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Air



FTIR AND METHOD 25A EMISSIONS TEST AT AN INTEGRATED IRON AND STEEL MANUFACTURING PLANT

**Youngstown Sinter Company of WCI Steel, Inc.
Youngstown, Ohio**



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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

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PREFACE

This 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. 2-12. 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. 4-20 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 4-20 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 11.

This report presents the procedures, schedule, and test results for an emissions test performed at Youngstown Sinter Company in Youngstown, Ohio. The emissions test used Fourier transform infrared (FTIR) sampling procedures to measure hazardous air pollutants (HAP's) and other pollutants and Method 25A to measure hydrocarbon species.

This report consists of one volume (396 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 directed Midwest Research Institute (MRI) to conduct emissions testing at iron and steel manufacturing facilities, specifically on sintering processes. The test request was initiated by 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). The test program was performed in August, 1997, under Work assignment No. 4-20, under EPA Contract No. 68-D2-0165. A draft report was submitted under Work Assignment No. 2-08, under Contract 68-W6-0048.

Initially, the project included two field tests: (1) a screening test with FTIR Method 320 to evaluate the data for detected HAP's, and (2) a separate FTIR emissions test at the same site after additional preparation based on the screening results. The emissions test was to include performance of the Method 301 spiking procedure with method validation for any detected HAP's. Immediately before the field test the EPA altered the Scope of Work for this project to include only one test for HAP screening and emissions measurements. No validation testing was performed.

The test was performed on the sintering process at the Youngstown Sinter Company of WCI Steel, Inc., in Youngstown, Ohio, using EPA Draft FTIR Method 320¹ and EPA Method 25A. Method 320 is an extractive test method based on Fourier Transform infrared spectroscopy, which uses quantitative analytical procedures described in the EPA FTIR Protocol.² Data were used to quantify and characterize HAP and other detected emissions and the performance of the control unit for MACT standard development for this industry.

1.2 PROJECT SUMMARY

The sintering process is used to agglomerate fine raw materials into a product suitable for charging into a blast furnace. It is a potentially significant source of HAP emissions, including both metal and organic compounds. The principal emission point at a sinter plant is the exhaust from the sintering machine windboxes. Air pollution controls for the Youngstown Sinter Company of WCI Steel, Inc. include a Strand baghouse to control particulate emissions from the

sintering machine windboxes. Testing was conducted at the stack (outlet) and inlet to the Strand baghouse to determine the measurable emissions released during the sintering process.

Three test runs were conducted by MRI at each location over a 3 day period concurrently with manual method testing conducted by Eastern Research Group, Inc. (ERG). The FTIR testing was done by alternating sampling between the Strand baghouse inlet and stack; the Method 25A testing was continuous at both locations. Summaries of the FTIR and Method 25A results are presented in Tables 1-1 and 1-2, respectively. Average estimated uncertainties for some target analytes, identified in the test request, and for some other HAP's, are presented in Section 4.5.

The emission include hydrocarbon compounds that were represented primarily by "hexane" in the draft report results. Since the draft report was submitted, MRI has measured laboratory reference of some non-HAP hydrocarbon compounds. The new reference spectra were included in the revised analysis of the WCI FTIR data. The revised results presented in Tables 1-1, B-1, and B-2 include measurements of 2-methyl-2-butene, but eight other non-HAP hydrocarbons were not detected. The hexane concentrations are slightly lower in the revised results, but the draft hexane results were fairly accurate. The toluene concentrations in the revised results are also lower compared to the draft report results.

The EPA Method 320 uses an extractive sampling procedure. A probe, pump, and heated line are used to transport gas from the test port to a gas distribution 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. A compact disk containing all spectral data is provided with this report.

The EPA Method 25A also uses an extractive sampling procedure. The same sample transport system was used for both the FTIR and Method 25A testing. Volume concentration data and results obtained from the samples were recorded and saved on computer media and reviewed after the test was completed.

TABLE 1-1. SUMMARY OF FTIR RESULTS AT WCI BAGHOUSE INLET AND OUTLET

Compound		Baghouse Inlet			Baghouse Outlet		
		Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Toluene	ppm	0.88	4.84	9.64	1.37	3.06	10.3
	lb/hr	4.1	21.0	44.7	5.91	12.8	43.3
	kg/hr	1.9	9.54	20.3	2.68	5.80	19.6
Hexane	ppm	11.4	10.8	8.8	11.3	10.8	9.2
	lb/hr	49.4	43.9	38.0	45.4	42.4	36.3
	kg/hr	22.4	19.9	17.2	20.6	19.2	16.5
Ethylene	ppm	5.70	6.30	5.64	6.02	6.45	5.38
	lb/hr	8.03	8.33	7.95	7.88	8.21	6.90
	kg/hr	3.64	3.78	3.60	3.57	3.72	3.13
Methane	ppm	121	125	107	124	127	107
	lb/hr	97.5	94.3	86.2	93.1	92.9	78.7
	kg/hr	44.2	42.8	39.1	42.2	42.1	35.7
Sulfur Dioxide	ppm	175	182	149	166	175	132
	lb/hr	563	551	478	497	507	387
	kg/hr	255	250	217	225	230	175
Carbon Monoxide	ppm	1531	1558	1486	1548	1548	1490
	lb/hr	2153	2057	2091	2022	1968	1907
	kg/hr	976.1	932.7	948.1	916.8	892.3	864.9
Ammonia	ppm	4.51	0.46	2.25	5.34	0.64	2.55
	lb/hr	3.86	0.37	1.93	4.24	0.49	1.98
	kg/hr	1.75	0.17	0.87	1.92	0.22	0.90
Formaldehyde	ppm	4.46	4.43	5.55	4.06	3.68	5.27
	lb/hr	6.73	6.28	8.38	5.69	5.01	7.23
	kg/hr	3.05	2.85	3.80	2.58	2.27	3.28
Hydrogen Chloride	ppm	6.67	ND	0.98	6.16	ND	1.33
	lb/hr	12.2		1.79	10.5		2.22
	kg/hr	5.54		0.81	4.75		1.01
2-Methyl-2-butene	ppm	0.97	1.88	0.76	1.60	1.73	ND
	lb/hr	3.45	6.30	2.72	5.31	5.60	
	kg/hr	1.56	2.86	1.23	2.41	2.54	

^a The absorbance intensity in the CO reference spectrum was much less than the intensities in the sample spectra so these average concentrations are approximate. Measuring high concentration CO reference spectra may produce more accurate concentrations.

TABLE 1-2. SUMMARY OF METHOD 25A RESULTS FOR HYDROCARBON EMISSIONS

Test Data				
Run Number	1	2	3	
Date	12-Aug-97	13-Aug-97	14-Aug-97	
Time	1335-2000	1055-1736	0810-1029	Average
Baghouse inlet				
Gaseous Concentration				
THC ^a Concentration, ppm (wet basis)	248.7	244.3	220.9	238.0
Methane Concentration, ppm (wet basis)	121.4	125.0	107.3	117.9
Emissions Data				
THC ^a Emission Rate, lb/hr	149.8	138.2	133.2	140.4
TGNMOC ^b				
Emission Rate, lb/hr	76.7	67.5	68.5	70.9
Baghouse stack				
Gaseous Concentration				
THC ^a Concentration, ppmc (wet basis)	197.7	211.5	172.6	194.0
Methane Concentration, ppm (wet basis)	124.7	127.8	107.6	120.0
Emissions Data				
THC ^a Emission Rate, lb/hr	110.7	115.3	94.7	106.9
TGNMOC ^b				
Emission Rate, lb/hr	40.9	45.6	35.7	40.7

^a THC = Total hydrocarbons (ppm carbon).^b TGNMOC = Total gaseous non-methane organic carbon.

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
WCI Steel, Inc. Manger, Environmental Control of YSC	Thomas O. Shepker	(330) 841-8392
WCI Steel, Inc. Environmental Engineer Environmental Control of WCI	Keith McGlaughlin	(330) 841-8162
U. S. EPA, EMC Work Assignment Manager Work Assignment 4-20 Work Assignment 2-12	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 4-20 Work Assignment 2-12	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 YOUNGSTOWN SINTER COMPANY'S SINTER PLANT

The material in Section 2 was prepared by Eastern Research Group (ERG) and provided to MRI by the EMC. It is included in this report without MRI review.

2.1 OVERVIEW

The primary purpose of the sinter plant is to recover the iron value from waste materials generated at iron and steel plants by converting the materials to a product that can be used in the blast furnace (as burden material). Many of these wastes have little or no value otherwise and would require disposal if they could not be recycled by this process. A secondary purpose of the sinter plant is to recover lime from wastes and to convert limestone to lime, which is used as a fluxing agent in the blast furnace. The raw material feed (sinter mix) consists of iron ore fines, chips from iron ore pellets, fine limestone, slag from the steelmaking furnace, scale from the steel rolling mill, blast furnace flue dust, coke breeze (undersize coke that cannot be used in the blast furnace), and dolomite.

There are currently 10 sinter plants in operation in the U.S. A total of 6 of these plants use scrubbers to control emissions from the sinter plant windbox, and 4 use a baghouse. The sinter plant at Youngstown Sinter Plant, Youngstown, OH, a wholly owned subsidiary of WCI Steel Company, was chosen for testing to evaluate hazardous air pollutants and emission control performance associated with sinter plants that use baghouses.

2.2 PROCESS DESCRIPTION

The Youngstown sinter plant is operated by Youngstown Sinter Company, a wholly owned subsidiary of WCI Steel. The plant was purchased from LTV Steel Company and was brought on line in June 1991. The sinter plant is located a few miles from the WCI Steel integrated iron and steel plant in Warren, OH. The integrated plant includes one blast furnace, a basic oxygen furnace (BOF) shop containing two BOF vessels, ladle metallurgy, continuous casting, rolling mills, and galvanizing lines. The sinter plant has a capacity of 60,000 tons per month (tpm) and operates 24 hours per day with 2 days scheduled downtime every seven days for routine maintenance. The major processing steps in the sinter plant include preparation of the sinter mix (feed material), sintering, discharge end operations (crushing and screening), and cooling of the sinter product. Figure 2-1 is a simplified schematic of the sintering process.

The typical feed composition of the sinter mix during the emission tests is shown in Table 2-1.

TABLE 2-1. SUMMARY OF SINTER MIX (FEED) COMPONENTS

Feed material	Composition (% of feed)	Feed Rate (tons/day)
Ore fines	27.70	880
Mill scale	12.79	406
Limestone	12.15	385
Flue dust	9.07	288
Coke breeze	0.63	20
BOF slag	16.51	524
Pellet chips	19.73	625
Dolomite	1.42	4.5

The raw materials are brought into the sinter plant by truck and are stored at the site. Two feeder tables blend mill scale, BOF slag, and crushed ore pellets by volume, and the mixture is transferred by conveyor to the sinter plant and fed into the sinter machine through a series of bins. Limestone, dolomite, coke fines, and cold fines recycled from the sintering process are also contained in bins and are blended into the mix. A "hearth layer" of material, which is undersize sinter material that is recycled from the screening operation, is first deposited on the grate bars of the sinter pallets so that the sinter mixture does not burn through to the grate, and then the feed mix is added to a depth of about 17 inches. The plant has found that a deeper bed results in fewer fines being generated.

The sinter feed passes through an ignition furnace, and the surface of the sinter feed is ignited with natural gas. The sinter pallets move continually through the ignition furnace at

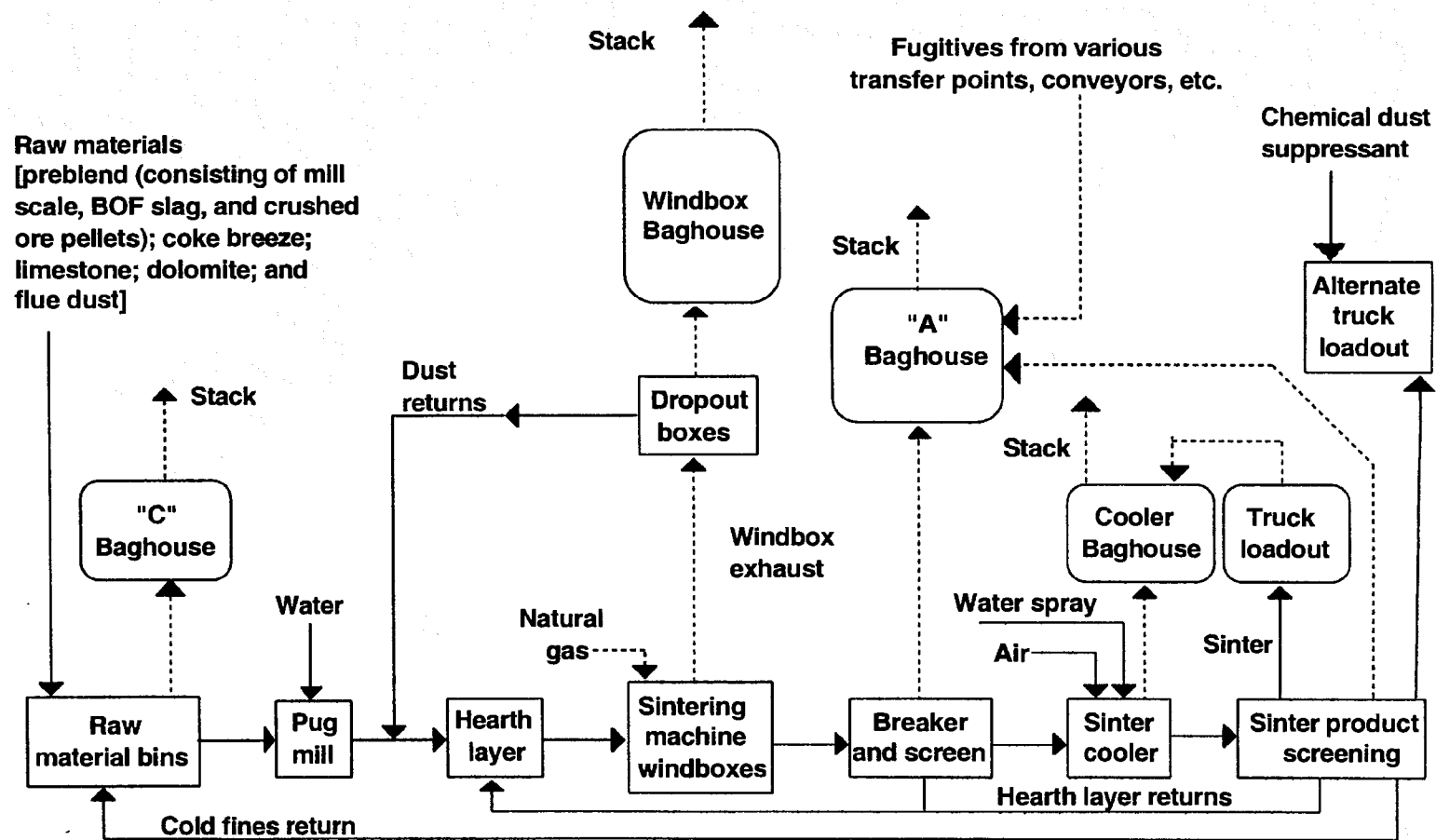


Figure 2-1. Schematic of material flow in sinter plant.

about 6.3 to 7.0 feet per minute over 21 vacuum chambers called "windboxes." A vacuum is created in the windbox by a fan that draws heat through the sinter bed and creates the fused "sintered" product.

The red hot sinter from the furnace continues to be transported on the pallets to the breaker, where it is crushed, screened, and discharged to a 250-foot linear four-stack sinter cooler. The sinter is removed from the cooler and transported by covered conveyor to the truck loadout station. The sinter plant has two truck loadout stations, and all of the sinter is transported to the blast furnace by truck. The larger station is evacuated to a hood which goes to the cooler baghouse; the building is open but has a curtain over each end to contain emissions with an opening for the trucks to enter and exit.

The smaller truck loadout station is used to provide more capacity and is normally used to handle production from the midnight shift; the station utilizes chemical dust suppression. The sinter is transferred by a covered conveyor from the sinter cooler to a storage building as needed, and is then transferred by a covered conveyor to the truck loadout station. Emissions from the sinter storage building are evacuated to the A baghouse. *SoLong*, manufactured by Midwest, is used for dust suppression at the truck loadout station. The chemical acts as a polymer and binds the dust to the sinter during truck loading; *SoLong* is applied to the sinter as the product exits the covered conveyor and drops into the bed of each truck. Very little emissions from the loading process were observed to escape capture at the larger truck loadout station. Some emissions were observed from the unenclosed area at the top of the conveyor and from the truck as the sinter was being loaded. Dust emissions were minimal but were noticeable depending on the truck being loaded. Sinter material that passes through the screens ("fines") is returned to the sinter process for use as the hearth layer or for addition to the sinter mix.

Several operating parameters are monitored and controlled to ensure proper operation of the sinter machine. These parameters include the feed rate of each of the ten feed bins, the sinter furnace temperature, the temperature profile through the various windboxes, draft on the windboxes, speed of the grate, and percent water in the feed. The percentage of oil in each of the feed materials is analyzed and the total amount of oil in the sinter feed is limited to less than 0.1 percent. To maintain the proper chemistry in the blast furnace, an important quality control parameter that is monitored is the sinter basicity:

$$(\text{CaO}+\text{MgO})/(\text{SiO}_2+\text{Al}_2\text{O}_3)$$

The sinter composition for the four tests days is summarized in Table 2-2 and shows that the sinter basicity ranged from 2.72 to 2.92.

TABLE 2-2. SUMMARY OF SINTER COMPOSITION

Component	Percent of total			
	Test 1 (08/12/97)	Test 2 (08/13/97)	Test 3 (08/14/97)	Test 4 (08/15/97)
Fe	53.23	52.23	52.42	52.20
SiO ₂	4.82	5.47	5.21	5.17
Al ₂ O ₃	0.90	0.98	0.91	0.89
CaO	14.69	15.30	15.03	15.40
MgO	2.09	2.16	2.23	2.28
Sinter basicity	2.90	2.72	2.84	2.92

2.3 EMISSION CONTROL EQUIPMENT

Emissions are generated in the process as sinter dust and combustion products are discharged through the grates and the 21 windboxes to a common collector main and are then collected by the strand baghouse. The pulse jet baghouse is manufactured by Environmental Elements and uses Nomex® bags that are coated with an acid-resistant finish. There are fourteen modules, each containing 306 bags. The bags are 6 inches in diameter and 15 feet in length, and the total cloth area for each module is 7,215 square feet. The gross air-to-cloth ratio is 3.96 acfm/ft² and the net air-to-cloth ratio, with one module off-line for cleaning is 4.26 acfm/ft².

The flow to the baghouse is approximately 400,000 cubic feet per minute. A preheat burner is used to minimize condensation and to bring the gas up to the desired inlet temperature. The dust is removed from the baghouse by rotary screw to bins where it is stored on the ground to gather moisture and is blended back into the sinter feed. The parameters associated with the baghouse that are monitored include the pressure drop across the baghouse, inlet temperature, stack temperature, damper percent, and fan amps.

Typical operating conditions associated with the baghouse are summarized in Table 2-3. Current State regulations limit particulate matter to 50 pounds per hour for the strand baghouse.

TABLE 2-3. TYPICAL BAGHOUSE PARAMETERS

Parameter	Typical value
Pressure drop	10 to 13 inches of water
Gas flow rate	400,000 scfm
Inlet temperature	235 to 270 F
Outlet temperature	120 F
Damper Percent	90%
Fan Amps	659-735

Three additional baghouses are used to control emissions from the sinter plant. The C baghouse, a pulse jet baghouse utilizing polyester bags, is used to control emissions from the material handling bins and the conveyors that transfer the sinter mix to the sinter machine. The cooler baghouse controls emissions from the sinter cooler and from the main truck loadout station. The baghouse is a shaker baghouse that utilizes Nomex® bags and contains nine compartments. Eight of the compartments are used for the cooler and one compartment is used for the truck loadout station. There are four 200 horsepower fans on the sinter cooler. The first fan is the dirtiest fan and is directed back to hoods on the sinter machine and sent back through as preheat air. The other 3 fans are ducted to the baghouse. In addition, the truck loadout station has a 70,000 cubic feet per minute fan. These baghouses were not evaluated as part of this test program.

The A baghouse that serves the discharge end of the sinter plant was evaluated as part of this test program. A schematic of A baghouse is shown in Figure 2-2. This baghouse controls emissions from discharge end emission points, including the hood before the sinter machine; the hood over sinter discharge; the sinter breaker and hot screen which is enclosed by a cloth curtain; the tail end of the sinter cooler; emissions from each of the ten sinter feed bins; a variety of transfer points for the transport of sinter, dust, and fines; and emissions from sinter bins located in the sinter overflow storage area. At any point where there is hot sinter, emissions are first ducted to a cyclone before going to the baghouse.

The plant sprays the roads twice per week to minimize dust emissions, except during the winter months. All of the baghouses are monitored on a weekly basis by an outside contractor, Fastway, Inc., to check the operation and for any visible opacity. A whole compartment is dye-tested if there is more than 5 percent visible emissions observed, and the broken bags are then replaced. Every other month, a complete compartment of either the strand or cooler baghouse is replaced; each compartment is replaced approximately every 3 years.

2.4 MONITORING RESULTS DURING THE TESTS

The operating parameters associated with the process and control device were recorded at 15-minute intervals throughout each test day. The process parameters that were monitored included the temperatures and the fan draft for the windboxes, percent water in the feed, sinter machine speed, and the temperature of each of the four cooling fans. In addition, the turn supervisor's report provided additional information, including tons per hour of pre-blend, and tons per 8-hour turn of limestone, dolomite, coke fines, and cold fines. The emission control device parameters that were monitored included the pressure drop across the baghouse, damper percent, inlet temperature, stack temperature, fan amps, and the pressure drop of each of the 14 compartments of the baghouse. Tables 2-4 and 2-5 present a summary of the range of values for these parameters for each test period. Table 2-6 presents a summary of the pressure drops of each compartment of the baghouse for the four days of testing.

The process and control device appeared to be stable throughout the four test days; consequently, sampling was conducted under normal and representative conditions. An examination of the monitoring data showed that the average pressure drop across the baghouse was 10.8, 12.0, 12.9 and 13.5 inches of water for the 4 test days. The pressure drop across the baghouse did increase slightly during each day of testing. On the third day, the compartments were double cleaned to try to reduce the pressure drop. The temperatures and draft of the windboxes varied somewhat during the tests; plant operators stated that the temperature of windboxes 19 and 20, should generally be 475° to 500°F to achieve proper burnthrough of the sinter bed.

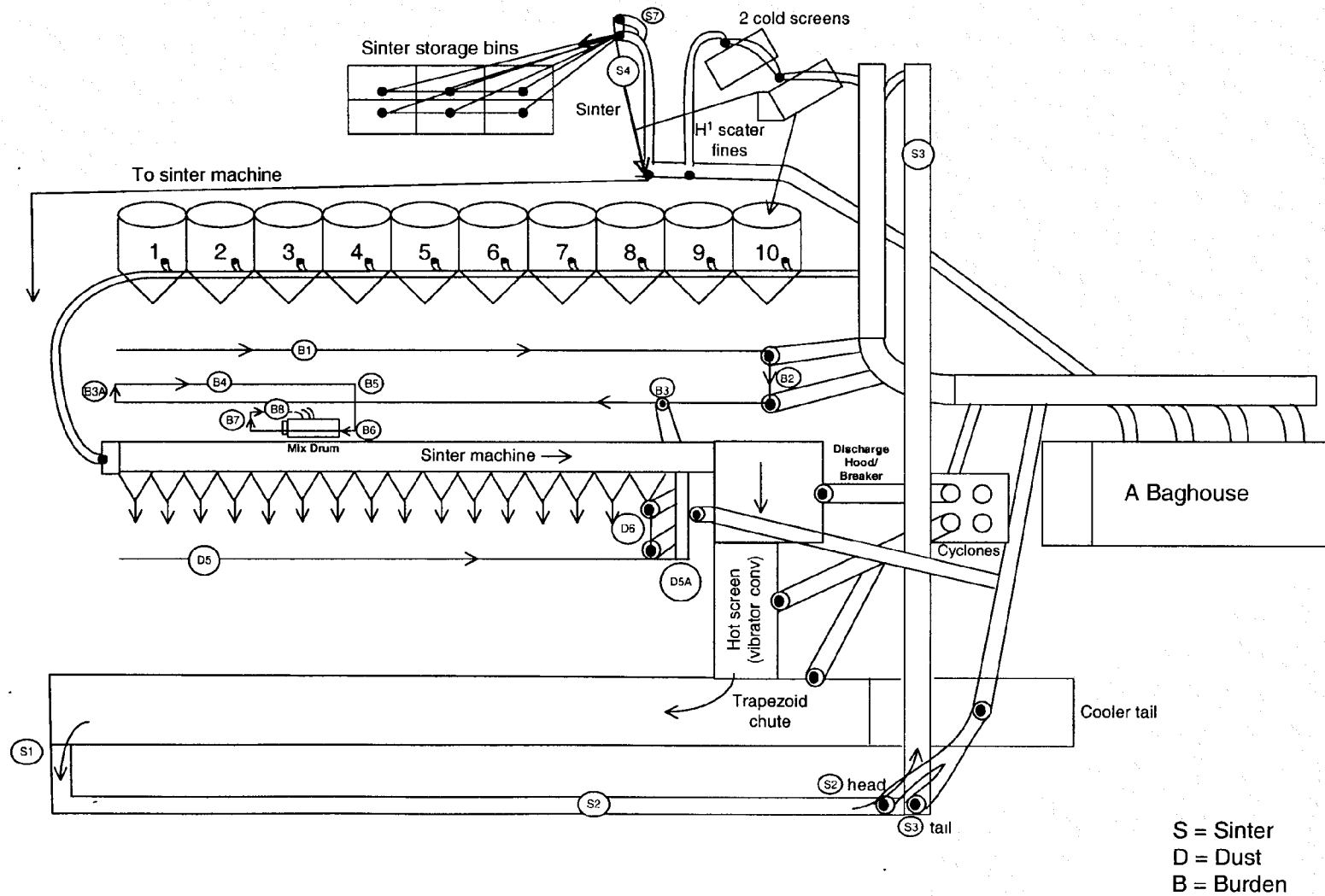


Figure 2-2. Schematic of pick-up points for a baghouse.

TABLE 2-4. PROCESS PARAMETER RANGES DURING THE TESTS

Parameter	Test 1 (8/12/97)	Test 2 (8/13/97)	Test 3 (8/14/97)	Test 4 (8/15/97)
Feed rate:				
Pre-blend (ore) (tons/hour)	120	120	120	120
Limestone (tons/turn)	144	114	167	
Dolomite (tons/turn)	43	39	43	
Coke fines (tons/turn)	19	17	18	
Cold fines (tons/turn)	1738	1545	1787	
Other parameters:				
Percent water	7.0 - 7.2	6.7 - 7.6	6.8 - 7.0	6.7 - 6.8
Grate speed (feet/min)	—	—	—	6.3 - 7.0
Windbox 1 temperature (F)	177-211	150-202	157-207	166-220
Windbox 1 draft (in. H ₂ O)	18.0-22.1	20.3-23.5	19.5-22.3	19.5-21.8
Windbox 3 temperature (F)	167-195	108-186	149-181	159-198
Windbox 3 draft (in. H ₂ O)	16.2-20.3	18.6-21.5	18.1-20.5	18.0-20.1
Windbox 13 temperature (F)	187-266	184-233	169-231	165-342
Windbox 13 draft (in. H ₂ O)	—	—	—	—
Windbox 18 temperature (F)	327-463	251-459	288-457	301-521
Windbox 18 draft (in. H ₂ O)	14.7-18.3	16.6-19.9	15.7-18.5	16.0-17.8
Windbox 19 temperature (F)	396-542	357-513	350-460	363-545
Windbox 19 draft (in. H ₂ O)	16.4-21.1	18.4-21.9	18.0-20.4	17.2-20.5
Windbox 20 temperature (F)	373-580	391-546	372-496	385-545
Windbox 20 draft (in. H ₂ O)	14.5-18.9	17.0-20.7	16.2-18.9	16.5-18.6
Windbox 21 temperature (F)	—	360-465	332-429	355-443
Windbox 21 draft (in. H ₂ O)	14.9-17.7	15.7-19.3	15.1-17.5	15.3-17.2
Cooling Fan Temperatures (°F)				
A	420-463	411-460	395-415	376-413
B	505-546	405-544	456-530	456-507
C	430-460	205-458	372-440	385-435
D	185-243	116-237	157-200	172-192

TABLE 2-5. CONTROL DEVICE OPERATING PARAMETERS — WINDBOX BAGHOUSE

Parameter	Test 1 (08/12/97)	Test 2 (08/13/97)	Test 3 (08/14/97)	Test 4 (08/15/97)
Pressure drop (in. H ₂ O)	9.30-11.87	10.60-12.59	11.61-13.57	12.09-14.12
Inlet Temp. (F)	242 - 265	217-253	211-245	217-236
Stack Temp. (F)	243 - 248	231-248	216-243	227-248
Fan amps	684 - 735	667-690	667-694	659-690
Damper (%)	88.9-90.1	89.5-91.2	88.8-90.9	89.0-90.8

TABLE 2-6. PRESSURE DROP ACROSS EACH COMPARTMENT
OF THE WINDBOX BAGHOUSE

Compartment Pressure Drop	Test 1 (08/12/97)	Test 2 (08/13/97)	Test 3 (08/14/97)	Test 4 (08/15/97)
1	7.0-8.6	6.8-9.3	7.0-9.6	8.6-9.9
2	8.2-9.2	6.7-9.6	6.9-9.8	8.0-10.0
3	7.1-8.6	8.6-9.8	9.4-10+	9.9-10+
4	5.6-8.0	6.8-8.8	7.4-9.8	7.9-10+
5	7.1-8.5	8.0-9.8	9.1-10+	10.0-10+
6	6.6-7.9	7.8-9.3	8.3-9.9	8.9-10+
7	6.4-8.0	7.1-9.4	8.9-10.0	9.7-10+
8	6.7-8.4	6.0-8.8	7.7-9.7	7.2-10+
9	7.6-9.4	8.6-9.9	9.4-10+	9.5-10+
10	7.1-9.0	7.8-9.7	9.3-10+	9.9-10+
11	6.8-8.9	7.3-9.4	8.5-10+	8.2-10+
12	7.6-9.4	8.8-10+	9.6-10+	10+
13	6.4-9.0	7.6-10+	9.8-10+	10.0-10+
14	6.4-9.2	7.6-10+	9.4-10+	8.5-10+
Total	9.9-11.5	10.0-11.5	11.4-12.3	12.0-13.0

During each run of testing performed on A baghouse, the pressure drops of each compartment and the pressure drop across the baghouse were monitored periodically, generally every 20 to 30 minutes. The plant does not monitor any other parameters on A baghouse; since the A baghouse is responsible for the capture and control of dust sources throughout the sintering process, malfunctions are readily apparent. Table 2-7 presents a summary of the pressure drops of each compartment and the pressure drop across the baghouse during each test period.

2.5 ANALYSIS OF MONITORING AND TEST RESULTS

Table 2-8 summarizes the emission results for each run for key pollutants from the outlet of the control device on the sinter strand, along with selected parameters that were monitored during the test. Only a few comparisons can be made because the process operated stably and consistently during the 3 test runs. One difference is that the pressure drop across the strand baghouse increased over the four days of testing, from an average of 10.78 on the first day of testing, to an average of 13.48 on the final day of testing. However, the results were fairly stable and did not appear to be impacted by the increased pressure drop over the course of testing.

Table 2-9 presents emission results for each run for key pollutants from the A baghouse outlet.

Particulate matter and HAP metal emissions were fairly steady over three runs. One interesting factor is that while particulate matter emissions during Run 2 were three times lower than during Run 1, and two times lower than during Run 3, HAP metal emissions were steady over the course of the three runs. The major metal HAPs that were found were lead and manganese; both were effectively captured and controlled by both the Strand baghouse and A baghouse.

Another interesting result is the very low emission rate of dioxins, relative to what had been reported from testing at German sinter plants. The German study reported concentrations of 23 to 68 ng TEQ/m³ from their initial studies and a range of 5 to 10 ng TEQ/m³ for plants that optimized and improved their operation. The results for this sinter plant was much lower, with an average concentration of 0.807 ng TEQ/m³. On the basis of sinter production, the Germans reported emission levels in the range of 10 to 100 µg/Mg of sinter compared to a measured level of 0.6 µg/Mg of sinter for this plant. The WCI sinter plant had emissions of dioxins and furans that were on the order of 10 to 100 times less than that reported for German sinter plants.

TABLE 2-7. PRESSURE DROP ACROSS EACH COMPARTMENT OF "A" BAGHOUSE

Compartment	Test 1 (08/15/97)	Test 2 & 3 (08/16/97)
1	2.6-3.8	3.0-4.7
2	2.8-3.7	3.7-5.5
3	4.7-5.5	1.5-2.0
4	4.4-6.0	5.5-7.4
Total	7.7-8.1	7.9-10.9

TABLE 2-8. STRAND BAGHOUSE SUMMARY OF RESULTS FOR EACH TEST RUN

Test Day	Units	Day 1	Day 2	Day 3	Day 4	Average
Sinter production	tons/hour	110	110	110	110	110
Baghouse ΔP	in. H ₂ O	10.78	12.00	12.88	13.48	12.28
Windbox 20 Temp.	F	474	467	446	457	461
Baghouse Inlet Temp.	F	252	240	230	231	238
Baghouse Outlet Temp.	F	246	240	230	238	238
Parameter	Units	Run 1	Run 2	Run 3	Runs 4 & 5	Average
PM ^a — outlet	lb/hr	2.35	0.71	1.30	Not necessary to do more than 3 runs	1.45
Pb — outlet	lb/hr	0.0220	0.0209	0.0229		0.0219
Mn — outlet	lb/hr	0.0080	0.0661	0.0158		0.0300
HAP metals — outlet	lb/hr	0.0628	0.1224	0.0681		0.0845
		Runs 1 & 2	Run 3	Run 4	Run 5	Average
Dioxin/furan congeners ^b	g/hr	Questionable data; unacceptable leak checks	2,142	2,444	2,186	2,257
Dioxin/furan TEQ ^c	g/hr		342	404	375	374
7 PAHs ^d	g/hr		28.90	34.75	33.88	32.51
16 PAHs	g/hr		510	457	575	514
Total PAHs	g/hr		691	634	755	693

^a PM = particulate matter^b D/F congeners are those dioxins and furans that have a toxicity equivalent factor relative to 2,3,7,8-TCDD^c D/F TEQ is the toxicity equivalent expressed relative to 2,3,7,8-TCDD^d PAH = polycyclic aromatic hydrocarbons

TABLE 2-9. A BAGHOUSE SUMMARY OF RESULTS FOR EACH TEST RUN

Parameter	Units	Run 1	Run 2	Run 3	Average
PM ^a — outlet	lb/hour	0.53	0.67	0.26	0.48
Mn — outlet	lb/hour	0.0033	0.036	0.016	0.019
HAP metals — outlet	lb/hour	0.012	0.046	0.028	0.029

^a PM = particulate matter

The dioxin results are not unexpected because there are basic differences between the operation of WCI's sinter plant and the German plants. The German study attributed the formation of dioxin to the presence of chlorinated organics, primarily in cutting oils, that were in the waste materials fed to the sintering process. In addition, they stated that the use of electrostatic precipitators contributed to recombination and formation of dioxin. In contrast, the WCI plant, like most U.S. integrated plants, has eliminated the purchase and use of chlorinated organics in their facility. Their rolling mill oils (lubricants and hydraulic fluids) do not contain chlorinated compounds. In addition, routine analysis of waste materials going to the sinter plant have not detected chlorinated solvent. Finally, the WCI plant does not use an electrostatic precipitator. Consequently, dioxin rates at WCI that are much lower than those reported by German sinter plants appear to be reasonable and explainable.

A surprising result is the emission rate of polycyclic aromatic hydrocarbons (PAHs) that was measured during the testing. Emissions for PAHs were slightly higher than particulate matter emissions from the outlet of the strand baghouse. These results were consistent over all test runs; even though the first two test runs resulted in questionable data, the results still are consistent with the remaining three test runs. It is not known if the higher emissions were present in the inlet stream or if the baghouse performed poorly in the capture and control of PAHs emissions, since inlet testing for PAHs was not performed. The major PAHs present in the outlet stream were naphthalene and 2-methylnaphthalene, with 3,660 and 2,920 pounds per year being emitted respectively.

Table 2-10 presents a summary of particulate matter and metal HAP results for the strand baghouse, including concentrations, efficiencies, annual emission rates, and emissions factors for each metal HAP. Table 2-11 presents similar results for polycyclic aromatic hydrocarbons and dioxins and furans. Table 2-12 presents a summary of results for the A baghouse for particulate

matter and metal HAPs. The information contained in Tables 2-11 and 2-12 does not contain efficiencies since inlet testing was not performed.

TABLE 2-10. STRAND BAGHOUSE SUMMARY OF RESULTS FOR PARTICULATE MATTER AND METAL HAPS

Pollutant — Particulate Matter	Inlet		Outlet		Efficiency	Annual Rate, ^b tpy		Emission Factor (lb/ton of sinter)	
	lb/hr	g/dscm	lb/hr	g/dscm	%	Inlet	Outlet	Inlet	Outlet
	1,520	1.23	1.45	0.0014	99.9	5,700	5.36	13.8	0.013
Pollutant — HAP Metals	Concentration ($\mu\text{g/dscm}$)		Emission rate (g/hr)		Efficiency (%)	Annual rate (tpy)		Emission factor (lb/t sinter)	
	Inlet	Outlet	Inlet	Outlet		Inlet	Outlet	Inlet	Outlet
Mercury	6.23	5.02	3.5	2.35	32.5	0.03	0.02	7.0×10^{-5}	4.7×10^{-5}
Arsenic	8.27	0.452	4.6	0.21	95.4	0.04	0.00	9.3×10^{-5}	4.2×10^{-6}
Beryllium	0.075	0.038	0.04	0.02	57.7	0.00	0.00	8.4×10^{-7}	3.6×10^{-7}
Cadmium	32.2	0.180	18.0	0.08	99.5	0.15	0.00	3.6×10^{-4}	1.7×10^{-6}
Cobalt	9.35	0.135	5.2	0.06	98.8	0.04	0.00	1.0×10^{-4}	1.3×10^{-6}
Chromium	90.2	4.47	50.5	2.09	95.9	0.41	0.02	1.0×10^{-3}	4.2×10^{-5}
Manganese	2230	29.1	1,247	13.62	98.9	10.16	0.11	2.5×10^{-2}	2.7×10^{-4}
Nickel	18.3	2.07	10.2	0.97	90.5	0.08	0.01	2.0×10^{-4}	1.9×10^{-5}
Lead	7153	21.3	4,001	9.97	99.8	32.61	0.08	8.0×10^{-2}	2.0×10^{-4}
Antimony	2.48	1.21	1.4	0.57	59.3	0.01	0.00	2.8×10^{-5}	1.1×10^{-5}
Selenium	23.1	18.0	12.9	8.42	34.7	0.11	0.07	2.6×10^{-4}	1.7×10^{-4}
HAP metals	9,573	82	5,354	38	99.3	44	0.31	1.1×10^{-1}	7.7×10^{-4}

^a PM = particulate matter^b Based on operation for 24 hours per day, 6 days per week, 52 weeks per year (7400 hours/year).

TABLE 2-11. STRAND BAGHOUSE SUMMARY OF RESULTS
FOR PAHS AND DIOXIN/FURANS

Pollutant — Polycyclic Aromatic Hydrocarbons (PAHs)	Outlet		Annual Emissions, Outlet of Control Device ^a	Emission Factor, Sinter Basis Outlet of Control Device
	g/hr	μg/dsc m	tpy	lb/ton
Benzo(a)anthracene	9.79	21.2	0.0799	1.96x10 ⁻⁴
Benzo(a)pyrene	0.956	2.07	0.0078	1.92x10 ⁻⁵
Benzo(b)fluoranthene	4.07	8.81	0.0332	8.16x10 ⁻⁵
Benzo(k)fluoranthene	1.29	2.79	0.0105	2.58x10 ⁻⁵
Chrysene	16.0	34.6	0.1305	3.21x10 ⁻⁴
Dibenzo(a,h)anthracene	<0.273	0.590	0.0022	5.47x10 ⁻⁶
Ideno(1,2,3-cd)pyrene	<0.200	0.433	0.0016	4.01x10 ⁻⁶
7 PAHs (Total)	32.6	70.7	0.266	6.53x10 ⁻⁴
Acenaphthene	8.80	19.0	0.072	1.76x10 ⁻⁴
Acenaphthylene	16.0	34.5	0.1305	3.21x10 ⁻⁴
Anthracene	20.4	44.2	0.1664	4.09x10 ⁻⁴
Benzo(g,h,i)perylene	<0.194	0.419	0.0016	3.89x10 ⁻⁶
Fluoranthene	56.3	122	0.459	1.13x10 ⁻³
Fluorene	18.8	40.3	0.1534	3.77x10 ⁻⁴
Naphthalene	221	478	1.80	4.43x10 ⁻³
Phenanthrene	115	250	0.938	2.30x10 ⁻³
Pyrene	25.3	54.8	0.206	5.07x10 ⁻⁴
16 PAHs (Total)	514	1114	4.19	1.03x10 ⁻²
2-methylnaphthalene	176	382	1.44	3.53x10 ⁻³
2-chloronaphthalene	0.804	1.74	0.0066	1.61x10 ⁻⁵
Benzo(e)pyrene	1.98	4.27	0.0162	3.97x10 ⁻⁵
Perylene	<0.257	0.557	0.0021	5.15x10 ⁻⁶
Total PAHs	693	1503	5.65	1.39x10 ⁻²

TABLE 2-11. (CONTINUED)

Pollutant — Polycyclic Aromatic Hydrocarbons (PAHs)	Outlet		Annual Emissions, Outlet of Control Device ^a	Emission Factor, Sinter Basis Outlet of Control Device
	g/hr	μg/dsc m	tpy	lb/ton
Pollutant — Dioxin/Furans	Outlet		Annual Emissions, Outlet of Control Device ^a	Emission Factor, Sinter Basis Outlet of Control Device
	μg/hr	ng/dsc m	grams/year	lb/ton
D/F congeners ^b	2,257	4.877	16.70	5.11x10 ⁻⁸
D/F TEQ ^c	374	0.807	2.77	8.48x10 ⁻⁹

^a Based on operation for 24 hours per day, 6 days per week, 52 weeks per year (7400 hours/year).

^b D/F congeners are those dioxins and furans that have a toxicity equivalent factor relative to 2,3,7,8-TCDD.

^c D/F TEQ is the toxicity equivalent expressed relative to 2,3,7,8-TCDD.

**TABLE 2-12. DISCHARGE END BAGHOUSE ("A") -- RESULTS FOR
PARTICULATE MATTER AND METAL HAPS**

Pollutant — Particulate Matter	Outlet		Emissions^b	Emission Factor
	lb/hr	gr/dscf	tpy	lb/ton sinter
PM ^a	0.48	0.0007	1.8	0.0044
Pollutant — Metal HAPs	Outlet		Emissions^b	Emission Factor
	g/hr	µg/dscm	tpy	lb/ton sinter
Arsenic	0.10	0.755	0.0008	2.4 x 10 ⁻⁶
Beryllium	0.013	0.098	0.0001	2.6 x 10 ⁻⁷
Cadmium	0.017	0.126	0.0001	3.4 x 10 ⁻⁷
Cobalt	0.039	0.292	0.0003	7.8 x 10 ⁻⁷
Chromium	1.2	8.92	0.0099	2.4 x 10 ⁻⁵
Mercury	0.29	2.13	0.0024	5.8 x 10 ⁻⁶
Manganese	8.4	62.3	0.070	1.7 x 10 ⁻⁴
Nickel	1.0	7.59	0.0084	2.0 x 10 ⁻⁵
Lead	1.1	7.88	0.0086	2.2 x 10 ⁻⁵
Antimony	0.48	3.57	0.0040	9.6 x 10 ⁻⁶
Selenium	0.43	3.21	0.0036	8.6 x 10 ⁻⁶
HAP metals	13.1	96.9	0.11	2.6 x 10⁻⁴

^a PM = particulate matter.

^b Based on operation for 24 hours per day, 6 days per week, 52 weeks per year (7400 hours/year).

3.0 TEST LOCATIONS

Figures 3-1 and 3-2 are drawings of the baghouse inlet and outlet test locations. The baghouse outlet stack and the baghouse inlet duct were sampled from the same trailer position.

3.1 BAGHOUSE INLET DUCT

The inlet location is a rectangular, horizontal, steel duct approximately 35 feet (ft) above the ground level. The duct dimensions are 11 ft inside height by 10 ft inside width.

One 3-in. test port on the top of the duct was used for the FTIR and Method 25A sampling. Other ports installed on the side walls of the duct were used for the manual testing.

3.2 BAGHOUSE OUTLET (STACK)

The test platform and test ports on the stack were located approximately 60-70 ft above ground level. Access to the stack platform was provided by a ladder on the exterior of the stack.

One 3-in. test port was installed 2 ft above the platform for the FTIR and Method 25A sampling. The four existing ports in the stack were used for the manual testing.

3.3 VOLUMETRIC FLOW

Table 3-1 summarizes the gas composition and flow data provided by ERG. ERG provided volumetric flow rates, moisture content, gas molecular weight, etc. as part of their manual testing; therefore, MRI did not conduct these tests.

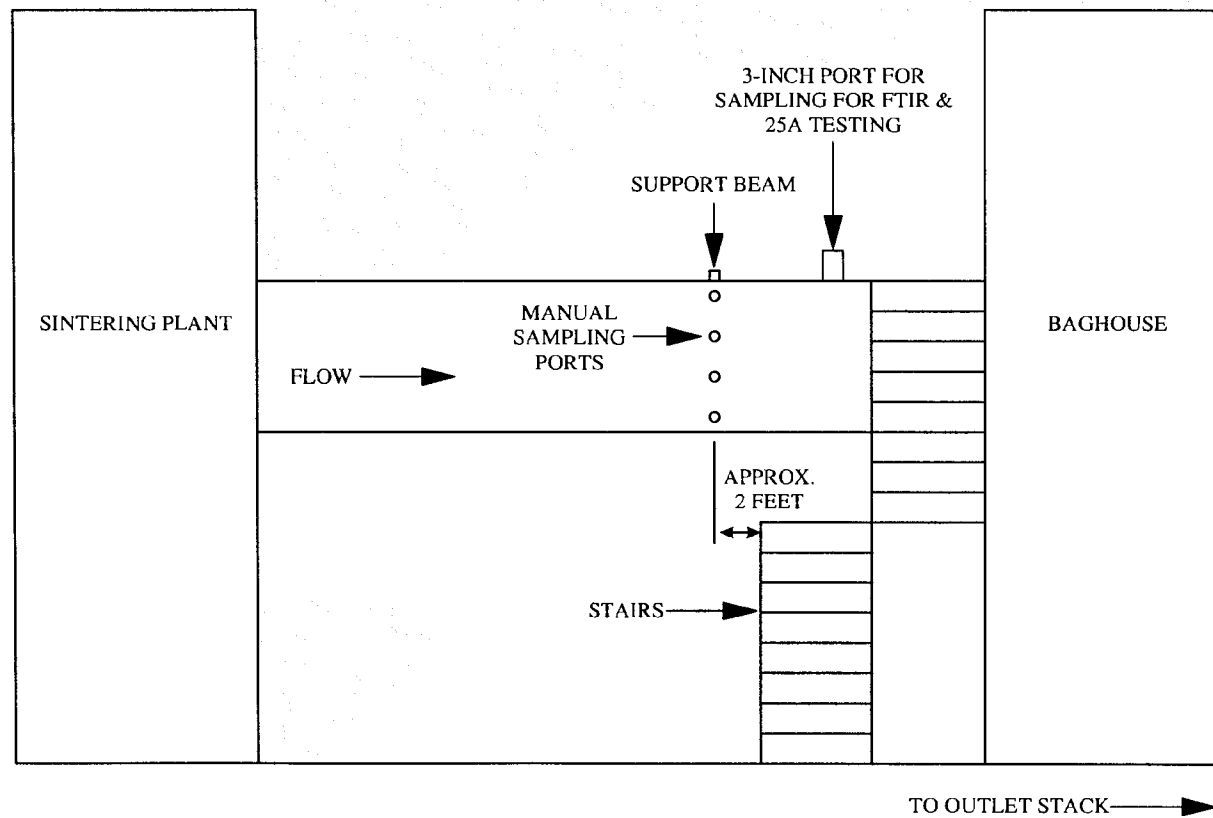


Figure 3-1. Test locations on the baghouse inlet duct.

