For formaldehyde the program used a spectrum in the EPA library. The program was used to analyze spectra of each of the spike mixtures, which were measured directly in the FTIR gas cell. Table 5-2 present the results from this analysis. Table 5-2 also shows the mass flow meter readings used to prepare the spike mixtures, the files names for the direct-to-cell measurements of each mixture, and the file names of the samples that were spiked with each mixture.

The measured concentrations in Table 5-2 were used to determine the percent recoveries in Tables 4-2 to 4-9: the SF_6 concentrations were used to determine the DF, and the toluene and formaldehyde concentrations were combined with DF to determine the C_{exp} and the percent recoveries for those analytes.

5.3.2.3 Determination of Percent Recovery

The expected concentration of the spiked component was determined using the tracer gas, SF_6. In the following discussion the "direct" measurement refers to the measured concentration in the spike mixture before it was added to the sample stream (i.e., the concentrations presented in Table 5-2).

The DF was determined by the ratio of the measured SF_6 concentration in the direct measurement of the spike mixture, $SF_{6(direct)}$, to the measured SF_6 concentration in the spiked samples, $SF_{6(spike)}$.

$$DF = \frac{SF_{6(direct)}}{SF_{6(spike)}} \tag{2}$$

The direct measurement of the analyte concentration in the spike mixture divided by DF gives the expected concentration for a 100 percent recovery of the analyte spike, C_{exp}.

$$C_{exp} = \frac{Analyte_{(direct)}}{DF} \tag{3}$$

where:

$analyte_{(direct)}$ = The concentration of either toluene or formaldehyde from the direct measurement of the spiked mixture (from Table 5-2).

TABLE 5-2. MEASURED ANALYTE CONCENTRATIONS AND MIXING FLOW RATES FOR THE SPIKE MIXTURES

Date	Spiked Sample Files	File name of Direct Measurement	Toluene[a] (ppm)	Toluene[b] (ppm)	SF_6 (ppm)	Formaldehyde (ppm)	Mixing Flow Rates (Lpm)[c]	
							Formaldehyde	Toluene
9/5/97	shksp101, 102, 107	average (sftol2, sftol3)	39.8	25.7	2.090	62.2	1.00	1.00
	coosp101, 102, 107							
9/8/97	outsp101	average (sf6tol4, sftol05)	29.7	19.1	2.191	50.1	1.00	1.00
	insp101, insp102							
	outsp111, insp117	sftol06	53.0	34.2	1.475	34.8	1.00	2.00
9/9/97	insp201, outsp201	sft0909a			3.841	41.8	2.00	xx
	insp205, outsp208	sft0909b	31.8	20.5	2.013	44.1	1.00	1.00
		average (sft0909a, sft909b)[d]				43.0		
9/10/97	insp301, outsp301	sft0910a	27.6	17.8	2.069	43.1	1.00	1.00
	outsp316, insp312	sft0910b			3.226	75.5	2.00	1.00

[a] Toluene concentration determined using EPA reference spectrum "153a4arc" deresolved to 2.0 cm^{-1}.

[b] Toluene concentration determined using spectrum of sample taken directly from 60 ppm toluene cylinder standard. See Section 3-4 and Table 15 for additional explanation.

[c] The mass flow meter on the Kintek (formaldehyde) vapor generator was used to control the SF_6 carrier gas flow. A separate mass flow meter was used to control the flow from the 60 ppm toluene gas standard.

[d] The formaldehyde concentration in these two mixtures can be averaged because increasing the carrier gas flow from 1.0 Lpm to 2.0 Lpm dilutes the formaldehyde concentration in the vapor generation output. This dilution is similar to using a 1.0 Lpm carrier gas flow and then mixing the vapor generation output with a 1.0 Lpm flow from the toluene standard.

The actual spike recovery in Tables 4-2 to 4-9 is the percent difference between the measured analyte concentrations in the spiked samples and C_{exp}.

$$\% \text{ Recovery} = \frac{\text{calc}}{\text{Cexp}} \times 100 \tag{4}$$

where:

calc = the analyte concentration in the spike samples, spiked – unspiked.

5.4 ANALYTICAL PROCEDURES

Analytical procedures in the EPA FTIR Protocol [2] were followed for this test. A computer program was prepared with reference spectra shown in Table 4-7. The computer program[6] used mathematical techniques based on a K-matrix analysis.[7]

Initially, the sample spectra were reviewed to determined appropriate input for the computer program. Next an analysis was run on the sample spectra using reference spectra listed in Tables 5-3 and 5-4. The estimated uncertainty results for the undetected species were reported in Tables 1-1 and 1-2. Finally, compounds undetected in the initial analysis were removed from the program and the spectra were analyzed again using reference spectra only for the detected compounds. The results from this second analytical run are summarized in Tables 1-1 and 1-2 and reported in Appendix B.

The same program that did the analysis calculated the residual spectra (the difference between the observed and least squares fit absorbance values). Three residuals, one for each of the three analytical regions, were calculated for each sample spectrum. All of the residuals were stored electronically and are included with the electronic copy of the sample data provided with this report. The computer program calculated the standard 1*sigma uncertainty for each analytical result, but the reported uncertainties are equal to 4*sigma. The program was modified to report as a non-detect any concentration less than 2*uncertainty.

The concentrations were corrected for differences in absorption path length and temperature between the reference and sample spectra.

$$C_{corr} = \left(\frac{L_r}{L_s}\right)\left(\frac{T_s}{T_r}\right) C_{calc} \tag{5}$$

where:

C_{corr} = concentration, corrected for path length and temperature.

C_{calc} = uncorrected sample concentration.

L_r = cell path length(s) (meters) used in recording the reference spectrum.

L_s = cell path length (meters) used in recording the sample spectra.

T_s = absolute temperature (Kelvin) of the sample gas when confined in the FTIR gas cell.

T_r = absolute temperature(s) (Kelvin) of gas cell used in recording the reference spectra.

The ambient pressure recorded over the three days of the test averaged about 755 mm Hg so no pressure correction was applied to the results.

The sample path length was estimated by measuring the number of laser passes through the infrared gas cell. These measurements were recorded in the data records. The actual sample path length, L_s was calculated by comparing the sample CTS spectra to CTS (reference) spectra in the EPA FTIR reference spectrum library. The reference CTS spectra, which were recorded with the toluene reference spectra and are included in the EPA library, were used as input for a K-matrix analysis of the CTS spectra collected at the Waupaca field test. The calculated average cell path length resulting from this analysis and the variation among the Waupaca sample CTS spectra are reported in Section 4.4.1.

5.4.1 Computer Program Input

The reference spectra used in the program input are summarized in Table 5-3 for the analysis of the cooling and shake-out housing data and in Table 5-4 for the analysis of the baghouse inlet and outlet data. Results from MRI's analysis are presented in Tables 1-1 and 1-2 and Tables B-1 to B-4.

The program input for the cupola baghouse inlet and outlet included spectra of water vapor, CO_2, methane, toluene, formaldehyde, HCl, and hexane. The toluene and formaldehyde were included to analyze the spiked samples. The program input for the cooling and shake-out samples was similar, but HCl was not included in the analysis.

Table 5-5 summarizes the program input used to analyze the CTS spectra recorded at the field test. The CTS spectra were analyzed as an independent determination of the cell path length. To analyze the CTS spectra, MRI used 0.25 cm^{-1} spectra "cts0814b" and "cts0814c." These reference CTS spectra were recorded on the same dates as the toluene reference spectra used in the analysis. These spectra were deresolved in the same way as the toluene reference

spectra: by using Section K.2.2 of the EPA FTIR protocol. The program analyzed the main two ethylene bands centered near 2,989 and 949 cm^{-1}. Table 5-6 summarizes the results of the CTS analysis. The cell path length from this analysis was used as L_S in equation 4.

5.4.2 EPA Reference Spectra

The formaldehyde and toluene spectra used in the MRI analysis were taken from the EPA reference spectrum library (http://www.epa.gov/ttn/emc/ftir.html). To deresolve the spectra to 2.0 cm^{-1}, the sampling resolution, the original sample and background interferograms were truncated to the first 8,192 data points. The new interferograms were then Fourier transformed using Norton-Beer medium apodization and no zero filling. The transformation parameters were chosen to agree with those used to collect the sample absorbance spectra. The new 2.0 cm^{-1} formaldehyde and toluene single-beam spectra were combined with their deresolved single-beam background spectra and converted to absorbance. This procedure was used to prepare spectral standards for the HAP's and other compounds included in the analyses.

TABLE 5-3. PROGRAM INPUT FOR ANALYSIS OF MOLD COOLING AND SHAKE-OUT HOUSING SAMPLE SPECTRA

Compound name	File name	Region No.	ISC[a]	Reference	
				Meters	T (K)
Water	194f2sub	1,2,3	100[a]		
Carbon monoxide	co20829a	1	167.1	22	394
Carbon dioxide	193b4a_a	1,2,3	415[a]		
Formaldehyde	087b4anb	3	100.0	11.25	373
Methane	196c1bsb	3	80.1	22	394
Toluene	153a4arc	3	103.0	3	298
Ethylene	CTS0820b	2	20.1	10.4	394
SF_6	Sf60819a	2	4.01	10.4	394
Hexane	0950709a	3	46.9	10.3	399
butane	but0715a	3	100.0	11.25	397.8
n-heptane	hep0716a	3	49.97	10.3	398.3
pentane	pen0715a	3	49.99	10.3	397.9
1-pentene	1pe0712a	3	50.1	10.3	399
2-methyl-1-pentene	2m1p716a	3	50.08	10.3	398.2

Compound name	File name	Region No.	ISC[a]	Reference	
				Meters	T (K)
2-methyl-2butene	2m2b716a	3	50.04	10.3	398.2
2-methyl-2-pentene	2m2p713a	3	51.4	10.3	398.6
Isooctane	1650715a	3	50.3	10.3	398.3
3-methylpentane	3mp0713a	3	50.0	10.3	398.5

Region No.	Upper cm^{-1}	Lower cm^{-1}
1	2,142.0	2,035.6
2	1,275.0	789.3
3	3,160.8	2,650.1

[a] Indicates an arbitrary concentration was used for the interferant.

TABLE 5-4. PROGRAM INPUT FOR ANALYSIS OF BAGHOUSE INLET AND OUTLET SAMPLE SPECTRA

Compound name	File name	Region No.	ISC [a]	Reference	
				Meters	T (K)
Water	194f2sub	1,2,3	100 [a]		
Carbon monoxide	co20829a	1	167.1	22	394
Sulfur Dioxide	198c1bsc	2	89.5	22	394
Carbon dioxide	193b4a_a	1,2,3	415 [a]		
Formaldehyde	087b4anb	3	100.0	11.25	373
HCl	097b4asd	3	72.2	2.25	373
Methane	196c1bsb	3	80.1	22	394
Toluene	153a4arc	3	103.0	3	298
Hexane	095a4asd	3	101.6	3	298
Ethylene	CTS0820b	2	20.1	10.4	394
SF_6	Sf60819a	2	4.01	10.4	394
Ammonia	174a4ast	2	500.0	3	298

Region No.	Upper cm^{-1}	Lower cm^{-1}
1	2,142.0	2,035.6
2	1,275.0	789.3
3	3,160.8	2,650.1

[a] Indicates an arbitrary concentration was used for the interferant.

TABLE 5-5. PROGRAM INPUT FOR ANALYSIS OF CTS SPECTRA AND PATH LENGTH DETERMINATION

Compound name	File name	ASC	ISC	% Difference
Ethylene [a]	cts0814b.spc	1.007	1.014	0.7349
Ethylene	cts0814c.spc	1.007	0.999	0.7350

[a] This spectrum was used in the analysis of the Waupaca CTS spectra.

TABLE 5-6. RESULTS OF PATH LENGTH DETERMINATION

CTS spectra	Path length calculations		
100 ppm Ethylene	Meters	Delta [a]	% Delta
CTS0904A	10.91	0.29	2.70
CTS0905A	10.81	0.18	1.71
CTS0905B	10.79	0.17	1.56
CTS0905C	10.60	-0.02	-0.23
CTS0908A	10.62	-0.01	-0.08
CTS0908B	10.61	-0.01	-0.12
CTS0908C	10.52	-0.11	-1.03
CTS0909A	10.50	-0.13	-1.19
CTS0909B	10.40	-0.23	-2.14
CTS0910A	10.46	-0.17	-1.57
CTS0910B	10.67	0.04	0.39
Average Path Length (m)	10.63		
Standard Deviation	0.16		

[a] The difference between the calculated and average values.

5.5 FTIR SYSTEM

A KVB/Analect Diamond 20 spectrometer was used to collect all of the data in this field test. The gas cell is a heated variable path (D-22H) gas cell from Infrared Analysis, Inc. The path length of the cell was set at 20 laser passes and measured to be about 10.6 meters using the CTS reference and sample spectra. The interior cell walls have been treated with a Teflon®

coating to minimize potential analyte losses. A mercury/cadmium/ telluride (MCT) liquid nitrogen detector was used. The spectra were recorded at a nominal resolution of 2.0 cm^{-1}.

The optical path length was measured by shining an He/Ne laser through the cell and adjusting the mirror tilt to obtain the desired number of laser spots on the field mirror. Each laser spot indicates two laser passes through the cell. The number of passes was recorded on the field data sheets in Appendix B. The path length in meters was determined by comparing calibration transfer standard (CTS, ethylene in nitrogen) spectra measured in the field to CTS spectra in the EPA reference spectrum library. The procedure for determining the cell path length is described in Section 5.4.

6.0 SUMMARY OF QA/QC PROCEDURES

6.1 SAMPLING AND TEST CONDITIONS

Before the test, sample lines were checked for leaks and cleaned by purging with moist air (250°F). Following this, the lines were checked for contamination using dry nitrogen. This is done by heating the sampling lines to 250°F and purging with dry nitrogen. The FTIR cell was filled with some of the purging nitrogen, and the spectrum of this sample was collected. This single-beam spectrum was converted to absorbance using a spectral background of pure nitrogen (99.9 percent) taken directly from a cylinder. The lines were checked again on site before sampling, after each change of location, and after spiking.

During sampling, spectra of at least 10 different samples were collected during each hour (five at each of two locations).

Each spectrum was assigned a unique file name and written to the hard disk and a backup disk under that file name. Each interferogram was also saved under a file name that identifies it with its corresponding absorbance spectrum. All background spectra and calibration spectra were also stored on disks with their corresponding interferograms.

Notes on each calibration and sample spectrum were recorded on hard copy data sheets. Listed below are some sampling and instrument parameters that were documented in these records.

Sampling Conditions

- Line temperature
- Process conditions
- Sample flow rate
- Ambient pressure
- Time of sample collection

Instrument Configuration

- Cell volume (for continuous measurements)
- Cell temperature
- Cell path length
- Instrument resolution
- Number of scans co-added
- Length of time to measure spectrum

- Time spectrum was collected
- Time and conditions of recorded background spectrum
- Time and conditions of relevant CTS spectra
- Apodization

Hard copy records were also kept of all flue gas measurements, such as sample flow, temperature, moisture, and diluent data.

Effluent was allowed to flow through the entire sampling system for at least 5 min before a sampling run started or after changing to a different test location. FTIR spectra were continuously monitored to ensure that there was no deviation in the spectral baseline greater than ±5 percent ($-0.02 \leq$ absorbance $\leq +0.02$). When this condition occurred, sampling was interrupted and a new background spectrum was collected. The run was then resumed until completed or until it was necessary to collect another background spectrum.

6.2 FTIR SPECTRA

For a detailed description of QA/QC procedures relating to data collection and analysis, refer to the "Protocol For Applying FTIR Spectrometry in Emission Testing".[2]

A spectrum of the CTS was recorded at the beginning and end of each test day. A leak check of the FTIR cell was also performed according to the procedures in references 1 and 2. The CTS gas was 100 ppm ethylene in nitrogen. The CTS spectrum provided a check on the operating conditions of the FTIR instrumentation, e.g., spectral resolution and cell path length. Ambient pressure was recorded whenever a CTS spectrum was collected. The CTS spectra were compared to CTS spectra in the EPA library. This comparison is used to quantify differences between the library spectra and the field spectra so library spectra of HAP's can be used in the quantitative analysis.

Two copies of all interferograms, processed backgrounds, sample spectra, and the CTS were stored on separate computer disks. Additional copies of sample and CTS absorbance spectra were also stored for data analysis. Sample absorbance spectra can be regenerated from the raw interferograms, if necessary. A copy of the data was provided with the draft report.

To measure HAP's detected in the gas stream MRI used spectra from the EPA library, when available.

7.0 REFERENCES

1. Test Method 320 (Draft) "Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy," *40 CFR Part 63, Appendix A.*

2. "Protocol for the Use of FTIR Spectrometry to Perform Extractive Emissions Testing at Industrial Sources," Revised, EPA Contract No. 68-D2-0165, Work Assignment 3-12, September, 1996.

3. "Method 301 - Field Validation of Pollutant Measurement Methods from Various Waste Media," *40 CFR Part 63, Appendix A.*

4. "Computer-Assisted Quantitative Infrared Spectroscopy," Gregory L. McClure (ed.), *ASTM Special Publication 934* (ASTM), 1987.

5. "Multivariate Least-Squares Methods Applied to the Quantitative Spectral Analysis of Multicomponent Mixtures," *Applied Spectroscopy*, 39(10), 73-84, 1985.

6. "An Examination of a Least Squares Fit FTIR Spectral Analysis Method," G. M. Plummer and W. K. Reagen, Air and Waste Management Association, Paper Number 96-WA65.03, 1996.

APPENDIX A

VOLUMETRIC FLOW DATA

A-1. MOLD COOLING AND SHAKE-OUT HOUSING LINE FLOW DATA

Project Waupaca Foundry, Plant No. 5, Tell City, IN
Project Number 3804.25 / 4701.08.08
Source Mold Cooling Line (Location C) / Shakeout Housing (Location D)
Sample Location Combined Cooling Line Duct / Shakout Housing Duct

Run Number		1a	1c
Date		5-Sep-97	5-Sep-97
Time		14:47	15:15
Barometric Pressure	P_{bar}	29.63	29.63
Velocity Head	$\sqrt{\Delta P_{avg}}$	0.9688	1.3576
Pitot Tube Coefficient	C_p	0.84	0.84
CO_2, dry basis	%	0	0
O_2, dry basis	%	20.9	20.9
N_2, dry basis	%	79.1	79.1
Static Pressure	P_s	0	0
Stack Pressure	P_g	29.63	29.63
Stack Temperature, °F	T_s	120.80	119.93
Stack Temperature, °R	T_s	580.80	579.93
Water Vapor, proportion	B_{ws}	0.026	0.054
Mole Fraction of dry gas		0.97	0.95
Dry Molecular Weight	M_d	28.84	28.84
Actual Molecular Weight	M_s	28.55	28.25
Gas Stream Velocity	ft/sec	57.65	81.14
Stack Diameter	in	34.25	34.25
Stack Area	ft^2	6.40	6.40
Actual Volumetric Flow	wacfm	22130	31149
Standard Volumetric Flow	dscfm	19399	26576
Standard Volumetric Flow	dscmm	549	753

Moisture Calculation With WB/DB Measurement

Run	cool	shake
Pbar	29.63	29.63
Ps	0	0
Ts(DB)	121.2	126.6
Ts(WB)	83.8	99.2
Psat	1.175	1.876

Calculations

Pstack	29.63	29.63
dT	37.4	27.4
Pp H2O	0.7795	1.5913
BWS	0.0263	0.0537

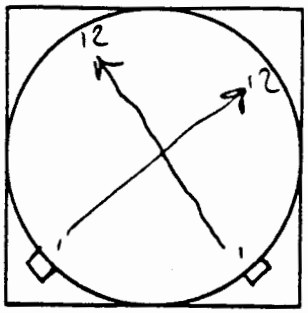
Project Waupaca Foundry, Plant No. 5, Tell City, IN
Project Number 3804.25 / 4701.08.08
Source Mold Cooling Line (Location C) / Shakeout Housing (Location I
Sample Location Combined Cooling Line Duct / Shakout Housing Duct
Stack Diameter in. 34.25 (both)
Cp 0.84 s-type pitot
Operators Neal/Edwards/Raile

Date	05-Sep-97	Date	05-Sep-97
Time	14:47	Time	15:15
Ts(DB)	121.2	Ts(DB)	126.6
Ts(WB)	83.8	Ts(WB)	99.2
Pbar	29.63	Pbar	29.63
Pt	NA	Pt	NA
Ps	0	Ps	0
O2 %	20.9	O2 %	20.9
CO2 %	0	CO2 %	0

		Run - Cool			Run - Shake		
Port	Point	dP	sqrt dP	Ts	dP	sqrt dP	Ts
NE	1	0.61	0.7810	97	1.85	1.3601	94.4
	2	0.83	0.9110	115	2	1.4142	99
	3	0.93	0.9644	120.4	2.05	1.4318	100.6
	4	0.85	0.9220	120.2	2.05	1.4318	99.6
	5	0.78	0.8832	121	2.05	1.4318	99.8
	6	0.83	0.9110	119.2	2.05	1.4318	101.2
	7	1.1	1.0488	119.4	1.9	1.3784	111.2
	8	1.2	1.0954	125.4	1.8	1.3416	115.8
	9	1.15	1.0724	125.4	1.75	1.3229	116
	10	1.25	1.1180	125.8	1.7	1.3038	122.6
	11	1.35	1.1619	126.4	1.8	1.3416	130.6
	12	1.25	1.1180	126.6	1.65	1.2845	130.4
SE	1	0.59	0.7681	101.4	1.9	1.3784	119
	2	0.96	0.9798	114.6	2.2	1.4832	123.8
	3	1.1	1.0488	115	2.3	1.5166	126.2
	4	1.15	1.0724	122.6	2.25	1.5000	127.6
	5	1.15	1.0724	123.2	2.35	1.5330	128.8
	6	1.1	1.0488	123.6	2.45	1.5652	129.6
	7	0.71	0.8426	126	1.75	1.3229	129.6
	8	0.69	0.8307	126	1.4	1.1832	133.4
	9	0.68	0.8246	126.2	1.35	1.1619	134
	10	0.71	0.8426	126.4	1.35	1.1619	135
	11	0.91	0.9539	126.2	1.4	1.1832	134.8
	12	0.96	0.9798	126.2	1.25	1.1180	135.4
	Average		0.9688	120.8		1.3576	119.9

VELOCITY TRAVERSE DATA

Project No. _____
Run No. Prelim Date 9/5/91
Plant WAPACA FOUNDRY
Sampling Location Shake-out
Operator(s) NEAL EDWARDS Raille
Barometric Pressure, ~~in. Hg~~ 752 mm Hg
Site to Barometer Elevation _____ ft.
Corrected Barometric Pressure _____
Pitot No. _____ Pitot Cp _____
T/C No. _____ Temp. Meter No. _____
Stack Area, sq.ft. _____
Static Pressure, in. H_2O _____
Assumed Moisture, % _____
Assumed $\%CO_2$ _____ Assumed $\%O_2$ _____
Initial Pitot Leak Check 1455 PASS
Final Pitot Leak Check 1522 PASS
Comments:



Traverse Point Layout

Start Time 1500 End Time 1520

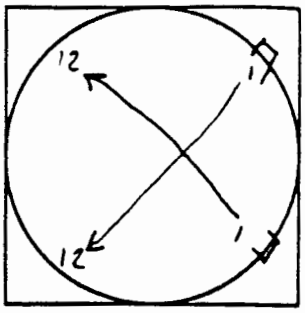
TRAVERSE POINT NUMBER	VELOCITY HEAD, Δp in. H_2O	STACK TEMP. °F	ROTATION ANGLE α	
SE 1	1.85	94.4		
2	2.00	~~96~~ 99.0		
3	2.05	100.6		125.2
4	2.05	99.6		
5	2.05	99.8		
6	2.05	101.2		
7	1.90	111.2		*
8	1.80	115.8		
9	1.75	116.0		
10	1.70	122.6		
11	1.80	130.6		
12	1.65	130.4		

TRAVERSE POINT NUMBER	VELOCITY HEAD, Δp in. H_2O	STACK TEMP. °F	ROTATION ANGLE α	
SW 1	1.90	119.0		
2	2.20	123.8		
3	2.30	126.2		
4	2.25	127.6		
5	2.35	128.8		
6	2.45	129.6		
7	1.75	129.6		
8	1.40	133.4		
9	1.35	134.0		
10	1.35	135.0		
11	1.40	134.8		
12	1.25	135.4		

93-3 SEV sur frm 020393

VELOCITY TRAVERSE DATA

Project No. _____
Run No. Prelim Date 9/5/97
Plant WAPPCA FOUNDRY
Sampling Location Cooling
Operator(s) Edwards, Neal, Raile
Barometric Pressure, ~~in. Hg~~ 752 mm Hg
Site to Barometer Elevation _____ ft.
Corrected Barometric Pressure _____
Pitot No. _____ Pitot Cp _____
T/C No. _____ Temp. Meter No. _____
Stack Area, sq.ft. _____
Static Pressure, in. H_2O _____
Assumed Moisture, % _____
Assumed $\%CO_2$ _____ Assumed $\%O_2$ _____
Initial Pitot Leak Check 1424 PASS
Final Pitot Leak Check 1449 PASS
Comments:



Traverse Point Layout

Start Time 1430 End Time 1407

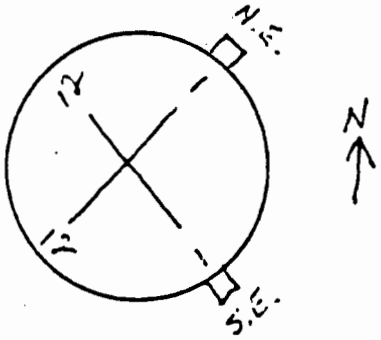
TRAVERSE POINT NUMBER	VELOCITY HEAD, Δp in. H_2O	STACK TEMP. °F	ROTATION ANGLE α	
SE 1	0.61	97.0		
2	0.83	115.0		
3	0.93	120.4		
4	0.85	120.2		
5	0.78	121.0		
6	0.83	119.2		
7	1.10	119.4		
8	1.20	125.4		
9	1.15	125.4		
10	1.25	125.8		
11	1.35	126.4		
12	1.25	126.6		

TRAVERSE POINT NUMBER	VELOCITY HEAD, Δp in. H_2O	STACK TEMP. °F	ROTATION ANGLE α	
NE 1	0.59	101.4		
2	0.96	114.6		
3	1.10	115.0		
4	1.15	122.6		
5	1.15	123.2		
6	1.10	123.6		
7	0.71	126.0		
8	0.69	126.0		
9	0.68	126.2		
10	0.71	126.4		
11	0.91	126.2		
12	0.96	126.2		

93-3 SEV sur frm 020393

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

ANT Waupaca Foundry Plant #5
TE 9-5-97
SAMPLING LOCATION Cooling Stack
INSIDE OF FAR WALL TO
OUTSIDE OF NIPPLE. (DISTANCE A) 37½"
INSIDE OF NEAR WALL TO
OUTSIDE OF NIPPLE. (DISTANCE B) 3¼"
STACK I.D., (DISTANCE A - DISTANCE B) 34¼"
NEAREST UPSTREAM DISTURBANCE
NEAREST DOWNSTREAM DISTURBANCE
CALCULATOR D. Neal



SCHEMATIC OF SAMPLING LOCATION

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	DISTANCE B	TRAVERSE POINT LOCATION FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)
N.E. 1	.021	34.25	0.719	3.25	3.97
2	.067	"	2.295	"	5.55
3	.118	"	4.042	"	7.29
4	.177	"	6.062	"	9.31
5	.25	"	8.563	"	11.81
6	.355	"	12.159	"	15.41
7	.645	"	22.091	"	25.34
8	~~.65~~ .75 DN	"	D.N. ~~22.263~~ 25.688	"	D.N. ~~25.51~~ 28.94
9	.823	"	28.188	"	31.44
10	.882	"	30.209	"	33.46
11	.933	"	31.955	"	35.21
12	.979	"	33.531	"	36.78
S.E. 1	.021	"	0.719	"	3.97
2	.067	"	2.295	"	5.55
3	.118	"	4.042	"	7.29
4	.177	"	6.062	"	9.31
5	.25	"	8.563	"	11.81
6	.355	"	12.159	"	15.41
7	.645	"	22.091	"	25.34
8	DN ~~.65~~ .75	"	25.688	"	28.94
9	.823	"	28.188	"	31.44
10	.882	"	30.209	"	33.46
11	.933	"	31.955	"	35.21
12	.979	"	33.531	"	36.78

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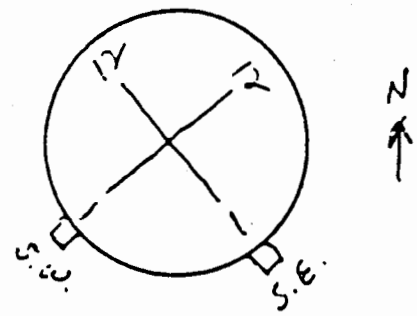
SE

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT Waupaca Foundry Plant #5
TE 9-5-97
SAMPLING LOCATION Shaker Stack
INSIDE OF FAR WALL TO
OUTSIDE OF NIPPLE. (DISTANCE A) 37½"
INSIDE OF NEAR WALL TO
OUTSIDE OF NIPPLE. (DISTANCE B) 3¼"
STACK I.D., (DISTANCE A - DISTANCE B) 34¼"
NEAREST UPSTREAM DISTURBANCE
NEAREST DOWNSTREAM DISTURBANCE
CALCULATOR D. Neal



SCHEMATIC OF SAMPLING LOCATION

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	DISTANCE B	TRAVERSE POINT LOCATION FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)
S.W. 1	.021	34.25	0.719	3.25	3.97
2	.067	"	2.295	"	5.55
3	.118	"	4.042	"	7.29
4	.177	"	6.067	"	9.31
5	.25	"	8.563	"	11.81
6	.355	"	12.159	"	15.41
7	.645	"	22.091	"	25.34
8	.75	"	25.688	"	28.94
9	.823	"	28.188	"	31.44
10	.882	"	30.209	"	33.46
11	.933	"	31.955	"	35.21
12	.979	"	33.531	"	36.78
S.E. 1 ~~12~~	.021	"	0.719	"	3.97
2	.067	"	2.295	"	5.55
3	.118	"	4.042	"	7.29
4	.177	"	6.067	"	9.31
5	.25	"	8.563	"	11.81
6	.355	"	12.159	"	15.41
7	.645	"	22.091	"	25.34
8	.75	"	25.688	"	28.94
9	.823	"	28.188	"	31.44
10	.882	"	30.209	"	33.46
11	.933	"	31.955	"	35.21
12	.979	"	33.531	"	36.78

PA (Dur) 232
4 72

FTIR FIELD DATA FORM

PROJECT NO. 3804-25-04-03 *(Moisture Data - Wet Bulb and Dry Bulb Method)* **BAROMETRIC:**

PLANT: Wampum **OPERATOR:** [illegible]

DATE	LOCATION	TIME 24-HR	DRY BULB TEMPERATURES t (F)	WET BULB TEMPERATURES tw (F)	BAROMETRIC PRESSURE Pbar ~~in Hg~~ mm Hg	TOTAL PRESSURE TP in. w.c.	VELOCITY HEAD Delta P in. w.c.	PITOT COEFFICIENT Cp	CARBON DIOXIDE Dry-basis % by vol.	OXYGEN Dry-basis % by vol.
9/5/97	Coldwater	1447	121.2	83.8	752		1.10	0.8400	0	20.9
9/6/97	Shakeoff	1515	126.6	99.2	752		1.80	0.8400	0	20.9

Collection of ORSAT BAGS

OXYGEN AND CARBON DIOXIDE BY ORSAT

Cooling Line
ORSAT
Start - 16:41
@ 1.00 lpm

17:01 = 0.75 lpm

Stop - 17:19

PROJECT NO. 3201-25 RUN NO. Stackpoint and Cooling
SAMPLE NO. 111 DATE 9/5/97
PLANT SAMPLING LOCATION Wauparce [illegible]
ANALYSIS TIME (24hr-CLOCK)
SAMPLE TYPE (BAG, GRAB)
OPERATOR

ORSAT LEAK CHECK BEFORE ANALYSIS:
BURETTE _____ CHANGE IN 4 MIN.
PIPETTES _____ CHANGE IN 4 MIN.
ORSAT LEAK CHECK AFTER ANALYSIS:
BURETTE _____ CHANGE IN 4 MIN.
PIPETTES _____ CHANGE IN 4 MIN.

Stackpoint Line
ORSAT
Start - 17:20
@ 1.00 lpm
17:40 - 1.0[illegible]

Stop: 17:50

GAS \ RUN	1		2		3		AVERAGE NET VOLUME
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET	
CO_2	1 2 3		1 2 3		1 2 3		
O_2 (NET IS SECOND READING MINUS ACTUAL CO_2 READING)	1 2 3		1 2 3		1 2 3		

91-18 SEV SURMAN wkshi 052191

Acceptance Criteria

CO_2	> 4%	.3% by Volume	O_2	≥ 15%	.2% by Volume
	≤ 4%	.2% by Volume		< 15%	.3% by Volume

Comments:

— Bags were collected off of sample line after manifold and condenser unit

OXYGEN AND CARBON DIOXIDE BY ORSAT

PROJECT NO. 3804-25-04-03 RUN NO. _____

SAMPLE NO. _____ DATE 9-5-97

PLANT SAMPLING LOCATION SHAKE OUT STACK

ANALYSIS TIME (24hr-CLOCK) 1910

SAMPLE TYPE (BAG, GRAB) _____

OPERATOR D. NEAL

ORSAT LEAK CHECK BEFORE ANALYSIS:

BURETTE PASS CHANGE IN 4 MIN.

PIPETTES PASS CHANGE IN 4 MIN.

ORSAT LEAK CHECK AFTER ANALYSIS:

BURETTE PASS CHANGE IN 4 MIN.

PIPETTES PASS CHANGE IN 4 MIN.

GAS \ RUN	1		2		3		AVERAGE NET VOLUME
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET	
CO_2	1 0.0 2 0.0 3 0.0	0.0	1 2 0.0 3		1 2 3		
O_2 (NET IS SECOND READING MINUS ACTUAL CO_2 READING)	1 20.9 2 20.9 3 20.9	20.9	1 2 20.9 3		1 2 3		

91-16 SEV SURMAN wksht 052191

Acceptance Criteria

CO_2	> 4%	.3% by Volume	O_2	≥ 15%	.2% by Volume
	≤ 4%	.2% by Volume		< 15%	.3% by Volume

Comments:

OXYGEN AND CARBON DIOXIDE BY ORSAT

PROJECT NO. 3804-25-04-03 RUN NO. _____

SAMPLE NO. _____ DATE 9-5-97

PLANT SAMPLING LOCATION Cooling Stack

ANALYSIS TIME (24hr-CLOCK) 1840

SAMPLE TYPE (BAG, GRAB) _____

OPERATOR D. Neal

ORSAT LEAK CHECK BEFORE ANALYSIS:

BURETTE Pass CHANGE IN 4 MIN.

PIPETTES Pass CHANGE IN 4 MIN.

ORSAT LEAK CHECK AFTER ANALYSIS:

BURETTE Pass CHANGE IN 4 MIN.

PIPETTES Pass CHANGE IN 4 MIN.

RUN / GAS	1		2		3		AVERAGE NET VOLUME
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET	
CO_2	1 0.0 2 0.0 3 0.0	0.0	1 2 0.0 3		1 2 3		
O_2 (NET IS SECOND READING MINUS ACTUAL CO_2 READING)	1 20.7 2 20.7 3 20.7	20.7	1 2 20.7 3		1 2 3		

91-16 SEV SURIMAN wksht 052191

Acceptance Criteria

CO_2	> 4%	.3% by Volume	O_2	≥ 15%	.2% by Volume
	≤ 4%	.2% by Volume		< 15%	.3% by Volume

Comments:

1-9

A-2. BAGHOUSE FLOW DATA

PARTICULATE/METALS EMISSIONS SAMPLING AND FLUE GAS PARAMETERS - CUPOLA BAGHOUSE INLET THE WAUPACA FOUNDRY - TELL CITY, INDIANA

Run No.	I-M29-1	I-M29-2	I-M29-3	Average
Date	9/9/97	9/10/97	9/10/97	
Total Sampling Time, min	240.5	240	240	
Average Sampling Rate, dscfm [a]	0.342	0.481	0.493	0.439
Sample Volume:				
dscf [b]	82.208	115.471	118.408	105.362
dscm [c]	2.328	3.270	3.353	2.984
Average Flue Gas Temp., °F	275	301	302	293
O_2 Concentration, % by Volume	10.9	9.5	8.8	9.7
CO_2 Concentration, % by Volume	10.8	11.6	12.4	11.6
Moisture, % by Volume	2.5	2.8	2.4	2.6
Flue Gas Volumetric Flow Rate:				
acfm [d]	39,900	58,900	59,300	52,700
dscfm [a]	26,800	38,200	38,500	34,500
dscmm [e]	759	1,080	1,090	976
Isokinetic Sampling Ratio, %	104.0	102.7	106.5	104.4

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Dry standard cubic feet at 68° F (20° C) and 1 atm.

[c] Dry standard cubic meters at 68° F (20° C) and 1 atm.

[d] Actual cubic feet per minute at exhaust gas conditions.

[e] Dry standard cubic meters per minute at 68° F (20° C) and 1 atm.

PARTICULATE/METALS EMISSIONS SAMPLING AND EXHAUST GAS PARAMETERS - CUPOLA BAGHOUSE OUTLET THE WAUPACA FOUNDRY - TELL CITY, INDIANA

Run No.	O-M29-1	O-M29-2	O-M29-3	Average
Date	9/9/97	9/10/97	9/10/97	
Total Sampling Time, min	240	240	240	
Average Sampling Rate, dscfm [a]	0.378	0.580	0.552	0.503
Sample Volume:				
dscf [b]	90.633	139.162	132.547	120.781
dscm [c]	2.566	3.941	3.753	3.420
Average Exhaust Gas Temp., °F	231	253	254	246
O_2 Concentration, % by Volume	12.7	11.0	11.0	11.6
CO_2 Concentration, % by Volume	8.8	10.1	10.0	9.6
Moisture, % by Volume	5.5	2.6	2.8	3.6
Exhaust Gas Volumetric Flow Rate:				
acfm [d]	45,000	69,600	68,200	60,900
dscfm [a]	32,100	49,700	48,500	43,400
dscmm [e]	908	1,410	1,370	1,230
Isokinetic Sampling Ratio, %	103.1	102.2	99.7	101.7

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Dry standard cubic feet at 68° F (20° C) and 1 atm.

[c] Dry standard cubic meters at 68° F (20° C) and 1 atm.

[d] Actual cubic feet per minute at exhaust gas conditions.

[e] Dry standard cubic meters per minute at 68° F (20° C) and 1 atm.

SVOHAPS EMISSIONS SAMPLING AND EXHAUST GAS PARAMETERS
CUPOLA BAGHOUSE OUTLET
THE WAUPACA FOUNDRY - TELL CITY, INDIANA

Run No.	BO-0010-1	BO-0010-2	BO-0010-3	Average
Date	9/9/97	9/10/97	9/10/97	
Total Sampling Time, min	240	240	240	
Average Sampling Rate, dscfm [a]	0.458	0.627	0.690	0.592
Sample Volume:				
dscf [b]	110.023	150.485	165.500	142.003
dscm [c]	3.116	4.261	4.686	4.021
Average Exhaust Gas Temp., °F	234	258	256	249
O_2 Concentration, % by Volume	12.7	11.0	11.0	11.6
CO_2 Concentration, % by Volume	8.8	10.1	10.0	9.6
Moisture, % by Volume	3.4	2.7	2.6	2.9
Exhaust Gas Volumetric Flow Rate:				
acfm [d]	46,600	66,700	69,300	60,900
dscfm [a]	33,800	47,200	49,200	43,400
dscmm [e]	957	1,340	1,390	1,230
Isokinetic Sampling Ratio, %	99.4	97.3	102.6	99.7

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Dry standard cubic feet at 68° F (20° C) and 1 atm.

[c] Dry standard cubic meters at 68° F (20° C) and 1 atm.

[d] Actual cubic feet per minute at exhaust gas conditions.

[e] Dry standard cubic meters per minute at 68° F (20° C) and 1 atm.

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PCDDs/PCDFs EMISSIONS SAMPLING AND EXHAUST GAS PARAMETERS
CUPOLA BAGHOUSE OUTLET
THE WAUPACA FOUNDRY - TELL CITY, INDIANA

Run No.	BO-23-1	BO-23-2	BO-23-3	Average
Date	9/9/97	9/10/97	9/10/97	
Total Sampling Time, min	240	240	240	
Average Sampling Rate, dscfm [a]	0.486	0.669	0.660	0.605
Sample Volume:				
dscf [b]	116.671	160.663	158.414	145.249
dscm [c]	3.304	4.549	4.486	4.113
Average Exhaust Gas Temp., °F	230	258	254	247
O_2 Concentration, % by Volume	12.7	11.0	11.0	11.6
CO_2 Concentration, % by Volume	8.8	10.1	10.0	9.6
Moisture, % by Volume	3.4	2.6	2.4	2.8
Exhaust Gas Volumetric Flow Rate:				
acfm [d]	49,400	69,500	68,800	62,600
dscfm [a]	36,000	49,200	49,100	44,800
dscmm [e]	1,020	1,390	1,390	1,270
Isokinetic Sampling Ratio, %	98.9	99.7	98.5	99.0

[a] Dry standard cubic feet per minute at 68° F (20° C) and 1 atm.

[b] Dry standard cubic feet at 68° F (20° C) and 1 atm.

[c] Dry standard cubic meters at 68° F (20° C) and 1 atm.

[d] Actual cubic feet per minute at exhaust gas conditions.

[e] Dry standard cubic meters per minute at 68° F (20° C) and 1 atm.

APPENDIX B

FTIR DATA

WAUPACA

Date	Time	Location	Spiked	Unspiked	Event/Notes
9/9/97	10:30	Outlet	X		
	10:39	Outlet		X	
	10:50-11:00	Inlet	X		
	11:08-11:17	Inlet		X	
	11:28-11:34	Outlet		X	
	11:44				N2 only direct to cell
	11:50				Background - N2 only
	11:57	Inlet		X	
	12:24	Outlet		X	
	12:50	Inlet		X	
	13:24	Outlet		X	
	13:50	Inlet		X	
	14:05	Outlet	X		
	14:11	Inlet	X		
	14:29				Spike direct to cell
	15:05				Outlet - air through
	15:25				Background - N2 only
9/10/97	7:20				Leak check inlet and outlet
	7:49	Inlet	X		
	8:00	Outlet	X		
	8:10				Direct to cell spike
	8:15-8:27	Outlet		X	
	8:34-8:47	Inlet		X	
	9:00				Background
	9:07	Outlet		X	
	9:40	Inlet		X	
	10:05	Outlet		X	
	10:29	Inlet		X	
	11:05				N2 only - Background
	11:17-11:37	Outlet		X	
	11:45-12:07	Inlet		X	
	12:15-12:33	Outlet		X	
	12:45	Outlet	X		
	12:53	Inlet	X		
	13:04				Spike direct to cell
	13:26				Background - N2 only

WAUPACA

Date	Time	Location	Spiked	Unspiked	Event/Notes
9/5/97	9:30				Background, N2 only
	9:45-9:53				Calibration
	9:55				Leak check
	10:15				Process down
	10:54				Background, N2 only
	11:20		X		SF6 to spike line
	13:15				Process restarted
	14:10				SF6 into Kintek
	14:20-14:24				N2 only
	15:23-15:33	Cooling stack	X		
	15:37-16:02		X		Shakeout probe in stack
	16:11-16:27	Cooling line		X	
	16:41-16:58	Shakeout line		X	
	17:21	Shakeout line			
	17:41	Cooling line		X	
	18:03	Cooling line		X	
	18:07	Shakeout line		X	
	18:29	Cooling line			
	18:50	Shakeout line	X		
	19:11	Cooling line	X		
	19:31				N2 only direct to cell
	19:45				N2 only - Background
9/8/97	10:24				Background - N2 only
	11:07				direct to cell - spike
	12:02-12:18	Inlet	X		
	12:27-12:40	Outlet	X		
	12:46				Background - N2 direct to cell
	12:56-13:27	Inlet		X	
	13:25				Changed outlet probe
	13:42-13:49	Inlet		X	
	14:04-14:15	Outlet		X	
	14:28-14:33	Inlet		X	
	14:45-15:09	Outlet		X	
	15:18-15:30	Inlet		X	
	15:39	Outlet		X	
	16:09	Inlet		X	
	16:39				Background - N2 only
	16:55	Outlet	X		
	17:05	Inlet	X		
	17:10				Probes pulled out of stack
	17:13				Inlet and Outlet pass leak checks
	17:28				N2 only
9/9/97	7:10				Inlet and Outlet pass leak checks
	7:27				Background
	7:47	Inlet	X		
	8:00	Outlet	X		
	8:11				Spike direct to cell
	8:24	Outlet	X		
	8:57-9:06				N2 only in background
	9:25-9:44	Outlet		X	
	9:54-10:08	Inlet		X	
	10:16	Outlet		X	

B-1. FTIR RESULTS TABLES

TABLE B-1. FTIR RESULTS FROM THE MOLD COOLING LINE

TABLE B-1. FTIR RESULTS AT MOLD COOLING LINE

			Toluene		Hexane		Ethylene		SF_6		Methane		CO		Formaldehyde	
Date	Time	File name [1]	ppm	Unc [2]	ppm	Unc	ppm	Unc	ppm [3]	Unc	ppm	Unc	ppm	Unc	ppm	Unc
9/5/97	**15:25**	**COOSP101**	**19.9**	**3.1**	**0.0**	**22.2**	**7.3**	**0.8**	**0.440**	**0.018**	**129.3**	**1.9**	**337.9**	**20.3**	**12.8**	**1.7**
	15:35	**COOSP102**	**20.8**	**3.5**	**0.0**	**25.0**	**8.1**	**0.9**	**0.445**	**0.019**	**139.9**	**2.1**	**354.1**	**23.6**	**12.3**	**1.9**
	16:12	COOUN103	19.5	4.0	0.0	31.5	13.5	0.8	0.000	0.019	189.6	2.7	438.4	27.5	0.0	3.0
	16:18	COOUN104	18.7	4.0	0.0	31.3	13.2	0.8	0.000	0.018	184.4	2.6	417.3	27.3	0.0	3.0
	16:23	COOUN105	19.3	4.0	0.0	31.9	13.6	0.8	0.000	0.019	189.1	2.7	425.0	27.9	0.0	3.1
	16:30	COOUN106	18.2	3.9	0.0	31.0	13.2	0.8	0.000	0.018	182.1	2.6	421.0	27.7	0.0	3.0
	17:07	19050001	19.0	4.0	0.0	31.0	13.6	0.8	0.000	0.019	189.5	2.6	441.9	27.1	0.0	3.0
	17:09	19050002	18.6	3.9	0.0	30.6	13.5	0.8	0.000	0.018	187.2	2.6	437.0	27.0	0.0	3.0
	17:12	19050003	18.6	3.9	0.0	30.7	13.6	0.8	0.000	0.018	187.3	2.6	436.8	27.3	0.0	3.0
	17:14	19050004	19.0	4.0	0.0	31.2	13.8	0.8	0.000	0.019	189.9	2.7	438.5	27.8	0.0	3.0
	17:16	19050005	18.7	4.0	0.0	31.1	13.7	0.8	0.000	0.019	188.1	2.6	434.0	27.9	0.0	3.0
	17:18	19050006	17.8	3.9	0.0	30.5	13.3	0.8	0.000	0.018	182.4	2.6	424.2	27.6	0.0	2.9
	17:20	19050007	17.2	3.8	0.0	30.1	13.1	0.8	0.000	0.018	178.9	2.6	419.4	27.7	0.0	2.9
	17:44	19050018	16.8	4.0	0.0	31.4	13.2	0.8	0.000	0.019	174.3	2.7	385.4	30.0	0.0	3.0
	17:46	19050019	17.4	4.1	0.0	31.9	13.5	0.8	0.000	0.019	177.4	2.7	389.7	30.3	0.0	3.1
	17:48	19050020	17.6	4.1	0.0	32.2	13.7	0.8	0.000	0.019	179.0	2.7	392.5	30.5	0.0	3.1
	17:50	19050021	17.9	4.2	0.0	32.7	13.9	0.8	0.000	0.019	181.4	2.8	396.6	30.8	0.0	3.2
	17:52	19050022	18.1	4.2	0.0	32.9	14.0	0.8	0.000	0.019	183.0	2.8	396.8	30.9	0.0	3.2
	17:54	19050023	18.2	4.1	0.0	32.5	14.1	0.8	0.000	0.019	184.2	2.8	399.5	30.7	0.0	3.1
	17:57	19050024	18.2	4.2	0.0	32.8	14.1	0.8	0.000	0.019	184.1	2.8	396.8	30.8	0.0	3.2
	17:59	19050025	17.3	4.1	0.0	32.3	13.6	0.8	0.000	0.019	178.6	2.7	387.2	30.4	0.0	3.1
	18:05	19050028	9.2	2.0	0.0	13.4	7.4	0.4	0.000	0.010	74.3	1.2	196.0	14.5	0.0	1.3
	18:29	19050039	16.6	3.9	0.0	30.7	13.5	0.8	0.000	0.019	179.9	2.6	397.0	28.4	0.0	3.0
	18:31	19050040	16.5	3.9	0.0	30.6	13.5	0.8	0.000	0.019	179.9	2.6	396.9	28.4	0.0	3.0
	18:33	19050041	16.5	3.9	0.0	30.8	13.5	0.8	0.000	0.019	180.3	2.6	395.8	28.4	0.0	3.0
	18:35	19050042	16.1	3.9	0.0	30.5	13.3	0.8	0.000	0.018	178.2	2.6	392.0	28.2	0.0	2.9
	19:11	**COOSP107**	**16.5**	**3.2**	**0.0**	**22.7**	**5.3**	**1.1**	**0.545**	**0.023**	**125.2**	**2.0**	**325.6**	**22.0**	**12.8**	**1.8**
		Average --->	17.5	3.9	0.0	30.6	13.3	0.8	0.000	0.018	178.5	2.6	402.3	28.1	0.0	3.0

[1] The samples indicated in bold type, "COOSP101, "COOSP102" and "COOSP107," were spiked with a mixture o f formaldehyde vapor, toluene vapor, and SF_6. The spike results are presented in Seciton 4.4, and the analyte spike procedure is discussed in Section 5.3.1.
[2] Unc is th estimated uncertainty in the measurement.
[3] SF_6 was spiked as a tracer gas to determine to spike dilution. SF_6 was not detected in the gas stream.