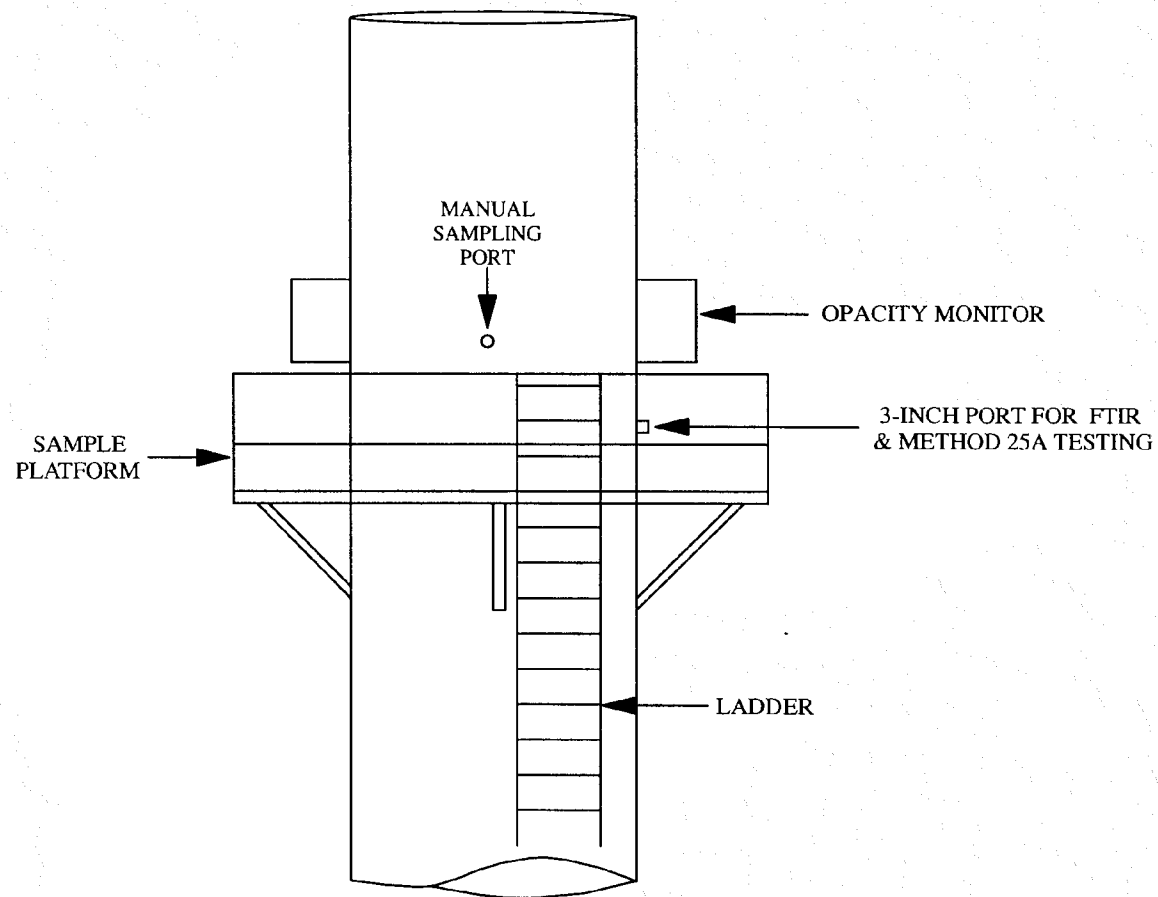


Figure 3-2. Test locations on the baghouse outlet stack.

TABLE 3-1. SOURCE GAS COMPOSITION AND FLOW SUMMARY

Test Data			
Run Number	1	2	3
Date	12-Aug-97	13-Aug-97	14-Aug-97
Baghouse inlet duct			
Oxygen, %	18.5	18.5	18.0
Carbon Dioxide, %	2.0	2.0	2.0
Moisture Content, %	6.2	5.1	6.7
Gas Stream Velocity, fpm	5,375	4,822	5,116
Volumetric Flow Rate, dscfm ^a	302,348	287,397	301,113
Baghouse outlet (stack)			
Oxygen, %	19.0	18.5	17.5
Carbon Dioxide, %	2.0	2.5	3.5
Moisture Content, %	7.4	7.0	6.5
Gas Stream Velocity, fpm	6,463	6,255	6,235
Volumetric Flow Rate, dscfm ^b	277,347	271,179	274,572

^a The flow data sheets in Appendix A incorrectly show the stack cross-sectional area of 9,160 in² and an incorrect volumetric flow was calculated. The correct inlet duct cross-sectional area was 12,690 in². This was confirmed in verbal communications with ERG, Inc., on 2/18/98. The corrected inlet duct cross-sectional area was used to calculate the inlet volumetric flow rates shown.

^b The outlet flows in Table 3-1 are averages of the two or three manual train results from each run.

4.0 RESULTS

4.1 TEST SCHEDULE

The test program at WCI Steel was run from August 11 to August 14, 1997. Table 4-1 summarizes the sampling schedule. A complete record of all Method 25A and FTIR sampling is in Appendices A and B. The FTIR and Method 25 A sampling was coordinated with the manual sampling conducted by ERG.

TABLE 4-1. TEST SCHEDULE AT WCI STEEL

Date	Task ^a
8/11/97	Arrive on site, attend safety meeting, and setup at inlet and outlet.
8/12/97	Test Run 1. FTIR and Method 25A in conjunction with manual methods by ERG. 13:35-20:00
8/13/97	Test Run 2. FTIR and Method 25A in conjunction with manual methods by ERG. 10:55-17:37
8/14/97	Test Run 3. FTIR and Method 25A in conjunction with manual methods by ERG. 08:10-10:29 Pack equipment and depart site

^a All of the testing was conducted at the baghouse inlet and outlet, which are described in Section 2.2.

4.2 FIELD TEST PROBLEMS AND CHANGES

During Run 2 the FTIR mercury/cadmium/telluride (MCT) liquid nitrogen cooled detector warmed up, and some data were lost. No results are reported for this period because the spectra could not be analyzed. These spectra are included on the data back-up disk and are noted in the data records. The MCT detector warming up had no effect on the 25A results, which are reported for the same period.

Run 3 was abbreviated to about two hours because the manual sampling runs could not be completed that day. The manual runs were redone the following day, but the EPA observer decided that enough FTIR data had already been collected and did not require MRI to continue testing on the following (fourth) day.

4.3 METHOD 25A RESULTS

Table 1-2 summarizes the Method 25A total hydrocarbon (THC) results at the baghouse inlet and outlet. The mass emissions data are presented as both THC and total gaseous non-methane organic (TGNMO) carbon. The TGNMO was calculated using the procedures outlined in Section 5.6.2 of this report using methane concentrations from the FTIR analysis.

The THC emissions were fairly steady during each test run except Run 3 at the baghouse outlet. Table 4-2 shows the minimum and maximum 1-minute average THC concentrations, as carbon, and the average concentration for each test run. At the baghouse inlet, the THC concentration ranged from 199.8 ppm carbon during Run 1 to 287.4 ppm carbon also during Run 1. At the baghouse outlet, the THC concentration ranged from 66.9 ppm carbon during Run 3 to 242.7 ppm carbon during Run 1. Without accounting for process variations during the testing periods, no absolute determinations can be made.

TABLE 4-2. MINIMUM AND MAXIMUM AND AVERAGE THC CONCENTRATIONS (ppmc)

Run No.	Minimum	Maximum	Average
<u>Baghouse inlet</u>			
1	199.8	287.4	248.7
2	208.5	265.2	244.2
3	204.3	262.8	220.8
<u>Baghouse stack</u>			
1	153.9	242.7	197.7
2	174.6	225.3	211.5
3	66.9	207.3	172.5

The complete Method 25A results are included in Appendix A. The concentrations presented were measured by MRI, and the mass emissions data, presented in Section 1.2, were calculated using volumetric flow results provided by ERG. The pre- and post-run calibrations and QA checks met the Method 25A criteria in all cases. Calibration QA results are included in Appendix A.

4.4 FTIR RESULTS

A summary of the FTIR results is presented in Table 1-1. Complete FTIR results at the inlet and outlet are presented in Tables B-1 to B-2 in Appendix B. The infrared spectra showed evidence of water vapor, carbon dioxide (CO_2), CO, methane, formaldehyde, sulfur dioxide (SO_2), toluene, ethylene, hexane, hydrogen chloride (HCl), 2-methyl-2-butene, and ammonia. The FTIR results are from a revised analysis that included reference spectra of eight additional hydrocarbon compounds. 2-methyl-2-butene was the only additional hydrocarbon compound detected, but the revised hexane and toluene concentrations are slightly lower compared to the draft report results. A description of the analytical procedures used to prepare the FTIR results is given in Section 5.4. The mass emission rates were calculated using flow data provided by ERG. Mass emission calculations for toluene include only the results from unspiked samples.

Initially, the computer program was set to report as zero any concentration that was less than four times the uncertainty. After the initial analysis there was clear evidence of HCl remaining in some of the residual spectra. The evidence for HCl is shown in the expanded portion of Figure 4-1 at the end of Section 4. The analytical program was then modified to measure HCl and to report any concentrations that were greater than 1.0 times the uncertainty. Note that in analysis both the calculated concentrations and uncertainties were the same. The only mathematical difference between the analyses was in how many of the concentrations the program actually reported (the other difference is that the first analysis did not account for HCl at all). The second analysis detected HCl only in the first run. It was first measured at the inlet about 3:00 PM, which was just prior to a process shut down at about 3:20 PM. The HCl was then measured for the remainder of the run at both locations even after the process restarted. Note that the calculated uncertainty for HCl is very similar for all of the spectra. The HCl concentrations reported Tables B-1 and B-2 were during periods when the HCl concentrations exceeded 1.0 times the uncertainties.

4.5 ANALYTE SPIKE RESULTS

For quality control a toluene gas standard was used for analyte spiking experiments. Preferably, a spike standard combines the analyte and the tracer gas in the same cylinder, but the SF_6 and toluene were contained in two separate cylinders. Therefore, the two components (SF_6 and toluene) were quantitatively mixed before being introduced into the sample gas stream.

The analyte spike results are presented in Table 4-3. Samples were spiked with a measured flow of toluene vapor during each run and at each location. The SF₆ tracer gas was spiked into the gas stream to determine the spike dilution factor. A description of the spike procedure is given in Section 5.3.1.

In most cases the calculated spike recoveries (using deresolved reference spectra from the EPA library) were greater than 130 percent, which is above the range allowed by Method 301 for a validation correction factor (between 70 and 130 percent). This does not reflect on the accuracy of the emissions results reported in Appendix B. The residual spectra (Figure 4-1), which show no significant (or negative) remaining absorbances, indicate that the computer program correctly measured the absorbances from the interfering species and the analytes.

An important factor contributing to the (calculated) high spike recoveries relates to the use of the toluene reference spectra. Method 320 specifies that library reference spectra be used in the spectral analysis. The toluene spike recoveries and all of the toluene results were obtained using reference spectra in the EPA library. Spectra of the toluene (spike) cylinder standard were recorded on site during the test. If these on-site spectra are used in the analysis, one obtains results about 38 percent lower (far right column in Table 4-3) than those obtained using the reference spectra.

TABLE 4-3. SUMMARY OF SPIKE RESULTS

Baghouse Inlet

Run	Toluene			SF ₆				Cexp	Tol (calc) Cexp	Library spectra ^a	Standard spectra ^b
	Average		Tol(calc)	Average		spike - unspike	DF			% Recovery	% Recovery
	Spike	Unspike	spike - unspike	Spike	Unspike						
1	26.0	7.1	18.9	0.361	0.000	0.361	5.6	10.9	8.0	174	107
2	43.9	9.5	34.3	0.367	0.000	0.367	3.6	22.2	12.1	155	95
3	40.1	9.7	30.4	0.346	0.000	0.338	4.0	20.4	10.0	149	92

Baghouse Outlet (Stack)

Run	Toluene			SF ₆				Cexp	Tol (calc) Cexp	Library spectra ^a	Standard spectra ^b
	Average		Tol(calc)	Average		spike - unspike	DF			% Recovery	% Recovery
	Spike	Unspike	spike - unspike	Spike	Unspike					% Recovery	% Recovery
1	23.1	8.5	14.6	0.297	0.000	0.297	6.8	9.0	5.6	163	100
2	39.0	7.5	31.5	0.328	0.000	0.328	4.1	19.8	11.7	159	98
3	40.3	10.8	29.6	0.322	0.000	0.322	4.2	19.4	10.1	152	94

Calculations of the dilution factor, DF and the expected toluene spike concentration, Cexp, are described in Section 5.3.

^a These recoveries were obtained using EPA library reference spectra for toluene.

^b These were obtained using spectra of the toluene cylinder standard measured on site.

Table 4-4 presents measured band areas of the EPA toluene reference spectra (deresolved to 2.0 cm⁻¹) and the spectra of the toluene cylinder standard measured while at the WCI test site. The comparison of the band areas does not agree with the comparison of the concentrations (corrected for path length and temperature). The comparisons differ by about 38 percent. This observed difference predicts that, if the spectra of the toluene cylinder standard are used in the analysis rather than the EPA library spectra, then the result would give a toluene concentration that is about 38 percent lower. This in fact happens when the computer program is modified to include the cylinder standard spectra.

This type of discrepancy is compound specific and the information in Table 4-4 does not apply to the results for any of the other compounds detected. In fact, the deresolved CTS (ethylene calibration) spectra give a path length result (Section 5.4.1) that is consistent with the observed number of laser passes and the instrument resolution. Additionally, this observed

TABLE 4-4. COMPARISON OF EPA REFERENCE SPECTRA TO SPECTRA OF TOLUENE CYLINDER STANDARD^a

Toluene Spectra	Source	Band Area	Region (cm ⁻¹)	Spectra comparison based on band areas		Comparison of spectra based on standard concentrations		
				Ratio (Ra)	=1/Ra	(ppm-m)/K	Ratio (Rc)	=1/Rc
153a4ara (2.0 cm ⁻¹)	EPA library	23.4	3160.8 - 2650.1	5.4	0.184	4.94	4.8	0.210
153a4arc (2.0 cm ⁻¹)	EPA library	4.3		1.0	1.000	1.04	1.0	1.000
ToldirA	WCI	21.2		4.9	0.203 ^a	3.13	3.0	0.332 ^a
ToldirB	WCI	21.1		4.9	0.204	3.13	3.0	0.332
ToldirC	WCI	21.0		4.9	0.205	3.13	3.0	0.332
153a4ara (2.0 cm ⁻¹)	EPA library	12.1	761.9 - 670.1	4.8	0.210	4.94	4.8	0.210
153a4arc (2.0 cm ⁻¹)	EPA library	2.6		1.0	1.000	1.04	1.0	1.000
ToldirA	WCI	11.0		4.3	0.232	3.13	3.0	0.332
ToldirB	WCI	11.2		4.4	0.228	3.13	3.0	0.332
ToldirC	WCI	11.2		4.4	0.227	3.13	3.0	0.332

^a The relevant comparison is Rc/Ra for spectra "ToldirA, ToldirB, and ToldirC" (about 61 percent).

discrepancy is not an artifact of the deresolution procedure because the band areas in the original 0.25 cm⁻¹ toluene spectra are nearly equal to the band areas in the deresolved 1.0 cm⁻¹ and 2.0 cm⁻¹ versions of these spectra.

A discrepancy of this type has the greatest affect on the difference, "spike - unspike" when the unspiked concentration is near zero. This is because two sets of reference spectra that disagree will yield the same answer for a zero concentration, but they will yield different answers for nonzero concentrations.

A similar disagreement between reference and standard spectra has been observed at least once previously.⁴ In that study, which is included in Appendix D, HCl was the analyte. The spike recovery results were not significantly affected because there was a stable unspiked HCl concentration and because both the spiked and unspiked HCl concentrations were large compared to the disagreement between the reference spectra and the spectra of the cylinder standard.

4.6 ESTIMATED UNCERTAINTIES

Estimated uncertainties for undetected HAP's are reported in Table 4-5. The spectra were analyzed for the target analytes listed in the test request and for other, principally hydrocarbon, species, which are in the EPA library of FTIR reference spectra. The procedure for estimating the uncertainties is described in Section 5.4. The compounds for which the spectra were analyzed and the analytical region(s) for each compound are given in Section 5.4. The reported uncertainties can be interpreted as the practical measurement limits imposed by the sampling conditions. The method of calculating uncertainties was identical to that used for the compounds reported in Appendix B and depends on the noise in the residuals (Figure 4-1).

TABLE 4-5. AVERAGE UNCERTAINTIES (ppm) OF UNDETECTED ANALYTES AT WCI STEEL

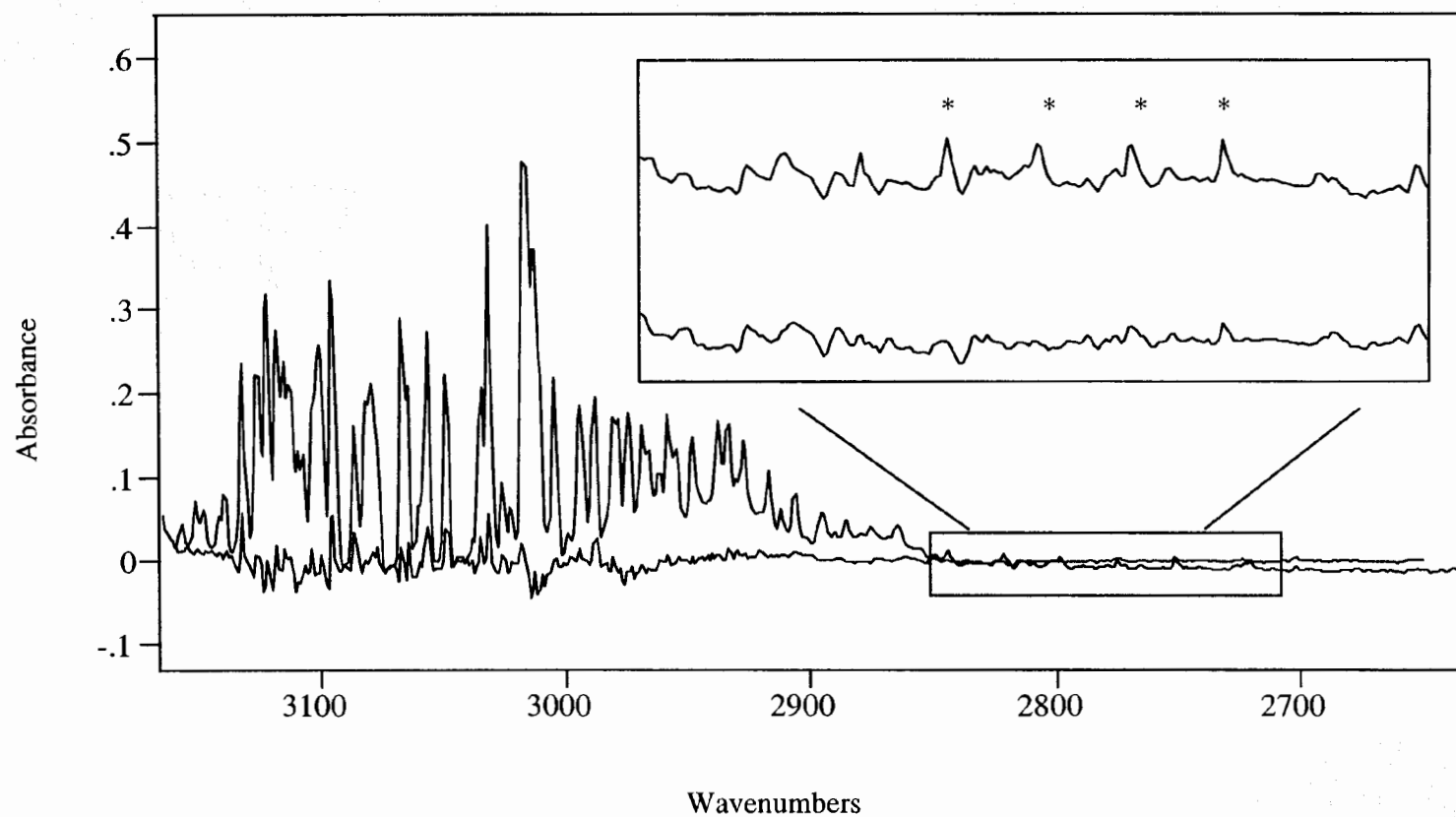
Compound ^a	Run 1		Run 2		Run 3	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Benzene (ch)	3.51	3.46	3.24	3.26	2.94	2.88
Methyl bromide (fp)	11.99	12.02	11.72	11.85	10.89	10.74
Methyl chloride (ch)	9.99	9.86	9.24	9.30	8.37	8.21
Methyl chloroform (fp)	1.00	1.00	0.98	0.99	0.91	0.90
1,1-Dichloroethane (fp)	1.37	1.37	1.34	1.35	1.24	1.23
1,3-Butadiene (fp)	1.40	1.40	1.37	1.38	1.27	1.25
Carbon tetrachloride (fp)	0.24	0.24	0.23	0.24	0.22	0.21
Chlorobenzene (fp)	3.28	3.29	3.21	3.24	2.98	2.94
Cumene (ch)	3.77	3.72	3.49	3.51	3.16	3.10
Ethyl benzene (ch)	9.87	9.74	9.13	9.19	8.27	8.11
Methylene chloride (fp)	2.01	2.02	1.97	1.99	1.83	1.80
Propionaldehyde (ch)	2.14	2.11	1.98	1.99	1.80	1.76
Styrene (fp)	2.73	2.73	2.66	2.69	2.48	2.44
1,1,2,2-Tetrachloroethane (fp)	0.97	0.97	0.95	0.96	0.88	0.87
p-Xylene (fp)	4.24	4.25	4.15	4.19	3.85	3.80
o-Xylene (ch)	7.05	6.96	6.52	6.50	5.91	5.79
m-Xylene (ch)	15.36	15.15	14.20	14.29	12.87	12.61
HCl (ch)	detected	detected	3.2	3.2	2.8	2.8
2,2,4-Trimethylpentane (ch)	0.85	0.84	0.79	0.79	0.71	0.70

^a Analytical Regions

(ch) - 2,650.1 - 3,160.8 cm⁻¹

(fp) - 789.3 - 1275.0 cm⁻¹

Procedure for estimating uncertainties is described in Section 5.4.3



The large figure shows part of spectrum "18120080" and its subtracted residual. The top trace in the expanded view shows the residual spectrum after the initial analysis before the spectra were analyzed for HCl (*). The bottom trace of the expanded view shows the residual spectrum after the second analysis, which measured HCl. This is discussed in Section 3-4.

Figure 4-1. Example of a sample spectrum and its subtracted residual spectrum.

5.0 TEST PROCEDURES

The procedures followed in this field test are described in EPA Method 320 for using FTIR spectroscopy to measure HAP's, the EPA Protocol for extractive FTIR testing at industrial point sources, and EPA Method 25A for measuring total gaseous organics. 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, conduct analyte spiking for quality control, and analyze the spectra for compounds not in the EPA library. Additionally, manual measurements of gas temperature, gas velocities, moisture, CO₂, and O₂ by ERG were used to calculate the mass emissions rates.

The extractive sampling system shown in Figure 5-1 was used to transport sample gas from the test ports to the FTIR instrument and the THC analyzers.

5.1 SAMPLING SYSTEM DESCRIPTION

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 nonreactive 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.

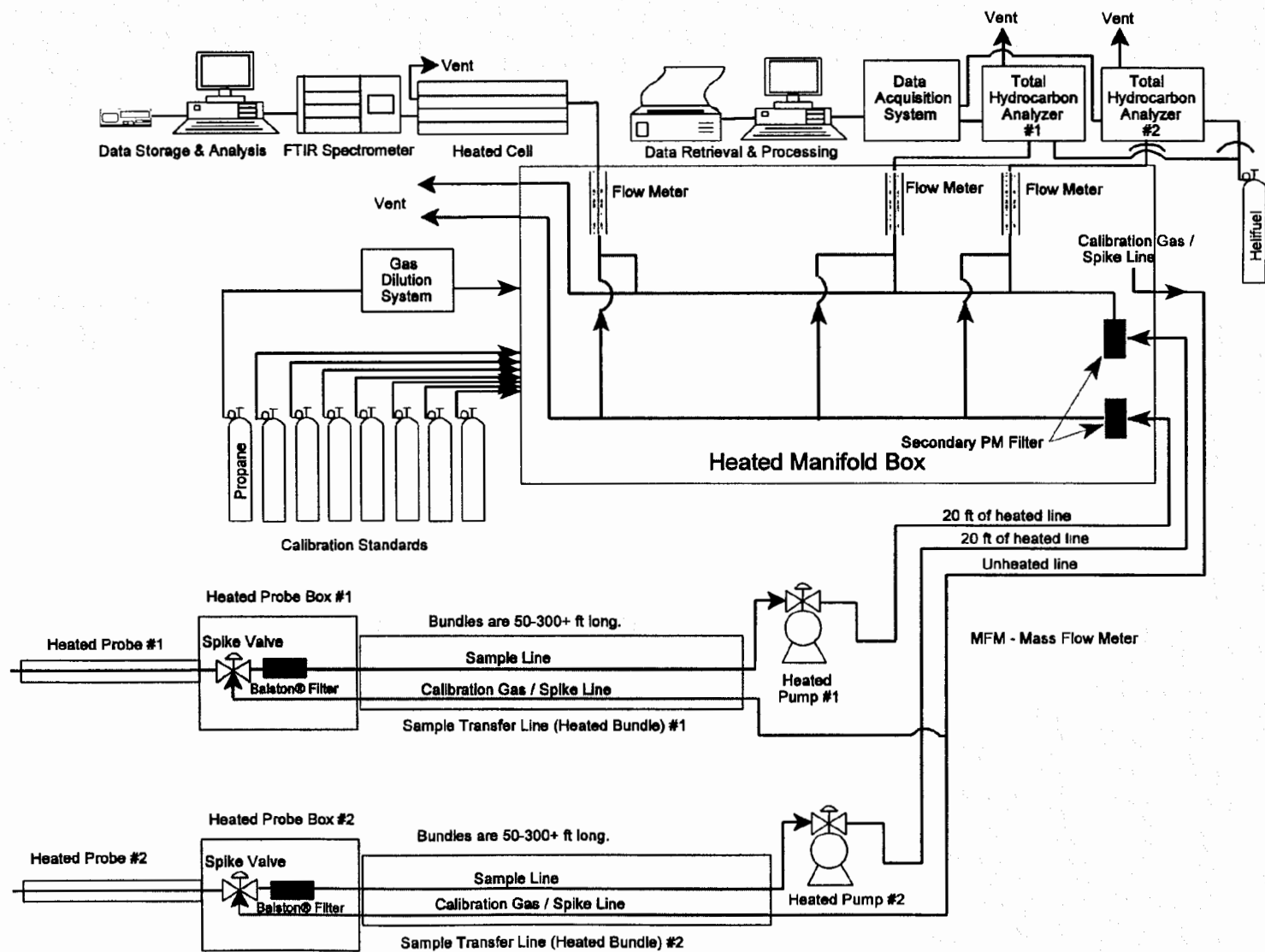


Figure 5-1. Sampling system schematic

The sample lines are standard heated sample lines with three $\frac{3}{8}$ inch Teflon tubes in 10, 25, 50, and 100 ft lengths. The pumps are heated single-head diaphragm pumps manufactured by either KNF Neuberger or Air Dimensions. These pumps are capable of sampling at rates up to 20 Lpm depending on the pressure drop created by the upstream components.

The heated gas distribution manifold was specially constructed for FTIR sampling by MRI. It is built onto a cart that can be operated inside the MRI mobile lab or elsewhere, 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, eight calibration gases, and has three individual outputs for analyzers. Also included on the cart is 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 at both the inlet duct and outlet stack of the Strand baghouse through the sample probe and transported to the gas distribution manifold. Inside the manifold the gas passed through separate secondary particulate filters. Downstream of the secondary filters, a portion of each sample gas stream was directed to separate THC analyzers; one to measure concentration of the inlet sample and another to measure concentration of the outlet sample. A portion of the remaining sample gas from each stream was either sent to the FTIR instrument for analysis or exhausted with the remaining portion of the gas stream being sampled (i.e., when the inlet sample was analyzed, the stack sample was exhausted and visa versa). This was accomplished by rotating the gas selection valves to allow the appropriate sample gas to pass to the instrument inlet port. The gas flow to the instruments was regulated by needle valves on rotameters at the manifold outlets.

The FTIR instrument was used to sample each location alternately, while the two THC analyzers were used to sample both locations simultaneously.

5.2 FTIR SAMPLING PROCEDURES

Figure 5-1 shows a schematic of the FTIR instrument and connections to the sample distribution manifold.

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

5.2.1 Batch Samples

In this procedure, a 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 open to vent the cell to ambient pressure, the spectrum of the static sample was recorded, and then the cell was evacuated for the next sample. This procedure was repeated to collect as many samples as possible during Run 1.

Batch sampling has the advantage that every sample is an independent sample. The time resolution of the measurements is limited by the interval required to pressurize the cell and record the spectrum. For this test the time resolution was 4 to 5 minutes. All of the spiked samples and all of the samples in Run 1 were collected using this procedure.

5.2.2 Continuous Sampling

The cell was filled as in the batch sampling procedure, but the cell inlet and outlet valves were then opened to keep gas continuously flowing through the cell. The inlet and outlet flows were regulated to keep the sample at ambient pressure. The flow through the cell was maintained at about 5 Lpm (about 0.7 cell volumes per min). The cell volume was about 7 L.

The FTIR instrument was automated to record spectra of the flowing sample about every 2 minutes. The analytical program was revised after the field tests and the spectra were analyzed to prepare the results reported in Section 4.

This procedure with automated data collection was used for all of the unspiked testing during Runs 2 and 3. 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) depended on the sample flow rate (through the cell), and the cell volume. The following explanation is taken from Performance Specification 15, for continuous operation of FTIR systems.

“The Time Constant (TC) 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." Therefore,

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

Performance Specification 15 defines $5 * TC$ as the minimum interval between independent samples. In this test, $5 * TC$ was about 7 minutes.

A stainless steel tube ran from the cell inlet connection point to the front 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.

5.3 ANALYTE SPIKING

Since no information about possible HAP emissions or flue gas composition was available for this source before the test, validation of specific HAP's at this test was not planned. MRI conducted spiking for QA purposes using a toluene (121 ppm in air) 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 quality assurance check that the sampling system can transport the spiked analyte(s) 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 validation³. No validation was performed at this field test.

The spike procedure follows Sections 9.2 and 13 of EPA draft 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 comprises about 1/10 or less of the spiked sample. The actual dilution ratio depends on the sample flow rate and the spike gas flow rate. The expected concentration (C_{exp} , the calculated 100 percent recovery) of the spiked component is determined using a tracer gas, 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 (100 percent) of the spiked analyte recovery.

In this test the analyte (121 ppm toluene in air) and the tracer gas (4.01 ppm SF₆ in nitrogen) were in separate cylinders. Flows from the two gas standards were passed through separate mass flow meters and then combined into one flow that was directed up the spike line and introduced into the sample stream at the back of the sampling probe. Because the two gases were mixed, the concentrations of each component were reduced in the combined spike gas flow. This had to be accounted for in the calculation of the spike dilution factor, DF. For example, the SF₆ concentration in the combined spike stream was

$$SF_{6(\text{direct})} = \frac{F_{SF_6}}{F_{\text{toluene}} + F_{SF_6}} * SF_{6(\text{standard})} \quad (2)$$

where:

SF_{6(direct)} = SF₆ in the spike mixture. This is used in place of the cylinder standard concentration.

F_{SF6} and F_{toluene} = the measured flows from the toluene and SF₆ cylinder standards.

SF_{6(standard)} = the concentration of the SF₆ cylinder standard.

The toluene concentration in the combined spike flow is calculated in the same way.

$$\text{toluene}_{(\text{direct})} = \frac{F_{\text{toluene}}}{F_{\text{toluene}} + F_{SF_6}} * \text{toluene}_{(\text{standard})} \quad (3)$$

The value, SF_{6(spike)} is compared to the measured SF₆ concentration in the spiked samples to determine the spike dilution factor:

$$DF = \frac{SF_{6(\text{direct})}}{SF_{6(\text{spike})}} \quad (4)$$

Where DF is the spike dilution factor in Section 9.2.2 of Method 320 and $SF_{6(\text{direct})}$ is calculated using Equation 2.

The calculated 100 percent recovery of the toluene spike is analogous to the expected concentration in Section 9.2.2 of Method 320. In this case:

$$C_{\text{exp}} = \frac{\text{toluene}_{(\text{direct})}}{\text{DF}} \quad (5)$$

where:

C_{exp} = expected toluene concentration in the spiked samples (100 percent recovery).

$\text{toluene}_{(\text{direct})}$ = from Equation 3.

DF = from Equation 4.

5.3.2 Analysis of Spiked Results

The toluene and SF_6 concentrations used in the evaluation of the spike recoveries in Table 4-3 were taken directly from the sample analyses reported in Appendix B. The concentrations in the spiked samples included a contribution from the spike gas and from any analyte present in the flue gas. The component of the toluene concentration attributed to the spike was determined by subtracting the average of the unspiked samples from the measured concentration in each spiked sample ("spiked - unspiked" in Table 4-3). The percent recovery was determined by comparing the differences, spiked - unspiked, to the calculated 100 percent recovery, C_{exp} in Section 5.3.1.

5.4 ANALYTICAL PROCEDURES

Analytical procedures in the EPA FTIR Protocol² were followed for this test. A computer program was prepared with reference spectra shown in Table 5-1. The computer program⁵ used mathematical techniques based on a K-matrix analysis.⁶

TABLE 5-1. PROGRAM INPUT FOR ANALYSIS OF SAMPLE SPECTRA

Compound name	File name	Region No.	ISC ^a	Reference	
				Meters	T (K)
Water	194c1bvh	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	087c1anb	3	100.0	11.25	373
Benzene	015a4ara	3	496.6	3	298
Methane	196c1bsb	3	80.1	22	394
Methyl bromide	106a4asb	2	485.3	3	298
Toluene	153a4arc	3	103.0	3	298
Methyl chloride	107a4asa	3	501.4	3	298
Methyl chloroform	108a4asc	2	98.8	3	298
1,1-dichloroethane	086b4asa	2	499.1	2.25	373
1,3-butadiene	023a4asc	2	98.4	3	298
Carbon tetrachloride	029a4ase	2	20.1	3	298
Chlorobenzene	037a4arc	2	502.9	3	298
Cumene	046a4asc	3	96.3	3	298
Ethyl benzene	077a4arb	3	515.5	3	298
Methylene chloride	117a4asa	2	498.5	3	298
Propionaldehyde	140b4anc	3	99.4	2.25	373
Styrene	147a4asb	2	550.7	3	298
1,1,2,2-tetrachloroethane	150b4asb	2	493.0	2.25	373
p-Xylene	173a4asa	2	488.2	3	298
o-Xylene	171a4asa	3	497.5	3	298
m-Xylene	172a4arh	2	497.8	3	298
Ethylene	CTS0813d	2	20.1	10.4	394
SF ₆	Sf60811a	2	4.01	10.4	394
Ammonia	174a4ast	2	500.0	3	298
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
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.

Initially, the spectra were reviewed to determine suitable input spectra for the computer program. Next an analysis was run on all of the sample spectra using all of the reference spectra listed in Table 5-1. The estimated uncertainty results for the undetected species were reported in Table 4-4. Finally, the undetected compounds were removed from the analysis and the spectra were analyzed again using reference spectra only for the detected compounds. The results from this second analytical run are summarized in Table 1-1 and reported in Appendix B. In addition to the detected compounds shown in Table 5-1, the spectra were analyzed for 2-methyl-2-pentene, 3-methylpentane, butane, 2-methyl-1-pentene, n-heptane, 1-pentene, 2-methyl-2-butene, and n-pentane in the revised analysis.

The same program that performed 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. Finally the computer program calculated the standard 1-sigma uncertainty for each analytical result, but the reported uncertainties are equal to 4*sigma.

The concentrations were corrected for differences in absorption path length and temperature between the reference and sample spectra using equation 6.

$$C_{\text{corr}} = \left(\frac{L_r}{L_s} \right) \left(\frac{T_s}{T_r} \right) C_{\text{calc}} \quad (6)$$

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

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

The ambient pressure recorded over the three days of the test averaged about 746 mm Hg. Because the sample pressure in the gas cell is equivalent to the ambient pressure, an addition concentration correction factor of about 2 percent was included in the reported concentrations.

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 calibration transfer standard (CTS) spectra to CTS 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 WCI field test.

5.4.1 Computer Program Input

Table 5-1 presents a summary of the reference spectra input for the computer program used to analyze the sample spectra. Table 5-2 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 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 5-3 summarizes the results of the CTS analysis. The cell path length from this analysis was used as L_s in equation 2.

5.4.2 EPA Reference Spectra

The toluene spectra used in the MRI analysis were taken from the EPA reference spectrum library (<http://www.epa.gov/ttn/emc/ftir.html>). 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} toluene single beam spectra were combined with their deresolved single beam background spectra and converted to absorbance. This same procedure was used to prepare spectral standards for the HAP's and other compounds that were included in the analysis.

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 10.4 meters using the CTS reference and sample spectra. The interior cell walls have been treated with a Teflon® coating to minimize potential analyte losses. An MCT liquid nitrogen detector was used. The spectra were recorded at a nominal resolution of 2.0 cm^{-1} .

TABLE 5-2. PROGRAM INPUT FOR ANALYSIS AND 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 WCI CTS spectra
Analytical Regions for CTS analysis were 842.5 cm^{-1} -1107 cm^{-1} and 2984.36 cm^{-1} -2992.38 cm^{-1}

TABLE 5-3. RESULTS OF PATH LENGTH DETERMINATION

CTS spectra 20.1 ppm Ethylene	Path length calculations		
	Meters	Delta ^a	% Delta
CTS0811B	10.51	0.10	0.92
CTS0811C	10.49	0.07	0.70
CTS0811D	10.51	0.10	0.93
CTS0813C	10.30	-0.11	-1.09
CTS0813D	10.25	-0.16	-1.56
CTS0814C	10.43	0.02	0.19
CTS0814D	10.49	0.07	0.68
CTS0814E	10.33	-0.08	-0.77
Average Path Length (M)	10.41		
Standard Deviation	0.103		

^a The difference between the calculated and average values.

The optical path length was measured by shining a 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 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 4.4.

5.6 CONTINUOUS EMISSIONS MONITORING FOR TOTAL HYDROCARBONS (THC)

The guidelines set forth in Method 25A were followed during the sampling at WCI with two exceptions. Section 7.2 of Method 25A specifies that the mid-level calibration gas be used for the drift determination. For this test program, the high-level calibration gas was used for the drift determination because it more closely approximated the measured THC concentrations. Also, Section 7.2 of Method 25A specifies an analyzer drift determination hourly during the test period, but this instruction was not followed.

There are two reasons the drift determination was not completed as specified. The first reason is for continuity in the FTIR and THC sampling. With run length exceeding four hours, this hourly drift determination would have involved off-line periods of up to 10 minutes each hour for the THC analyzers and possibly the FTIR instrument. The loss of this time could affect the results if significant process events had occurred during these periods. The second reason is that experience with the analyzers MRI was using show them to be stable over extended periods when they are operated in a climate controlled environment.

The need to do hourly drift determinations is somewhat diminished when the stability of the analyzer is known and when the possibility that being off-line could affect the representativeness of both the FTIR and THC results.

5.6.1 Total Hydrocarbon Sampling Procedures

The THC sampling was conducted continuously from both locations by using of two separate analyzers. The same sample systems used for the FTIR sampling were used for the THC sampling. Sample gas was directed to the analyzers through a separate set of rotameters and control valves. Each test run was conducted from the start to the end of the manual test runs completed by ERG. A summary of specific procedures used is given below.

A brief description of each system component follows.

- **THC Analyzer-** The THC concentration was measured using a flame ionization detector (FID). MRI used two J.U.M. Model VE-7 analyzers. The THC analyzers were operated on the zero to 100 ppm range throughout the test period. The fuel for the FID is 40 percent hydrogen and 60 percent helium mixture.
- **Data Acquisition System-** MRI used LABTECH notebook (Windows version), which is an integrated system that provides data acquisition, monitoring and control. The system normally writes data to a disk in the background while performing foreground tasks or displaying data in real time. The averaging period set for this test was one minute.
- **Calibration Gases-** Calibration gases were prepared from an EPA Protocol 1 cylinder of propane (5278 ppm propane in nitrogen) using an Environics Model 2020 gas dilution system that complies with the requirements of EPA Method 205. High, medium, and low standard gases were generated to perform analyzer calibration checks. The raw data are recorded in ppm as propane but are converted to an as carbon basis for reporting. Appendix C contains copies of mass flow meter calibration records and calibration gas certifications

5.6.2 Hydrocarbon Emission Calculations

The hydrocarbon data is presented as both THC and TGNMO emissions in Table 4-1. To do this the THC emission data was first converted to an as carbon basis using Equation 7, and then the THC emission rate was calculated using Equation 9.

$$C_c = K C_{meas} \quad (7)$$

where:

C_c = organic concentration as carbon, ppmv.

C_{meas} = organic concentration as measured, ppmv.

K = carbon equivalent correction factor, 3 for propane.

The TGNMO concentration was calculated by subtracting the methane concentration measured by the FTIR from C_c (Equation 8). The emission rate was then calculated using Equation 9.

$$C_{\text{TGNMO}} = C_{\text{C}} - C_{\text{CH}_4} \quad (8)$$

where:

C_{TGNMO} = total gaseous nonmethane organic concentration, ppmv.

C_{CH_4} = methane concentration in gas stream, ppmv.

$$E_{\text{TGNMO/THC}} = \frac{\frac{C_{\text{TGNMO/Cc}}}{(1-B_{\text{ws}})} \times \text{MW} \times Q_{\text{std}} \times 60}{385.3 \times 10^6} \quad (9)$$

where:

$E_{\text{TGNMO/THC}}$ = TGNMO or THC mass emission rate, lb/hr.

B_{ws} = moisture fraction in gas stream

MW = molecular Weight of Carbon, 12 lb/lb-mole.

Q_{std} = volumetric Flowrate corrected to standard conditions, dscfm.

60 = conversion to hours, min/hr.

385.3 = molar Volume, ft³/mole at standard conditions.

10⁶ = conversion for decimal fraction to ppm.

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 was 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. Below are listed 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 minutes 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 ($-0.02 \leq \text{absorbance} \leq +0.02$). Whenever 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 CTS spectrum 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 20.1 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 were 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 spectra 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.

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

6.3 METHOD 25A

6.3.1 Initial Checks

Before starting the first run, the following system checks were performed:

1. Zero and Span check of the analyzer;
2. Analyzer linearity check at intermediate levels; and
3. Response time of the system.

Calibration criteria for Method 25A is ± 5 percent of calibration gas value.

6.3.2 Daily Checks

The following checks were made for each test run.

1. Zero/Span calibration and Linearity check before each test run; and
2. Final Zero and Span calibrations of the analyzer at the end of each test run.

The difference between initial and final zero and span checks agreed within ± 3 percent of the instrument span.

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. "Validation of EPA FTIR Method For Measuring HCl," T. J. Geyer and G. M. Plummer, Air and Waste Management Association Paper Number 97-MP74.05, 1997.
5. "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.
6. "Computer-Assisted Quantitative Infrared Spectroscopy," Gregory L. McClure (ed.), **ASTM Special Publication 934** (ASTM), 1987.