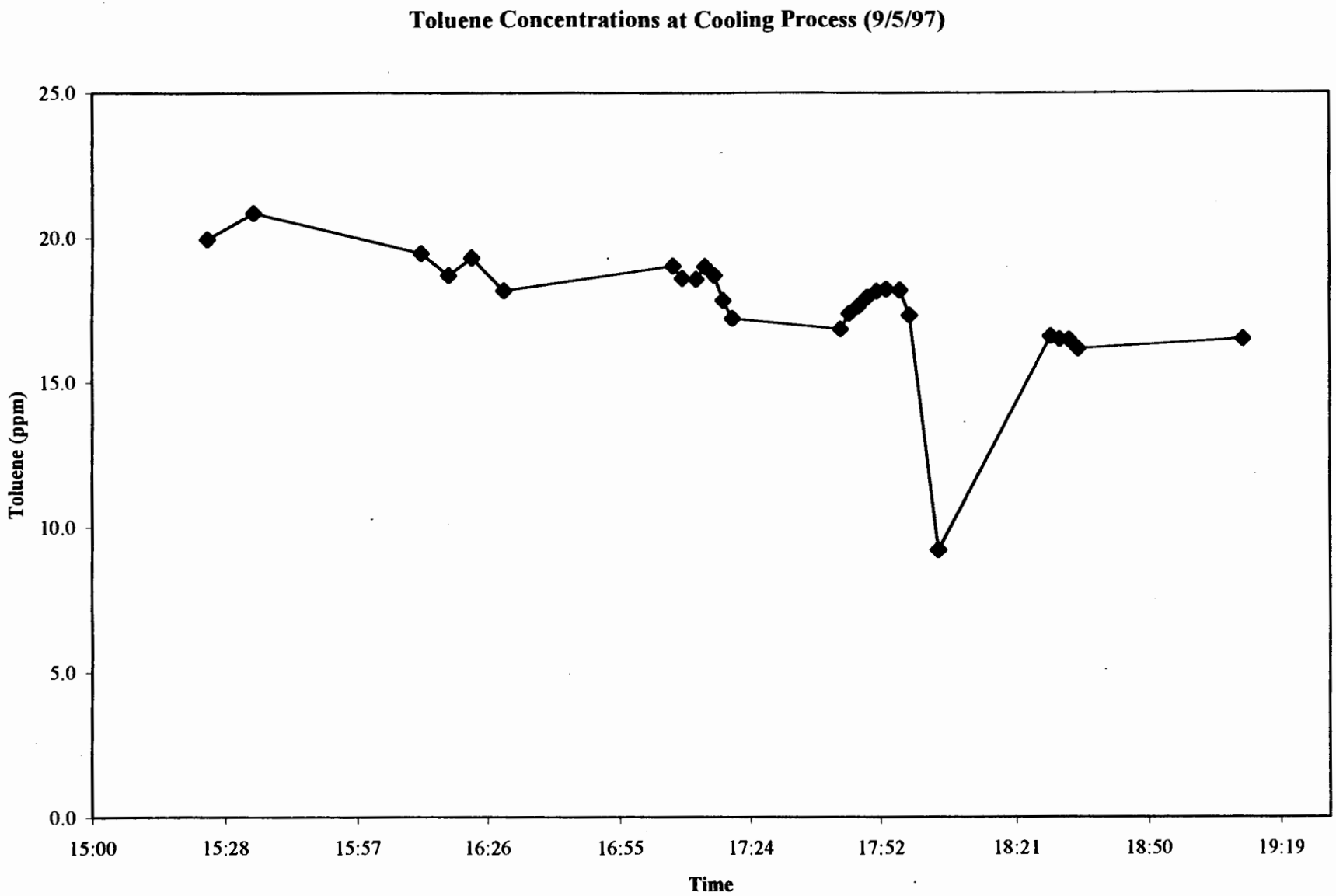
TABLE B-1. Continued.

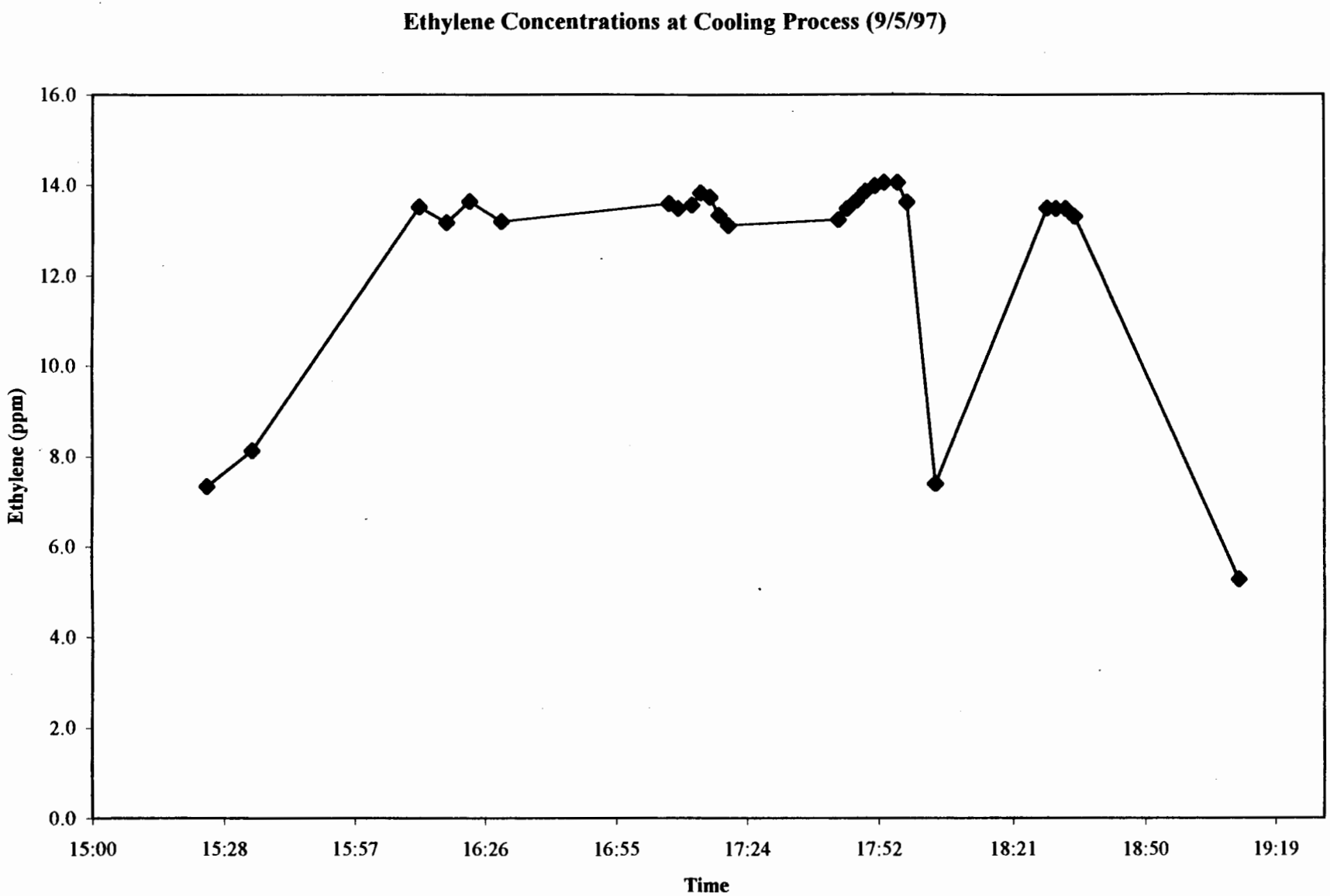
Date	Time	File name [1]	3-Methylepentane ppm	3-Methylepentane Unc [2]	1-Pentene ppm	1-Pentene Unc
9/5/97	**15:25**	**COOSP101**	**3.9**	**1.2**	**10.9**	**3.3**
	15:35	**COOSP102**	**4.3**	**1.4**	**11.1**	**3.7**
	16:12	COOUN103	5.6	1.7	18.0	4.0
	16:18	COOUN104	5.4	1.7	17.1	3.9
	16:23	COOUN105	5.6	1.7	17.7	4.0
	16:30	COOUN106	5.5	1.7	16.9	3.9
	17:07	19050001	5.9	1.7	18.1	3.9
	17:09	19050002	5.8	1.7	17.8	3.9
	17:12	19050003	5.8	1.7	18.0	3.9
	17:14	19050004	5.8	1.7	18.4	4.0
	17:16	19050005	5.8	1.7	18.2	3.9
	17:18	19050006	5.6	1.7	17.6	3.9
	17:20	19050007	5.4	1.7	17.2	3.8
	17:44	19050018	5.3	1.7	17.6	4.0
	17:46	19050019	5.4	1.7	18.2	4.0
	17:48	19050020	5.5	1.8	18.5	4.1
	17:50	19050021	5.5	1.8	18.8	4.1
	17:52	19050022	5.6	1.8	19.0	4.2
	17:54	19050023	5.6	1.8	19.1	4.1
	17:57	19050024	5.6	1.8	19.0	4.2
	17:59	19050025	5.4	1.8	18.2	4.1
	18:05	19050028	2.1	0.8	13.2	1.8
	18:29	19050039	5.5	1.7	18.0	3.9
	18:31	19050040	5.5	1.7	18.0	3.9
	18:33	19050041	5.5	1.7	18.1	3.9
	18:35	19050042	5.4	1.7	17.8	3.9
	19:11	**COOSP107**	**7.8**	**0.7**	**0.0**	**11.7**
		Average --->	5.4	1.7	17.9	3.9

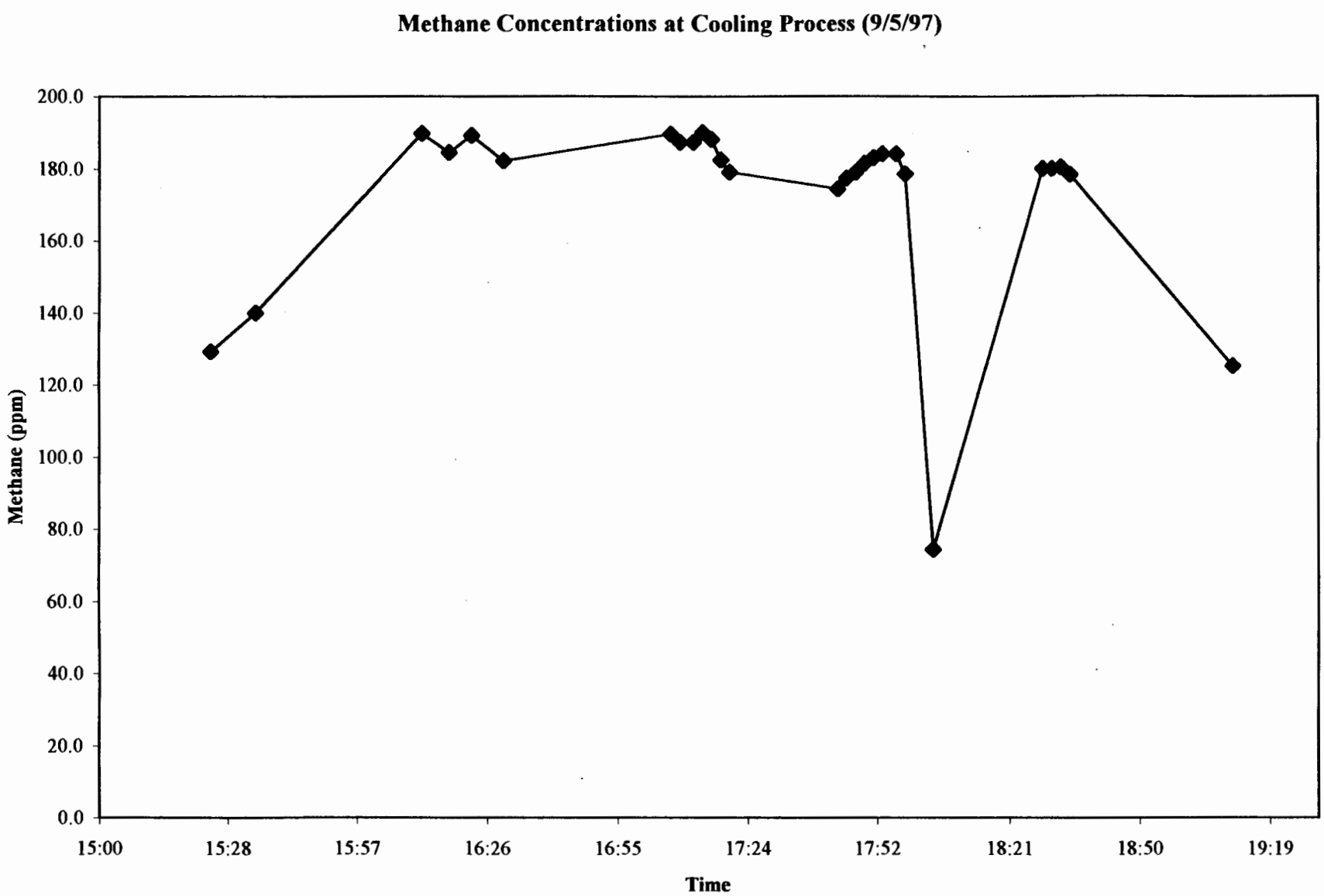
[1] The samples indicated in bold type, "COOSP101, "COOSP102" and "COOSP107," were spiked with a mixture o f formaldehyde vapor, toluene vapor, and SF_6. The spike results are presented in Seciton 4.4, and the analyte spike procedure is discussed in Section 5.3.1.

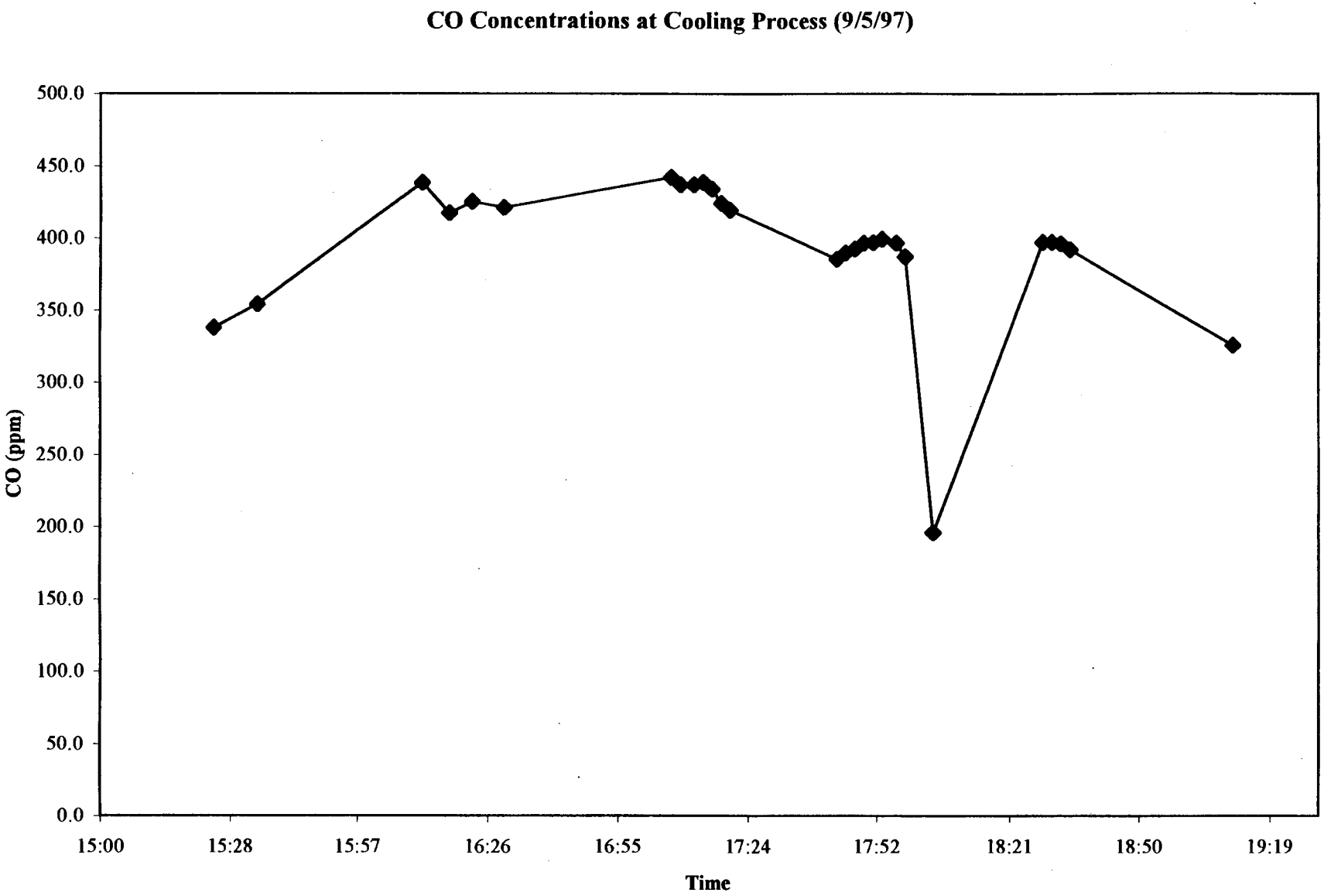
[2] Unc is th estimated uncertainty in the measurement.

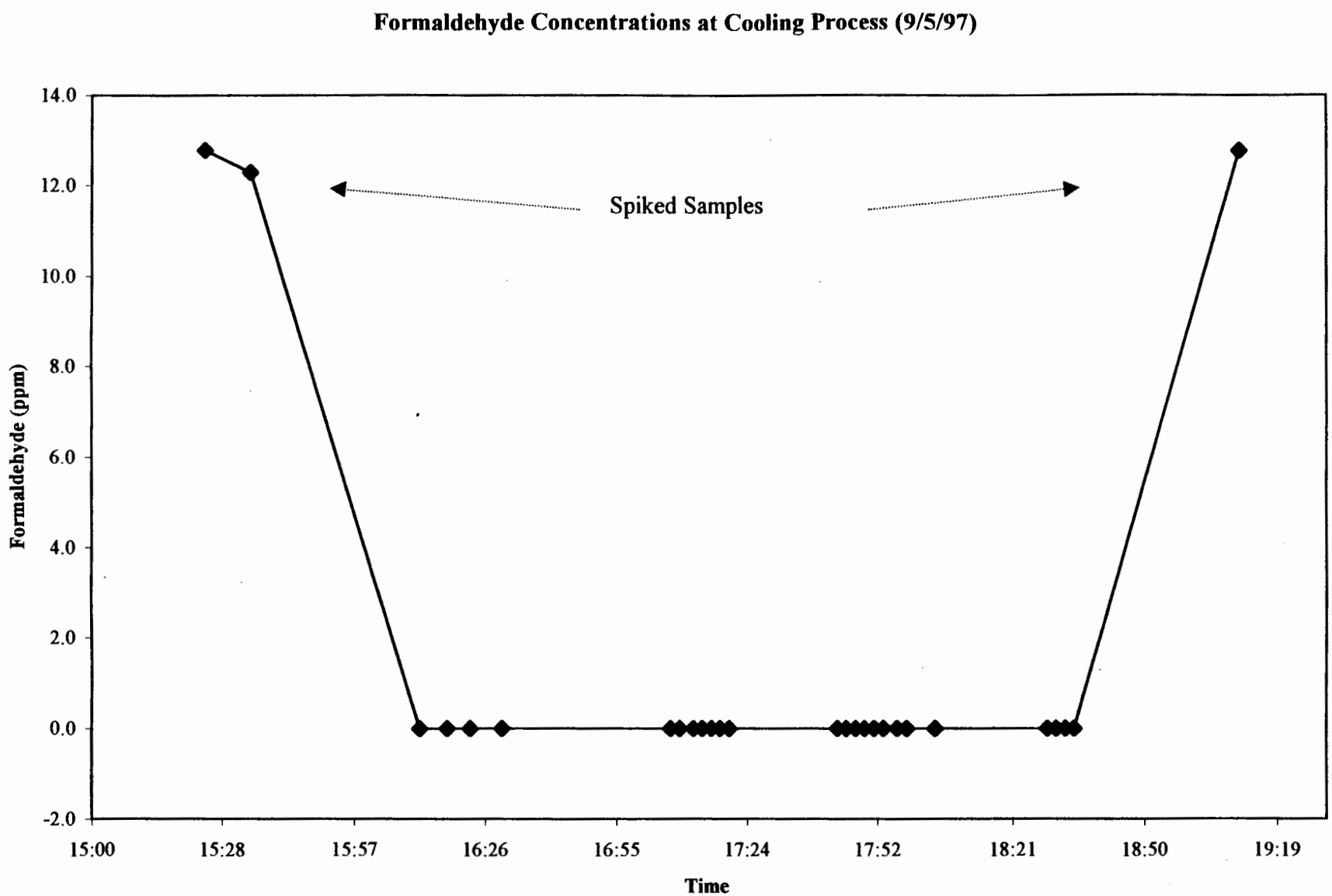
[3] SF_6 was spiked as a tracer gas to determine to spike dilution. SF_6 was not detected in the gas stream.


Toluene Concentrations at Cooling Process (9/5/97)
25.0
20.0
15.0
10.0
5.0
0.0
Toluene (ppm)
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time

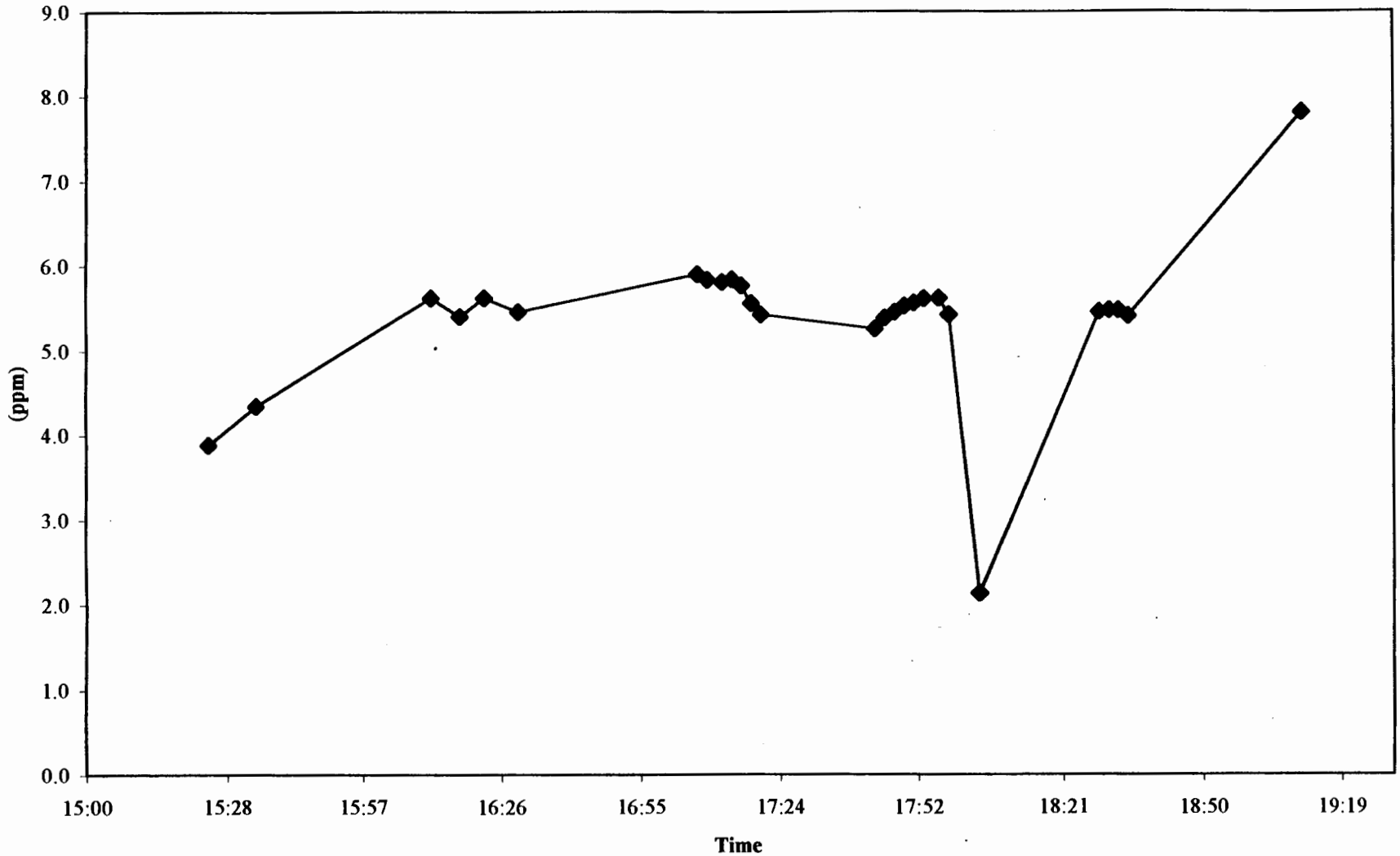

Ethylene Concentrations at Cooling Process (9/5/97)
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
Ethylene (ppm)
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time


Methane Concentrations at Cooling Process (9/5/97)
Methane (ppm)
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time


CO Concentrations at Cooling Process (9/5/97)
500.0
450.0
400.0
350.0
300.0
250.0
200.0
150.0
100.0
50.0
0.0
CO (ppm)
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time


Formaldehyde Concentrations at Cooling Process (9/5/97)
Spiked Samples
Formaldehyde (ppm)
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time

3-Methylpentane Concentrations at Cooling Process (9/5/97)



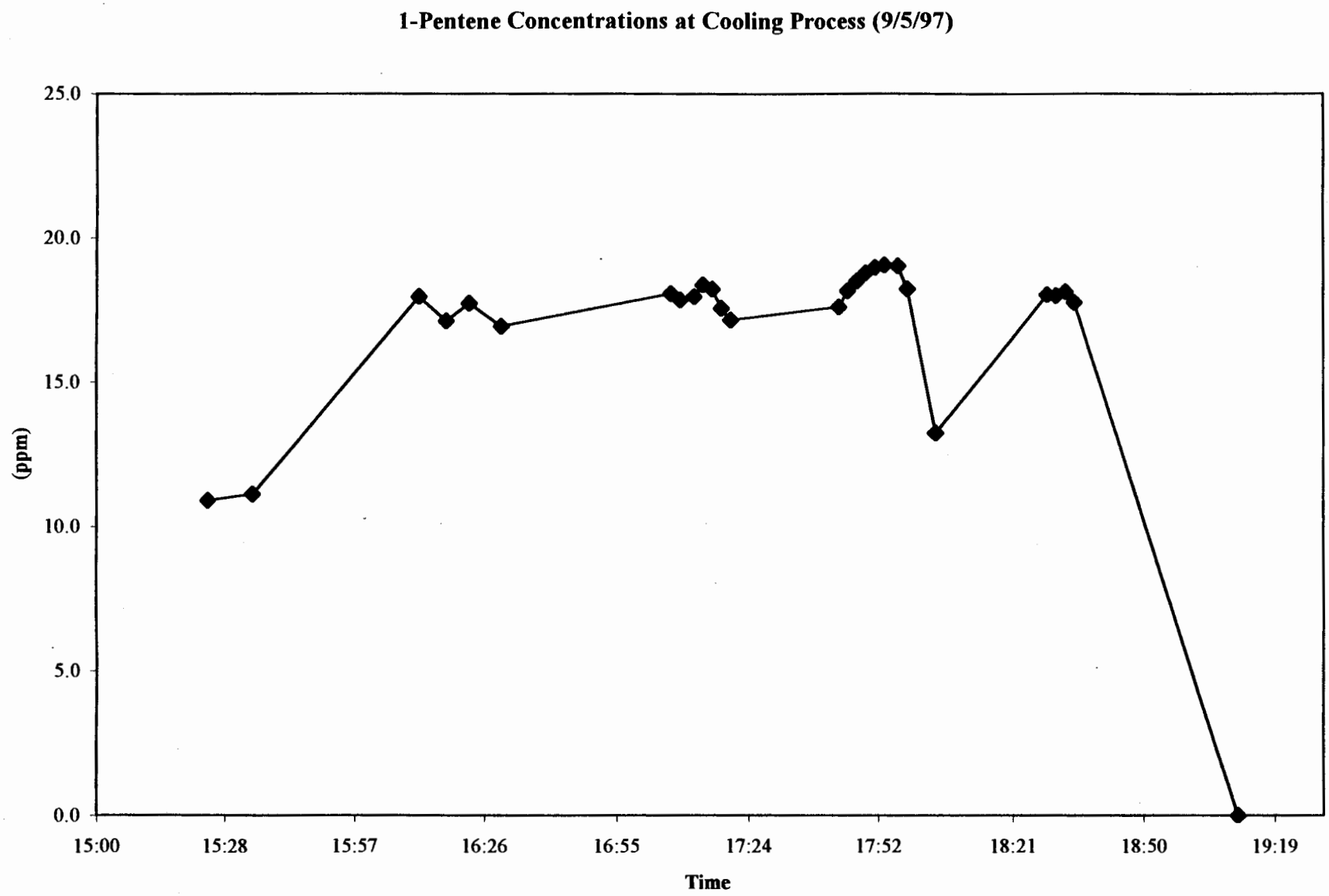

1-Pentene Concentrations at Cooling Process (9/5/97)
25.0
20.0
15.0
10.0
5.0
0.0
(ppm)
15:00
15:28
15:57
16:26
16:55
17:24
17:52
18:21
18:50
19:19
Time

TABLE B-2. FTIR RESULTS FROM THE SHAKE-OUT HOUSING LINE

TABLE B-2. FTIR RESULTS AT THE SHAKE-OUT HOUSING

			Toluene		Hexane		Ethylene		SF_6		Methane		CO		Formaldehyde	
Date	Time	File name [1]	ppm	Unc [2]	ppm	Unc	ppm	Unc	ppm	Unc	ppm	Unc	ppm	Unc	ppm	Unc
9/5/97	**15:51**	**SHKSP101**	**11.1**	**1.8**	**0.0**	**12.1**	**0.0**	**0.8**	**0.464**	**0.016**	**10.7**	**1.2**	**91.1**	**13.3**	**14.6**	**1.0**
	16:05	**SHKSP102**	**11.5**	**1.8**	**0.0**	**11.8**	**0.0**	**0.8**	**0.479**	**0.016**	**13.7**	**1.2**	**93.4**	**12.8**	**14.7**	**1.0**
	16:42	SHKUN103	0.0	2.5	3.0	0.2	1.8	0.6	0.000	0.014	12.2	1.0	78.1	13.8	0.0	1.2
	16:50	SHKUN104	0.0	3.4	0.0	17.1	2.7	0.7	0.000	0.017	17.8	1.6	96.9	17.8	0.0	1.7
	16:55	SHKUN105	0.0	3.6	0.0	18.0	2.9	0.8	0.000	0.018	19.1	1.7	96.2	18.3	0.0	1.7
	17:00	SHKUN106	0.0	3.5	0.0	17.4	2.8	0.7	0.000	0.018	18.4	1.6	94.8	17.9	0.0	1.7
	17:22	19050008	15.4	3.3	0.0	21.9	10.9	0.6	0.000	0.014	130.2	1.9	331.7	21.9	0.0	2.1
	17:24	19050009	0.0	2.4	0.0	12.2	2.1	0.5	0.000	0.012	15.5	1.2	76.1	13.0	0.0	1.2
	17:27	19050010	0.0	3.8	0.0	18.8	3.0	0.8	0.000	0.019	19.0	1.8	102.7	20.1	0.0	1.8
	17:29	19050011	0.0	3.5	0.0	17.4	2.6	0.8	0.000	0.018	16.0	1.6	100.5	19.0	0.0	1.7
	17:31	19050012	0.0	3.3	0.0	16.6	2.6	0.7	0.000	0.017	16.8	1.5	97.2	18.4	0.0	1.6
	17:33	19050013	0.0	3.7	0.0	18.3	3.2	0.8	0.000	0.019	21.4	1.7	104.1	19.9	0.0	1.8
	17:35	19050014	0.0	3.8	0.0	18.8	3.4	0.8	0.000	0.019	23.5	1.7	106.1	20.4	0.0	1.8
	17:37	19050015	0.0	3.8	0.0	18.8	3.3	0.8	0.000	0.019	22.4	1.7	102.4	20.4	0.0	1.8
	18:10	19050030	0.0	3.8	0.0	19.1	3.3	0.8	0.000	0.019	23.0	1.7	91.4	20.2	0.0	1.8
	18:12	19050031	0.0	3.9	0.0	19.2	3.3	0.8	0.000	0.020	22.7	1.7	98.6	20.6	0.0	1.9
	18:14	19050032	0.0	4.0	0.0	20.1	3.5	0.9	0.000	0.020	24.1	1.8	97.5	21.2	0.0	1.9
	18:16	19050033	0.0	3.7	0.0	18.6	3.2	0.8	0.000	0.019	22.5	1.7	85.8	19.7	0.0	1.8
	18:18	19050034	0.0	3.9	0.0	19.3	3.3	0.8	0.000	0.020	23.6	1.7	89.4	20.4	0.0	1.9
	18:20	19050035	0.0	3.7	0.0	18.4	3.2	0.8	0.000	0.019	22.8	1.7	90.2	19.8	0.0	1.8
	18:22	19050036	0.0	3.9	0.0	19.2	3.3	0.8	0.000	0.020	23.6	1.7	86.9	20.3	0.0	1.9
	18:57	**SHKSP107**	**9.3**	**2.0**	**0.0**	**11.8**	**0.0**	**1.4**	**0.605**	**0.026**	**11.7**	**1.2**	**70.3**	**13.2**	**18.7**	**1.1**
		Average --->	0.8	3.5	0.2	17.3	3.4	0.8	0.000	0.018	26.0	1.6	106.7	19.1	0.0	1.7

[1] The samples indicated in bold type, "SHKSP101, "SHKSP102" and "SHKSP107," were spiked with a mixture of formaldehyde vapor, toluene vapor, and SF_6. The spike results are presented in Seciton 4.4, and the analyte spike procedure is discussed in Section 5.3.1.

[2] Unc is th estimated uncertainty in the measurement.

[3] SF_6 was spiked as a tracer gas to determine to spike dilution. SF_6 was not detected in the gas stream.

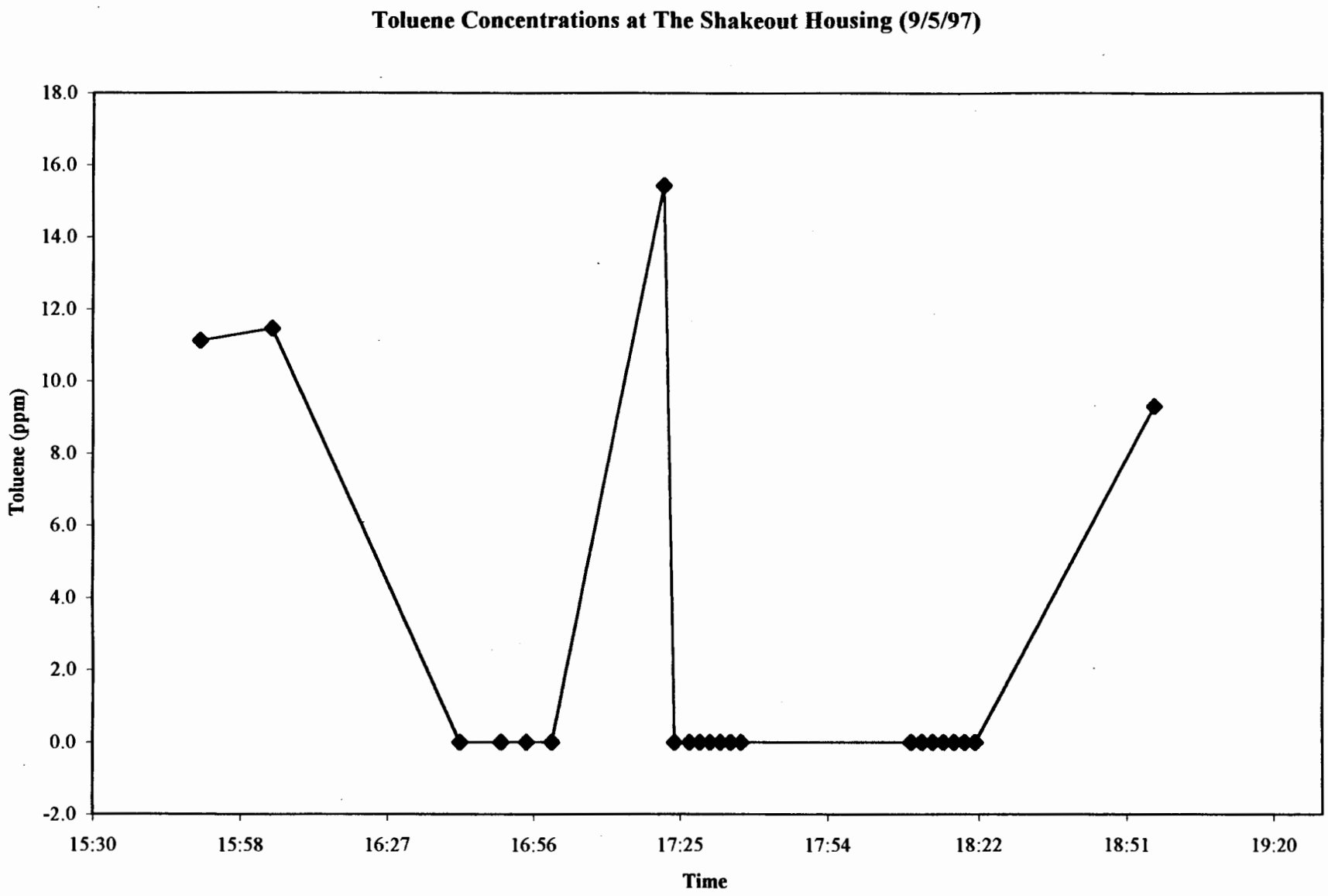
TABLE B-2. Continued.

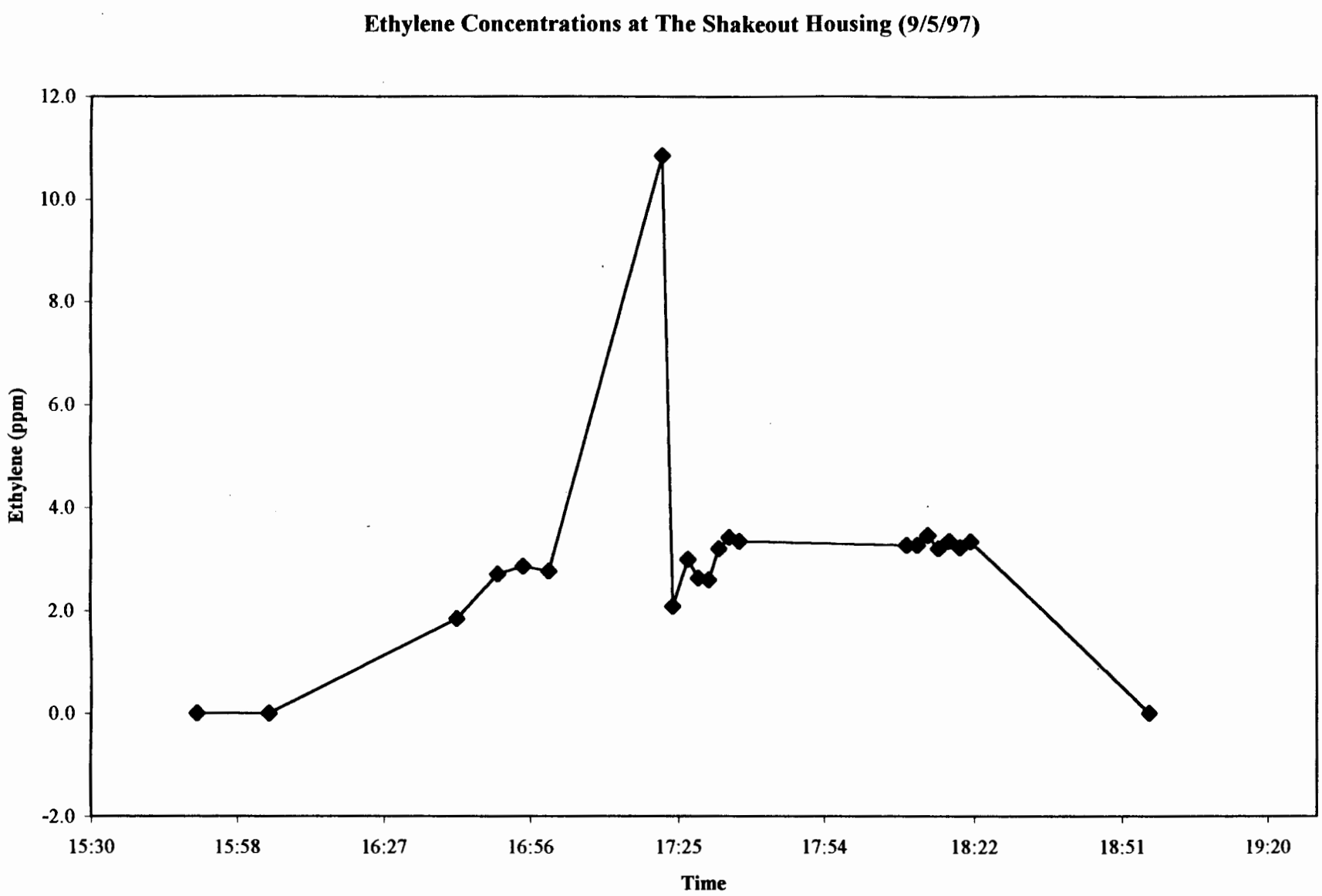
Date	Time	File name [1]	3-Methylepentane		Butane		1-Pentene		2-Methyl-2butene	
			ppm	Unc [2]	ppm	Unc	ppm	Unc	ppm	Unc
9/5/97	**15:51**	**SHKSP101**	**0.0**	**1.0**	**0.0**	**3.9**	**0.0**	**6.2**	**6.4**	**1.0**
	16:05	**SHKSP102**	**0.0**	**1.0**	**0.0**	**3.8**	**0.0**	**6.1**	**6.6**	**1.0**
	16:42	SHKUN103	0.0	0.9	0.0	2.8	0.0	6.4	0.0	0.9
	16:50	SHKUN104	4.2	0.5	0.0	4.0	0.0	8.8	8.2	1.2
	16:55	SHKUN105	4.5	0.5	0.0	4.3	0.0	9.3	8.9	1.2
	17:00	SHKUN106	4.3	0.5	0.0	4.1	0.0	9.0	8.5	1.2
	17:22	19050008	3.6	1.2	0.0	24.9	17.5	2.9	0.0	6.3
	17:24	19050009	3.6	0.4	0.0	13.9	0.0	6.3	6.8	0.9
	17:27	19050010	3.7	1.4	4.8	4.5	0.0	9.7	9.5	1.4
	17:29	19050011	4.4	0.5	0.0	4.1	0.0	8.9	8.5	1.2
	17:31	19050012	3.8	0.5	0.0	3.9	0.0	8.5	6.9	1.1
	17:33	19050013	3.2	1.3	5.3	4.3	0.0	9.4	7.8	1.3
	17:35	19050014	3.4	1.4	5.7	4.4	0.0	9.7	8.5	1.4
	17:37	19050015	3.3	1.4	5.6	4.4	0.0	9.7	8.3	1.4
	18:10	19050030	3.1	1.4	5.6	4.4	0.0	9.8	7.5	1.4
	18:12	19050031	3.1	1.4	5.7	4.5	0.0	9.9	7.5	1.4
	18:14	19050032	3.4	1.4	6.1	4.7	0.0	10.3	8.3	1.5
	18:16	19050033	3.0	1.3	5.5	4.3	0.0	9.6	7.0	1.3
	18:18	19050034	3.1	1.4	5.7	4.5	0.0	9.9	7.3	1.4
	18:20	19050035	3.0	1.3	5.4	4.3	0.0	9.5	6.9	1.3
	18:22	19050036	3.1	1.4	5.7	4.5	0.0	9.9	7.2	1.4
	18:57	**SHKSP107**	**0.0**	**1.1**	**0.0**	**3.8**	**0.0**	**6.1**	**8.4**	**1.1**
		Average --->	3.4	1.0	3.2	5.8	0.9	8.8	7.0	1.5

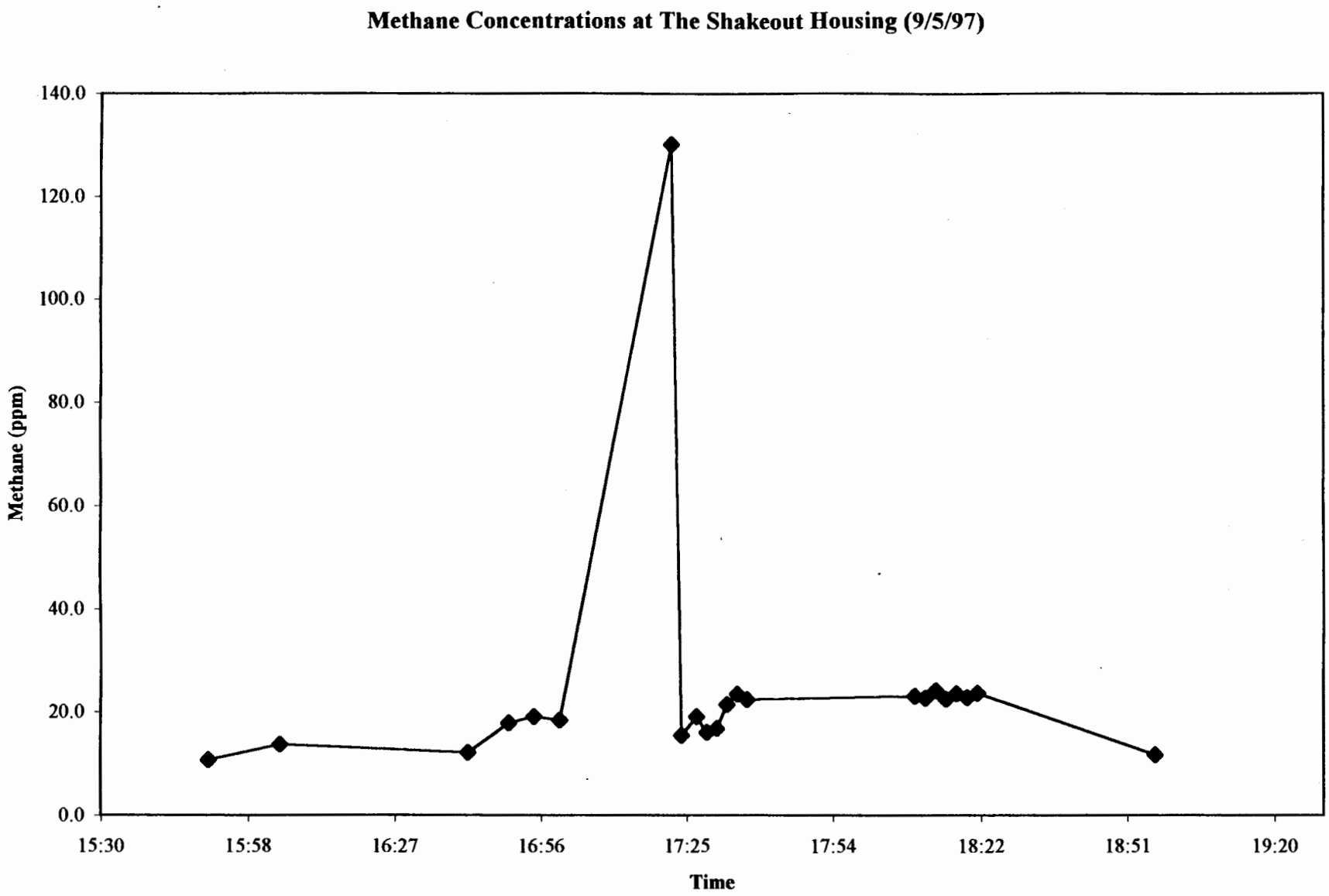
[1] The samples indicated in bold type, "SHKSP101, "SHKSP102" and "SHKSP107," were spiked with a mixture of formaldehyde vapor, toluene vapor, and SF_6. The spike results are presented in Seciton 4.4, and the analyte spike procedure is discussed in Section 5.3.1.