APPENDIX A

METHOD 25A AND VOLUMETRIC FLOW DATA

A-1 METHOD 25A RESULTS

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1335	83.4	68.7
1336	83.7	67.9
1337	83.7	68.9
1338	83.1	68.4
1339	83.5	68.1
1340	83.1	68.6
1341	83.4	68.2
1342	84.4	68.4
1343	84.5	68.9
1344	86.9	69.8
1345	87.2	70.8
1346	87.8	70.9
1347	87.8	71.0
1348	87.7	70.9
1349	87.1	70.7
1350	86.8	70.6
1351	86.4	69.9
1352	85.7	69.7
1353	86.0	69.6
1354	84.6	69.7
1355	84.5	68.8
1356	84.6	69.1
1357	85.7	69.3
1358	86.4	70.1
1359	86.2	70.2
1400	86.5	70.2
1401	87.9	70.3
1402	88.2	72.0
1403	88.4	71.3
1404	87.6	71.1
1405	87.7	70.8
1406	86.9	71.0
1407	87.1	70.8
1408	86.7	71.1
1409	84.9	69.5
1410	84.9	69.1
1411	85.1	69.0
1412	86.5	69.5
1413	86.2	70.1
1414	85.2	69.2
1415	84.9	69.2
1416	84.3	68.7
1417	84.6	68.7
1418	88.5	71.3
1419	89.1	72.7
1420	88.4	72.1
1421	88.3	72.5

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1422	88.0	71.1
1423	95.8	80.9
1424	90.5	74.8
1425	87.6	71.5
1426	88.6	71.8
1427	87.4	70.9
1428	88.2	70.4
1429	90.1	72.0
1430	87.7	71.1
1431	88.3	70.9
1432	88.6	71.4
1433	89.6	71.1
1434	90.5	73.0
1435	89.9	71.7
1436	89.3	72.7
1437	86.6	71.1
1438	84.8	69.6
1439	84.8	69.6
1440	87.4	71.8
1441	84.7	70.9
1442	83.4	69.5
1443	81.5	67.3
1444	81.5	66.9
1445	82.2	67.1
1446	84.8	67.5
1447	85.9	69.5
1448	85.9	69.7
1449	85.5	69.5
1450	84.6	68.9
1451	83.1	66.1
1452	82.4	61.4
1453	82.5	62.4
1454	81.6	62.4
1455	80.9	60.2
1456	80.4	61.4
1457	80.5	63.2
1458	78.3	61.5
1459	80.0	60.6
1500	81.5	61.5
1501	81.7	64.5
1502	82.2	66.7
1503	82.2	67.0
1504	82.8	67.6
1505	84.3	68.2
1506	83.5	69.1
1507	84.3	68.7
1508	84.9	69.4

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1509	84.7	69.5
1510	85.5	69.1
1511	85.3	69.5
1512	86.8	70.0
1513	87.6	70.7
1514	89.4	72.1
1515	90.4	73.6
1516	90.4	73.2
1517	90.7	73.4
1518	90.0	72.7
1519	89.7	72.3
1520	91.0	72.1
1521	92.0	74.6
Process off Line (1522-1551)		
1552	81.0	58.9
1553	80.4	58.6
1554	82.6	61.7
1555	81.9	65.8
1556	79.7	61.8
1557	79.8	60.6
1558	79.0	57.9
1559	83.2	63.3
1600	83.9	64.9
1601	85.5	67.9
1602	87.0	69.3
1603	89.0	70.4
1604	90.0	71.7
1605	90.4	71.9
1606	91.1	72.0
1607	91.0	72.4
1608	90.4	72.0
1609	89.6	71.6
1610	89.2	71.3
1611	88.8	71.1
1612	89.1	71.1
1613	89.2	71.8
1614	88.3	71.3
1615	88.7	71.1
1616	89.0	71.5
1617	88.3	71.4
1618	88.2	71.0
1619	87.8	70.7
1620	88.1	71.2
1621	87.6	71.2
1622	86.6	69.9
1623	85.6	63.3

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1624	85.0	66.5
1625	85.2	63.9
1626	84.1	61.5
1627	88.2	66.8
1628	89.6	70.7
1629	88.7	70.3
1630	89.2	69.6
1631	89.8	71.8
1632	90.2	72.6
1633	90.4	72.0
1634	89.7	72.1
1635	89.0	72.0
1636	84.4	64.2
1637	83.0	61.3
1638	82.7	60.6
1639	82.9	60.3
1640	83.0	60.4
1641	83.6	60.8
1642	85.4	61.2
1643	87.3	68.2
1644	88.6	70.2
1645	90.3	72.0
1646	90.8	73.1
1647	91.4	73.7
1648	92.4	74.2
1649	95.2	75.8
1650	93.8	76.2
1651	92.7	75.2
1652	91.6	74.1
1653	89.9	73.3
1654	88.0	71.5
1655	89.8	71.8
1656	86.6	71.8
1657	84.6	70.0
1658	88.0	70.0
1659	84.4	67.4
1700	77.1	61.4
1701	73.8	57.3
1702	70.6	55.1
1703	68.4	53.5
1704	66.8	52.0
1705	68.4	52.1
1706	66.6	51.3
1707	67.2	51.8
1708	67.5	51.7
1709	68.6	52.6
1710	68.8	52.9

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1711	70.3	53.5
1712	72.3	54.7
1713	72.6	55.4
1714	72.4	55.2
1715	73.0	55.3
1716	73.9	56.1
1717	74.1	56.4
1718	74.7	56.6
1719	75.9	57.2
1720	77.1	58.2
1721	78.7	58.5
1722	80.6	59.7
1723	81.0	60.3
1724	82.6	60.8
1725	83.7	61.9
1726	84.4	62.1
1727	85.0	62.3
1728	85.9	62.9
1729	87.0	69.3
1730	83.8	66.5
1731	82.6	62.6
1732	81.4	60.5
1733	81.7	60.2
1734	84.3	62.3
1735	86.5	68.9
1736	87.5	70.8
1737	87.9	72.0
1738	88.2	71.6
1739	87.8	72.4
1740	86.2	71.3
1741	85.2	70.1
1742	82.3	69.1
1743	79.4	62.9
1744	78.5	59.7
1745	76.7	58.6
1746	75.8	57.2
1747	75.2	57.2
1748	75.2	56.8
1749	75.6	56.7
1750	76.2	56.9
1751	77.5	58.1
1752	76.1	57.2
1753	76.2	56.8
1754	78.7	57.9
1755	79.7	59.0
1756	81.6	59.8
1757	83.0	60.7

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1758	83.7	61.5
1759	88.8	65.7
1800	92.1	73.2
1801	91.0	73.4
1802	89.1	72.6
1803	86.3	70.6
1804	81.8	64.3
1805	80.5	60.2
1806	79.3	59.2
1807	77.5	57.9
1808	77.4	57.6
1809	76.9	57.5
1810	76.9	57.4
1811	76.3	56.9
1812	75.8	56.8
1813	76.2	56.6
1814	75.8	56.6
1815	76.2	56.8
1816	76.1	56.9
1817	76.2	56.8
1818	76.4	57.0
1819	77.2	57.5
1820	78.0	57.5
1821	78.1	57.9
1822	78.5	57.5
1823	79.9	57.8
1824	82.0	64.0
1825	81.8	66.3
1826	81.5	65.4
1827	81.6	66.0
1828	80.1	65.9
1829	79.9	64.9
1830	80.6	65.5
1831	80.5	65.9
1832	81.5	66.3
1833	80.6	66.5
1834	81.7	66.6
1835	81.8	67.2
1836	82.2	67.0
1837	82.3	67.6
1838	81.1	67.1
1839	82.5	67.4
1840	80.3	66.6
1841	80.3	65.9
1842	78.4	65.8
1843	75.7	58.4
1844	74.6	57.2

WCI

Run 1

Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1845	73.8	56.0
1846	73.5	55.6
1847	73.5	55.3
1848	73.8	55.5
1849	74.9	55.8
1850	74.3	56.2
1851	74.8	56.0
1852	74.9	56.5
1853	77.0	59.2
1854	79.1	64.4
1855	78.8	62.9
1856	79.9	64.8
1857	78.8	63.0
1858	78.1	59.7
1859	79.8	60.8
1900	80.7	65.5
1901	80.9	66.2
1902	81.7	66.3
1903	83.2	67.5
1904	84.6	69.5
1905	83.7	68.7
1906	84.5	69.1
1907	82.9	68.4
1908	82.6	68.0
1909	82.0	67.7
1910	82.8	68.0
1911	81.8	68.1
1912	80.8	67.1
1913	80.2	66.6
1914	80.6	66.8
1915	80.1	66.4
1916	80.1	66.5
1917	80.4	66.1
1918	80.4	66.8
1919	79.3	66.1
1920	78.8	65.1
1921	78.1	65.1
1922	78.3	65.1
1923	77.9	65.0
1924	78.1	65.1
1925	78.3	65.3
1926	78.6	65.3
1927	79.2	66.0
1928	79.5	66.1
1929	80.7	66.6
1930	81.5	67.4
1931	81.9	68.1

WCI

Run 1

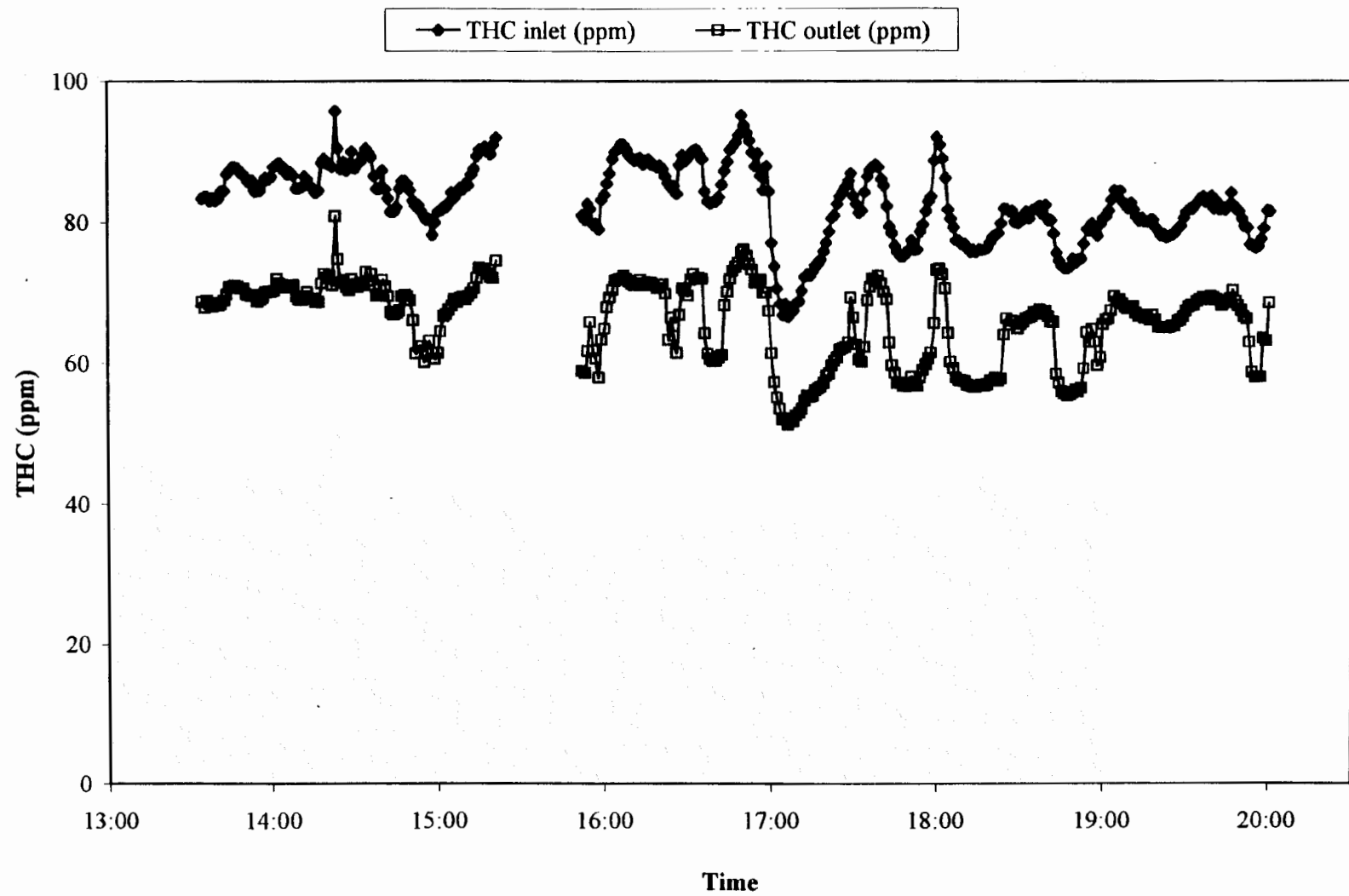
Date: 8/12/97

Project No.: 3804-20-03-02-02/4701-08-10

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1932	81.9	68.3
1933	82.4	68.3
1934	82.9	68.8
1935	83.4	69.1
1936	83.7	69.4
1937	82.9	69.4
1938	83.1	69.0
1939	83.8	69.5
1940	82.0	69.4
1941	82.9	69.1
1942	81.8	68.9
1943	82.1	68.3
1944	81.8	68.7
1945	82.4	68.6
1946	84.2	69.2
1947	82.4	70.4
1948	81.8	68.8
1949	81.6	68.2
1950	80.5	67.5
1951	79.5	66.6
1952	79.3	66.3
1953	76.9	63.0
1954	76.8	58.7
1955	76.4	58.0
1956	76.6	58.1
1957	77.7	58.1
1958	79.2	63.6
1959	81.7	63.2
2000	81.6	68.6
	<u>Inlet</u>	<u>Outlet</u>
Minimum	66.6	51.3
Maximum	95.8	80.9
Average	82.9	65.9

THC Concentrations vs. Time (Run 1, 8/12/97)



WCI
Run 2
Date: 8/13/97
Project No.: 3804-20-03-02-01
Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1055	82.5	70.5
1056	83.0	71.5
1057	81.6	70.3
1058	81.4	70.0
1059	81.0	69.8
1100	80.2	69.5
1101	80.8	69.8
1102	79.8	69.2
1103	79.9	69.0
1104	79.8	69.3
1105	79.0	68.9
1106	79.9	69.3
1107	81.3	69.9
1108	81.4	70.5
1109	81.7	70.2
1110	81.6	70.6
1111	81.8	70.6
1112	83.3	71.4
1113	83.2	71.7
1114	83.0	71.2
1115	84.1	72.3
1116	83.9	72.3
1117	84.2	72.4
1118	84.6	72.7
1119	84.4	72.5
1120	84.8	72.7
1121	85.4	72.6
1122	85.7	73.2
1123	85.7	73.9
1124	84.2	73.1
1125	82.6	72.0
1126	81.1	70.9
1127	82.1	71.2
1128	83.1	72.1
1129	80.9	71.4
1130	80.2	70.1
1131	78.4	65.8
1132	79.0	68.6
1133	79.2	69.0
1134	79.5	69.4
1135	79.5	69.7
1136	79.8	69.8
1137	79.2	69.6
1138	79.8	69.7

WCI

Run 2

Date: 8/13/97

Project No.: 3804-20-03-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1139	79.4	69.6
1140	79.7	69.7
1141	80.3	70.0
1142	79.9	69.8
1143	80.8	70.0
1144	81.9	70.9
1145	82.8	72.2
1146	82.5	71.6
1147	83.1	72.2
1148	83.1	72.1
1149	82.4	71.7
1150	83.1	71.6
1151	84.5	72.2
1152	84.2	74.0
1153	82.5	71.7
1154	81.5	71.0
1155	81.5	70.8
1156	80.8	70.9
1157	80.3	69.9
1158	80.3	70.1
1159	81.6	70.3
1200	81.8	71.0
1201	81.8	70.4
1202	83.2	71.2
1203	83.1	71.1
1204	84.3	71.6
1205	85.0	73.1
1206	82.5	71.6
1207	84.6	72.5
1208	84.7	72.7
1209	85.6	71.9
1210	84.9	72.6
1211	85.2	72.1
1212	83.2	71.1
1213	82.0	70.3
1214	82.1	70.1
1215	82.1	69.8
1216	83.0	70.7
1217	81.2	69.9
1218	83.2	70.4
1219	81.5	70.3
1220	81.8	70.0
1221	83.3	71.1
1222	81.3	69.3

WCI

Run 2

Date: 8/13/97

Project No.: 3804-20-03-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1223	80.7	69.1
1224	82.0	69.8
1225	85.0	72.3
1226	88.4	75.1
1227	86.0	74.5
1228	83.6	72.1
1229	82.0	71.1
1230	82.8	70.4
1231	84.4	72.4
1232	84.7	72.9
1233	84.0	72.7
1234	81.6	70.8
1235	80.1	69.8
1236	79.8	68.7
1237	79.4	69.1
1238	78.7	68.3
1239	81.8	70.1
1240	80.0	69.8
1241	80.6	69.1
1242	80.1	70.3
1243	79.7	68.9
1244	80.1	69.2
1245	81.1	69.6
1246	82.3	70.5
1247	82.0	71.2
1248	82.5	71.1
1249	83.5	72.7
1250	82.2	71.7
1251	80.5	70.6
1252	78.6	69.1
1253	78.7	68.2
1254	78.9	68.8
1255	79.0	69.2
1256	78.8	68.9
1257	78.8	68.8
1258	78.9	68.9
1259	79.2	68.8
1300	79.6	69.3
1301	79.8	69.3
1302	81.1	70.2
1303	82.4	71.2
1304	82.2	71.8
1305	82.4	71.5
1306	84.3	72.5

WCI

Run 2

Date: 8/13/97

Project No.: 3804-20-03-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1307	84.5	73.0
1308	84.9	72.7
1309	85.6	73.5
1310	84.6	72.8
1311	84.5	72.9
1312	84.1	72.4
1313	83.9	72.2
1314	84.9	72.7
1315	85.1	73.1
1316	84.4	72.6
1317	84.2	72.0
1318	83.9	72.3
1319	83.7	72.2
1320	84.1	72.3
1321	83.9	72.3
1322	84.0	72.4
1323	84.1	72.5
1324	84.0	72.6
1325	83.7	72.4
1326	83.2	72.1
1327	83.3	71.8
1328	83.0	72.1
1329	82.9	71.8
1330	83.8	72.1
1331	83.0	72.1
1332	83.6	72.0
1333	83.9	73.0
1334	84.3	71.9
1335	85.1	73.7
1336	83.9	72.7
1337	84.0	72.3
1338	84.3	73.0
1339	84.5	72.7
1340	83.0	72.0
1341	82.1	71.0
1342	82.7	70.9
1343	88.2	74.7
1344	84.8	74.4
1345	81.6	70.6
1346	82.8	71.8
1347	81.9	70.6
1348	81.5	70.7
1349	81.9	70.3
1350	82.5	70.9

WCI
 Run 2
 Date: 8/13/97
 Project No.: 3804-20-03-02-01
 Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1351	82.1	71.0
1352	82.2	70.5
1353	83.1	71.0
1354	82.3	70.7
Manual Sampling Down (1355-1529)		
1530	78.9	68.5
1531	79.4	68.9
1532	79.2	68.8
1533	79.9	69.2
1534	80.7	69.9
1535	81.7	70.8
1536	81.4	70.8
1537	81.1	70.0
1538	82.7	71.2
1539	81.8	71.1
1540	81.8	70.6
1541	81.8	70.6
1542	81.1	70.3
1543	81.2	70.5
1544	80.5	70.1
1545	79.5	69.3
1546	79.1	69.1
1547	78.1	68.3
1548	78.0	68.4
1549	76.7	67.6
1550	77.1	67.6
1551	76.3	67.8
1552	74.8	66.8
1553	74.0	65.9
1554	73.4	65.7
1555	71.3	63.4
1556	70.4	58.2
1557	73.2	62.7
1558	73.2	64.5
1559	72.8	65.0
1600	73.2	65.3
1601	73.8	66.1
1602	75.5	67.3
1603	76.6	68.5
1604	77.9	69.4
1605	78.5	69.7
1606	82.6	72.0
1607	83.6	73.6

WCI
 Run 2
 Date: 8/13/97
 Project No.: 3804-20-03-02-01
 Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1608	84.9	74.3
1609	84.3	74.2
1610	83.6	73.8
1611	83.7	73.1
1612	83.2	73.2
1613	84.7	73.6
1614	83.8	73.1
1615	83.2	72.8
1616	83.1	72.3
1617	82.9	72.3
1618	82.5	72.2
1619	82.1	71.7
1620	82.0	71.7
1621	81.2	71.1
1622	81.1	70.9
1623	81.6	71.5
Process Down (1624-1659)		
1700	69.5	61.9
1701	70.8	62.4
1702	72.7	64.0
1703	75.0	65.6
1704	77.4	66.8
1705	79.9	69.2
1706	81.9	70.6
1707	83.7	72.0
1708	85.2	72.9
1709	86.7	74.2
1710	86.0	74.2
1711	84.1	72.7
1712	81.6	70.8
1713	80.0	69.3
1714	79.2	68.4
1715	79.5	68.9
1716	78.8	68.2
1717	80.6	68.9
1718	80.0	69.3
1719	79.7	69.1
1720	79.3	68.6
1721	79.4	68.5
1722	78.3	68.1
1723	77.8	67.8
1724	77.2	67.4
1725	76.8	67.0

WCI

Run 2

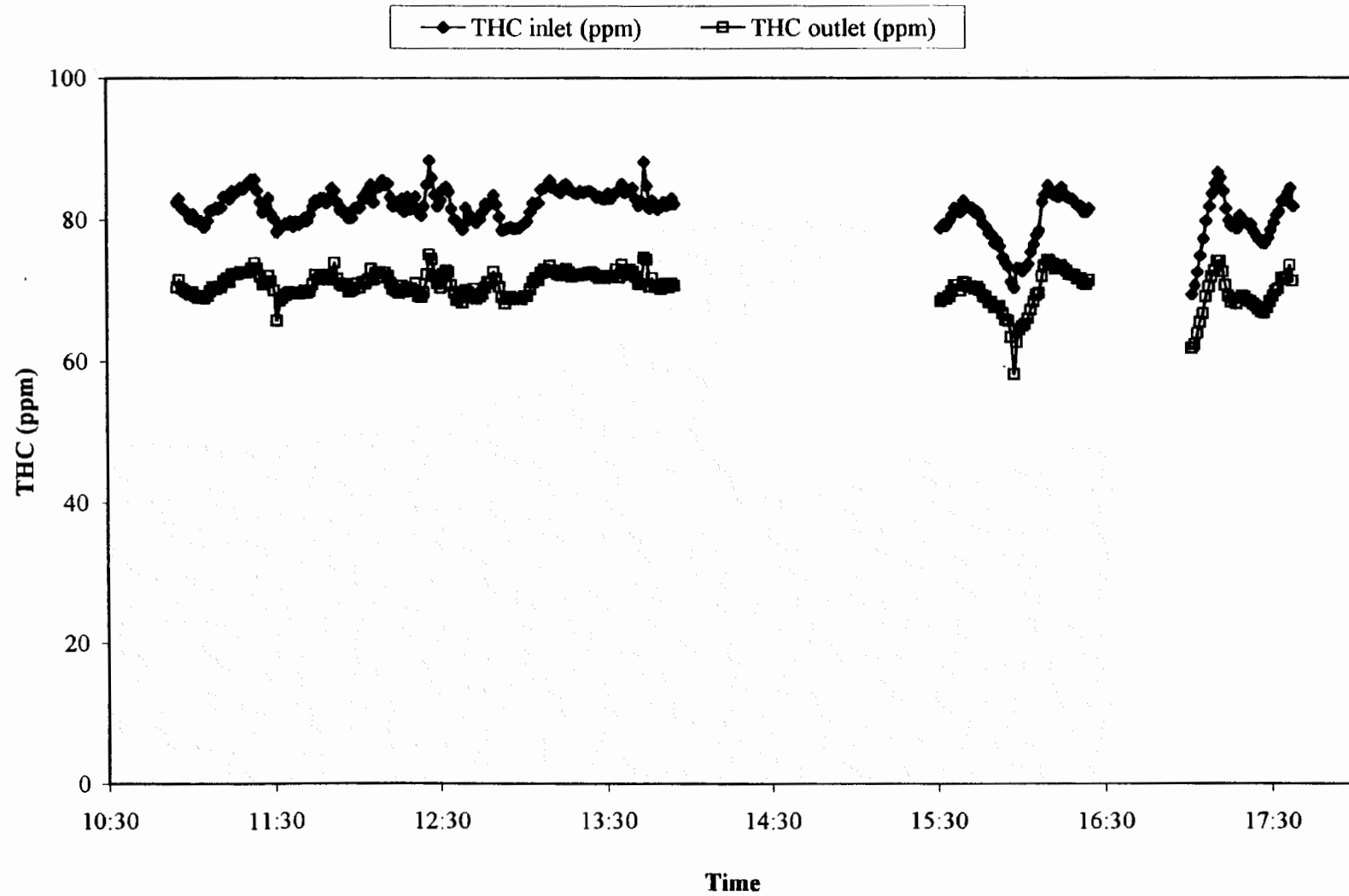
Date: 8/13/97

Project No.: 3804-20-03-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1726	76.7	66.9
1727	77.5	67.6
1728	78.6	68.5
1729	79.6	69.3
1730	80.7	70.0
1731	81.2	70.3
1732	82.7	71.8
1733	82.7	71.6
1734	83.9	71.9
1735	84.5	73.6
1736	81.9	71.4
	<u>Inlet</u>	<u>Outlet</u>
Minimum	69.5	58.2
Maximum	88.4	75.1
Average	81.4	70.5

THC Concentrations vs. Time (Run 2, 8/13/97)



WCI

Run 3

Date: 8/14/97

Project No. 3804-20-02-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
810	71.5	56.5
811	78.2	60.2
812	77.1	62.8
813	75.2	59.5
814	78.3	60.5
815	79.1	22.3
816	77.0	27.9
817	76.9	58.8
818	79.3	60.6
819	80.8	62.4
820	81.6	64.0
821	81.7	63.1
822	82.3	64.6
823	82.9	64.2
824	85.2	66.1
825	81.7	64.8
826	82.8	63.5
827	85.1	65.1
828	87.6	69.1
829	82.4	66.2
830	79.4	63.0
831	77.9	61.0
832	75.7	60.5
833	74.6	58.5
834	75.2	58.9
835	73.9	58.5
836	73.5	57.8
837	72.8	57.9
838	72.5	57.2
839	72.4	57.0
840	72.9	57.2
841	74.7	58.5
842	74.7	59.7
843	76.5	59.8
844	74.6	60.2
845	72.6	58.0
846	72.5	57.1
847	73.2	57.7
848	72.7	57.8
849	72.8	57.8
850	71.7	57.5
851	71.8	57.0
852	71.7	57.0
853	71.2	57.2

WCI

Run 3

Date: 8/14/97

Project No. 3804-20-02-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
854	70.9	56.7
855	71.2	56.7
856	70.0	56.7
857	69.4	55.5
858	68.5	55.4
859	68.1	54.7
900	69.0	55.0
901	69.0	55.5
902	69.0	55.4
903	69.7	56.0
904	70.2	56.6
905	70.9	56.8
906	72.0	57.4
907	72.5	57.6
908	73.6	58.3
909	74.4	58.5
910	74.9	58.4
911	75.9	68.3
912	76.0	Spike
913	75.1	Spike
914	73.4	Spike
915	71.8	Spike
916	70.8	Spike
917	71.0	Spike
918	71.0	Spike
919	70.4	Spike
920	69.8	Spike
921	69.9	Spike
922	70.0	Spike
923	70.7	Spike
924	70.7	Spike
925	71.0	Spike
926	71.6	Spike
927	72.8	Spike
928	71.9	Spike
929	72.7	Spike
930	73.4	Spike
931	73.4	Spike
932	73.8	Spike
933	75.8	Spike
934	75.2	Spike
935	74.0	Spike
936	73.5	Spike
937	72.9	Spike

WCI

Run 3

Date: 8/14/97

Project No. 3804-20-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
938	71.9	Spike
939	71.5	Spike
940	71.6	Spike
941	71.9	56.6
942	Spike	55.5
943	Spike	55.2
944	Spike	54.1
945	Spike	53.8
946	Spike	52.8
947	Spike	53.1
948	Spike	53.3
949	Spike	53.4
950	Spike	53.8
951	Spike	53.5
952	Spike	54.0
953	Spike	53.8
954	Spike	54.4
955	Spike	55.6
956	Spike	55.0
957	Spike	55.4
958	Spike	55.2
959	Spike	56.9
1000	Spike	57.1
1001	Spike	58.2
1002	Spike	57.5
1003	Spike	56.7
1004	Spike	57.4
1005	Spike	58.1
1006	Spike	58.0
1007	Spike	58.1
1008	Spike	57.6
1009	72.5	59.8
1010	71.6	58.8
1011	76.1	62.5
1012	71.6	61.2
1013	70.5	57.7
1014	70.5	58.4
1015	70.4	57.8
1016	72.3	58.8
1017	71.8	59.2
1018	71.0	58.4
1019	70.7	57.9
1020	70.6	57.6
1021	70.9	58.3

WCI

Run 3

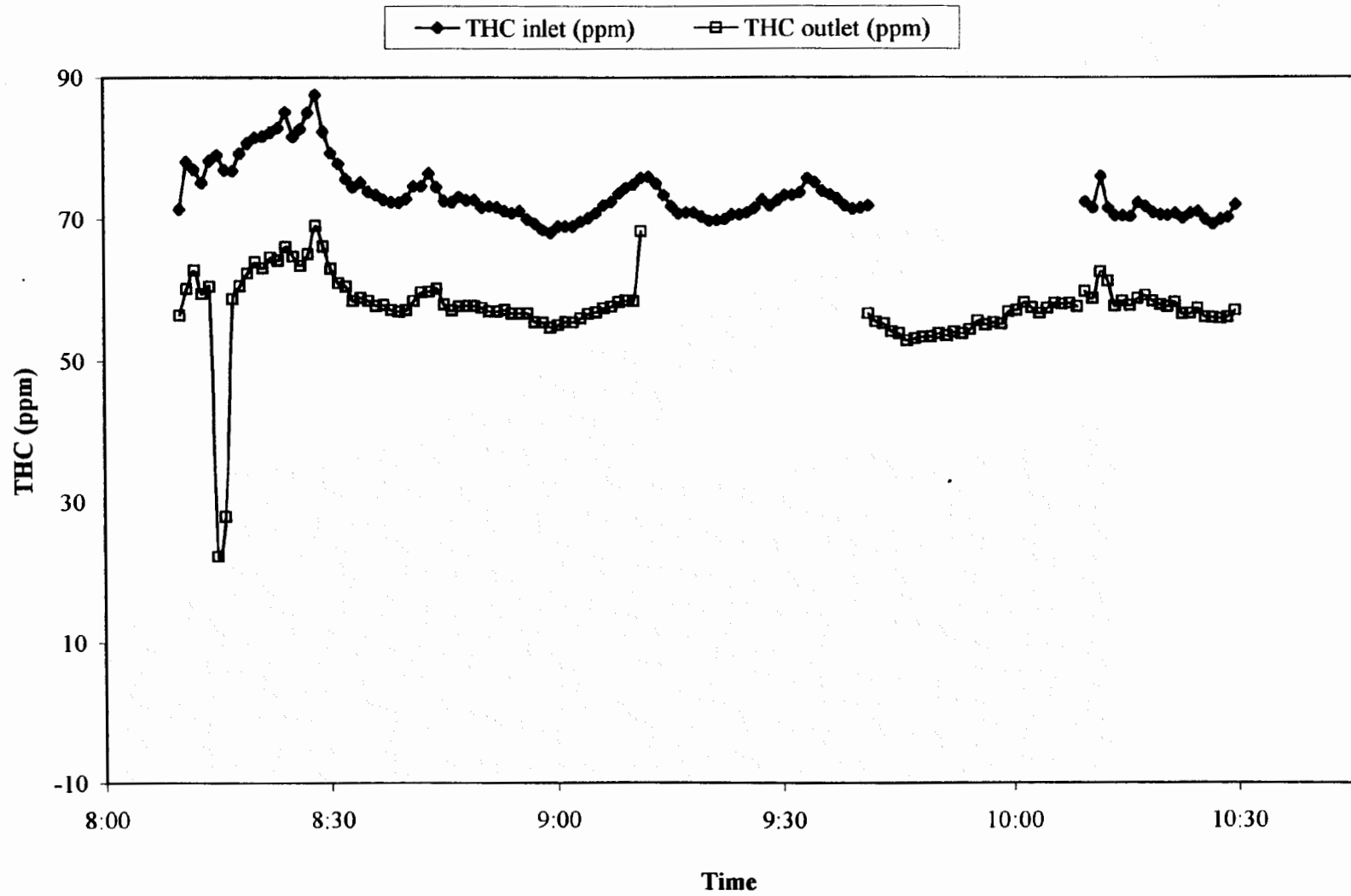
Date: 8/14/97

Project No. 3804-20-02-02-01

Operator: Gulick

Time (24 hour)	THC inlet (ppm)	THC outlet (ppm)
1022	70.2	56.6
1023	70.9	56.7
1024	71.1	57.4
1025	70.0	56.2
1026	69.4	56.1
1027	70.0	56.0
1028	70.3	56.2
1029	72.1	57.1
	<u>Inlet</u>	<u>Outlet</u>
Minimum	68.1	22.3
Maximum	87.6	69.1
Average	73.6	57.5

THC Concentrations vs. Time (Run 3, 8/14/97)



A-2 METHOD 25A CALIBRATION AND QA CHECK DATA

Calibration Error Determination For 8/12/97

	Cal Gas Value	Measured Value	Difference as % of Cal Gas	Pass/ Fail
THC 1 Inlet	0.0	0.0	0.0	Pass
	90.4	91.0	0.7	Pass
	50.4	50.7	0.6	Pass
	35.2	34.7	1.4	Pass
THC 2 Outlet	0.0	0.1	0.1	Pass
	90.4	92.4	2.2	Pass
	50.4	50.8	0.8	Pass
	35.2	34.8	1.1	Pass

Pass/Fail Criteria is +/- 5% of Calibration gas.

Calibration Drift Determination for 8/12/97

Zero Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	0.0	-1.3	1.3	Pass
THC 2 Outlet	0.1	0.6	0.5	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.

Pass/Fail Criteria is +/- 3% of Instrument Span.

Span Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	91.0	91.4	0.4	Pass
THC 2 Outlet	92.4	90.6	1.8	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.

Pass/Fail Criteria is +/- 3% of Instrument Span.

Calibration Error Determination For 8/13/97

	Cal Gas Value	Measured Value	Difference as % of Cal Gas	Pass/ Fail
THC 1 Inlet	0.0	0.3	0.3	Pass
	90.4	90.4	0.0	Pass
	50.4	49.8	1.2	Pass
	35.2	34.1	3.1	Pass
THC 2 Outlet	0.0	0.5	0.5	Pass
	90.4	91.4	1.1	Pass
	50.4	50.8	0.8	Pass
	35.2	35.1	0.3	Pass

Pass/Fail Criteria is +/- 5% of Calibration gas.

Calibration Drift Determination for 8/13/97

Zero Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	0.3	0.0	0.3	Pass
THC 2 Outlet	0.5	1.8	1.3	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.

Pass/Fail Criteria is +/- 3% of Instrument Span.

Span Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	90.4	90.5	0.1	Pass
THC 2 Outlet	91.4	92.0	0.6	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.

Pass/Fail Criteria is +/- 3% of Instrument Span.

Calibration Error Determination For 8/14/97

	Cal Gas Value	Measured Value	Difference as % of Cal Gas	Pass/ Fail
THC 1 Inlet	0.0	0.3	0.3	Pass
	90.4	90.7	0.3	Pass
	50.4	51.1	1.4	Pass
	35.2	35.8	1.7	Pass
THC 2 Outlet	0.0	0.6	0.6	Pass
	90.4	91.5	1.2	Pass
	50.4	50.4	0.0	Pass
	35.2	34.4	2.3	Pass

Pass/Fail Criteria is +/- 5% of Calibration gas.

Calibration Drift Determination for 8/14/97

Zero Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	0.3	-0.1	0.4	Pass
THC 2 Outlet	0.6	0.3	0.3	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.
Pass/Fail Criteria is +/- 3% of Instrument Span.

Span Drift

	Initial Value	Final Value	Difference as % of Span	Pass/ Fail
THC 1 Inlet	90.7	89.5	1.2	Pass
THC 2 Outlet	91.5	90.5	1.0	Pass

Instrument Span for THC 1 and THC 2 is 100 ppm.
Pass/Fail Criteria is +/- 3% of Instrument Span.

Response Times

THC 1
Inlet

47 seconds

THC 2
Outlet

55 seconds

A-3 VOLUMETRIC FLOW DATA

Facility:	WCI
Date:	08/12/97
Location:	Stack
Run Number:	7
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (In Hg)	29.26
Absolute Stack Pressure (In H2O)	29.23
Stack Static Pressure (in H2O)	-0.40
Average Stack Temperature (°F)	246.09
Stack Area (sq in)	9180.88
Actual Meter Volume (cu ft)	224.695
Average Meter Pressure (In H2O)	2.77
Average Meter Temperature (°F)	99.06
Moisture Collected (g)	368.50
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9880
Nozzle Diameter (In)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.860
Standard Metered Volume (dscf)	206.501
Standard Metered Volume (dscm)	5.848
Stack Moisture (%V)	7.76
Mole Fraction Dry Stack Gas	0.922
Dry Molecular Weight	29.26
Wet Molecular Weight	28.39
Stack Gas Velocity (fpm)	6473.79
Stack Gas Velocity (mpm)	1973.21
Volumetric Flow Rate (acfm)	411844.44
Volumetric Flow Rate (acmm)	11663.43
Volumetric Flow Rate (dscfm)	277479.87
Volumetric Flow Rate (dscmm)	7858.23
Percent Isokinetic	97.10
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/13/87
Location:	Stack
Run Number:	2
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.00
Absolute Stack Pressure (in H ₂ O)	29.04
Stack Static Pressure (in H ₂ O)	0.60
Average Stack Temperature (°F)	238.50
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	213.980
Average Meter Pressure (in H ₂ O)	2.55
Average Meter Temperature (°F)	77.34
Moisture Collected (g)	341.00
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9880
Nozzle Diameter (in)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.844
Standard Metered Volume (dscf)	202.664
Standard Metered Volume (dscm)	5.739
Stack Moisture (%V)	7.35
Mole Fraction Dry Stack Gas	0.928
Dry Molecular Weight	29.26
Wet Molecular Weight	28.43
Stack Gas Velocity (fpm)	6326.64
Stack Gas Velocity (mpm)	1928.36
Volumetric Flow Rate (acfm)	402483.68
Volumetric Flow Rate (acmm)	11398.34
Volumetric Flow Rate (dscfm)	273586.26
Volumetric Flow Rate (dscmm)	7747.96
Percent Isokinetic	96.65
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/14/97
Location:	Stack
Run Number:	3
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (In Hg)	29.04
Absolute Stack Pressure (in H2O)	29.08
Stack Static Pressure (In H2O)	0.60
Average Stack Temperature (°F)	230.91
Stack Area (sq in)	9180.88
Actual Meter Volume (cu ft)	209.885
Average Meter Pressure (in H2O)	2.48
Average Meter Temperature (°F)	86.61
Moisture Collected (g)	283.00
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9880
Nozzle Diameter (in)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.815
Standard Metered Volume (dscf)	195.672
Standard Metered Volume (dscm)	5.541
Stack Moisture (%V)	6.38
Mole Fraction Dry Stack Gas	0.936
Dry Molecular Weight	29.26
Wet Molecular Weight	28.54
Stack Gas Velocity (fpm)	6224.21
Stack Gas Velocity (mpm)	1897.14
Volumetric Flow Rate (acfm)	395967.06
Volumetric Flow Rate (acmm)	11213.79
Volumetric Flow Rate (dscfm)	275333.02
Volumetric Flow Rate (dscmm)	7797.43
Percent Isokinetic	92.72
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/12/97
Location:	BH/Inlet
Run Number:	1
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.30
Absolute Stack Pressure (in H ₂ O)	27.09
Stack Static Pressure (in H ₂ O)	-30.00
Average Stack Temperature (°F)	257.53
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	107.497
Average Meter Pressure (in H ₂ O)	0.67
Average Meter Temperature (°F)	90.61
Moisture Collected (g)	140.10
Carbon Dioxide Concentration (%V)	2.0
Oxygen Concentration (%V)	18.5
Nitrogen Concentration (%V)	79.5
Dry Gas Meter Factor	0.9840
Nozzle Diameter (in)	0.150
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.415
Standard Metered Volume (dscf)	99.613
Standard Metered Volume (dscm)	2.821
Stack Moisture (%V)	6.22
Mole Fraction Dry Stack Gas	0.938
Dry Molecular Weight	29.06
Wet Molecular Weight	28.37
Stack Gas Velocity (fpm)	5375.30
Stack Gas Velocity (mpm)	1638.39
Volumetric Flow Rate (acfm)	341962.03
Volumetric Flow Rate (acmm)	9684.36
Volumetric Flow Rate (dscfm)	213664.28
Volumetric Flow Rate (dscmm)	6050.97
Percent Isokinetic	100.70
Percent Excess Air	738.85
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WGI
Date:	08/13/97
Location:	BH/Inlet
Run Number:	2
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.06
Absolute Stack Pressure (in H ₂ O)	26.85
Stack Static Pressure (in H ₂ O)	-30.00
Average Stack Temperature (°F)	218.96
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	104.344
Average Meter Pressure (in H ₂ O)	0.58
Average Meter Temperature (°F)	78.21
Moisture Collected (g)	111.60
Carbon Dioxide Concentration (%V)	2.0
Oxygen Concentration (%V)	18.5
Nitrogen Concentration (%V)	79.5
Dry Gas Meter Factor	0.9840
Nozzle Diameter (in)	0.150
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.408
Standard Metered Volume (dscf)	97.867
Standard Metered Volume (dscm)	2.772
Stack Moisture (%V)	5.10
Mole Fraction Dry Stack Gas	0.949
Dry Molecular Weight	29.06
Wet Molecular Weight	28.50
Stack Gas Velocity (fpm)	4821.75
Stack Gas Velocity (mpm)	1469.67
Volumetric Flow Rate (acfm)	306746.70
Volumetric Flow Rate (acmm)	8687.07
Volumetric Flow Rate (dscfm)	203150.98
Volumetric Flow Rate (dscmm)	5753.24
Percent Isokinetic	104.06
Percent Excess Air	738.85
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/14/97
Location:	BH/Inlet
Run Number:	3
Sample Type:	Metals
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.10
Absolute Stack Pressure (in H2O)	26.89
Stack Static Pressure (in H2O)	-30.00
Average Stack Temperature (°F)	216.67
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	109.508
Average Meter Pressure (in H2O)	0.61
Average Meter Temperature (°F)	80.08
Moisture Collected (g)	157.20
Carbon Dioxide Concentration (%V)	2.0
Oxygen Concentration (%V)	18.0
Nitrogen Concentration (%V)	80.0
Dry Gas Meter Factor	0.9840
Nozzle Diameter (in)	0.150
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.430
Standard Metered Volume (dscf)	103.200
Standard Metered Volume (dscm)	2.923
Stack Moisture (%V)	6.70
Mole Fraction Dry Stack Gas	0.933
Dry Molecular Weight	29.04
Wet Molecular Weight	28.30
Stack Gas Velocity (fpm)	5116.05
Stack Gas Velocity (mpm)	1559.37
Volumetric Flow Rate (acfm)	325469.09
Volumetric Flow Rate (acmm)	9217.28
Volumetric Flow Rate (dscfm)	212954.17
Volumetric Flow Rate (dscmm)	6030.86
Percent Isokinetic	104.68
Percent Excess Air	573.98
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/12/97
Location:	Stack
Run Number:	1
Sample Type:	Dioxin
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (In Hg)	29.26
Absolute Stack Pressure (In H2O)	29.26
Stack Static Pressure (In H2O)	-0.04
Average Stack Temperature (°F)	250.19
Stack Area (sq In)	9160.88
Actual Meter Volume (cu ft)	228.017
Average Meter Pressure (In H2O)	2.63
Average Meter Temperature (°F)	99.53
Moisture Collected (g)	341.40
Carbon Dioxide Concentration (%V)	0.5
Oxygen Concentration (%V)	20.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9960
Nozzle Diameter (In)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.878
Standard Metered Volume (dscf)	210.832
Standard Metered Volume (dscm)	5.971
Stack Moisture (%V)	7.09
Mole Fraction Dry Stack Gas	0.929
Dry Molecular Weight	28.90
Wet Molecular Weight	28.13
Stack Gas Velocity (fpm)	8452.52
Stack Gas Velocity (mpm)	1966.73
Volumetric Flow Rate (acfm)	410491.39
Volumetric Flow Rate (acmm)	11625.12
Volumetric Flow Rate (dscfm)	277213.98
Volumetric Flow Rate (dscmm)	7850.70
Percent Isokinetic	99.23
Percent Excess Air	5513.72
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/13/97
Location:	Stack
Run Numb.	2
Sample Ty.	Dioxin
Total Sampling Time (min)	120.0
Corrected Barometric Pressure (in Hg)	29.00
Absolute Stack Pressure (in H ₂ O)	29.04
Stack Static Pressure (in H ₂ O)	0.60
Average Stack Temperature (°F)	245.69
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	107.856
Average Meter Pressure (in H ₂ O)	2.27
Average Meter Temperature (°F)	79.38
Moisture Collected (g)	148.70
Carbon Dioxide Concentration (%V)	0.5
Oxygen Concentration (%V)	20.6
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9960
Nozzle Diameter (in)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.854
Standard Metered Volume (dscf)	102.513
Standard Metered Volume (dscm)	2.903
Stack Moisture (%V)	6.32
Mole Fraction Dry Stack Gas	0.937
Dry Molecular Weight	28.90
Wet Molecular Weight	28.21
Stack Gas Velocity (fpm)	6188.65
Stack Gas Velocity (mpm)	1886.30
Volumetric Flow Rate (acfm)	393704.79
Volumetric Flow Rate (acmm)	11149.72
Volumetric Flow Rate (dscfm)	267835.10
Volumetric Flow Rate (dscmm)	7585.09
Percent Isokinetic	99.88
Percent Excess Air	5513.72
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/13/97
Location:	Stack
Run Number:	3
Sample Type:	Dioxin
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.00
Absolute Stack Pressure (in H ₂ O)	29.04
Stack Static Pressure (in H ₂ O)	0.60
Average Stack Temperature (°F)	233.63
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	224.147
Average Meter Pressure (in H ₂ O)	2.53
Average Meter Temperature (°F)	81.05
Moisture Collected (g)	358.40
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9960
Nozzle Diameter (in)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.886
Standard Metered Volume (dscf)	212.543
Standard Metered Volume (dscm)	6.019
Stack Moisture (%V)	7.37
Mole Fraction Dry Stack Gas	0.926
Dry Molecular Weight	29.26
Wet Molecular Weight	28.43
Stack Gas Velocity (fpm)	6249.71
Stack Gas Velocity (mpm)	1904.91
Volumetric Flow Rate (acfm)	397589.40
Volumetric Flow Rate (acmm)	11259.73
Volumetric Flow Rate (dscfm)	272116.18
Volumetric Flow Rate (dscmm)	7706.33
Percent Isokinetic	101.91
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/14/97
Location:	Stack
Run Number:	4
Sample Type:	Dioxin
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	29.04
Absolute Stack Pressure (in H ₂ O)	29.08
Stack Static Pressure (in H ₂ O)	0.60
Average Stack Temperature (°F)	235.66
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	222.820
Average Meter Pressure (in H ₂ O)	2.51
Average Meter Temperature (°F)	89.59
Moisture Collected (g)	310.80
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.6
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9960
Nozzle Diameter (in)	0.193
Pilot Constant	0.84
Average Sampling Rate (dscfm)	0.868
Standard Metered Volume (dscf)	208.287
Standard Metered Volume (dscm)	5.899
Stack Moisture (%V)	6.57
Mole Fraction Dry Stack Gas	0.934
Dry Molecular Weight	29.26
Wet Molecular Weight	28.52
Stack Gas Velocity (fpm)	6245.02
Stack Gas Velocity (mpm)	1903.48
Volumetric Flow Rate (acfm)	397290.73
Volumetric Flow Rate (acmm)	11251.27
Volumetric Flow Rate (dscfm)	273811.90
Volumetric Flow Rate (dscmm)	7754.35
Percent Isokinetic	99.25
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

Facility:	WCI
Date:	08/15/97
Location:	Stack
Run Number:	5
Sample Type:	Dioxin
Total Sampling Time (min)	240.0
Corrected Barometric Pressure (in Hg)	28.96
Absolute Stack Pressure (in H2O)	29.00
Stack Static Pressure (in H2O)	0.60
Average Stack Temperature (°F)	232.06
Stack Area (sq in)	9160.88
Actual Meter Volume (cu ft)	222.592
Average Meter Pressure (in H2O)	2.41
Average Meter Temperature (°F)	86.34
Moisture Collected (g)	297.40
Carbon Dioxide Concentration (%V)	3.5
Oxygen Concentration (%V)	17.5
Nitrogen Concentration (%V)	79.0
Dry Gas Meter Factor	0.9960
Nozzle Diameter (in)	0.193
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.869
Standard Metered Volume (dacf)	208.642
Standard Metered Volume (dscm)	5.909
Stack Moisture (%V)	6.30
Mole Fraction Dry Stack Gas	0.937
Dry Molecular Weight	29.26
Wet Molecular Weight	28.55
Stack Gas Velocity (fpm)	6112.46
Stack Gas Velocity (mpm)	1863.08
Volumetric Flow Rate (acfm)	388858.13
Volumetric Flow Rate (acmm)	11012.46
Volumetric Flow Rate (dscfm)	269443.87
Volumetric Flow Rate (dscmm)	7630.65
Percent Isokinetic	101.03
Percent Excess Air	519.01
Concentration (g/dscm)	0.00
Concentration (kg/hr)	0.00
Concentration (ppmv)	0.00
Emissions (lb/hr)	0.00

APPENDIX B

FTIR DATA

B-1 FTIR RESULTS

TABLE B-1. FTIR RESULTS (ppm) AT THE WCI BAGHOUSE INLET

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/12/97	10:35	IS0811A	26.2	1.6	6.1	0.19	3.5	0.75	80.3	1.1	115.0	2.9
	10:37	IS0811B	26.1	1.6	5.9	0.19	3.1	0.75	78.1	1.1	114.7	2.9
	10:41	IS0811C	25.9	1.5	5.6	0.19	2.9	0.74	75.7	1.0	106.6	2.9
	10:43	IS0811D	25.8	1.5	5.4	0.19	2.8	0.73	72.4	1.0	101.4	2.9
	10:53	IU0811A	6.2	1.6	6.1	0.20	3.5	0.72	80.6	1.1	115.6	3.1
	11:00	IU0811B	6.9	1.8	7.2	0.22	4.1	0.76	96.4	1.2	140.3	3.3
	11:02	IU0811C	7.5	2.1	8.3	0.48	4.9	0.83	113.1	1.3	168.9	3.6
	11:05	IU0811D	7.7	2.2	9.0	0.50	5.2	0.86	119.2	1.4	176.6	3.7
	11:07	IU0811E	7.8	2.2	9.3	0.50	5.3	0.87	122.5	1.4	183.7	3.7
	13:32	18120022	5.5	2.3	11.3	0.52	6.3	0.92	129.2	1.5	196.2	3.9
	13:34	18120023	5.5	2.3	11.2	0.52	6.1	0.91	126.5	1.4	191.1	3.9
	13:37	18120024	5.3	2.3	11.1	0.52	5.9	0.90	125.6	1.4	181.8	3.8
	13:39	18120025	5.1	2.3	11.1	0.52	5.9	0.90	125.0	1.4	174.5	3.8
	13:41	18120026	5.0	2.3	11.2	0.52	5.9	0.90	124.9	1.4	167.8	3.8
	13:43	18120027	5.3	2.3	11.3	0.53	6.0	0.90	126.1	1.5	167.3	3.8
	13:46	18120028	5.5	2.3	11.6	0.53	6.2	0.92	129.6	1.5	175.4	3.9
	13:48	18120029	5.4	2.3	11.7	0.53	6.2	0.91	129.0	1.5	181.8	3.9
	13:50	18120030	5.2	2.3	11.7	0.53	6.1	0.91	128.4	1.5	187.0	3.9
	13:53	18120031	5.2	2.3	11.6	0.53	6.1	0.90	127.9	1.5	184.7	3.9
	13:55	18120032	5.0	2.3	11.5	0.53	6.0	0.90	126.4	1.5	179.8	3.8
	13:57	18120033	4.9	2.3	11.5	0.53	6.0	0.90	127.0	1.5	178.0	3.9
	13:59	18120034	5.2	2.3	11.6	0.54	6.1	0.91	128.2	1.5	176.1	3.9
	14:01	18120035	5.0	2.3	11.8	0.54	6.1	0.91	130.4	1.5	173.3	3.9
	14:03	18120036	4.9	2.3	11.8	0.54	6.1	0.91	131.1	1.5	176.1	3.9
	14:05	18120037	4.9	2.3	11.8	0.54	6.1	0.91	131.1	1.5	177.8	3.9
	14:32	18120049	3.9	2.4	11.5	0.55	6.4	0.93	132.0	1.5	164.1	4.0

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a,b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/12/97	14:34	18120050	3.8	2.4	11.6	0.55	6.4	0.93	133.1	1.5	168.7	4.0
	14:36	18120051	3.8	2.4	11.8	0.55	6.4	0.93	134.8	1.5	176.3	4.0
	14:37	18120052	0.0	2.6	11.5	0.54	6.3	0.93	130.6	1.5	182.6	4.0
	14:40	18120053	0.0	2.6	11.3	0.54	6.1	0.93	131.4	1.5	178.0	3.9
	14:42	18120054	0.0	2.6	11.2	0.54	6.1	0.93	129.6	1.5	176.0	3.9
	14:44	18120055	0.0	2.6	11.1	0.54	5.9	0.91	125.1	1.5	171.2	3.9
	14:46	18120056	0.0	2.6	11.2	0.54	6.0	0.91	126.3	1.5	173.5	3.9
	14:48	18120057	0.0	2.6	11.4	0.54	6.1	0.92	128.5	1.5	179.3	3.9
	14:50	18120058	0.0	2.6	11.3	0.54	6.1	0.92	128.3	1.5	175.4	3.9
	14:52	18120059	0.0	2.6	11.1	0.54	6.0	0.92	126.7	1.5	172.2	3.9
	14:54	18120060	0.0	2.6	11.0	0.54	5.9	0.91	125.7	1.5	179.1	3.9
	14:57	18120061	0.0	2.6	10.9	0.54	5.8	0.91	124.5	1.5	183.4	3.9
	14:59	18120062	0.0	2.6	10.8	0.53	5.7	0.90	123.9	1.5	179.1	3.9
	15:01	18120063	0.0	2.6	10.9	0.54	5.8	0.91	125.7	1.5	179.5	3.9
	15:03	18120064	0.0	2.6	11.0	0.54	5.9	0.91	126.6	1.5	177.5	3.9
	15:05	18120065	0.0	2.6	11.1	0.54	5.9	0.91	128.3	1.5	176.3	3.9
	15:07	18120066	0.0	2.6	11.1	0.54	6.0	0.92	128.9	1.5	172.1	3.9
	15:09	18120067	0.0	2.6	11.3	0.54	6.0	0.92	129.3	1.5	167.7	3.9
	16:48	18120091	0.0	2.8	12.3	0.59	6.3	1.0	130.3	1.7	182.4	4.1
	16:50	18120092	0.0	2.8	12.6	0.60	6.5	1.0	134.6	1.7	193.2	4.2
	16:52	18120093	0.0	2.8	12.6	0.59	6.5	1.0	133.8	1.7	206.5	4.2
	16:54	18120094	0.0	2.8	12.4	0.59	6.3	1.0	129.7	1.7	212.0	4.1
	16:56	18120095	0.0	2.8	12.3	0.59	6.2	1.0	128.3	1.7	214.2	4.1
	16:58	18120096	0.0	2.8	12.0	0.58	5.9	0.95	128.1	1.6	206.2	4.0
	17:00	18120097	0.0	3.8	11.9	0.27	4.9	0.91	128.4	1.6	136.0	3.9
	17:02	18120098	0.0	3.7	10.8	0.27	4.2	0.88	120.9	1.5	99.9	3.8

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/12/97	17:05	18120099	0.0	3.6	10.1	0.27	4.4	0.87	112.8	1.5	162.8	3.7
	17:07	18120100	0.0	3.6	10.0	0.26	4.4	0.87	107.0	1.5	191.6	3.7
	17:09	18120101	0.0	3.6	10.1	0.26	4.5	0.87	105.5	1.5	193.1	3.7
	17:11	18120102	0.0	3.6	10.3	0.26	4.6	0.88	105.9	1.5	189.1	3.7
	17:13	18120103	0.0	3.7	10.6	0.27	4.9	0.89	108.9	1.5	182.0	3.8
	17:15	18120104	0.0	3.7	10.9	0.27	5.0	0.90	111.4	1.6	172.5	3.9
	17:17	18120105	0.0	3.8	10.9	0.28	5.0	0.90	113.4	1.6	160.2	3.8
	17:20	18120106	0.0	3.8	11.1	0.27	5.1	0.89	114.4	1.6	150.9	3.8
	17:22	18120107	0.0	3.8	11.4	0.28	5.4	0.91	117.9	1.6	150.9	3.9
	17:24	18120108	0.0	3.9	11.8	0.28	5.6	0.92	120.5	1.6	152.6	3.9
	17:26	18120109	0.0	4.0	12.1	0.29	5.8	0.94	122.6	1.6	161.9	4.0
	17:28	18120110	0.0	4.0	12.4	0.29	5.9	0.95	124.9	1.6	185.0	4.0
	17:30	18120111	0.0	4.0	12.5	0.29	6.0	0.95	124.9	1.6	200.5	4.0
	17:32	18120112	0.0	4.0	12.2	0.29	5.8	0.94	122.2	1.6	186.1	4.0
	17:34	18120113	0.0	4.0	12.2	0.29	5.9	0.94	123.1	1.6	172.2	4.0
	17:37	18120114	0.0	4.1	12.5	0.29	6.2	1.0	129.5	1.7	172.1	4.1
	17:39	18120115	0.0	4.1	12.8	0.30	6.2	1.0	130.8	1.7	181.6	4.1
	17:41	18120116	0.0	4.1	12.7	0.29	6.1	1.0	128.5	1.7	186.1	4.1
	17:43	18120117	0.0	4.1	12.2	0.29	5.8	0.95	123.7	1.7	175.7	4.1
	17:45	18120118	0.0	4.0	11.7	0.29	5.6	0.93	118.9	1.6	162.2	4.0
	17:47	18120119	0.0	3.9	11.4	0.29	5.4	0.93	115.7	1.6	150.7	3.9
	17:49	18120120	0.0	3.9	11.2	0.29	5.4	0.93	114.7	1.6	146.7	3.9
	17:52	18120121	0.0	3.9	11.3	0.29	5.4	0.93	116.0	1.6	144.0	4.0
	17:54	18120122	0.0	4.0	11.4	0.29	5.5	0.94	116.9	1.6	135.8	4.0
	18:19	18120134	0.0	3.7	9.9	0.28	4.9	0.89	103.8	1.6	153.1	3.8
	18:21	18120135	0.0	3.7	9.9	0.28	5.0	0.89	105.0	1.6	154.6	3.8

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a,b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/12/97	18:24	18120136	0.0	3.9	10.9	0.28	5.3	0.91	112.8	1.6	167.9	3.9
	18:26	18120137	0.0	4.0	11.6	0.29	5.6	0.93	118.8	1.6	181.6	4.0
	18:28	18120138	0.0	4.0	11.9	0.29	5.7	0.94	119.3	1.7	183.6	4.0
	18:30	18120139	0.0	4.0	11.8	0.29	5.6	0.93	117.9	1.6	181.2	4.0
	18:32	18120140	0.0	4.0	11.9	0.29	5.7	0.94	118.3	1.7	178.6	4.0
	18:34	18120141	0.0	4.0	12.0	0.29	5.7	0.94	119.3	1.7	173.7	4.0
	18:36	18120142	0.0	4.1	12.0	0.30	5.8	0.95	120.4	1.7	174.4	4.0
	18:38	18120143	0.0	4.1	12.0	0.30	5.8	1.0	120.7	1.7	176.9	4.1
	18:41	18120144	0.0	4.1	11.8	0.30	5.7	0.95	119.6	1.7	181.9	4.0
	18:43	18120145	0.0	4.0	11.5	0.30	5.5	0.94	117.5	1.7	179.9	4.0
	18:45	18120146	0.0	3.9	11.2	0.29	5.2	0.92	113.4	1.7	171.0	3.9
	18:47	18120147	0.0	3.9	10.9	0.29	5.1	0.92	111.6	1.6	164.2	3.9
	18:49	18120148	0.0	3.9	10.9	0.29	5.1	0.92	112.2	1.7	161.6	3.9
	18:51	18120149	0.0	3.9	10.8	0.29	5.1	0.92	112.1	1.7	159.1	3.9
	18:53	18120150	0.0	3.9	10.3	0.29	5.2	0.92	110.1	1.6	167.3	3.9
	18:56	18120151	0.0	3.9	10.6	0.29	5.4	0.93	114.1	1.6	180.2	4.0
	18:58	18120152	0.0	3.9	10.5	0.29	5.3	0.92	111.2	1.6	169.5	3.9
	19:00	18120153	0.0	3.9	10.6	0.29	5.5	0.93	113.6	1.7	161.6	4.0
	19:02	18120154	0.0	4.0	11.1	0.29	5.7	0.95	117.2	1.7	163.2	4.0
	19:04	18120155	0.0	4.0	11.5	0.30	5.9	1.0	121.0	1.7	164.9	4.1
	19:08	18120156	0.0	4.0	11.5	0.30	5.9	1.0	119.0	1.7	166.5	4.1
	19:10	18120157	0.0	4.0	11.5	0.30	5.8	1.0	118.8	1.7	166.0	4.1
	19:13	18120158	0.0	4.0	11.4	0.30	5.7	1.0	116.8	1.7	168.6	4.1
	19:15	18120159	0.0	4.0	11.2	0.30	5.7	1.0	116.8	1.7	169.6	4.1
	19:17	18120160	0.0	4.0	11.2	0.29	5.7	0.95	115.8	1.7	180.6	4.0
	19:19	18120161	0.0	4.0	11.2	0.29	5.6	0.95	114.9	1.7	189.3	4.0

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/12/97	19:21	18120162	0.0	3.9	11.0	0.29	5.5	0.94	113.3	1.7	187.2	4.0
	19:23	18120163	0.0	3.9	11.0	0.29	5.5	0.94	112.8	1.7	178.0	4.0
	19:25	18120164	0.0	3.9	11.1	0.29	5.5	0.93	113.1	1.7	171.1	4.0
	19:28	18120165	0.0	3.9	11.1	0.29	5.5	0.93	114.0	1.7	171.6	4.0
	19:30	18120166	0.0	3.9	11.4	0.29	5.6	0.94	115.9	1.7	181.6	4.0
	19:32	18120167	0.0	3.9	11.9	0.29	5.6	0.94	117.9	1.7	187.3	4.0
	19:34	18120168	0.0	4.0	12.0	0.29	5.7	0.94	119.2	1.7	188.3	4.0
	19:36	18120169	0.0	4.0	12.1	0.29	5.7	0.94	120.2	1.7	191.3	4.0
	19:38	18120170	0.0	4.0	12.1	0.30	5.7	0.94	120.6	1.7	194.0	4.0
	19:40	18120171	0.0	4.0	12.1	0.30	5.7	0.94	120.6	1.7	195.8	4.0
	19:42	18120172	0.0	4.0	12.0	0.30	5.7	0.94	119.5	1.7	195.0	4.0
	19:45	18120173	0.0	4.0	11.9	0.30	5.7	0.94	119.6	1.7	194.9	4.0
8/12/97 Average -->			0.9	3.4	11.4	0.39	5.7	0.93	121.4	1.6	175.2	3.9

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a,b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/13/97	9:30	IU0813A	9.3	2.1	8.4	0.47	5.4	0.83	110.3	1.3	127.9	3.6
	9:36	IU0813B	9.8	2.1	9.0	0.49	5.7	0.85	113.9	1.3	127.1	3.6
	9:42	IU0813C	9.7	2.1	9.3	0.49	5.7	0.86	113.8	1.3	138.2	3.7
	9:45	IU0813D	9.5	2.1	9.3	0.49	5.7	0.86	112.3	1.3	146.2	3.7
	10:00	IS0813A	43.9	1.8	6.8	0.41	3.2	0.78	81.4	1.1	115.4	3.0
	10:06	IS0813B	44.3	1.6	6.9	0.20	3.1	0.77	81.1	1.1	108.6	3.0
	10:11	IS0813C	43.8	1.6	6.8	0.20	3.0	0.77	80.3	1.1	109.3	3.0
	10:16	IS0813D	43.4	1.6	6.6	0.20	2.9	0.77	78.6	1.1	104.4	3.0
	11:23	18130001	7.7	2.3	11.2	0.53	6.7	0.93	131.5	1.5	204.2	3.9
	11:25	18130002	7.5	2.3	11.1	0.54	6.6	0.91	129.5	1.5	202.0	3.9
	11:28	18130003	7.2	2.3	11.0	0.53	6.4	0.91	129.7	1.5	192.4	3.9
	11:30	18130004	6.9	2.3	10.9	0.53	6.3	0.91	127.8	1.5	184.4	3.9
	11:32	18130005	6.8	2.3	10.7	0.53	6.2	0.90	125.1	1.5	179.2	3.9
	11:34	18130006	6.6	2.3	10.7	0.53	6.2	0.90	124.3	1.5	184.8	3.9
	11:36	18130007	6.7	2.3	10.8	0.52	6.3	0.90	124.0	1.5	188.1	3.9
	11:38	18130008	6.6	2.3	10.8	0.52	6.4	0.90	123.7	1.5	187.3	3.9
	11:40	18130009	6.6	2.3	10.8	0.52	6.3	0.90	124.4	1.5	184.2	3.8
	11:42	18130010	6.5	2.3	10.9	0.52	6.3	0.90	124.8	1.5	182.1	3.9
	11:45	18130011	6.5	2.3	11.0	0.53	6.5	0.90	127.8	1.5	179.4	3.9
	11:47	18130012	6.6	2.3	11.1	0.53	6.5	0.91	129.0	1.5	181.2	3.9
	11:49	18130013	6.7	2.3	11.1	0.53	6.5	0.91	128.9	1.5	181.0	3.9
	11:51	18130014	6.5	2.3	11.1	0.53	6.5	0.91	130.9	1.5	184.6	3.9
	11:53	18130015	6.4	2.3	11.2	0.53	6.5	0.91	129.9	1.5	192.3	3.9
	12:43	18130038	4.9	2.3	10.7	0.53	6.3	0.91	126.6	1.5	170.2	3.9
	12:45	18130039	4.7	2.3	10.9	0.53	6.3	0.91	125.6	1.5	167.5	3.9
	12:48	18130040	4.6	2.3	11.1	0.53	6.4	0.92	128.1	1.5	174.3	3.9

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/13/97	12:50	18130041	4.6	2.4	11.2	0.54	6.5	0.92	130.7	1.5	181.9	3.9
	12:52	18130042	4.5	2.3	11.1	0.54	6.5	0.92	128.2	1.5	195.2	3.9
	12:54	18130043	4.2	2.3	11.0	0.53	6.3	0.91	125.1	1.5	193.3	3.9
	12:56	18130044	4.1	2.3	10.9	0.53	6.3	0.90	125.0	1.5	183.9	3.9
	12:58	18130045	3.9	2.3	10.8	0.53	6.3	0.90	124.7	1.5	183.0	3.9
	13:00	18130046	4.0	2.3	10.9	0.53	6.3	0.90	125.0	1.5	185.2	3.9
	13:02	18130047	4.0	2.3	10.9	0.54	6.3	0.91	126.2	1.5	185.5	3.9
	15:53	18130094	0.0	2.5	10.4	0.25	6.0	0.88	116.8	1.4	169.3	3.8
	15:55	18130095	0.00	2.5	9.8	0.25	5.7	0.86	110.9	1.4	162.3	3.7
	15:57	18130096	0.00	2.5	9.5	0.25	5.6	0.86	109.6	1.5	162.7	3.7
	16:00	18130097	0.00	2.6	9.9	0.26	5.8	0.88	114.7	1.5	174.2	3.8
	16:04	18130098	0.0	2.7	10.5	0.28	5.9	1.1	121.6	1.6	181.9	4.2
8/13/97 Average --->			4.8	2.4	10.8	0.49	6.3	0.91	125.0	1.5	182.6	3.9

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/14/97	8:32	18140001	12.1	1.9	8.5	0.23	7.5	0.86	111.6	1.3	133.9	3.7
	8:34	18140002	11.7	2.1	8.6	0.49	6.2	0.87	112.8	1.3	142.7	3.7
	8:36	18140003	11.7	2.1	8.8	0.48	5.8	0.87	111.5	1.3	144.0	3.7
	8:38	18140004	11.6	2.1	8.8	0.48	5.7	0.86	109.8	1.3	141.0	3.7
	8:40	18140005	11.4	2.1	8.9	0.48	5.7	0.86	109.4	1.3	135.1	3.7
	8:42	18140006	11.4	2.1	9.1	0.49	5.8	0.86	112.6	1.3	132.2	3.7
	8:50	18140007	10.8	2.1	9.1	0.48	5.6	0.85	107.9	1.3	141.5	3.6
8/14/97	8:52	18140008	10.7	2.1	9.1	0.48	5.5	0.84	107.1	1.3	151.3	3.6
	8:54	18140009	10.4	2.1	9.0	0.47	5.4	0.84	106.3	1.3	164.0	3.6
	8:56	18140010	10.3	2.1	9.0	0.47	5.4	0.84	105.2	1.3	167.4	3.6
	8:59	18140011	10.0	2.0	8.8	0.47	5.3	0.83	102.8	1.3	154.6	3.5
	9:36	18140028	9.0	2.0	9.1	0.24	5.6	0.97	113.2	1.4	147.5	3.7
	9:38	18140029	8.7	2.0	9.0	0.24	5.5	0.97	109.9	1.3	158.9	3.7
	9:40	18140030	8.4	2.0	8.9	0.24	5.5	0.96	107.6	1.3	162.4	3.7
Spike On	9:42	18140031	8.8	1.9	8.8	0.24	5.2	0.94	103.6	1.3	158.9	3.6
	9:44	18140032	7.4	1.8	8.1	0.23	4.7	0.90	94.2	1.3	149.6	3.5
	9:46	18140033	24.3	1.7	6.8	0.20	3.5	0.81	77.1	1.1	121.9	3.1
	9:48	18140034	36.9	1.5	5.7	0.19	2.8	0.75	66.8	1.0	101.0	2.9
	9:50	18140035	39.6	1.5	5.4	0.19	2.6	0.74	64.9	1.0	93.4	2.9
	9:53	18140036	40.2	1.5	5.3	0.19	2.6	0.74	64.5	1.0	92.5	2.9
	9:55	18140037	40.4	1.5	5.3	0.19	2.6	0.74	65.0	1.0	93.8	2.8
Spike Off	9:57	18140038	40.3	1.5	5.3	0.19	2.6	0.74	65.7	1.0	94.7	2.8
	9:59	18140039	23.6	1.7	6.4	0.21	3.6	0.81	79.7	1.1	117.3	3.1
	10:01	18140040	10.0	1.8	7.4	0.22	4.5	0.87	92.5	1.2	133.3	3.4
	10:03	18140041	7.1	1.8	7.7	0.23	4.7	0.87	93.6	1.3	136.8	3.4
	10:05	18140042	6.5	1.8	7.9	0.23	5.1	0.79	94.9	1.3	139.1	3.4

TABLE B-1. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Inlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/14/97	10:08	18140043	6.4	1.9	8.1	0.23	5.2	0.80	97.4	1.3	146.7	3.4
	10:10	18140044	7.3	2.0	8.9	0.24	5.7	0.83	108.2	1.3	162.4	3.6
	10:12	18140045	7.6	2.0	9.3	0.25	5.9	0.85	116.0	1.4	164.1	3.6
8/14/97 Average -->			9.6	2.0	8.8	0.36	5.6	0.86	107.3	1.3	148.7	3.6

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	10:35	IS0811A	1202	22.5	0.0	0.4	5.3	1.0	0.0	2.4	0.0	1.1
	10:37	IS0811B	1183	22.1	0.0	0.4	5.1	1.0	0.0	2.4	0.0	1.1
	10:41	IS0811C	1153	21.3	0.0	0.4	5.0	0.9	0.0	2.3	0.0	1.1
	10:43	IS0811D	1132	20.8	0.0	0.4	4.8	0.9	0.0	2.3	0.0	1.0
	10:53	IU0811A	1186	22.7	0.0	0.4	5.8	1.0	0.0	2.4	0.0	1.1
	11:00	IU0811B	1308	26.0	0.0	0.5	7.0	1.1	0.0	2.6	0.0	1.2
	11:02	IU0811C	1456	31.2	0.0	0.5	7.9	1.2	0.0	2.9	1.8	1.3
	11:05	IU0811D	1507	33.4	0.0	0.5	8.7	1.2	0.0	3.0	2.3	1.3
	11:07	IU0811E	1531	34.4	0.0	0.5	9.5	1.2	0.0	3.0	2.6	1.3
	13:32	18120022	1588	36.5	5.6	0.5	5.6	1.3	0.0	3.1	2.7	1.4
	13:34	18120023	1566	35.7	4.6	0.5	5.6	1.3	0.0	3.1	2.4	1.4
	13:37	18120024	1556	35.2	4.1	0.5	5.6	1.3	0.0	3.0	2.3	1.4
	13:39	18120025	1554	35.3	3.9	0.5	5.7	1.3	0.0	3.0	2.3	1.4
	13:41	18120026	1553	35.4	4.0	0.5	5.7	1.3	0.0	3.0	2.3	1.4
	13:43	18120027	1559	35.7	4.3	0.5	5.6	1.3	0.0	3.1	2.3	1.4
	13:46	18120028	1579	36.7	4.9	0.5	5.7	1.3	0.0	3.1	2.5	1.4
	13:48	18120029	1580	36.5	5.4	0.5	5.7	1.3	0.0	3.1	2.6	1.4
	13:50	18120030	1585	36.7	5.5	0.5	5.8	1.3	0.0	3.1	2.5	1.4
	13:53	18120031	1572	36.3	5.0	0.5	5.7	1.3	0.0	3.1	2.4	1.4
	13:55	18120032	1559	35.9	4.5	0.5	5.7	1.3	0.0	3.1	2.3	1.4
	13:57	18120033	1569	36.2	4.6	0.5	5.9	1.3	0.0	3.1	2.4	1.4
	13:59	18120034	1578	36.5	4.6	0.5	5.7	1.3	3.5	3.1	2.6	1.4
	14:01	18120035	1583	36.8	4.7	0.5	5.7	1.3	0.0	3.1	2.5	1.4
	14:03	18120036	1583	36.8	5.0	0.5	5.7	1.3	0.0	3.1	2.6	1.4
	14:05	18120037	1574	36.4	5.6	0.5	5.6	1.3	0.0	3.1	2.4	1.4
	14:32	18120049	1563	36.8	6.7	0.6	4.8	1.4	0.0	3.2	2.1	1.5

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	14:34	18120050	1572	37.0	6.8	0.6	4.9	1.4	0.0	3.2	2.2	1.5
	14:36	18120051	1578	37.2	6.8	0.6	5.3	1.4	0.0	3.2	2.4	1.5
	14:37	18120052	1565	37.2	6.3	0.6	5.1	1.4	3.7	3.2	3.4	1.4
	14:40	18120053	1537	36.3	5.7	0.5	5.2	1.4	4.1	3.2	3.1	1.4
	14:42	18120054	1525	36.0	5.2	0.5	5.3	1.4	4.2	3.2	3.0	1.4
	14:44	18120055	1522	35.7	5.1	0.5	5.2	1.3	4.4	3.2	2.8	1.3
	14:46	18120056	1547	36.5	5.1	0.5	5.2	1.4	4.4	3.2	2.9	1.3
	14:48	18120057	1564	37.1	5.0	0.5	5.1	1.4	4.5	3.2	3.0	1.4
	14:50	18120058	1554	37.0	4.9	0.5	5.1	1.4	4.6	3.2	3.0	1.4
	14:52	18120059	1542	36.5	5.2	0.5	5.0	1.4	4.5	3.2	2.8	1.3
	14:54	18120060	1534	36.1	5.6	0.5	5.0	1.3	4.4	3.2	2.6	1.3
	14:57	18120061	1529	36.0	5.3	0.5	5.0	1.3	4.6	3.2	2.4	1.3
	14:59	18120062	1520	35.7	4.7	0.5	5.0	1.3	5.0	3.2	2.2	1.3
	15:01	18120063	1537	36.2	4.3	0.5	4.9	1.4	5.3	3.2	2.4	1.3
	15:03	18120064	1556	36.7	4.4	0.5	5.0	1.4	5.2	3.2	2.5	1.3
	15:05	18120065	1561	36.9	4.3	0.5	4.9	1.4	5.2	3.2	2.6	1.4
	15:07	18120066	1565	37.2	4.1	0.5	4.9	1.4	5.3	3.2	2.6	1.4
	15:09	18120067	1578	37.5	4.0	0.5	4.9	1.4	5.4	3.2	2.7	1.4
	16:48	18120091	1600	40.7	5.7	0.6	4.7	1.5	7.8	3.5	2.5	1.5
	16:50	18120092	1615	41.6	6.0	0.6	4.8	1.5	7.2	3.5	2.6	1.5
	16:52	18120093	1626	41.7	6.0	0.6	4.9	1.5	7.0	3.5	2.5	1.5
	16:54	18120094	1610	40.9	5.5	0.6	4.9	1.5	7.3	3.5	2.3	1.5
	16:56	18120095	1584	40.0	4.8	0.6	4.9	1.5	7.7	3.5	2.0	1.5
	16:58	18120096	1544	38.3	3.9	0.6	4.8	1.5	8.7	3.5	1.8	1.5
	17:00	18120097	1327	31.1	3.5	0.5	4.5	1.4	9.5	3.3	0.0	1.4
	17:02	18120098	1216	28.3	4.0	0.5	4.1	1.4	9.5	3.2	0.0	1.4

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	17:05	18120099	1319	30.4	3.4	0.5	4.2	1.4	10.3	3.2	0.0	1.4
	17:07	18120100	1387	31.8	2.7	0.5	4.2	1.3	11.7	3.2	0.0	1.3
	17:09	18120101	1410	32.7	2.5	0.5	4.2	1.3	11.8	3.2	0.0	1.3
	17:11	18120102	1422	33.0	2.7	0.5	4.2	1.4	11.1	3.2	0.0	1.4
	17:13	18120103	1438	33.8	2.8	0.5	4.3	1.4	10.7	3.2	0.0	1.4
	17:15	18120104	1474	35.3	3.3	0.5	4.4	1.4	10.1	3.3	0.0	1.4
	17:17	18120105	1495	35.8	3.8	0.5	4.3	1.4	9.2	3.3	0.0	1.4
	17:20	18120106	1498	36.0	4.2	0.5	4.2	1.4	8.8	3.3	0.0	1.4
	17:22	18120107	1527	37.2	4.2	0.5	4.2	1.4	8.4	3.4	0.0	1.4
	17:24	18120108	1540	37.9	4.4	0.5	4.3	1.4	7.9	3.4	0.0	1.4
	17:26	18120109	1544	38.2	5.0	0.6	4.2	1.5	7.0	3.4	0.0	1.5
	17:28	18120110	1560	39.3	5.4	0.6	4.5	1.5	6.6	3.5	0.0	1.5
	17:30	18120111	1561	39.3	5.1	0.6	4.6	1.5	6.9	3.5	0.0	1.5
	17:32	18120112	1526	38.1	4.5	0.6	4.5	1.5	7.8	3.5	0.0	1.5
	17:34	18120113	1515	37.8	4.3	0.6	4.3	1.5	7.6	3.5	0.0	1.5
	17:37	18120114	1543	39.2	4.6	0.6	4.5	1.5	7.1	3.5	0.0	1.5
	17:39	18120115	1565	40.2	4.7	0.6	4.8	1.5	6.7	3.6	0.0	1.5
	17:41	18120116	1565	40.1	4.5	0.6	5.0	1.5	6.8	3.5	0.0	1.5
	17:43	18120117	1536	39.0	4.2	0.6	4.9	1.5	7.2	3.5	0.0	1.5
	17:45	18120118	1501	37.6	3.8	0.6	4.9	1.5	7.6	3.5	0.0	1.5
	17:47	18120119	1491	37.3	3.4	0.5	4.9	1.5	8.4	3.5	0.0	1.5
	17:49	18120120	1484	37.0	3.3	0.5	4.7	1.5	8.3	3.5	0.0	1.5
	17:52	18120121	1486	36.9	3.1	0.5	4.7	1.5	8.3	3.5	0.0	1.5
	17:54	18120122	1499	37.5	3.1	0.6	4.7	1.5	8.3	3.5	0.0	1.5
	18:19	18120134	1431	33.9	4.6	0.5	2.8	1.4	11.0	3.3	0.0	1.4
	18:21	18120135	1437	34.2	4.8	0.5	2.8	1.4	10.5	3.3	0.0	1.4

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	18:24	18120136	1486	36.3	5.1	0.5	3.8	1.5	9.4	3.4	0.0	1.5
	18:26	18120137	1524	38.0	5.0	0.6	4.5	1.5	8.7	3.5	0.0	1.5
	18:28	18120138	1534	38.5	4.6	0.6	4.8	1.5	8.5	3.5	0.0	1.5
	18:30	18120139	1531	38.3	4.3	0.6	4.7	1.5	8.3	3.5	0.0	1.5
	18:32	18120140	1538	38.7	3.9	0.6	4.8	1.5	8.2	3.5	0.0	1.5
	18:34	18120141	1542	39.0	3.7	0.6	4.9	1.5	8.0	3.5	0.0	1.5
	18:36	18120142	1546	39.5	3.5	0.6	5.0	1.5	7.9	3.6	0.0	1.5
	18:38	18120143	1546	39.5	3.5	0.6	5.0	1.5	7.6	3.6	0.0	1.5
	18:41	18120144	1543	39.4	3.3	0.6	5.0	1.5	7.8	3.6	0.0	1.5
	18:43	18120145	1526	38.6	2.7	0.6	4.9	1.5	8.3	3.6	0.0	1.5
	18:45	18120146	1508	37.6	2.4	0.5	5.0	1.5	8.9	3.5	0.0	1.5
	18:47	18120147	1496	37.1	2.2	0.5	4.9	1.5	9.1	3.5	0.0	1.5
	18:49	18120148	1490	36.9	2.1	0.5	4.6	1.5	9.2	3.5	0.0	1.5
	18:51	18120149	1488	36.9	2.3	0.5	4.5	1.5	8.9	3.5	0.0	1.5
	18:53	18120150	1489	36.4	3.0	0.5	3.1	1.5	9.2	3.5	0.0	1.5
	18:56	18120151	1521	37.5	3.1	0.6	2.8	1.5	9.5	3.5	0.0	1.5
	18:58	18120152	1491	36.4	3.3	0.5	2.8	1.5	9.9	3.5	0.0	1.5
	19:00	18120153	1508	37.2	3.5	0.6	2.9	1.5	10.0	3.5	0.0	1.5
	19:02	18120154	1543	38.6	3.8	0.6	2.8	1.5	9.9	3.5	0.0	1.5
	19:04	18120155	1560	39.2	4.3	0.6	2.8	1.5	9.5	3.6	0.0	1.5
	19:08	18120156	1551	39.2	4.8	0.6	2.8	1.5	9.5	3.6	0.0	1.5
	19:10	18120157	1552	39.0	5.0	0.6	2.9	1.5	9.7	3.6	0.0	1.5
	19:13	18120158	1544	38.7	5.3	0.6	2.8	1.5	9.5	3.6	0.0	1.5
	19:15	18120159	1544	38.6	5.6	0.6	2.7	1.5	9.7	3.6	0.0	1.5
	19:17	18120160	1550	38.7	5.7	0.6	2.7	1.5	9.6	3.5	0.0	1.5
	19:19	18120161	1550	38.6	5.8	0.6	2.7	1.5	9.5	3.5	0.0	1.5

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	19:21	18120162	1546	38.3	5.9	0.6	2.6	1.5	9.7	3.5	0.0	1.5
	19:23	18120163	1544	38.3	5.7	0.6	2.6	1.5	10.0	3.5	0.0	1.5
	19:25	18120164	1543	38.2	5.5	0.6	2.7	1.5	10.3	3.5	0.0	1.5
	19:28	18120165	1544	38.1	5.7	0.6	2.6	1.5	10.1	3.5	0.0	1.5
	19:30	18120166	1556	38.5	5.8	0.6	2.8	1.5	9.8	3.5	0.0	1.5
	19:32	18120167	1570	39.1	5.4	0.6	3.5	1.5	9.4	3.5	0.0	1.5
	19:34	18120168	1572	39.3	5.3	0.6	3.8	1.5	8.9	3.5	0.0	1.5
	19:36	18120169	1573	39.4	5.3	0.6	3.8	1.5	8.4	3.5	0.0	1.5
	19:38	18120170	1565	39.1	5.3	0.6	3.8	1.5	8.2	3.6	0.0	1.5
	19:40	18120171	1563	39.1	4.8	0.6	3.8	1.5	8.3	3.6	0.0	1.5
	19:42	18120172	1558	38.9	4.3	0.6	3.8	1.5	8.5	3.6	0.0	1.5
	19:45	18120173	1560	39.0	4.1	0.6	3.8	1.5	8.5	3.6	0.0	1.5
8/12/97 Average ---->			1531	37.2	4.5	0.5	4.5	1.4	6.7	3.4	1.0	1.4

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/13/97	9:30	IU0813A	1411	30.0	0.0	0.5	6.4	1.2	0.0	2.8	1.6	1.3
	9:36	IU0813B	1451	31.5	0.6	0.5	6.4	1.2	0.0	2.9	1.9	1.3
	9:42	IU0813C	1451	31.6	1.3	0.5	6.3	1.2	0.0	2.9	2.0	1.3
	9:45	IU0813D	1448	31.5	1.7	0.5	6.1	1.2	0.0	2.9	2.0	1.3
	10:00	IS0813A	1212	22.9	0.6	0.4	5.4	1.0	0.0	2.4	1.2	1.1
	10:06	IS0813B	1184	22.2	0.0	0.4	5.3	1.0	0.0	2.4	0.0	1.1
	10:11	IS0813C	1169	22.0	0.0	0.4	5.3	1.0	0.0	2.4	0.0	1.1
	10:16	IS0813D	1170	21.9	0.0	0.4	5.2	1.0	0.0	2.4	0.0	1.1
	11:23	18130001	1602	37.8	0.7	0.5	5.5	1.3	0.0	3.2	2.6	1.4
	11:25	18130002	1599	37.4	0.0	0.5	5.5	1.3	0.0	3.2	2.5	1.4
	11:28	18130003	1577	36.6	0.0	0.5	5.5	1.3	0.0	3.2	2.4	1.4
	11:30	18130004	1562	36.2	0.0	0.5	5.6	1.3	0.0	3.2	2.3	1.4
	11:32	18130005	1558	35.9	0.0	0.5	5.4	1.3	0.0	3.1	2.1	1.4
	11:34	18130006	1565	36.2	0.0	0.5	5.5	1.3	0.0	3.1	2.2	1.4
	11:36	18130007	1566	36.2	0.0	0.5	5.5	1.3	0.0	3.1	2.2	1.4
	11:38	18130008	1571	36.4	0.0	0.5	5.4	1.3	0.0	3.1	2.2	1.4
	11:40	18130009	1577	36.5	0.0	0.5	5.3	1.3	0.0	3.1	2.1	1.4
	11:42	18130010	1580	36.6	0.0	0.5	5.3	1.3	0.0	3.1	2.2	1.4
	11:45	18130011	1585	36.7	0.0	0.5	5.3	1.3	0.0	3.1	2.4	1.4
	11:47	18130012	1594	37.1	0.5	0.5	5.3	1.3	0.0	3.1	2.5	1.4
	11:49	18130013	1591	36.9	0.8	0.5	5.2	1.3	0.0	3.1	2.5	1.4
	11:51	18130014	1585	36.8	1.1	0.5	5.2	1.3	0.0	3.2	2.7	1.4
	11:53	18130015	1582	36.8	1.3	0.5	5.4	1.3	0.0	3.1	2.6	1.4
	12:43	18130038	1536	34.8	1.5	0.5	4.1	1.3	0.0	3.1	1.7	1.4
	12:45	18130039	1549	35.6	1.7	0.5	4.4	1.3	0.0	3.1	1.9	1.4
	12:48	18130040	1567	36.5	1.6	0.5	5.0	1.3	0.0	3.1	2.3	1.4

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/13/97	12:50	18130041	1568	36.7	1.7	0.5	5.2	1.3	0.0	3.2	2.3	1.4
	12:52	18130042	1570	36.7	1.8	0.5	5.3	1.3	0.0	3.2	2.2	1.4
	12:54	18130043	1555	36.0	0.9	0.5	5.3	1.3	0.0	3.1	2.1	1.4
	12:56	18130044	1547	35.7	0.0	0.5	5.2	1.3	0.0	3.1	2.0	1.4
	12:58	18130045	1546	35.7	0.0	0.5	5.2	1.3	0.0	3.1	2.1	1.4
	13:00	18130046	1550	35.9	0.0	0.5	5.2	1.3	0.0	3.1	1.9	1.4
	13:02	18130047	1560	36.3	0.0	0.5	5.2	1.3	0.0	3.1	2.1	1.4
	15:53	18130094	1518	33.4	0.0	0.5	1.8	1.3	0.0	3.1	0.0	1.4
	15:55	18130095	1473	31.7	0.0	0.5	0.0	1.3	0.0	3.0	0.0	1.4
	15:57	18130096	1459	31.5	0.0	0.5	0.0	1.3	0.0	3.1	0.0	1.4
	16:00	18130097	1501	33.2	0.0	0.5	0.0	1.4	0.0	3.2	0.0	1.5
	16:04	18130098	1544	36.3	0.0	0.6	0.0	1.4	0.0	3.4	0.0	1.6
8/13/97 Average -->			1558	35.9	0.5	0.5	4.4	1.3	0.0	3.1	1.9	1.4

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/14/97	8:32	18140001	1451	31.1	4.4	0.5	6.3	1.2	0.0	2.8	0.0	1.3
	8:34	18140002	1489	32.8	3.7	0.5	6.6	1.2	2.9	2.8	1.6	1.3
	8:36	18140003	1493	32.9	3.2	0.5	6.6	1.2	3.0	2.8	1.6	1.3
	8:38	18140004	1492	32.9	2.9	0.5	6.5	1.2	3.2	2.8	1.6	1.3
	8:40	18140005	1487	32.8	3.0	0.5	6.2	1.2	2.9	2.8	1.5	1.3
	8:42	18140006	1498	33.2	3.2	0.5	6.2	1.2	0.0	2.9	1.5	1.3
	8:50	18140007	1504	33.1	3.3	0.5	6.1	1.2	0.0	2.8	1.4	1.3
	8:52	18140008	1495	32.7	3.5	0.5	6.1	1.2	0.0	2.8	1.3	1.3
	8:54	18140009	1489	32.4	3.5	0.5	6.1	1.2	0.0	2.8	1.3	1.3
	8:56	18140010	1485	32.3	3.0	0.5	5.9	1.2	3.1	2.7	1.3	1.3
	8:59	18140011	1468	31.6	2.6	0.5	5.8	1.2	3.5	2.7	1.3	1.2
	9:36	18140028	1530	34.5	2.2	0.5	4.9	1.2	0.0	2.9	0.0	1.4
	9:38	18140029	1524	34.4	2.2	0.5	5.1	1.2	0.0	2.9	0.0	1.3
	9:40	18140030	1522	34.4	2.2	0.5	5.2	1.2	0.0	2.9	0.0	1.3
	Spike On	18140031	1485	32.8	2.4	0.5	5.3	1.2	0.0	2.8	0.0	1.3
	9:44	18140032	1416	30.1	2.3	0.5	4.9	1.1	0.0	2.7	0.0	1.3
	9:46	18140033	1270	24.8	2.0	0.4	5.1	1.0	0.0	2.4	0.0	1.1
	9:48	18140034	1177	22.0	1.6	0.4	4.5	0.9	2.8	2.2	0.0	1.04
	9:50	18140035	1157	21.5	1.5	0.4	4.2	0.9	2.6	2.2	0.0	1.02
	9:53	18140036	1158	21.5	1.4	0.4	4.0	0.9	2.6	2.2	0.0	1.02
	9:55	18140037	1170	21.7	1.3	0.4	4.0	0.9	2.5	2.2	0.0	1.02
	Spike Off	18140038	1175	21.8	1.1	0.4	3.9	0.9	2.3	2.2	0.0	1.02
	9:59	18140039	1297	25.5	0.8	0.4	4.0	1.0	0.0	2.4	0.0	1.1
	10:01	18140040	1394	28.9	0.6	0.5	4.2	1.1	0.0	2.6	0.0	1.2
	10:03	18140041	1419	29.8	0.0	0.5	4.3	1.1	0.0	2.7	0.0	1.3
	10:05	18140042	1427	30.2	0.0	0.5	4.3	1.1	0.0	2.7	0.0	1.3

TABLE B-1. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Inlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/14/97	10:08	18140043	1449	30.9	0.0	0.5	4.3	1.2	0.0	2.7	0.0	1.3
	10:10	18140044	1504	33.2	0.0	0.5	4.4	1.2	0.0	2.9	0.0	1.3
	10:12	18140045	1509	33.7	0.0	0.5	4.5	1.2	0.0	3.0	0.0	1.4
8/14/97 Average -->			1486	32.6	2.3	0.5	5.6	1.2	1.0	2.8	0.8	1.3

^a Bold face type indicates samples that were spiked with toluene or SF₆.

^b Shaded rows indicate times when the process was down.

^c Unc is the estimated uncertainty in each measurement. Typically the uncertainty is greater for lower concentrations.