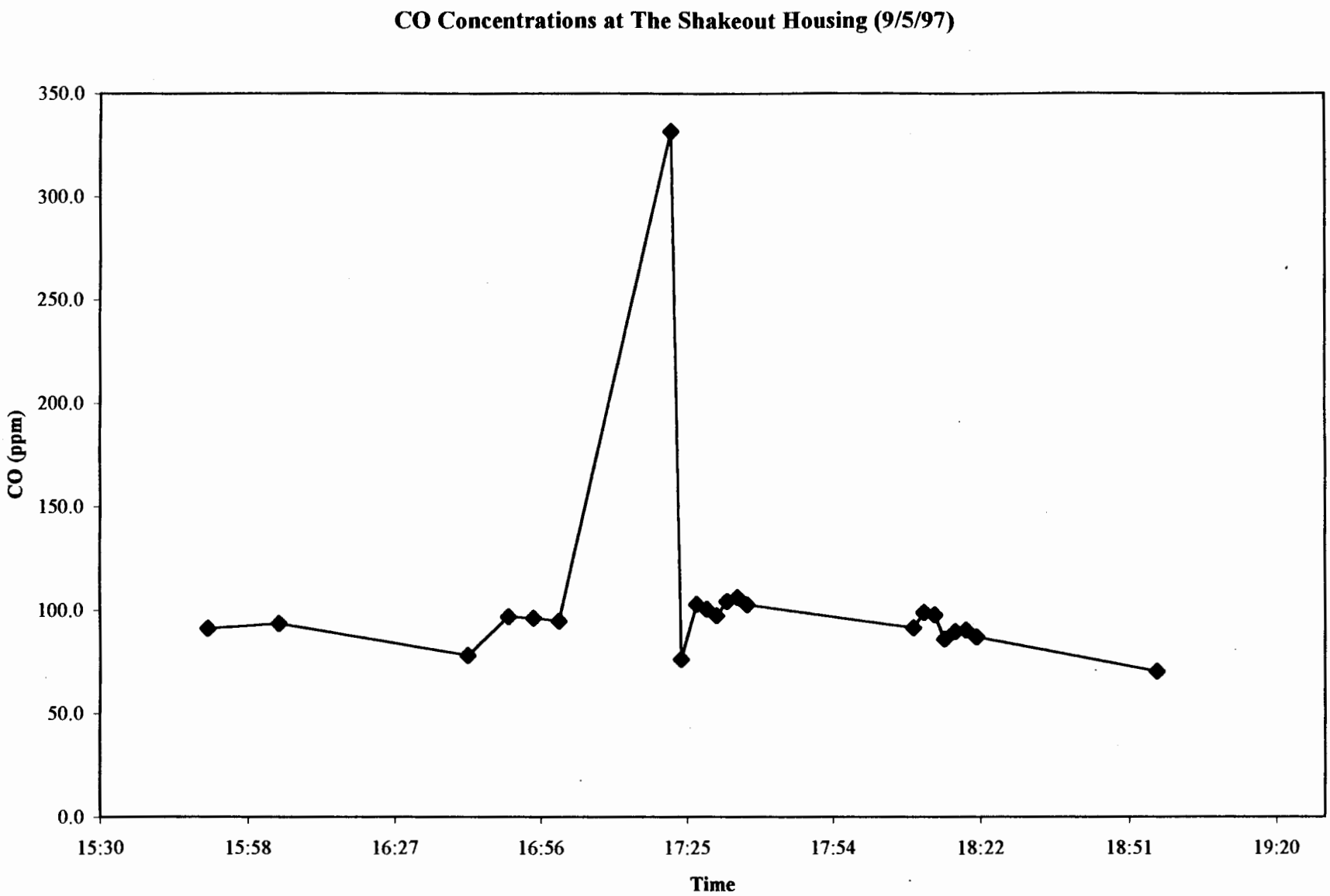
[2] Unc is th estimated uncertainty in the measurement.

[3] SF_6 was spiked as a tracer gas to determine to spike dilution. SF_6 was not detected in the gas stream.

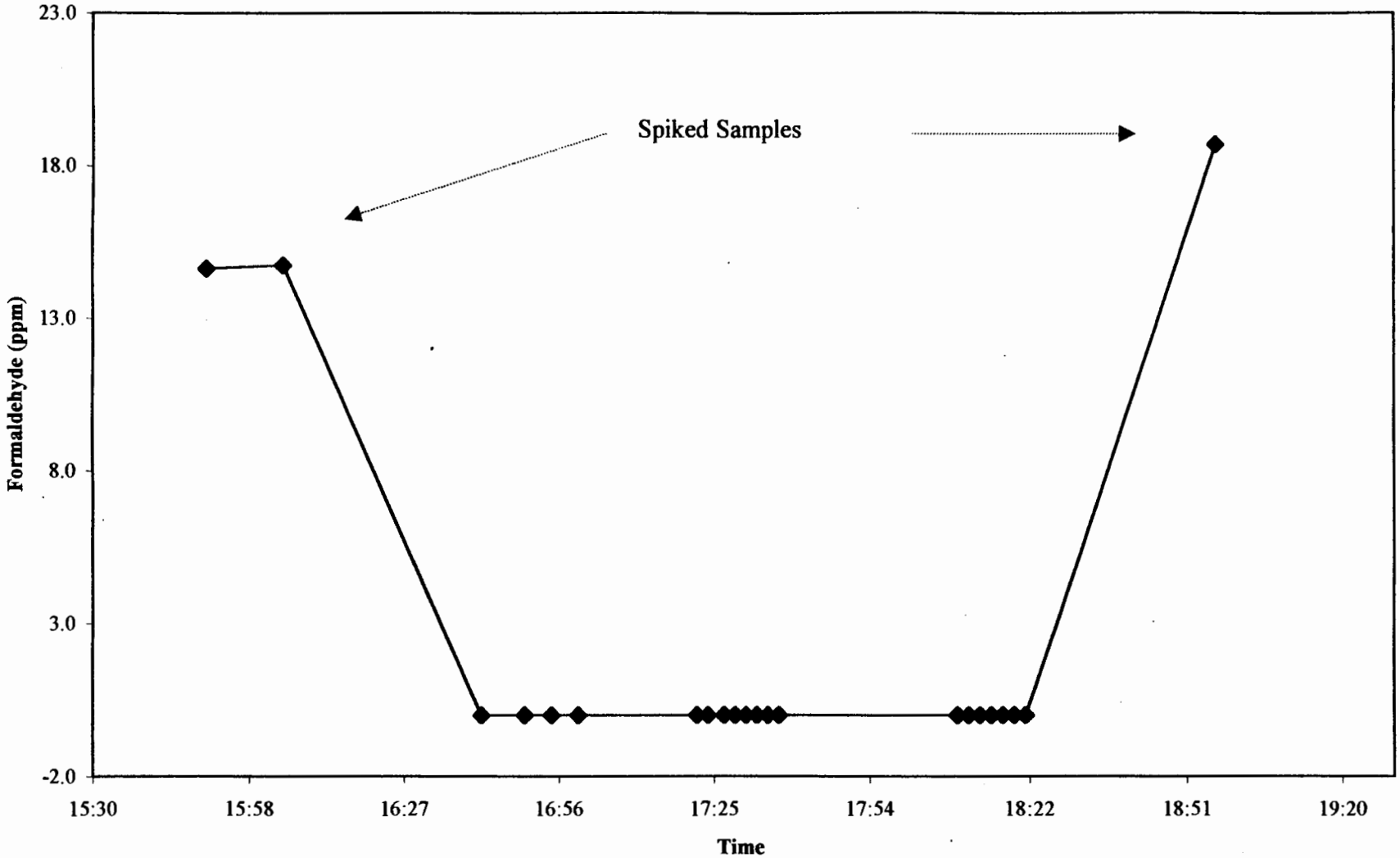

Toluene Concentrations at The Shakeout Housing (9/5/97)
Toluene (ppm)
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time

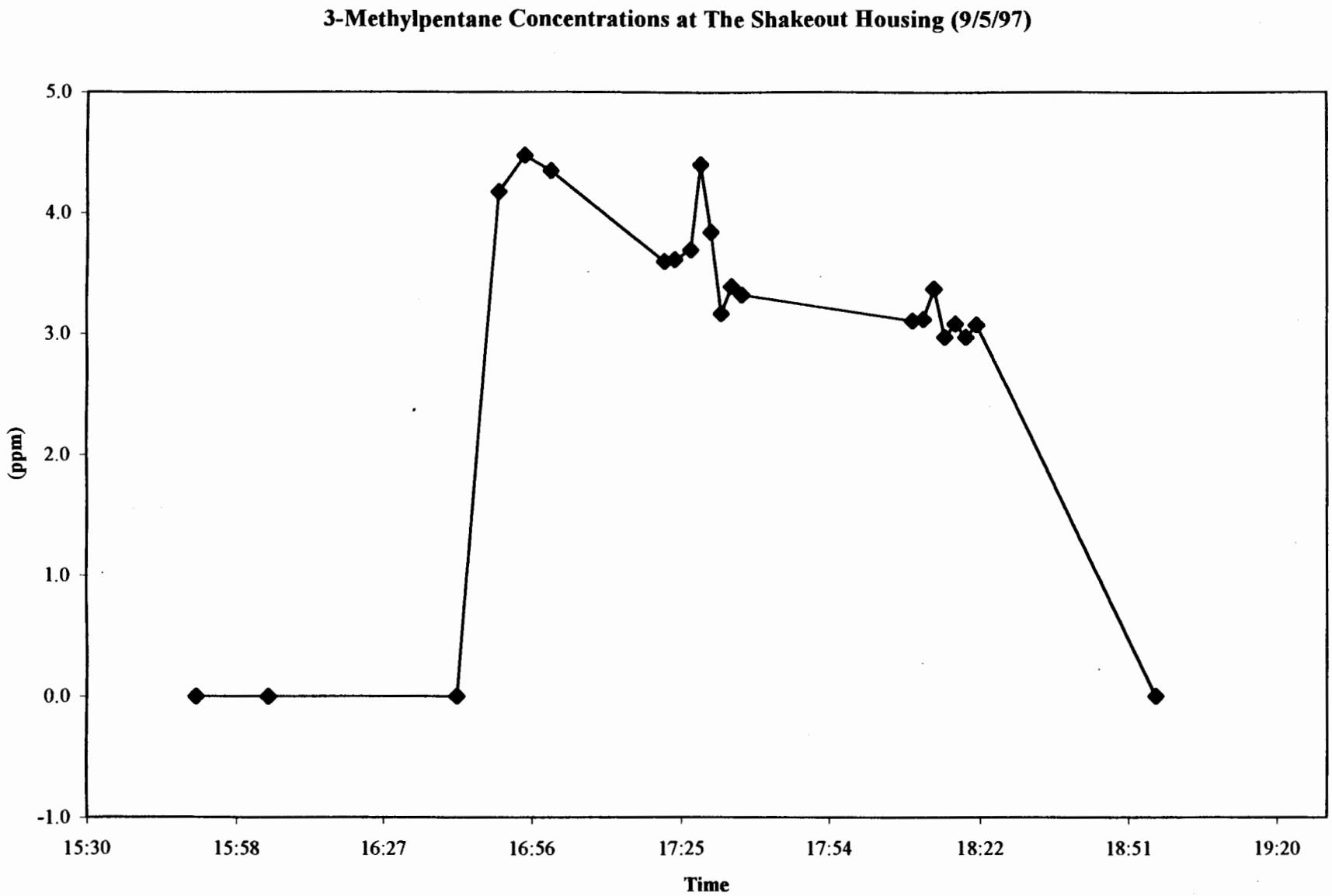

Ethylene Concentrations at The Shakeout Housing (9/5/97)
12.0
10.0
8.0
6.0
4.0
2.0
0.0
-2.0
Ethylene (ppm)
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time

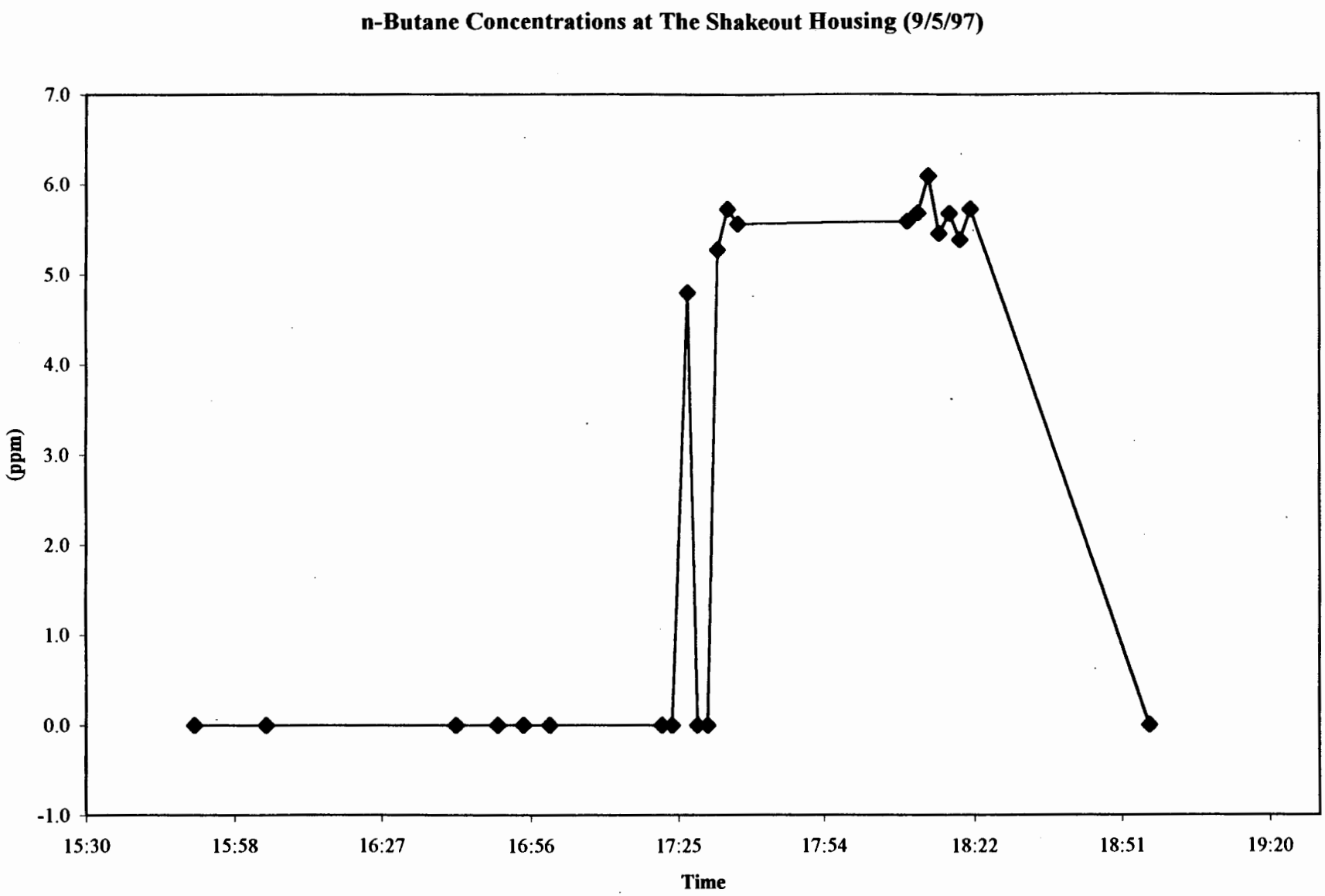

Methane Concentrations at The Shakeout Housing (9/5/97)
Methane (ppm)
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time


CO Concentrations at The Shakeout Housing (9/5/97)
CO (ppm)
350.0
300.0
250.0
200.0
150.0
100.0
50.0
0.0
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time

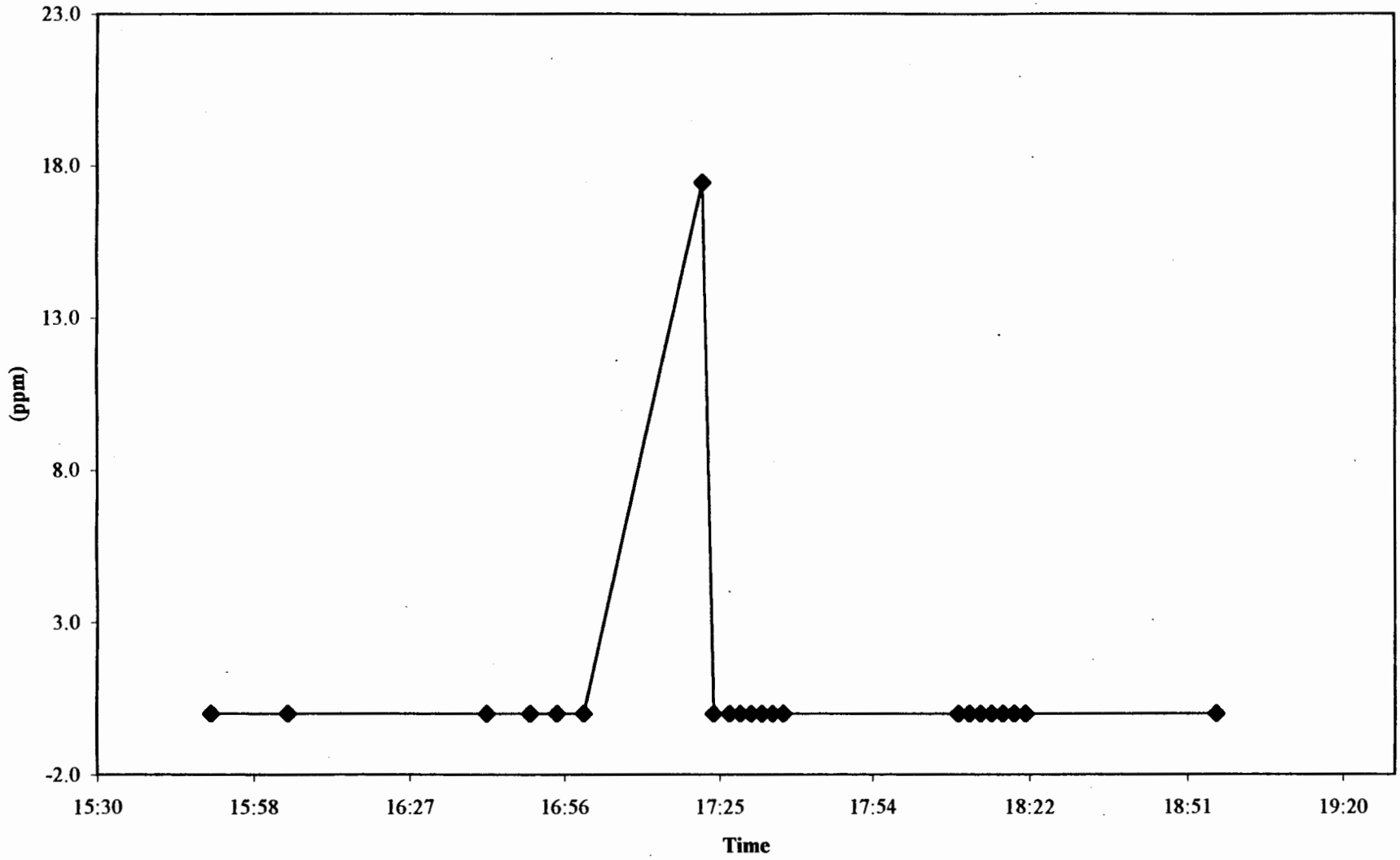
Formaldehyde Concentrations at The Shakeout Housing (9/5/97)




3-Methylpentane Concentrations at The Shakeout Housing (9/5/97)
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
(ppm)
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time


n-Butane Concentrations at The Shakeout Housing (9/5/97)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
(ppm)
15:30
15:58
16:27
16:56
17:25
17:54
18:22
18:51
19:20
Time

1-Pentene Concentrations at The Shakeout Housing (9/5/97)



2-Methyl-2-Butene Concentrations at The Shakeout Housing (9/5/97)

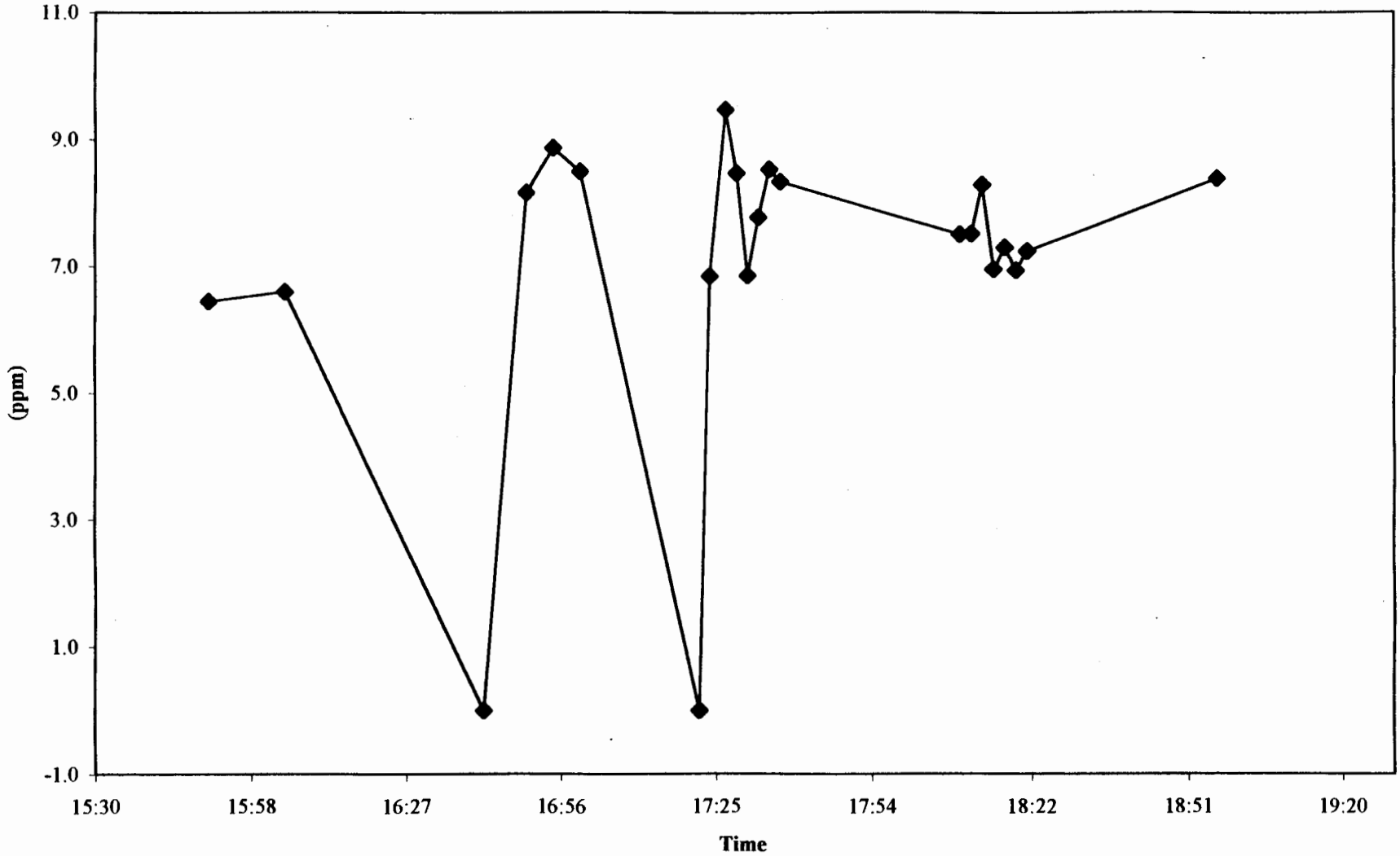


TABLE B-3. FTIR RESULTS AT THE BAGHOUSE INLET

TABLE B-3. FTIR RESULTS AT THE CUPOLA BAGHOUSE INLET

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
9/8/97	12:02	INLSP101	17.58	2.37	10.27	2.08	3.43	0.84	17.37	1.17
	12:09	INLSP102	17.11	2.15	10.73	1.89	3.16	0.76	17.66	1.07
	12:18	INLSP103	3.80	0.52	0.00	0.43	0.00	0.17	1.56	0.24
	12:59	INLUN104	31.73	3.15	0.00	2.78	5.24	1.12	0.00	1.57
	13:05	INLUN105	30.94	3.22	0.00	2.85	5.31	1.14	0.00	1.61
	13:13	INLUN106	34.37	3.32	0.00	2.94	5.48	1.18	0.00	1.66
	13:20	INLUN107	29.57	3.23	0.00	2.86	5.15	1.15	0.00	1.62
	13:30	INLUN108	41.78	3.45	0.00	3.05	5.79	1.23	0.00	1.73
	13:45	INLUN109	27.15	3.23	0.00	2.86	4.99	1.15	0.00	1.62
	13:53	INLUN110	27.25	3.26	0.00	2.89	5.05	1.16	0.00	1.64
	14:30	INLUN111	31.64	3.27	0.00	2.90	5.12	1.16	0.00	1.64
	14:35	INLUN112	34.65	3.08	0.00	2.73	4.95	1.09	0.00	1.54
	14:40	INLUN113	32.32	3.08	0.00	2.73	4.84	1.09	0.00	1.54
	15:21	INLUN114	30.83	3.53	0.00	3.13	5.14	1.25	0.00	1.77
	15:27	INLUN115	29.90	3.29	0.00	2.91	4.87	1.17	0.00	1.65
	15:33	INLUN116	31.63	3.27	0.00	2.89	4.86	1.16	0.00	1.64
	16:11	19080015	43.96	3.59	0.00	3.18	5.42	1.27	0.00	1.80
	16:14	19080016	43.10	3.59	0.00	3.18	5.38	1.27	0.00	1.80
	16:16	19080017	41.10	3.57	0.00	3.15	5.29	1.27	0.00	1.78
	16:31	19080024	27.81	3.38	0.00	2.98	5.32	1.19	4.30	1.55
	17:07	INLSP117	12.99	2.15	24.42	1.46	3.97	0.76	14.64	0.91
	Average -->		33.51	3.32	0.00	2.94	5.19	1.18	0.25	1.66

TABLE B-3. FTIR RESULTS AT THE CUPOLA BAGHOUSE INLET

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
9/9/97	7:49	INLSP201	7.74	2.52	0.00	2.23	3.38	0.89	16.37	1.16
	9:55	INLUN202	33.77	3.53	0.00	3.13	5.48	1.25	0.00	1.77
	10:03	INLUN203	35.02	3.58	0.00	3.18	5.53	1.27	0.00	1.80
	10:11	INLUN204	33.29	3.62	0.00	3.21	5.49	1.28	0.00	1.82
	10:52	INLSP205	12.62	2.68	14.15	2.35	4.00	0.95	17.34	1.33
	11:01	INLSP206	25.69	3.85	0.00	3.41	17.29	1.36	3.74	1.62
	11:09	INLUN207	22.85	4.55	0.00	4.04	5.79	1.62	0.00	2.28
	11:20	INLUN208	18.72	3.61	0.00	3.20	4.51	1.28	0.00	1.81
	12:00	19090001	18.49	3.15	0.00	2.79	4.66	1.12	0.00	1.58
	12:03	19090002	19.77	3.16	0.00	2.81	4.69	1.12	0.00	1.59
	12:05	19090003	20.61	3.17	0.00	2.81	4.73	1.13	0.00	1.59
	12:07	19090004	21.24	3.14	0.00	2.79	4.71	1.11	0.00	1.58
	12:09	19090005	21.67	3.13	0.00	2.78	4.74	1.11	0.00	1.57
	12:11	19090006	22.17	3.14	0.00	2.78	4.72	1.11	0.00	1.57
	12:13	19090007	22.60	3.12	0.00	2.77	4.72	1.11	0.00	1.57
	12:16	19090008	23.19	3.14	0.00	2.79	4.77	1.11	0.00	1.58
	12:52	19090023	24.18	3.53	0.00	3.13	5.10	1.25	0.00	1.77
	12:54	19090024	25.19	3.57	0.00	3.17	5.21	1.27	0.00	1.79
	12:56	19090025	25.79	3.60	0.00	3.20	5.25	1.28	0.00	1.81
	12:59	19090026	26.36	3.62	0.00	3.22	5.33	1.28	0.00	1.82
	13:01	19090027	27.41	3.59	0.00	3.19	5.33	1.27	0.00	1.80
	13:03	19090028	29.70	3.64	0.00	3.24	5.46	1.29	0.00	1.83
	13:05	19090029	31.42	3.66	0.00	3.25	5.56	1.30	0.00	1.84
	13:07	19090030	32.28	3.64	0.00	3.23	5.59	1.29	0.00	1.83
	13:09	19090031	32.84	3.65	0.00	3.24	5.61	1.29	0.00	1.83
	13:12	19090032	33.68	3.68	0.00	3.27	5.62	1.31	0.00	1.85
	13:14	19090033	34.50	3.73	0.00	3.32	5.69	1.33	0.00	1.88
	13:16	19090034	34.82	3.85	0.00	3.42	5.83	1.37	0.00	1.93

TABLE B-3. FTIR RESULTS AT THE CUPOLA BAGHOUSE INLET

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
	13:18	19090035	34.71	3.90	0.00	3.46	5.87	1.39	0.00	1.96
	13:20	19090036	34.45	3.89	0.00	3.46	5.85	1.38	0.00	1.95
	13:52	19090051	29.84	3.89	0.00	3.46	5.63	1.38	0.00	1.96
	13:54	19090052	30.25	3.86	0.00	3.42	5.64	1.37	0.00	1.94
	13:57	19090053	29.86	3.60	0.00	3.19	5.35	1.28	0.00	1.81
	14:19	INSP209	11.67	2.65	15.61	2.32	4.08	0.94	18.60	1.31
	Average -->		27.69	3.56	0.00	3.16	5.28	1.27	0.00	1.79

TABLE B-3. FTIR RESULTS AT THE CUPOLA BAGHOUSE INLET

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
9/10/97	7:53	INLSP301	17.02	2.70	12.51	2.37	3.93	0.95	16.24	1.34
	8:37	INLUN302	39.90	3.67	0.00	3.25	5.12	1.30	0.00	1.84
	8:44	INLUN303	38.41	3.74	0.00	3.31	5.12	1.33	0.00	1.87
	8:50	INLUN304	35.86	3.55	0.00	3.14	4.72	1.26	0.00	1.78
	9:11	19100001	24.82	3.20	0.00	2.84	5.29	1.14	0.00	1.61
	9:41	19100015	38.26	3.43	0.00	3.04	5.50	1.22	0.00	1.72
	9:43	19100016	40.25	3.45	0.00	3.06	5.56	1.23	0.00	1.73
	9:45	19100017	42.10	3.51	0.00	3.11	5.67	1.24	0.00	1.76
	9:47	19100018	42.41	3.51	0.00	3.11	5.65	1.25	0.00	1.76
	9:49	19100019	40.58	3.47	0.00	3.08	5.51	1.23	0.00	1.74
	9:52	19100020	39.16	3.48	0.00	3.08	5.47	1.23	0.00	1.74
	9:54	19100021	38.11	3.46	0.00	3.07	5.41	1.23	0.00	1.74
	9:56	19100022	37.21	3.45	0.00	3.06	5.34	1.23	0.00	1.73
	9:58	19100023	36.49	3.44	0.00	3.05	5.31	1.22	0.00	1.73
	10:00	19100024	36.72	3.45	0.00	3.06	5.32	1.23	0.00	1.73
	10:33	19100039	35.63	3.31	0.00	2.93	4.92	1.17	0.00	1.66
	10:35	19100040	32.74	3.29	0.00	2.91	4.78	1.17	0.00	1.65
	10:37	19100041	28.97	3.26	0.00	2.88	4.62	1.16	0.00	1.63
	10:39	19100042	25.04	3.22	0.00	2.85	4.41	1.14	0.00	1.61
	10:41	19100043	21.66	3.18	0.00	2.82	4.29	1.13	0.00	1.59
	10:43	19100044	20.30	3.22	0.00	2.85	4.20	1.14	0.00	1.61
	10:45	19100045	20.09	3.26	0.00	2.89	4.21	1.15	0.00	1.63
	10:48	19100046	21.09	3.33	0.00	2.94	4.32	1.18	0.00	1.67
	10:50	19100047	22.46	3.37	0.00	2.98	4.40	1.19	0.00	1.69
	10:52	19100048	23.61	3.35	0.00	2.96	4.41	1.19	0.00	1.68
	10:54	19100049	24.49	3.38	0.00	2.99	4.46	1.20	0.00	1.69
	10:56	19100050	24.57	3.37	0.00	2.99	4.40	1.19	0.00	1.69
	10:58	19100051	23.93	3.34	0.00	2.96	4.33	1.18	0.00	1.67

TABLE B-3. FTIR RESULTS AT THE CUPOLA BAGHOUSE INLET

Date	Time	File name	HCl		Toluene		Methane		Formaldehyde	
			ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
	11:49	INLUN305	36.39	3.19	0.00	2.82	5.07	1.13	0.00	1.60
	11:55	INLUN306	25.60	3.00	0.00	2.66	4.54	1.06	0.00	1.50
	11:58	INLUN307	22.57	2.96	0.00	2.62	4.35	1.05	0.00	1.48
	12:00	INLUN308	20.30	2.95	0.00	2.61	4.27	1.05	0.00	1.48
	12:03	INLUN309	17.37	2.92	0.00	2.59	4.13	1.04	0.00	1.46
	12:05	INLUN310	15.92	2.90	0.00	2.57	4.06	1.03	0.00	1.45
	12:10	INLUN311	15.49	2.97	0.00	2.63	4.04	1.05	0.00	1.49
	12:56	INLSP312	7.24	1.95	13.36	1.71	2.63	0.69	31.93	0.97
	Average -->		29.66	3.31	0.00	2.93	4.80	1.17	0.00	1.66

TABLE B-4. FTIR RESULTS AT THE BAGHOUSE OUTLET

TABLE B-4. FTIR RESULTS AT THE CUPOLA BAGHOUSE OUTLET.

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
9/8/97	12:30	OUTSP101	15.24	2.59	7.59	2.27	3.26	0.92	14.95	1.28
	12:41	OUTSP102	3.63	0.60	0.00	0.50	0.00	0.20	1.68	0.28
	14:06	OUTUN103	20.97	2.90	0.00	2.57	4.58	1.03	0.00	1.45
	14:11	OUTUN104	22.67	2.91	0.00	2.58	4.61	1.03	0.00	1.46
	14:18	OUTUN105	23.28	2.93	0.00	2.60	4.63	1.04	0.00	1.47
	14:47	OUTUN106	22.02	3.04	0.00	2.69	4.65	1.08	0.00	1.52
	14:54	OUTUN107	21.36	3.19	0.00	2.82	4.77	1.13	0.00	1.59
	15:00	OUTUN108	21.39	3.11	0.00	2.75	4.63	1.10	0.00	1.55
	15:05	OUTUN109	21.56	2.99	0.00	2.65	4.48	1.06	0.00	1.50
	15:12	OUTUN110	21.24	3.00	0.00	2.66	4.44	1.06	0.00	1.50
	15:41	19080001	20.78	3.33	0.00	2.95	4.73	1.18	0.00	1.67
	15:43	19080002	21.94	3.39	0.00	3.00	4.83	1.20	0.00	1.70
	15:46	19080003	22.29	3.41	0.00	3.02	4.85	1.21	0.00	1.71
	15:48	19080004	22.49	3.41	0.00	3.02	4.86	1.21	0.00	1.71
	15:50	19080005	21.90	3.41	0.00	3.02	4.79	1.21	0.00	1.71
	15:52	19080006	21.77	3.41	0.00	3.02	4.76	1.21	0.00	1.71
	15:54	19080007	21.96	3.41	0.00	3.02	4.76	1.21	0.00	1.71
	15:56	19080008	22.21	3.42	0.00	3.03	4.75	1.21	0.00	1.71
	15:59	19080009	21.99	3.40	0.00	3.01	4.73	1.20	0.00	1.70
	16:01	19080010	22.41	3.40	0.00	3.01	4.73	1.20	0.00	1.70
	16:03	19080011	22.81	3.38	0.00	2.99	4.73	1.20	0.00	1.69
	16:05	19080012	23.12	3.38	0.00	2.99	4.75	1.20	0.00	1.69
	16:22	19080020	29.85	3.42	0.00	3.03	4.95	1.21	0.00	1.71
	16:24	19080021	30.04	3.44	0.00	3.04	4.95	1.22	0.00	1.72
	16:26	19080022	29.91	3.44	0.00	3.04	4.94	1.22	0.00	1.72
	16:29	19080023	29.32	3.43	0.00	3.04	4.88	1.22	0.00	1.72
	16:57	OUTSP111	14.33	2.41	21.20	1.63	4.13	0.85	12.93	1.01
Average -->			23.30	3.27	0.00	2.90	4.74	1.16	0.00	1.64

TABLE B-4. FTIR RESULTS AT THE CUPOLA BAGHOUSE OUTLET.

Date	Time	File name	HCl ppm	HCl Uncertainty	Toluene ppm	Toluene Uncertainty	Methane ppm	Methane Uncertainty	Formaldehyde ppm	Formaldehyde Uncertainty
9/9/97	8:02	OUTSP201	10.80	2.68	0.00	2.36	3.47	0.95	13.38	1.23
	8:26	OUTSP202	1.65	0.42	0.00	0.33	0.00	0.13	1.23	0.19
	9:27	OUTUN203	18.03	3.26	0.00	2.90	5.04	1.16	0.00	1.64
	9:33	OUTUN204	20.30	3.20	0.00	2.85	4.98	1.14	0.00	1.61
	9:40	OUTUN205	21.34	3.22	0.00	2.86	5.01	1.14	0.00	1.62
	9:47	OUTUN206	21.33	3.22	0.00	2.86	5.00	1.14	0.00	1.62
	10:19	OUTUN207	21.10	3.35	0.00	2.97	4.93	1.19	0.00	1.68
	10:33	OUTSP208	14.80	2.56	11.22	2.24	3.79	0.90	14.10	1.27
	10:41	OUTUN209	18.78	3.46	0.00	3.06	4.81	1.23	0.00	1.73
	11:31	OUTUN210	12.43	3.42	0.00	3.03	4.33	1.21	0.00	1.71
	11:37	OUTUN211	12.04	3.24	0.00	2.87	4.09	1.15	0.00	1.62
	12:24	19090011	16.08	4.81	0.00	4.26	6.95	1.71	0.00	2.41
	12:26	19090012	13.23	3.09	0.00	2.75	4.54	1.10	0.00	1.55
	12:28	19090013	13.66	3.18	0.00	2.82	4.64	1.13	0.00	1.60
	12:33	19090014	13.94	3.23	0.00	2.87	4.67	1.15	0.00	1.62
	12:35	19090015	13.88	3.23	0.00	2.87	4.67	1.15	0.00	1.62
	12:37	19090016	13.96	3.27	0.00	2.90	4.69	1.16	0.00	1.64
	12:39	19090017	14.18	3.31	0.00	2.94	4.72	1.18	0.00	1.66
	12:41	19090018	14.00	3.31	0.00	2.94	4.71	1.17	0.00	1.66
	12:44	19090019	13.67	3.30	0.00	2.93	4.69	1.17	0.00	1.66
	12:46	19090020	13.70	3.32	0.00	2.95	4.70	1.18	0.00	1.67
	13:27	19090039	17.22	3.58	0.00	3.18	5.12	1.27	0.00	1.80
	13:29	19090040	17.22	3.52	0.00	3.12	5.06	1.25	0.00	1.77
	13:31	19090041	17.36	3.52	0.00	3.13	5.06	1.25	0.00	1.77
	13:33	19090042	17.35	3.57	0.00	3.17	5.10	1.27	0.00	1.79
	13:35	19090043	16.85	3.50	0.00	3.10	5.02	1.24	0.00	1.76
	13:37	19090044	16.90	3.47	0.00	3.08	5.00	1.23	0.00	1.74
	13:39	19090045	17.17	3.50	0.00	3.11	5.02	1.24	0.00	1.76
	13:42	19090046	17.00	3.46	0.00	3.07	5.00	1.23	0.00	1.74
	13:44	19090047	17.13	3.45	0.00	3.06	4.96	1.22	0.00	1.73
	13:46	19090048	18.07	3.75	0.00	3.33	5.25	1.33	0.00	1.88
Average -->			16.30	3.39	0.39	3.01	4.88	1.20	0.00	1.70

TABLE B-4. FTIR RESULTS AT THE CUPOLA BAGHOUSE OUTLET.