TABLE B-2. FTIR RESULTS (ppm) AT THE WCI BAGHOUSE OUTLET

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc	
8/12/97	11:17	OU0811A	7.6	2.1	8.8	0.5	5.0	0.8	108.7	1.3	149.8	3.5	
	11:19	OU0811B	8.5	2.2	9.6	0.5	5.6	0.9	118.5	1.4	167.7	3.7	
	11:21	OU0811C	9.0	2.2	10.1	0.5	6.0	0.9	123.5	1.4	174.2	3.7	
	11:24	OU0811D	8.8	2.2	10.0	0.5	5.9	0.8	120.2	1.4	171.3	3.6	
	11:32	OS0811A	22.9	2.0	8.8	0.5	4.3	0.9	99.7	1.3	143.2	3.3	
	11:34	OS0811B	23.2	2.0	8.7	0.4	4.2	0.9	99.0	1.2	141.5	3.3	
	11:39	OS0811C	23.0	1.9	8.3	0.4	4.0	0.8	95.1	1.2	137.3	3.3	
	11:41	OS0811D	23.1	2.0	8.5	0.4	4.1	0.9	96.6	1.2	146.2	3.3	
	14:08	18120038	4.8	2.3	11.6	0.5	6.1	0.9	130.0	1.5	175.3	3.9	
	14:10	18120039	4.7	2.3	11.4	0.5	6.0	0.9	127.2	1.5	172.3	3.8	
	14:12	18120040	4.6	2.3	11.4	0.5	5.9	0.9	127.3	1.5	174.2	3.8	
	14:14	18120041	4.5	2.3	11.3	0.5	6.0	0.9	128.0	1.5	176.8	3.9	
	14:16	18120042	4.3	2.3	11.3	0.5	6.0	0.9	126.4	1.5	169.2	3.8	
	14:18	18120043	4.4	2.4	11.4	0.5	6.1	0.9	130.7	1.5	163.4	3.9	
	14:20	18120044	4.4	2.4	11.5	0.5	6.3	0.9	132.6	1.5	163.4	3.9	
	14:24	18120045	4.3	2.4	11.7	0.6	6.4	0.9	141.0	1.5	156.2	4.0	
	14:26	18120046	4.2	2.4	11.7	0.6	6.4	0.9	139.3	1.5	151.2	4.0	
	14:28	18120047	4.0	2.4	11.5	0.6	6.4	0.9	132.9	1.5	153.1	4.0	
	14:30	18120048	3.9	2.4	11.5	0.6	6.3	0.9	132.9	1.5	156.7	4.0	
	15:12	18120068	0.0	2.6	11.4	0.5	6.1	0.9	129.7	1.5	163.4	3.9	
	15:14	18120069	0.0	2.6	11.5	0.5	6.2	0.9	132.3	1.6	164.5	4.0	
	15:16	18120070	0.0	2.6	11.4	0.6	6.4	0.9	134.0	1.6	164.9	4.0	
	15:18	18120071	0.0	2.6	11.4	0.5	6.3	0.9	134.4	1.6	160.5	4.0	
	15:20	18120072	0.0	2.6	11.4	0.5	6.3	0.9	135.2	1.6	159.4	4.0	
	Process Down	15:22	18120073	0.0	2.6	11.2	0.5	5.7	0.9	133.0	1.5	119.2	3.9
		15:24	18120074	0.0	2.5	10.6	0.5	4.8	0.9	124.4	1.5	89.6	3.8
		15:26	18120075	0.0	2.4	10.2	0.5	4.5	0.9	119.0	1.4	91.5	3.7

TABLE B-2. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Outlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
Process Down 8/12/97	15:29	18120076	0.0	2.4	10.0	0.2	4.6	0.9	117.5	1.4	132.5	3.6
	15:31	18120077	0.0	2.3	9.1	0.2	4.5	0.8	107.1	1.4	179.4	3.5
	15:33	18120078	0.0	3.3	9.0	0.2	4.4	0.8	101.3	1.4	190.3	3.5
	15:35	18120079	0.0	3.3	9.1	0.2	4.5	0.8	99.5	1.4	192.3	3.5
	15:54	18120080	0.0	2.5	10.4	0.3	5.4	0.9	113.6	1.5	167.1	3.8
	15:56	18120081	0.0	2.5	10.8	0.3	5.6	0.9	115.7	1.5	169.6	3.8
	16:03	18120082	0.0	2.7	11.3	0.6	6.3	0.9	125.9	1.6	153.0	4.0
	16:05	18120083	0.0	2.7	11.5	0.6	6.5	1.0	128.2	1.6	166.2	4.1
	16:07	18120084	0.0	2.7	11.6	0.6	6.5	1.0	128.9	1.6	173.6	4.1
	16:09	18120085	0.0	2.7	11.6	0.6	6.5	1.0	127.8	1.6	176.7	4.1
	16:11	18120086	0.0	2.7	11.6	0.6	6.4	0.9	126.6	1.6	180.4	4.0
	16:14	18120087	0.0	2.7	11.7	0.6	6.4	0.9	126.5	1.6	180.5	4.0
	16:17	18120088	0.0	2.7	11.7	0.6	6.4	0.9	126.3	1.6	181.0	4.0
	16:19	18120089	0.0	2.7	11.7	0.6	6.3	0.9	125.7	1.6	184.3	4.0
	16:21	18120090	0.0	2.7	11.7	0.6	6.3	0.9	125.5	1.6	187.7	4.0
	17:56	18120123	0.0	4.0	11.7	0.3	5.7	0.9	119.9	1.7	132.2	4.0
	17:58	18120124	0.0	4.0	11.9	0.3	5.9	1.0	122.5	1.7	140.1	4.1
	18:00	18120125	0.0	4.1	12.0	0.3	6.3	1.0	129.4	1.7	160.9	4.2
	18:02	18120126	0.0	4.2	12.2	0.3	6.6	1.0	131.1	1.7	183.4	4.2
	18:04	18120127	0.0	4.0	11.4	0.3	5.9	0.9	117.7	1.7	176.4	4.0
	18:06	18120128	0.0	3.8	10.5	0.3	5.3	0.9	107.5	1.6	165.1	3.9
	18:09	18120129	0.0	3.8	10.1	0.3	5.0	0.9	103.8	1.6	165.4	3.8
	18:11	18120130	0.0	3.7	10.0	0.3	4.9	0.9	103.3	1.6	165.9	3.8
	18:13	18120131	0.0	3.7	9.8	0.3	4.9	0.9	102.5	1.6	167.1	3.8
	18:15	18120132	0.0	3.7	9.8	0.3	4.9	0.9	102.5	1.6	162.7	3.8
	18:17	18120133	0.0	3.7	9.8	0.3	4.9	0.9	103.2	1.6	156.8	3.8
8/12/97 Average -->			1.4	3.0	11.3	0.5	6.0	0.9	124.7	1.6	166.4	4.0

TABLE B-2. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Outlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/13/97	10:34	OS0813A	37.0	1.6	6.9	0.2	2.9	0.8	81.8	1.1	115.3	3.0
	10:38	OS0813B	39.6	1.8	7.9	0.2	3.6	0.8	96.3	1.2	137.8	3.3
	10:44	OS0813C	39.7	1.8	8.2	0.2	3.9	0.8	98.6	1.2	145.4	3.3
	10:49	OS0813D	39.8	1.8	8.3	0.2	3.9	0.8	98.3	1.2	127.9	3.3
	11:02	OU0813A	7.6	2.3	10.3	0.5	6.5	0.9	124.8	1.4	188.4	3.9
	11:07	OU0813B	7.4	2.3	10.4	0.5	6.5	0.9	124.1	1.5	180.6	3.9
	11:12	OU0813C	7.5	2.3	10.6	0.5	6.6	0.9	125.7	1.5	176.6	3.9
	11:18	OU0813D	7.5	2.3	10.8	0.5	6.7	0.9	128.8	1.5	186.5	3.9
	11:55	18130016	6.2	2.3	11.0	0.5	6.4	0.9	128.5	1.5	195.6	3.9
	11:58	18130017	6.2	2.3	10.9	0.5	6.4	0.9	126.8	1.5	197.0	3.8
	12:01	18130018	6.3	2.3	10.8	0.5	6.5	0.9	127.1	1.5	190.4	3.9
	12:03	18130019	6.4	2.3	10.8	0.5	6.5	0.9	127.9	1.5	182.8	3.9
	12:05	18130020	6.5	2.3	10.9	0.5	6.6	0.9	129.9	1.5	180.1	3.9
	12:07	18130021	6.4	2.3	11.0	0.5	6.7	0.9	131.1	1.5	182.4	3.9
	12:09	18130022	6.4	2.3	11.0	0.5	6.8	0.9	131.8	1.5	190.8	4.0
	12:11	18130023	6.4	2.4	11.0	0.5	6.8	0.9	131.7	1.5	202.0	4.0
	12:13	18130024	6.2	2.3	10.9	0.5	6.7	0.9	128.8	1.5	204.0	3.9
	12:16	18130025	6.0	2.3	10.9	0.5	6.6	0.9	126.7	1.5	202.1	3.9
	12:18	18130026	5.9	2.3	10.9	0.5	6.6	0.9	127.1	1.5	206.4	3.9
	12:20	18130027	5.8	2.3	10.9	0.5	6.5	0.9	128.0	1.5	205.4	3.9
	12:22	18130028	5.7	2.3	10.9	0.5	6.5	0.9	128.9	1.5	193.9	3.9
	12:24	18130029	5.6	2.3	10.8	0.5	6.5	0.9	125.7	1.5	183.0	3.9
	12:26	18130030	5.9	2.3	11.1	0.5	6.6	0.9	132.1	1.5	186.8	3.9
	12:28	18130031	6.0	2.4	11.2	0.5	6.7	0.9	134.0	1.5	190.7	4.0
	12:30	18130032	5.8	2.3	11.1	0.5	6.5	0.9	128.8	1.5	191.3	3.9
	12:33	18130033	5.7	2.3	11.1	0.5	6.5	0.9	132.6	1.5	187.6	3.9
	12:35	18130034	5.4	2.3	11.0	0.5	6.5	0.9	130.5	1.5	186.3	3.9

TABLE B-2. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Outlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/13/97	12:37	18130035	5.2	2.3	10.9	0.5	6.4	0.9	125.2	1.5	184.1	3.9
	12:39	18130036	5.1	2.3	10.8	0.5	6.3	0.9	123.9	1.5	181.7	3.9
	12:41	18130037	5.0	2.3	10.8	0.5	6.3	0.9	126.6	1.5	177.8	3.9
	13:05	18130048	4.3	2.3	11.1	0.5	6.4	0.9	128.5	1.5	185.8	3.9
	13:07	18130049	4.5	2.4	11.0	0.5	6.6	0.9	129.5	1.5	188.3	4.0
	13:09	18130050	4.7	2.4	11.0	0.5	6.7	0.9	131.1	1.5	190.3	4.0
	13:11	18130051	4.8	2.4	11.1	0.5	6.8	0.9	132.0	1.5	186.6	4.0
	13:13	18130052	4.7	2.4	11.0	0.5	6.6	0.9	130.5	1.5	190.7	3.9
	13:15	18130053	4.6	2.4	11.1	0.5	6.6	0.9	130.6	1.5	198.0	4.0
	13:17	18130054	4.6	2.4	11.1	0.5	6.6	0.9	129.5	1.5	196.7	3.9
	13:20	18130055	4.5	2.3	11.1	0.5	6.6	0.9	129.0	1.5	194.2	3.9
	13:22	18130056	4.6	2.4	11.1	0.5	6.6	0.9	129.4	1.5	193.1	3.9
	13:24	18130057	4.5	2.4	11.1	0.5	6.6	0.9	130.0	1.5	190.9	3.9
	13:26	18130058	4.4	2.4	11.1	0.5	6.5	0.9	129.3	1.5	190.3	3.9
	13:28	18130059	4.2	2.3	11.1	0.5	6.5	0.9	128.1	1.5	187.7	3.9
	13:30	18130060	4.2	2.4	11.1	0.5	6.5	0.9	128.0	1.5	185.0	3.9
	13:32	18130061	4.2	2.4	11.1	0.5	6.5	0.9	128.8	1.5	177.3	3.9
	13:34	18130062	4.1	2.4	11.1	0.5	6.5	0.9	130.2	1.5	169.9	4.0
	13:37	18130063	4.0	2.4	11.1	0.5	6.6	0.9	131.0	1.5	167.2	4.0
	13:39	18130064	4.0	2.4	11.1	0.5	6.6	0.9	131.0	1.5	170.0	4.0
	13:45	18130065	0.0	2.6	10.7	0.5	6.5	0.9	132.9	1.5	176.6	3.9
	13:47	18130066	0.0	2.5	10.6	0.5	6.4	0.9	129.6	1.5	173.3	3.9
	13:49	18130067	0.0	2.5	10.6	0.5	6.4	0.9	127.0	1.5	170.9	3.9
	13:52	18130068	0.0	2.5	10.6	0.5	6.5	0.9	126.9	1.5	171.7	3.9
	15:00	18130069	0.0	2.6	10.6	0.5	6.5	0.9	133.3	1.5	143.8	3.9
	15:02	18130070	0.0	2.6	10.5	0.5	6.4	0.9	129.4	1.5	147.5	3.9
	15:04	18130071	0.0	2.5	10.5	0.5	6.4	0.9	127.2	1.5	148.8	3.9

TABLE B-2. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Outlet Results)

Date	Time	File Name ^{a, b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/13/97	15:06	18130072	0.0	2.5	10.5	0.5	6.4	0.9	126.3	1.5	155.0	3.9
	15:08	18130073	0.0	2.5	10.4	0.5	6.2	0.9	125.7	1.5	151.8	3.9
	15:10	18130074	0.0	2.5	10.7	0.3	6.1	0.9	122.9	1.5	145.7	3.9
	15:13	18130075	0.0	2.5	10.7	0.3	6.0	0.9	122.9	1.5	145.1	3.9
	15:15	18130076	0.0	2.5	10.4	0.5	6.1	0.9	123.8	1.5	147.4	3.9
	15:17	18130077	0.0	2.5	10.5	0.5	6.2	0.9	126.0	1.5	147.5	3.9
	15:19	18130078	0.0	2.6	10.6	0.5	6.3	0.9	127.0	1.5	148.0	3.9
	15:21	18130079	0.0	2.5	10.6	0.5	6.3	0.9	127.1	1.5	154.3	3.9
	15:23	18130080	0.0	2.5	10.6	0.5	6.3	0.9	125.9	1.5	159.8	3.9
	15:25	18130081	0.0	2.5	10.6	0.5	6.2	0.9	125.6	1.5	160.7	3.9
	15:28	18130082	0.0	2.5	10.6	0.5	6.2	0.9	124.7	1.5	157.7	3.9
	15:30	18130083	0.0	2.5	10.5	0.5	6.1	0.9	123.7	1.5	153.8	3.9
	15:32	18130084	0.0	2.5	10.5	0.5	6.1	0.9	123.7	1.5	149.8	3.9
	15:34	18130085	0.0	2.5	10.6	0.5	6.2	0.9	124.8	1.5	148.0	3.9
	15:36	18130086	0.0	2.6	10.7	0.5	6.4	0.9	126.8	1.5	145.5	3.9
	15:38	18130087	0.0	2.6	10.7	0.5	6.5	0.9	127.6	1.5	142.2	3.9
	15:40	18130088	0.0	2.6	10.7	0.5	6.5	0.9	127.1	1.5	147.0	3.9
	15:42	18130089	0.0	2.6	10.7	0.5	6.4	0.9	126.1	1.5	154.7	3.9
	15:45	18130090	0.0	2.6	10.6	0.5	6.3	0.9	124.7	1.5	159.6	3.9
	15:47	18130091	0.0	2.5	10.5	0.5	6.3	0.9	122.4	1.5	159.9	3.9
	15:49	18130092	0.0	2.5	10.3	0.5	6.2	0.9	120.1	1.5	164.4	3.8
	15:51	18130093	0.0	2.5	10.6	0.3	6.1	0.9	119.4	1.5	172.4	3.8
8/13/97 Average -->			3.1	2.4	10.8	0.5	6.4	0.9	127.8	1.5	174.6	3.9

TABLE B-2. Continued. (Toluene, Hexane, Ethylene, Methane and SO₂ Outlet Results)

Date	Time	File Name ^{a,b}	Toluene	Unc ^c	Hexane	Unc	Ethylene	Unc	Methane	Unc	SO ₂	Unc
8/14/97	9:01	18140012	10.7	1.9	9.2	0.2	5.3	0.8	103.5	1.3	140.5	3.5
	9:04	18140013	10.6	1.9	9.2	0.2	5.3	0.8	104.6	1.3	126.8	3.5
	9:06	18140014	10.9	1.9	9.4	0.2	5.4	0.8	106.0	1.3	120.1	3.6
	9:08	18140015	10.9	1.9	9.7	0.2	5.6	0.8	108.0	1.3	121.2	3.6
	9:10	18140016	10.8	1.9	9.8	0.2	5.7	0.9	110.4	1.3	130.8	3.7
	9:12	18140017	18.4	2.0	9.5	0.5	4.4	0.9	98.9	1.3	127.9	3.4
	9:14	18140018	36.9	1.7	8.1	0.2	3.8	0.8	90.5	1.2	129.1	3.2
	9:16	18140019	40.0	1.7	7.3	0.2	3.5	0.8	85.7	1.1	129.0	3.2
	9:18	18140020	40.4	1.7	7.0	0.2	3.5	0.8	84.3	1.1	126.8	3.1
	9:21	18140021	40.4	1.7	6.9	0.2	3.4	0.8	83.3	1.1	122.7	3.1
	9:23	18140022	40.6	1.7	6.8	0.2	3.4	0.8	82.6	1.1	119.9	3.1
	9:25	18140023	36.5	1.7	7.0	0.2	3.7	0.8	86.2	1.1	124.4	3.2
	9:27	18140024	16.2	1.9	8.4	0.2	4.9	0.9	102.6	1.3	145.5	3.5
	9:29	18140025	10.4	1.9	8.9	0.2	5.3	1.0	108.5	1.3	143.1	3.7
	9:31	18140026	9.3	2.0	9.0	0.2	5.4	1.0	110.0	1.3	136.8	3.7
	9:33	18140027	9.1	2.0	9.1	0.2	5.5	1.0	112.6	1.3	137.1	3.7
8/14/97 Average -->			10.3	1.9	9.2	0.2	5.4	0.9	107.6	1.3	132.2	3.6

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	11:17	OU0811A	1429	30.3	0.0	0.5	7.0	1.2	0.0	2.8	1.6	1.2
	11:19	OU0811B	1508	33.3	0.0	0.5	7.0	1.2	0.0	2.9	2.1	1.3
	11:21	OU0811C	1534	34.2	0.0	0.5	6.6	1.3	0.0	3.0	2.2	1.3
	11:24	OU0811D	1517	33.3	0.0	0.5	6.5	1.2	0.0	2.9	2.0	1.3
	11:32	OS0811A	1407	28.6	0.6	0.5	6.1	1.1	0.0	2.6	1.5	1.2
	11:34	OS0811B	1401	28.2	0.6	0.5	6.1	1.1	0.0	2.6	1.4	1.2
	11:39	OS0811C	1368	27.2	0.7	0.5	5.8	1.1	3.4	2.6	1.2	1.2
	11:41	OS0811D	1397	28.2	0.7	0.5	6.0	1.1	3.4	2.6	1.4	1.2
	14:08	18120038	1554	35.7	5.8	0.5	5.2	1.3	0.0	3.1	2.3	1.4
	14:10	18120039	1547	35.2	5.8	0.5	4.9	1.3	3.6	3.1	2.2	1.4
	14:12	18120040	1546	35.3	5.7	0.5	4.8	1.3	3.8	3.1	2.2	1.4
	14:14	18120041	1545	35.3	5.7	0.5	4.8	1.3	3.8	3.1	2.2	1.4
	14:16	18120042	1539	35.1	5.7	0.5	4.8	1.3	3.9	3.1	2.2	1.4
	14:18	18120043	1548	35.5	5.9	0.5	4.7	1.3	3.8	3.1	2.2	1.4
	14:20	18120044	1561	36.2	6.1	0.5	4.7	1.3	3.7	3.2	2.4	1.4
	14:24	18120045	1542	35.8	6.3	0.6	4.9	1.4	0.0	3.2	2.4	1.5
	14:26	18120046	1534	35.7	6.5	0.6	4.9	1.4	0.0	3.2	2.2	1.5
	14:28	18120047	1548	36.3	6.6	0.6	4.8	1.4	0.0	3.2	2.1	1.5
	14:30	18120048	1551	36.4	6.7	0.6	4.9	1.4	0.0	3.2	2.1	1.5
	15:12	18120068	1581	37.8	3.9	0.5	4.9	1.4	5.4	3.2	2.9	1.4
	15:14	18120069	1592	38.1	4.6	0.6	4.5	1.4	5.0	3.3	3.0	1.4
	15:16	18120070	1600	38.2	5.2	0.6	3.9	1.4	5.3	3.3	2.9	1.4
	15:18	18120071	1596	38.1	5.5	0.6	3.7	1.4	5.5	3.3	2.8	1.4
	15:20	18120072	1583	37.7	5.8	0.6	3.6	1.4	5.6	3.3	2.8	1.4
	15:22	18120073	1406	32.0	6.2	0.5	3.4	1.3	5.9	3.2	2.6	1.3
	15:24	18120074	1285	28.5	6.5	0.5	3.3	1.3	6.2	3.1	1.9	1.3

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formaldehyde	Unc	HCl	Unc	2-Methyl-2-butene	Unc
Process Down	15:26	18120075	1238	27.1	6.7	0.5	3.2	1.3	6.4	3.0	1.4	1.3
	15:29	18120076	1258	27.3	6.7	0.5	3.3	1.3	6.5	3.0	0.0	1.3
	15:31	18120077	1305	27.7	5.9	0.5	3.1	1.2	8.0	2.9	0.0	1.3
Process Down	15:33	18120078	1339	28.5	4.9	0.5	3.2	1.2	9.6	2.9	0.0	1.2
	15:35	18120079	1356	29.0	4.5	0.5	3.3	1.2	10.2	2.9	0.0	1.2
8/12/97	15:54	18120080	1498	34.4	5.6	0.5	3.6	1.3	8.6	3.1	0.0	1.3
	15:56	18120081	1520	35.4	5.5	0.5	3.6	1.3	8.6	3.1	0.0	1.3
	16:03	18120082	1594	39.0	5.8	0.6	3.6	1.4	8.5	3.3	2.1	1.4
	16:05	18120083	1614	39.8	6.0	0.6	3.6	1.4	8.1	3.4	2.3	1.4
	16:07	18120084	1615	39.9	6.1	0.6	3.6	1.4	7.9	3.4	2.4	1.4
	16:09	18120085	1608	39.4	6.3	0.6	3.6	1.4	7.9	3.4	2.3	1.4
	16:11	18120086	1606	39.2	6.4	0.6	3.6	1.4	7.9	3.3	2.2	1.4
	16:14	18120087	1605	39.0	6.1	0.6	3.6	1.4	8.2	3.3	2.2	1.4
	16:17	18120088	1602	39.1	6.1	0.6	3.7	1.4	8.4	3.3	2.2	1.4
	16:19	18120089	1591	38.6	6.0	0.6	3.6	1.4	8.6	3.3	2.2	1.4
	16:21	18120090	1584	38.5	5.9	0.6	3.7	1.4	8.8	3.3	2.1	1.4
	17:56	18120123	1519	38.3	3.5	0.6	4.6	1.5	7.6	3.5	0.0	1.5
	17:58	18120124	1534	38.8	3.9	0.6	4.5	1.5	7.1	3.5	0.0	1.5
	18:00	18120125	1575	40.4	4.2	0.6	3.8	1.5	7.0	3.6	0.0	1.5
	18:02	18120126	1596	41.3	4.2	0.6	3.6	1.5	7.1	3.6	0.0	1.5
	18:04	18120127	1530	38.3	4.6	0.6	3.6	1.5	7.7	3.5	0.0	1.5
	18:06	18120128	1461	35.3	4.7	0.5	3.4	1.4	8.8	3.4	0.0	1.4
	18:09	18120129	1441	34.6	4.4	0.5	3.3	1.4	9.6	3.4	0.0	1.4
	18:11	18120130	1433	34.1	4.2	0.5	3.2	1.4	10.1	3.3	0.0	1.4
	18:13	18120131	1428	33.9	4.1	0.5	3.0	1.4	10.6	3.3	0.0	1.4
	18:15	18120132	1428	33.8	4.1	0.5	2.9	1.4	10.8	3.3	0.0	1.4

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/12/97	18:17	18120133	1428	33.8	4.3	0.5	2.9	1.4	11.0	3.3	0.0	1.4
8/12/97 Average -->			1548	37.1	5.3	0.6	4.1	1.4	6.2	3.3	1.6	1.4

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/13/97	10:34	OS0813A	1173	21.8	0.0	0.4	4.4	1.0	0.0	2.4	0.0	1.1
	10:38	OS0813B	1311	25.6	0.0	0.5	4.8	1.1	0.0	2.6	0.0	1.2
	10:44	OS0813C	1329	26.1	0.0	0.5	4.7	1.1	0.0	2.7	0.0	1.2
	10:49	OS0813D	1327	26.2	0.0	0.5	4.7	1.1	0.0	2.7	0.0	1.2
	11:02	OU0813A	1544	35.6	0.0	0.5	5.0	1.3	0.0	3.1	2.0	1.4
	11:07	OU0813B	1544	35.6	0.0	0.5	5.1	1.3	0.0	3.1	2.1	1.4
	11:12	OU0813C	1560	36.5	0.0	0.5	5.1	1.3	0.0	3.1	2.2	1.4
	11:18	OU0813D	1578	36.8	0.0	0.6	5.0	1.3	0.0	3.1	2.3	1.4
	11:55	18130016	1574	36.4	1.3	0.5	5.3	1.3	0.0	3.1	2.5	1.4
	11:58	18130017	1566	35.9	1.2	0.5	5.2	1.3	0.0	3.1	2.4	1.4
	12:01	18130018	1559	35.6	1.1	0.5	4.4	1.3	0.0	3.1	2.1	1.4
	12:03	18130019	1561	35.8	1.0	0.5	4.2	1.3	0.0	3.1	2.1	1.4
	12:05	18130020	1575	36.3	1.1	0.5	4.2	1.3	0.0	3.2	2.1	1.4
	12:07	18130021	1585	36.8	1.4	0.5	4.2	1.3	0.0	3.2	2.1	1.4
	12:09	18130022	1590	36.9	1.7	0.5	4.3	1.3	0.0	3.2	2.2	1.4
	12:11	18130023	1592	37.2	1.9	0.6	4.3	1.3	0.0	3.2	2.1	1.4
	12:13	18130024	1583	36.8	1.6	0.5	4.4	1.3	0.0	3.2	2.0	1.4
	12:16	18130025	1571	36.3	1.2	0.5	4.4	1.3	0.0	3.1	1.9	1.4
	12:18	18130026	1564	36.0	0.9	0.5	4.4	1.3	0.0	3.1	1.9	1.4
	12:20	18130027	1556	35.6	0.8	0.5	4.4	1.3	0.0	3.1	1.9	1.4
	12:22	18130028	1542	35.1	0.6	0.5	4.4	1.3	0.0	3.1	1.8	1.4
	12:24	18130029	1535	34.7	0.0	0.5	4.2	1.3	0.0	3.1	1.8	1.4
	12:26	18130030	1556	35.7	0.7	0.5	4.3	1.3	0.0	3.2	2.1	1.4
	12:28	18130031	1569	36.2	1.1	0.6	4.3	1.3	0.0	3.2	2.1	1.4
	12:30	18130032	1564	35.9	1.5	0.5	4.3	1.3	0.0	3.1	1.9	1.4
	12:33	18130033	1548	35.3	1.4	0.5	4.3	1.3	0.0	3.2	2.0	1.4

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/13/97	12:35	18130034	1545	35.2	1.3	0.5	4.3	1.3	0.0	3.1	2.0	1.4
	12:37	18130035	1542	35.1	1.3	0.5	4.3	1.3	0.0	3.1	1.8	1.4
	12:39	18130036	1537	34.8	1.4	0.5	4.2	1.3	0.0	3.1	1.8	1.4
	12:41	18130037	1536	34.8	1.4	0.5	4.2	1.3	0.0	3.1	1.8	1.4
	13:05	18130048	1566	36.5	0.0	0.5	5.1	1.3	0.0	3.2	2.1	1.4
	13:07	18130049	1568	36.4	0.0	0.6	4.3	1.3	0.0	3.2	2.0	1.4
	13:09	18130050	1571	36.4	0.0	0.6	4.1	1.3	0.0	3.2	2.0	1.4
	13:11	18130051	1562	36.1	0.0	0.6	3.9	1.3	0.0	3.2	1.9	1.4
	13:13	18130052	1564	35.9	0.6	0.5	3.9	1.3	0.0	3.2	1.9	1.4
	13:15	18130053	1579	36.4	0.7	0.5	3.9	1.3	0.0	3.2	1.9	1.4
	13:17	18130054	1573	36.2	0.0	0.6	3.9	1.3	0.0	3.2	1.9	1.4
	13:20	18130055	1568	36.0	0.0	0.6	3.9	1.3	0.0	3.1	1.9	1.4
	13:22	18130056	1565	35.9	0.0	0.6	3.9	1.3	0.0	3.1	1.8	1.4
	13:24	18130057	1571	36.0	0.0	0.5	3.9	1.3	0.0	3.1	1.9	1.4
	13:26	18130058	1563	35.8	0.7	0.5	3.9	1.3	0.0	3.1	1.9	1.4
	13:28	18130059	1557	35.6	0.9	0.5	4.0	1.3	0.0	3.1	1.9	1.4
	13:30	18130060	1559	35.8	1.0	0.5	3.9	1.3	0.0	3.1	1.9	1.4
	13:32	18130061	1559	35.8	1.0	0.5	3.8	1.3	0.0	3.2	1.9	1.4
	13:34	18130062	1556	35.8	1.2	0.5	3.9	1.3	0.0	3.2	1.9	1.4
	13:37	18130063	1550	35.6	1.4	0.6	3.8	1.3	0.0	3.2	1.9	1.4
	13:39	18130064	1549	35.6	1.6	0.6	3.7	1.4	0.0	3.2	1.7	1.4
	13:45	18130065	1531	34.8	1.8	0.5	3.4	1.3	0.0	3.2	2.5	1.3
	13:47	18130066	1522	34.5	1.7	0.5	3.3	1.3	0.0	3.2	2.3	1.3
	13:49	18130067	1527	34.7	1.4	0.5	3.4	1.3	0.0	3.1	2.4	1.3
	13:52	18130068	1526	34.7	1.3	0.5	3.4	1.3	0.0	3.1	2.4	1.3
	15:00	18130069	1510	34.1	0.8	0.5	3.0	1.3	0.0	3.2	1.7	1.3

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

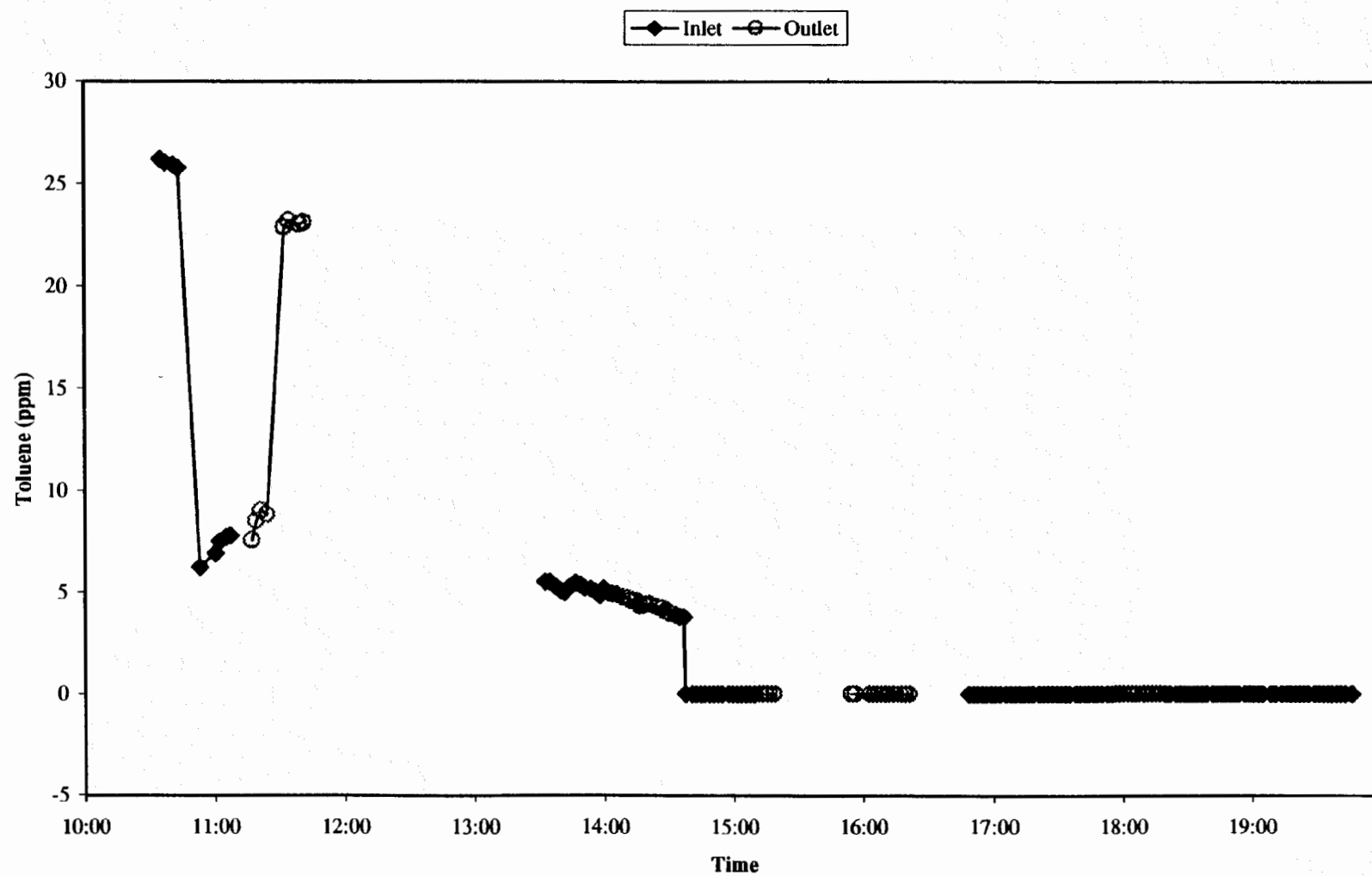
Date	Time	File Name ^{a,b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/13/97	15:02	18130070	1508	34.0	0.6	0.5	2.9	1.3	0.0	3.2	1.6	1.3
	15:04	18130071	1506	34.0	0.0	0.5	3.0	1.3	0.0	3.2	1.5	1.3
	15:06	18130072	1514	34.4	0.0	0.5	3.1	1.3	0.0	3.1	1.5	1.3
	15:08	18130073	1506	34.0	0.0	0.5	3.1	1.3	0.0	3.1	1.4	1.3
	15:10	18130074	1507	33.9	0.0	0.5	3.0	1.3	0.0	3.1	0.0	1.3
	15:13	18130075	1517	34.2	0.0	0.5	3.0	1.3	0.0	3.1	0.0	1.3
	15:15	18130076	1530	34.8	0.0	0.5	3.1	1.3	0.0	3.1	1.4	1.3
	15:17	18130077	1537	35.1	0.0	0.6	2.9	1.3	0.0	3.2	1.4	1.3
	15:19	18130078	1547	35.3	0.0	0.5	2.9	1.3	0.0	3.2	1.5	1.3
	15:21	18130079	1542	35.0	0.0	0.5	3.1	1.3	0.0	3.2	1.6	1.3
	15:23	18130080	1528	34.7	0.0	0.5	3.0	1.3	0.0	3.1	1.5	1.3
	15:25	18130081	1517	34.3	0.0	0.5	3.0	1.3	0.0	3.1	1.4	1.3
	15:28	18130082	1521	34.3	0.0	0.5	2.9	1.3	0.0	3.1	1.4	1.3
	15:30	18130083	1521	34.3	0.0	0.5	2.9	1.3	0.0	3.1	1.4	1.3
	15:32	18130084	1517	34.3	0.0	0.5	3.0	1.3	0.0	3.1	1.4	1.3
	15:34	18130085	1519	34.6	0.0	0.5	2.9	1.3	0.0	3.2	1.4	1.3
	15:36	18130086	1525	35.0	0.0	0.6	2.9	1.3	0.0	3.2	1.5	1.3
	15:38	18130087	1531	35.1	0.0	0.6	2.8	1.3	0.0	3.2	1.4	1.3
	15:40	18130088	1541	35.4	0.0	0.5	2.9	1.3	0.0	3.2	1.5	1.3
	15:42	18130089	1547	35.6	0.6	0.5	2.9	1.3	0.0	3.2	1.5	1.3
	15:45	18130090	1552	35.5	0.6	0.5	2.8	1.3	0.0	3.2	1.4	1.3
	15:47	18130091	1551	35.1	0.0	0.5	2.6	1.3	0.0	3.1	1.4	1.3
	15:49	18130092	1547	34.8	0.0	0.5	2.4	1.3	0.0	3.1	1.4	1.3
	15:51	18130093	1536	34.2	0.0	0.5	2.2	1.3	0.0	3.1	0.0	1.4
8/13/97 Average -->			1548	35.4	0.6	0.5	3.7	1.3	0.0	3.1	1.7	1.4

TABLE B-2. Continued. (CO, Ammonia, Formaldehyde, HCl and 2-Methyl-2-butene Outlet Results)

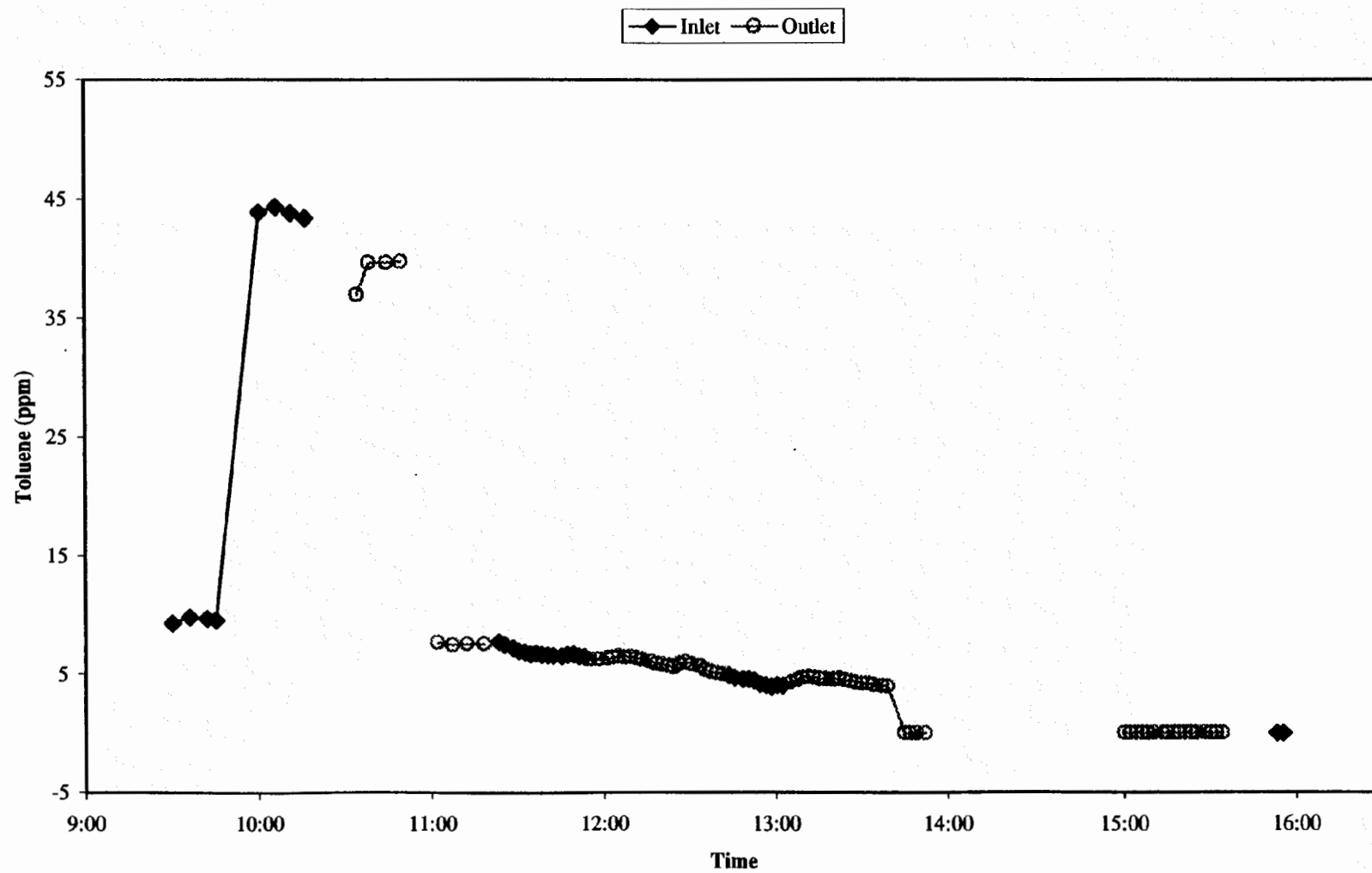
Date	Time	File Name ^{a, b}	CO	Unc ^c	Ammonia	Unc	Formal- dehyde	Unc	HCl	Unc	2-Methyl- 2-butene	Unc
8/14/97	9:01	18140012	1461	31.3	2.6	0.5	5.8	1.1	3.4	2.7	0.0	1.2
	9:04	18140013	1465	31.4	2.8	0.5	5.6	1.2	3.1	2.7	0.0	1.2
	9:06	18140014	1475	31.7	3.2	0.5	5.3	1.2	2.8	2.8	0.0	1.3
	9:08	18140015	1494	32.6	3.4	0.5	5.4	1.2	0.0	2.8	0.0	1.3
	9:10	18140016	1507	33.1	3.5	0.5	5.3	1.2	0.0	2.8	0.0	1.3
	9:12	18140017	1414	29.2	3.7	0.5	5.5	1.1	0.0	2.7	1.7	1.2
	9:14	18140018	1342	26.5	3.7	0.4	5.4	1.1	0.0	2.5	0.0	1.1
	9:16	18140019	1309	25.4	3.5	0.4	5.2	1.0	2.5	2.4	0.0	1.1
	9:18	18140020	1286	24.8	3.2	0.4	4.9	1.0	2.8	2.4	0.0	1.1
	9:21	18140021	1282	24.7	2.9	0.4	4.8	1.0	2.8	2.4	0.0	1.1
Spike Off	9:23	18140022	1284	24.7	2.8	0.4	4.7	1.0	2.9	2.4	0.0	1.1
	9:25	18140023	1317	25.7	2.6	0.4	4.7	1.0	2.9	2.4	0.0	1.1
	9:27	18140024	1455	30.9	2.2	0.5	4.9	1.1	0.0	2.7	0.0	1.3
	9:29	18140025	1498	33.0	1.9	0.5	4.93	1.2	0.0	2.8	0.0	1.3
	9:31	18140026	1515	33.8	1.9	0.5	4.93	1.2	0.0	2.9	0.0	1.3
	9:33	18140027	1523	34.2	2.0	0.5	4.88	1.2	0.0	2.9	0.0	1.4
8/14/97 Average --->			1490	32.6	2.5	0.5	5.3	1.2	1.3	2.8	0.0	1.3

^a Bold face type indicates samples that were spiked with toluene or SF₆.^b Shaded rows indicate times when the process was down.^c Unc is the estimated uncertainty in each measurement. Typically the uncertainty is greater for lower concentrations.

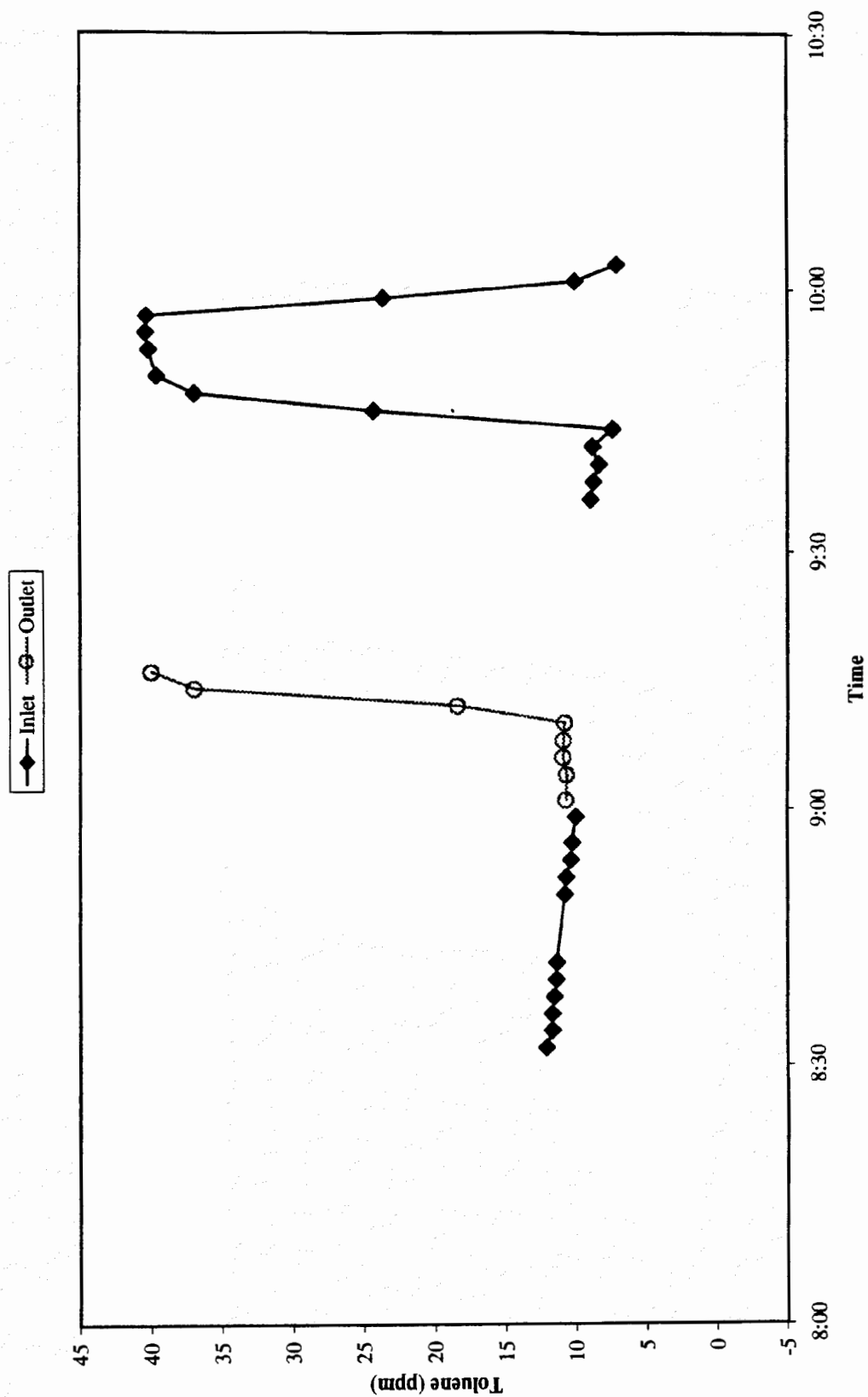
Toluene Concentrations at WCI Inlet and Outlet (8/12/97)



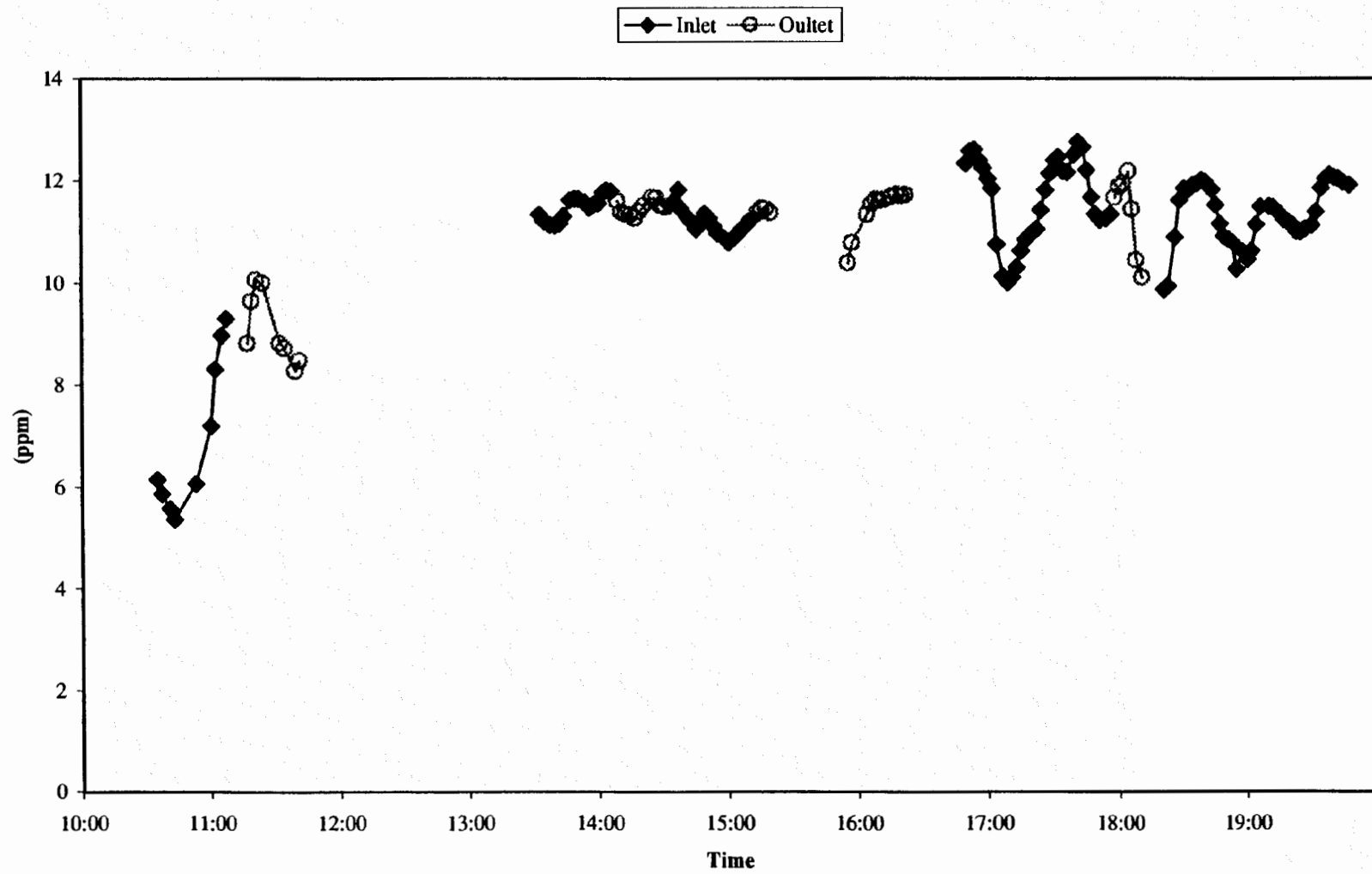
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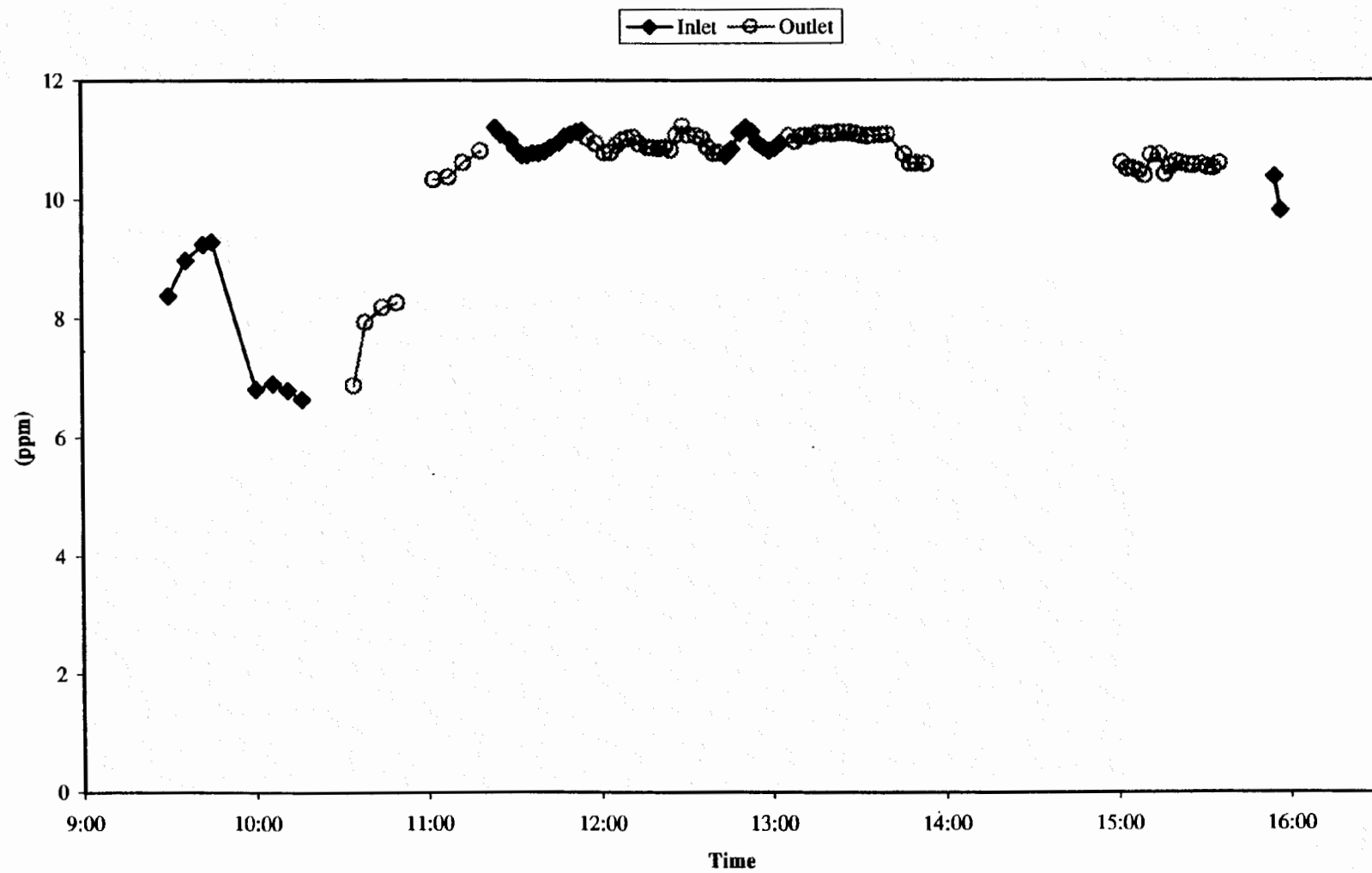
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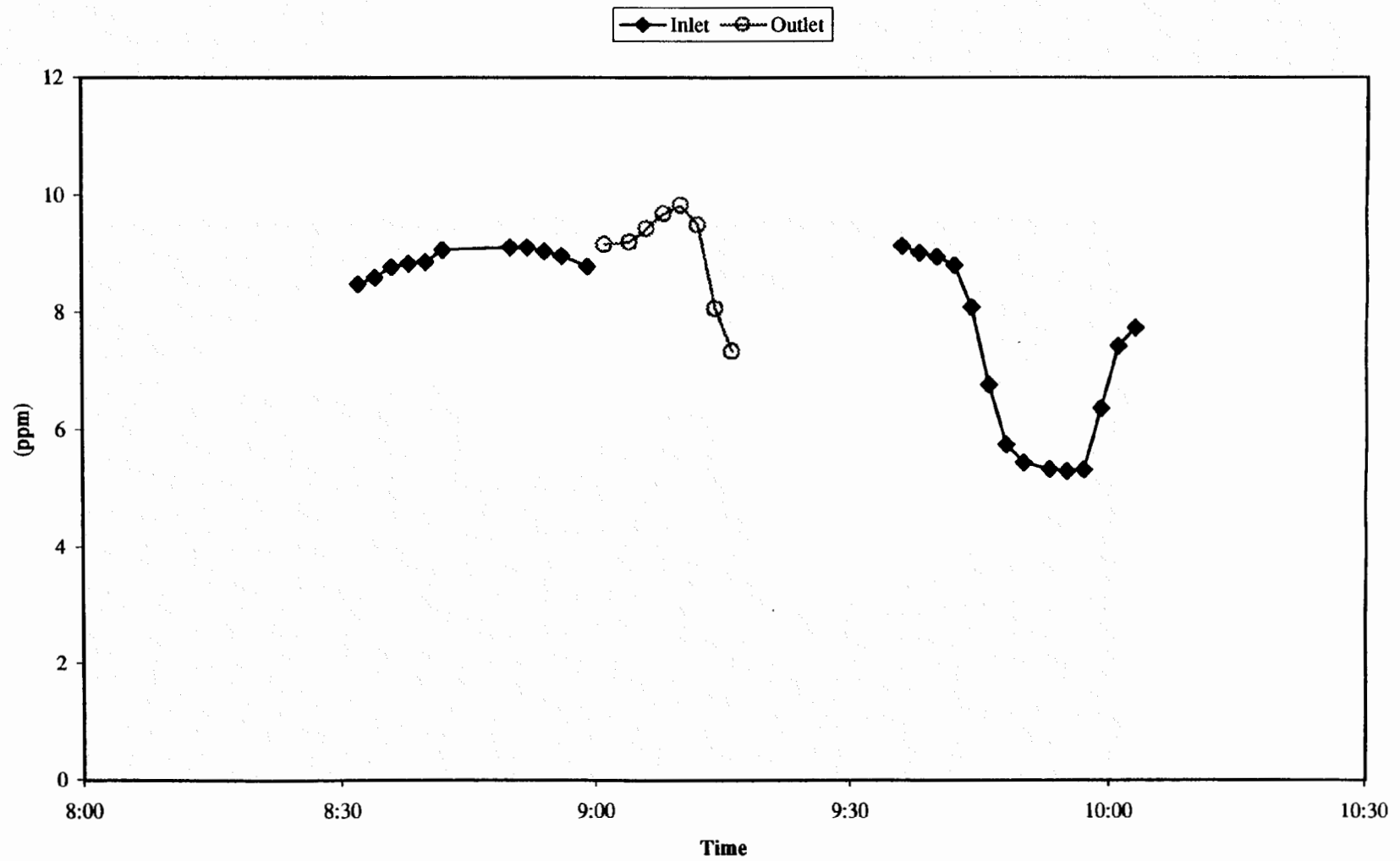
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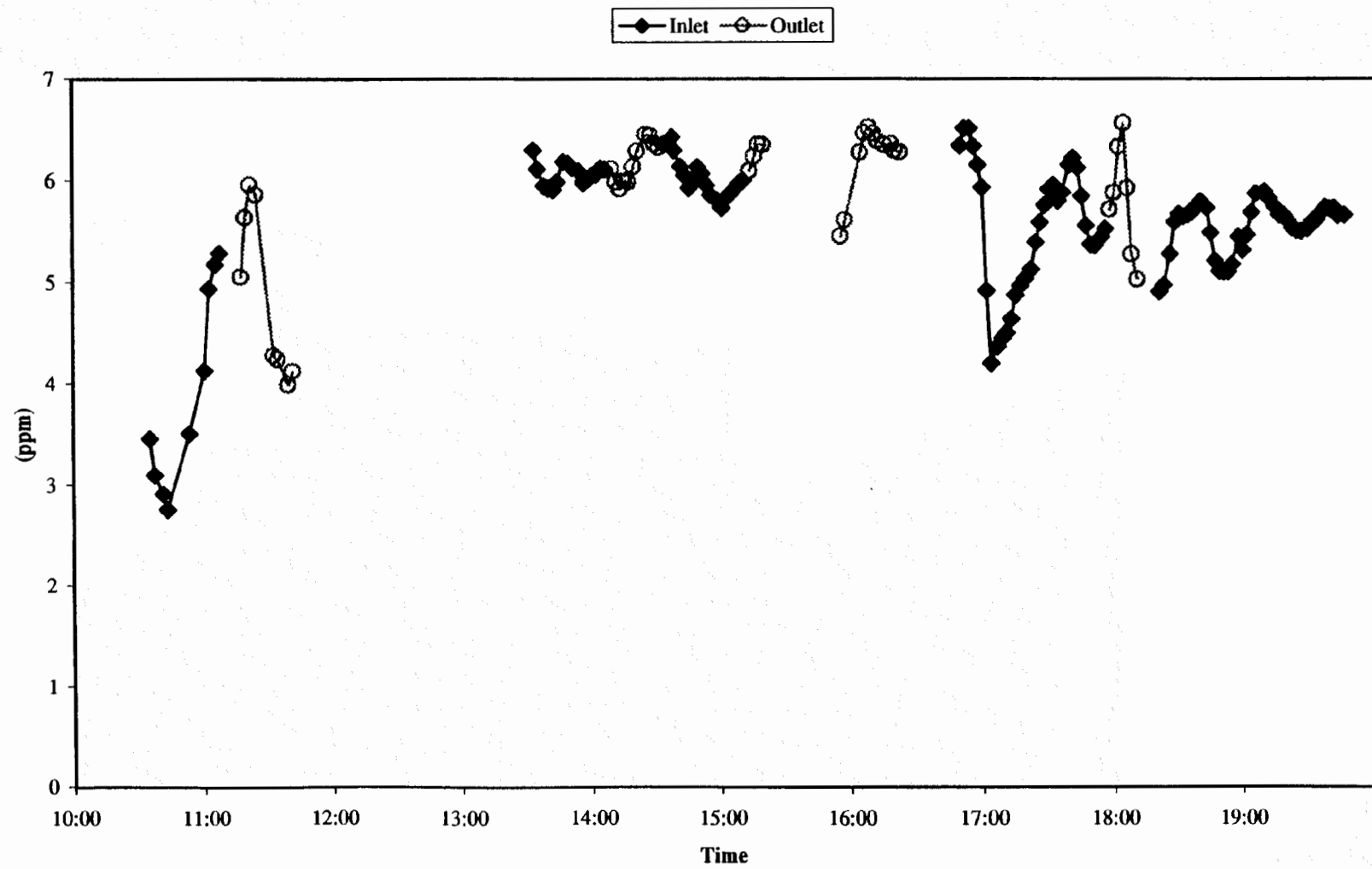
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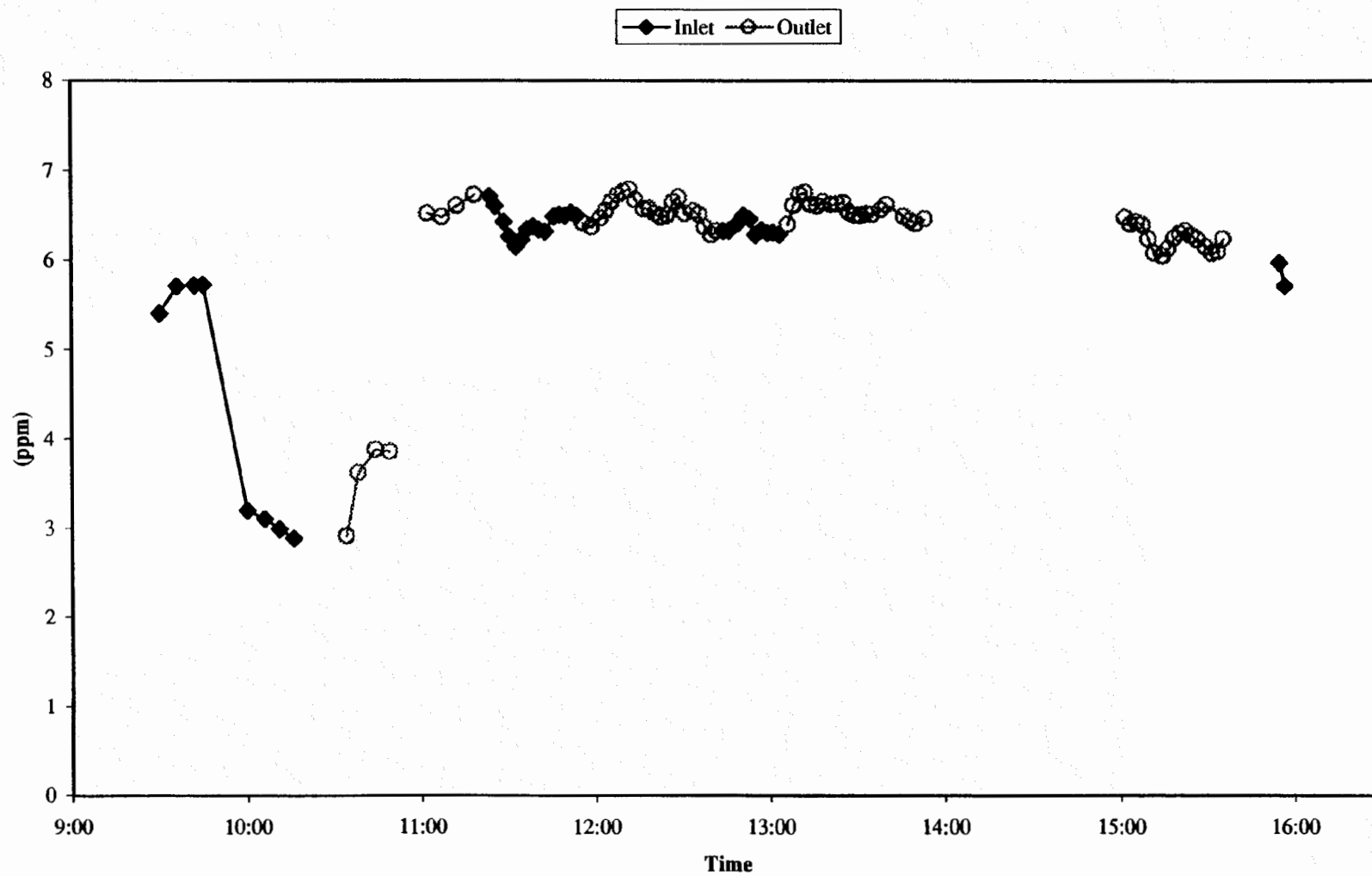
Hexane Concentrations at WCI (8/14/97)



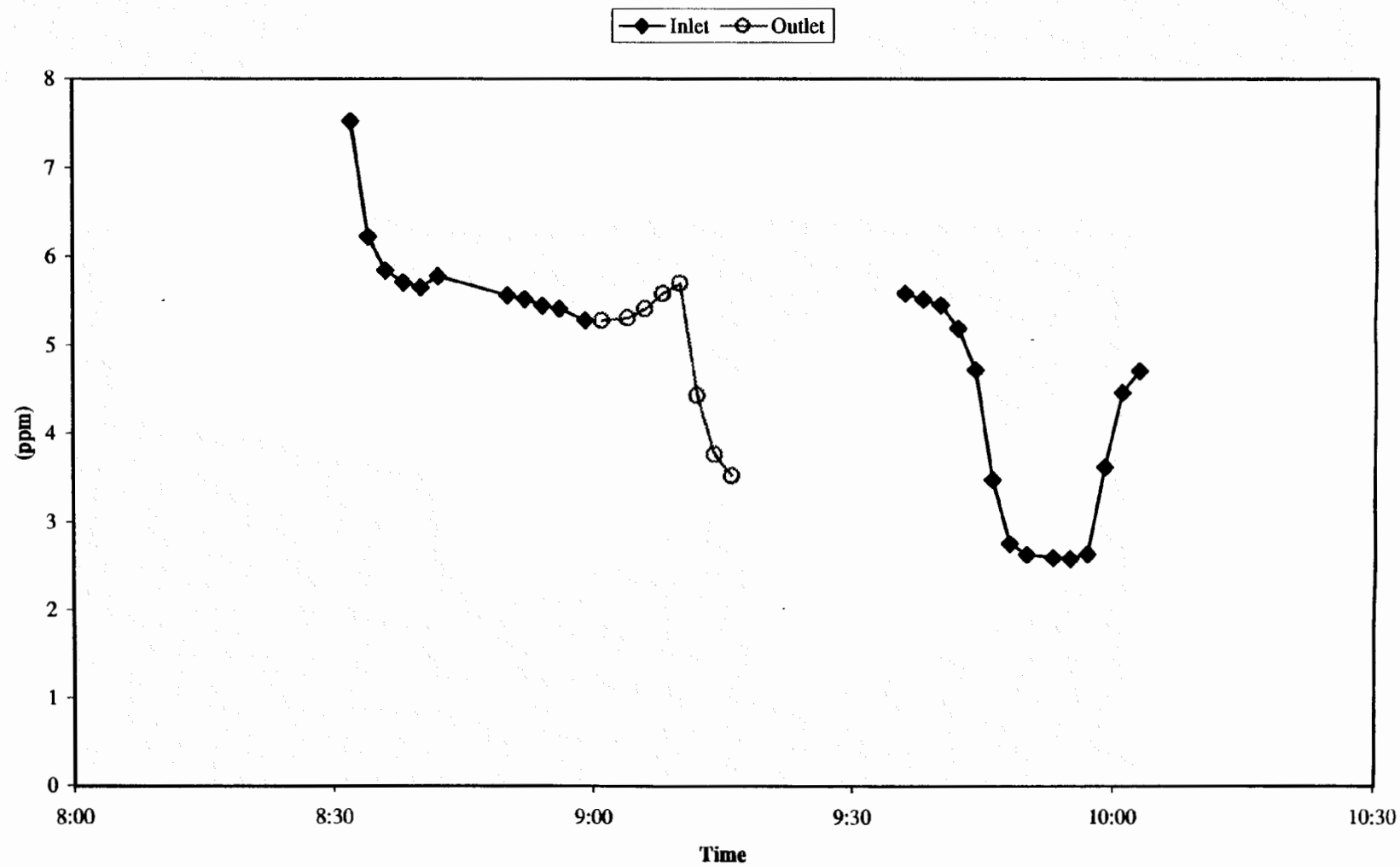
Ethylene Concentrations at WCI (8/12/97)



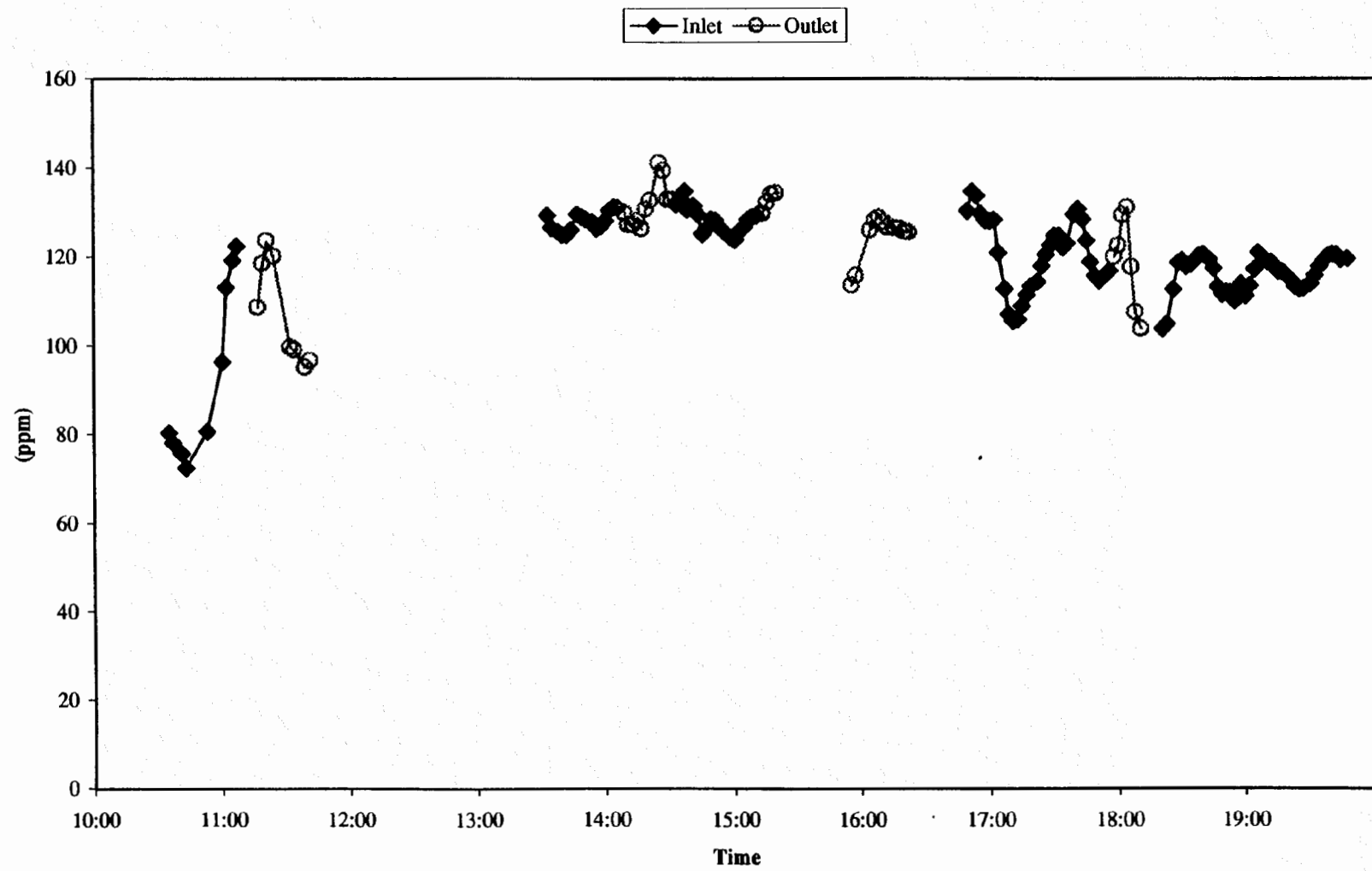
Ethylene Concentrations at WCI (8/13/97)



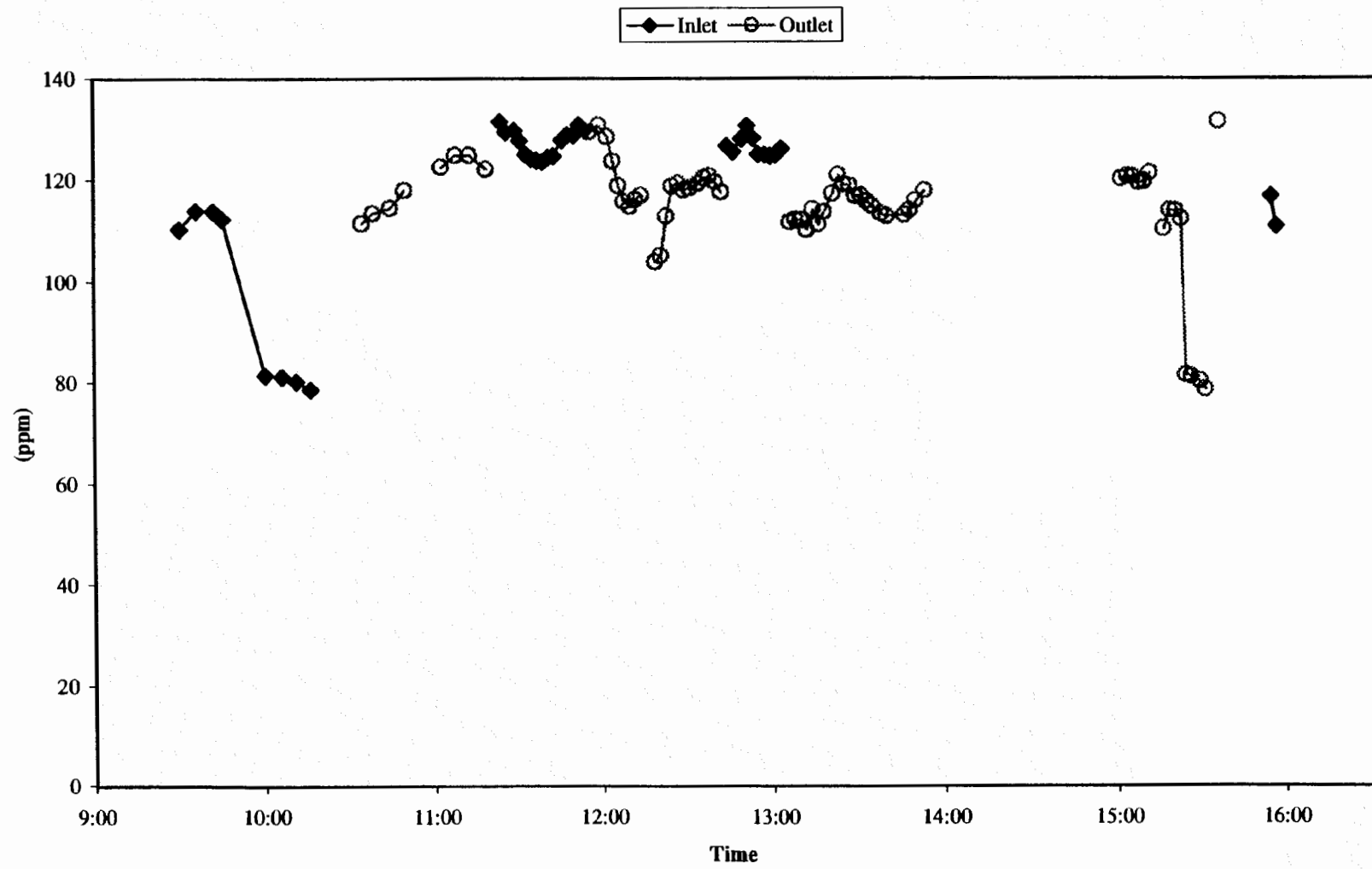
Ethylene Concentrations at WCI Inlet (8/14/97)



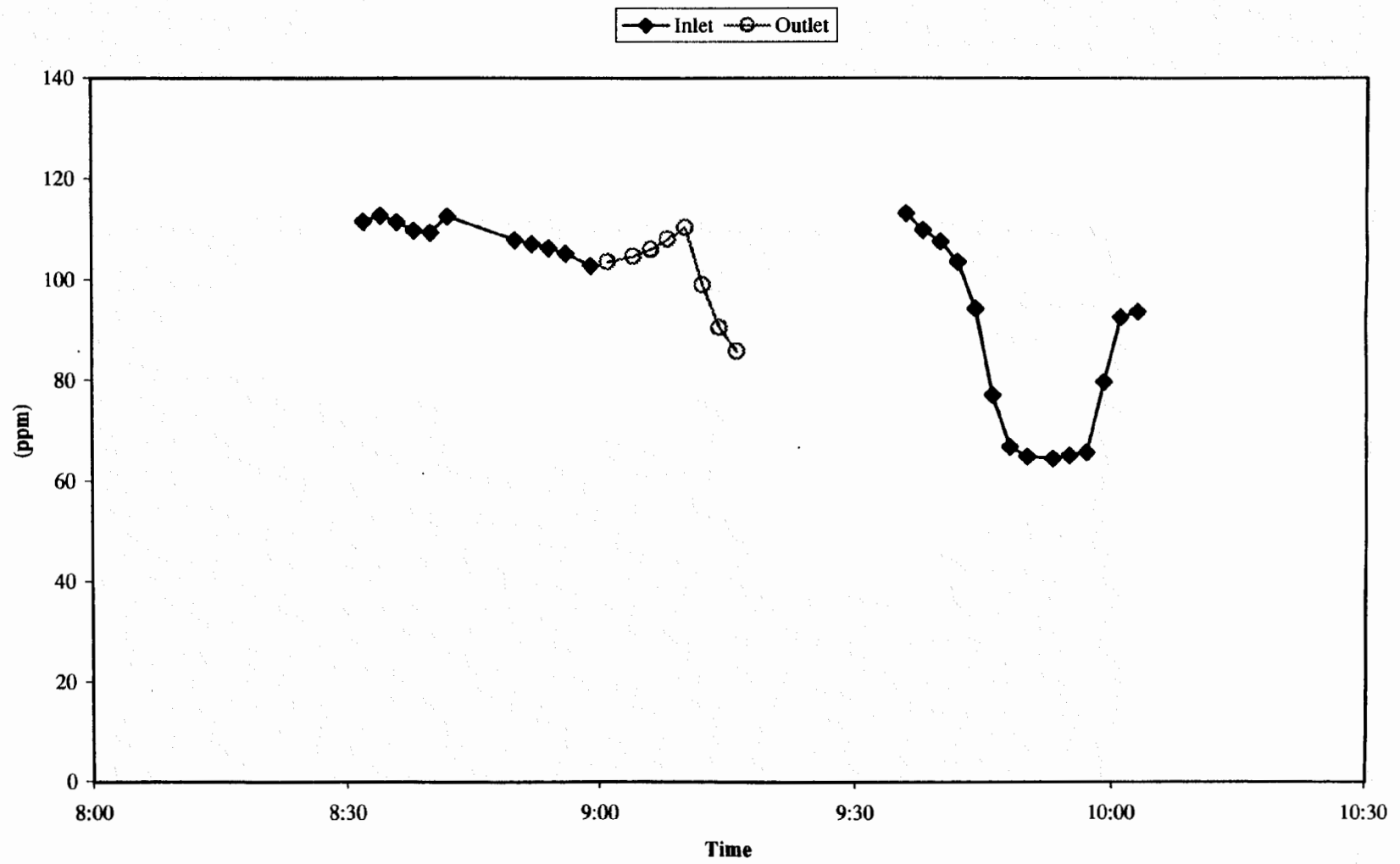
Methane Concentrations at WCI (8/12/97)



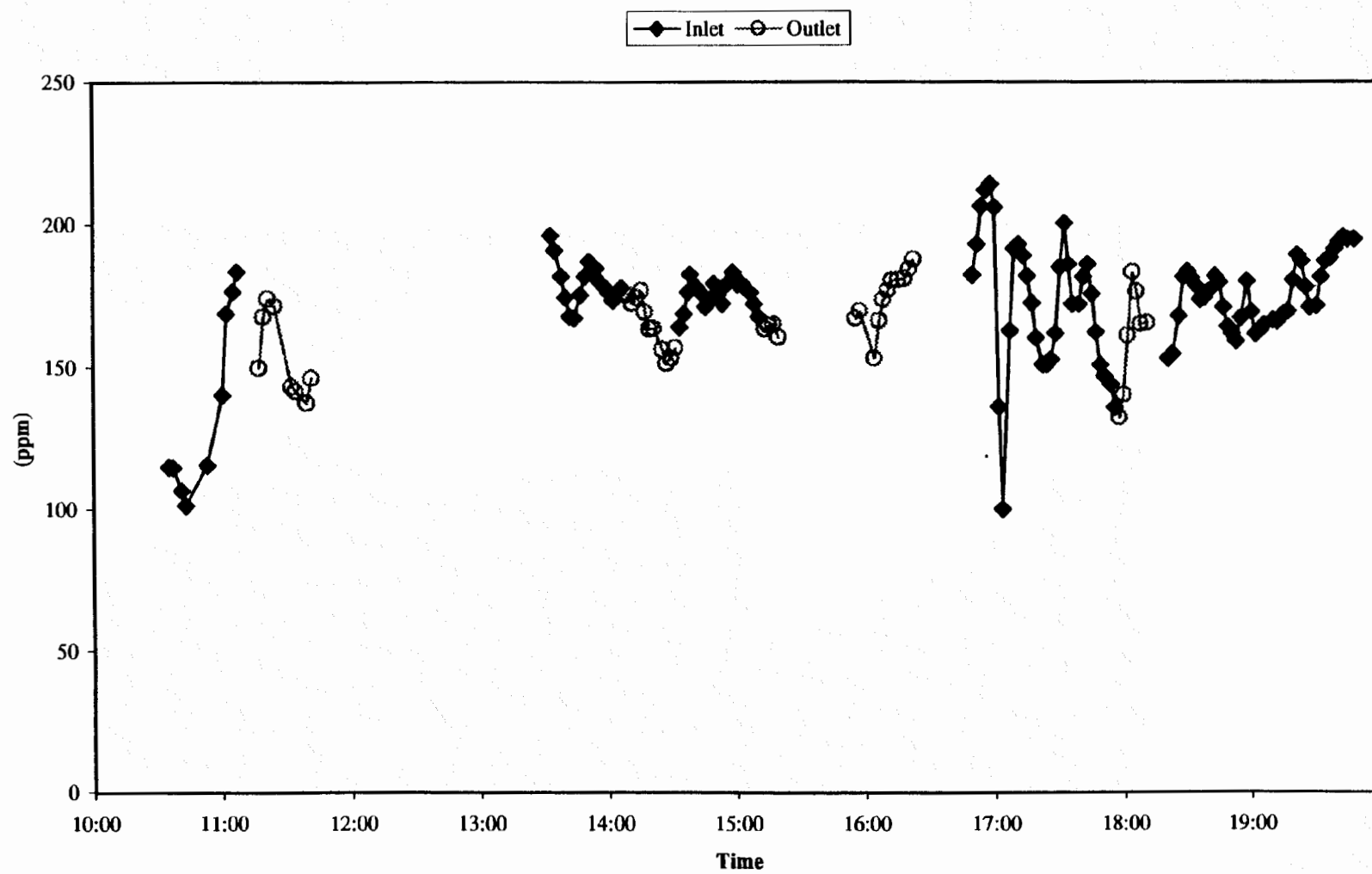
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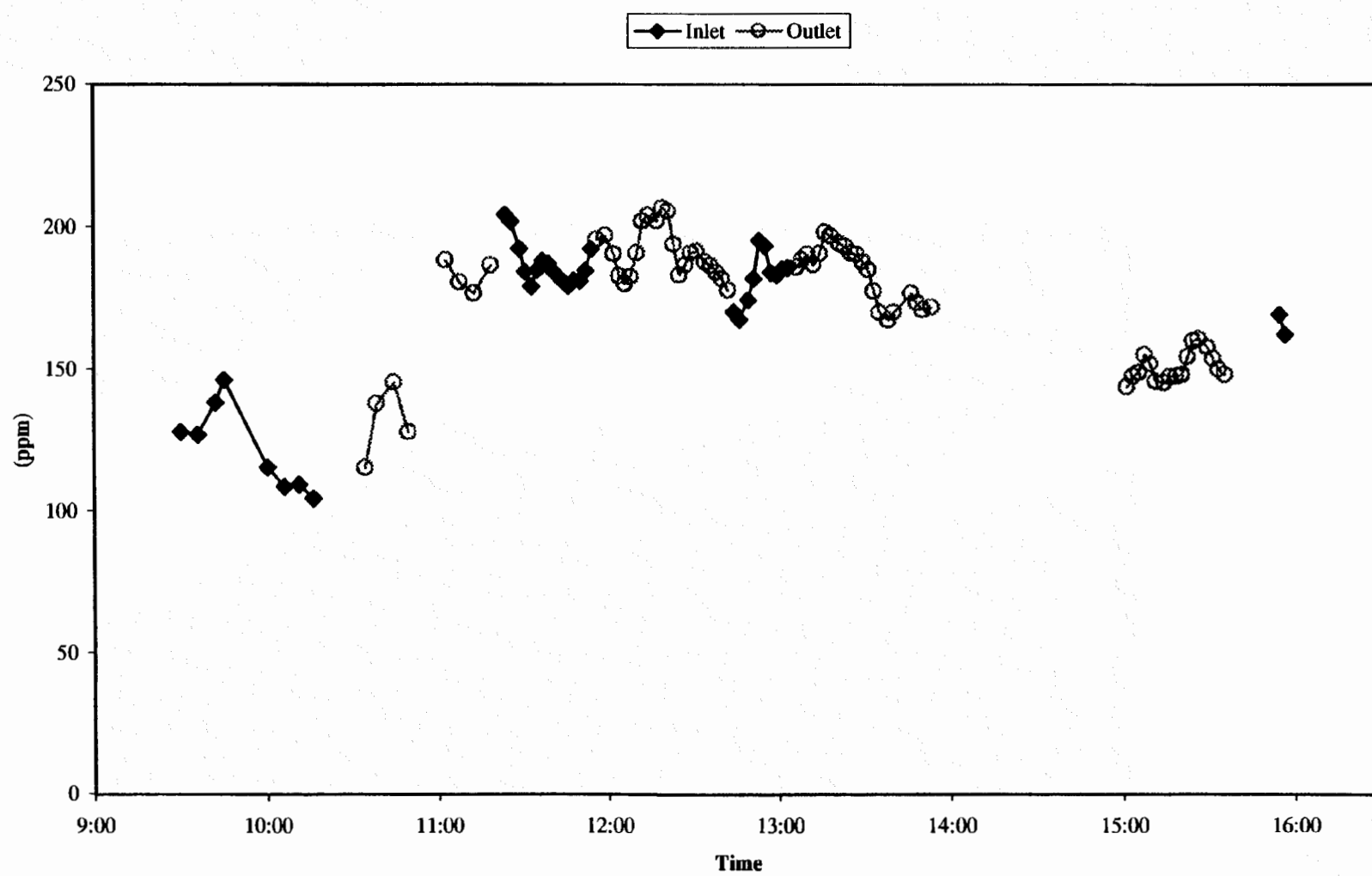
Methane Concentrations at WCI (8/14/97)



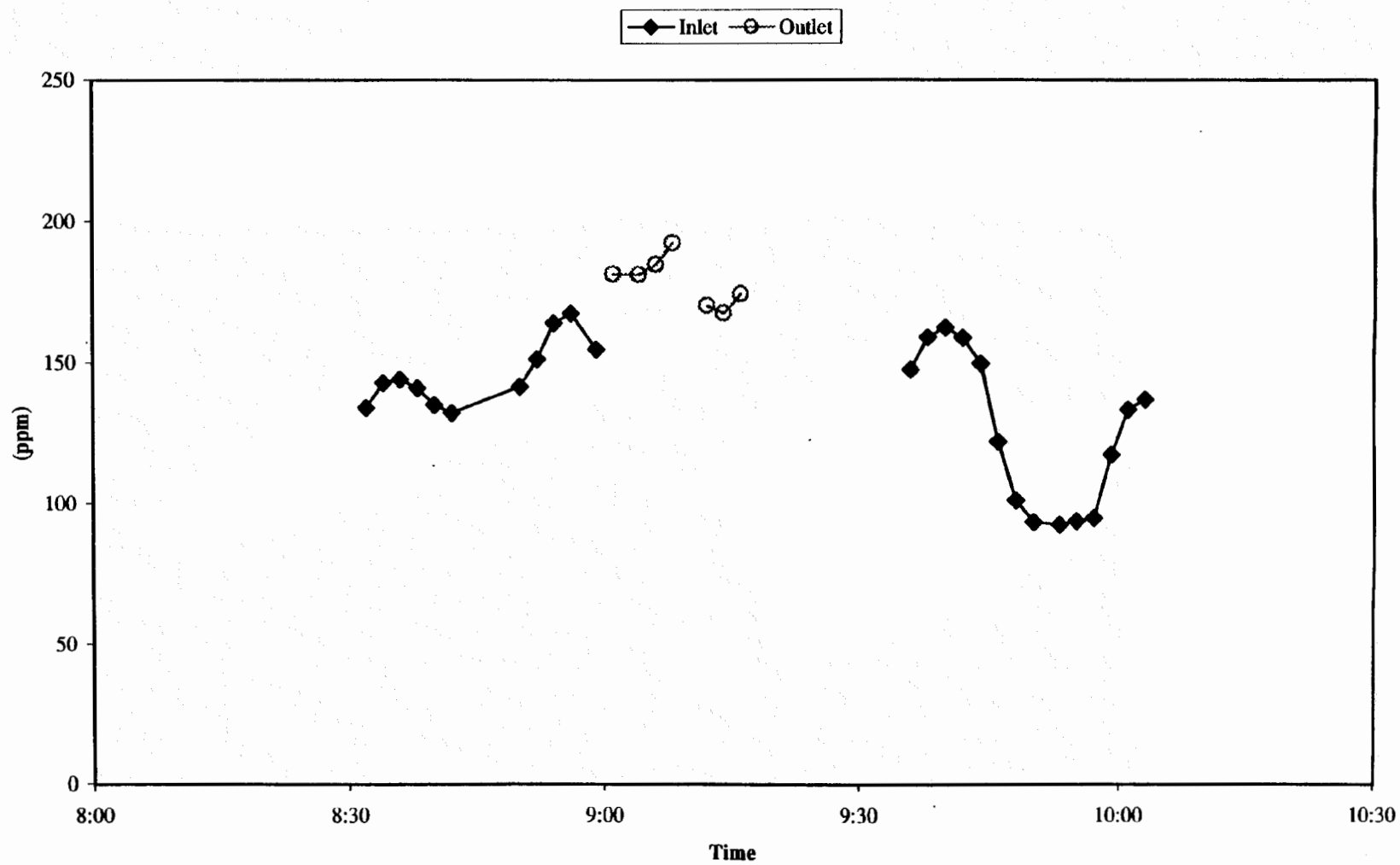
SO₂ Concentrations at WCI (8/12/97)



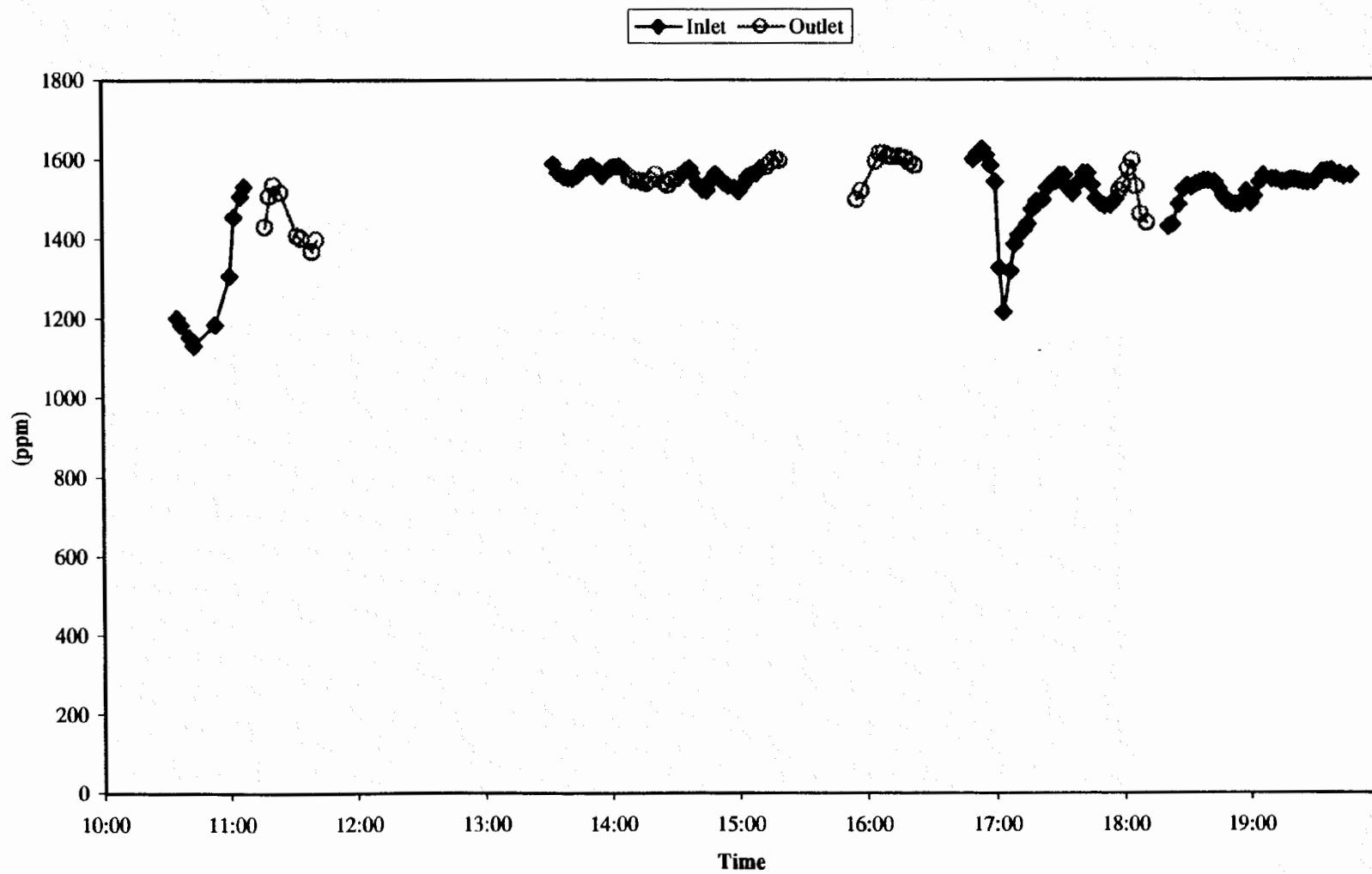
SO₂ Concentrations at WCI (8/13/97)



SO₂ Concentrations at WCI (8/14/97)



CO Concentrations at WCI (8/12/97)



B-2 FTIR FIELD DATA RECORDS

Data Sheet: FTIR Batch Samples: WCI. EPA Work Assignment 4-20.