			HCl		Toluene		Methane		Formaldehyde	
Date	Time	File name	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty	ppm	Uncertainty
9/10/97	8:03	OUTSP301	15.73	2.71	9.98	2.37	3.69	0.96	13.96	1.34
	8:19	OUTUN302	19.45	3.18	0.00	2.81	4.17	1.13	0.00	1.59
	8:25	OUTUN303	20.97	3.34	0.00	2.96	4.43	1.18	0.00	1.67
	8:31	OUTUN304	21.67	3.36	0.00	2.97	4.39	1.19	0.00	1.68
	9:13	19100002	25.50	3.21	0.00	2.85	5.32	1.14	0.00	1.61
	9:15	19100003	26.02	3.21	0.00	2.85	5.32	1.14	0.00	1.61
	9:17	19100004	26.29	3.19	0.00	2.84	5.30	1.13	0.00	1.60
	9:19	19100005	26.47	3.19	0.00	2.83	5.27	1.13	0.00	1.60
	9:21	19100006	27.13	3.21	0.00	2.85	5.27	1.14	0.00	1.61
	9:24	19100007	28.19	3.22	0.00	2.86	5.30	1.14	0.00	1.62
	9:26	19100008	28.71	3.20	0.00	2.84	5.27	1.14	0.00	1.61
	9:28	19100009	28.96	3.22	0.00	2.86	5.29	1.14	0.00	1.62
	9:30	19100010	28.59	3.25	0.00	2.88	5.30	1.15	0.00	1.63
	9:32	19100011	28.79	3.25	0.00	2.88	5.29	1.15	0.00	1.63
	10:07	19100027	27.90	3.23	0.00	2.86	4.93	1.15	0.00	1.62
	10:09	19100028	27.98	3.19	0.00	2.83	4.86	1.13	0.00	1.60
	10:11	19100029	27.34	3.13	0.00	2.77	4.83	1.11	0.00	1.57
	10:13	19100030	26.53	3.11	0.00	2.76	4.76	1.11	0.00	1.56
	10:15	19100031	25.42	3.09	0.00	2.74	4.69	1.10	0.00	1.55
	10:17	19100032	24.07	3.06	0.00	2.71	4.57	1.09	0.00	1.53
	10:20	19100033	22.81	3.04	0.00	2.69	4.49	1.08	0.00	1.52
	10:22	19100034	21.97	3.03	0.00	2.68	4.43	1.07	0.00	1.51
	10:24	19100035	21.62	3.02	0.00	2.67	4.40	1.07	0.00	1.51
	10:26	19100036	22.64	3.04	0.00	2.69	4.42	1.08	0.00	1.52
	11:20	OUTUN305	16.00	2.92	0.00	2.59	4.49	1.04	0.00	1.47
	11:26	OUTUN306	19.13	2.92	0.00	2.59	4.55	1.04	0.00	1.46
	11:35	OUTUN307	21.27	2.98	0.00	2.65	4.67	1.06	0.00	1.50
	11:41	OUTUN308	22.53	3.05	0.00	2.71	4.80	1.08	0.00	1.53
	12:18	OUTUN309	13.41	2.96	0.00	2.62	4.25	1.05	0.00	1.48
	12:21	OUTUN310	13.98	2.96	0.00	2.62	4.23	1.05	0.00	1.48
	12:24	OUTUN311	14.70	2.97	0.00	2.63	4.26	1.05	0.00	1.49
	12:29	OUTUN312	15.18	3.05	0.00	2.70	4.37	1.08	0.00	1.53
	12:31	OUTUN313	15.32	3.10	0.00	2.75	4.42	1.10	0.00	1.56
	12:33	OUTUN314	15.43	3.09	0.00	2.73	4.40	1.10	0.00	1.55
	12:36	OUTUN315	15.72	3.08	0.00	2.73	4.43	1.09	0.00	1.54
	12:48	OUTSP316	10.09	2.29	10.73	2.01	3.13	0.81	25.75	1.13
Average -->			22.58	3.12	0.00	2.76	4.74	1.11	0.00	1.56

B-2. FTIR FIELD DATA RECORDS

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 758 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/4/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
19:11	BKG0904b		Nitrogen in cell	500	2	130C				
	CTS0904a		20 ppm ethylene	250	2	130C				BKG0904b

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 755 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/5/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
9:30	BKG0905a	20m	Nitrogen flowing	500	2	130C			5 lpm	
	BKGCHK01		N2 only as nitrogen absorbance	250	2	130C			5 lpm	BKG0905a
9:45	CTS0905a		20 ppm ethylene	250	2	130C			5 lpm	BKG0905a
9:53	CTS0905b		20 ppm ethylene							
9:55			Leak check - cell at vacuum							
10:00	SF60905a		4 ppm SF6 - direct to cell	250	2	130C			5 lpm	BKG0905a
	SF60905b		4 ppm SF6 - direct to cell	250	2	130C			5 lpm	BKG0905a
10:11	TOL0905a		Toluene 60 ppm - direct to cell	250	2	130C			5 lpm	BKG0905a
10:15			Process down							
	SFTOL01		SF6 through kintek at 1 lpm and mixed with							
			toluene at 1 lpm, formaldehyde only been in							
			oven for 90 min @ 100C							
			(Bad leak in Kintek)							
10:54	BKG0905b		N2	500	2	130C			5 lpm	
11:20	SF60905c		SF6 @4.97 lpm to spike line	250	2	130C	spike		5 lpm	
			Shakeout - sample rate @ 4.0 lpm							BKG0905b
	SF60905d		SF6 @4.97 lpm - sample rate @ 4.0 lpm	250	2	130C	spike		5 lpm	BKG0905b
			Spike line ** Kintek was venting -VOID samples							
	SF60905e		SF6 @ 5.00 lpm - sample rate @ 4.0 lpm	250	2	130C	spike		5 lpm	BKG0905b
13:15			Process restarted - Computer clock is 1-hr							
			ahead of recorded times							
13:30			Refilled Dewar							
13:53	FORMAL01	20m	Formaldehyde in N2 - direct to cell	250	2	130C			1 lpm	BKG0905b
			1.0 lpm @ 100C - Permeation tube=94,000nanoL/min (~90 ppm)							
			Serial # 2214							
14:10	SFTOL02		SF6 4 ppm @ 1 lpm into Kintek	250	2	130C	spike		2 lpm	BKG0905b
			Toluene 60 ppm, 1 lpm into MFC							
			formaldehyde @ 100C and 1 lpm							
14:20	N2ONLY01		N2 only	250	2	130C			5 lpm	BKG0905b
14:24	BKG0905c		Background - N2 only	500	2	130C			5 lpm	
15:00			Probe inserted in cooling stack w/ spike							
15:23	COOSP101		Cooling stack	250	2	130C	spike		3.5 lpm	BKG0905c
			Spiking w/SF6 (4ppm) and formaldehyde at 1.0 lpm							
			and 100C and toluene (60ppm) @ 1.0 lpm							
			cell flow = 3.5 lpm, vent flow = 2 lpm							

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 755 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/5/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
15:33	COOSP102		Cooling w/spike	250	2	130C	spike			BKG 0905c
15:37			Shake out - probe in stack with spike							
15:39	SHKSP101		Shakeout w/spike	250	2	130C	spike			BKG 0905c
			same spike							
16:02	SHKSP102		Shakeout w/spike	250	2	130C	spike			BKG 0905c
16:11	COOUN103	20m	Cooling line only	250	2	130C	UN	cooling line	5 lpm	0905c
16:15	COOUN104	20m	Cooling line only	250	2	130C	UN	cooling line	5 lpm	0905c
16:21	COOUN105	20m	Cooling line only	250	2	130C	UN	cooling line	5 lpm	0905c
16:27	COOUN106	20m	Cooling line only	250	2	130C	UN	cooling line	5 lpm	0905c
16:41	SHKUN103	20m	Shakeout line	250	2	130C	UN	shakeout	5 lpm	0905c
16:48	SHKUN104	20m	Shakeout line	250	2	130C	UN	shakeout	5 lpm	0905c
16:53	SHKUN105	20m	Shakeout line	250	2	130C	UN	shakeout	5 lpm	0905c
16:58	SHKUN106	20m	Shakeout line	250	2	130C	UN	shakeout	5 lpm	0905c
			Start continuous process software							
17:05	19050001		Cooling							
	1905007		Stop - last good file							
	1905008		Change to shakeout							
	1905009		Evacuated cell							
17:21	19050010		Shakeout	250	2	130C	UN	shakeout	5 lpm	0905c
17:38	19050015		Stop							
	19050016		Switch lines and evacuate cell	250	2	130C	UN	shakeout	5 lpm	0905c
	19050017		Switch lines and evacuate cell	250	2	130C	UN	shakeout	5 lpm	0905c
			Continuous Software							
17:41	19050018		Cooling line	250	2	274	UN		5 lpm	0905c
17:58	19050025		Stop							
	19050026		Change line - Evacuate cell	250	2	274	UN		5 lpm	0905c
	19050027		Change line - Evacuate cell	250	2	274	UN		5 lpm	0905c

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 755 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/5/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
18:03	19050028		Cooling (**BAD**)	250	2	274	UN		5 lpm	0905c
			Reevacuate cell							
18:07	19050030		Shakeout	250	2	274	UN		5 lpm	0905c
	19050036		Stop							
	19050037-38		Change line - Evacuate cell							
18:29	19050039		Cooling line	250	2	274	UN		5 lpm	0905c
18:35	1905042		Stop							
18:50	SHKSP107		Shakeout w/spike	250	2	274	spike		3.0 lpm	0905c
			spike is SF6 (4ppm) @ 1.0 lpm							
			formaldehyde @ 100C @ 1.0 lpm							
			Toluene (60 ppm) @ 1.0 lpm							
			Sample rate 3.0 lpm							
			vent ~ 1.0 lpm							
19:11	COOSP107		Cooling line w/spike	250	2	274	spike		3.0 lpm	0905c
			Spike = 1.0 lpm SF6 in formaldehyde @ 100C							
			w/1.0 lpm toluene (Toluene is 60 ppm and SF6							
			is 4 ppm)							
19:23	SFTOL03		Direct to cell	250	2	274	spike		2.0 lpm	0905c
			spike mix 1.0 lpm SF6 @ 4 ppm							
			and 1.0 lpm toluene							
			and formaldehyde @ 100C							
			Formaldehyde @ ~ 94,000 nanoL/min							
19:31	N2ONLY02		N2 only direct to cell	250	2	274			5.0 lpm	0905c
19:38	CTS0905c		Ethylene 20 ppm	250	2	274			5.0 lpm	0905c
19:45	BKG0905d		N2 only - Background	500	2	274	UN		5.0 lpm	

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 747 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/8/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
9:45			Detector filled							
10:24	BKG0908a	20m	Background - N2 only	500	2	274		dynamic	5.0 lpm	
10:36	N2ONLY03		N2 only	250	2	274		dynamic	5.0 lpm	BKG0908a
10:45	CTS0908a		20 ppm Ethylene	250	2	274		dynamic	5.0 lpm	BKG0908a
10:50	CTS0908b		20 ppm Ethylene	250	2	274		dynamic	5.0 lpm	BKG0908a
10:59			Changed filter line #2 - inlet flow = 12 lpm							
11:07	SFTOL04		Direct to cell spike	250	2	274		dynamic	2.0 lpm	BKG0908a
			SF6 4 ppm @ 1.0 lpm							
			w/ formaldehyde @ 100C and toluene 60 ppm							
			@ 1.0 lpm (toluene = 60.6 ppm) from Scott							
			gas cylinder #ALM052730 MRI PO# 029872							
			(FORMALDEHYDE ABSORBANCE WAS LESS THAN 9/5/97)							
	SFTOL05		same as above	250	2	274		dynamic	2.0 lpm	BKG0908a
12:02	INLSP101	20m	Spike to inlet - formaldehyde OK	250	2	274		dynamic	3.0 lpm	BKG0908a
			Cell flow = 3.0 lpm							
			Vent flow = 2.0 lpm							
12:09	INLSP102		same as above	250	2	274		dynamic	3.0 lpm	BKG0908a
12:18	INLSP103		N2 only - flood N2 into line	250	2	274		dynamic	3.0 lpm	BKG0908a
			spike = 10 lpm of N2 only							
			(Line #1 = outlet, Line #2 = inlet)							
			Inlet and outlet leak checks good							
12:27	OUTSP101		Spike w/toluene 60 ppm @ 1.0 lpm	250	2	275	SP	dynamic	3.0 lpm	BKG0908a
			formaldehyde @ 100C and SF6 @ 1.0 lpm							
			cell flow = 3.0 lpm							
			vent flow = 2.0 lpm - OUTLET-							
12:40	OUTSP102		N2 only - Sample line	250	2	275		dynamic	5.0 lpm	BKG0908a
			Outlet sample line							
12:46	BKG0908b		Background - N2 direct to cell	500	2	275		dynamic	5.0 lpm	

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 747 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/8/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
12:56	INLUN104		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:02	INLUN105		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:10	INLUN106		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:17	INLUN107		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:27	INLUN108		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:28			Shut probe box of outlet down to check pitots							
			Changed outlet probe							
13:42	INLUN109		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
13:49	INLUN110		Inlet sample	250	2	274	UN	dynamic	5.0 lpm	0908b
14:04	OUTUN103		Outlet	250	2	275	UN	dynamic	5.0 lpm	0908b
14:09	OUTUN104		Outlet	250	2	275	UN	dynamic	5.0 lpm	0908b
14:15	OUTUN105		Outlet	250	2	275	UN	dynamic	5.0 lpm	0908b
14:28	INLUN111		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
14:33	INLUN112		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
14:38	INLUN113		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
14:45	OUTUN106		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
14:51	OUTUN107		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
14:58	OUTUN108		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
15:03	OUTUN109		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
15:09	OUTUN110		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
15:18	INLUN114		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
15:23			Refilled dewar							
15:25	INLUN115		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
15:30	INLUN116		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 747 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/8/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
			Outlet start continuous sampling							
15:39	19080001		Outlet	250	2	274	UN	dynamic	5.0 lpm	0908b
16:04	19080012		Stop							
			Change to inlet evacuate 19080013 and 19080014							
16:09	19080015		Inlet	250	2	274	UN	dynamic	5.0 lpm	0908b
16:15	19080017		Stop							
	19080018-19		Evacuate cell							
	19080020		Outlet - Continuous software	250	2	273	UN	dynamic	5.0 lpm	0908b
16:28	19080023		Stop							
16:39	BKG0908c		N2 only - Background	500	2	274	UN	dynamic	5.0 lpm	
16:55	OUTSP111		Spike - toluene 60 ppm @ 2.0 lpm	500	2	274	spike	dynamic	5.0 lpm	0908c
			SF6 4ppm @ 1.0 lpm and formaldehyde @ 100C							
			toluene high in this spike							
17:05	INLSP117		Spike - same as above				spike			
17:11	CTS0908c		Ethylene 20 ppm, direct to cell	250	2	274		dynamic	5.0 lpm	0908c
17:21	SFTO106		Spike direct to cell							
			Same as spikes above							
			Toluene, 60 ppm @ 2.0 lpm							
			SF6 4ppm @ 1.0 lpm							
			Formaldehyde @ 100C							
17:10			Probes pulled out of stack							
17:13			Inlet and outlet pass leak check							
17:28	N2ONLY04		N2 Only	250	2	274		dynamic	5.0 lpm	0908c
17:33	OUTAIR01		Air only	250	2	274		dynamic	5.0 lpm	0908c
17:39	INAIR01		Air only	250	2	274		dynamic	5.0 lpm	0908c

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 745 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/9/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
7:10			Passed inlet and outlet check							
7:15			filled detector							
7:22	EVC0908		Evacuated cell - 0.9 mm Hg	250						0908c
7:27	BKG0909a	20m	Background	500	2	275		dynamic	5.0 lpm	
7:37	CTS0909a		Ethylene 20 ppm	250	2	274		dynamic	5.0 lpm	0909a
7:47	INSP201		Inlet - spike	250	2	274	SP	dynamic	3.0 lpm	0909a
			SF6 - 4ppm @ 2.0 lpm							
			with formaldehyde @ 100C							
			Cell flow = 3.0 lpm, vent flow = 2.0 lpm							
8:00	OUTSP201		Outlet spike	250	2	274	SP	dynamic	3.0 lpm	0909a
			same as above (no toluene)							
			Cell leak check @ vacuum 0.6 mmHg in 60 sec							
8:11	SFT0909a		Spike direct to cell	250	2	274	SP	dynamic	3.0 lpm	0909a
			same as mix above (no toluene)							
8:24	OUTSP202		N2 only in line , OUTLET	250	2	274	SP	dynamic	3.0 lpm	0909a
8:57	BKG0909b		N2 only, background	500	2	274		dynamic	5.0 lpm	
9:06	BKG0909c		N2 only, background	500	2	274		dynamic	5.0 lpm	
9:25	OUTUN203		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909c
9:31	OUTUN204		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909c
9:36	OUTUN205		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909c
9:44	OUTUN206		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909c
			Purge and evaluate							
9:54	INLUN202		INLET	250	2	275	UN	dynamic	5.0 lpm	0909c
10:00	INLUN203		INLET	250	2	275	UN	dynamic	5.0 lpm	0909c
10:08	INLUN204		INLET	250	2	275	UN	dynamic	5.0 lpm	0909c
10:16	OUTUN207		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909c
10:30	OUTSP208		Outlet spike	250	2	275	spike	dynamic	3.0 lpm	0909c
			Toluene, 60 ppm @ 1.0 lpm							
			SF6, 4ppm @ 1.0 lpm w/form @ 100C							
			Cell flow = 3.0 lpm							
			Vent flow = 3.0 lpm							
10:39	OUTUN209		Outlet only	250	2	275	UN	dynamic	3.0 lpm	0909c
10:30			Manual sampling started							

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 745 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/9/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
10:50	INLSP205		Inlet spike	250	2	275	SP	dynamic	3.0 lpm	0909c
			at inlet probe box							
			toluene = 1.0 lpm, SF6 = 1.0 lpm, form = 100C							
			Cell flow = 3.0 lpm, vent = 2.0 lpm							
11:00	INSP207		Inlet only - No spike	250	2	274	UN	dynamic	3.0 lpm	0909c
11:00			Process went to lower production							
11:08	INUN207		Inlet	250	2	274	UN	dynamic	3.0 lpm	0909c
11:17	INUN208		Inlet	250	2	274	UN	dynamic	3.0 lpm	0909c
11:24			Refilled N2 detector							
11:28	OUTUN210		Outlet							
11:34	OUTUN211		Outlet							
11:44	NIT0909a		N2 only - direct to cell	250	2	274		dynamic	3.0 lpm	0909c
11:50	BKG0909d		Background - N2	500	2	275		dynamic	5.0 lpm	
			Start continuous software							
11:57	19090001		Inlet	250	2	275	UN	dynamic	5.0 lpm	0909d
	19090008		Stop							
	19090009		Evacuate cell							
	19090010		Evacuate cell							
	19090011		Bad							
12:24	19090012		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909d
12:46	19090020		Stop							
12:50	19090023		Inlet	250	2	275	UN	dynamic	5.0 lpm	0909d
13:20	19090036		Stop							
	19090037-38		Evacuate cell							
13:24	19090039		Outlet	250	2	275	UN	dynamic	5.0 lpm	0909d
13:45	19090048		Stop							
13:50	19090051		Inlet	250	2	275	UN	dynamic	5.0 lpm	0909d
13:56	19090053		Stop							
14:05	OUTSP112		Outlet - spike toluene, 60 ppm @ 1.0 lpm and	250	2	274	SP	dynamic	2.0 lpm	0909d
			SF6, 4ppm w/formaldehyde perm tube @ 100C							
			carried at 1.0 lpm, cell flow =2.0 lpm, vent flow = 2.0 lpm							
14:11			Outlet pulled from stack - passed leak check							
	INSP209		Inlet spike (same as above)	250	2	274	SP	dynamic	2.0 lpm	0909d
14:29	SFT0909b		Spike - direct to cell (same as above)	250	2	274	SP	dynamic	2.0 lpm	0909d
14:36	TOL0909a		Toluene, 60 ppm, direct	250	2	274	SP	dynamic		0909d
14:49	FRM0909a		Formaldehyde @ 1.0 lpm and 100 C	250	2	274	SP	dynamic	1.0 lpm	0909d
14:57	CTS0909b		Ethylene, 20 ppm	250	2	275		static		0909d
15:05	OUTAIR02		Outlet - air through	250	2	275		dynamic	5.0 lpm	0909d
15:13	INLAIR02		Inlet air sample line							
15:25	BKG0909e		N2 only	500	2	274			5.0 lpm	

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 745 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/10/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
7:20			Leak check inlet and outlet							
7:30	BKG0910a		N2 only background	500	2	274			5.0 lpm	
7:38	CTS0910a		Ethylene 20 ppm	250	2	274			5.0 lpm	0910a
7:49	INLSP301		Spike - Inlet	250	2	274	SP	dynamic	2.5 lpm	0910a
			SF6 4ppm @ 1.0 lpm w/form @ 100C							
			and toluene 60 ppm @ 1.0 lpm							
			Cell = 2.5 lpm, vent = 2.0 lpm							
8:00	OUTSP301		Spike - outlet (same as above)	250	2	274	SP	dynamic	2.5 lpm	0910a
8:04			Cell leak check under vacuum 1mmHg in 99sec							
8:10	SFT0910a		Direct to cell spike (same as above)	250	2	274	SP	dynamic	2.0 lpm	0910a
8:10			Manual sampling started							
8:15	OUTUN302		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:22	OUTUN303		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:27	OUTUN304		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:34	INLUN302		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:41	INLUN303		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:47	INLUN304		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910a
8:55	N20910a		N2 only	250						
9:00	BKG0910b		Background	500	2	275		dynamic	5.0 lpm	
9:07	19100001		Outlet - continuous software	250	2	275	UN	dynamic	5.0 lpm	0910b
9:31	19100011		Stop							
	1910012-14		Evacuate cell							
9:40	1910015		Inlet	250	2	275	UN	dynamic	5.0 lpm	0910b
9:59	1910024		Stop							
10:05	1910027		Outlet	250	2	275	UN	dynamic	5.0 lpm	0910b
10:25	1910036		Stop							
10:29	1910039		Inlet	250	2	275	UN	dynamic	5.0 lpm	0910b
10:58	1910051		Stop							
11:05	BKG0910c		N2 only - background	500	2	275		dynamic	5.0 lpm	0910c
11:17	OUTUN305		Outlet	250	2	275	UN	dynamic	5.0 lpm	0910c
11:23	OUTUN306		Outlet	250	2	275	UN	dynamic	5.0 lpm	0910c
11:32	OUTUN307		Outlet	250	2	275	UN	dynamic	5.0 lpm	0910c
11:37	OUTUN308		Outlet	250	2	275	UN	dynamic	5.0 lpm	0910c

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 4701-08-08 **BAROMETRIC:** 745 mm Hg

PLANT: Waupaca Foundary Inc. **DATE:** 9/10/97 **OPERATOR:** LMH

SAMPLE TIME	FILE NAME	PATH		NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
11:45	INUN305		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
11:52	INUN306		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
11:55	INUN307		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
11:57	INUN308		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:00	INUN309		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:02	INUN310		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:07	INUN311		Inlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:15	OUTUN309		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:18	OUTUN310		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:20	OUTUN311		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:25	OUTUN312		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:28	OUTUN313		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:30	OUTUN314		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:33	OUTUN315		Outlet	250	2	274	UN	dynamic	5.0 lpm	0910c
12:45	OUTSP316		Outlet spike	250	2	274	SP	dynamic	3.0 lpm	0910c
			SF6 4ppm @ 2.0 lpm w/formaldehyde @ 110C							
			and toluene 60 ppm @ 1.0 lpm							
			cell = 3.0 lpm, vent = 2.0 lpm							
12:53	INSP312		Inlet - spike (same as above)	250	2	274	SP	dynamic	3.0 lpm	0910c
13:04	SFT0910b		Spike direct to cell	250	2	274	SP	dynamic	3.0 lpm	0910c
13:11	CTS0910b		Ethylene 20 ppm	250	2	274		dynamic	5.0 lpm	0910c
13:18	N20910b		N2 only in cell	250	2	274		dynamic	5.0 lpm	0910c
13:26	BKG0910d		Background - N2	500						

Cooling + Shakeout

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** ~~XX~~ 758

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/4/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (°C)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
15:11	BKg090416		Nitrogen in Cell	500	2	130 ~~250~~				
	CT090416		20 ppm Ethylene	250	2	130				BKg090416

JMH

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FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 754.8 ~ 755

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/5/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (°C)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
9:30	BK60905a	20m	Nitrogen Flowing	500	2	130	—	—	5 lpm	
	BK64101		N_2 only ~~as~~ Nitrogen Absorbance -	250	2	"	--	—	"	BK60905a
9:45	CTS0905a		20ppm ethylene	250	"	"			"	"
9:53	CTS0905b		20ppm ethylene							
9:55	Leak Check - Cell at Vacuum - <									
10:03	SF60905a		4ppm - SF_6 - Direct to Cell	250	"	"			"	"
	SF60905b		" - Direct to Cell	"	"	"			"	"
10:11	TOL0905a		Toluene 60ppm - Direct to Cell	"	"	"			"	"
— Process Down —			~10:15							
	~~SF~~									
	SFTOL91		SF_6 Through Kintek at 1 lpm and mixed w/ Toluene at 1 lpm Formaldehyde only been in Oven for 10min @ 100°C } Bad Leak in Kintek							
13:54	BK60905b		N_2	500	2	130	—	—	5 lpm	
11:20	SF60905c	**	SF_6 @ 4.97 lpm to Spike Line	250	2	130	Spike	—	5 lpm	
			Shakeout - Sample Rate 6 ~~5~~ 4.5 lpm				Spike			BK60905b
	SF60905d	**	SF_6 @ 4.97 lpm - Sample Rate 4.0 lpm	250	2	130	Spike	-	5 lpm	"
			Spiked	** Kintek was venting - Void Samples						
	SF60905e		SF_6 @ 5.00 lpm - Sample Rate 4.0 lpm	250	2	130	Spike	—	5 lpm	"
* Process Restarted			13:15 —							
- Refilled Dryer		13:30								

MIDWEST RESEARCH INSTITUTE My Documents/FTIRFORM/Fieldata3.XLS 08-27-97

* Computer clock is one hour ahead of recorded times

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 755

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/5/97 **OPERATOR:** SMK

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
13:53	FORMAL01	20m	Formaldehyde in N_2 - Direct to Cell	250	2	130°C			1 lpm	BKG0905b
			1.0 lpm @ 100°C - Formation Tube = 914,000 nanol/min							
			Serial # 2214		≈ 90 ppm					
~14:10	SFTOC02									
		-	SF_6 (4ppm) @ 1 lpm into Kintek	250	2	130°C	SPIKE		2 lpm	BKG0905b
			Toluene 60ppm 1 lpm into MFC							
			Formaldehyde @ 100°C and 1 lpm							
14:20	N2ONLY01		N_2 only	"	"	"			5 lpm	"
14:24	BKG0905c		Background - N_2 only	500	2	130°C			5 lpm	
15:00 - Probe inserted in Cooling Stack w/ SPIKE										
15:23	COOSP101		Cooling Stack -	250	2	130°C	SPIKED		3.5 lpm	BKG0905c
			Spiking w/ SF_6 (4ppm) and form at 1.0 lpm							
			and 100°C and Tol at 1.0 lpm (60ppm)							
			Coil flow = 3.5 lpm Vent flow = 2 lpm							
15:33	COOSP102		Cooling w/ spike	250	2	130°C	SPIKED			"
15:37	- SHAKE OUT		Probe in Stack w/ SPIKE							
15:39	SHKSP101		Shakeout w/ spike	250	2	130°C	Spiked			"
			Same spike							
16:02	SHKSP102		Shakeout w/ spike	"	"	"	"			"

Coil Press 757

MIDWEST RESEARCH INSTITUTE My Documents/FTIRFORM/Fieldata3.XLS 08-27-97

* Computer clock is 1 hr ahead of data sheets

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 755

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/5/97 **OPERATOR:** JMH

Cell Press ~757

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
		C:\wasp\COOL\								
16:11	COOUN103	20m	Cooling Line only	250	2	130°C	UN	cooling line	5 lpm	0905c
16:15	COOUN104	"	"	"	"	"	"	"	"	"
16:21	COOUN105	"	"	"	"	"	"	"	"	"
16:27	COOUN106	"	"	"	"	"	"	"	"	"
		C:\wasp\shake\								
16:41	SHKUN103	20m	Shakeout Line	250	2	130	UN	shakeout	5 lpm	0905c
16:48	SHKUN104	"	"	"	"	"	"	"	"	
16:53	SHKUN105	"	"	"	"	"	"	"	"	"
16:58	SHKUN106	"	"	"	"	"	"	"	"	"
→ Start Continuous Process Software										
	C:\wasp\0905\									
17:05	~~[illegible]~~		Cooling							
	1905001									
	1905006	- -	Stop - last good file							
	1905007	Change	to Shake Out							
	1905009		Evacuated Cell							
~~[illegible]~~ 17:21	1905010		Shakeout	"	"	"	"	"	"	"
17:38	1905015		Stop							
"	1905016 and 1905017		Switch Lines - Evacuate Cell	"	"	"	"	"	"	"

* all samples dynamic

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 755

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 1/5/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
	c:\ftir\0905	-	CONTINUOUS SOFTWARE -							
17:41	19050018		Cooling Line	250	2	274	UN		5 lpm	09050
17:58	19050025		Stop							
	19050026 -	19050027	- Change Line - Evacuate Cell	"	"	"	"		"	"
18:03	19050028	Bod -	~~Shakeout~~ 3H Cooling	"	"	"	"		"	"
	Recevacuate cell									
18:07	19050030		Shakeout	"	"	"	"		"	"
	19050036		Stop							
	19050037 -	38	change Line Evacuate Cell							
18:29	19050039		Cooling Line	"	"	"	"		"	"
18:35	1905042		Stop							
	~~Guest107~~									
18:50	SHKSP107		shake out w/ SPIKE	250	"	274	SPIKE		3.0 lpm	"
			SPIKE IS SF_6 (1ppm) @ 1.0 lpm							
			formaldehyde @ 100°C @ 1.0 lpm							
			Toluene (6ppm) @ 1.0 lpm							
			Sample Rate 3.0 lpm							
			Vent ~ 1.0 lpm							

Cell Pressure ~757

* Gas clock is +1 hr compared to recorded data sh

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FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:**

PLANT: Waupaca Foundry, Inc. Tell City, IN **DATE:** 7/5/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
18:11	CC05P107		Cooling Line w/ Spike	250	2	274	Spiked		3.0 lpm	0/05c
			Spike = 1.0 lpm SF_6 in formaldehyde							
			@ 100°C w/ 1.0 lpm Toluene							
			Toluene is 6 ppm							
			and SF_6 is 4 ppm							
19:23	SFTOL03		Direct to Cell	"	"	"	"		2.0 lpm	"
			Spike Mix 1.0 lpm SF_6 64 ppm							
			and 1.0 lpm Toluene		Formaldehyde @ ~ 94,000 nanoL/min					
			and Formaldehyde @ 100°C							
18:31	N2ONLY02		N_2 only Direct to cell	"	"	"	"		5.0 lpm	"
19:38	CTS09c05c		Ethylene 20 ppm	"	"	"	"		5.0 lpm	"
19:45	BKg09c05d		N_2 only - Background	500	2	274	un		5.0 lpm	

Page 1.

Run #1 - Cupola

* Computer Clock updated to Actual Time

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 747mm Hg = 29.44"

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/8/97 **OPERATOR:** JMK

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
		Detectors filled 9:45								
10:24	Bkg0708a	20m	Background - N_2 only	500	2	274	—	dynamic	5.00lpm	
10:36	n2only03		N_2 only	250	2	274	—	"	"	Bkg0708a
10:45	cts0708a		20ppm Ethylene	"	"	"		"	"	"
10:50	cts0708b		"	"	"	"		"	"	"
10:57	Changed	filter Line #2 - Inlet - flow = 12lpm								
11:07	(JK) sf6014		Direct to Cell - SPIKE	250	2	274	—	"	2.0lpm	"
			SF_6 - 4ppm - @ 1.0lpm							
			w/ formaldehyde @100°C			Cell Press = 750mmHg				
**	— formaldehyde absorbance was less than 9/5/97		and Tol - 60ppm - @ 1.0lpm							
			Tolvent = 60.6ppm							
			from Scott Gas Cylinder # ALM052730							
			MRI PO # 029872							
	SFTOL05		"	"	"	"		"	"	"
12:02	inlsp01	20m	SPIKE to inlet	"	"	"		"	3.0lpm	"
			cell flow = 3.0 lpm							
			vent flow = 2.0 lpm	formaldehyde OK				"	"	"
12:09	inlsp02		"	"	"	"				
12:18	inlsp03		N2ONLY - flood N_2 into line	"	"	"		"	"	"
			Spike = 10lpm of N_2 only							

Cell Press = 756

Line #1 = outlet line #2 = inlet

Page 7

RUN #1

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 717mmHg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/8/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
			Inlet and Outlet Leak checks	Good -						
	~~OUTSP101~~		~~SPIKE w/ Toluene 60ppm @ 1lpm~~							
			~~N2 only~~ JH							
12:27	OUTSP101		SPIKE w/ Toluene 60ppm @ 1lpm	250	2	275	SP	dynamic	3.0lpm	Bkg 090896
			formaldyhyde @ 100°C and SF6 @ 1lpm							
			cell flow = 3.0lpm							
			Vent flow = 2.0lpm - OUTLET	-						
12:40	OUTSP102		N2 ONLY - 3610 Sample Line	"	"	"	"	"	"	"
			OUTLET Sample Line							
12:46	bkg091086		Background - N2 direct to cell	500	2	275	—	"	5.0lpm	
START 12:56	inlun104		Inlet Sample	250	"	274	UN	dynamic	5.0lpm	091086
13:02	inlun105		"	"	"	"	"	"	"	"
13:10	inlun106		"	"	"	"	"	"	"	"
13:17	inlun107		"	"	"	"	"	"	"	"
13:27	inlun108		"	"	"	"	"	"	"	"
13:25 -	shut	Probe Box	of Outlet down to check Pitots		- Changed	outlet	Probe			
13:42	inlun109		"	"	"	"	"	"	"	"
13:49	inlun110		"	"	"	"	"	"	"	"
14:04	OUTun103		Outlet	250	2	275	UN	dynamic	5.0lpm	"
14:09	OUTun104		"	"	"	"	"	"	"	"
14:15	OUTun105		"	"	"	"	"	"	"	"

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 747 mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/8/97 **OPERATOR:** JMN

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
14:28	inlun111		inlet	250	2	274	un	dynamic	5.0 lpm	09086
14:33	inlun112		inlet	"	"	"	"	"	"	"
14:38	inlun113		"	"	"	"	"	"	"	"
14:45	outun106		Outlet	"	"	"	"	"	"	"
14:51	outun107		"							→
14:58	outun108		"							→
15:03	outun109		"							→ Cal Press: 75.3
15:09	outun110		"							→
~~TT~~										
15:18	inlun114		inlet	"						→
15:25	inlun115		"							→
15:30	inlun116		"							→
OUTLET										
Start Continuous Sampling										
15:39	19080001		Outlet	250	2	274	un	dynamic	5.0 lpm	09086
16:04	19080012		— Stop							
inlet										
	Change to inlet execute 19080013 and 19080014									
16:09	19080015		INLET	250	"	"	"	"	"	"
16:15	19080017		STOP							

15:23 Refilled Dewar

19080018 and 19080019 — Evacuate Cell

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 747 mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/8/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
	19080020		OUTLET - Continuous Software	250	2	273	un	dynamic	5.0 lpm	09086
STOP 16:28	19080023		STOP							
16:39	Ukg0908c		N_2 only - Background	500	2	274	un	dynamic	5.0 lpm	
16:55	OUTSP111		SPIKE - Toluene 6ppm @ 2.0 lpm "	"	"	"	Spike	"	3.0 lpm	09086
			- SF_6, 4ppm, @ 1.0 lpm and							
			formaldehyde @ 100°C → Tol was higher in this spike							
17:05	INLSP117		SPIKE - Same as above							
17:11	Ct50908c		Ethylene 20ppm, direct to cell	250	2	274	-	"	5.0 lpm	"
17:21	SF10N96		SPIKE DIRECT TO CELL	250	2	274	-	"	3.0 lpm	"
			Same as spikes above							
			Tol, 6ppm @ 2.0 lpm							
			SF_6 4ppm @ 1.0 lpm							
			and formaldehyde @ 100°C							
17:13	Inlet and Outlet Pass Leak Check									
17:10	Probes pulled out of stack									
17:28	nOcilly04		N_2 Only	"					5.00 lpm	09086
17:33	Outair01		AIR only	250	2	274	-	dyn	5.00 lpm	"
17:39	Inair01		AIR only	"	"		"	"	"	"

Page H

Run #2
(Waupaca)

* Some files not corrected for Date and Time

FTIR FIELD DATA FORM

(FTIR Sampling Data)

Taylor Barometer
MRI # 4-2101

PROJECT NO. 3804-25

BAROMETRIC: 715 mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/9/97

OPERATOR: JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
7:10	Passed	inlet and	outlet leak check							
7:15	filled	detector								
7:22	Evac09		Evacuated Cell - 0.9mmHg	250						0908c
7:27	BKG0909a	20meters	Background	500	2	275	—	dynamic	5.0	
7:37	cts0909a		Ethylene 20ppm	250	2	274	—	"	"	0909a
7:47	insp201	**	INLET - SPIKE	250	2	274	SP	"	3.0 lpm	0909a
			SF_6 - 4ppm @ 2.0 lpm							
			with Formaldehyde @ 100°C							
		JH	~~and Toluene, 66ppm, @ 10 lpm~~	* NOTE Different MIX!!						
			Cell flow = 3.0 lpm	Toluene was not in!						
			Vent flow = 2.0 lpm							
8:00	outsp201		Outlet Spike	"	"	"	"	"	"	"
			Same as above (No Toluene)							
Cell	Leak Check @ Vacuum		0.6mmHg in 60 sec							
8:11	SFT0909a		SPIKE DIRECT TO CELL	"	"	"	"	"	"	"
			Same mix as above (No Toluene)							
8:24	outsp202		N_2 only in line, OUTLET	"	"	"	"	"	"	"

Run 61 [illegible] SPIKE to INLET [illegible]

FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 745mmHg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 09/09/97 **OPERATOR:** [illegible]

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
8:57	BKg0909b		N_2 only, Background	500	2	274	—	dynamic	5.0 lpm	
	~~BKg0909a~~		"	"	"	"		"	"	
9:06	BKg0909c		~~OUTLET~~ N_2 Background							
9:25	OUTUN203		OUTLET	250	2	275	UW	"	5.0 lpm	BKg0909c
	~~FTS STARTED~~		~~SAMPLING @ 9:30am~~							
9:31	OUTUN204		"	"	"	"	"	"	"	"
9:36	OUTUN205		"	"	"	"	"	"	"	"
9:44	OUTUN206		"	"	"	"	"	"	"	"
		Purge & Evacuate								
9:54	INLUN202		INLET	"	"	"	"	"	"	"
10:00	INLUN203		"	"	"	"	"	"	"	"
10:08	INLUN204		"	"	"	"	"	"	"	"
10:16	OUTUN207		OUTLET	"	"	"	"	"	"	"
10:30	OUTSP208		OUTLET SPIKE	"	"	"	"	"	3.0 lpm	"
			Toluene, 60ppm, @ 1.0 lpm							
			SF_6, 4ppm @ 1.0 lpm w/ Form @ 100°C							
			Cell flow = 3.0 lpm							
			Vent flow = 3.0 lpm							
10:39	OUTUN209		OUTLET ONLY	"	"	"	"	"	"	"
10:30	Manual Sampling Started									

Run 2

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/9/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
10:50	INLSP205		INLET SPIKE	250	2	275	SP	Dynamic	3.0lpm	0909c
			AT INLET PROBE BOX							
			Tol = 1.0lpm, SF6 = 1.0lpm, Form = 100°C							
			Cell flow = 3.0lpm, Vent = 2.0lpm							
11:00	INSP206		INLET ONLY - NO SPIKE	"	"	"	"	"	"	"
~11:00 -	PROCESS	WENT	~~DOWN~~ TO LOWER PRODUCTION							
11:08	INUN207		INLET	"	"	"	"	"	"	"
11:17	INUN208		"	"	"	"	"	"	"	"
11:24	Refilled	N_2	~~Purge~~ - Detector							
11:28	OUTUN210		OUTLET							
11:34	OUTUN211		OUTLET							
11:44	NIT0909a		N_2 only - Direct to Cell	"	"	"	"	"	"	"
11:50	BKG0909d		Background - N_2	500	2	275	—	Dynamic	5.0lpm	—
	START continuous		Software							
11:57	19090001		INLET	250	"	"	UN	"	"	0909d
	19090008		Stop							
	19090009 and 0010		evacuate cell + recall - Bad -							
12:24	19090012		OUTLET	"	"	"	"	"	"	"
12:46	19090020		Stop							

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/9/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
										BKg 0909d
12:50	19090033		INLET	250	2	275	UN	dynamic	5.0 lpm	
13:20	19090036		STOP							
	19090037 and	19090038	Evacuate Cell							
13:24	19090039		OUTLET	"	"	"	"	"	"	"
13:45	19090048		STOP							
13:50	19090051		INLET	"	"	"	"	"	"	"
13:56	19090053		STOP							
14:05	OUTSP112		OUTLET - SPIKE	251	2	274	SP	dynamic	2.0 lpm	0909d
			Toluene, 60ppm @ 1 lpm and							
			SF_6, 4ppm, w/ Formaldehyde Prim Tube @ 100°C							
			carried @ 1.0 lpm							
			Cell flow = 2.0 lpm, Vent flow = 2.0 lpm							
14:11	outlet pulled from Stack - Passed Leak Check									
	INSP209		INLET - SPIKE	"	"	"	"	"	2.0 lpm	0909d
			Same as Above							
14:29	SFT0909b		SPIKE - Direct to Cell	"	"	"	"	"	2.0 lpm	"
			Same as Above							
14:36	TOL0909a		Toluene 60ppm, Direct	"	"	"	"	"		"
14:49	FAM0901a		Formaldehyde @ 1.0 lpm and 100°C ≈ 94,000 ppm							

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25

BAROMETRIC: ~~(HI)~~ 745 mmHg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/9/97

OPERATOR: JMB

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
14:57	CTS0909b		Ethylene, 20 ppm	250	2	275	—	~~dynamic~~ static	~~5 lpm~~	0909d
	~~AUTOM~~									
15:05	OUTAIR02		OUTLET — AIR THROUGH	"	"	"	"	dynamic	5.0 lpm	"
15:13	INLAIR02		INLET-AIR Sample Line							
15:25	bkg0909e		N_2 only	500	2	274	"		5.0 lpm	—

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Run 43 - Cupola

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745 mm Hg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/16/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
7:20	Leak Check INLET and Outlet									
7:30	BKg0916a		N_2 only Background	500	2	274	—	—	5.0 lpm	—
7:38	ethy0916a		ethylene 20ppm	250	2	274	-	-	5.0 lpm	BKg0916a
7:49	INLSP301		SPIKE - INLET	"	"	"	SP	dynamic	2.5 lpm	"
			SF_6 4ppm @ 1.0 lpm w/ Freon @ 100°C							
			and Tol 6ppm @ 1.0 Lpm							
			Cell = 2.5 lpm, Vent = 2.0 lpm							
~~7:49~~ 8:00	OUTSP301		SPIKE - OUTLET	"	"	"	"	"	"	"
			Same as above							
8:04	Cell Leak Check		under Vacuum — 1mm Hg in 99sec							
8:10	SFT0916a		Direct to Cell - SPIKE	"	"	"	"	"	2.0 lpm	"
			Same as above							
8:10	Manual	Sampling	Started							
8:15	OUTUN302		OUTLET	250	2	274	UN	dynamic	5.0 lpm	BKg0916a
8:22	OUTUN303		"	"	"	"	"	"	"	"
8:27	OUTUN304		"	"	"	"	"	"	"	"
8:34	INLUN302		INLET	250	2	274	UN	dynamic	5.0 lpm	"
8:41	INLUN303		"	"	"	"	"	"	"	"
8:47	INLUN304									

Cell Press ~75 mm Hg

FTIR Start

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745 mmHg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/10/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
8:55	N_2 0910c		N_2 only	250						
9:03	bkg0910b		Background	500	2	275	—	dynamic	5.0lpm	—
9:07	1910 0001		Outlet - Continuous Soft	Where 250	2	"	UN	"	5.0lpm	0910b
9:31	1910 0011		Stop							
	Swcwatr	Cell	1910012 - 14							
9:40	1910015		Inlet	250	2	275	UN	"	5.0lpm	0910b
9:59	1910024		Stop							
10:05	1910027		Outlet	"	"	"	"	"	"	"
10:25	1910036		Stop							
10:29	1910039		Inlet	"	"	"	"	"	"	"
10:58	1910051		Stop							
11:05	~~bkg0910c~~ ~~N_2 0910c~~		N_2 only - Background	500	"	"	"	"	"	"
11:17	OUTUN305		Outlet	250	"	"	"	"	"	0910c
11:23	OUTUN306		"	"	"	"	"	"	"	"
11:32	OUTUN307		"	"	"	"	"	"	"	"
11:37	OUTUN308		"	"	"	"	"	"	"	"

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FTIR FIELD DATA FORM

PROJECT NO. 3804-25 *(FTIR Sampling Data)* **BAROMETRIC:** 745mmHg

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/10/97 **OPERATOR:** JMN

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
11:45	INUN305		INLET	250	2	274	UN	dynamic	5.0lpm	0910c
11:52	INUN306		"							
11:55	INUN307		"							
11:57	INUN308		"							
12:00	INUN309		"							
12:02	INUN310		"							
12:07	INUN311		"							
12:15	OUTUN309		Outlet	"	"	"	"	"	"	"
12:18	OUTUN310		"							
12:16	OUTUN311		"							
12:25	OUTUN312		"							
12:28	OUTUN313		"							
12:30	OUTUN314		"							
12:33	OUTUN315		"							
12:45	OUTSP316		OUTLET - SPIKE,	250	2	274	SP	dynamic	3.0lpm	0910c
			~~SF_6 4ppm~~, @ 2.0lpm w/ formaldehyde @ ~~1.0~~			110°C				
			and Tol, 600 ppm, @ 1.0lpm							
			Cell - 3.0lpm, Vent - 2.0lpm							
12:53	INSP312		Inlet - SPIKE	"	"	"	"	"	"	"
			Same as above							
	~~[illegible]~~		~~N_2 only~~							
13:04	SPT0910b		SPIKE DIRECT TO CELL	"	"	"	"	"	"	"

FTIR FIELD DATA FORM

(FTIR Sampling Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745

PLANT: Waupaca Foundry, Inc. **Tell City, IN** **DATE:** 9/10/97 **OPERATOR:** JMH

SAMPLE TIME	FILE NAME	PATH	LOCATION / NOTES	NUMBER SCANS	RES (cm-1)	CELL TEMP (F)	SPIKED/ UNSPIKED	SAMPLE COND.	SAMPLE FLOW	BKG
13:11	c:ts0910b		Ethylene 20ppm	250	2	274	—	dynamic	5 slpm	0910c
13:18	N20910b		N2 only in cell	"	"	"	"	"	"	"
13:26	bkg0910c		Background - N2	500						

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B-3. FTIR FLOW AND TEMPERATURE READINGS

Cooling & Shakeout

FTIR FIELD DATA FORM

(Sampling Location Data)

PROJECT NO. 3011-25 **BAROMETRIC:** 755

PLANT: Waupaca Fall City, IN **DATE:** 9/5/97 **OPERATOR:** JMH

~~INLET~~ COOL

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
15:38	1.1	130	302	303	
16:17	1.1	136	305	302	
17:44	1.1	137	306	302	
18:02	1.1	138	305	302	
18:23	1.1	134	305	300	

~~OUTLET~~ SHAKEOUT

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
15:38	2.1	131	303	299	
16:18	2.1	126	303	300	
17:44	2.2	129	299	299	
18:02	2.1	131	301	301	
18:23	2.1	132	299	300	

Run #1

FTIR FIELD DATA FORM

(Sampling Location Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 717 mm Hg

PLANT: ~~[illegible]~~ Wampum **DATE:** 9/8/97 **OPERATOR:** YMH

INLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
12:53	1.6	321	305	302	
13:55	0.95	305	304	302	
14:10	0.84	319	302	302	
14:38	0.80	315	303	302	
15:04	1.1	317	303	302	
15:40	0.9	313	305	302	
16:11	0.6	317	305	302	
16:54	0.79	304	303	302	

Changed out Probe

OUTLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
12:53	0.07	265	297	300	
13:55	0.09	264	234	187	
14:10	0.08	264	279	238	
14:38	0.08	261	301	297	
15:04	0.09	262	301	302	
15:40	0.08	259	298	300	
16:11	0.08	261	298	300	
16:54	0.07	258	301	297	

Run #2

FTIR FIELD DATA FORM

(Sampling Location Data)

PROJECT NO. 3804-25

BAROMETRIC: 745 mm Hg

PLANT: Wrupacia Tell City

DATE: 09/09/97

OPERATOR: YMK

INLET

SAMPLE TIME **	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
9:06	0.95	308	304	302	
10:03	0.75	307	304	302	
11:03	0.20	297	305	304	
11:35	0.32	298	304	302	
12:18	0.65	297	301	302	
13:06	2.0	294	304	302	
13:44	2.0	296	302	302	
14:14	2.5	295	302	302	

OUTLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
9:09	0.08	243	301	300	
10:03	0.08	245	299	300	
11:03	0.06	247	299	300	
11:35	0.06	239	301	300	
12:18	0.05	236	297	300	
13:06	0.06	232	300	300	
13:44	0.05	2.33	302	300	

out of stack @ 14:11

** ... changes quickly · Range of 0.01 ...

Run #3

FTIR FIELD DATA FORM

(Sampling Location Data)

PROJECT NO. 3804-25 **BAROMETRIC:** 745 mm Hg

PLANT: Wanpaco Tell City, IN **DATE:** 07/10/97 **OPERATOR:** JMH

INLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
7:44	5.5	307	305	302	
8:16	2.6	295	306	302	
10:21	6.0	305	303	302	
11:43	3.0	323	303	302	
12:27	1.8	315	305	303	

JMH

OUTLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
7:44	0.1	245	299	300	
8:16	0.06	244	302	300	
10:21	0.14	256	299	300	
11:43	0.09	261	302	300	
12:27	0.11	263	299	300	

JMH

121

Run #3

FTIR FIELD DATA FORM

(Sampling Location Data)

PROJECT NO. 3804-25

BAROMETRIC: 745 mm Hg

PLANT: Wanpaco Tell City, IN

DATE: 07/10/97

OPERATOR: JMH

INLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
7:44	5.5	307	305	302	
8:16	2.6	295	306	302	
10:21	6.0	305	303	302	
11:43	3.0	323	303	302	
12:27	1.8	315	305	303	

JMH

OUTLET

SAMPLE TIME	DELTA P IN. H2O	STACK TEMP.	PROBE TEMP.	FILTER TEMP.	
7:44	0.1	245	299	300	
8:16	0.06	244	302	300	
10:21	0.14	256	299	300	
11:43	0.09	261	302	300	
12:27	0.11	263	299	300	

JMH

122

B-4 HYDROCARBON REFERENCE SPECTRA

Reference Spectra of Hydrocarbon Compounds

The purpose of measuring reference spectra of some hydrocarbon compounds was to aid the analyses of FTIR sample spectra from iron and steel foundries and from integrated iron and steel plants. Four facilities were tested at these sources. At each facility hydrocarbon compounds were detected in the emissions. Because the EPA library of FTIR reference spectra contains only spectra of hazardous air pollutant (HAP) compounds, only quantitative reference spectra of hexane and isooctane were available to analyze the sample hydrocarbon emissions. As a result the hydrocarbon emissions were represented primarily by "hexane" in the draft report results. Many hydrocarbon compounds have infrared spectra which are similar to that of hexane in the spectral region near 2900 cm^{-1}. MRI selected nine candidate hydrocarbon compounds and measured their reference spectra in the laboratory. In addition MRI measured new high-temperature reference spectra of hexane and isooctane. The new reference spectra of these 11 compounds were included in revised analyses of the sample spectra. The FTIR results presented in the revised test reports show the measured concentrations of the detected hydrocarbons and also show revised concentrations of hexane and toluene. The hexane concentrations, in particular, are generally lower because the infrared absorbance from the hydrocarbon emissions is partly measured by the new reference spectra. As an example, figure B-1 illustrates the similarities among a sample spectrum and reference spectra of hexane and n-heptane.

MRI prepared a laboratory plan specifying the procedures for measuring the reference spectra. The EPA-approved laboratory plan is included in this appendix. The data sheets, check lists and other documentation are also included. During the measurements some minor changes were made to the laboratory plan procedures. These changes don't affect the data quality, but did allow the measurements to be completed in less time. This was necessary because the plan review process was more length than anticipated.

The following changes were to the procedures. The spectra were measured at 1.0 cm^{-1} resolution, which was the highest resolution of the sample spectra. It was unnecessary to use a heated line connection between the mass flow meter and the gas cell because the gas temperature in the cell was maintained without the heated line. Leak checks were conducted at positive pressure only because all of the laboratory measurements were conducted at ambient pressure. The reference spectra, CTS spectra, and background spectra will be provided on a disk with a separate reference spectrum report.

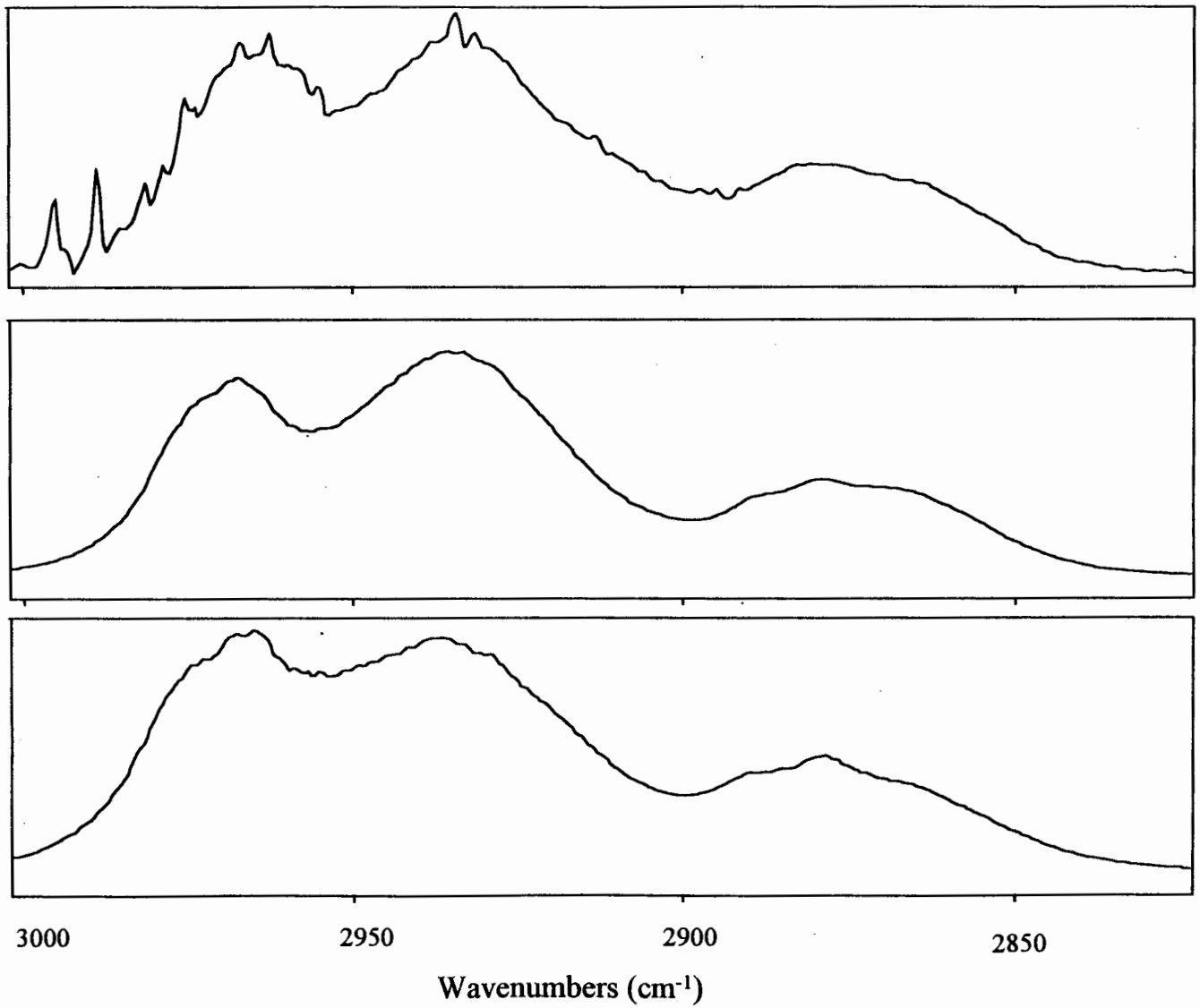

Figure B-1. Top trace, example sample spectrum; middle trace, n-heptane reference spectrum; bottom trace, n-hexane reference Spectrum.

LABORATORY PLAN FOR REFERENCE SPECTRUM MEASUREMENTS

DRAFT

Prepared for

Office of Air Quality Planning and Standards
Emissions, Monitoring and Analysis Division
Emission Measurement Center (MD-19)
Research Triangle Park, North Carolina 27711
Mr. Michael Ciolek
Work Assignment Manager

EPA Contract No. 68-D-98-027
Work Assignment 2-12 and 2-13
MRI Project No. 4951-12 and 4951-13

June 14, 1999

MIDWEST RESEARCH INSTITUTE 5520 Dillard Road, Suite 100, Cary, NC 27511-9232 • (919) 851-8181

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FIGURE AND TABLE LIST

Figure 1. Measurement system configuration 4

TABLE 1. ORGANIC COMPOUNDS SELECTED FOR THE LABORATORY STUDY 3

Laboratory Plan For Reference Spectrum Measurements
EPA Contract No. 68-D-98-027, Work Assignments 2-12 and 2-13
MRI Work Assignments 4951-12 and 4951-13

1.0 INTRODUCTION

In 1997 Midwest Research Institute (MRI) completed FTIR field tests at two iron and steel sintering facilities and at two iron and steel foundries. The tests were completed under EPA Contract No. 68-D2-0165, work assignments 4-20 and 4-25 for the sintering plants and foundries, respectively. The draft test reports were completed in 1998 under EPA Contract No. 68-W6-0048, work assignment 2-08, tasks 11 and 08 for the sintering plants and foundries, respectively.

Results from the data analyses indicated that the emissions from some locations included a mixture of hydrocarbon compounds, one of which was hexane. The EPA spectral library of FTIR reference spectra is comprised primarily of hazardous air pollutants (HAPs) identified in Title III of the 1990 Clean Air Act Amendments and, therefore, contains a limited number of aliphatic hydrocarbon compounds. MRI will measure reference spectra of some additional organic compounds that may have been part of the sample mixtures. The new reference spectra will be used in revised analyses of the sample spectra. The revised analyses will provide a better measure of the non-hexane sample components and, therefore, more accurate hexane measurements.

A Quality Assurance Project Plan (QAPP) was submitted for each source under EPA Contract No. 68-D2-0165, work assignments 4-20 and 4-25. When the QAPPs were prepared it was not anticipated that laboratory measurements would be required. This document describes the laboratory procedures and is an addition to the QAPPs.

This document outlines the technical approach and specifies the laboratory procedures that will be followed to measure the FTIR reference spectra. Electronic copies of the new reference spectra will be submitted to EPA with corresponding documentation. The laboratory procedures are consistent with EPA's Protocol for the Use of Extractive Fourier Transform Infrared (FTIR) Spectrometry for the Analyses of Gaseous Emissions From Stationary Sources, revised 1996.

1.1 Objective

The objective is to obtain accurate hexane measurements from FTIR spectra recorded at field tests at iron and steel sintering plants and at steel foundry plants. The approach is to measure reference spectra of some organic compounds that are not included in the EPA reference spectrum library and then use these new reference spectra in revised analyses of the field test spectra. The revised analyses will provide better discrimination of the hexane component from the absorbance bands of the organic mixture.

1.2 Background

Spectra of samples measured at the field test sites contained infrared absorbance features that may be due to a mixture of non-aromatic organic compounds. The samples were measured using quantitative reference spectra in the EPA library and the hexane reference spectra provided the best model for the observed absorbance features. The EPA library contains a limited number of reference spectra, primarily HAPs, listed in Title III of the 1990 Clean Air Act Amendments, which includes hexane. To obtain accurate measurements of target components it is helpful to use reference spectra of all compounds in the sample gas mixture. In this case it was decided to measure reference spectra of some additional organic compounds, which are similar in structure and have spectral features similar to hexane. The revised analyses will measure the sample absorbance in the 2900 cm^{-1} region using a combination of the hexane and new reference spectra. The revised analyses should provide more accurate hexane measurements, by measuring the non-hexane sample components more accurately.

2.0 TECHNICAL APPROACH

The analytical region used to measure hexane lies near 2900 cm^{-1}. Other aliphatic hydrocarbons with structures similar to hexane exhibit similar absorbance band shapes in this region. MRI viewed spectra of aliphatic organic compounds to identify some likely components of the sample spectra. Table 1 identifies the compounds that were selected for reference spectrum measurements. Cylinder standards of the selected compounds will be purchased from a commercial gas supplier. The standards will be about 50 ppm of the analyte in a balance of nitrogen. The cylinders will contain gravimetric standards (analytical accuracy of ±1 percent) in a balance of nitrogen.

2.1 Measurement System

A controlled, measured flow of the gas standard will be directed from the cylinder to the infrared gas cell. The gas cell is a CIC Photonics Pathfinder. This is a variable path White cell with an adjustable path length from 0.4 to 10 meters. The path lengths have been verified by measurements of ethylene spectra compared to ethylene spectra in the EPA FTIR spectral library. The inner cell surface is nickel coated alloy to minimize reactions of corrosive compounds with the cell surfaces. The cell windows are ZnSe. The cell is heat-wrapped and insulated. Temperature controllers and digital readout are used to control and monitor the cell temperature in two heating zones. The gas temperature inside the cell will be recorded using a T-type thermocouple temperature probe inserted through a 1/4 in. Swagelok fitting. The gas temperature will be maintained at about 120°C. Documentation of the temperature probe and thermometer calibration will be provided with the report.

TABLE 1. ORGANIC COMPOUNDS SELECTED FOR THE LABORATORY STUDY

Compound Name	Boiling Point (°C)
n–hexane[a]	69
n-heptane	98.4
Pentane	36.1
isooctane[a]	99.2
1-pentene	30
2-methyl,1-pentene	60.7
2-methyl,2-butene	38.6
2-methyl,2-pentene	67.3
3-methylpentane	63.3
Butane	-0.5

[a] Hexane and isooctane are HAPs. Their reference spectra will be re-measured because the reference spectra in the EPA library were measured at ambient temperature.

The instrument is an Analect Instruments (Orbital Sciences) RFX-65 optical bench equipped with a mercury-cadmium-telluride (MCT) detector. The RFX-65 instrument is capable of measuring spectra at 0.125 cm^{-1} resolution. The reference spectra will be measured at 0.25 cm^{-1} or 0.50 cm^{-1} resolution. Gas pressure in the sample cell will be measured using an Edwards barocell pressure sensor equipped with an Edwards model 1570 digital readout. A record of the pressure sensor calibration will be provided with the report.

A continuous flow of the gas standard will be maintained through the cell as the spectra are recorded. A mass flow meter will be used to monitor the gas flow (Sierra Instruments, Inc., model No. 822S-L-2-OK1-PV1-V1-A1, 0 to 5 liters per minute).

The instrument system will be configured to measure 0.25 cm^{-1} or 0.50 cm^{-1} resolution spectra. The measurement configuration is shown in Figure 1. Calibration transfer standards (CTS) will be measured each day before any reference spectra are measured and after reference spectra measurements are completed for the day.

2.2 Procedure

Information will be recorded in a laboratory notebook. Additionally, the instrument operator will use check lists to document that all procedures are completed. There will be three checklists for: (1) daily startup prior to any reference measurements, (2) reference spectrum measurements, and (3) daily shut down after reference measurements are completed. Example checklists are at the end of this document.

The information recorded in the laboratory notebook includes; the cell temperature, ambient pressure, background, CTS and spectrum file names, sample temperatures and pressures for each measurement, cell path length settings, number of background and sample scans, instrument

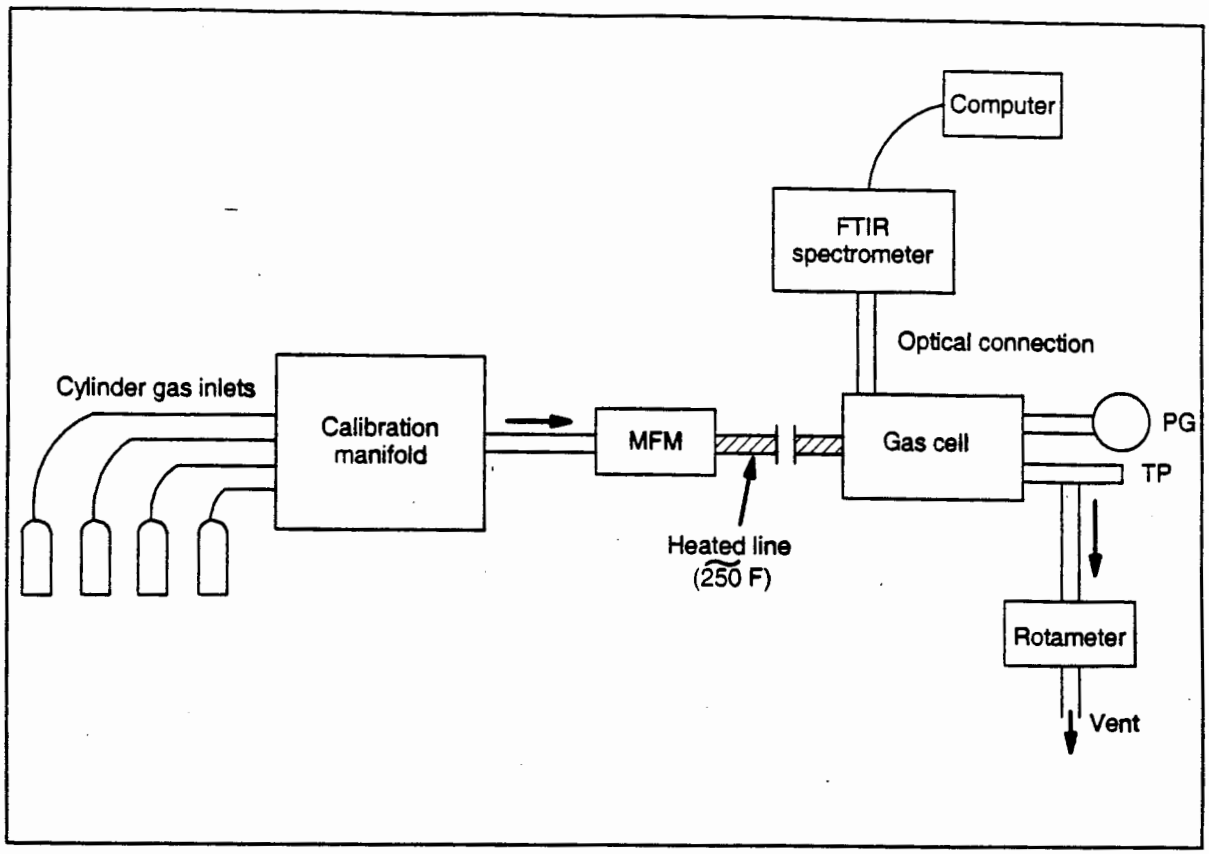

Figure 1. Measurement system configuration.
PG = pressure gauge; TP = temperature probe; MFM = mass flow meter.

resolution, gas standard concentration, sample cylinder identification, and sample flow rates for each measurement. Certificates of Analysis for all gas standards used in the project will be provided with the report.

The MCT detector will be cooled with liquid nitrogen and allowed to stabilize before measurements begin.

The cell will be filled with dry nitrogen and vented to ambient pressure. The pressure, in torr, will be recorded from the digital barocell readout. The cell will then be evacuated and leak checked under vacuum to verify that the vacuum pressure leak, or out-gassing, is no greater than 4 percent of the cell volume within a 1-minute period. The cell will then be filled with nitrogen and a background will be recorded as the cell is continuously purged with dry nitrogen. After the background spectrum is completed the cell will be evacuated and filled with the CTS gas. The CTS spectrum will be recorded as the cell is continuously purged with the CTS gas standard. The purge flow rates will be 0.5 to 1.0 LPM (liters per minute) as measured by the mass flow meter.

After the background and CTS measurements are completed the cell will be filled with a reference gas sample. The reference spectra will be recorded as the cell is continuously purged at 0.5 to 1.0 LPM with gas standard. The gas flow will be monitored with a mass flow meter before the gas enters a heated line, and with a rotameter after the gas exits the cell. The mass flow meter is calibrated for nitrogen in the range 0 to 5 LPM. The purpose of the heated line connection is to help maintain the gas temperature inside the cell. This may only require placing a heat wrap on the line where the gas enters the cell.

The gas temperature of each nitrogen background, CTS, and reference gas will be recorded as its spectrum is collected.

Several preliminary spectra will be recorded to verify that the in-cell gas concentration has stabilized. Stabilization usually occurs within 5 minutes after the gas is first introduced into the cell with the measurement system that will be used for this project. Duplicate (or more) reference spectra will be collected for each flowing sample. The second reference spectrum will be recorded at least 5 minutes after the first spectrum is completed while the continuous gas flow is maintained.

At least 100 scans will be co-added for all background, CTS , and reference interferograms.

A new background single beam spectrum will be recorded for each new compound or more frequently if the absorbance base line deviates by more than ±0.02 absorbance units from zero absorbance in the analytical region.

After reference spectrum measurements are completed each day, the background and CTS measurements will be repeated.

The CTS gas will be an ethylene gas standard, either 30 or 100ppm in nitrogen (±1 percent) or methane (about 50 ppm in nitrogen, ±1 percent). The methane CTS may be particularly suitable for the analytical region near 2900 cm^{-1}.

3.0 QUALITY ASSURANCE AND QUALITY CONTROL

The following procedures will be followed to assure data quality.

3.1 Spectra Archiving

Two copies of all recorded spectra will be stored, one copy on the computer hard drive and a second copy on an external storage medium. The raw interferograms will be stored in addition to the absorbance spectra. After the data are collected, the absorbance spectra will be converted to Grams (Galactic Industries) spectral format. The spectra will be reviewed by a second analyst and all of the spectra, including the Grams versions will be provided with a report and documentation of the reference spectra.

3.2 CTS Spectra

The CTS spectra will provide a record of the instrument stability over the entire project. The precision of the CTS absorbance response will be analyzed and reported. All of the CTS spectra will be archived with the background and reference spectra.

3.3 Sample Pressure

The barocell gauge calibration will be NIST traceable and will be documented in the reference spectrum report. The ambient pressure will be recorded daily and all of the samples will be maintained near ambient pressure within the IR gas cell.

3.4 Sample Temperature

The IR gas cell is equipped with a heating jacket and temperature controllers. The temperature controller readings will be recorded whenever spectra are recorded. Additionally, the temperature of each gas sample will be measured as its spectrum is collected using a calibrated temperature probe and digital thermometer. The calibration record will be provided with the reference spectrum report. The gas sample will be preheated before entering the cell by passing through a heated 20 ft. Teflon line. The Teflon line temperature will be maintained at about 120°C. The line temperature controllers will be adjusted to keep the gas sample temperature near 120°C.

3.5 Spectra

MRI will record parameters used to collect each interferogram and to generate each absorbance spectrum. These parameters include: spectral resolution, number of background and sample scans, cell path length, and apodization. The documentation will be sufficient to allow an independent analyst to reproduce the reference absorbance spectra from the raw interferograms.

3.6 Cell Path Length

The cell path length for various settings is provided by the manufacturer's documentation. The path length will be verified by comparing ethylene CTS spectra to ethylene CTS spectra in the EPA spectral library.

3.7 Reporting

A report will be prepared that describes the reference spectrum procedures. The report will include documentation of the laboratory activities, copies of data sheets and check lists, and an electronic copy of all spectra and interferograms.

3.8 Documentation

Laboratory analysts will use three check lists to document data recording activities. The check lists are appended to this plan. The checklists: (1) record start up activities such as instrument settings, background and CTS spectra, (2) record reference spectra activities, and (3) record daily shut down procedures, including post-reference spectra background and CTS measurements.

In addition to the check lists the operator will record notations in a laboratory notebook. Copies of the check lists and note book pages will be provided with the reference spectrum report.

A draft of the reference spectrum report will be provided with the revised test reports. The reference spectrum report will then be finalized and submitted separately.

Project No. _____

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: _____ OPERATOR: _____

			Initials
Check cell temperature			_____
Verify temperature using thermocouple probe and hand-held readout			_____
Purge cell with dry nitrogen and vent to ambient pressure			_____
Record ambient pressure in cell, (P_b)			_____
Vacuum Leak Check Procedure:			
Evacuate cell to baseline pressure.			_____
Isolate cell (close cell inlet and cell outlet)			_____
Record time and baseline pressure (P_{min})	_____ Time	_____ P_{min}	_____
Leave cell isolated for one minute			
Record time and cell pressure (P_{max})	_____ Time	_____ P_{max}	_____
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		_____ ΔP	_____
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		_____ $\% V_L$	_____
$\lvert \% V_L \rvert$ should be < 4			

Record Nitrogen Background

Purge cell with dry nitrogen	_____
Verify cell is as dry as previous background	_____
Record ambient pressure using cell Barocell gauge	_____
Record nitrogen flow rate (about sampling flow rate)	_____
Collect Background (**AQBK**) under continuous flow and ambient pressure	_____
Record information in data book.	_____
Copy Background to C-drive and backup using batch file.	_____

Record CTS Spectrum

Record Cell path length setting	
Evacuate Cell	_____
Fill Cell with CTS gas	_____
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	_____
Record cylinder ID Number	_____
Record CTS gas cylinder identity and concentration	_____
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	_____
Record Barytron pressure during collect	_____
Record information on "Background and Calibrations" data sheet.	_____
Verify that spectrum and interferogram were copied to directories.	_____
Record CTS Spectrum File Name	_____

Reviewed by: _____ Date: _____

Project No. _____

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: _____ OPERATOR: _____

	Initials
Reference Spectrum Sample	
Start Time	_____
Record Cell path length setting	_____
Record Background Spectrum File Name	_____
Record CTS Spectrum File Name	_____
Record Compound Name	_____
Record Cylinder Identification Number	_____
Record Cylinder Concentration	_____
Record Spectrum File Name	_____
Fill cell to ambient pressure with gas from cylinder standard	_____
Open cell outlet vent valve	_____
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	_____
Allow to equilibrate for 5 minutes	_____
Record sample pressure in cell	_____
Record sample flow rate through cell	_____
Start spectrum collect program	_____
Record information in data book	_____
Copy Spectrum and Interferogram to backup directories	_____
End Time	_____

Reviewed by: _____ Date: _____

Project No. _____

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: _____ OPERATOR: _____

	Initials
Purge sample from cell using ambient air or nitrogen	_____
Record Nitrogen Background	_____
Purge cell with dry nitrogen	_____
Verify cell is as dry as previous background	_____
Record ambient pressure using cell Barocell gauge	_____
Record nitrogen flow rate (about sampling flow rate)	_____
Collect Background (**AQBK**) under continuous flow and ambient pressure	_____
Record information in data book.	_____
Copy Background to C-drive and backup using batch file.	_____
Record CTS Spectrum	
Evacuate Cell	_____
Fill Cell with CTS gas	_____
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	_____
Record cylinder ID Number	_____
Record CTS gas cylinder identity and concentration	_____
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	_____
Record Barytron pressure during collect	_____
Record information on "Background and Calibrations" data sheet.	_____
Verify that spectrum and interferogram were copied to directories.	_____
Record CTS Spectrum File Name	_____
Close cylinders	_____
Evacuate or Purge CTS from cell using nitrogen	_____
Leave cell under low nitrogen purge or under vacuum	_____
Fill MCT detector dewar	_____

Reviewed by: _____ Date: _____

SHIPPING ORDER

MIDWEST RESEARCH INSTITUTE

425 Volker Boulevard, Kansas City, Missouri 64110

☐ TRANSFER ☐ RETURN FOR CREDIT

☐ EXCHANGE ☐ RETURN FOR REPAIR

144099

REFER TO THIS NO. IN ALL CORRESPONDENCE

TO Midwest Research Institute
5520 DILLARD ROAD
SUITE 100
Cary NC 27511-9232

DATE 5-10-99

VIA Best Way	☐ A.M. ☐ P.M. ☐ PREPAID ☐ COLLECT	INSURE: ☐ YES ☐ NO AMOUNT
REQUESTED BY Dave Upton	Charge No. 041 or Bill Recipient Acct. No.	REFERENCE

QUANTITY	DESCRIPTION OF MATERIAL	PRESENT LOCATION
1	Barocel pressure transducer	
1	Edwards Indicator	
1	HH21 Omega Indicator	
1	Type T thermocouple	

SIGNED Dave Upton

Victor

PACKING SLIP

MRI 11 (Rev. 8/92)

Attachment 1
Instrument Found Out of Tolerance

Instrument: 1570 indicator with W60041111 Capacitance Manometer

Manufacturer: Edwards

MRI Number: ~~N/A~~ FE 3U Y-6472 & Y-6473

Serial Number: 4237 & 80412159

Acceptance Criteria: MFG Accuracy

Date of calibration or test that revealed the out of tolerance condition: 5-6-99

Date of previous calibration: UNKNOWN

Responsible person: Tom Geyer (Must receive a copy of this report)

Tested/Calibrated by: Dave Upton Date: 5-6-99

Reviewed by: [signature] Date: 5/6/1999

Unit read 7.9 to 9.7 torr high. Adjusted to mfg tolerance.

I hereby certify that I have received a copy of this report and will notify the appropriate people and take the appropriate actions necessary to determine what data may have been corrupted and what corrective actions are indicated.

Signed: [signature] (Responsible person)

Date: 5/14/99

Attachment 1
Pressure Gauge Calibration Data Sheet

MRI No. ~~N/A~~ Y-6472 & Y-6473 Model No. / Type 1570 with W60041111 Serial No. 4237 S/N 80412159
Report No.

Noun Barocel pressure indicator Ambient Temperature 73°F Ambient Humidity 41%

Applied Pressure	Initial Check	Final Check	Tolerance ±	Pass	Fail
500 TORR	507.9 TORR	500.5 TORR	1.2 TORR	✓	
600 TORR	608.3 TORR	600.2 TORR	1.4 TORR	✓	
700 TORR	708.6 TORR	699.9 TORR	1.5 TORR	✓	
750 TORR	758.7 TORR	749.8 TORR	1.6 TORR	✓	
800 TORR	808.8 TORR	799.7 TORR	1.7 TORR	✓	
900 TORR	909.2 TORR	899.4 TORR	1.9 TORR	✓	
1000 TORR	1009.7 TORR	999.3 TORR	2.1 TORR	✓	

Cumulative uncertainties of the standards used to perform this calibration did not exceed the requirements of MRI-0701 and ISO 10012-1.

Standards Used: MRI No.	Date Calibrated	Date Due Calibration
Y-6413	1-22-99	1-22-00

Notes/Adjustments/Repairs/Modifications:
Transducer total accuracy specifications: 0.15% Rdg (accuracy) + 0.01% FS (repeatability) + 0.005% FS (zero temp coef) + 0.02% Rdg (span temp coef). Indicator total accuracy: 0.005% RDG + 2 counts. Total system accuracy above.
adjusted indicator zero + fullscale and transducer zero

Limitations for use:
Not calibrated below 500 TORR

Date Calibrated: 5-6-99 Date Due Recalibration: 5-6-00 Cal Interval: 12 month

Calibration Performed by: Dave Cotton Date: 5-6-99

Reviewed by: [signature] Date: 5/6/1999

Attachment
Calibration Data Sheet

MRI No.: — Model No./Type: TTSS-18G-12 Serial No.: T99130 Report No.: —

Noun: "T" Thermocouple Ambient Temperature: 74° F Ambient Humidity: 31%

Applied temperature	Initial check		Final check	Tolerance ±	Pass	Fail
°C	mv.	Calculated °C	Same	°C		
-25.0	-0.9237	-24.5	Same	1.0	✓	
0.0	0.0059	0.2	Same	1.0	✓	
100.0	4.2749	99.9	Same	1.0	✓	
150.0	6.7043	150.0	Same	1.1	✓	
200.0	9.2911	200.1	Same	1.5	✓	
300.0	14.8765	300.3	Same	2.2	✓	
400.0	20.8913	400.3	Same	3.0		

Cumulative uncertainties of the standards used to perform this calibration did not exceed the requirements of MRI-0701 and ISO 10012-1.

Standards used: MRI No.	Date calibrated	Date due calibration
Y-5835	12-4-98	12-4-99
012601	5-26-98	5-26-99
012600	4-23-99	4-23-00
012693	1-7-99	1-7-00

Notes/Adjustments/Repairs/Modifications:
N/A

Limitations for use:
N/A

Date Calibrated: 5-7-99 Date Due Recalibration: 5-7-00 Cal Interval: 1 Year

Calibration Performed by: Dave Cotton Date: 5-7-99

Reviewed by: [signature] Date: 5-10-99

Attachment
Calibration Data Sheet

MRI No.: Y-6474 Model No./Type: HH21 Serial No.: T-206821 Report No.: —

Noun: Thermocouple Thermometer Ambient Temperature: 74° F Ambient Humidity: 28°

Applied temperature	Initial check	Final check	Tolerance ±	Pass	Fail
"T" -200°C	-200.4°C	Same	0.8°C	✓	
"T" -100°C	-100.3°C		0.7°C	✓	
"T" 0°C	-0.2°C		0.6°C	✓	
"T" 100°C	99.7°C		0.7°C	✓	
"T" 150°C	149.7°C		0.75°C	✓	
"T" 200°C	199.6°C		0.8°C	✓	
"T" 300°C	299.7°C		0.9°C	✓	
"T" 400°C	399.7°C	Same	1.0°C	✓	

Cumulative uncertainties of the standards used to perform this calibration did not exceed the requirements of MRI-0701 and ISO 10012-1.

Standards used: MRI No.	Date calibrated	Date due calibration
Y-5835	12-4-98	12-4-99

Notes/Adjustments/Repairs/Modifications:
NONE

Limitations for use:
Type T thermocouples only

Date Calibrated: 5-7-99 Date Due Recalibration: 5-7-00 Cal Interval: 1 Year

Calibration Performed by: Dan Upton Date: 5-7-99

Reviewed by: [illegible] Date: 5-10-99

Scott Specialty Gases

Shipped From: 6141 EASTON ROAD, BLDG 1 PO BOX 310
PLUMSTEADVILLE PA 18949-0310
Phone: 215-766-8861 Fax: 215-766-2070

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
SCOTT KLAMM
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 01-01788-006
PO#: 033452
ITEM #: 01021951 5AL
DATE: 3/31/98

CYLINDER #: ALM025384
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +/-5%

BLEND TYPE : CERTIFIED WORKING STD

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
ETHYLENE	20.	PPM	20.0	PPM
NITROGEN		BALANCE		BALANCE

ANALYST: Genya Kogut
GENYA KOGUT

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34162-005
PO#: 038546
ITEM #: 12022751 1AL
DATE: 5/26/99

CYLINDER #: ALM046483
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
METHANE	50.	PPM	52.6	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: B.M. BECTON

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34162-004
PO#: 038546
ITEM #: 12022232 1AL
DATE: 5/25/99

CYLINDER #: ALM045092
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/25/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
N-HEXANE	50.	PPM	49.6	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34167-006
PO#: 038545
ITEM #: 1202M2034951AL
DATE: 5/27/99

CYLINDER #: ALM037409
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/27/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
3-METHYLPENTANE	50.	PPM	50.0	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34162-006
PO#: 038546
ITEM #: 1202P2000801AL
DATE: 5/27/99

CYLINDER #: ALM041358
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/27/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
N-PENTANE	50.	PPM	49.99	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

C E R T I F I C A T E O F A N A L Y S I S

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34167-005
PO#: 038545
ITEM #: 1202M2034941AL
DATE: 5/26/99

CYLINDER #: ALM054078
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
2-METHYL-2-PENTENE	50.	PPM	51.4	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: B.M. BECTON

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34167-004
PO#: 038545
ITEM #: 1202M2034961AL
DATE: 5/26/99

CYLINDER #: ALM005876
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
2-METHYL 2-BUTENE	50.	PPM	50.04	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34167-003
PO#: 038545
ITEM #: 1202M2034971AL
DATE: 5/26/99

CYLINDER #: ALM017936
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
2-METHYL-1-PENTENE	50.	PPM	50.08	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34167-002
PO#: 038545
ITEM #: 1202P2019421AL
DATE: 5/27/99

CYLINDER #: ALM041929
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/27/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
1-PENTENE	50.	PPM	50.1	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: B.M. BECTON

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34162-003
PO#: 038546
ITEM #: 1202N2007311AL
DATE: 5/26/99

CYLINDER #: AAL21337
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
N-HEPTANE	50.	PPM	49.97	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: L. TAYLOR

Scott Specialty Gases

Shipped From: 1750 EAST CLUB BLVD
DURHAM NC 27704
Phone: 919-220-0803 Fax: 919-220-0808

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH

CROSSROADS CORP PARK
5520 DILLARD RD,SUITE 100
CARY NC 27511

PROJECT #: 12-34162-001
PO#: 038546
ITEM #: 12021152 1AL
DATE: 5/25/99

CYLINDER #: ALM020217
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/25/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
N-BUTANE	50.	PPM	51.3	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST: B.M. Becton
B.M. BECTON

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7-7-99

OPERATOR: Geyer

			Initials
Check cell temperature			
Verify temperature using thermocouple probe and hand-held readout			23.8 °C
Purge cell with dry nitrogen and vent to ambient pressure			JG
Record ambient pressure in cell, (P_b)			749.9 torr
~~Vacuum~~ **Leak Check Procedure:** (positive Pressure)			JG
Evacuate cell to baseline pressure.			
Isolate cell (close cell inlet and cell outlet)			
Record time and baseline pressure (P_{min})	11:07:45 Time	775.4 P_{min}	JG
Leave cell isolated for one minute			
Record time and cell pressure (P_{max})	11:06:45 Time	775.7 P_{max}	JG
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.3 ΔP	JG
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		1.16% $\% V_L$	JG
$\lvert \% V_L \rvert$ should be < 4			

(of difference between P_{max} and $P_{ambient}$)

Record Nitrogen Background		
Purge cell with dry nitrogen		JG
Verify cell is as dry as previous background		JG
Record ambient pressure using cell Barocell gauge		749.9
Record nitrogen flow rate (about sampling flow rate)		0.8 LPM (751.5 torr)
Collect Background (AQBK) under continuous flow and ambient pressure		0.8 LPM JG
Record information in data book.		JG
Copy Background to C-drive and backup using batch file.		JG BK60707A

Record CTS Spectrum		
Record Cell path length setting		
Purge ~~Evacuate Cell~~		10.03 JG
Fill Cell with CTS gas		11:23 JG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)		1.0 LPM
Record cylinder ID Number		AØ18472
Record CTS gas cylinder identity and concentration		Ethylene in N_2 (104.4 ppm)
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.		
Record Barytron pressure during collect		751.8
~~Record information on "Background and Calibrations" data sheet.~~		JG
Verify that spectrum and interferogram were copied to directories.		JG
Record CTS Spectrum File Name		CTS0707A

Reviewed by: JGeyer

Date: 7/7/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/7/99

OPERATOR: T. Bayer

			Initials
Check cell temperature			
Verify temperature using thermocouple probe and hand-held readout			
Purge cell with dry nitrogen and vent to ambient pressure			
Record ambient pressure in cell, (P_b)			
Vacuum Leak Check Procedure:			
Evacuate cell to baseline pressure.			
Isolate cell (close cell inlet and cell outlet)			
Record time and baseline pressure (P_{min})			
Leave cell isolated for one minute	Time	P_{min}	
Record time and cell pressure (P_{max})			
Calculate "leak rate" for 1 minute	Time	P_{max}	
$\Delta P = P_{min} - P_{max}$			
Calculate "leak rate" as percentage of total pressure		ΔP	
$\% V_L = (\Delta P/P_b) * 100$			
$\lvert \% V_L \rvert$ should be < 4		$\% V_L$	

Record Nitrogen Background	
Purge cell with dry nitrogen	
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	BK60707A

Record CTS Spectrum	
Record Cell path length setting	10.03
Purge **Evacuate Cell**	
Fill Cell with CTS gas	
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	1.0 LPM
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration	20.0 ppm Ethylene in N
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	
Record Barytron pressure during collect	~~752.0~~ 751.8
~~Record information on "Background and Calibrations" data sheet.~~	
Verify that spectrum and interferogram were copied to directories.	
Record CTS Spectrum File Name	CTS0707B

Reviewed by: T. Bayer

Date: 7/7/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7-8-99

OPERATOR: T. Geyer

			Initials
Check cell temperature			24.0 °C
Verify temperature using thermocouple probe and hand-held readout			d/c
Purge cell with dry nitrogen and vent to ambient pressure			
Record ambient pressure in cell, (P_b)			750.5 torr
~~Vacuum~~ Leak Check Procedure:			
Positive Pressure — Evacuate (Purge) cell to baseline (approximate) pressure.			JG
Isolate cell (close cell inlet and cell outlet)			JG
Record time and baseline pressure (P_{min})	10:36:10 (Time)	779.0 (P_{min})	JG
Leave cell isolated for one minute			
Record time and cell pressure (P_{max})	10:35:10 (Time)	779.3 (P_{max})	JG
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.3 (ΔP)	JG
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		1.04 ($\% V_L$)	JG
$\lvert \% V_L \rvert$ should be < 4			

% of $P_{max} - P_{ambient}$

Record Nitrogen Background

Purge cell with dry nitrogen	JG
Verify cell is as dry as previous background	d/c
Record ambient pressure using cell Barocell gauge	750.5
Record nitrogen flow rate (about sampling flow rate)	0.47 LPM
Collect Background (AQBK) under continuous flow and ambient pressure	JG
Record information in data book.	JG
Copy Background to C-drive and backup using batch file.	JG

Record CTS Spectrum

Record Cell path length setting	
~~Evacuate Cell~~	10.03
Purge ~~Fill~~ Cell with CTS gas	JG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	JG
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration	20 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	JG
Record Barytron pressure during collect	752.7 @ 0.9061
Record information on "Background and Calibrations" data sheet.	JG
Verify that spectrum and interferogram were copied to directories.	JG
Record CTS Spectrum File Name	CTS0708A

Reviewed by: [signature] Date: 7/8/99

Project No. 4951 - 12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/9/99

OPERATOR: J. Geyer

			Initials
Check ~~cell~~ gas in cell temperature			
Verify temperature using thermocouple probe and hand-held readout			JB
Purge cell with dry nitrogen and vent to ambient pressure			126.6 °C
Record ambient pressure in cell, (P_b)			751.4 torr
~~Vacuum~~ Leak Check Procedure:			
Positive Pressure Purge ~~Evacuate~~ Isolate cell ~~to baseline~~ above ambient pressure.			
Isolate cell (close cell inlet and cell outlet)			JG
Record time and baseline pressure (P_{min})	11:08:30 Time	770.8 P_{min}	JG
Leave cell isolated for one minute			JG
Record time and cell pressure (P_{max}) Start	11:09:30 Time	771.5 P_{max}	JG
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.7 ΔP	JG
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		3.5	JG
$\lvert \% V_L \rvert$ should be < 4		% V_L	
		% of $P_{max} - P_{ambient}$	
Record Nitrogen Background			
Purge cell with dry nitrogen			JG
Verify cell is as dry as previous background			JG
Record ambient pressure using cell Barocell gauge			JG
Record nitrogen flow rate (about sampling flow rate)			0.49 LPM
Collect Background (AQBK) under continuous flow and ambient pressure			JG
Record information in data book.			JG
Copy Background to C-drive and backup using batch file.			JG
Record CTS Spectrum			
Record Cell path length setting			10.03
Purge ~~Evacuate~~ Cell			JG
Fill Cell with CTS gas			JG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)			JG
Record cylinder ID Number			ALM025384
Record CTS gas cylinder identity and concentration			20.0 ppm Sulfur
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.			JG
Record Barytron pressure during collect			JG
Record information on "Background and Calibrations" data sheet.			JG
Verify that spectrum and interferogram were copied to directories.			
Record CTS Spectrum File Name			CTS0709A

Reviewed by: J. Geyer

Date: 7/9/99

Project No. 4951-12, 17

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/12/99

OPERATOR: T. Gayes

			Initials
Check cell temperature			
Verify temperature using thermocouple probe and hand-held readout			126.5°C TG
Purge cell with dry nitrogen and vent to ambient pressure			TG
Record ambient pressure in cell, (P_b)			753.3 torr
~~Vacuum~~ Leak Check Procedure:			TG
Pressure Evacuate cell to baseline pressure. (Purge, Maximum)			
Isolate cell (close cell inlet and cell outlet)			TG
Record time and baseline pressure (P_{min})	8:52:30 Time	773.3 P_{min}	TG
Leave cell isolated for one minute			TG
Record time and cell pressure (P_{max})	8:51:30 Time	774.0 P_{max}	TG
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.70 ΔP	TG
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		3.4% $\% V_L$	TG
$\lvert \% V_L \rvert$ should be < 4			

% of $P_{max} - P_{ambient}$

Record Nitrogen Background	
Purge cell with dry nitrogen	1.1 LPM
Verify cell is as dry as previous background	TG
Record ambient pressure using cell Barocell gauge	753.3
Record nitrogen flow rate (about sampling flow rate)	1.1 LPM
Collect Background (AQBK) under continuous flow and ambient pressure	TG
Record information in data book.	TG
Copy Background to C-drive and backup using batch file.	TG

Record CTS Spectrum	
Record Cell path length setting	10.03 TG
~~Evacuate Cell~~	—
Fill Cell with CTS gas	TG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	TG 1.17 LPM
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration	20.0 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	TG
Record Barytron pressure during collect	756.1
Record information on "Background and Calibrations" data sheet.	TG
Verify that spectrum and interferogram were copied to directories.	TG
Record CTS Spectrum File Name	C50712A

Reviewed by: [signature] Date: 7/12/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/13/99

OPERATOR: T. Geyer

			Initials
Check cell temperature			126.4 °C
Verify temperature using thermocouple probe and hand-held readout			752.9 torr
Purge cell with dry nitrogen and vent to ambient pressure			
Record ambient pressure in cell, (P_b)			752.9 torr
~~**Vacuum Leak Check Procedure:**~~ overpressure?			
Pressure Evacuate cell to baseline pressure.			
Isolate cell (close cell inlet and cell outlet)			
Record time and baseline pressure (P_{min})	10:42:03 Time	771.4 P_{min}	
Leave cell isolated for one minute			
Record time and cell pressure (P_{max})	10:41:03 Time	771.8 P_{max}	
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.4 ΔP	
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		2.1 $\% V_L$	
$\lvert \% V_L \rvert$ should be < 4			

% of $P_{max} - P_{ambient}$

Record Nitrogen Background

Purge cell with dry nitrogen	
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	

Record CTS Spectrum

Record Cell path length setting	
Ref ~~Evacuate~~ Cell	10.03
Fill Cell with CTS gas	
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	
Record cylinder ID Number	AL402538 4
Record CTS gas cylinder identity and concentration	200 ppm ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	
Record Barytron pressure during collect	
Record information on "Background and Calibrations" data sheet.	
Verify that spectrum and interferogram were copied to directories.	
Record CTS Spectrum File Name	CS0713A

Reviewed by: J Thomas Cox

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/15/99

OPERATOR: T. Geyer

			Initials
Check cell temperature			
Verify temperature using thermocouple probe and hand-held readout			TG
Purge cell with dry nitrogen and vent to ambient pressure			125.6
Record ambient pressure in cell, (P_b)			TG
Vacuum Leak Check Procedure:			754.8 torr
Purge ~~Evacuate~~ cell to baseline pressure. (P_{min})			TG
Isolate cell (close cell inlet and cell outlet)			TG
Record time and baseline pressure (P_{min})	11:28:15 Time	774.4 P_{min}	TG
Leave cell isolated for one minute			TG
Record time and cell pressure (P_{max})	11:27:05 Time	774.8 P_{max}	TG
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.4 ΔP	TG
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		2.0% $\% V_L$	TG
$\lvert \% V_L \rvert$ should be < 4			

% of $P_{max} - P_{ambient}$

Record Nitrogen Background	
Purge cell with dry nitrogen	TG
Verify cell is as dry as previous background	TG
Record ambient pressure using cell Barocell gauge	754.8 TG
Record nitrogen flow rate (about sampling flow rate)	1.03 LPM
Collect Background (AQBK) under continuous flow and ambient pressure	TG
Record information in data book.	TG
Copy Background to C-drive and backup using batch file.	TG

Record CTS Spectrum	
Record Cell path length setting	10.03
Purge ~~Evacuate~~ Cell	TG
Fill Cell with CTS gas	TG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	TG
Record cylinder ID Number	AL-M 25381
Record CTS gas cylinder identity and concentration	20.0 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	TG
Record Barytron pressure during collect	757.1
Record information on "Background and Calibrations" data sheet.	TG
Verify that spectrum and interferogram were copied to directories.	TG
Record CTS Spectrum File Name	CT0715A

Reviewed by: TG

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up Procedure

DATE: 7/6/99

OPERATOR: T. Gayer

			Initials
Check cell temperature			
Verify temperature using thermocouple probe and hand-held readout			7/6
Purge cell with dry nitrogen and vent to ambient pressure			125.3 °C
Record ambient pressure in cell, (P_b)			7/6
Vacuum Leak Check Procedure:			755.2
Purge ~~Evacuate~~ cell to ~~baseline~~ P_{max} pressure.			7/6
Isolate cell (close cell inlet and cell outlet)			7/6
Record time and baseline pressure (P_{min})	9:52:40 (Time)	778.5 torr (P_{min})	7/6
Leave cell isolated for one minute			
Record time and cell pressure (P_{max})	9:51:40 (Time)	779.2 (P_{max})	7/6
Calculate "leak rate" for 1 minute			
$\Delta P = P_{min} - P_{max}$		0.7 (ΔP)	7/6
Calculate "leak rate" as percentage of total pressure			
$\% V_L = (\Delta P/P_b) * 100$		2.9 ($\% V_L$)	7/6
$\lvert \% V_L \rvert$ should be < 4			

% of P_{max} - $P_{ambient}$

Record Nitrogen Background	
Purge cell with dry nitrogen	7/6
Verify cell is as dry as previous background	7/6
Record ambient pressure using cell Barocell gauge	7/6 755.2
Record nitrogen flow rate (about sampling flow rate)	0.99 LPM
Collect Background (AQBK) under continuous flow and ambient pressure	7/6
Record information in data book.	7/6
Copy Background to C-drive and backup using batch file.	7/6

Record CTS Spectrum	
Record Cell path length setting	
Purge ~~Evacuate~~ Cell	1057
Fill Cell with CTS gas	7/6
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	7/6
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration	20.0 ppm
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	7/6
Record Barytron pressure during collect	757.1
Record information on "Background and Calibrations" data sheet.	7/6
Verify that spectrum and interferogram were copied to directories.	7/6
Record CTS Spectrum File Name	CTS0716A

Reviewed by: 7/6

Date: 7/6/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13

BAROMETRIC: 749.9 torr

SITE: NCO Laboratory

DATE: 7/7/99

OPERATOR: T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD
10:47	BKG0707A	10.03	N_2 through cell @ 0.8 LPM	500	1.0	23.8	751.5	–	NB/Med
11:37	CTS0707A	"	104.4 ppm ethylene @ 1.0 LPM	500	1.0	23.8	751.8	707A	"
12:45	CTS0707B	"	20.0 ppm ethylene 1.0 LPM	500	1.0	23.8	751.8	707A	"
~~13:31~~	~~BKG0707B~~	~~5.07~~	~~N_2 @ 0.8 LPM~~	~~500~~	~~1.0~~	~~23.5~~	~~751.2~~	~~–~~	~~"~~
1345	CTS0707C	10.03	200 ppm ethylene @ 1.0 LPM	500	1.0	23.5	751.7	707A	NB/Med

Did not do

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Date 7-7-99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 750.5

SITE: NCO Laboratory **DATE:** 7/8/99 **OPERATOR:** T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD
10:40	BK0708A	10.03	N_2 through cell @ 0.47 LPM after realign.	500	1.0	24.0	751.3	—	NB/mod
11:32	CTS0708A	10.03	20.0 ppm Ethylene @ 0.90 LPM	500	1.0	23.8	752.7	708A	"
16:05	BK0708B	10.03	N_2 @ 0.50 LPM GAIN is now 2R for source signal as cold cell (~70%). Did not realign after heating	500	1.0	126.5°C	749.9	—	NB/mod
16:26	CTS0708B	10.03	Ethylene @ 0.97 LPM 20.0 ppm	500	1.0	126.4°C	750.8	708B	NB/mod

Ambient = 749.0

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Date 7/8/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 751.4

SITE: NCO Laboratory **DATE:** 7/9/99 **OPERATOR:** T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD
13:13	BKG0709A	10.03	N_2 @ 0.99 LPM	500	1.0	126.2	751.3	—	[illegible]
13:44	CTS0709A	10.03	20 ppm ethylene @ 0.89 LPM	500	1.0	126.3	752.0	A	"
14:14	1960709A	10.03	52.6 ppm methane in N_2 @ 1.09 LPM ALM046483	500	1.0	126.3	752.2	A	"
14:27	1960709B	10.03	52.6 ppm methane in N_2 @ 1.10 LPM ALM046483	500	1.0	126.3	752.1	A	"
14:55	BKG0709B	10.03	N2 @ ~~0.45~~ 0.90 LPM	500	1.0	126.3	751.3 ~~750.4~~	✓	"
16:03	CTS0709B	10.03	20.0 ppm ethylene @ 0.93 LPM	500	1.0	126.1	751.0	B	"
16:25	1960709C ~~B~~	10.03	52.6 ppm methane in N_2 @ 1.11 LPM ALM046483	500	1.0	126.1	751.3	B	"
16:39	1960709D	10.03	52.6 ppm methane @ 1.10 LPM	500	1.0	126.1	751.2	B	"

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Date 7/7/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 752.9

SITE: NCO Laboratory **DATE:** 7/13/99 **OPERATOR:** T. Gayer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD	
11:00	BKG0713A	10.03	N_2 through cell @ 0.95 LPM ~~higher purge today for this BKG~~	500	1.0	125.3	754.9	–	N/B mod	Gain = 2
11:15	BKG0713B	10.03	Same conditions RaBo because compartmentalize dryer	500	1.0	125.4	754.9	–	"	"
11:54	BKG0713C	10.03	Same conditions w/ dryer cabinet 0.97 LPM	500	1.0	125.4	755.0	–	"	"
12:30	CTS0713A	10.03	20.0 ppm Ethylene in N_2 Alltech 25384 @ 0.95 LPM	500	1.0	125.5	755.0	713 C	"	"
13:03	CTS0713B	10.03	" @ 0.92 LPM	500	1.0	125.6	754.8	"	"	"
13:22	1960713A	10.03	52.6 ppm Methane @ 1.02 LPM	500	1.0	125.5	755.1	713 C	"	"
14:53	CTS0713C	10.03	20.0 ppm Ethylene @ 0.99 LPM	500	1.0	125.4	754.7	713C	"	"
15:08	1960713B	10.03	52.6 ppm Methane @ 1.12 LPM	500	1.0	125.4	755.0	713 C	"	"

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Date 7/15/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 753.3

SITE: NCO Laboratory **DATE:** 7/12/99 **OPERATOR:** T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD	
10:40	BKG0712A	10.03	N_2 through cell @ 1.12 LPM	500	1.0	126.0	756.3	—	NB/Mod	GAIN=2
10:55	CTS0712A	10.03	20.0 ppm Ethylene in N_2 @ 1.06 LPM	500	1.0	126.0	756.1	712 A	"	"
11:10	CTS0712B	10.03	20. ppm Ethylene @ 0.79 LPM (712A was ~10% low)	500	1.0	126.2	755.9	"	"	"
12:20	1960712A	10.03	52.6 ppm Methane in N_2 @ 0.99 LPM ALMOST 48.3	500	1.0	126.1	755.9	712 A	"	"
12:45	BKG0712B	10.03	N_2 @ 1.11 LPM	500	1.0	126.1	756.0	—	"	"
14:40	CTS0712C	10.03	20.0 ppm Ethylene @ 1.10 LPM	500	1.0	126.3	755.6	712 B	"	"
14:56	1960712B	10.03	52.6 ppm Methane in N_2 @ 1.06 LPM	500	1.0	126.4	755.5	712 B	"	"