Date	Sample time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond	Sample Flow	BKG
	10:00	is0813a		First spiked inlet sample				S			
	10:05	is0813b		Second spiked inlet sample				S			
	10:10	is0813c		Third spiked inlet sample				S			
	10:15	is0813d		Fourth spiked inlet sample				S			
		msu0813d		Spiked minus unspiked; tol larger than yesterday (OK!)							
	10:29			Start tol (121ppm) flow 2.00 lpm, SF6 (4.0ppm) 1.0 lpm to outlet spike, total flow ~14.5lpm							
	10:34	os0813a		First spiked outlet sample							
	10:39	os0813b		Second spiked outlet sample							
	10:44	os0813c		Third spiked outlet sample							
	10:49	os0813d		Fourth spiked outlet sample							
	10:51			Spike gases off, valve closed							
	10:55			Run 2 starts at outlet							
	11:02	ou0813a		First unspiked outlet sample							
	11:07	ou0813b		Second unspiked outlet sample							
	11:12	ou0813c		Third unspiked outlet sample							
	11:17	ou0813d		Fourth unspiked outlet sample							
	11:22			Switch to continuous mode at inlet							
	11:24	180130001		First of Run 2 continuous mode spectra	250	2.0	130C	U	H/W	3.3lpm	A(500)
	11:55			Inlet port switch starts							
	11:58	18130016		Switch to continuous mode at outlet							
	12:15	~18130025		Inlet port switch ends							
	12:45	181300038		Switch to continuous mode at inlet							
	13:05	~18130047		Outlet port switch starts							
	13:06			Switch to continuous mode at outlet						4.0lpm	
	13:54	18130068		Stop analysis, Waiting for repairs to outlet dioxin train and test restart							
	14:57	18130069		Start continuous mode at outlet							
	15:30	18130079		Re-start manual testing at outlet							
	15:56	~18130094		Start continuous mode at inlet						3.0lpm	
	16:23	18130106		Manual testing stopped because of process disruption							
	16:24	18130106		Start continuous mode at outlet (process down)							
	17:00			Manual testing restarted							
	17:15			Inlet port switch starts							
	17:37			FTIR run ends (manual testing to continue)							
	17:40			Start CTS flow to cell 7.5 lpm P=745							
	17:45			Data since 16:28 lost - detector warmed up - 15 minutes of manual test comparison lost.							
	17:49	CTS0813C		20 ppm ETY							
	17:51	CTS0813D		20 ppm ETY							
	17:54			Start N2 flow to cell							
8/14/97	7:26			Initial CTS (a,b) with old bkg no good; NH3 seems to be gone							
	8:21	bkg0814a			500	2.0	130C				A
	8:22	cts0814c		P=747.1							
	8:26	cts0814d									
	8:28			Start inlet flow to cell						3.5lpm	
	8:30			Start of FTIR Run 3							
	8:45	18140006		Stop for data check							
	8:48			Data check OK							
	8:50	18140007		Restart FTIR testing							
	9:03	~18140012		Switch to continuous mode at outlet						4.5lpm	

Data Sheet: FTIR Batch Samples: WCI. EPA Work Assignment 4-20.

Date	Sample time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
	9:10	~18140016		Start spike flow to outlet 1.0lpm SF6 (4.0 ppm) + 2.00lpm TOL (121ppm); total flow ~10.5lpm							
	9:25	~18140022		Spike flows off							
	9:40	~18140028		Switch to continuous mode at inlet						3.5lpm	
	9:43	~18140030		Start spike flow to inlet 1.0lpm SF6 (4.0 ppm) + 2.00lpm TOL (121ppm); total flow ~9.0lpm							
	9:58	~18140037		Spike flows off							
	10:13	~18140045		Stop for data check							
	10:21	cts0814e									

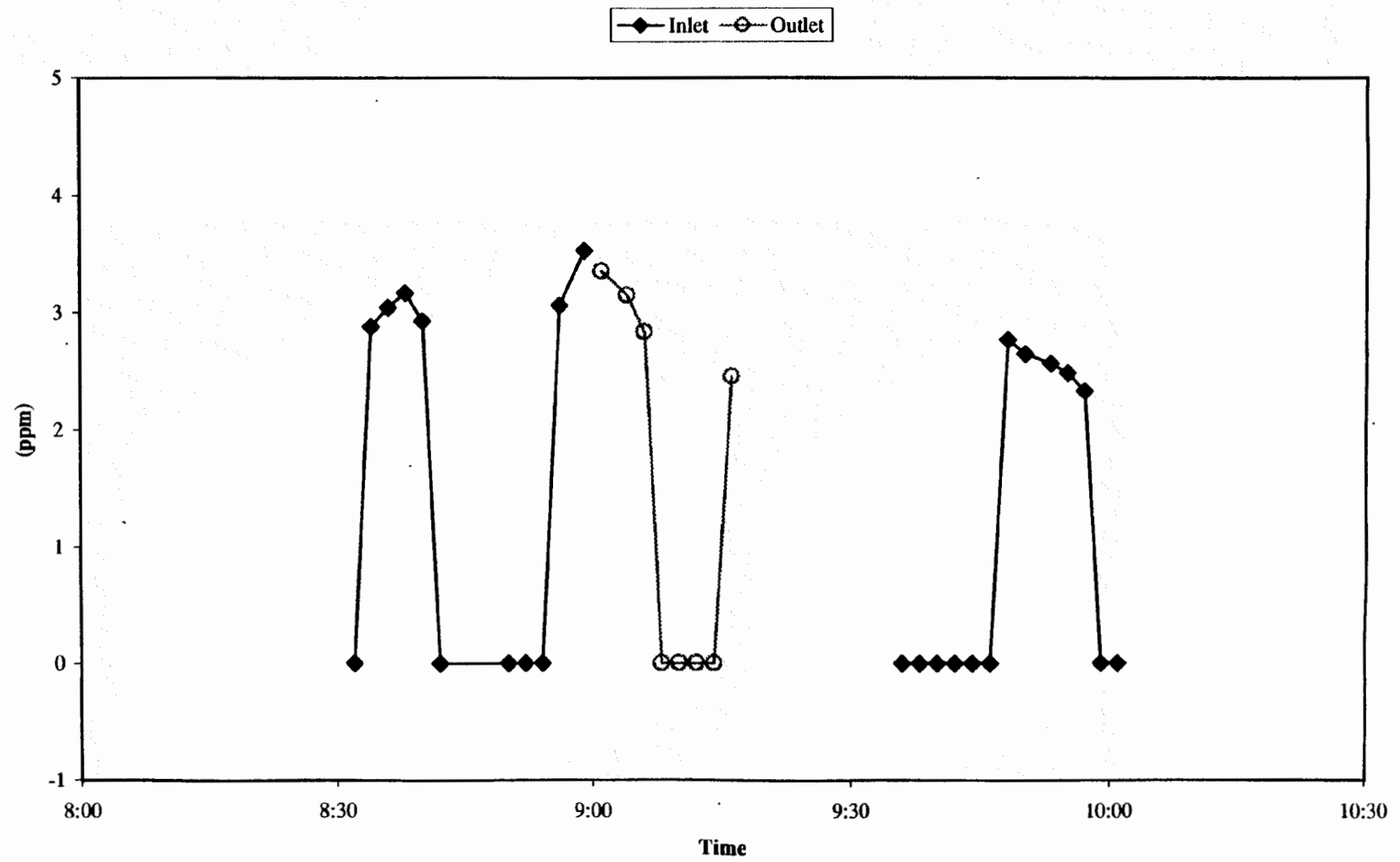
Data Sheet: FTIR Batch Samples WCI. EPA Work Assignment 4-20.

Date	Sample time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
8/11/97	8:00			Truck location							
	~9:30			Safety briefing							
	11:30			Computer failure, Syquest mistakenly plugged into MIDAC card.							
	12:30			Discover MIDAC card and cable destroyed.							
	~14:30			System running w/o Syquest (removed MIDAC card)							
	~15:00	bkgatcd.asf		BKG nearly identical to KC 8/6/97 "A," RMS 2500-2600 ~.0001AU for two 250 scan ratio							
	~17:00			System running w/ Syquest (new M/F 25 pin cable)							
	until 21:00			Inlet/outlet setup; final inlet probe set-up and samp sys leak checks remain; LH has re-plumbed and tested spiking system.							
8/12/97	5:45			Spreadsheet prep							
	9:02	CTS0811A	E 20 passes	20 ppm ETY 7.5lpm 5 min; P=748.5 N2 BKG0811e 500 scans	250	2.0	130C				E
	9:18			CTSA,B show tellon outgasses and features in CH region							
				Switch to N2 7.5 lpm P=748.9 for new BKG							
	9:21	BKG0811F			500						
	9:25			Switch to 20ppm ETY 6.0 lpm P=749.3							
	9:27			Inlet and outlet systems pass leak checks							
	9:28	CTS0811C			250						F
	9:31	CTS0811D			250						
	9:33			CTS comparison and quality good; switch to N2							
	10:16			Start SF6 4.0 ppm direct flow 5lpm							
	10:21	SF60811a		SF6 direct 4.0ppm 5.5lpm P=748.9, recorded as inlet spec	250						
	10:24			SF6 absorbance ~0.67AU; switch to N2							
	10:29			Start tol (121ppm) flow 1.00 lpm, SF6 (4.0ppm) 1.0 lpm to inlet spike, total flow ~10lpm							
	10:35	is0811a		Start first spiked inlet stack spectrum 4lpm	250						
	10:38	is0811b		Start second spiked inlet stack spectrum							
	10:40	is0811c		Start third spiked inlet stack spectrum							
	10:42	is0811d		Start fourth spiked inlet stack spectrum							
	10:45			Spike gases off							
	10:52	iu0811a		First unspiked inlet stack spectrum 4 lpm;							
	10:54			Can see SF6 and tol in calc'd spectrum smu0811a							
	11:00	iu0811b		Second unspiked inlet stack spectrum							
	11:02	iu0811c		Third unspiked inlet stack spectrum							
	11:04	iu0811d		Fourth unspiked inlet stack spectrum							
	11:07	iu0811e		Fourth unspiked inlet stack spectrum							
	11:08			Inlet filter T and flow have dropped; repairs begun							
	11:09			Switch to outlet stream 3.0 lpm							
	11:16	ou0811a		First unspiked outlet stack spectrum 4.0 lpm; P=746.5							
	11:19	ou0811b		Second unspiked outlet stack spectrum							
	11:21	ou0811c		Third unspiked outlet stack spectrum							
	11:24	ou0811d		Fourth unspiked outlet stack spectrum							
	11:27			Start tol (121ppm) flow 1.00 lpm, SF6 (4.0ppm) 1.0 lpm to outlet spike; total flow ~12.0lpm							
	11:32	os811a		First spiked outlet stack spectrum 4.0 lpm; P=746.6							
	11:34	os811b		Second spiked outlet stack spectrum							
	11:38			Can see SF6 and tol in calc'd spectrum smu0811b							
	11:39			Third spiked outlet stack spectrum							
	11:41			Fourth spiked outlet stack spectrum							
	11:44			Start Tol direct 121ppm 5.0lpm							
	11:51	toldira		Toluene direct 121ppm 6.5lpm P=749, recorded as outlet spec							

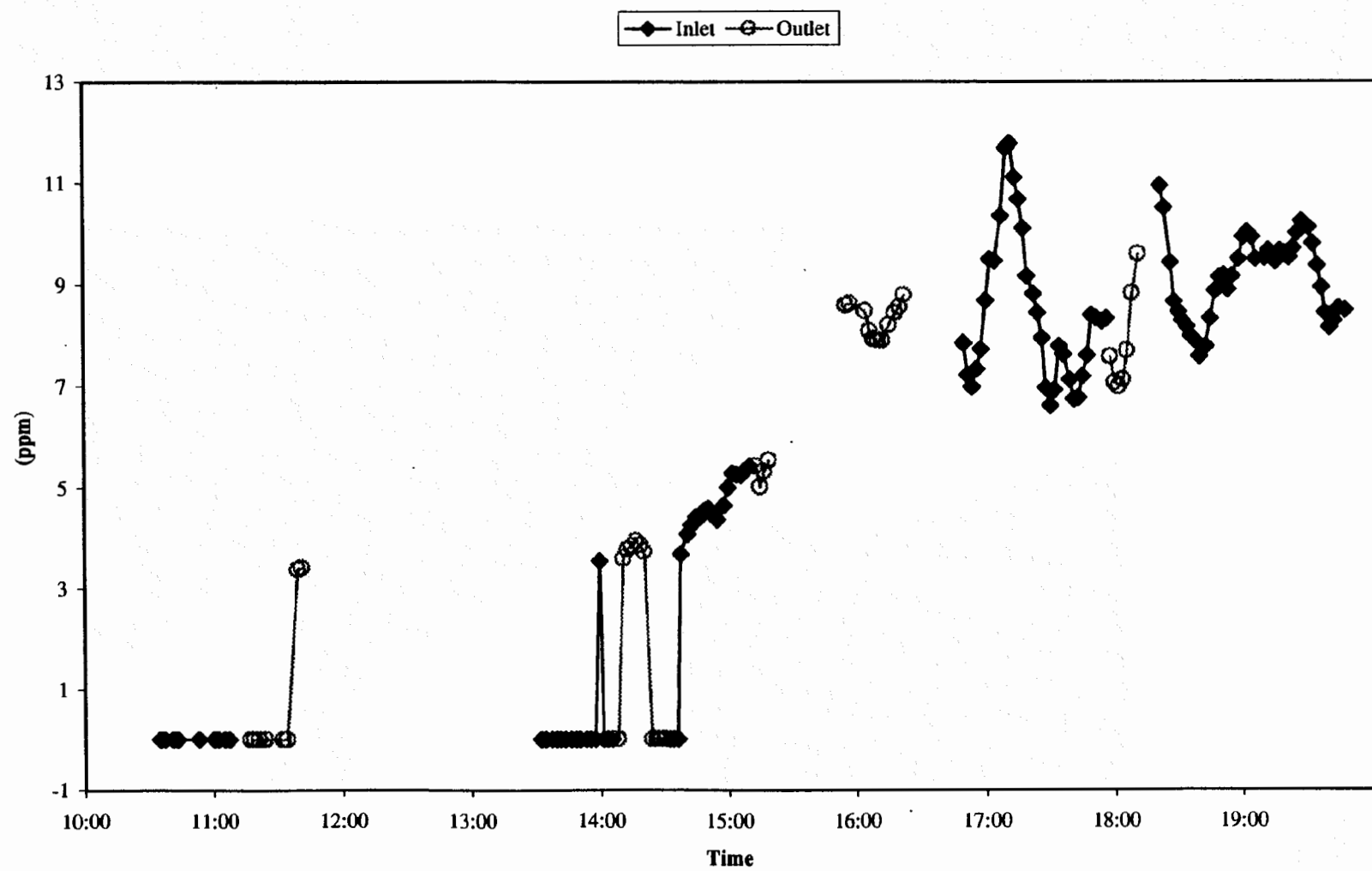
Data Sheet FTIR Batch Samples: WCI EPA Work Assignment 4-20.

Date	Sample time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond	Sample Flow	BKG
	11:54	toldirb		Toluene direct 121ppm 6.5lpm P=749; recorded as outlet spec							
	11:59	toldirc		Toluene direct 121ppm 6.5lpm P=749; recorded as outlet spec							
	12:03			Switch to inlet flow 2.5 lpm							
	12:04			Switching to continuous mode 250 scans							
	12:07			Start continuous mode; stop to change # scans from 50							
	12:14			Discovered error in date! Reset date in computer to Tuesday, August 12							
	12:40			Start continuous mode again in 0812 sub-dir, 50 scans							
				May have a throughput problem; stopping to check							
	12:50			No throughput problem- "mon" mode actually reset gain to 2R (was at 4R)							
	12:52			Re-fill detector							
	12:59			Started cont mod w/ 50 scans per spectrum as set in wci.aqp, still wrong, aborted							
	13:07			Edited wci.prm file to set aqp file (this was the problem) and gain to proper values							
	13:21			Inlet samples: Back on line using gain =4R, 250scans, T=130C, P=745.4							
8/12/97	13:35	18120022		Run 1 start, continuous mode, prmfile=WCI, AQP=WCI	250	2.0	130C	U	H/W	4.0lpm	F(500)
	13:49			May have had data glitch caused by inadvertent keyboard entry after file 27							
	14:07	18120038		Switch to outlet (4 lpm) and record meterbox data							
	14:35	18120049		Switch to inlet (4 lpm) and record meterbox data							
	14:35			Manual method port change at inlet starts							
	14:45			Manual method inlet train back on line							
	15:15			Switch to outlet (5 lpm) and record meterbox data							
	15:22			Audible process change, outlet delta(p) (prev. steady at 2.4) drops to 0.7; this is the first of the usual palette (pellet?) changes							
	15:37			FTIR data check for first "half" of Run 1.							
				Gain actually at 8 in these data; will need to scale and transform BKG* all, regenerate abs files							
	15:52			Process back on line after 30 min delay							
	15:53			Record meterbox data							
	15:53	181200(80,81)		Re-start monitor mode for gain test, two spectra completed							
	16:03	18120082		Resume FTIR outlet analysis							
	16:18			Manual method port change at outlet starts.							
	16:19			Record meterbox data							
	16:20			Manual method port change at inlet starts							
	16:47	18120091		Resume FTIR testing at inlet	250			U		4.0lpm	F(500)
	16:50			Record meterbox data							
	17:03			Re-fill detector							
	17:46			Record meterbox data							
	17:52	~18120120		Manual method port change at outlet completed; testing resumed without notice from ERG or EPA							
	17:58			Switch FTIR sampling to outlet (unaware of test resumption)	250			U		4.5lpm	F(500)
	18:22			Switch FTIR sampling to inlet							
	18:24			Record meterbox data							
8/13/97	9:00			Record several "ice" and other spectra; note NH3 contamination in system							
	9:25			Start inlet sample flow to cell 3.0 lpm							
	9:30	iu0813a		First unspiked inlet sample	250	2.0	130C	U	H/W	3.0lpm	A(500)
	9:35	iu0813b		Second unspiked inlet sample				U			
	9:41	iu0813c		Third unspiked inlet sample				U			
	9:45	iu0813d		Fourth unspiked inlet sample				U			
	9:52			Start tol (121ppm) flow 2.00 lpm, SF6 (4.0ppm) 1.0 lpm to inlet spike, total ~12.0lpm							
	9:56			SF6 clearly in "1st" spectrum							

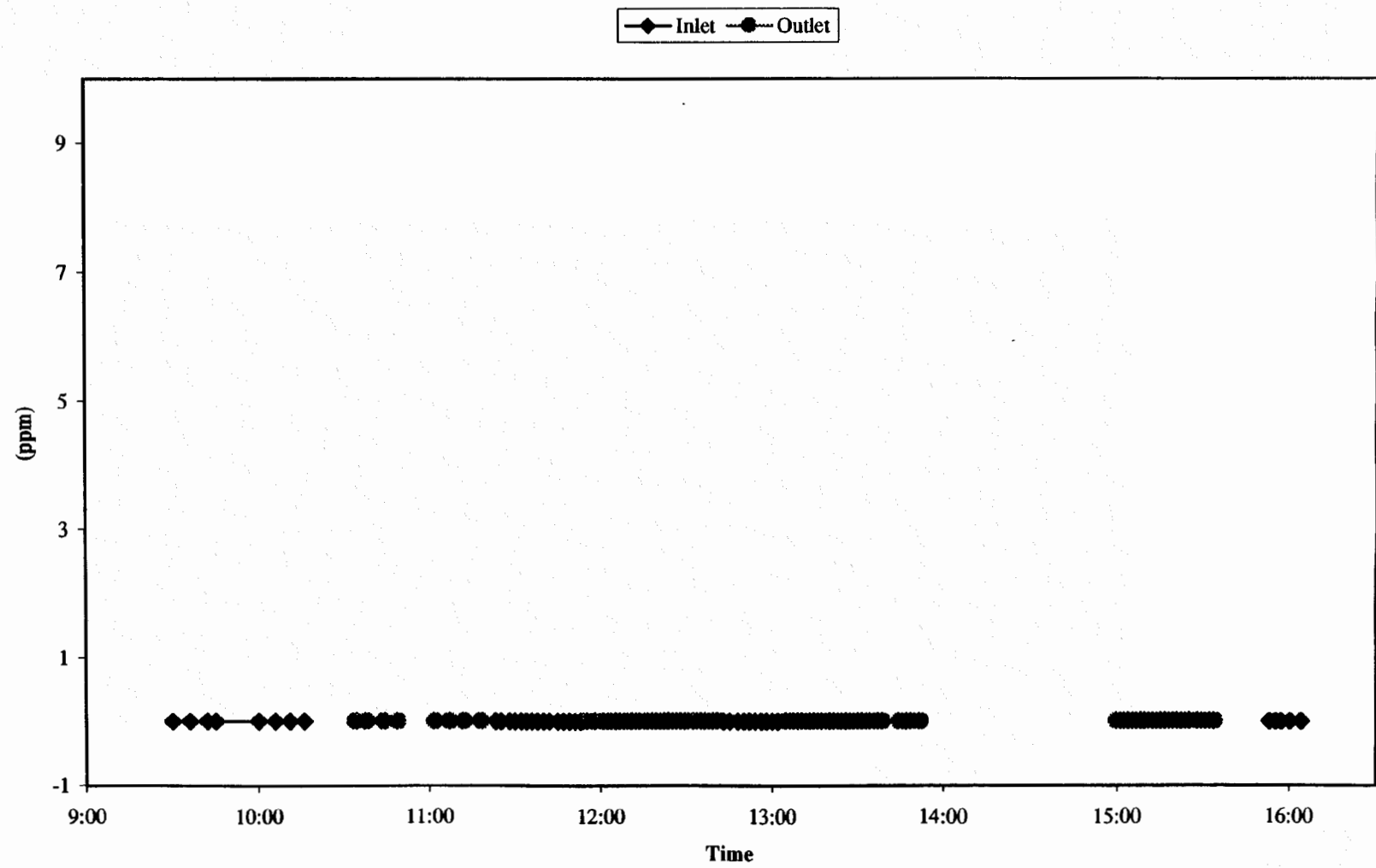
HCl Concentrations at WCI (8/14/97)



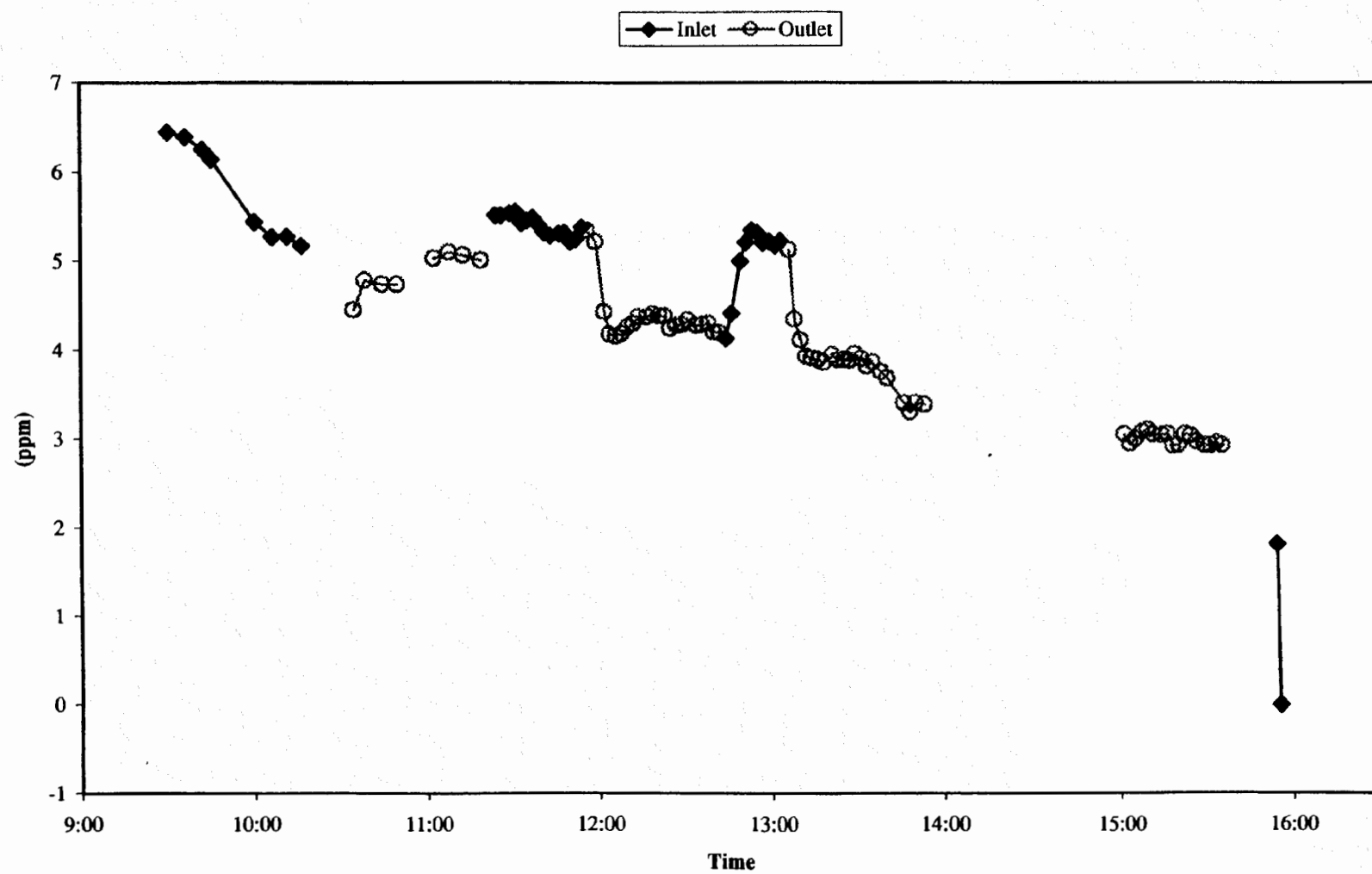
HCl Concentrations at WCI (8/12/97)



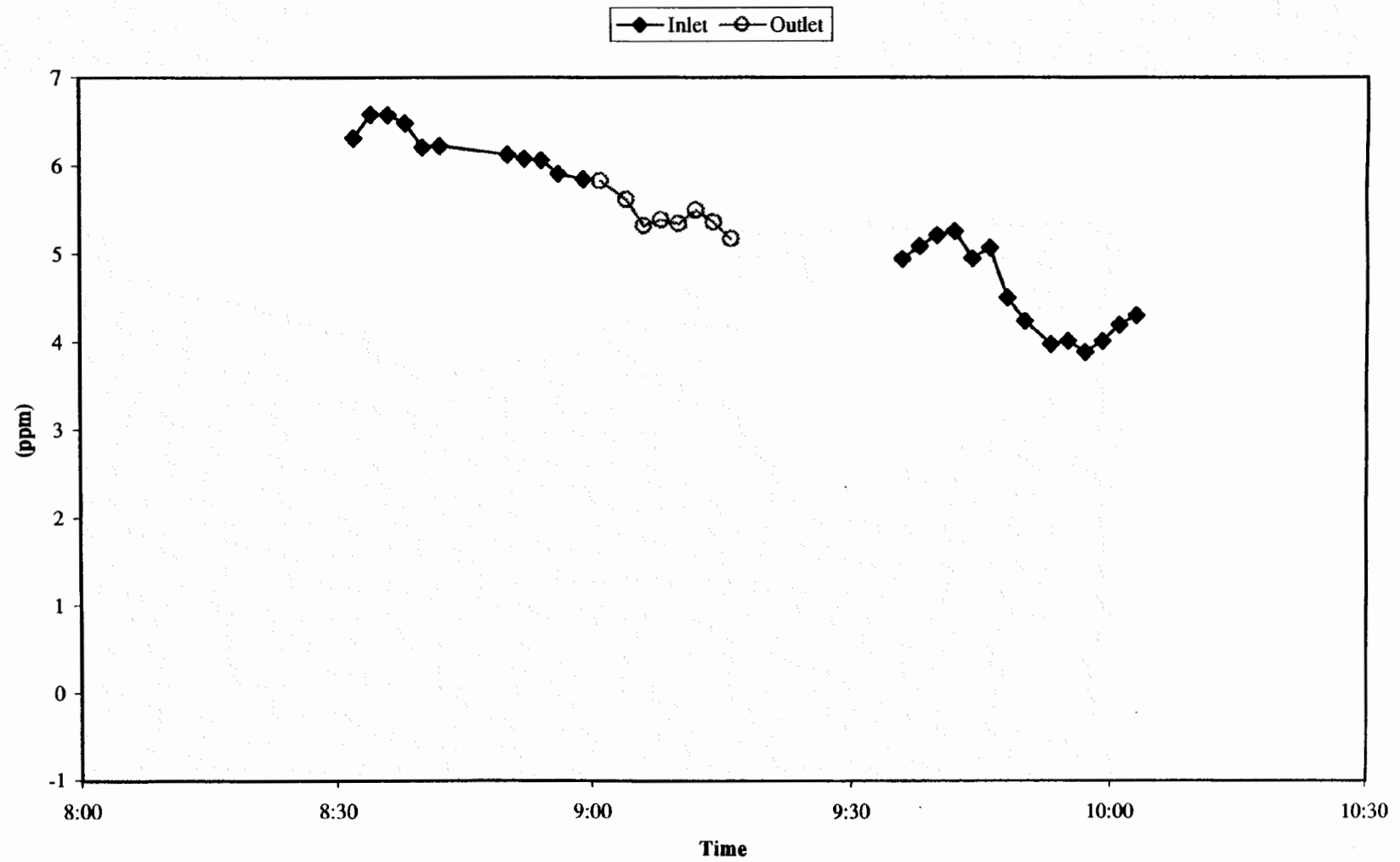
HCl Concentrations at WCI (8/13/97)



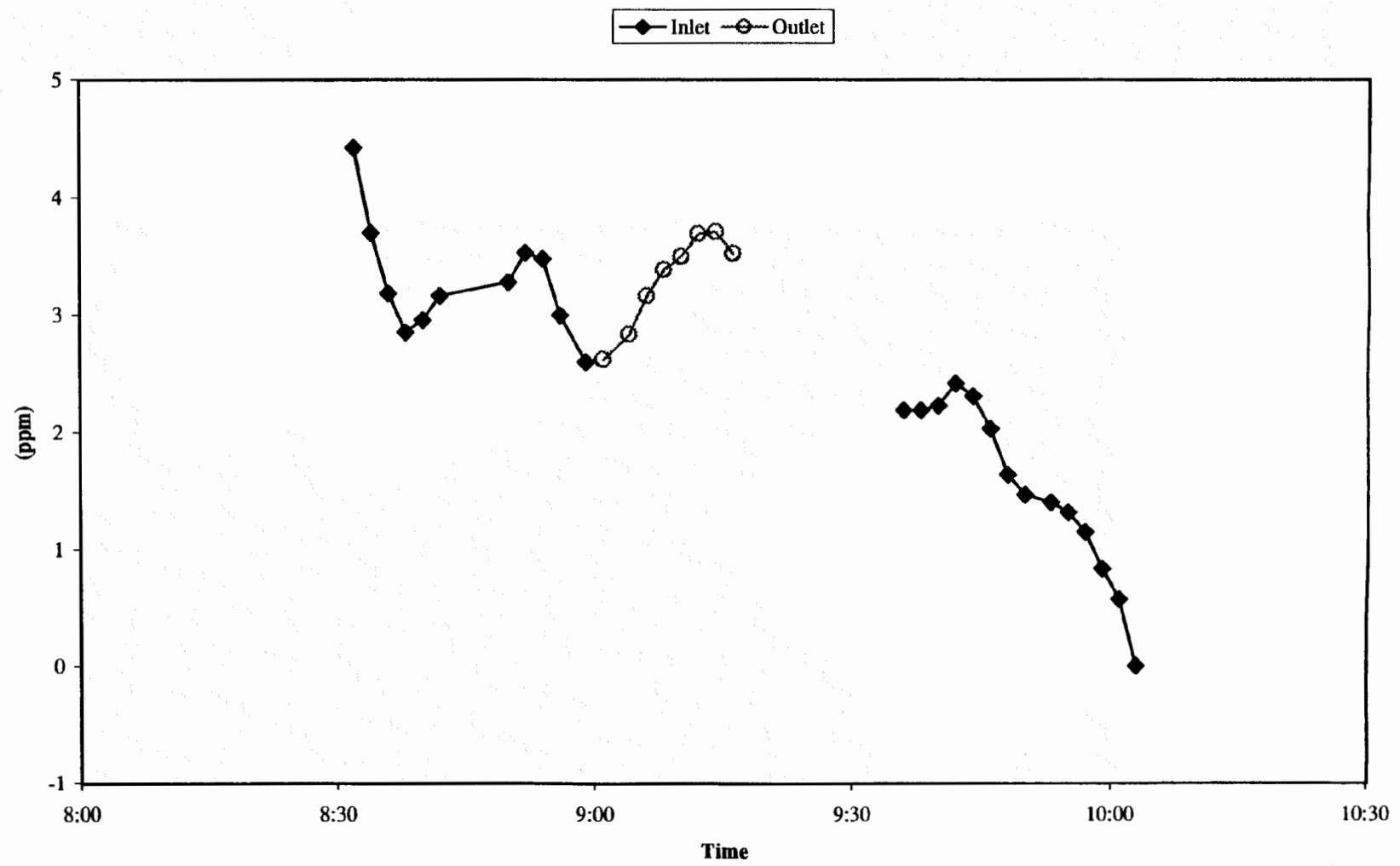
Formaldehyde Concentrations at WCI (8/13/97)



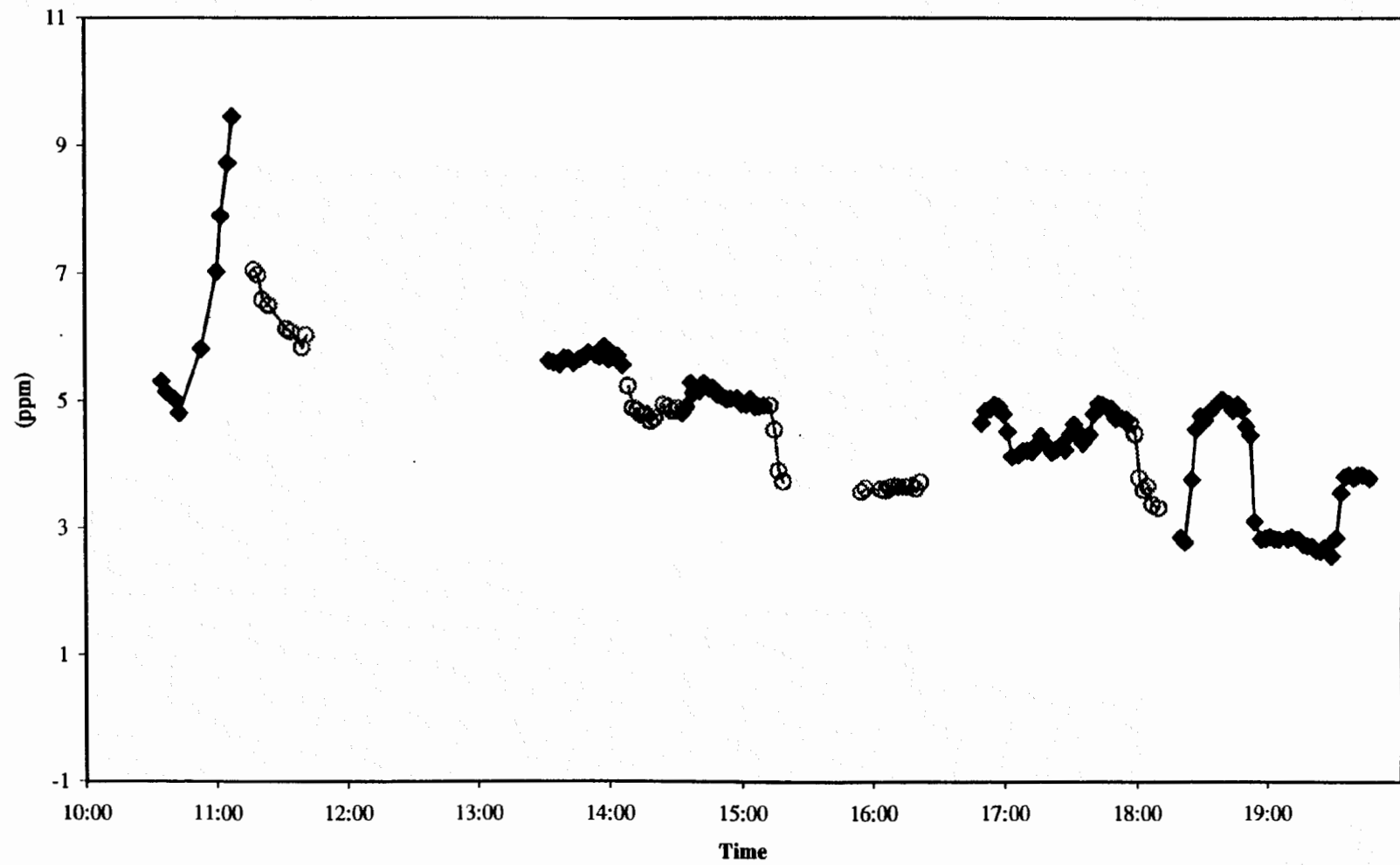
Formaldehyde Concentrations at WCI (8/14/97)



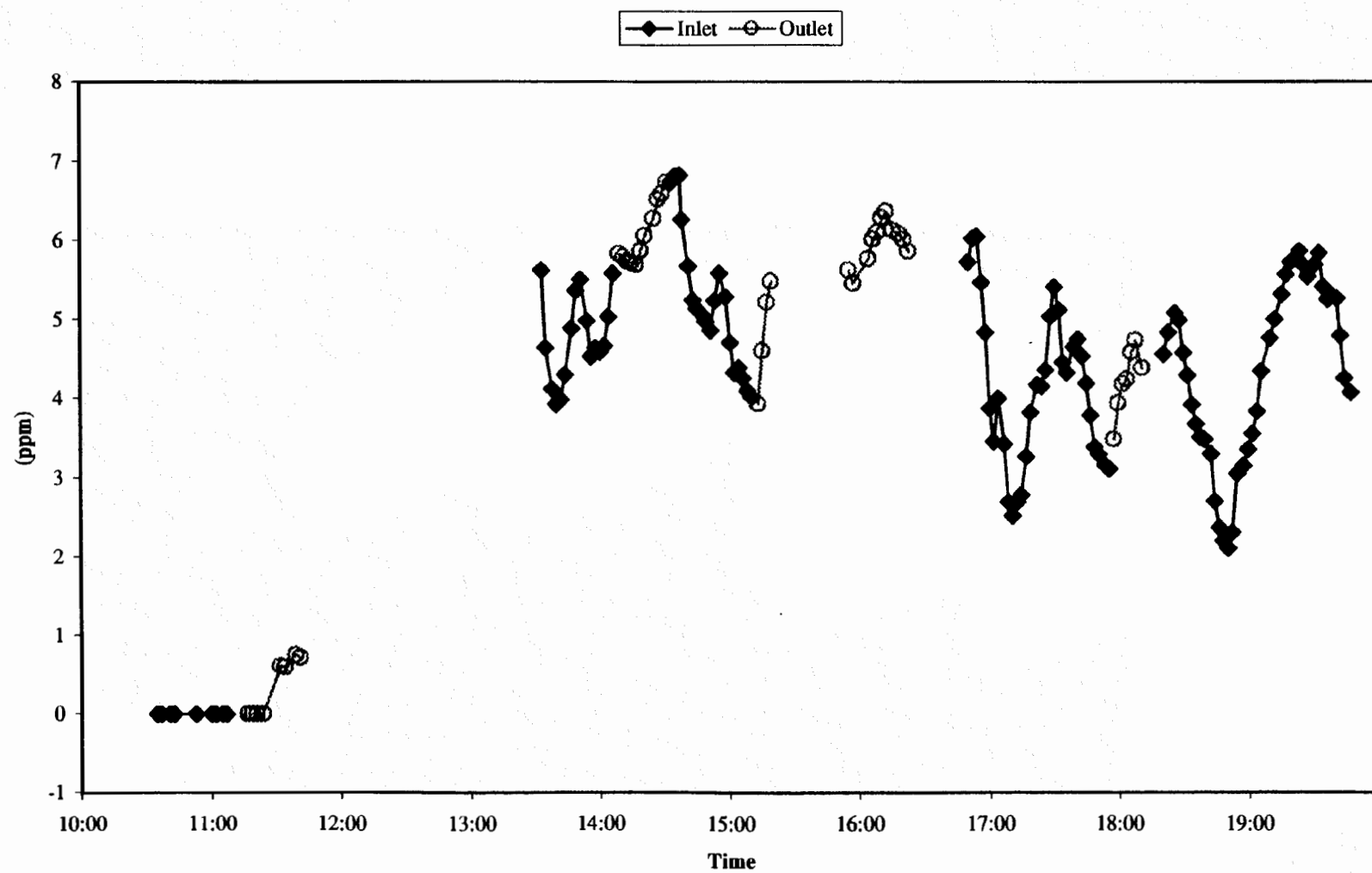
Ammonia Concentrations at WCI (8/14/97)



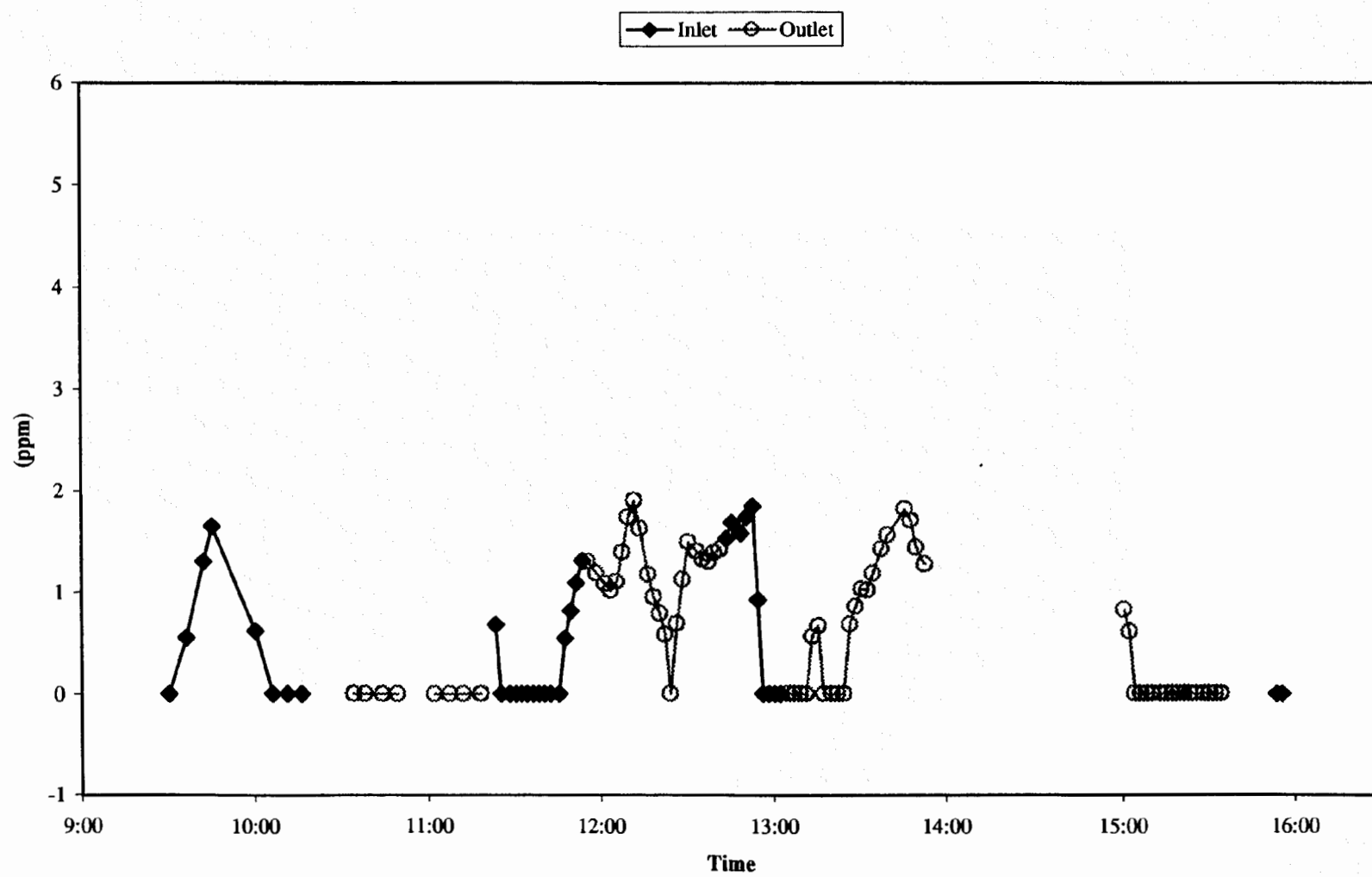
Formaldehyde Concentrations at WCI (8/12/97)



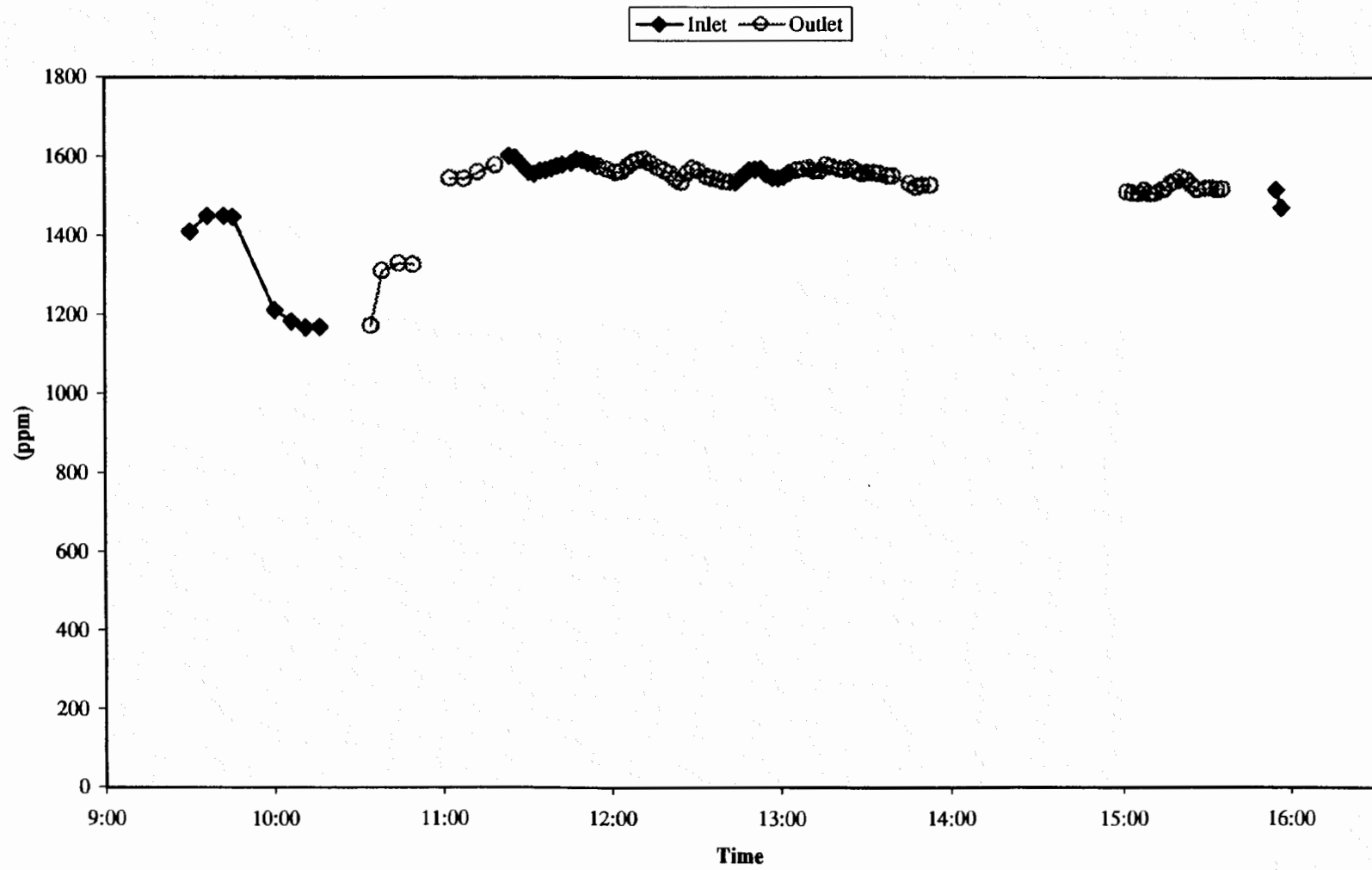
Ammonia Concentrations at WCI (8/12/97)



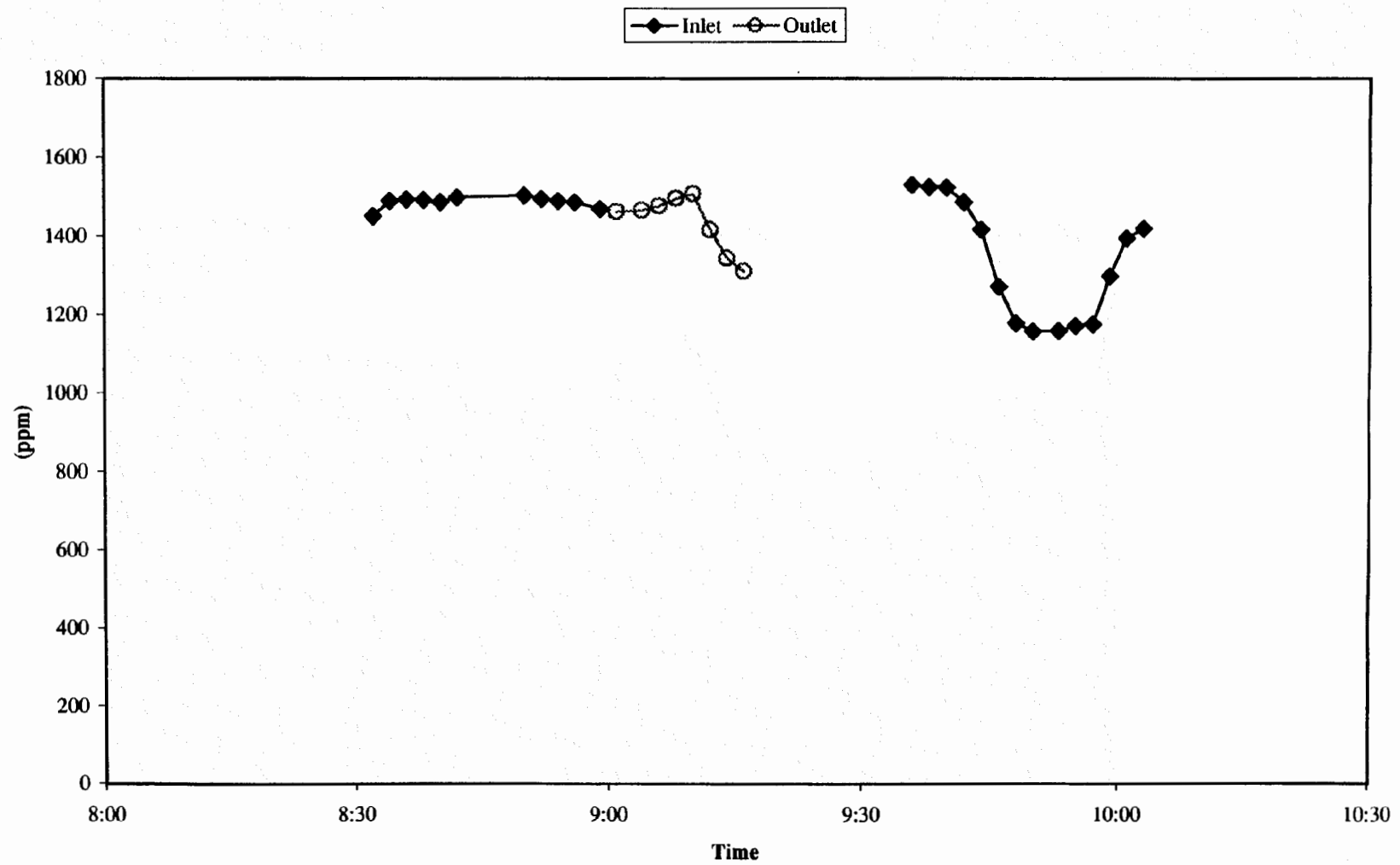
Ammonia Concentrations at WCI (8/13/97)



CO Concentrations at WCI (8/13/97)



CO Concentrations at WCI (8/14/97)



B-3 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.