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Date 7/15/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 754.8 torr

SITE: NCO Laboratory **DATE:** 7/15/99 **OPERATOR:** T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD	
11:40	BK60715A	10.03	N2 through cell @ 1.09 LPM	500	1.0	125.5	757.1 ~~754.8~~	–	N/B and	Gain=2
12:15	CT50715A	10.03	20.0 ppm Ethylene @ 1.07 LPM ALM025384	500	1.0	125.2	757.1	A	"	"
12:36	196 0715A	10.03	52.6 ppm Methane @ 1.09 LPM	500	1.0	125.2	757.0	A	"	"
1403	BK60715B	10.03	N2 @ 1.00 LPM	500	1.0	125.1	756.6	–	"	"
16:00	CT50715B	10.03	20.0 ppm Ethylene 0.97 LPM ALA025384	500	1.0	125.0	755.8	715B	"	"
16:20	196 0715B	10.03	ALM 46483 @ 1.05 LPM Methane (52.6 ppm)	500	1.0	125.0 ~~1.05 LPM~~	755.8	715B	"	"

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Date 7/15/99

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13 BAROMETRIC: 755.2

SITE: NCO Laboratory DATE: 7/16/99 OPERATOR: T. Gayer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (°F)	Gas PRESSURE	BKG	APOD	
11:25	BK0716A	10.03	N_2 through cell @ 0.98 LPM	500	1.0	125.12	757.2	–	NB/mod	Gain = 2
12:39	CT0716A	10.03	20.0 ppm Ethylene @ 1.00 LPM	500	1.0	125.2	757.0	716A	"	"
12:51	1960716A	10.03	52.6 ppm Methane ALM046483 @ 1.09 LPM	500	1.0	125.1	757.2	716A	"	"
15:15	CTS0716B	10.03	20.0 ppm Ethylene ARM025384 @ 1.0 LPM	500	1.0	125.2	756.1	716A	"	"
15:33	1960716A	10.03	52.6 ppm Methane @ 0.99 LPM	500	1.0	125.2°C	756.1	716A	"	"

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Date 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-9-99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	
Start Time (data collect)	Hexane
Record Cell path length setting	15:24
Record Background Spectrum File Name	10.03
Record CTS Spectrum File Name	BK60709B
Record Compound Name	CTS0709A,B / 1960709
Record Cylinder Identification Number	Hexane
Record Cylinder Concentration	AL#45092
Record Spectrum File Name	49.6 ppm
Fill cell to ambient pressure with gas from cylinder standard	0950709A
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	TG
Allow to equilibrate for 5 minutes	1.00 LPM
Record sample pressure in cell	TG
Record sample flow rate through cell	757.3 torr
Start spectrum collect program	1.002 LPM
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	TG
	15:34
	126.2°C

Reviewed by: [signature]

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-9-99

OPERATOR: T. Gayor

	Initials
Reference Spectrum Sample	HEXANE 0950709B
Start Time 15:36	10.03
Record Cell path length setting	BH60709B
Record Background Spectrum File Name	C750709A,B 1960707A-
Record CTS Spectrum File Name	
Record Compound Name	HEXANE
Record Cylinder Identification Number	AL045092
Record Cylinder Concentration	49.6 ppm
Record Spectrum File Name	0950709B
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.09 LPM
Allow to equilibrate for 5 minutes	Continued from "A"
Record sample pressure in cell	751.5 ppm
Record sample flow rate through cell	1.09 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	15:46
	126.1 °C

Reviewed by: [signature]

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-12-99

OPERATOR: T. Gayer

	Initials
Reference Spectrum Sample	
Start Time	13:14 1-pentene
Record Cell path length setting	13:14
Record Background Spectrum File Name	10.03
Record CTS Spectrum File Name	BKG0712 B
Record Compound Name	CTS 0712 A-C, 1968
Record Cylinder Identification Number	1-Pentene
Record Cylinder Concentration	AL40419 29
Record Spectrum File Name	50.1 ppm
Fill cell to ambient pressure with gas from cylinder standard	IPE0712A
Open cell outlet vent valve	N/A
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	N/A
Allow to equilibrate for 5 minutes	1.00 LPM
Record sample pressure in cell	N/A
Record sample flow rate through cell	765.6
Start spectrum collect program	1.00 LPM
Record information in data book	N/A
Copy Spectrum and Interferogram to backup directories	N/A
End Time	N/A
	13:24
	126.0°C

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Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/12/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	1-Pentene
Start Time	13:38
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60712B
Record CTS Spectrum File Name	CTS0712A-C 1960712
Record Compound Name	1-Pentene
Record Cylinder Identification Number	ALM#41929
Record Cylinder Concentration	50.1 ppm
Record Spectrum File Name	1PE0712B
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.03 LPM
Allow to equilibrate for 5 minutes continued from A	TG
Record sample pressure in cell	755.6
Record sample flow rate through cell	1.03 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	13:48
	126.1°C

Reviewed by: [signature]

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-12-99

OPERATOR: T Geyer

	Initials
Reference Spectrum Sample	~~HFPO~~ n-heptane
Start Time	14:10
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60712B
Record CTS Spectrum File Name	CTS0712A-C 1960712A
Record Compound Name	n-heptane
Record Cylinder Identification Number	AAL 21339
Record Cylinder Concentration	49.99 ppm
Record Spectrum File Name	HSP0712A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.00 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	755.2 torr
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	14:20

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Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/12/99

OPERATOR: T. Gayle

	Initials
Reference Spectrum Sample	n-heptane
Start Time	14:20
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK0712 B
Record CTS Spectrum File Name	CTS0712 A-C, 1960712 A,
Record Compound Name	n-heptane
Record Cylinder Identification Number	AAL 21337
Record Cylinder Concentration	49.99 ppm
Record Spectrum File Name	HEP0712 B
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.00 LPM
Allow to equilibrate for 5 minutes Continued from A	TG
Record sample pressure in cell	755.3
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	14:39

Reviewed by: T. Gayle

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Gayer

	Initials
Reference Spectrum Sample	2-methyl-2-pentene
Start Time	13:46
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60713C
Record CTS Spectrum File Name	CTS0713A-C 19607131
Record Compound Name	2-methyl-2-pentene
Record Cylinder Identification Number	ALM054078
Record Cylinder Concentration	51.4 ppm
Record Spectrum File Name	2M2P713A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.00 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	754.9
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	14:55

Reviewed by: [signature]

Date: 7/15/99

Project No. 4961-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Geyer

		Initials
Reference Spectrum Sample		2-methyl-2-pentene
Start Time		13:56
Record Cell path length setting	10.03	
Record Background Spectrum File Name		BK60713C
Record CTS Spectrum File Name		CT10713AC (960713A,B
Record Compound Name		2-methyl-2 pentene
Record Cylinder Identification Number		ALM054078
Record Cylinder Concentration		51.4 ppm
Record Spectrum File Name		2M2P7138
Fill cell to ambient pressure with gas from cylinder standard		TG
Open cell outlet vent valve		TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate		TG
Allow to equilibrate for 5 minutes	Continued from A	TG
Record sample pressure in cell		754.9
Record sample flow rate through cell		0.98 LPM
Start spectrum collect program		TG
Record information in data book		TG
Copy Spectrum and Interferogram to backup directories		TG
End Time		14:05

Reviewed by: T. Geyer

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: J. Geyer

	Initials
Reference Spectrum Sample	3-methyl pentane
Start Time	14:21
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60713C
Record CTS Spectrum File Name	CTS0713A-C 1960713A,B
Record Compound Name	3-methylpentane
Record Cylinder Identification Number	ALM037409
Record Cylinder Concentration	50.0 ppm
Record Spectrum File Name	3MP0713A
Fill cell to ambient pressure with gas from cylinder standard	JG
Open cell outlet vent valve	JG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.00 LPM
Allow to equilibrate for 5 minutes	JG
Record sample pressure in cell	754.6
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	JG
Record information in data book	JG
Copy Spectrum and Interferogram to backup directories	JG
End Time	14:30

Reviewed by: J. Geyer

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Gayer

	Initials
Reference Spectrum Sample	3-methylpentane
Start Time	14:34
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60713C
Record CTS Spectrum File Name	CTS0713A-C 1960713A
Record Compound Name	3-methylpentane
Record Cylinder Identification Number	IALM 37409
Record Cylinder Concentration	50.0 ppm
Record Spectrum File Name	3MP0713B
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	0.98 LPM
Allow to equilibrate for 5 minutes Continued for A	TG
Record sample pressure in cell	754.6 torr
Record sample flow rate through cell	0.96 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	1444

Reviewed by: TG

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Gayer

	Initials
Reference Spectrum Sample	(2,2,4-trimethylpentane isooctane
Start Time (start purge)	12:52
Record Cell path length setting	10.03
Record Background Spectrum File Name	BKG0715A
Record CTS Spectrum File Name	CTS0715A,B 1960715A
Record Compound Name	isooctane
Record Cylinder Identification Number	~~AC10715B~~ ALU044
Record Cylinder Concentration	50.3 ppm
Record Spectrum File Name	1650715A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.06 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	757.0
Record sample flow rate through cell	1.02 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	13:08
	125.2 °C

Reviewed by: [signature]

Date: 7/16/99

Project No. 7951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	Isooctane
Start Time	1310
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK0715A
Record CTS Spectrum File Name	CTS0715A,B 1960715A,B
Record Compound Name	Isooctane
Record Cylinder Identification Number	AL004469B
Record Cylinder Concentration	50.3 ppm
Record Spectrum File Name	1650715A
Fill cell to ambient pressure with gas from cylinder standard continued from A	TJG
Open cell outlet vent valve	TJG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	0.98
Allow to equilibrate for 5 minutes	TJG
Record sample pressure in cell	756.9
Record sample flow rate through cell	0.98
Start spectrum collect program	TJG
Record information in data book	TJG
Copy Spectrum and Interferogram to backup directories	TJG
End Time	

Reviewed by: [signature]

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	n-Pentane
Start Time	14:30
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK60715B
Record CTS Spectrum File Name	CTS0715A,B M60715A,B
Record Compound Name	n-Pentane
Record Cylinder Identification Number	ALM024358
Record Cylinder Concentration	49.99
Record Spectrum File Name	Pen0715A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	TG 1.07 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	756.4
Record sample flow rate through cell	1.09 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	14:48
	124.9 °C

Reviewed by: [signature]

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	n-Butane
Start Time	15:06
Record Cell path length setting	10.03
Record Background Spectrum File Name	BKG0715B
Record CTS Spectrum File Name	CTS0715A,B 19G0715A
Record Compound Name	Butane
Record Cylinder Identification Number	ALM020217
Record Cylinder Concentration	51.3 ppm
Record Spectrum File Name	BuT0715A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.06 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	756.1 torr
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	TG 15:23
	124.9°C

Reviewed by: 7/16 [signature]

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample continued from "A"	Butane
Start Time	10.03
Record Cell path length setting	10.03
Record Background Spectrum File Name	7128
Record CTS Spectrum File Name	CT 8712A,B 196072
Record Compound Name	Butane
Record Cylinder Identification Number	AL402021
Record Cylinder Concentration	51.3 ppm
Record Spectrum File Name	BUT0715A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	1.02
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	756.1
Record sample flow rate through cell	1.02
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	15:40

124.9.2

Reviewed by: TG

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	2-methyl-2-butene
Start Time	13:08
Record Cell path length setting	10.03
Record Background Spectrum File Name	BK607160A
Record CTS Spectrum File Name	CTS07160A,B 1960716A
Record Compound Name	2-M
Record Cylinder Identification Number	AL40058716
Record Cylinder Concentration	50.04 ppm
Record Spectrum File Name	2M2B716A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	TG
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	756.8
Record sample flow rate through cell	1.00 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	13:30
	125.2°C

Reviewed by: [signature]

Date: 7/16/99

Project No. 4451-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Gayer

		Initials
Reference Spectrum Sample		
Start Time	13:30	2-methyl-2-butene
Record Cell path length setting		10.03
Record Background Spectrum File Name		
Record CTS Spectrum File Name		BK60716A
Record Compound Name		CTS0716A,B MT60716A,B
Record Cylinder Identification Number		2/6
Record Cylinder Concentration		ALH005876
Record Spectrum File Name		50.04 ppm
Fill cell to ambient pressure with gas from cylinder standard		2M28716B
Open cell outlet vent valve		2/6
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate		2/6
Allow to equilibrate for 5 minutes	Contained from "A"	2/6
Record sample pressure in cell		2/6
Record sample flow rate through cell		756.6 torr
Start spectrum collect program		0.95 LPM
Record information in data book		2/6
Copy Spectrum and Interferogram to backup directories		2/6
End Time		2/6
		1540

Reviewed by: [signature]

Date: 7/16/99

Project No. _____

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: _____

OPERATOR: _____

	Initials
Reference Spectrum Sample	
Start Time	_____
Record Cell path length setting	_____
Record Background Spectrum File Name	_____
Record CTS Spectrum File Name	_____
Record Compound Name	_____
Record Cylinder Identification Number	_____
Record Cylinder Concentration	_____
Record Spectrum File Name	_____
Fill cell to ambient pressure with gas from cylinder standard	_____
Open cell outlet vent valve	_____
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	_____
Allow to equilibrate for 5 minutes	_____
Record sample pressure in cell	_____
Record sample flow rate through cell	_____
Start spectrum collect program	_____
Record information in data book	_____
Copy Spectrum and Interferogram to backup directories	_____
End Time	_____

Reviewed by: _____

Date: _____

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T Geyer

	Initials
Reference Spectrum Sample	2-methyl-1-pentene
Start Time	1349
Record Cell path length setting	10.03
Record Background Spectrum File Name	2K60716A
Record CTS Spectrum File Name	CTS0716A 19600716
Record Compound Name	7/6
Record Cylinder Identification Number	AL40 17936
Record Cylinder Concentration	50.08 ppm
Record Spectrum File Name	2M1P716A
Fill cell to ambient pressure with gas from cylinder standard	7/6
Open cell outlet vent valve	7/6
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	7/6 1.05LPM
Allow to equilibrate for 5 minutes	7/6
Record sample pressure in cell	756.4
Record sample flow rate through cell	1.06 LPM
Start spectrum collect program	7/6
Record information in data book	
Copy Spectrum and Interferogram to backup directories	
End Time	

Reviewed by: _____ Date: _____

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	2-methyl-1-pentene
Start Time (continued from "A")	10.03
Record Cell path length setting	716A
Record Background Spectrum File Name	CTS716A 196716A
Record CTS Spectrum File Name	
Record Compound Name	
Record Cylinder Identification Number	AL401793B
Record Cylinder Concentration	50.08
Record Spectrum File Name	2M1P716B
Fill cell to ambient pressure with gas from cylinder standard	716
Open cell outlet vent valve	716
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	716
Allow to equilibrate for 5 minutes	716
Record sample pressure in cell	756.3
Record sample flow rate through cell	0.97 LPM
Start spectrum collect program	716
Record information in data book	716
Copy Spectrum and Interferogram to backup directories	716
End Time	1440

Reviewed by: _____ Date: _____

Project No. 4951-12-13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Geyer

	Initials
Reference Spectrum Sample	n-heptane
Start Time	1425
Record Cell path length setting	10.03
Record Background Spectrum File Name	716 A
Record CTS Spectrum File Name	CTS0716A 19660716A
Record Compound Name	TG
Record Cylinder Identification Number	ARL21337
Record Cylinder Concentration	49.97 ppm
Record Spectrum File Name	HEP0716A
Fill cell to ambient pressure with gas from cylinder standard	TG
Open cell outlet vent valve	TG
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	0.97 LPM
Allow to equilibrate for 5 minutes	TG
Record sample pressure in cell	TG 756.1
Record sample flow rate through cell	0.95 LPM
Start spectrum collect program	TG
Record information in data book	TG
Copy Spectrum and Interferogram to backup directories	TG
End Time	1450

Reviewed by: _____ Date: _____

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Gayer

	Initials
Reference Spectrum Sample	
Start Time continued from "A"	n-heptane
Record Cell path length setting	—
Record Background Spectrum File Name	10.03
Record CTS Spectrum File Name	716A
Record Compound Name	(T)0716A (960716A
Record Cylinder Identification Number	—
Record Cylinder Concentration	AAL 21377
Record Spectrum File Name	49.97
Fill cell to ambient pressure with gas from cylinder standard	HG.P0716A
Open cell outlet vent valve	7/16
Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate	7/16
Allow to equilibrate for 5 minutes	7/16
Record sample pressure in cell	7/16
Record sample flow rate through cell	756.1
Start spectrum collect program	0.96 LPM
Record information in data book	7/16
Copy Spectrum and Interferogram to backup directories	7/16
End Time	7/16
	15:00

Reviewed by: _____ Date: _____

FTIR DATA FORM

Sampling Data

PROJECT NO. 4951-12 and 13

BAROMETRIC: 751.4

SITE: NCO Laboratory

DATE: 7/9/99

OPERATOR: T. Geyer

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
15:24	0950709A	10.03	Hexane (49.6 ppm in N_2) @ 1.0 LPM AL M045092	500	1.0	126.2 ~~751.3~~	1.0 LPM	torr 751.3	709 B
15:36	0950709B	10.03	Hexane, 49.6 ppm in N_2 AL M045092	500	1.0	126.1	1.09 LPM	751.5	709 B

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07-07-99

Reviewed by [signature]
Date 7/9/99

FTIR DATA FORM

Sampling Data

PROJECT NO. 4951-12 and 13

BAROMETRIC: 752.9

SITE: NCO Laboratory

DATE: 7/13/99

OPERATOR: T. Geyer

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
13:46	2M2P713A	10.03	2-methyl-2-pentene ALM# 54078 51.4 ppm	500	1.0	125.6	1.00 LPM	754.9	713C
13:56	2M2P713B	10.03	"	500	1.0	125.6	0.98 LPM	754.9	713C
14:21	3MP0713A	10.03	3-Methylpentane (50.0 ppm) ALM# 37409	500	1.0	125.5	1.00 LPM	754.6	713C
14:34	3MP0713B	10.03	"	500	1.0	125.5	0.98 LPM	754.6	713C

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Reviewed by [signature]
Date 7/13/99

FTIR DATA FORM

Sampling Data

PROJECT NO. 4951-12 and 13

BAROMETRIC: 753.1

SITE: NCO Laboratory

DATE: 7/12/99

OPERATOR: T. Geyer

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
13:14	1PE0712A	10.03	50.1 ppm 1-Pentene ALM# ~~[illegible]~~ 419 29	500	1.0	126.0	1.00 LPM	755.6	B
13:38	1PE0712B	10.03	50.1 ppm 1-Pentene	500	1.0	126.1	1.03 LPM	755.6	B
14:10	HEP0712A	10.03	49.97 ppm n-heptane AAL 21337	500	1.0	126.2	1.00 LPM	755.2	B
14:20	HEP0712B	10.03	"	500	1.0	126.4	1.00 LPM	755.3	B

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Reviewed by [illegible]
Date 7/12/99

FTIR DATA FORM

Sampling Data

PROJECT NO. 4951-12 and 13

BAROMETRIC: 754.8 Torr

SITE: NCO Laboratory

DATE: 7/15/99

OPERATOR: T. Geyer

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
12:58	1650715A	10.03	Iso octane 50.3 ppm in N_2 ALM044693	500	1.0	125.3	1.02 LPM	756.9	715A
13:13 ~~13:40~~	1650715B	10.03	Isooctane @ 0.98 LPM	500	1.0	125.2°c	0.98 LPM	756.9	715A
14:37	PEN0715A	10.03	n-Pentane @ 1.09 LPM	500	1.0	124.9°c	1.09	756.2	715 B
14:48	PEN0715B	10.03	n-Pentane @ ALM0241358	500	1.0	124.9	1.10	756.5	715 B
15:13 ~~15:04~~	But0715A	10.03	Butane 51.3 ppm ALM020217	500	1.0	124.8	1.00 LPM	756.1	715 B
15:30	BUT0715B	10.03	51.3 ppm Butane	500	1.0	124.9	1.02 LPM	756.0	715 B

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07-07-99

Reviewed by [illegible]
Date 7/14/99

FTIR DATA FORM

Sampling Data

PROJECT NO. 4951-12 and 13 **BAROMETRIC:** 755.2

SITE: NCO Laboratory **DATE:** 7/16/99 **OPERATOR:** T. Geyer

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
13:16	~~2M2~~ 2M28716A	10.03	2-methyl-2-butene AL M00 5876, 50.04 ppm	500	1.0	125.2	1.00 LPM	756.8	716A
1330	2M2A716A 2M28 0716A	10.03	2-methyl-2-butene	500	1.0	125.2	0.95 LPM	756.6	716A
1352	2M1P716A	10.03	2-methyle-1-pentene AL M01 1936 50.08 ppm	500	1.0	125.2	1.06 LPM	756.4	716A
1410	2M1P716B	10.03	2-methyl-1-pentene	500	1.0	125.1	0.97	756.3	716A
1440	HEP0716A	10.03	n-heptane 49.97 ppm AAL 21337 {Re-measure because #2 CTS was low}	500	1.0	125.3	0.95 LPM	756.1	716A
1450	HEP0716B	10.03	n-heptane	500	1.0	125.3	0.96 LPM	756.1	716A

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07-07-99

Reviewed by [illegible]
Date 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 9-7-99

OPERATOR: T. Geyer

	Initials
Purge sample from cell using ambient air or nitrogen	
Record Nitrogen Background	
Purge cell with dry nitrogen	TG
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
Evacuate Cell	
Fill Cell with CTS gas	
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	
Record cylinder ID Number	
Record CTS gas cylinder identity and concentration	
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	
Record Barytron pressure during collect	
Record information on "Background and Calibrations" data sheet.	
Verify that spectrum and interferogram were copied to directories.	
Record CTS Spectrum File Name	CTS0707e
Close cylinders	TG
Evacuate or Purge CTS from cell using nitrogen	TG
Leave cell under low nitrogen purge or under vacuum	TG
Fill MCT detector dewar	TG

Reviewed by: T. Geyer

Date: 7/7/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/8/99

OPERATOR: T. Geyer

	Initials
Cell has been heated for ~ 4 hrs. and is still stabilizing. This Background & CTS are for stability to tomorrow's measurements	
Purge sample from cell using ambient air or nitrogen	JG
Record Nitrogen Background	
Purge cell with dry nitrogen	JG
Verify cell is as dry as previous background	no JG
Record ambient pressure using cell Barocell gauge	749.0
Record nitrogen flow rate (about sampling flow rate)	0.50 LPM
Collect Background (AQBK) under continuous flow and ambient pressure	JG
Record information in data book.	JG
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
Purge ~~Evacuate~~ Cell	JG
Fill Cell with CTS gas	JG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	0.97 LPM
Record cylinder ID Number	ALM25384
Record CTS gas cylinder identity and concentration	20 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	
Record Barytron pressure during collect	751.0 torr
Record information on "Background and Calibrations" data sheet.	JG
Verify that spectrum and interferogram were copied to directories.	
Record CTS Spectrum File Name	CTS0708B
Close cylinders	JG
Evacuate or Purge CTS from cell using nitrogen	JG
Leave cell under low nitrogen purge or under vacuum	JG
Fill MCT detector dewar	

Reviewed by: [signature]

Date: 7/8/99

Project No. 4951-12-13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/9/99

OPERATOR: T. Geyer

	Initials
Purge sample from cell using ambient air or nitrogen	
Record Nitrogen Background	
Purge cell with dry nitrogen	AG
Verify cell is as dry as previous background	AG
Record ambient pressure using cell Barocell gauge	749.1
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure 7098	
Record information in data book.	AG
Copy Background to C-drive and backup using batch file.	AG
Record CTS Spectrum	
Purge Evacuate Cell	AG
Fill Cell with CTS gas	AG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	AG
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration Also methane - see data sheet. AG	20 ppm ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	AG
Record Barytron pressure during collect	AG 751.1
Record information on "Background and Calibrations" data sheet.	AG
Verify that spectrum and interferogram were copied to directories.	AG
Record CTS Spectrum File Name	CTS0709B
Close cylinders	AG
Evacuate or Purge CTS from cell using nitrogen	AG
Leave cell under low nitrogen purge or under vacuum	AG
Fill MCT detector dewar	–

Reviewed by: [signature]

Date: 7/12/99

Project No. 4951-12-13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/12/99

OPERATOR: T. Geyer

	Initials
Purge sample from cell using ambient air or nitrogen	
Record Nitrogen Background	
Purge cell with dry nitrogen	[illegible]
Verify cell is as dry as previous background	[illegible]
Record ambient pressure using cell Barocell gauge	753.1 [illegible]
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
~~Evacuate Cell~~	
Fill Cell with CTS gas	[illegible]
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	1.10 LPM
Record cylinder ID Number	AL#25384
Record CTS gas cylinder identity and concentration	20.01ppm ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	[illegible]
Record Barytron pressure during collect	755.3
Record information on "Background and Calibrations" data sheet.	[illegible]
Verify that spectrum and interferogram were copied to directories.	[illegible]
Record CTS Spectrum File Name	CTS0712C
Close cylinders	[illegible]
Evacuate or Purge CTS from cell using nitrogen	[illegible]
Leave cell under low nitrogen purge or under vacuum	[illegible]
Fill MCT detector dewar	

used Background "B"

Reviewed by: [illegible]

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/13/99

OPERATOR: T. Geyer

	Initials
Purge sample from cell using ambient air or nitrogen	
Record Nitrogen Background	
Purge cell with dry nitrogen	
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	wd BK0713C
Record information in data book.	
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
Evacuate Cell	
Fill Cell with CTS gas	
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	
Record cylinder ID Number	ALM0 25354
Record CTS gas cylinder identity and concentration	20 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	
Record Barytron pressure during collect	754.7
Record information on "Background and Calibrations" data sheet.	
Verify that spectrum and interferogram were copied to directories.	
Record CTS Spectrum File Name	CTS0713C
Close cylinders	
Evacuate or Purge CTS from cell using nitrogen	
Leave cell under low nitrogen purge or under vacuum	
Fill MCT detector dewar	

Reviewed by: R. Gy

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/15/99

OPERATOR: T. Geyer

	Initials
Purge sample from cell using ambient air or nitrogen	
Record Nitrogen Background	
Purge cell with dry nitrogen	
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate)	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
Evacuate Cell	TJG
Fill Cell with CTS gas	TJG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	TJG
Record cylinder ID Number	ALM025384
Record CTS gas cylinder identity and concentration	20 ppm Ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	TJG
Record Barytron pressure during collect	TJG
Record information on "Background and Calibrations" data sheet.	TJG
Verify that spectrum and interferogram were copied to directories.	TJG
Record CTS Spectrum File Name	TJG CTS0715B
Close cylinders	TJG
Evacuate or Purge CTS from cell using nitrogen	TJG
Leave cell under low nitrogen purge or under vacuum	TJG
Fill MCT detector dewar	—

used BKG "B"

Reviewed by: TJG

Date: 7/16/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/16/99

OPERATOR: T. Gayer

	Initials
Purge sample from cell using ambient air or nitrogen	TG
Record Nitrogen Background	
Purge cell with dry nitrogen	
Verify cell is as dry as previous background	
Record ambient pressure using cell Barocell gauge	
Record nitrogen flow rate (about sampling flow rate) using "BK60716B"	
Collect Background (AQBK) under continuous flow and ambient pressure	
Record information in data book.	
Copy Background to C-drive and backup using batch file.	
Record CTS Spectrum	
~~Evacuate~~ purge Cell	TG
Fill Cell with CTS gas	TG
Open cell outlet and purge cell with CTS at sampling rate (1 to 5 LPM)	TG
Record cylinder ID Number	AL402538 4
Record CTS gas cylinder identity and concentration	20.0 ppm ethylene
Record and copy spectrum and interferogram to C-drive and back up using CTS batch file.	TG
Record Barytron pressure during collect	TG
Record information on "Background and Calibrations" data sheet.	TG
Verify that spectrum and interferogram were copied to directories.	TG
Record CTS Spectrum File Name	CTS0716B
Close cylinders	TG
Evacuate or Purge CTS from cell using nitrogen	TG
Leave cell under low nitrogen purge or under vacuum	TG
Fill MCT detector dewar	—

Reviewed by: T. Gayer

Date: 7/16/99

APPENDIX C

CALIBRATION GAS CERTIFICATES

Scott Specialty Gases

Shipped From:
6141 EASTON ROAD PO BOX 310
PLUMSTEADVILLE PA 18949-0310
Phone: 215-766-8861 Fax: 215-766-2070

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
TOM GEYER
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 01-88514-002
PO#: 029257
ITEM #: 01023822 1AL
DATE: 4/10/97

CYLINDER #: ALM010610
FILL PRESSURE: 1980 PSIG

ANALYTICAL ACCURACY: +-5%

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
SULFUR HEXAFLUORIDE	4.	PPM	3.89	PPM
NITROGEN		BALANCE		BALANCE

ANALYST: T. Ludwig
T. LUDWIG

Scott Specialty Gases

Shipped From: 1290 COMBERMERE STREET
TROY MI 48083
Phone: 248-589-2950 Fax: 248-589-2134

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
MELISSA TUCKER; # 026075
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 05-97268-002
PO#: 026075
ITEM #: 05023822 4A
DATE: 6/03/96

CYLINDER #: A7853
FILL PRESSURE: 2000 PSI

ANALYTICAL ACCURACY: +/- 2%
PRODUCT EXPIRATION: 6/03/1997

BLEND TYPE : CERTIFIED MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
SULFUR HEXAFLUORIDE	4.	PPM	4.01	PPM
NITROGEN		BALANCE		BALANCE

CERTIFIED MASTER GAS

ANALYST: Sarah Reister

125

Scott Specialty Gases

Shipped From: 6141 EASTON ROAD PO BOX 310
PLUMSTEADVILLE PA 18949-0310
Phone: 215-766-8861 Fax: 215-766-2070

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
TOM GEYER
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 01-88514-001
PO#: 029257
ITEM #: 01021951 1AL
DATE: 3/25/97

CYLINDER #: ALM023940
FILL PRESSURE: 2000 PSIG
BLEND TYPE : GRAVIMETRIC MASTER GAS

ANALYTICAL ACCURACY: +-1%

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
ETHYLENE	20.	PPM	20.01	PPM
NITROGEN		BALANCE		BALANCE

CERTIFIED

ANALYST: G. Kogut
GENYA KOGUT

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Scott Specialty Gases

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CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
LANCE HENNING
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 05-16958-001
PO#: 031195
ITEM #: 05021951 1A
DATE: 9/02/97

CYLINDER #: A7649
FILL PRESSURE: 2000 PSI

ANALYTICAL ACCURACY: +/- 1%
PRODUCT EXPIRATION: 9/03/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
ETHYLENE	20.	PPM	19.38	PPM
NITROGEN		BALANCE		BALANCE

GRAVIMETRIC MASTER GAS

CERTIFIED TO HAVE BEEN BLENDED AGAINST NIST TRACEABLE WEIGHTS AND VERIFIED CORRECT BY INDEPENDENT ANALYSIS.

ANALYST: Sarah Reuter

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Scott Specialty Gases

Shipped From: 6141 EASTON ROAD PO BOX 310
PLUMSTEADVILLE PA 18949-0310
Phone: 215-766-8861 Fax: 215-766-2070

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
DAVE ALBURTY, X1525
425 VOLKER BLVD

KANSAS CITY MO 64110

PROJECT #: 01-89796-004
PO#: 029872
ITEM #: 01023912 4AL
DATE: 5/13/97

CYLINDER #: ALM052730 ANALYTICAL ACCURACY: +/- 2%
FILL PRESSURE: 2000 PSIG
BLEND TYPE : CERTIFIED MASTER GAS

COMPONENT	REQUESTED GAS CONC MOLES		ANALYSIS (MOLES)	
TOLUENE	60.	PPM	60.6	PPM
AIR		BALANCE		BALANCE

CERTIFIED

ANALYST: G. Kogut
GENYA KOGUT

APPENDIX D

TEST METHODS

D-1 EPA METHOD 320

Appendix A of part 63 is amended by adding, in numerical order, Methods 320 and 321 to read as follows:

Appendix A to Part 63-Test Methods

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TEST METHOD 320

MEASUREMENT OF VAPOR PHASE ORGANIC AND INORGANIC EMISSIONS BY EXTRACTIVE FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

1.0 Introduction.

Persons unfamiliar with basic elements of FTIR spectroscopy should not attempt to use this method. This method describes sampling and analytical procedures for extractive emission measurements using Fourier transform infrared (FTIR) spectroscopy. Detailed analytical procedures for interpreting infrared spectra are described in the "Protocol for the Use of Extractive Fourier Transform Infrared (FTIR) Spectrometry in Analyses of Gaseous Emissions from Stationary Sources," hereafter referred to as the "Protocol." Definitions not given in this method are given in appendix A of the Protocol. References to specific sections in the Protocol are made throughout this Method. For additional information refer to references 1 and 2, and other EPA reports, which describe the use of FTIR spectrometry in specific field measurement applications and validation tests. The sampling procedure described here is

extractive. Flue gas is extracted through a heated gas transport and handling system. For some sources, sample conditioning systems may be applicable. Some examples are given in this method. Note: sample conditioning systems may be used providing the method validation requirements in Sections 9.2 and 13.0 of this method are met.

1.1 Scope and Applicability.

1.1.1 Analytes. Analytes include hazardous air pollutants (HAPs) for which EPA reference spectra have been developed. Other compounds can also be measured with this method if reference spectra are prepared according to section 4.6 of the protocol.

1.1.2 Applicability. This method applies to the analysis of vapor phase organic or inorganic compounds which absorb energy in the mid-infrared spectral region, about 400 to 4000 cm^{-1} (25 to 2.5 µm). This method is used to determine compound-specific concentrations in a multi-component vapor phase sample, which is contained in a closed-path gas cell. Spectra of samples are collected using double beam infrared absorption spectroscopy. A computer program is used to analyze spectra and report compound concentrations.

1.2 Method Range and Sensitivity. Analytical range and sensitivity depend on the frequency-dependent analyte absorptivity, instrument configuration, data collection parameters, and gas stream composition. Instrument factors

include: (a) spectral resolution, (b) interferometer signal averaging time, (c) detector sensitivity and response, and (d) absorption path length.

1.2.1 For any optical configuration the analytical range is between the absorbance values of about .01 (infrared transmittance relative to the background = 0.98) and 1.0 (T = 0.1). (For absorbance > 1.0 the relation between absorbance and concentration may not be linear.)

1.2.2 The concentrations associated with this absorbance range depend primarily on the cell path length and the sample temperature. An analyte absorbance greater than 1.0, can be lowered by decreasing the optical path length. Analyte absorbance increases with a longer path length. Analyte detection also depends on the presence of other species exhibiting absorbance in the same analytical region. Additionally, the estimated lower absorbance (A) limit (A = 0.01) depends on the root mean square deviation (RMSD) noise in the analytical region.

1.2.3 The concentration range of this method is determined by the choice of optical configuration.

1.2.3.1 The absorbance for a given concentration can be decreased by decreasing the path length or by diluting the sample. There is no practical upper limit to the measurement range.

1.2.3.2 The analyte absorbance for a given concentration

may be increased by increasing the cell path length or (to some extent) using a higher resolution. Both modifications also cause a corresponding increased absorbance for all compounds in the sample, and a decrease in the signal throughput. For this reason the practical lower detection range (quantitation limit) usually depends on sample characteristics such as moisture content of the gas, the presence of other interferants, and losses in the sampling system.

1.3 Sensitivity. The limit of sensitivity for an optical configuration and integration time is determined using appendix D of the Protocol: Minimum Analyte Uncertainty, (MAU). The MAU depends on the RMSD noise in an analytical region, and on the absorptivity of the analyte in the same region.

1.4 Data Quality. Data quality shall be determined by executing Protocol pre-test procedures in appendices B to H of the protocol and post-test procedures in appendices I and J of the protocol.

1.4.1 Measurement objectives shall be established by the choice of detection limit (DL_i) and analytical uncertainty (AU_i) for each analyte.

1.4.2 An instrumental configuration shall be selected. An estimate of gas composition shall be made based on previous test data, data from a similar source or information

gathered in a pre-test site survey. Spectral interferants shall be identified using the selected DL_i and AU_i and band areas from reference spectra and interferant spectra. The baseline noise of the system shall be measured in each analytical region to determine the MAU of the instrument configuration for each analyte and interferant (MIU_i).

1.4.3 Data quality for the application shall be determined, in part, by measuring the RMS (root mean square) noise level in each analytical spectral region (appendix C of the Protocol). The RMS noise is defined as the RMSD of the absorbance values in an analytical region from the mean absorbance value in the region.

1.4.4 The MAU is the minimum analyte concentration for which the AU_i can be maintained; if the measured analyte concentration is less than MAU_i, then data quality are unacceptable.

2.0 Summary of Method.

2.1 Principle. References 4 through 7 provide background material on infrared spectroscopy and quantitative analysis. A summary is given in this section.

2.1.1 Infrared absorption spectroscopy is performed by directing an infrared beam through a sample to a detector. The frequency-dependent infrared absorbance of the sample is measured by comparing this detector signal (single beam spectrum) to a signal obtained without a sample in the beam

path (background).

2.1.2 Most molecules absorb infrared radiation and the absorbance occurs in a characteristic and reproducible pattern. The infrared spectrum measures fundamental molecular properties and a compound can be identified from its infrared spectrum alone.

2.1.3 Within constraints, there is a linear relationship between infrared absorption and compound concentration. If this frequency dependent relationship (absorptivity) is known (measured), it can be used to determine compound concentration in a sample mixture.

2.1.4 Absorptivity is measured by preparing, in the laboratory, standard samples of compounds at known concentrations and measuring the FTIR "reference spectra" of these standard samples. These "reference spectra" are then used in sample analysis: (1) compounds are detected by matching sample absorbance bands with bands in reference spectra, and (2) concentrations are measured by comparing sample band intensities with reference band intensities.

2.1.5 This method is self-validating provided that the results meet the performance requirement of the QA spike in sections 8.6.2 and 9.0 of this method, and results from a previous method validation study support the use of this method in the application.

2.2 Sampling and Analysis. In extractive sampling a probe

assembly and pump are used to extract gas from the exhaust of the affected source and transport the sample to the FTIR gas cell. Typically, the sampling apparatus is similar to that used for single-component continuous emission monitor (CEM) measurements.

2.2.1 The digitized infrared spectrum of the sample in the FTIR gas cell is measured and stored on a computer. Absorbance band intensities in the spectrum are related to sample concentrations by what is commonly referred to as Beer's Law.

$$A_i = a_i \, b \, c_i \qquad \textbf{(1)}$$

where:

A_i = absorbance at a given frequency of the ith sample component.

a_i = absorption coefficient (absorptivity) of the ith sample component.

b = path length of the cell.

c_i = concentration of the ith sample component.

2.2.2 Analyte spiking is used for quality assurance (QA). In this procedure (section 8.6.2 of this method) an analyte is spiked into the gas stream at the back end of the sample probe. Analyte concentrations in the spiked samples are compared to analyte concentrations in unspiked samples.

Since the concentration of the spike is known, this procedure can be used to determine if the sampling system is removing the spiked analyte(s) from the sample stream.

2.3 Reference Spectra Availability. Reference spectra of over 100 HAPs are available in the EPA FTIR spectral library on the EMTIC (Emission Measurement Technical Information Center) computer bulletin board service and at internet address http://info.arnold.af.mil/epa/welcome.htm. Reference spectra for HAPs, or other analytes, may also be prepared according to section 4.6 of the Protocol.

2.4 Operator Requirements. The FTIR analyst shall be trained in setting up the instrumentation, verifying the instrument is functioning properly, and performing routine maintenance. The analyst must evaluate the initial sample spectra to determine if the sample matrix is consistent with pre-test assumptions and if the instrument configuration is suitable. The analyst must be able to modify the instrument configuration, if necessary.

2.4.1 The spectral analysis shall be supervised by someone familiar with EPA FTIR Protocol procedures.

2.4.2 A technician trained in instrumental test methods is qualified to install and operate the sampling system. This includes installing the probe and heated line assembly, operating the analyte spike system, and performing moisture and flow measurements.

3.0 Definitions.

See appendix A of the Protocol for definitions relating to infrared spectroscopy. Additional definitions are given in sections 3.1 through 3.29.

3.1 Analyte. A compound that this method is used to measure. The term "target analyte" is also used. This method is multi-component and a number of analytes can be targeted for a test.

3.2 Reference Spectrum. Infrared spectrum of an analyte prepared under controlled, documented, and reproducible laboratory conditions according to procedures in section 4.6 of the Protocol. A library of reference spectra is used to measure analytes in gas samples.

3.3 Standard Spectrum. A spectrum that has been prepared from a reference spectrum through a (documented) mathematical operation. A common example is de-resolving of reference spectra to lower-resolution standard spectra (Protocol, appendix K to the addendum of this method). Standard spectra, prepared by approved, and documented, procedures can be used as reference spectra for analysis.

3.4 Concentration. In this method concentration is expressed as a molar concentration, in ppm-meters, or in (ppm-meters)/K, where K is the absolute temperature (Kelvin). The latter units allow the direct comparison of concentrations from systems using different optical

configurations or sampling temperatures.

3.5 Interferant. A compound in the sample matrix whose infrared spectrum overlaps with part of an analyte spectrum. The most accurate analyte measurements are achieved when reference spectra of interferants are used in the quantitative analysis with the analyte reference spectra. The presence of an interferant can increase the analytical uncertainty in the measured analyte concentration.

3.6 Gas Cell. A gas containment cell that can be evacuated. It is equipped with the optical components to pass the infrared beam through the sample to the detector. Important cell features include: path length (or range if variable), temperature range, materials of construction, and total gas volume.

3.7 Sampling System. Equipment used to extract the sample from the test location and transport the sample gas to the FTIR analyzer. This includes sample conditioning systems.

3.8 Sample Analysis. The process of interpreting the infrared spectra to obtain sample analyte concentrations. This process is usually automated using a software routine employing a classical least squares (cls), partial least squares (pls), or K- or P- matrix method.

3.9 One hundred percent line. A double beam transmittance spectrum obtained by combining two background single beam spectra. Ideally, this line is equal to 100 percent

transmittance (or zero absorbance) at every frequency in the spectrum. Practically, a zero absorbance line is used to measure the baseline noise in the spectrum.

3.10 Background Deviation. A deviation from 100 percent transmittance in any region of the 100 percent line. Deviations greater than ± 5 percent in an analytical region are unacceptable (absorbance of 0.021 to -0.022). Such deviations indicate a change in the instrument throughput relative to the background single beam.

3.11 Batch Sampling. A procedure where spectra of discreet, static samples are collected. The gas cell is filled with sample and the cell is isolated. The spectrum is collected. Finally, the cell is evacuated to prepare for the next sample.

3.12 Continuous Sampling. A procedure where spectra are collected while sample gas is flowing through the cell at a measured rate.

3.13 Sampling resolution. The spectral resolution used to collect sample spectra.

3.14 Truncation. Limiting the number of interferogram data points by deleting points farthest from the center burst (zero path difference, ZPD).

3.15 Zero filling. The addition of points to the interferogram. The position of each added point is interpolated from neighboring real data points. Zero

filling adds no information to the interferogram, but affects line shapes in the absorbance spectrum (and possibly analytical results).

3.16 Reference CTS. Calibration Transfer Standard spectra that were collected with reference spectra.

3.17 CTS Standard. CTS spectrum produced by applying a de-resolution procedure to a reference CTS.

3.18 Test CTS. CTS spectra collected at the sampling resolution using the same optical configuration as for sample spectra. Test spectra help verify the resolution, temperature and path length of the FTIR system.

3.19 RMSD. Root Mean Square Difference, defined in EPA FTIR Protocol, appendix A.

3.20 Sensitivity. The noise-limited compound-dependent detection limit for the FTIR system configuration. This is estimated by the MAU. It depends on the RMSD in an analytical region of a zero absorbance line.

3.21 Quantitation Limit. The lower limit of detection for the FTIR system configuration in the sample spectra. This is estimated by mathematically subtracting scaled reference spectra of analytes and interferences from sample spectra, then measuring the RMSD in an analytical region of the subtracted spectrum. Since the noise in subtracted sample spectra may be much greater than in a zero absorbance spectrum, the quantitation limit is generally much higher

than the sensitivity. Removing spectral interferences from the sample or improving the spectral subtraction can lower the quantitation limit toward (but not below) the sensitivity.

3.22 Independent Sample. A unique volume of sample gas; there is no mixing of gas between two consecutive independent samples. In continuous sampling two independent samples are separated by at least 5 cell volumes. The interval between independent measurements depends on the cell volume and the sample flow rate (through the cell).

3.23 Measurement. A single spectrum of flue gas contained in the FTIR cell.

3.24 Run. A run consists of a series of measurements. At a minimum a run includes 8 independent measurements spaced over 1 hour.

3.25 Validation. Validation of FTIR measurements is described in sections 13.0 through 13.4 of this method. Validation is used to verify the test procedures for measuring specific analytes at a source. Validation provides proof that the method works under certain test conditions.

3.26 Validation Run. A validation run consists of at least 24 measurements of independent samples. Half of the samples are spiked and half are not spiked. The length of the run is determined by the interval between independent samples.

3.27 Screening. Screening is used when there is little or no available information about a source. The purpose of screening is to determine what analytes are emitted and to obtain information about important sample characteristics such as moisture, temperature, and interferences. Screening results are semi-quantitative (estimated concentrations) or qualitative (identification only). Various optical and sampling configurations may be used. Sample conditioning systems may be evaluated for their effectiveness in removing interferences. It is unnecessary to perform a complete run under any set of sampling conditions. Spiking is not necessary, but spiking can be a useful screening tool for evaluating the sampling system, especially if a reactive or soluble analyte is used for the spike.

3.28 Emissions Test. An FTIR emissions test is performed according specific sampling and analytical procedures. These procedures, for the target analytes and the source, are based on previous screening and validation results. Emission results are quantitative. A QA spike (sections 8.6.2 and 9.2 of this method) is performed under each set of sampling conditions using a representative analyte. Flow, gas temperature and diluent data are recorded concurrently with the FTIR measurements to provide mass emission rates for detected compounds.

3.29 Surrogate. A surrogate is a compound that is used in

a QA spike procedure (section 8.6.2 of this method) to represent other compounds. The chemical and physical properties of a surrogate shall be similar to the compounds it is chosen to represent. Under given sampling conditions, usually a single sampling factor is of primary concern for measuring the target analytes: for example, the surrogate spike results can be representative for analytes that are more reactive, more soluble, have a lower absorptivity, or have a lower vapor pressure than the surrogate itself.

4.0 Interferences.

Interferences are divided into two classifications: analytical and sampling.

4.1 Analytical Interferences. An analytical interference is a spectral feature that complicates (in extreme cases may prevent) the analysis of an analyte. Analytical interferences are classified as background or spectral interference.

4.1.1 Background Interference. This results from a change in throughput relative to the single beam background. It is corrected by collecting a new background and proceeding with the test. In severe instances the cause must be identified and corrected. Potential causes include: (1) deposits on reflective surfaces or transmitting windows, (2) changes in detector sensitivity, (3) a change in the infrared source output, or (4) failure in the instrument electronics. In

routine sampling throughput may degrade over several hours. Periodically a new background must be collected, but no other corrective action will be required.

4.1.2 Spectral Interference. This results from the presence of interfering compound(s) (interferant) in the sample. Interferant spectral features overlap analyte spectral features. Any compound with an infrared spectrum, including analytes, can potentially be an interferant. The Protocol measures absorbance band overlap in each analytical region to determine if potential interferants shall be classified as known interferants (FTIR Protocol, section 4.9 and appendix B). Water vapor and CO_2 are common spectral interferants. Both of these compounds have strong infrared spectra and are present in many sample matrices at high concentrations relative to analytes. The extent of interference depends on the (1) interferant concentration, (2) analyte concentration, and (3) the degree of band overlap. Choosing an alternate analytical region can minimize or avoid the spectral interference. For example, CO_2 interferes with the analysis of the 670 cm^{-1} benzene band. However, benzene can also be measured near 3000 cm^{-1} (with less sensitivity).

4.2 Sampling System Interferences. These prevent analytes from reaching the instrument. The analyte spike procedure is designed to measure sampling system interference, if any.

4.2.1 Temperature. A temperature that is too low causes condensation of analytes or water vapor. The materials of the sampling system and the FTIR gas cell usually set the upper limit of temperature.

4.2.2 Reactive Species. Anything that reacts with analytes. Some analytes, like formaldehyde, polymerize at lower temperatures.

4.2.3 Materials. Poor choice of material for probe, or sampling line may remove some analytes. For example, HF reacts with glass components.

4.2.4 Moisture. In addition to being a spectral interferant, condensed moisture removes soluble compounds.

5.0 Safety.

The hazards of performing this method are those associated with any stack sampling method and the same precautions shall be followed. Many HAPs are suspected carcinogens or present other serious health risks. Exposure to these compounds should be avoided in all circumstances. For instructions on the safe handling of any particular compound, refer to its material safety data sheet. When using analyte standards, always ensure that gases are properly vented and that the gas handling system is leak free. (Always perform a leak check with the system under maximum vacuum and, again, with the system at greater than ambient pressure.) Refer to section 8.2 of this method for

leak check procedures. This method does not address all of the potential safety risks associated with its use. Anyone performing this method must follow safety and health practices consistent with applicable legal requirements and with prudent practice for each application.

6.0 Equipment and Supplies.

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

The equipment and supplies are based on the schematic of a sampling system shown in Figure 1. Either the batch or continuous sampling procedures may be used with this sampling system. Alternative sampling configurations may also be used, provided that the data quality objectives are met as determined in the post-analysis evaluation. Other equipment or supplies may be necessary, depending on the design of the sampling system or the specific target analytes.

6.1 Sampling Probe. Glass, stainless steel, or other appropriate material of sufficient length and physical integrity to sustain heating, prevent adsorption of analytes, and to transport analytes to the infrared gas cell. Special materials or configurations may be required in some applications. For instance, high stack sample temperatures may require special steel or cooling the probe.

For very high moisture sources it may be desirable to use a dilution probe.

6.2 Particulate Filters. A glass wool plug (optional) inserted at the probe tip (for large particulate removal) and a filter (required) rated for 99 percent removal efficiency at 1-micron (e.g., Balston™) connected at the outlet of the heated probe.

6.3 Sampling Line/Heating System. Heated (sufficient to prevent condensation) stainless steel, polytetrafluoroethane, or other material inert to the analytes.

6.4 Gas Distribution Manifold. A heated manifold allowing the operator to control flows of gas standards and samples directly to the FTIR system or through sample conditioning systems. Usually includes heated flow meter, heated valve for selecting and sending sample to the analyzer, and a by-pass vent. This is typically constructed of stainless steel tubing and fittings, and high-temperature valves.

6.5 Stainless Steel Tubing. Type 316, appropriate diameter (e.g., 3/8 in.) and length for heated connections. Higher grade stainless may be desirable in some applications.

6.6 Calibration/Analyte Spike Assembly. A three way valve assembly (or equivalent) to introduce analyte or surrogate spikes into the sampling system at the outlet of the probe upstream of the out-of-stack particulate filter and the FTIR

analytical system.

6.7 Mass Flow Meter (MFM). These are used for measuring analyte spike flow. The MFM shall be calibrated in the range of 0 to 5 L/min and be accurate to ± 2 percent (or better) of the flow meter span.

6.8 Gas Regulators. Appropriate for individual gas standards.

6.9 Polytetrafluoroethane Tubing. Diameter (e.g., 3/8 in.) and length suitable to connect cylinder regulators to gas standard manifold.

6.10 Sample Pump. A leak-free pump (e.g., KNF™), with by-pass valve, capable of producing a sample flow rate of at least 10 L/min through 100 ft of sample line. If the pump is positioned upstream of the distribution manifold and FTIR system, use a heated pump that is constructed from materials non-reactive to the analytes. If the pump is located downstream of the FTIR system, the gas cell sample pressure will be lower than ambient pressure and it must be recorded at regular intervals.

6.11 Gas Sample Manifold. Secondary manifold to control sample flow at the inlet to the FTIR manifold. This is optional, but includes a by-pass vent and heated rotameter.

6.12 Rotameter. A 0 to 20 L/min rotameter. This meter need not be calibrated.

6.13 FTIR Analytical System. Spectrometer and detector,

capable of measuring the analytes to the chosen detection limit. The system shall include a personal computer with compatible software allowing automated collection of spectra.

6.14 FTIR Cell Pump. Required for the batch sampling technique, capable of evacuating the FTIR cell volume within 2 minutes. The pumping speed shall allow the operator to obtain 8 sample spectra in 1 hour.

6.15 Absolute Pressure Gauge. Capable of measuring pressure from 0 to 1000 mmHg to within ± 2.5 mmHg (e.g., Baratron™).

6.16 Temperature Gauge. Capable of measuring the cell temperature to within ± 2°C.

6.17 Sample Conditioning. One option is a condenser system, which is used for moisture removal. This can be helpful in the measurement of some analytes. Other sample conditioning procedures may be devised for the removal of moisture or other interfering species.

6.17.1 The analyte spike procedure of section 9.2 of this method, the QA spike procedure of section 8.6.2 of this method, and the validation procedure of section 13 of this method demonstrate whether the sample conditioning affects analyte concentrations. Alternatively, measurements can be made with two parallel FTIR systems; one measuring conditioned sample, the other measuring unconditioned

sample.

6.17.2 Another option is sample dilution. The dilution factor measurement must be documented and accounted for in the reported concentrations. An alternative to dilution is to lower the sensitivity of the FTIR system by decreasing the cell path length, or to use a short-path cell in conjunction with a long path cell to measure more than one concentration range.

7.0 Reagents and Standards.

7.1 Analyte(s) and Tracer Gas. Obtain a certified gas cylinder mixture containing all of the analyte(s) at concentrations within ± 2 percent of the emission source levels (expressed in ppm-meter/K). If practical, the analyte standard cylinder shall also contain the tracer gas at a concentration which gives a measurable absorbance at a dilution factor of at least 10:1. Two ppm SF_6 is sufficient for a path length of 22 meters at 250 °F.

7.2 Calibration Transfer Standard(s). Select the calibration transfer standards (CTS) according to section 4.5 of the FTIR Protocol. Obtain a National Institute of Standards and Technology (NIST) traceable gravimetric standard of the CTS (± 2 percent).

7.3 Reference Spectra. Obtain reference spectra for each analyte, interferant, surrogate, CTS, and tracer. If EPA reference spectra are not available, use reference spectra

prepared according to procedures in section 4.6 of the EPA FTIR Protocol.

8.0 Sampling and Analysis Procedure.

Three types of testing can be performed: (1) screening, (2) emissions test, and (3) validation. Each is defined in section 3 of this method. Determine the purpose(s) of the FTIR test. Test requirements include: (a) AU_i, DL_i, overall fractional uncertainty, OFU_i, maximum expected concentration ($CMAX_i$), and t_{AN} for each, (b) potential interferants, (c) sampling system factors, e.g., minimum absolute cell pressure, (P_{min}), FTIR cell volume (V_{SS}), estimated sample absorption pathlength, L_S', estimated sample pressure, P_S', T_S', signal integration time (t_{SS}), minimum instrumental linewidth, MIL, fractional error, and (d) analytical regions, e.g., m = 1 to M, lower wavenumber position, FL_m, center wavenumber position, FC_m, and upper wavenumber position, FU_m, plus interferants, upper wavenumber position of the CTS absorption band, FFU_m, lower wavenumber position of the CTS absorption band, FFL_m, wavenumber range FNU to FNL. If necessary, sample and acquire an initial spectrum. From analysis of this preliminary spectrum determine a suitable operational path length. Set up the sampling train as shown in Figure 1 or use an appropriate alternative configuration. Sections 8.1 through 8.11 of this method provide guidance on pre-test calculations in the EPA

protocol, sampling and analytical procedures, and post-test protocol calculations.

8.1 Pretest Preparations and Evaluations. Using the procedure in section 4.0 of the FTIR Protocol, determine the optimum sampling system configuration for measuring the target analytes. Use available information to make reasonable assumptions about moisture content and other interferences.

8.1.1 Analytes. Select the required detection limit (DL_i) and the maximum permissible analytical uncertainty (AU_i) for each analyte (labeled from 1 to i). Estimate, if possible, the maximum expected concentration for each analyte, $CMAX_i$. The expected measurement range is fixed by DL_i and $CMAX_i$ for each analyte (i).

8.1.2 Potential Interferants. List the potential interferants. This usually includes water vapor and CO_2, but may also include some analytes and other compounds.

8.1.3. Optical Configuration. Choose an optical configuration that can measure all of the analytes within the absorbance range of .01 to 1.0 (this may require more than one path length). Use Protocol sections 4.3 to 4.8 for guidance in choosing a configuration and measuring CTS.

8.1.4. Fractional Reproducibility Uncertainty (FRU_i). The FRU is determined for each analyte by comparing CTS spectra taken before and after the reference spectra were measured.

The EPA para-xylene reference spectra were collected on 10/31/91 and 11/01/91 with corresponding CTS spectra "cts1031a," and "cts1101b." The CTS spectra are used to estimate the reproducibility (FRU) in the system that was used to collect the references. The FRU must be < AU. Appendix E of the protocol is used to calculate the FRU from CTS spectra. Figure 2 plots results for 0.25 cm^{-1} CTS spectra in EPA reference library: S_3 (cts1101b - cts1031a), and S_4 [(cts1101b + cts1031a)/2]. The RMSD (SRMS) is calculated in the subtracted baseline, S_3, in the corresponding CTS region from 850 to 1065 cm^{-1}. The area (BAV) is calculated in the same region of the averaged CTS spectrum, S_4.

8.1.5 Known Interferants. Use appendix B of the EPA FTIR Protocol.

8.1.6 Calculate the Minimum Analyte Uncertainty, MAU (section 1.3 of this method discusses MAU and protocol appendix D gives the MAU procedure). The MAU for each analyte, i, and each analytical region, m, depends on the RMS noise.

8.1.7 Analytical Program. See FTIR Protocol, section 4.10. Prepare computer program based on the chosen analytical technique. Use as input reference spectra of all target analytes and expected interferants. Reference spectra of additional compounds shall also be included in the program

if their presence (even if transient) in the samples is considered possible. The program output shall be in ppm (or ppb) and shall be corrected for differences between the reference path length, L_R, temperature, T_R, and pressure, P_R, and the conditions used for collecting the sample spectra. If sampling is performed at ambient pressure, then any pressure correction is usually small relative to corrections for path length and temperature, and may be neglected.

8.2 Leak-check.

8.2.1 Sampling System. A typical FTIR extractive sampling train is shown in Figure 1. Leak check from the probe tip to pump outlet as follows: Connect a 0- to 250-mL/min rate meter (rotameter or bubble meter) to the outlet of the pump. Close off the inlet to the probe, and record the leak rate. The leak rate shall be ≤ 200 mL/min.

8.2.2 Analytical System Leak check. Leak check the FTIR cell under vacuum and under pressure (greater than ambient). Leak check connecting tubing and inlet manifold under pressure.

8.2.2.1 For the evacuated sample technique, close the valve to the FTIR cell, and evacuate the absorption cell to the minimum absolute pressure P_{min}. Close the valve to the pump, and determine the change in pressure ΔP_v after 2 minutes.

8.2.2.2 For both the evacuated sample and purging techniques, pressurize the system to about 100 mmHg above

atmospheric pressure. Isolate the pump and determine the change in pressure ΔP_p after 2 minutes.

8.2.2.3 Measure the barometric pressure, P_b in mmHg.

8.2.2.4 Determine the percent leak volume $\%V_L$ for the signal integration time t_{ss} and for ΔP_{max}, i.e., the larger of ΔP_v or ΔP_p, as follows:

$$\%V_L = 50\, t_{SS} \frac{\Delta P_{max}}{P_{SS}} \qquad \textbf{(2)}$$

where 50 = 100% divided by the leak-check time of 2 minutes.

8.2.2.5 Leak volumes in excess of 4 percent of the FTIR system volume V_{ss} are unacceptable.

8.3 Detector Linearity. Once an optical configuration is chosen, use one of the procedures of sections 8.3.1 through 8.3.3 to verify that the detector response is linear. If the detector response is not linear, decrease the aperture, or attenuate the infrared beam. After a change in the instrument configuration perform a linearity check until it is demonstrated that the detector response is linear.

8.3.1 Vary the power incident on the detector by modifying the aperture setting. Measure the background and CTS at three instrument aperture settings: (1) at the aperture setting to be used in the testing, (2) at one half this aperture and (3) at twice the proposed testing aperture.

Compare the three CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system. If test aperture is the maximum aperture, collect CTS spectrum at maximum aperture, then close the aperture to reduce the IR throughput by half. Collect a second background and CTS at the smaller aperture setting and compare the spectra again.

8.3.2 Use neutral density filters to attenuate the infrared beam. Set up the FTIR system as it will be used in the test measurements. Collect a CTS spectrum. Use a neutral density filter to attenuate the infrared beam (either immediately after the source or the interferometer) to approximately 1/2 its original intensity. Collect a second CTS spectrum. Use another filter to attenuate the infrared beam to approximately 1/4 its original intensity. Collect a third background and CTS spectrum. Compare the CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system.

8.3.3 Observe the single beam instrument response in a frequency region where the detector response is known to be zero. Verify that the detector response is "flat" and equal to zero in these regions.

8.4 Data Storage Requirements. All field test spectra shall be stored on a computer disk and a second backup copy must stored on a separate disk. The stored information

includes sample interferograms, processed absorbance spectra, background interferograms, CTS sample interferograms and CTS absorbance spectra. Additionally, documentation of all sample conditions, instrument settings, and test records must be recorded on hard copy or on computer medium. Table 1 gives a sample presentation of documentation.

8.5 Background Spectrum. Evacuate the gas cell to ≤ 5 mmHg, and fill with dry nitrogen gas to ambient pressure (or purge the cell with 10 volumes of dry nitrogen). Verify that no significant amounts of absorbing species (for example water vapor and CO_2) are present. Collect a background spectrum, using a signal averaging period equal to or greater than the averaging period for the sample spectra. Assign a unique file name to the background spectrum. Store two copies of the background interferogram and processed single-beam spectrum on separate computer disks (one copy is the back-up).

8.5.1 Interference Spectra. If possible, collect spectra of known and suspected major interferences using the same optical system that will be used in the field measurements. This can be done on-site or earlier. A number of gases, e.g. CO_2, SO_2, CO, NH_3, are readily available from cylinder gas suppliers.

8.5.2 Water vapor spectra can be prepared by the following

procedure. Fill a sample tube with distilled water. Evacuate above the sample and remove dissolved gasses by alternately freezing and thawing the water while evacuating. Allow water vapor into the FTIR cell, then dilute to atmospheric pressure with nitrogen or dry air. If quantitative water spectra are required, follow the reference spectrum procedure for neat samples (protocol, section 4.6). Often, interference spectra need not be quantitative, but for best results the absorbance must be comparable to the interference absorbance in the sample spectra.

8.6 Pre-Test Calibrations

8.6.1 Calibration Transfer Standard. Evacuate the gas cell to ≤ 5 mmHg absolute pressure, and fill the FTIR cell to atmospheric pressure with the CTS gas. Alternatively, purge the cell with 10 cell volumes of CTS gas. (If purge is used, verify that the CTS concentration in the cell is stable by collecting two spectra 2 minutes apart as the CTS gas continues to flow. If the absorbance in the second spectrum is no greater than in the first, within the uncertainty of the gas standard, then this can be used as the CTS spectrum.) Record the spectrum.

8.6.2 QA Spike. This procedure assumes that the method has been validated for at least some of the target analytes at the source. For emissions testing perform a QA spike. Use

a certified standard, if possible, of an analyte, which has been validated at the source. One analyte standard can serve as a QA surrogate for other analytes which are less reactive or less soluble than the standard. Perform the spike procedure of section 9.2 of this method. Record spectra of at least three independent (section 3.22 of this method) spiked samples. Calculate the spiked component of the analyte concentration. If the average spiked concentration is within 0.7 to 1.3 times the expected concentration, then proceed with the testing. If applicable, apply the correction factor from the Method 301 of this appendix validation test (not the result from the QA spike).

8.7 Sampling. If analyte concentrations vary rapidly with time, continuous sampling is preferable using the smallest cell volume, fastest sampling rate and fastest spectra collection rate possible. Continuous sampling requires the least operator intervention even without an automated sampling system. For continuous monitoring at one location over long periods, Continuous sampling is preferred. Batch sampling and continuous static sampling are used for screening and performing test runs of finite duration. Either technique is preferred for sampling several locations in a matter of days. Batch sampling gives reasonably good time resolution and ensures that each spectrum measures a

discreet (and unique) sample volume. Continuous static (and continuous) sampling provide a very stable background over long periods. Like batch sampling, continuous static sampling also ensures that each spectrum measures a unique sample volume. It is essential that the leak check procedure under vacuum (section 8.2 of this method) is passed if the batch sampling procedure is used. It is essential that the leak check procedure under positive pressure is passed if the continuous static or continuous sampling procedures are used. The sampling techniques are described in sections 8.7.1 through 8.7.2 of this method.

8.7.1 Batch Sampling. Evacuate the absorbance cell to ≤ 5 mmHg absolute pressure. Fill the cell with exhaust gas to ambient pressure, isolate the cell, and record the spectrum. Before taking the next sample, evacuate the cell until no spectral evidence of sample absorption remains. Repeat this procedure to collect eight spectra of separate samples in 1 hour.

8.7.2 Continuous Static Sampling. Purge the FTIR cell with 10 cell volumes of sample gas. Isolate the cell, collect the spectrum of the static sample and record the pressure. Before measuring the next sample, purge the cell with 10 more cell volumes of sample gas.

8.8 Sampling QA and Reporting.

8.8.1 Sample integration times shall be sufficient to

achieve the required signal-to-noise ratio. Obtain an absorbance spectrum by filling the cell with N_2. Measure the RMSD in each analytical region in this absorbance spectrum. Verify that the number of scans used is sufficient to achieve the target MAU.

8.8.2 Assign a unique file name to each spectrum.

8.8.3 Store two copies of sample interferograms and processed spectra on separate computer disks.

8.8.4 For each sample spectrum, document the sampling conditions, the sampling time (while the cell was being filled), the time the spectrum was recorded, the instrumental conditions (path length, temperature, pressure, resolution, signal integration time), and the spectral file name. Keep a hard copy of these data sheets.

8.9 Signal Transmittance. While sampling, monitor the signal transmittance. If signal transmittance (relative to the background) changes by 5 percent or more (absorbance = -.02 to .02) in any analytical spectral region, obtain a new background spectrum.

8.10 Post-test CTS. After the sampling run, record another CTS spectrum.

8.11 Post-test QA.

8.11.1 Inspect the sample spectra immediately after the run to verify that the gas matrix composition was close to the expected (assumed) gas matrix.

8.11.2 Verify that the sampling and instrumental parameters were appropriate for the conditions encountered. For example, if the moisture is much greater than anticipated, it may be necessary to use a shorter path length or dilute the sample.

8.11.3 Compare the pre- and post-test CTS spectra. The peak absorbance in pre- and post-test CTS must be ± 5 percent of the mean value. See appendix E of the FTIR Protocol.

9.0 Quality Control.

Use analyte spiking (sections 8.6.2, 9.2 and 13.0 of this method) to verify that the sampling system can transport the analytes from the probe to the FTIR system.

9.1 Spike Materials. Use a certified standard (accurate to ± 2 percent) of the target analyte, if one can be obtained. If a certified standard cannot be obtained, follow the procedures in section 4.6.2.2 of the FTIR Protocol.

9.2 Spiking Procedure. QA spiking (section 8.6.2 of this method) is a calibration procedure used before testing. QA spiking involves following the spike procedure of sections 9.2.1 through 9.2.3 of this method to obtain at least three spiked samples. The analyte concentrations in the spiked samples shall be compared to the expected spike concentration to verify that the sampling/analytical system is working properly. Usually, when QA spiking is used, the

method has already been validated at a similar source for the analyte in question. The QA spike demonstrates that the validated sampling/analytical conditions are being duplicated. If the QA spike fails then the sampling/analytical system shall be repaired before testing proceeds. The method validation procedure (section 13.0 of this method) involves a more extensive use of the analyte spike procedure of sections 9.2.1 through 9.2.3 of this method. Spectra of at least 12 independent spiked and 12 independent unspiked samples are recorded. The concentration results are analyzed statistically to determine if there is a systematic bias in the method for measuring a particular analyte. If there is a systematic bias, within the limits allowed by Method 301 of this appendix, then a correction factor shall be applied to the analytical results. If the systematic bias is greater than the allowed limits, this method is not valid and cannot be used.

9.2.1 Introduce the spike/tracer gas at a constant flow rate of ≤ 10 percent of the total sample flow, when possible. (Note: Use the rotameter at the end of the sampling train to estimate the required spike/tracer gas flow rate.) Use a flow device, e.g., mass flow meter (± 2 percent), to monitor the spike flow rate. Record the spike flow rate every 10 minutes.

9.2.2 Determine the response time (RT) of the system by continuously collecting spectra of the spiked effluent until the spectrum of the spiked component is constant for 5 minutes. The RT is the interval from the first measurement until the spike becomes constant. Wait for twice the duration of the RT, then collect spectra of two independent spiked gas samples. Duplicate analyses of the spiked concentration shall be within 5 percent of the mean of the two measurements.

9.2.3 Calculate the dilution ratio using the tracer gas as follows:

$$DF = \frac{SF_{6(spk)}}{SF_{6(dir)}} \qquad \textbf{(3)}$$

where:

$$CS = DF * Spike_{dir} + Unspike(1-DF) \qquad \textbf{(4)}$$

DF = Dilution factor of the spike gas; this value shall be ≥10.

$SF_{6(dir)}$ = SF_6 (or tracer gas) concentration measured directly in undiluted spike gas.

$SF_{6(spk)}$ = Diluted SF_6 (or tracer gas) concentration measured in a spiked sample.

$Spike_{dir}$ = Concentration of the analyte in the spike standard measured by filling the FTIR cell directly.

CS = Expected concentration of the spiked samples.

Unspike = Native concentration of analytes in unspiked samples

10.0 Calibration and Standardization.

10.1 Signal-to-Noise Ratio (S/N). The RMSD in the noise must be less than one tenth of the minimum analyte peak absorbance in each analytical region. For example if the minimum peak absorbance is 0.01 at the required DL, then RMSD measured over the entire analytical region must be $\leq$ 0.001.

10.2 Absorbance Path length. Verify the absorbance path length by comparing reference CTS spectra to test CTS spectra. See appendix E of the FTIR Protocol.

10.3 Instrument Resolution. Measure the line width of appropriate test CTS band(s) to verify instrument resolution. Alternatively, compare CTS spectra to a reference CTS spectrum, if available, measured at the nominal resolution.

10.4 Apodization Function. In transforming the sample interferograms to absorbance spectra use the same

apodization function that was used in transforming the reference spectra.

10.5 FTIR Cell Volume. Evacuate the cell to ≤ 5 mmHg. Measure the initial absolute temperature (T_i) and absolute pressure (P_i). Connect a wet test meter (or a calibrated dry gas meter), and slowly draw room air into the cell. Measure the meter volume (V_m), meter absolute temperature (T_m), and meter absolute pressure (P_m); and the cell final absolute temperature (T_f) and absolute pressure (P_f). Calculate the FTIR cell volume V_{SS}, including that of the connecting tubing, as follows:

$$V_{SS} = \frac{V_m \frac{P_m}{T_m}}{\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]} \qquad \textbf{(5)}$$

11.0 Data Analysis and Calculations.

Analyte concentrations shall be measured using reference spectra from the EPA FTIR spectral library. When EPA library spectra are not available, the procedures in section 4.6 of the Protocol shall be followed to prepare reference spectra of all the target analytes.

11.1 Spectral De-resolution. Reference spectra can be converted to lower resolution standard spectra (section 3.3

of this method) by truncating the original reference sample and background interferograms. Appendix K of the FTIR Protocol gives specific deresolution procedures. Deresolved spectra shall be transformed using the same apodization function and level of zero filling as the sample spectra. Additionally, pre-test FTIR protocol calculations (e.g., FRU, MAU, FCU) shall be performed using the de-resolved standard spectra.

11.2 Data Analysis. Various analytical programs are available for relating sample absorbance to a concentration standard. Calculated concentrations shall be verified by analyzing residual baselines after mathematically subtracting scaled reference spectra from the sample spectra. A full description of the data analysis and calculations is contained in the FTIR Protocol (sections 4.0, 5.0, 6.0 and appendices). Correct the calculated concentrations in the sample spectra for differences in absorption path length and temperature between the reference and sample spectra using equation 6,

$$C_{corr} = \left(\frac{L_r}{L_s}\right)\left(\frac{T_s}{T_r}\right)\left(\frac{P_r}{P_s}\right) C_{calc} \qquad \textbf{(6)}$$

where:

C_{corr} = Concentration, corrected for path length.

C_{calc} = Concentration, initial calculation (output of the analytical program designed for the compound).

L_r = Reference spectra path length.

L_s = Sample spectra path length.

T_s = Absolute temperature of the sample gas, K.

T_r = Absolute gas temperature of reference spectra, K.

P_s = Sample cell pressure.

P_r = Reference spectrum sample pressure.

12.0 Method Performance.

12.1 Spectral Quality. Refer to the FTIR Protocol appendices for analytical requirements, evaluation of data quality, and analysis of uncertainty.

12.2 Sampling QA/QC. The analyte spike procedure of section 9 of this method, the QA spike of section 8.6.2 of this method, and the validation procedure of section 13 of this method are used to evaluate the performance of the sampling system and to quantify sampling system effects, if any, on the measured concentrations. This method is self-validating provided that the results meet the performance requirement of the QA spike in sections 9.0 and 8.6.2 of this method and results from a previous method validation study support the use of this method in the application. Several factors can contribute to uncertainty in the measurement of spiked samples. Factors which can be controlled to provide better accuracy in the spiking procedure are listed in sections 12.2.1 through 12.2.4 of this method.

12.2.1 Flow meter. An accurate mass flow meter is accurate to ± 1 percent of its span. If a flow of 1 L/min is monitored with such a MFM, which is calibrated in the range of 0-5 L/min, the flow measurement has an uncertainty of 5 percent. This may be improved by re-calibrating the meter at the specific flow rate to be used.

12.2.2 Calibration gas. Usually the calibration standard is certified to within ± 2 percent. With reactive analytes, such as HCl, the certified accuracy in a commercially available standard may be no better than ± 5 percent.

12.2.3 Temperature. Temperature measurements of the cell shall be quite accurate. If practical, it is preferable to measure sample temperature directly, by inserting a thermocouple into the cell chamber instead of monitoring the cell outer wall temperature.

12.2.4 Pressure. Accuracy depends on the accuracy of the barometer, but fluctuations in pressure throughout a day may be as much as 2.5 percent due to weather variations.

13.0 Method Validation Procedure.

This validation procedure, which is based on EPA Method 301 (40 CFR part 63, appendix A), may be used to validate this method for the analytes in a gas matrix. Validation at one source may also apply to another type of source, if it can be shown that the exhaust gas characteristics are similar at both sources.

13.1 Section 5.3 of Method 301 (40 CFR part 63, appendix A), the Analyte Spike procedure, is used with these modifications. The statistical analysis of the results follows section 6.3 of EPA Method 301. Section 3 of this method defines terms that are not defined in Method 301.

13.1.1 The analyte spike is performed dynamically. This means the spike flow is continuous and constant as spiked samples are measured.

13.1.2 The spike gas is introduced at the back of the sample probe.

13.1.3 Spiked effluent is carried through all sampling components downstream of the probe.

13.1.4 A single FTIR system (or more) may be used to collect and analyze spectra (not quadruplicate integrated sampling trains).

13.1.5 All of the validation measurements are performed sequentially in a single "run" (section 3.26 of this method).

13.1.6 The measurements analyzed statistically are each independent (section 3.22 of this method).

13.1.7 A validation data set can consist of more than 12 spiked and 12 unspiked measurements.

13.2 Batch Sampling. The procedure in sections 13.2.1 through 13.2.2 may be used for stable processes. If process emissions are highly variable, the procedure in section

13.2.3 shall be used.

13.2.1 With a single FTIR instrument and sampling system, begin by collecting spectra of two unspiked samples. Introduce the spike flow into the sampling system and allow 10 cell volumes to purge the sampling system and FTIR cell. Collect spectra of two spiked samples. Turn off the spike and allow 10 cell volumes of unspiked sample to purge the FTIR cell. Repeat this procedure until the 24 (or more) samples are collected.

13.2.2 In batch sampling, collect spectra of 24 distinct samples. (Each distinct sample consists of filling the cell to ambient pressure after the cell has been evacuated.)

13.2.3 Alternatively, a separate probe assembly, line, and sample pump can be used for spiked sample. Verify and document that sampling conditions are the same in both the spiked and the unspiked sampling systems. This can be done by wrapping both sample lines in the same heated bundle. Keep the same flow rate in both sample lines. Measure samples in sequence in pairs. After two spiked samples are measured, evacuate the FTIR cell, and turn the manifold valve so that spiked sample flows to the FTIR cell. Allow the connecting line from the manifold to the FTIR cell to purge thoroughly (the time depends on the line length and flow rate). Collect a pair of spiked samples. Repeat the procedure until at least 24 measurements are completed.

13.3 Simultaneous Measurements With Two FTIR Systems. If unspiked effluent concentrations of the target analyte(s) vary significantly with time, it may be desirable to perform synchronized measurements of spiked and unspiked sample. Use two FTIR systems, each with its own cell and sampling system to perform simultaneous spiked and unspiked measurements. The optical configurations shall be similar, if possible. The sampling configurations shall be the same. One sampling system and FTIR analyzer shall be used to measure spiked effluent. The other sampling system and FTIR analyzer shall be used to measure unspiked flue gas. Both systems shall use the same sampling procedure (i.e., batch or continuous).

13.3.1 If batch sampling is used, synchronize the cell evacuation, cell filling, and collection of spectra. Fill both cells at the same rate (in cell volumes per unit time).

13.3.2 If continuous sampling is used, adjust the sample flow through each gas cell so that the same number of cell volumes pass through each cell in a given time (i.e. $TC_1 = TC_2$).

13.4 Statistical Treatment. The statistical procedure of EPA Method 301 of this appendix, section 6.3 is used to evaluate the bias and precision. For FTIR testing a validation "run" is defined as spectra of 24 independent samples, 12 of which are spiked with the analyte(s) and 12

of which are not spiked.

13.4.1 Bias. Determine the bias (defined by EPA Method 301 of this appendix, section 6.3.2) using equation 7:

$$B = S_m - CS \tag{7}$$

where:

B = Bias at spike level.

S_m = Mean concentration of the analyte spiked samples.

CS = Expected concentration of the spiked samples.

13.4.2 Correction Factor. Use section 6.3.2.2 of Method 301 of this appendix to evaluate the statistical significance of the bias. If it is determined that the bias is significant, then use section 6.3.3 of Method 301 to calculate a correction factor (CF). Analytical results of the test method are multiplied by the correction factor, if $0.7 \leq CF \leq 1.3$. If is determined that the bias is significant and CF > ± 30 percent, then the test method is considered to "not valid."

13.4.3 If measurements do not pass validation, evaluate the sampling system, instrument configuration, and analytical system to determine if improper set-up or a malfunction was the cause. If so, repair the system and repeat the validation.

14.0 Pollution Prevention.

The extracted sample gas is vented outside the enclosure containing the FTIR system and gas manifold after the analysis. In typical method applications the vented sample volume is a small fraction of the source volumetric flow and its composition is identical to that emitted from the source. When analyte spiking is used, spiked pollutants are vented with the extracted sample gas. Approximately 1.6 x 10^{-4} to 3.2 x 10^{-4} lbs of a single HAP may be vented to the atmosphere in a typical validation run of 3 hours. (This assumes a molar mass of 50 to 100 g, spike rate of 1.0 L/min, and a standard concentration of 100 ppm). Minimize emissions by keeping the spike flow off when not in use.

15.0 Waste Management.

Small volumes of laboratory gas standards can be vented through a laboratory hood. Neat samples must be packed and disposed according to applicable regulations. Surplus materials may be returned to supplier for disposal.

16.0 References.

1. "Field Validation Test Using Fourier Transform Infrared (FTIR) Spectrometry To Measure Formaldehyde, Phenol and Methanol at a Wool Fiberglass Production Facility." Draft. U.S. Environmental Protection Agency Report, EPA Contract No. 68D20163, Work Assignment I-32, September 1994.

2. "FTIR Method Validation at a Coal-Fired Boiler".

Prepared for U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No.: EPA-454/R95-004, NTIS No.: PB95-193199. July, 1993.

3. "Method 301 - Field Validation of Pollutant Measurement Methods from Various Waste Media," **40 CFR part 63, appendix A**.

4. "Molecular Vibrations; The Theory of Infrared and Raman Vibrational Spectra," E. Bright Wilson, J. C. Decius, and P. C. Cross, Dover Publications, Inc., 1980. For a less intensive treatment of molecular rotational-vibrational spectra see, for example, "Physical Chemistry," G. M. Barrow, chapters 12, 13, and 14, McGraw Hill, Inc., 1979.

5. "Fourier Transform Infrared Spectrometry," Peter R. Griffiths and James de Haseth, **Chemical Analysis**, **83**, 16-25,(1986), P. J. Elving, J. D. Winefordner and I. M. Kolthoff (ed.), John Wiley and Sons.

6. "Computer-Assisted Quantitative Infrared Spectroscopy," Gregory L. McClure (ed.), **ASTM Special Publication 934** (ASTM), 1987.

7. "Multivariate Least-Squares Methods Applied to the Quantitative Spectral Analysis of Multicomponent Mixtures," **Applied Spectroscopy**, **39**(10), 73-84, 1985.

Table 1. EXAMPLE PRESENTATION OF SAMPLING DOCUMENTATION.

Sample Time	Spectrum File Name	Background File Name	Sample conditioning	Process condition

Sample Time	Spectrum File	Interferogram	Resolution	Scans	Apodization	Gain	CTS Spectrum

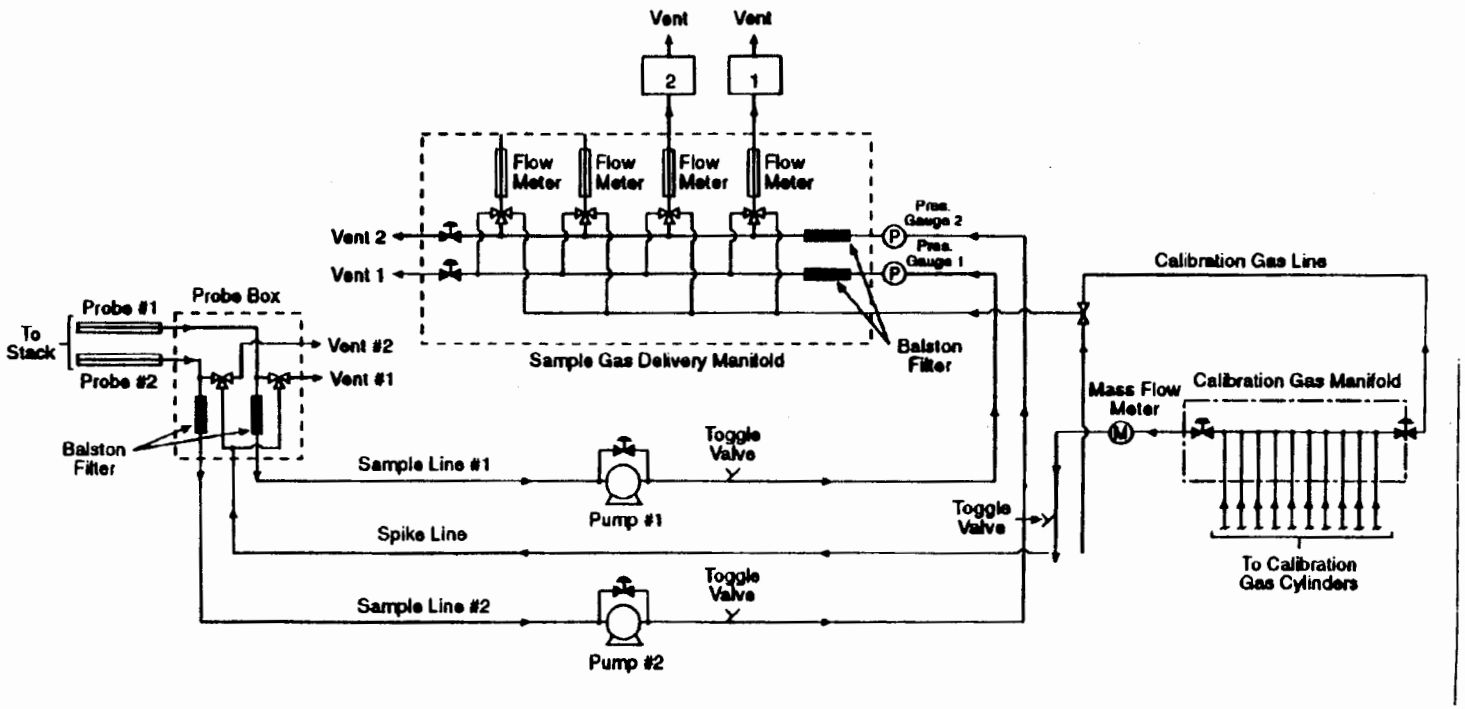

Figure 1. Extractive FTIR sampling system.

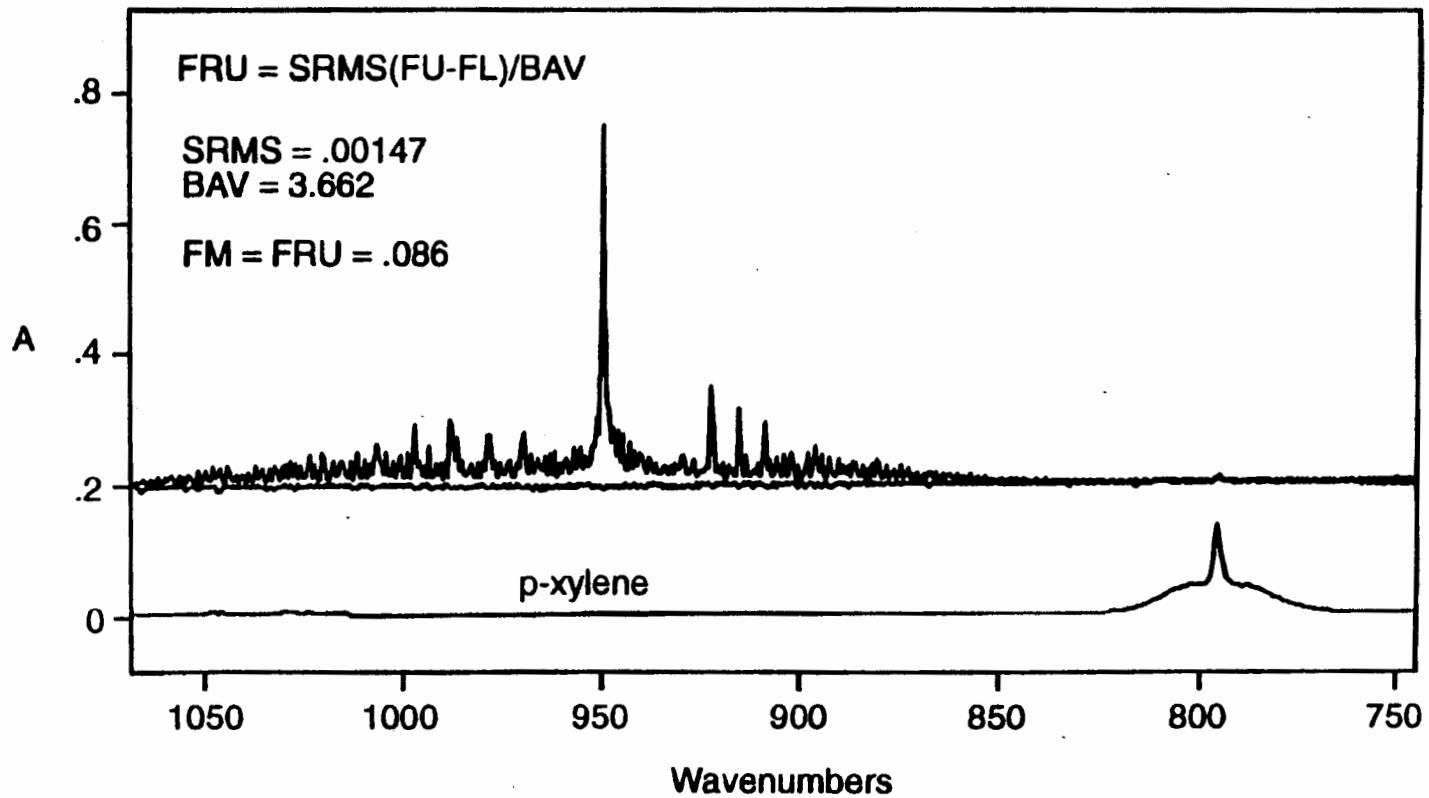

Figure 2. Fractional Reproducibility. Top: average of cts1031a and cts1101b. Bottom: Reference spectrum of p-xylene.

D-2 EPA FTIR PROTOCOL

PROTOCOL FOR THE USE OF EXTRACTIVE FOURIER TRANSFORM INFRARED (FTIR) SPECTROMETRY FOR THE ANALYSES OF GASEOUS EMISSIONS FROM STATIONARY SOURCES

INTRODUCTION

The purpose of this document is to set general guidelines for the use of modern FTIR spectroscopic methods for the analysis of gas samples extracted from the effluent of stationary emission sources. This document outlines techniques for developing and evaluating such methods and sets basic requirements for reporting and quality assurance procedures.

1.0 NOMENCLATURE

1.1 Appendix A lists definitions of the symbols and terms used in this Protocol, many of which have been taken directly from American Society for Testing and Materials (ASTM) publication E 131-90a, entitled "Terminology Relating to Molecular Spectroscopy."

1.2 Except in the case of background spectra or where otherwise noted, the term "spectrum" refers to a double-beam spectrum in units of absorbance vs. wavenumber (cm^{-1}).

1.3 The term "Study" in this document refers to a publication that has been subjected to EPA- or peer-review.

2.0 APPLICABILITY AND ANALYTICAL PRINCIPLE

2.1 Applicability. This Protocol applies to the determination of compound-specific concentrations in single- and multiple-component gas phase samples using double-beam absorption spectroscopy in the mid-infrared band. It does not specifically address other FTIR applications, such as single-beam spectroscopy, analysis of open-path (non-enclosed) samples, and continuous measurement techniques. If multiple spectrometers, absorption cells, or instrumental linewidths are used in such analyses, each distinct operational configuration of the system must be evaluated separately according to this Protocol.

2.2 Analytical Principle.

2.2.1 In the mid-infrared band, most molecules exhibit characteristic gas phase absorption spectra that may be recorded by FTIR systems. Such systems consist of a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements for the transfer of infrared radiation between components, and gas flow control and measurement components. Adjunct and integral computer systems are used for controlling

the instrument, processing the signal, and for performing both Fourier transforms and quantitative analyses of spectral data.

2.2.2 The absorption spectra of pure gases and of mixtures of gases are described by a linear absorbance theory referred to as Beer's Law. Using this law, modern FTIR systems use computerized analytical programs to quantify compounds by comparing the absorption spectra of known (reference) gas samples to the absorption spectrum of the sample gas. Some standard mathematical techniques used for comparisons are classical least squares, inverse least squares, cross-correlation, factor analysis, and partial least squares. Reference A describes several of these techniques, as well as additional techniques, such as differentiation methods, linear baseline corrections, and non-linear absorbance corrections.

3.0 GENERAL PRINCIPLES OF PROTOCOL REQUIREMENTS

The characteristics that distinguish FTIR systems from gas analyzers used in instrumental gas analysis methods (e.g., EPA Methods 6C and 7E) are: (1) Computers are necessary to obtain and analyze data; (2) chemical concentrations can be quantified using previously recorded infrared reference spectra; and (3) analytical assumptions and results, including possible effects of interfering compounds, can be evaluated after the quantitative analysis. The following general principles and requirements of this Protocol are based on these characteristics.

3.1 Verifiability and Reproducibility of Results. Store all data and document data analysis techniques sufficient to allow an independent agent to reproduce the analytical results from the raw interferometric data.

3.2 Transfer of Reference Spectra. To determine whether reference spectra recorded under one set of conditions (e.g., optical bench, instrumental linewidth, absorption pathlength, detector performance, pressure, and temperature) can be used to analyze sample spectra taken under a different set of conditions, quantitatively compare "calibration transfer standards" (CTS) and reference spectra as described in this Protocol. (Note: The CTS may, but need not, include analytes of interest). To effect this, record the absorption spectra of the CTS (a) immediately before and immediately after recording reference spectra and (b) immediately after recording sample spectra.

3.3 Evaluation of FTIR Analyses. The applicability, accuracy, and precision of FTIR measurements are influenced by a number of interrelated factors, which may be divided into two classes:

3.3.1 Sample-Independent Factors. Examples are system configuration and performance (e.g., detector sensitivity and infrared source output), quality and applicability of reference

absorption spectra, and type of mathematical analyses of the spectra. These factors define the fundamental limitations of FTIR measurements for a given system configuration. These limitations may be estimated from evaluations of the system before samples are available. For example, the detection limit for the absorbing compound under a given set of conditions may be estimated from the system noise level and the strength of a particular absorption band. Similarly, the accuracy of measurements may be estimated from the analysis of the reference spectra.

3.3.2 Sample-Dependent Factors. Examples are spectral interferants (e.g., water vapor and CO_2) or the overlap of spectral features of different compounds and contamination deposits on reflective surfaces or transmitting windows. To maximize the effectiveness of the mathematical techniques used in spectral analysis, identification of interferants (a standard initial step) and analysis of samples (includes effects of other analytical errors) are necessary. Thus, the Protocol requires post-analysis calculation of measurement concentration uncertainties for the detection of these potential sources of measurement error.

4.0 PRE-TEST PREPARATIONS AND EVALUATIONS

Before testing, demonstrate the suitability of FTIR spectrometry for the desired application according to the procedures of this section.

4.1 Identify Test Requirements. Identify and record the test requirements described below in 4.1.1 through 4.1.5. These values set the desired or required goals of the proposed analysis; the description of methods for determining whether these goals are actually met during the analysis comprises the majority of this Protocol.

4.1.1 Analytes (specific chemical species) of interest. Label the analytes from $i = 1$ to I.

4.1.2 Analytical uncertainty limit (AU_i). The AU_i is the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration in the sample.

4.1.3 Required detection limit for each analyte (DL_i, ppm). The detection limit is the lowest concentration of an analyte for which its overall fractional uncertainty (OFU_i) is required to be less than its analytical uncertainty limit (AU_i).

4.1.4 Maximum expected concentration of each analyte ($CMAX_i$, ppm).

4.2 Identify Potential Interferants. Considering the chemistry of the process or results of previous Studies, identify potential interferants, i.e., the major effluent constituents and any relatively minor effluent constituents that possess either strong absorption characteristics or strong structural similarities to any analyte of interest. Label them 1 through N_j, where the subscript "j" pertains to potential interferants. Estimate the concentrations of these compounds in the effluent ($CPOT_j$, ppm).

4.3 Select and Evaluate the Sampling System. Considering the source, e.g., temperature and pressure profiles, moisture content, analyte characteristics, and particulate concentration), select the equipment for extracting gas samples. Recommended are a particulate filter, heating system to maintain sample temperature above the dew point for all sample constituents at all points within the sampling system (including the filter), and sample conditioning system (e.g., coolers, water-permeable membranes that remove water or other compounds from the sample, and dilution devices) to remove spectral interferants or to protect the sampling and analytical components. Determine the minimum absolute sample system pressure (P_{min}, mmHg) and the infrared absorption cell volume (V_{SS}, liter). Select the techniques and/or equipment for the measurement of sample pressures and temperatures.

4.4 Select Spectroscopic System. Select a spectroscopic configuration for the application. Approximate the absorption pathlength (L_S', meter), sample pressure (P_S', kPa), absolute sample temperature T_S', and signal integration period (t_{SS}, seconds) for the analysis. Specify the nominal minimum instrumental linewidth (MIL) of the system. Verify that the fractional error at the approximate values P_S' and T_S' is less than one half the smallest value AU_i (see Section 4.1.2).

4.5 Select Calibration Transfer Standards (CTS's). Select CTS's that meet the criteria listed in Sections 4.5.1, 4.5.2, and 4.5.3.

> <u>Note</u>: It may be necessary to choose preliminary analytical regions (see Section 4.7), identify the minimum analyte linewidths, or estimate the system noise level (see Section 4.12) before selecting the CTS. More than one compound may be needed to meet the criteria; if so, obtain separate cylinders for each compound.

4.5.1 The central wavenumber position of each analytical region lies within 25 percent of the wavenumber position of at least one CTS absorption band.

4.5.2 The absorption bands in 4.5.1 exhibit peak absorbances greater than ten times the value RMS_{EST} (see Section 4.12) but less than 1.5 absorbance units.

4.5.3 At least one absorption CTS band within the operating range of the FTIR instrument has an instrument-independent linewidth no greater than the narrowest analyte absorption band; perform and document measurements or cite Studies to determine analyte and CTS compound linewidths.

4.5.4 For each analytical region, specify the upper and lower wavenumber positions (FFU_m and FFL_m, respectively) that bracket the CTS absorption band or bands for the associated analytical region. Specify the wavenumber range, FNU to FNL, containing the absorption band that meets the criterion of Section 4.5.3.

4.5.5 Associate, whenever possible, a single set of CTS gas cylinders with a set of reference spectra. Replacement CTS gas cylinders shall contain the same compounds at concentrations within 5 percent of that of the original CTS cylinders; the entire absorption spectra (not individual spectral segments) of the replacement gas shall be scaled by a factor between 0.95 and 1.05 to match the original CTS spectra.

4.6 Prepare Reference Spectra.

Note: Reference spectra are available in a permanent soft copy from the EPA spectral library on the EMTIC (Emission Measurement Technical Information Center) computer bulletin board; they may be used if applicable.

4.6.1 Select the reference absorption pathlength (L_R) of the cell.

4.6.2 Obtain or prepare a set of chemical standards for each analyte, potential and known spectral interferants, and CTS. Select the concentrations of the chemical standards to correspond to the top of the desired range.

4.6.2.1 Commercially-Prepared Chemical Standards. Chemical standards for many compounds may be obtained from independent sources, such as a specialty gas manufacturer, chemical company, or commercial laboratory. These standards (accurate to within ±2 percent) shall be prepared according to EPA Protocol 1 (see Reference D) or shall be traceable to NIST standards. Obtain from the supplier an estimate of the stability of the analyte concentration; obtain and follow all the supplier's recommendations for recertifying the analyte concentration.

4.6.2.2 Self-Prepared Chemical Standards. Chemical standards may be prepared as follows: Dilute certified commercially prepared chemical gases or pure analytes with ultra-pure carrier (UPC) grade nitrogen according to the barometric and volumetric techniques generally described in Reference A, Section A4.6.

4.6.3 Record a set of the absorption spectra of the CTS {R1}, then a set of the reference spectra at two or more concentrations in duplicate over the desired range (the top of the range must be less than 10 times that of the bottom), followed by a second set of CTS spectra {R2}. (If self-prepared standards are used, see Section 4.6.5 before disposing of any of the standards.) The maximum accepted standard concentration-pathlength product (ASCPP) for each compound shall be higher than the maximum estimated concentration-pathlength products for both analytes and known interferants in the effluent gas. For each analyte, the minimum ASCPP shall be no greater than ten times the concentration-pathlength product of that analyte at its required detection limit.

4.6.4 Permanently store the background and interferograms in digitized form. Document details of the mathematical process for generating the spectra from these interferograms. Record the sample pressure (P_R), sample temperature (T_R), reference absorption pathlength (L_R), and interferogram signal integration period (t_{SR}). Signal integration periods for the background interferograms shall be $\geq t_{SR}$. Values of P_R, L_R, and t_{SR} shall not deviate by more than ±1 percent from the time of recording {R1} to that of recording {R2}.

4.6.5 If self-prepared chemical standards are employed and spectra of only two concentrations are recorded for one or more compounds, verify the accuracy of the dilution technique by analyzing the prepared standards for those compounds with a secondary (non-FTIR) technique as follows:

4.6.5.1 Record the response of the secondary technique to each of the four standards prepared.

4.6.5.2 Perform a linear regression of the response values (dependant variable) versus the accepted standard concentration (ASC) values (independent variable), with the regression constrained to pass through the zero-response, zero ASC point.

4.6.5.3 Calculate the average fractional difference between the actual response values and the regression-predicted values (those calculated from the regression line using the four ASC values as the independent variable).

4.6.5.4 If the average fractional difference value calculated in Section 4.6.5.3 is larger for any compound than the corresponding AU_i, the dilution technique is not sufficiently accurate and the reference spectra prepared are not valid for the analysis.

4.7 Select Analytical Regions. Using the general considerations in Section 7 of Reference A and the spectral characteristics of the analytes and interferants, select the analytical regions for the application. Label them m = 1 to M. Specify the lower, center and upper wavenumber positions of each

analytical region (FL_m, FC_m, and FU_m, respectively). Specify the analytes and interferants which exhibit absorption in each region.

4.8 Determine Fractional Reproducibility Uncertainties. Using Appendix E, calculate the fractional reproducibility uncertainty for each analyte (FRU_i) from a comparison of {R1} and {R2}. If $FRU_i > AU_i$ for any analyte, the reference spectra generated in Section 4.6 are not valid for the application.

4.9 Identify Known Interferants. Using Appendix B, determine which potential interferant affects the analyte concentration determinations. If it does, relabel the potential interferant as "known" interferant, and designate these compounds from k = 1 to K. Appendix B also provides criteria for determining whether the selected analytical regions are suitable.

4.10 Prepare Computerized Analytical Programs.

4.10.1 Choose or devise mathematical techniques (e.g, classical least squares, inverse least squares, cross-correlation, and factor analysis) based on Equation 4 of Reference A that are appropriate for analyzing spectral data by comparison with reference spectra.

4.10.2 Following the general recommendations of Reference A, prepare a computer program or set of programs that analyzes all the analytes and known interferants, based on the selected analytical regions (4.7) and the prepared reference spectra (4.6). Specify the baseline correction technique (e.g., determining the slope and intercept of a linear baseline contribution in each analytical region) for each analytical region, including all relevant wavenumber positions.

4.10.3 Use programs that provide as output [at the reference absorption pathlength (L_R), reference gas temperature (T_R), and reference gas pressure (P_R)] the analyte concentrations, the known interferant concentrations, and the baseline slope and intercept values. If the sample absorption pathlength (L_S), sample gas temperature (T_S) or sample gas pressure (P_S) during the actual sample analyses differ from L_R, T_R, and P_R, use a program or set of programs that applies multiplicative corrections to the derived concentrations to account for these variations, and that provides as output both the corrected and uncorrected values. Include in the report of the analysis (see Section 7.0) the details of any transformations applied to the original reference spectra (e.g., differentiation), in such a fashion that all analytical results may be verified by an independent agent from the reference spectra and data spectra alone.

4.11 Determine the Fractional Calibration Uncertainty. Calculate the fractional calibration uncertainty for each analyte (FCU_i) according to Appendix F, and compare these values to the

fractional uncertainty limits (AU_i; see Section 4.1). If $FCU_i > AU_i$), either the reference spectra or analytical programs for that analyte are unsuitable.

4.12 Verify System Configuration Suitability. Using Appendix C, measure or obtain estimates of the noise level (RMS_{EST}, absorbance) of the FTIR system; alternatively, construct the complete spectrometer system and determine the values RMS_{Sm} using Appendix G. Estimate the minimum measurement uncertainty for each analyte (MAU_i, ppm) and known interferant (MIU_k, ppm) using Appendix D. Verify that (a) $MAU_i < (AU_i)(DL_i)$, $FRU_i < AU_i$, and $FCU_i < AU_i$ for each analyte and that (b) the CTS chosen meets the requirements listed in Section 4.5.

5.0 SAMPLING AND ANALYSIS PROCEDURE

5.1 Analysis System Assembly and Leak-Test. Assemble the analysis system. Allow sufficient time for all system components to reach the desired temperature. Then determine the leak-rate (L_R) and leak volume (V_L), where $V_L = L_R\ t_{SS}$. Leak volumes shall be ≤4 percent of V_{SS}.

5.2 Verify Instrumental Performance. Measure the noise level of the system in each analytical region using the procedure of Appendix G. If any noise level is higher than that estimated for the system in Section 4.12, repeat the calculations of Appendix D and verify that the requirements of Section 4.12 are met; if they are not, adjust or repair the instrument and repeat this section.

5.3 Determine the Sample Absorption Pathlength. Record a background spectrum. Then, fill the absorption cell with CTS at the pressure P_R and record a set of CTS spectra {R3}. Store the background and unscaled CTS single beam interferograms and spectra. Using Appendix H, calculate the sample absorption pathlength (L_S) for each analytical region. The values L_S shall not differ from the approximated sample pathlength L_S' (see Section 4.4) by more than 5 percent.

5.4 Record Sample Spectrum. Connect the sample line to the source. Either evacuate the absorption cell to an absolute pressure below 5 mmHg before extracting a sample from the effluent stream into the absorption cell, or pump at least ten cell volumes of sample through the cell before obtaining a sample. Record the sample pressure P_S. Generate the absorbance spectrum of the sample. Store the background and sample single beam interferograms, and document the process by which the absorbance spectra are generated from these data. (If necessary, apply the spectral transformations developed in Section 5.6.2). The resulting sample spectrum is referred to below as S_S.

Note: Multiple sample spectra may be recorded according to the procedures of Section 5.4 before performing Sections 5.5 and 5.6.

5.5 Quantify Analyte Concentrations. Calculate the unscaled analyte concentrations RUA_i and unscaled interferant concentrations RUI_K using the programs developed in Section 4. To correct for pathlength and pressure variations between the reference and sample spectra, calculate the scaling factor $R_{LPS} = (L_R P_R T_S)/(L_S P_S T_R)$. Calculate the final analyte and interferant concentrations $RSA_i = R_{LPS} RUA_i$ and $RSI_k = R_{LPS} RUI_k$.

5.6 Determine Fractional Analysis Uncertainty. Fill the absorption cell with CTS at the pressure P_S. Record a set of CTS spectra {R4}. Store the background and CTS single beam interferograms. Using Appendix H, calculate the fractional analysis uncertainty (FAU) for each analytical region. If the FAU indicated for any analytical region is larger than the required accuracy requirements determined in Section 4.1, then comparisons to previously recorded reference spectra are invalid in that analytical region, and the analyst shall perform one or both of the following procedures:

5.6.1 Perform instrumental checks and adjust the instrument to restore its performance to acceptable levels. If adjustments are made, repeat Sections 5.3, 5.4 (except for the recording of a sample spectrum), and 5.5 to demonstrate that acceptable uncertainties are obtained in all analytical regions.

5.6.2 Apply appropriate mathematical transformations (e.g., frequency shifting, zero-filling, apodization, smoothing) to the spectra (or to the interferograms upon which the spectra are based) generated during the performance of the procedures of Section 5.3. Document these transformations and their reproducibility. Do not apply multiplicative scaling of the spectra, or any set of transformations that is mathematically equivalent to multiplicative scaling. Different transformations may be applied to different analytical regions. Frequency shifts shall be smaller than one-half the minimum instrumental linewidth, and must be applied to all spectral data points in an analytical region. The mathematical transformations may be retained for the analysis if they are also applied to the appropriate analytical regions of all sample spectra recorded, and if all original sample spectra are digitally stored. Repeat Sections 5.3, 5.4 (except the recording of a sample spectrum), and 5.5 to demonstrate that these transformations lead to acceptable calculated concentration uncertainties in all analytical regions.

6.0 POST-ANALYSIS EVALUATIONS

Estimate the overall accuracy of the analyses performed in Section 5 as follows:

6.1 Qualitatively Confirm the Assumed Matrix. Examine each analytical region of the sample spectrum for spectral evidence of unexpected or unidentified interferants. If found, identify the interfering compounds (see Reference C for guidance) and add them to the list of known interferants. Repeat the procedures of Section 4 to include the interferants in the uncertainty calculations and analysis procedures. Verify that the MAU and FCU values do not increase beyond acceptable levels for the application requirements. Re-calculate the analyte concentrations (Section 5.5) in the affected analytical regions.

6.2 Quantitatively Evaluate Fractional Model Uncertainty (FMU). Perform the procedures of either Section 6.2.1 or 6.2.2:

6.2.1 Using Appendix I, determine the fractional model error (FMU) for each analyte.

6.2.2 Provide statistically determined uncertainties FMU for each analyte which are equivalent to two standard deviations at the 95% confidence level. Such determinations, if employed, must be based on mathematical examinations of the pertinent sample spectra (not the reference spectra alone). Include in the report of the analysis (see Section 7.0) a complete description of the determination of the concentration uncertainties.

6.3 Estimate Overall Concentration Uncertainty (OCU). Using Appendix J, determine the overall concentration uncertainty (OCU) for each analyte. If the OCU is larger than the required accuracy for any analyte, repeat Sections 4 and 6.

7.0 REPORTING REQUIREMENTS

[Documentation pertaining to virtually all the procedures of Sections 4, 5, and 6 will be required. Software copies of reference spectra and sample spectra will be retained for some minimum time following the actual testing.]

8.0 REFERENCES

A) Standard Practices for General Techniques of Infrared Quantitative Analysis (American Society for Testing and Materials, Designation E 168-88).

B) The Coblentz Society Specifications for Evaluation of Research Quality Analytical Infrared Reference Spectra (Class II); Anal. Chemistry 47, 945A (1975); **Appl. Spectroscopy 444, pp. 211-215, 1990.**

C) Standard Practices for General Techniques for Qualitative Infrared Analysis, American Society for Testing and Materials, Designation E 1252-88.

D) "Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Emissions Monitors (Protocol Number 1)," June 1978, Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/4-77-027b, August 1977.

APPENDIX A

DEFINITIONS OF TERMS AND SYMBOLS

A.1 Definitions of Terms

absorption band - a contiguous wavenumber region of a spectrum (equivalently, a contiguous set of absorbance spectrum data points) in which the absorbance passes through a maximum or a series of maxima.

absorption pathlength - in a spectrophotometer, the distance, measured in the direction of propagation of the beam of radiant energy, between the surface of the specimen on which the radiant energy is incident and the surface of the specimen from which it is emergent.

analytical region - a contiguous wavenumber region (equivalently, a contiguous set of absorbance spectrum data points) used in the quantitative analysis for one or more analyte.

Note: The quantitative result for a single analyte may be based on data from more than one analytical region.

apodization - modification of the ILS function by multiplying the

interferogram by a weighing function whose magnitude varies with retardation.

background spectrum - the single beam spectrum obtained with all system components without sample present.

baseline - any line drawn on an absorption spectrum to establish a reference point that represents a function of the radiant power incident on a sample at a given wavelength.

Beers's law - the direct proportionality of the absorbance of a compound in a homogeneous sample to its concentration.

calibration transfer standard (CTS) gas - a gas standard of a compound used to achieve and/or demonstrate suitable quantitative agreement between sample spectra and the reference spectra; see Section 4.5.1.

compound - a substance possessing a distinct, unique molecular structure.

concentration (c) - the quantity of a compound contained in a unit quantity of sample. The unit "ppm" (number, or mole, basis) is recommended.

concentration-pathlength product - the mathematical product of concentration of the species and absorption pathlength. For

reference spectra, this is a known quantity; for sample spectra, it is the quantity directly determined from Beer's law. The units "centimeters-ppm" or "meters-ppm" are recommended.

derivative absorption spectrum - a plot of rate of change of absorbance or of any function of absorbance with respect to wavelength or any function of wavelength.

double beam spectrum - a transmission or absorbance spectrum derived by dividing the sample single beam spectrum by the background spectrum.

Note: The term "double-beam" is used elsewhere to denote a spectrum in which the sample and background interferograms are collected simultaneously along physically distinct absorption paths. Here, the term denotes a spectrum in which the sample and background interferograms are collected at different times along the same absorption path.

fast Fourier transform (FFT) - a method of speeding up the computation of a discrete FT by factoring the data into sparse matrices containing mostly zeros.

flyback - interferometer motion during which no data are recorded.

Fourier transform (FT) - the mathematical process for converting an amplitude-time spectrum to an amplitude-frequency spectrum, or vice versa.

Fourier transform infrared (FTIR) spectrometer - an analytical system that employs a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements that transfer infrared radiation between components, and a computer system. The time-domain detector response (interferogram) is processed by a Fourier transform to yield a representation of the detector response vs. infrared frequency.

Note: When FTIR spectrometers are interfaced with other instruments, a slash should be used to denote the interface; e.g., GC/FTIR; HPCL/FTIR, and the use of FTIR should be explicit; i.e., FTIR not IR.

frequency, ν - the number of cycles per unit time.

infrared - the portion of the electromagnetic spectrum containing wavelengths from approximately 0.78 to 800 microns.

interferogram, $I(\sigma)$ - record of the modulated component of the interference signal measured as a function of retardation by the detector.

interferometer - device that divides a beam of radiant energy into two or more paths, generate an optical path difference between the beams, and recombines them in order to produce repetitive interference maxima and minima as the optical retardation is varied.

linewidth - the full width at half maximum of an absorption band in units of wavenumbers (cm^{-1}).

mid-infrared - the region of the electromagnetic spectrum from approximately 400 to 5000 cm^{-1}.

pathlength - see "absorption pathlength."

reference spectra - absorption spectra of gases with known chemical compositions, recorded at a known absorption pathlength, which are used in the quantitative analysis of gas samples.

retardation, σ - optical path difference between two beams in an interferometer; also known as "optical path difference" or "optical retardation."

scan - digital representation of the detector output obtained during one complete motion of the interferometer's moving assembly or assemblies.

scaling - application of a multiplicative factor to the absorbance values in a spectrum.

single beam spectrum - Fourier-transformed interferogram, representing the detector response vs. wavenumber.

Note: The term "single-beam" is used elsewhere to denote any spectrum in which the sample and background interferograms are recorded on the same physical absorption path; such usage differentiates such spectra from those generated using interferograms recorded along two physically distinct absorption paths (see "double-beam spectrum" above). Here, the term applies (for example) to the two spectra used directly in the calculation of transmission and absorbance spectra of a sample.

standard reference material - a reference material, the composition or properties of which are certified by a recognized standardizing agency or group.

Note: The equivalent ISO term is "certified reference material."

transmittance, T - the ratio of radiant power transmitted by the sample to the radiant power incident on the sample. Estimated in FTIR spectroscopy by forming the ratio of the single-beam sample and background spectra.

wavenumber, $\bar{v}$ - the number of waves per unit length.

Note: The usual unit of wavenumber is the reciprocal centimeter, cm^{-1}. The wavenumber is the reciprocal of the wavelength, λ, when λ is expressed in centimeters.

zero-filling - the addition of zero-valued points to the end of a measured interferogram.

Note: Performing the FT of a zero-filled interferogram results in correctly interpolated points in the computed spectrum.

A.2 Definitions of Mathematical Symbols

A, absorbance - the logarithm to the base 10 of the reciprocal of the transmittance (T).

$$A = \log_{10}\left(\frac{1}{T}\right) = -\log_{10}T \qquad (1)$$

AAI_{im} - band area of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i) .

AAV_{im} - average absorbance of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i) .

ASC, accepted standard concentration - the concentration value assigned to a chemical standard.

ASCPP, accepted standard concentration-pathlength product - for a chemical standard, the product of the ASC and the sample absorption pathlength. The units "centimeters-ppm" or "meters-ppm" are recommended.

AU_i, analytical uncertainty limit - the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration determined in the analysis.

AVT_m - average estimated total absorbance in the mth analytical region.

$CKWN_k$ - estimated concentration of the kth known interferant.

$CMAX_i$ - estimated maximum concentration of the ith analyte.

$CPOT_j$ - estimated concentration of the jth potential interferant.

DL_i, **required detection limit** - for the ith analyte, the lowest concentration of the analyte for which its overall fractional uncertainty (OFU_i) is required to be less than the analytical uncertainty limit (AU_i).

FC_m - center wavenumber position of the mth analytical region.

FAU_i, **fractional analytical uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the mathematical comparison of reference and sample spectra.

FCU_i, **fractional calibration uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in Beer's law modeling of the reference spectra concentrations.

FFL_m - lower wavenumber position of the CTS absorption band associated with the mth analytical region.

FFU_m - upper wavenumber position of the CTS absorption band associated with the mth analytical region.

FL_m - lower wavenumber position of the mth analytical region.

FMU_i, **fractional model uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the absorption model employed.

FN_L - lower wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

FN_U - upper wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

FRU_i, **fractional reproducibility uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the reproducibility of spectra from the FTIR system.

FU_m - upper wavenumber position of the mth analytical region.

IAI_{jm} - band area of the jth potential interferant in the mth analytical region, at its expected concentration ($CPOT_j$).

IAV_{im} - average absorbance of the ith analyte in the mth analytical region, at its expected concentration ($CPOT_j$).

$ISC_{i \text{ or } k}$, indicated standard concentration - the concentration from the computerized analytical program for a single-compound reference spectrum for the ith analyte or k^{th} known interferant.

kPa - kilo-Pascal (see Pascal).

L_S' - estimated sample absorption pathlength.

L_R - reference absorption pathlength.

L_S - actual sample absorption pathlength.

MAU_i - mean of the MAU_{im} over the appropriate analytical regions.

MAU_{im}, minimum analyte uncertainty - the calculated minimum concentration for which the analytical uncertainty limit (AU_i) in the measurement of the ith analyte, based on spectral data in the mth analytical region, can be maintained.

MIU_j - mean of the MIU_{jm} over the appropriate analytical regions.

MIU_{jm}, minimum interferant uncertainty - the calculated minimum concentration for which the analytical uncertainty limit $CPOT_j/20$ in the measurement of the jth interferant, based on spectral data in the mth analytical region, can be maintained.

MIL, minimum instrumental linewidth - the minimum linewidth from the FTIR system, in wavenumbers.

<u>Note</u>: The MIL of a system may be determined by observing an absorption band known (through higher resolution examinations) to be narrower than indicated by the system. The MIL is fundamentally limited by the retardation of the interferometer, but is also affected by other operational parameters (e.g., the choice of apodization).

N_i - number of analytes.

N_j - number of potential interferants.

N_k - number of known interferants.

N_{scan} - the number of scans averaged to obtain an interferogram.

OFU_i - the overall fractional uncertainty in an analyte concentration determined in the analysis ($OFU_i = MAX\{FRU_i, FCU_i, FAU_i, FMU_i\}$).

Pascal (Pa) - metric unit of static pressure, equal to one Newton per square meter; one atmosphere is equal to 101,325 Pa;

1/760 atmosphere (one Torr, or one millimeter Hg) is equal to 133.322 Pa.

P_{min} - minimum pressure of the sampling system during the sampling procedure.

P_S' - estimated sample pressure.

P_R - reference pressure.

P_S - actual sample pressure.

RMS_{Sm} - measured noise level of the FTIR system in the mth analytical region.

RMSD, root mean square difference - a measure of accuracy determined by the following equation:

$$RMSD = \sqrt{\left(\frac{1}{n}\right) \sum_{i=1}^{n} e_i^2} \qquad (2)$$

where:

n = the number of observations for which the accuracy is determined.

e_i = the difference between a measured value of a property and its mean value over the n observations.

Note: The RMSD value "between a set of n contiguous absorbance values (A_i) and the mean of the values" (A_M) is defined as

$$RMSD = \sqrt{\left(\frac{1}{n}\right) \sum_{i=1}^{n} (A_i - A_M)^2} \qquad (3)$$

RSA_i - the (calculated) final concentration of the ith analyte.

RSI_k - the (calculated) final concentration of the kth known interferant.

t_{scan}, scan time - time used to acquire a single scan, not including flyback.

t_S, signal integration period - the period of time over which an interferogram is averaged by addition and scaling of individual scans. In terms of the number of scans N_{scan} and scan time t_{scan}, $t_S = N_{scan} t_{scan}$.

t_{SR} - signal integration period used in recording reference spectra.

t_{SS} - signal integration period used in recording sample spectra.

T_R - absolute temperature of gases used in recording reference spectra.

T_S - absolute temperature of sample gas as sample spectra are recorded.

TP, Throughput - manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

V_{SS} - volume of the infrared absorption cell, including parts of attached tubing.

W_{ik} - weight used to average over analytical regions k for quantities related to the analyte i; see Appendix D.

Note that some terms are missing, e.g., BAV_m, OCU, $RMSS_m$, SUB_S, SIC_i, SAC_i, S_S

APPENDIX B

IDENTIFYING SPECTRAL INTERFERANTS

B.1 General

B.1.1 Assume a fixed absorption pathlength equal to the value L_S'.

B.1.2 Use band area calculations to compare the relative absorption strengths of the analytes and potential interferants. In the mth analytical region (FL_m to FU_m), use either rectangular or trapezoidal approximations to determine the band areas described below (see Reference A, Sections A.3.1 through A.3.3); document any baseline corrections applied to the spectra.

B.1.3 Use the average total absorbance of the analytes and potential interferants in each analytical region to determine whether the analytical region is suitable for analyte concentration determinations.

Note: The average absorbance in an analytical region is the band area divided by the width of the analytical region in wavenumbers. The average total absorbance in an analytical region is the sum of the average absorbances of all analytes and potential interferants.

B.2 Calculations

B.2.1 Prepare spectral representations of each analyte at the concentration $CL_i = (DL_i)(AU_i)$, where DL_i is the required detection limit and AU_i is the maximum permissible analytical uncertainty. For the mth analytical region, calculate the band area (AAI_{im}) and average absorbance (AAV_{im}) from these scaled analyte spectra.

B.2.2 Prepare spectral representations of each potential interferant at its expected concentration ($CPOT_j$). For the mth analytical region, calculate the band area (IAI_{jm}) and average absorbance (IAV_{jm}) from these scaled potential interferant spectra.

B.2.3 Repeat the calculation for each analytical region, and record the band area results in matrix form as indicated in Figure B.1.

B.2.4 If the band area of any potential interferant in an analytical region is greater than the one-half the band area of any analyte (i.e., $IAI_{jm} > 0.5\ AAI_{im}$ for any pair ij and any m), classify the potential interferant as known interferant. Label the known interferants k = 1 to K. Record the results in matrix form as indicated in Figure B.2.

B.2.5 Calculate the average total absorbance (AVT_m) for each analytical region and record the values in the last row of the matrix described in Figure B.2. Any analytical region where AVT_m >2.0 is unsuitable.

FIGURE B.1 Presentation of Potential Interferant Calculations

	Analytical Regions 1 . . . . . M
Analyte Labels	
1	AAI_{11} . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . AAI_{IM}
Potential Interferant Labels	
1	IAI_{11} . . . IAI_{1M}
.	. .
.	. .
J	IAI_{J1} . . . IAI_{JM}

FIGURE B.2 Presentation of Known Interferant Calculations

	Analytical Regions 1 . . . . . M
Analyte Labels	
1	AAI_{11} . . . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . . . AAI_{IM}
Known Interferant Labels	
1	IAI_{11} . . . . . IAI_{1M}
.	. .
.	. .
K	IAI_{K1} . . . . . IAI_{KM}
Total Average Absorbance	AVT_1 AVT_M

APPENDIX C

ESTIMATING NOISE LEVELS

C.1 General

C.1.1 The root-mean-square (RMS) noise level is the standard measure of noise in this Protocol. The RMS noise level of a contiguous segment of a spectrum is defined as the RMS difference (RMSD) between the absorbance values which form the segment and the mean value of that segment (see Appendix A).

C.1.2 The RMS noise value in double-beam absorbance spectra is assumed to be inversely proportional to: (a) the square root of the signal integration period of the sample single beam spectra from which it is formed, and (b) to the total infrared power transmitted through the interferometer and absorption cell.

C.1.3 Practically, the assumption of C.1.2 allow the RMS noise level of a complete system to be estimated from the following four quantities:

(a) RMS_{MAN} - the noise level of the system (in absorbance units), without the absorption cell and transfer optics, <u>under those conditions necessary to yield the specified minimum instrumental linewidth</u>, e.g., Jacquinot stop size.

(b) t_{MAN} - the manufacturer's signal integration time used to determine RMS_{MAN}.

(c) t_{SS} - the signal integration time for the analyses.

(d) TP - the manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

C.2 Calculations

C.2.1 Obtain the values of RMS_{MAN}, t_{MAN}, and TP from the manufacturers of the equipment, or determine the noise level by direct measurements with the completely constructed system proposed in Section 4.

C.2.2 Calculate the noise value of the system (RMS_{EST}) as follows:

$$RMS_{EST} = RMS_{MAN} \; TP \sqrt{\frac{t_{ss}}{t_{MAN}}} \qquad (4)$$

APPENDIX D

ESTIMATING MINIMUM CONCENTRATION MEASUREMENT UNCERTAINTIES (MAU and MIU)

D.1 General

Estimate the minimum concentration measurement uncertainties for the ith analyte (MAU_i) and j^{th} interferant (MIU_j) based on the spectral data in the mth analytical region by comparing the analyte band area in the analytical region (AAI_{im}) and estimating or measuring the noise level of the system (RMS_{EST} or RMS_{Sm}).

Note: For a single analytical region, the MAU or MIU value is the concentration of the analyte or interferant for which the band area is equal to the product of the analytical region width (in wavenumbers) and the noise level of the system (in absorbance units). If data from more than one analytical region is used in the determination of an analyte concentration, the MAU or MIU is the mean of the separate MAU or MIU values calculated for each analytical region.

D.2 Calculations

D.2.1 For each analytical region, set RMS = RMS_{Sm} if measured (Appendix G), or set RMS = RMS_{EST} if estimated (Appendix C).

D.2.2 For each analyte associated with the analytical region, calculate

$$MAU_{im} = (RMS)(DL_i)(AU_i)\frac{(FU_m - FL_m)}{AAI_{im}} \qquad (5)$$

D.2.3 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $MAU_i = MAU_{im}$.

D.2.4 If a number of analytical regions are used to calculate the concentration of the ith analyte, set MAU_i equal to the weighted mean of the appropriate MAU_{im} values calculated above; the weight for each term in the mean is equal to the fraction of the total wavenumber range used for the calculation represented by each analytical region. Mathematically, if the set of analytical regions employed is {m'}, then the MAU for each analytical region is

$$MAU_i = \sum_{k \in \{m'\}} W_{ik} \; MAU_{ik} \tag{6}$$

where the weight W_{ik} is defined for each term in the sum as

$$W_{ik} = (FM_k - FL_k) \left(\sum_{p \in \{m'\}} [FM_p - FL_p] \right)^{-1} \tag{7}$$

D.2.5 Repeat Sections D.2.1 through D.2.4 to calculate the analogous values MIU_j for the interferants j = 1 to J. Replace the value $(AU_i)(DL_i)$ in the above equations with $CPOT_j/20$; replace the value AAI_{im} in the above equations with IAI_{jm}.

APPENDIX E

DETERMINING FRACTIONAL REPRODUCIBILITY UNCERTAINTIES (FRU)

E.1 General

To estimate the reproducibility of the spectroscopic results of the system, compare the CTS spectra recorded before and after preparing the reference spectra. Compare the difference between the spectra to their average band area. Perform the calculation for each analytical region on the portions of the CTS spectra associated with that analytical region.

E.2 Calculations

E.2.1 The CTS spectra {R1} consist of N spectra, denoted by S_{1i}, i=1, N. Similarly, the CTS spectra {R2} consist of N spectra, denoted by S_{2i}, i=1, N. Each S_{ki} is the spectrum of a single compound, where i denotes the compound and k denotes the set {Rk} of which S_{ki} is a member. Form the spectra S_3 according to $S_{3i} = S_{2i}-S_{1i}$ for each i. Form the spectra S_4 according to $S_{4i} = [S_{2i}+S_{1i}]/2$ for each i.

E.2.2 Each analytical region m is associated with a portion of the CTS spectra S_{2i} and S_{1i}, for a particular i, with lower and upper wavenumber limits FFL_m and FFU_m, respectively.

E.2.3 For each m and the associated i, calculate the band area of S_{4i} in the wavenumber range FFU_m to FFL_m. Follow the guidelines of Section B.1.2 for this band area calculation. Denote the result by BAV_m.

E.2.4 For each m and the associated i, calculate the RMSD of S_{3i} between the absorbance values and their mean in the wavenumber range FFU_m to FFL_m. Denote the result by $SRMS_m$.

E.2.5 For each analytical region m, calculate the quantity

$$FM_m = SRMS_m(FFU_m-FFL_m)/BAV_m$$

E.2.6 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FRU_i = FM_m$.

E.2.7 If a number p_i of analytical regions are used to calculate the concentration of the ith analyte, set FRU_i equal to the weighted mean of the appropriate FM_m values calculated above. Mathematically, if the set of analytical regions employed is {m'}, then

$$FRU_i = \sum_{k \in \{m'\}} W_{ik} FM_k \qquad (8)$$

where the W_{ik} are calculated as described in Appendix D.

APPENDIX F

DETERMINING FRACTIONAL CALIBRATION UNCERTAINTIES (FCU)

F.1 General

F.1.1 The concentrations yielded by the computerized analytical program applied to each single-compound reference spectrum are defined as the indicated standard concentrations (ISC's). The ISC values for a single compound spectrum should ideally equal the accepted standard concentration (ASC) for one analyte or interferant, and should ideally be zero for all other compounds. Variations from these results are caused by errors in the ASC values, variations from the Beer's law (or modified Beer's law) model used to determine the concentrations, and noise in the spectra. When the first two effects dominate, the systematic nature of the errors is often apparent; take steps to correct them.

F.1.2 When the calibration error appears non-systematic, apply the following method to estimate the fractional calibration uncertainty (FCU) for each compound. The FCU is defined as the mean fractional error between the ASC and the ISC for all reference spectra with non-zero ASC for that compound. The FCU for each compound shall be less than the required fractional uncertainty specified in Section 4.1.

F.1.3 The computerized analytical programs shall also be required to yield acceptably low concentrations for compounds with ISC=0 when applied to the reference spectra. The limits chosen in this Protocol are that the ISC of each reference spectrum for each analyte or interferant shall not exceed that compound's minimum measurement uncertainty (MAU or MIU).

F.2 Calculations

F.2.1 Apply each analytical program to each reference spectrum. Prepare a similar table as that in Figure F.1 to present the ISC and ASC values for each analyte and interferant in each reference spectrum. Maintain the order of reference file names and compounds employed in preparing Figure F.1.

F.2.2 For all reference spectra in Figure F.1, verify that the absolute value of the ISC's are less than the compound's MAU (for analytes) or MIU (for interferants).

F.2.3 For each analyte reference spectrum, calculate the quantity (ASC-ISC)/ASC. For each analyte, calculate the mean of these values (the FCU_i for the ith analyte) over all reference spectra. Prepare a similar table as that in Figure F.2 to present the FCU_i and analytical uncertainty limit (AU_i) for each analyte.

FIGURE F.1

Presentation of Accepted Standard Concentrations (ASC's)
and Indicated Standard Concentrations (ISC's)

Compound Name	Reference Spectrum File Name	ASC (ppm)	ISC (ppm) Analytes i=1.....I			Interferants j=1.....J		

FIGURE F.2

Presentation of Fractional Calibration Uncertainties (FCU's)
and Analytical Uncertainties (AU's)

Analyte Name	FCU (%)	AU (%)

APPENDIX G

MEASURING NOISE LEVELS

G.1 General

The root-mean-square (RMS) noise level is the standard measure of noise. The RMS noise level of a contiguous segment of a spectrum is the RMSD between the absorbance values that form the segment and the mean value of the segment (see Appendix A).

G.2 Calculations

G.2.1 Evacuate the absorption cell or fill it with UPC grade nitrogen at approximately one atmosphere total pressure.

G.2.2 Record two single beam spectra of signal integration period t_{SS}.

G.2.3 Form the double beam absorption spectrum from these two single beam spectra, and calculate the noise level RMS_{Sm} in the M analytical regions.

APPENDIX H

DETERMINING SAMPLE ABSORPTION PATHLENGTH (L_S) AND FRACTIONAL ANALYTICAL UNCERTAINTY (FAU)

H.1 General

Reference spectra recorded at absorption pathlength (L_R), gas pressure (P_R), and gas absolute temperature (T_R) may be used to determine analyte concentrations in samples whose spectra are recorded at conditions different from that of the reference spectra, i.e., at absorption pathlength (L_S), absolute temperature (T_S), and pressure (P_S). Appendix H describes the calculations for estimating the fractional uncertainty (FAU) of this practice. It also describes the calculations for determining the sample absorption pathlength from comparison of CTS spectra, and for preparing spectra for further instrumental and procedural checks.

H.1.1 Before sampling, determine the sample absorption pathlength using least squares analysis. Determine the ratio L_S/L_R by comparing the spectral sets {R1} and {R3}, which are recorded using the same CTS at L_S and L_R, and T_S and T_R, but both at P_R.

H.1.2 Determine the fractional analysis uncertainty (FAU) for each analyte by comparing a scaled CTS spectral set, recorded at L_S, T_S, and P_S, to the CTS reference spectra of the same gas, recorded at L_R, T_R, and P_R. Perform the quantitative comparison after recording the sample spectra, based on band areas of the spectra in the CTS absorbance band associated with each analyte.

H.2 Calculations

H.2.1 Absorption Pathlength Determination. Perform and document separate linear baseline corrections to each analytical region in the spectral sets {R1} and {R3}. Form a one-dimensional array $\mathbf{A}_R$ containing the absorbance values from all segments of {R1} that are associated with the analytical regions; the members of the array are A_{Ri}, i = 1, n. Form a similar one-dimensional array $\mathbf{A}_S$ from the absorbance values in the spectral set {R3}; the members of the array are A_{Si}, i = 1, n. Based on the model $\mathbf{A}_S = r\mathbf{A}_R + \mathbf{E}$, determine the least-squares estimate of r', the value of r which minimizes the square error $\mathbf{E}^2$. Calculate the sample absorption pathlength $L_S = r'(T_S/T_R)L_R$.

H.2.2 Fractional Analysis Uncertainty. Perform and document separate linear baseline corrections to each analytical region in the spectral sets {R1} and {R4}. Form the arrays $\mathbf{A}_S$ and $\mathbf{A}_R$ as described in Section H.2.1, using values from {R1} to form $\mathbf{A}_R$, and values from {R4} to form $\mathbf{A}_S$. Calculate the values

$$NRMS_E = \sqrt{\sum_{i=1}^{n} \left[A_{Si} - \left(\frac{T_R}{T_S}\right)\left(\frac{L_S}{L_R}\right)\left(\frac{P_S}{P_R}\right) A_{Ri} \right]^2} \tag{9}$$

and

$$IA_{AV} = \frac{1}{2} \sum_{i=1}^{n} \left[A_{Si} + \left(\frac{T_R}{T_S}\right)\left(\frac{L_S}{L_R}\right)\left(\frac{P_S}{P_R}\right) A_{Ri} \right] \tag{10}$$

The fractional analytical uncertainty is defined as

$$FAU = \frac{NRMS_E}{IA_{AV}} \tag{11}$$

APPENDIX I

DETERMINING FRACTIONAL MODEL UNCERTAINTIES (FMU)

I.1 General

To prepare analytical programs for FTIR analyses, the sample constituents must first be assumed; the calculations in this appendix, based upon a simulation of the sample spectrum, verify the appropriateness of these assumptions. The simulated spectra consist of the sum of single compound reference spectra scaled to represent their contributions to the sample absorbance spectrum; scaling factors are based on the indicated standard concentrations (ISC) and measured (sample) analyte and interferant concentrations, the sample and reference absorption pathlengths, and the sample and reference gas pressures. No band-shape correction for differences in the temperature of the sample and reference spectra gases is made; such errors are included in the FMU estimate. The actual and simulated sample spectra are quantitatively compared to determine the fractional model uncertainty; this comparison uses the reference spectra band areas and residuals in the difference spectrum formed from the actual and simulated sample spectra.

I.2 Calculations

I.2.1 For each analyte (with scaled concentration RSA_i), select a reference spectrum SA_i with indicated standard concentration ISC_i. Calculate the scaling factors

$$RA_i = \frac{T_R\, L_S\, P_S\, RSA_i}{T_S\, L_R\, P_R\, ISC_i} \qquad (12)$$

and form the spectra SAC_i by scaling each SA_i by the factor RA_i.

I.2.2 For each interferant, select a reference spectrum SI_k with indicated standard concentration ISC_k. Calculate the scaling factors

$$RI_k = \frac{T_R\, L_S\, P_S\, RSI_k}{T_S\, L_R\, P_R\, ISC_k} \qquad (13)$$

and form the spectra SIC_k by scaling each SI_k by the factor RI_k.

I.2.3 For each analytical region, determine by visual inspection which of the spectra SAC_i and SIC_k exhibit absorbance bands within the analytical region. Subtract each spectrum SAC_i

and SIC_k exhibiting absorbance from the sample spectrum S_S to form the spectrum SUB_S. To save analysis time and to avoid the introduction of unwanted noise into the subtracted spectrum, it is recommended that the calculation be made (1) only for those spectral data points within the analytical regions, and (2) for each analytical region separately using the original spectrum S_S.

I.2.4 For each analytical region m, calculate the RMSD of SUB_S between the absorbance values and their mean in the region FFU_m to FFL_m. Denote the result by $RMSS_m$.

I.2.5 For each analyte i, calculate the quantity

$$FM_m = \frac{RMSS_m (FFU_m - FFL_m) AU_i DL_i}{AAI_i RSA_i} \tag{14}$$

for each analytical region associated with the analyte.

I.2.6 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FMU_i = FM_m$.

I.2.7 If a number of analytical regions are used to calculate the concentration of the ith analyte, set FM_i equal to the weighted mean of the appropriate FM_m values calculated above. Mathematically, if the set of analytical regions employed is $\{m'\}$, then

$$FMU_i = \sum_{k \in \{m'\}} W_{ik} FM_k \tag{15}$$

where W_{ik} is calculated as described in Appendix D.

APPENDIX J

DETERMINING OVERALL CONCENTRATION UNCERTAINTIES (OCU)

The calculations in previous sections and appendices estimate the measurement uncertainties for various FTIR measurements. The lowest possible overall concentration uncertainty (OCU) for an analyte is its MAU value, which is an estimate of the absolute concentration uncertainty when spectral noise dominates the measurement error. However, if the product of the largest fractional concentration uncertainty (FRU, FCU, FAU, or FMU) and the measured concentration of an analyte exceeds the MAU for the analyte, then the OCU is this product. In mathematical terms, set $OFU_i = MAX\{FRU_i, FCU_i, FAU_i, FMU_i\}$ and $OCU_i = MAX\{RSA_i*OFU_i, MAU_i\}$.

APPENDIX K

SPECTRAL DE-RESOLUTION PROCEDURES

K.1 General.

High resolution reference spectra can be converted into lower resolution standard spectra for use in quantitative analysis of sample spectra. This is accomplished by truncating the number of data points in the original reference sample and background interferograms.

De-resolved spectra must meet the following requirements to be used in quantitative analysis.

(a) The resolution must match the instrument sampling resolution. This is verified by comparing a de-resolved CTS spectrum to a CTS spectrum measured on the sampling instrument.

(b) The Fourier transformation of truncated interferograms (and their conversion to absorbance spectra) is performed using the same apodization function (and other mathematical corrections) used in converting the sample interferograms into absorbance spectra.

K.2 Procedures

This section details three alternative procedures using two different commercially available software packages. A similar procedures using another software packages is acceptable if it is based on truncation of the original reference interferograms and the results are verified by Section K.3.

K.2.1 KVB/Analect Software Procedure - The following example converts a 0.25 cm^{-1} 100 ppm ethylene spectrum (cts0305a) to 1 cm^{-1} resolution. The 0.25 cm^{-1} CTS spectrum was collected during the EPA reference spectrum program on March 5, 1992. The original data (in this example) are in KVB/Analect FX-70 format.

(i) **decomp cts0305a.aif,0305dres,1,16384,1**

"decomp" converts cts0305a to an ASCII file with name 0305dres. The resulting ASCII interferogram file is truncated to 16384 data points. Convert background interferogram (bkg0305a.aif) to ASCII in the same way.

(ii) **compose 0305dres,0305dres.aif,1**

"Compose" transforms truncated interferograms back to spectral format.

(iii) **IG2SP 0305dres.aif,0305dres.dsf,3,1,low cm^{-1},high cm^{-1}**

"IG2SP" converts interferogram to a single beam spectrum using Norton-Beer medium apodization, 3, and no zero filling, 1. De-resolved interferograms should be transformed using the same apodization and zero filling that will be used to collect sample spectra. Choose the desired low and high frequencies, in cm^{-1}. Transform the background interferogram in the same way.

(iv) **DVDR 0305dres.dsf,bkg0305a.dsf,0305dres.dlf**

"DVDR" ratios the transformed sample spectrum against the background.

(v) **ABSB 0305dres.dlf,0305dres.dlf**

"ABSB" converts the spectrum to absorbance.

The resolution of the resulting spectrum should be verified by comparison to a CTS spectrum collected at the nominal resolution. Refer to Section K.3.

K.2.2 Alternate KVB/Analect Procedure -- In either DOS (FX-70) or Windows version (FX-80) use the "Extract" command directly on the interferogram.

(i) **EXTRACT CTS0305a.aif,0305dres.aif,1,16384**

"Extract" truncates the interferogram to data points from to 16384 (or number of data points for desired nominal resolution). Truncate background interferogram in the same way.

(ii) Complete steps (iii) to (v) in Section K.2.1.

K.2.3 Grams™ Software Procedure - Grams™ is a software package that displays and manipulates spectra from a variety of instrument manufacturers. This procedure assumes familiarity with basic functions of Grams™.

This procedure is specifically for using Grams to truncate and transform reference interferograms that have been imported into Grams from the KVB/Analect format. Table K-1 shows data files and parameter values that are used in the following procedure.

The choice of all parameters in the ICOMPUTE.AB call of step 3 below should be fixed to the shown values, with the exception of the "Apodization" parameter. This parameter should be set (for both background and sample single beam conversions) to the type of apodization function chosen for the de-resolved spectral library.

TABLE K-1. GRAMS DATA FILES AND DE-RESOLUTION PARAMETERS.

Desired Nominal Spectral Resolution (cm^{-1})	Data File Name	Parameter "N" Value
0.25	Z00250.sav	65537
0.50	Z00500.sav	32769
1.0	Z01000.sav	16385
2.0	Z02000.sav	8193

(i) **Import** using "File/Import" the desired *.aif file. Clear all open data slots.

(ii) **Open** the resulting *.spc interferogram as file #1.

(iii) **Xflip** - *If the x-axis is increasing from left to right, and the ZPD burst appears near the left end of the trace, omit this step.*

In the "Arithmetic/Calc" menu item input box, type the text below. Perform the calculation by clicking on "OK" (once only), and, when the calculation is complete, click the "Continue" button to proceed to step (iv). Note the comment in step (iii) regarding the trace orientation.

xflip:#s=#s(#0,#N)+50

(iv) **Run ICOMPUTE.AB** from "Arithmetic/Do Program" menu. Ignore the "subscripting error," if it occurs.

The following menu choices should be made before execution of the program (refer to Table K-1 for the correct choice of "**N**":)

First: **N** Last: **0** Type: **Single Beam**
Zero Fill: **None** Apodization: **(as desired)**
Phasing: **User**
Points: **1024** Interpolation: **Linear** Phase:
Calculate

(v) **As** in step (iii), in the "**Arithmetic/Calc**" menu item enter and then run the following commands (refer to Table 1 for appropriate "**FILE,**" which may be in a directory other than "c:\mdgrams.")

setffp 7898.8805, 0 : loadspc "c:\mdgrams\ FILE" : #2=#s+#2

(vi) Use "**Page Up**" to activate file #2, and then use the "**File/Save As**" menu item with an appropriate file name to save the result.

K.3 Verification of New Resolution

K.3.1 Obtain interferograms of reference sample and background spectra. Truncate interferograms and convert to absorbance spectra of desired nominal resolution.

K.3.2 Document the apodization function, the level of zero filling, the number of data points, and the nominal resolution of the resulting de-resolved absorbance spectra. Use the identical apodization and level of zero filling when collecting sample spectra.

K.3.3 Perform the same de-resolution procedure on CTS interferograms that correspond with the reference spectra (reference CTS) to obtain de-resolved CTS standard spectra (CTS standards). Collect CTS spectra using the sampling resolution and the FTIR system to be used for the field measurements (test CTS). If practical, use the same pathlength, temperature, and standard concentration that were used for the reference CTS. Verify, by the following procedure that CTS linewidths and intensities are the same for the CTS standards and the test CTS.

K.3.4 After applying necessary temperature and pathlength corrections (document these corrections), subtract the CTS standard from the test CTS spectrum. Measure the RMSD in the resulting subtracted spectrum in the analytical region(s) of the CTS band(s). Use the following equation to compare this RMSD to the test CTS band area. The ratio in equation 7 must be no greater than 5 percent (0.05).

$$\frac{RMSS_i \times n(FFU_i - FFL_i)}{A_{CTS\text{-}test}} \leq .05 \qquad \textbf{(16)}$$

RMSS=RMSD in the ith analytical region in subtracted result, test CTS minus CTS standard.

n=number of data points per cm^{-1}. Exclude zero filled points.

FFU_i &=The upper and lower limits (cm^{-1}), respectively, of the FFL_i analytical region.

$A_{test\text{-}CTS}$=band area in the ith analytical region of the test CTS.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before comp

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15. SUPPLEMENTARY NOTES

16. ABSTRACT

The purpose of this testing program was to obtain emissions data by using FTIR sampling at iron foundries, specifically on cupola emission control devices as well as pouring, cooling, and shake-out operations to support a national emission standard for hazardous air pollutants (NESHAP).

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
MACT	MACT Support for the Iron & Steel Foundry Industry	

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