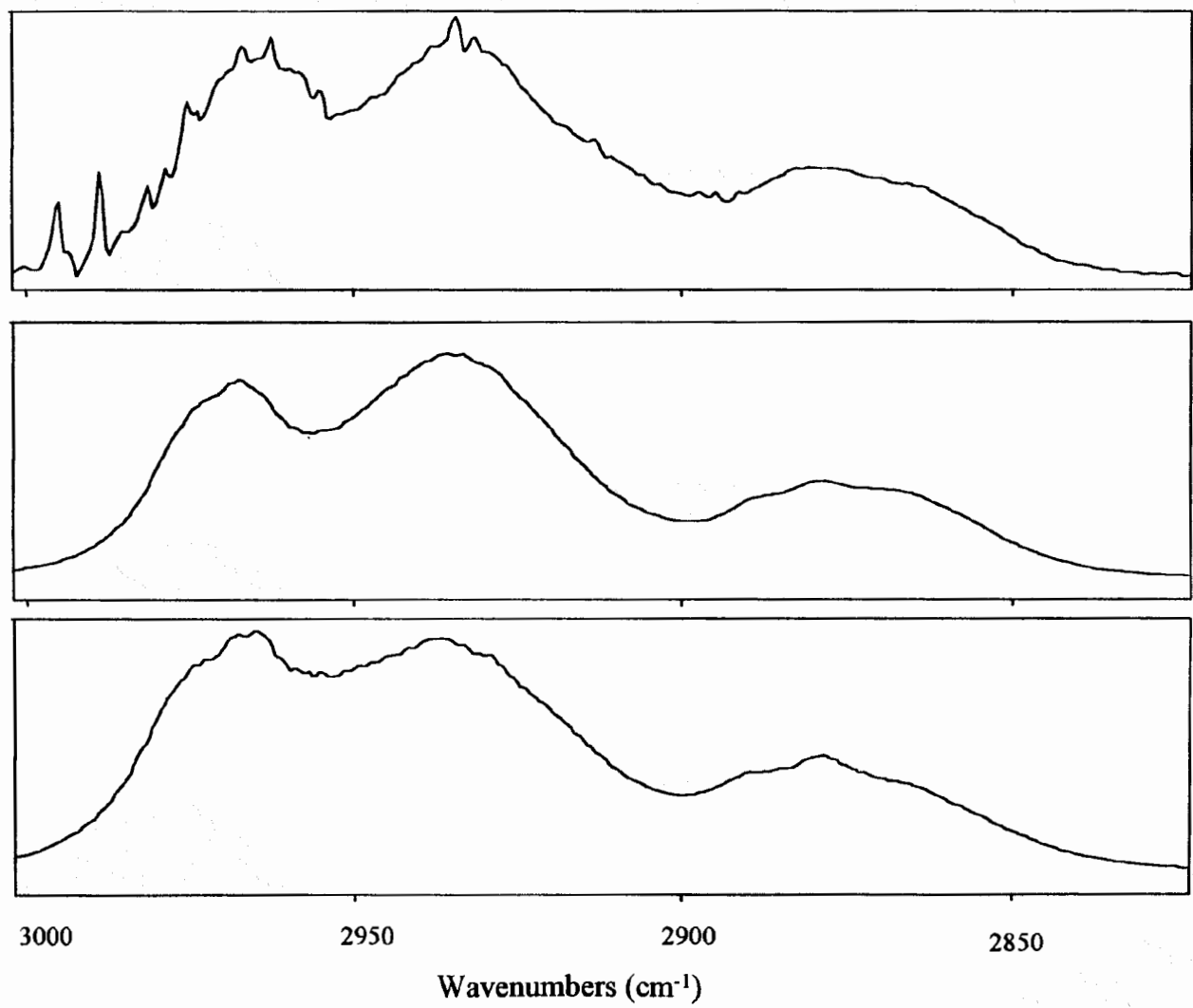


Figure B-1. Top trace, example sample spectrum; middle trace, n-heptane reference spectrum; bottom trace, n-hexane reference Spectrum.



LABORATORY PLAN FOR REFERENCE SPECTRUM MEASUREMENTS

DRAFT

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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

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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

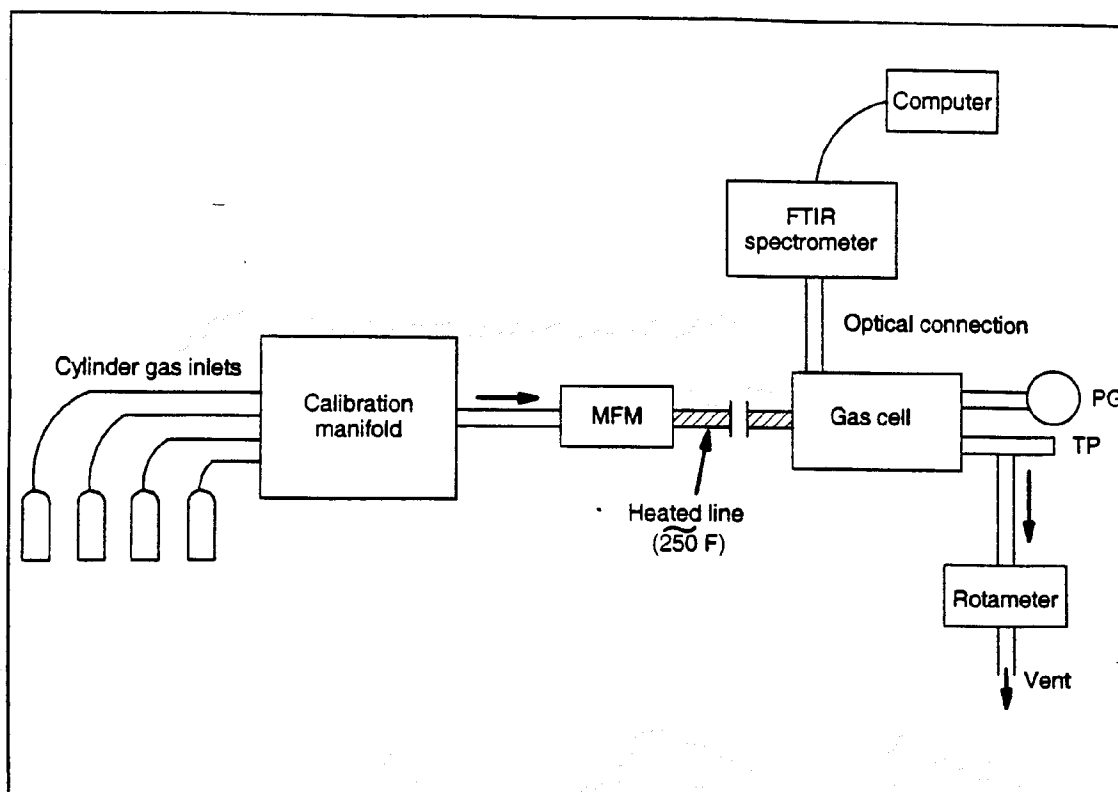


Figure 1. Measurement system configuration.

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.

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

DATE 5-10-99

VIA <u>Best Way</u>	<input type="checkbox"/> A.M. <input type="checkbox"/> PREPAID <input type="checkbox"/> P.M. <input type="checkbox"/> COLLECT	INSURE: <input type="checkbox"/> YES <input type="checkbox"/> NO AMOUNT
REQUESTED BY: <u>Dee Upton</u>	Charge No. <u>041</u> or Bill Recipient Acct. No.	REFERENCE

QUANTITY	DESCRIPTION OF MATERIAL	PRESENT LOCATION
1	Barocel pressure transducer	
1	Edwards Indicator	
1	H#21 Omega Indicator	
1	Type T thermocouple	

SIGNED

PACKING SLIP

Attachment 1
Instrument Found Out of Tolerance

Instrument: 1570 indicator with W60041111 Capacitance Manometer

Manufacturer: Edwards

MRI Number: ~~N/A~~ 4-6472 + 4-6473

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: Keith D. Smith 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: Thane By (Responsible person)

Date: 5/14/99

Attachment 1

Pressure Gauge Calibration Data Sheet

EE 24
MRI No. N/A 4-6472 Model No. / Type 1570 Serial No. 4237
Report No. with W6004111 S/N 80412159

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
<u>4-6413</u>	<u>1-22-99</u>	<u>1-22-00</u>

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: Keith S. Miller 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. <u> </u> calculated °C <u> </u>	<u> </u>	°C		
-25.0	-0.9237	-24.5	1.0	✓	
0.0	0.0059	0.2	1.0	✓	
100.0	4.2749	99.9	1.0	✓	
150.0	6.7043	150.0	1.1	✓	
200.0	9.2911	200.1	1.5	✓	
300.0	14.8765	300.3	2.2	✓	
400.0	20.8913	400.3	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
<u>Y-5835</u>	<u>12-4-98</u>	<u>12-4-99</u>
<u>012601</u>	<u>5-26-98</u>	<u>5-26-99</u>
<u>012600</u>	<u>4-23-99</u>	<u>4-23-00</u>
<u>012693</u>	<u>1-7-99</u>	<u>1-7-00</u>

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: Don Cpton Date: 5-7-99

Reviewed by: Steve A. B. Smith Jr. Date: 5-10-99

Attachment Calibration Data Sheet

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

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

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
<u>Y-5835</u>	<u>12-4-98</u>	<u>12-4-99</u>

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: Don Lipton Date: 5-7-99

Reviewed by: Steve a Parrell Jr Date: 5-10-99

Scott Specialty GasesShipped
From:6141 EASTON ROAD, BLDG 1
PLUMSTEADVILLE PA 18949-0310
Phone: 215-766-8861PO BOX 310
Fax: 215-766-2070**C E R T I F I C A T E O F A N A L Y S I S**MIDWEST RESEARCH
SCOTT KLAMM
425 VOLKER BLVDPROJECT #: 01-01788-006
PO#: 033452
ITEM #: 01021951 5AL
DATE: 3/31/98

KANSAS CITY

MO 64110

CYLINDER #: ALM025384
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: +/-5%

BLEND TYPE : CERTIFIED WORKING STD

COMPONENTETHYLENE
NITROGEN**REQUESTED GAS****CONC MOLES**

20.

PPM

BALANCE

ANALYSIS**(MOLES)**

20.0

PPM

BALANCE

ANALYST:

Genya Rogut
GENYA ROGUT



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DURHAM
Phone: 919-220-0803

NC 27704

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

CYLINDER #: ALM046483
FILL PRESSURE: 2000 PSIG

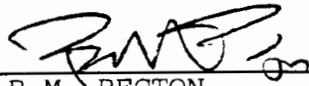
ANALYTICAL ACCURACY: +-1%
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

<u>COMPONENT</u>	<u>REQUESTED GAS</u>	<u>ANALYSIS</u>
	<u>CONC MOLES</u>	<u>(MOLES)</u>
METHANE	50. PPM	52.6 PPM
NITROGEN	BALANCE	BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST:


B.M. BECTON

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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: $\pm 1\%$
PRODUCT EXPIRATION: 5/25/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

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

NIST TRACEABLE BY WEIGHT

ANALYST: 

L. TAYLOR



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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 #: 12-34167-006

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

NIST TRACEABLE BY WEIGHT

ANALYST:


L. TAYLOR

Scott Specialty Gases

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1750 EAST CLUB BLVD
DURHAM
Phone: 919-220-0803

NC 27704

Fax: 919-220-0808

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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: $\pm 1\%$
PRODUCT EXPIRATION: 5/27/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT	REQUESTED GAS		ANALYSIS	
	CONC MOLES		(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
Phone: 919-220-0803

NC 27704

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: $\pm 1\%$
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

<u>COMPONENT</u>	<u>REQUESTED GAS</u>	<u>ANALYSIS</u>
	<u>CONC MOLES</u>	<u>(MOLES)</u>
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
Phone: 919-220-0803

NC 27704

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

CYLINDER #: ALM005876
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: $\pm 1\%$
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

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

NIST TRACEABLE BY WEIGHT

ANALYST:


L. TAYLOR



Scott Specialty Gases

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From:

1750 EAST CLUB BLVD
DURHAM
Phone: 919-220-0803

NC 27704

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

CYLINDER #: ALM017936
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: $\pm 1\%$
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

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 #: 12-34167-002
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		ANALYSIS	
	CONC	MOLES	(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
Phone: 919-220-0803

NC 27704

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

CYLINDER #: AAL21337
FILL PRESSURE: 2000 PSIG

ANALYTICAL ACCURACY: $\pm 1\%$
PRODUCT EXPIRATION: 5/26/2000

BLEND TYPE : GRAVIMETRIC MASTER GAS

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

NIST TRACEABLE BY WEIGHT

ANALYST:


L. TAYLOR

Attachment Calibration Data Sheet

MRI No.: Y-6474 Model No./Type: HH21 Serial No.: T-206821 Report No.:
Noun: Thermocouple Ambient Temperature: 74° F Ambient Humidity: 28°
Thermometer

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
<u>Y-5835</u>	<u>12-4-98</u>	<u>12-4-99</u>

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: Don Ipton Date: 5-7-99

Reviewed by: Steve A. Powell Jr Date: 5-10-99

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 #: 12-34162-001
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		ANALYSIS	
	CONC	MOLES	(MOLES)	
N-BUTANE	50.	PPM	51.3	PPM
NITROGEN		BALANCE		BALANCE

NIST TRACEABLE BY WEIGHT

ANALYST:


B.M. BECTON

Project No 4951-12, 13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7-7-99OPERATOR: Geyer**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_a)**Vacuum Leak Check Procedure:** (positive pressure)

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

Record time and cell pressure (P_{max})

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_a) * 100$$

| $\% V_L$ | should be < 4

Initials

23.8 °CJLB749.9 torrJLB11:07:45

Time

775.4 P_{min} 11:08:45

Time

775.7 P_{max} 0.3 ΔP 1.16% $\% V_L$ (of difference between P_{max} and $P_{ambient}$)**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

Purge 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: JLBDate: 7/7/99JLBJLB749.90.8 LPM (751.5 torr)0.8 LPM JLBJLBJLB

BK60707A

10.03 JLB11.23 JLB1.0 LPMAB18472Ethylene N2 (104.4 ppm)751.0JLBCT50707A

Project No 4951-12, 13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/7/99OPERATOR: T. Geyer**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_a)**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

Record time and cell pressure (P_{max})

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_a) * 100$$

| $\% V_L$ | should be < 4

Initials

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

~~Purge~~ 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: T. GeyerDate: 7/7/99

10.03

2/6

2/6

1.0 LPM 40

ALM025384

20.0 ppm ethylene in N2

40

752.0 751.8 40

CTS0707B

Project No 4951-12,13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7-8-99OPERATOR: T. Geys**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_a)

Initials

24.0 °C2/0750.5 torr**Vacuum Leak Check Procedure:**Positive
PressureEvacuate cell to ^{Purge} ~~baseline~~ ^{Maximum} pressure.

Isolate cell (close cell inlet and cell outlet)

Record time and baseline pressure (P_{min})10:36:10779.0

Leave cell isolated for one minute

Time

 P_{min} Record time and cell pressure (P_{max})10:35:10779.3

Calculate "leak rate" for 1 minute

Time

 P_{max}

$$\Delta P = P_{min} - P_{max}$$

0.3

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_a) * 100$$

 ΔP 1.04

$$|\% V_L| \text{ should be } < 4$$

 $\% V_L$ $\% \text{ of } P_{max} - P_{ambient}$ **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

Evacuate Cell

9/11 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: T. GeysDate: 7/8/9910.032/02/0AL402528420 ppm ethylene2/0752.7 @ 0.906f2/02/0CT50108A

Project No 4951-12,13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/9/99OPERATOR: J. Geyer*gas in cell*
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:

Positive Pressure Purge Evacuate cell to *about ambient* baseline pressure.

Isolate cell (close cell inlet and cell outlet)

Record time and baseline pressure (P_{min})

Leave cell isolated for one minute

Record time and cell pressure (P_{max})

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_b) * 100$$

| $\% V_L$ | 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

Purge 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: J. GeyerDate: 7/9/99

Initials

J/G126.6 °C751.4 torr__

Project No 4951-12, 17MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/12/99OPERATOR: T. Geyer**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:***Pressure* ~~Evacuate~~ ^{Purge} 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

Record time and cell pressure (P_{max})

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_b) * 100$$

| $\% V_L$ | should be < 4

Initials

126.5°C JLB2/6753.3 Torr2/6773.32/62/68:52:30

Time

 P_{min} 8:51:30

Time

774.0 P_{max} 0.70 ΔP 3.4% $\% V_L$ 7.7 $P_{max} - P_{ambient}$ **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.

1.1 LPM2/6753.31.1 LPM2/62/62/6**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

10.03 2/62/62/61.17 LPMALM02538420.0 ppm Ethylene2/6756.12/62/66150712AReviewed by: M. J. B.Date: 7/12/99

Project No 4951-12,13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/13/99OPERATOR: T. Geyer**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_a)**Vacuum Leak Check Procedure:** overpressurePressure Evacuate cell to baseline pressure.

Isolate cell (close cell inlet and cell outlet)

Record time and baseline pressure (P_{min}) 10:42:03

Leave cell isolated for one minute

Record time and cell pressure (P_{max}) 10:41:03

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_a) * 100$$

| $\% V_L$ | should be < 4

Initials

126.4 °C752.9 Torr2/6752.9 Torr2/62/62/62/62/62/62/62/62/62/6**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.

2/6 $P_{max} - P_{ambient}$ 2/62/62/62/62/62/62/62/6**Record CTS Spectrum**

Record Cell path length setting

Q 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

10.03 2/62/62/62/6ALH02538420011m2/62/62/62/6CT50713AReviewed by: J. H. [signature]Date: 7/15/99

Project No 4951-12, 13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/15/99OPERATOR: T. Geyer**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_a)**Vacuum Leak Check Procedure:**~~Purge~~ Evacuate cell to baseline pressure. (1 min)

Isolate cell (close cell inlet and cell outlet)

Record time and baseline pressure (P_{min})

Leave cell isolated for one minute

Record time and cell pressure (P_{max})

Calculate "leak rate" for 1 minute

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_a) * 100$$

| $\% V_L$ | 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

~~Purge~~ 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: T. GeyerDate: 7/16/99

Initials

2/6125.62/6754.8 torr2/62/62/6774.4 P_{min} 774.8 P_{max} 0.4 ΔP 2.0% $\% V_L$ $\% \text{ of } P_{max} - P_{ambient}$ 2/62/6754.81.03 LPM2/62/62/62/610.032/62/6ALH 153820.0 ppm ethylene2/6757.12/62/6CT5715A

Project No 4951-12, 13MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Start up ProcedureDATE: 7/6/99OPERATOR: T. Gayer

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:

~~Purge~~ Evacuate cell to ~~baseline~~ ^{pressure} pressure.

Isolate cell (close cell inlet and cell outlet)

Record time and baseline pressure (P_{min}) 9:52:40Leave cell isolated for one minute TimeRecord time and cell pressure (P_{max}) 9:51:40Calculate "leak rate" for 1 minute Time

$$\Delta P = P_{min} - P_{max}$$

Calculate "leak rate" as percentage of total pressure

$$\% V_L = (\Delta P / P_b) * 100$$

 $|\% V_L|$ should be < 4

Initials

2/6
125.3 °C
2/6
755.22/62/62/62/62/62/62/62/62/6 $\% \text{ of } P_{max} - P_{ambient}$

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.

2/62/62/62/62/62/62/6

Record CTS Spectrum

Record Cell path length setting

~~Purge~~ 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

10.872/62/62/62/62/62/62/62/62/6Reviewed by: 2/6Date: 7/6/99

FTIR DATA FORM

Background and Calibration SpectraPROJECT NO. 4951-12 and 13BAROMETRIC: 749.9 hPa

SITE: NCO Laboratory

DATE: 7/7/99OPERATOR: J. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD
10:47	BK60707A	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	"
13:41	BK60707B	5.07	N₂ @ 0.8 LPM	500	1.0	23.5	751.2	-	"
1345	CTS0707C	10.03	20.0 ppm ethylene @ 1.0 LPM	500	1.0	23.5	751.1	707A	NB/med

Did not
Do

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13BAROMETRIC: 750.5

SITE: NCO Laboratory

DATE: 7/8/99OPERATOR: T. Beyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (°F)	Gas PRESSURE	BKG	APOD
10:40	BK60708A	10.03	N ₂ through cell @ 0.47 LPM after realign.	500	1.0	24.0	751.3	-	NO/mad
11:32	CTS0708A	10.03	20.0 ppm Ethylene @ 0.90 LPM	500	1.0	23.8	752.7	708A	"
16:05	BK60708B	10.03	N ₂ @ 0.50 LPM GAIN is now 2R for some signal as cold cell (~70%). Did not realign after heating	500	1.0	126.5°C	749.9	-	NO/mad
16:26	CTS0708B	10.03	Ethylene @ 0.97 LPM 20.0 ppm	500	1.0	126.4°C	750.8	708B	NO/mad

Ameliant
= 749.0

"

FTIR DATA FORM

Background and Calibration Spectra

PROJECT NO. 4951-12 and 13BAROMETRIC: 751.4

SITE: NCO Laboratory

DATE: 7/7/99OPERATOR: T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD
13:13	BK60709A	10.03	N ₂ @ 0.49 LPM	500	1.0	126.2	751.3	—	no/level
13:44	CT50709A	10.03	20 ppm Styrene @ 0.89 LPM	500	1.0	126.3	752.0	A	"
14:14	1960709A	10.03	52.6 ppm Methane in N ₂ @ 0.44 LPM ALM 46483	500	1.0	126.3	752.2	A	"
14:27	1960709B	10.03	52.6 ppm Methane in N ₂ @ 1.16 LPM ALM 46483	500	1.0	126.3	752.1	A	"
14:55	BK60709B	10.03	N ₂ @ 0.45 LPM 0.90	500	1.0	126.3	751.3 750.4	✓	"
16:03	CT50709B	10.03	20.0 ppm Styrene @ 0.43 LPM	500	1.0	126.1	751.0	B	"
16:25	1960709B ^C JG	10.03	52.6 ppm Methane in N ₂ @ 1.11 LPM ALM 46483	500	1.0	126.1	751.3	B	"
16:34	1960709D	10.03	52.6 ppm Methane @ 1.10 LPM	500	1.0	126.1	751.2	B	"

FTIR DATA FORM

Background and Calibration SpectraPROJECT NO. 4951-12 and 13BAROMETRIC: 753.3

SITE: NCO Laboratory

DATE: 7/12/99OPERATOR: T. Geyer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (F)	Gas PRESSURE	BKG	APOD	
10:40	BK60712A	10.03	N ₂ through cell @ 1.12 LPM	500	1.0	126.0	756.3	-	NO/APOD	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	20.0 ppm Ethylene @ 0.79 (712A was ~10% low) LPM	500	1.0	126.2	755.9	"	"	"
12:20	1960712A	10.03	52.6 ppm Methane in N ₂ @ 0.99 LPM ALUMINA 483	500	1.0	126.1	755.9	712 A	"	"
12:45	BK60712A	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	"	"

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	BK60713A	10.03	N ₂ sample cell @ 0.95 LPM higher range today for this BKG	500	1.0	125.3	754.9	-	1/10 mod
11:15	BK60713B	10.03	Same conditions ReO ₂ became completely clear	500	1.0	125.4	754.9	-	"
11:54	BK60713C	10.03	Same conditions w/ dryer Cabinet 0.97 LPM	500	1.0	125.4	755.0	-	"
12:30	CT50713A	10.03	20.0 ppm Ethylene in N ₂ ALM# 25384 @ 0.95 LPM	500	1.0	125.5	755.0	713 C	"
13:03	CT50713B	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	CT50713C	10.03	20.0 ppm Ethylene @ 0.99 LPM	500	1.0	125.4	754.7	713 C	"
15:08	1960713B	10.03	52.6 ppm Methane @ 1.12 LPM	500	1.0	125.4	755.0	713 C	"

Gain = 2

"

"

"

"

"

"

"

7/15/99

FTIR DATA FORM

Background and Calibration Spectra

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

SITE: NCO Laboratory

DATE: 7/15/99OPERATOR: 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	N ₂ through cell @ 1.09 LPM	500	1.0	125.5	757.1 754.8	-	N/D MD
12:15	CT50715A	10.03	20.0 ppm Ethylene @ 1.07 LPM ALM025387	500	1.0	125.2	757.1	A	"
12:36	1960715A	10.03	52.6 ppm Methane @ 1.09 LPM	500	1.0	125.2	757.0	A	"
14:03	BK60715B	10.03	N ₂ @ 1.00 LPM	500	1.0	125.1	756.6	-	"
16:00	CT50715B	10.03	20.0 ppm Ethylene 0.97 LPM ALM025387	500	1.0	125.0	755.8	715B	"
16:20	1960715B	10.03	ALM0 46483 @ 1.05 LPM Methane (52.6 ppm)	500	1.0	125.0 105.0	755.8	715B	"

Gain = 2

"

"

"

"

"

FTIR DATA FORM

Background and Calibration SpectraPROJECT NO. 4951-12 and 13BAROMETRIC: 755.2

SITE: NCO Laboratory

DATE: 7/16/99OPERATOR: T. Gayer

TIME	FILE NAME	(Dial) PATH	NOTES	NUMBER SCANS	Resolution (cm-1)	Gas TEMP (°F)	Gas PRESSURE	BKG	APOD
11:25	BK60716A	10.03	N ₂ through cell @ 0.98 LPM	500	1.0	125.12	757.2	-	NB/Mod
12:39	CTK0716A	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 ALMS 46483 @ 1.09 LPM	500	1.0	125.1	757.2	716A	"
15:15	CT50716B	10.03	20.0 ppm Ethylene ALMS 25384 @ 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	"

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-9-99

OPERATOR: T. Geyer

Reference Spectrum Sample

Start Time (data collected)

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

Initials

H. Geyer

15:24

10.03

BK60709 D

CT50709 A, B / 1960709 A

Hexane

ALM45092

49.6 ppm

0950709 A

8/6

3/6

1.00 LPM

8/6

751.3 torr

1.002 PM

8/6

8/6

8/6

15:34

126.2 °C

Reviewed by: T. Geyer

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-9-99

OPERATOR: T. Geyer

Reference Spectrum Sample

Start Time 15:26
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

Initials

HEXANE 0950709B

10.03

BH60709A

C750709A, D 1960707 A-D

HEXANE

AL4045092

49.6 ppm

0950709B

26

46

1.09 LPM

Continued from "A"

751.5 ppm

1.09 LPM

4/6

46

4/6

15:46

126.1 °C

Reviewed by: T. Geyer

Date: 7/15/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-12-99

OPERATOR: T. Gayes

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

Initials

13:11 1-pentene
13:14
10.03
BKG0712 B
CTS0712 A-C, 1968
1-Pentene
AL6041929
50.1 ppm
195.0712 A
296
40
1.00 LPM
90
765.6
1.00 LPM
770
816
816
13:21
126.0°C

Reviewed by: T. Gayes

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

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

1 - Pentene

12:38

10.03

BK60712 B

CT50712 A-C

1960712 A

1-Pentene

ALM41929

50.1 ppm

1P60712 B

96

8/6

1.03 LPM

96

755.6

1.03 LPM

96

96

96

13:48

126.1°C

Reviewed by:

1/6

Date:

7/15/99

Project No 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7-12-99

OPERATOR: J Geyer

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

Initials

14:10 n-heptane

14:10

10.03

8460712 A

CT50712 A-C 196072 A, B

n-heptane

HAL 21337

49.97 ppm

H5P0712 A

416

216

1.00 LPM

516

755.2 torr

1.00 LPM

416

416

216

14:20

Reviewed by: J Geyer

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/12/99

OPERATOR: T. Gayle

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 *continued from A*
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

Initials

n-heptane
14:20
10.03
BK00120
CT50712 AC, 1960712 A, B
n-heptane
PAL 21337
49.97 ppm
H410712 B
36
1/6
1.00 LPM
36
755.9
1.00 LPM
36
36
1/6
14:30

Reviewed by: T. Gayle

Date: 7/12/99

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Boyer

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

2-methyl-2-pentane

13:46

10.03

BK60713.C

CT30713A-C 1960713A

2-methyl-2-pentane

DL4954078

51.4 ppm

2M21713A

8/6

8/6

1.00 LPM

8/6

754.9

1.00 LPM

8/6

8/6

8/6

14:55

Reviewed by: 

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

Start Time
Record Cell path length setting 10.03
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 continued from A
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

2-methyl-2-pentene
13.56
BK60713C
CT0713AC (960713A,B)
2-methyl-2-pentene
ALM054078
51.4 ppm
2M2P713B
016
716
716
716
754.9
0.98 LPM
716
716
716
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: L. Geyer

Initials

Reference Spectrum Sample

Start Time

3-methyl pentane

Record Cell path length setting

14.21

Record Background Spectrum File Name

10.03

Record CTS Spectrum File Name

8460713 C

Record Compound Name

150713A C 1960713A B

Record Cylinder Identification Number

3-methyl pentane

Record Cylinder Concentration

ALMO 37409

Record Spectrum File Name

50.0 ppm

Fill cell to ambient pressure with gas from cylinder standard

3M P0713A

Open cell outlet vent valve

dllo

Adjust sample flow through cell to 0.5 to 1 LPM. Record flow rate

66

Allow to equilibrate for 5 minutes

1.00 LPM

Record sample pressure in cell

76

Record sample flow rate through cell

754.6

Start spectrum collect program

1.00 LPM

Record information in data book

76

Copy Spectrum and Interferogram to backup directories

7/0

End Time

7/6

14:30

Reviewed by: L. 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

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 *Continued for A*
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

3-methylpentane
14:34
10.03
9K60713C
CT50713A-C 1960713A
3-methylpentane
ALMS 37409
50.0 ppm
3MP0713B
8/6
8/6
0.98 LPM
754.6 Torr
0.98 LPM
8/6
8/6
8/6
1444

Reviewed by: 8/6

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Geyer

Reference Spectrum Sample

Start Time (start purge)
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

Initials

(2,2,4-trimethylpentane
150 octane
12:52
10.03
BK60715A
CT50715A, 13 460715A
150 octane
~~ALH044~~ ALH044
50.3 ppm
1650715A
2/6
2/6
1.06 LPM
2/6
757.0
1.02 LPM
2/6
2/6
2/6
13:08
125.2 °C

Reviewed by: T. Geyer

Date: 7/16/99

Project No. 7951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/5/79

OPERATOR: T. Bayer

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 *continued from A*

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

Initials

Isocytone

1310

10.03

DK60715A

CT50715A, B 1960715A, D

Isocytone

ALMO4469B

50.3 ppm

1650715A

7/6

7/6

0.98

7/6

156.9

0.98

7/6

7/6

7/6

Reviewed by: 7/6 up

Date: 7/16/79

Project No 4551-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/13/99

OPERATOR: T. Gayes

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

Initials

n-Butane

14:30

12.03

0160715B

CT50715A, B 060715A, B

n-Butane

ALH624358

49.99

Pen0715A

2/6

2/6

2/6 1.07 LPM

1/6

756.4

1.09 LPM

2/6

2/6

2/6

14:48

124.9 °C

Reviewed by: T. Gayes

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

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

n-Butane

15:06

10.03

81560715 B

CT50715A,B 1960715A

Butane

ALM020217

51.3 ppm

81560715A

7/16

7/16

1.06 LPM

7/16

756.1 torr

1.00 LPM

7/16

7/16

7/16

7/16 15:23

124.9 °C

Reviewed by: 7/16/99 7/16/99

Date: 7/16/99

Project No. 4951-12, 17

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/15/99

OPERATOR: T. Geyer

Reference Spectrum Sample

continued from "A"

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

Initials

Butane

10.00

10.00

712.0

CTD712A.B 196072f

Butane

AL40202v7

51.3 ppm

BUT0715A

7/6

7/6

1.02

7/6

756.1

1.02

7/6

7/6

7/6

15:40

124.9.9

Reviewed by: 7/16/99

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

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

2-methyl-2-butene

13:08

10.03

2K607K0A

CT50710A,B 1960710A

AK

AL4005876

50.04 ppm

2M20710A

AK

AK

AK

AK

156.8

1.00 LPM

AK

AK

AK

13:30

125.2°C

Reviewed by: AK

Date: 7/16/99

Project No. 4451-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Gayle

Reference Spectrum Sample

Start Time

13:30

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

Initials

2-methyl-2-butene

10-03

PK60716A

CTS0716A,B PK60716A,B

8/6

AL4005816

50.04 ppm

2m20716B

8/6

8/6

8/6

8/6

756.6 Torr

0.95 LPM

8/6

8/6

8/6

1540

Continue from A

Reviewed by: 8/6/99

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. Gyer

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

2-methyl-1-pentene

1349

10.03

2K60716 A

0750716 A 1960716

2/6

AL4017936

50.08 ppm

2M1P716 A

2/6

2/6

5/6 1.05 LPM

5/6

756.4

1.06 LPM

2/6

Reviewed by: _____

Date: _____

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/10/99

OPERATOR: T. Geyen

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

Initials

2-methyl-1-pentene

10.03

716A

CT5716A 176716A

AL4017936

50.08

2M1P7160

0/0

0/0

0/0

0/0

756.3

0.97 LPM

0/0

0/0

0/0

1440

Reviewed by: _____

Date: _____

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Geyer

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

Initials

n-heptane

1425

10.03

716 A

CT150716 A 1960716 A

76

ARL21337

49.97 ppm

4280716 A

7/6

9/6

0.97 LPM

16

16 756.1

0.95 LPM

9/6

7/6

9/6

1450

Reviewed by: _____

Date: _____

Project No. 4951-12, 13

MIDWEST RESEARCH INSTITUTE
FTIR Reference Spectrum Checklist

DATE: 7/16/99

OPERATOR: T. Geyer

Reference Spectrum Sample

Start Time continued from "A"
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

Initials

H. Kesteno
12
10.03
716A
CT0716A 1960716A
-
ALA 21337
49.97
H6.P0716A
3/6
3/6
3/6
3/6
756.1
0.916 LPM
3/6
3/6
3/6
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. Boyer

[illegible]

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	1PE0702A	10.03	50.1 ppm 1-Pentane 419.29 ALMP 21337	500	1.0	126.0	1.00 LPM	755.6	B
13:38	1PE0712B	10.03	50.1 ppm 1-Pentane	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	HEP0712D	10.03	"	500	1.0	126.4	1.00 LPM	755.3	B

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	713 C
13:56	2M2P713B	10.03	"	500	1.0	125.6	0.98 LPM	754.9	713 C
14:21	3MPO713A	10.03	3-methyl-pentane (50.0 ppm) ALM# 37409	500	1.0	125.5	1.00 LPM	754.6	713 C
14:34	3MPO713B	10.03	"	500	1.0	125.5	0.98 LPM	754.6	713 C

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 ALMO44693	500	1.0	125.3	1.02 LPM	756.9	715A
13:13 12:44	1650715B	10.03	iso octane @ 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 @ ALMO241358	500	1.0	124.9	1.10	756.5	715 B
13 15:00	BUT0715A	10.03	Butane 51.3 ppm ALMO20217	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

FTIR DATA FORM
Sampling Data

PROJECT NO. 4951-12 and 13

BAROMETRIC: 755.2

SITE: NCO Laboratory

DATE: 7/16/99

OPERATOR: T. Gyar

Time	File Name	(Dial) Path	NOTES	Scans	Resolution (cm-1)	Gas Temp (°C)	Flow Rate	Gas Pressure	BKG
1316	2M28716A 2M28716A	10.03	2-methyl-2-butanol ALM005876, 60.04 ppm	500	1.0	126.2	1.00 LPM	756.8	716 A
1330	2M28716B 2M28716A	10.03	2-methyl-2-butanol	500	1.0	125.2	0.95 LPM	756.6	716 A
1352	2M18716A	10.03	2-methyl-1-pentanol ALM017936 50.08 ppm	500	1.0	125.2	1.06 LPM	756.4	716 A
1410	2M18716B	10.03	2-methyl-1-pentanol	500	1.0	125.1	0.97	756.3	716 A
1440	H2P0716A	10.03	n-heptane { 49.97 ppm AAL21337 } Ge-Mass spec became #2 CTS was low	500	1.0	125.3	0.85 LPM	756.1	716 A
1450	H2P0716B	10.03	n-heptane	500	1.0	125.3	0.96 LPM	756.1	716 A

Project No 4951-12, 13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7-7-99

OPERATOR: T. Gayer

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: T. Gayer

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

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

Purge 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: T. Geyer

Date: 7/8/99

Project No 4951-12513

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

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 709.6

Record information in data book.

Copy Background to C-drive and backup using batch file.

Record CTS Spectrum

~~Purge~~
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 *Also Methane - see data sheet.*

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: *[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

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.

*used
Background "B"*

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:

T. Geyer

Date:

7/15/99

Project No 4981-12, 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

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.

*no
BKG0713C*

Record CTS Spectrum

~~Evacuate~~ 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

2/6
2/6
2/6 ALM 25384
2/6 20 ppm ethylene
2/6 754.7
2/6
2/6
CTS0713C

Close cylinders

Evacuate or Purge CTS from cell using nitrogen

Leave cell under low nitrogen purge or under vacuum

Fill MCT detector dewar

2/6
2/6
2/6
2/6

Reviewed by: *T. Geyer*

Date: 7/15/99

Project No. 4951-12,13

MIDWEST RESEARCH INSTITUTE
DAILY CHECKLIST
Shut Down Procedure

DATE: 7/15/99

OPERATOR: T. Gay

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.

used BKG "B"

Record CTS Spectrum

2/6
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

2/6
2/6
2/6
ALMB25384
20 ppm ethylene
2/6
2/6
2/6
2/6
2/6
CTS0715B

Close cylinders

Evacuate or Purge CTS from cell using nitrogen

Leave cell under low nitrogen purge or under vacuum

Fill MCT detector dewar

2/6
2/6
2/6
—

Reviewed by: 2/6

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

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

~~Purge~~ 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: T. Gayer

Date: 7/16/99

APPENDIX C

EQUIPMENT CALIBRATION CERTIFICATES

C-1 CALIBRATION GAS CERTIFICATES



Scott Specialty Gases

Shipped
From:6141 EASTON ROAD
PLUMSTEADVILLE
Phone: 215-766-8861

PA 18949-0310

PO BOX 310

Fax: 215-766-2070

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
DAVE ALBURTY, X1525
425 VOLKER BLVDPROJECT #: 01-89796-005
PO#: 029872
ITEM #: 01023912 4AL
DATE: 5/13/97

KANSAS CITY

MO 64110

CYLINDER #: ALM057730

ANALYTICAL ACCURACY: +/- 2%

FILL PRESSURE: 2000 PSIG

BLEND TYPE : CERTIFIED MASTER GAS

COMPONENTTOLUENE
AIRREQUESTED GAS
CONC MOLES

120.

PPM

BALANCE

ANALYSIS
(MOLES)

121.

PPM

BALANCE

CERTIFIED

ANALYST:

GENYA ROGUT



Scott Specialty Gases

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

Fax: 248-589-2134

CERTIFICATE OF ANALYSIS

MIDWEST RESEARCH
MELISSA TUCKER; # 026075
425 VOLKER BLVD

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

KANSAS CITY

MO 64110

CYLINDER #: A7853
FILL PRESSURE: 2000 PSI

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

BLEND TYPE : CERTIFIED MASTER GAS

COMPONENT
SULFUR HEXAFLUORIDE
NITROGEN

REQUESTED GAS
CONC MOLES
4. PPM
BALANCE

ANALYSIS
(MOLES)
4.01 PPM
BALANCE

CERTIFIED MASTER GAS

ANALYST:

Mark Riste

Scott Specialty GasesShipped
From:6141 EASTON ROAD
PLUMSTEADVILLE
Phone: 215-766-8861

PA 18949-0310

PO BOX 310

Fax: 215-766-2070

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

MIDWEST RESEARCH
TOM GEYER
425 VOLKER BLVDPROJECT #: 01-88514-001
PO#: 029257
ITEM #: 01021951 LAL
DATE: 3/25/97

KANSAS CITY

MO 64110

CYLINDER #: ALM023940

ANALYTICAL ACCURACY: +-1%

FILL PRESSURE: 2000 PSIG

BLEND TYPE : GRAVIMETRIC MASTER GAS

COMPONENT

ETHYLENE

NITROGEN

REQUESTED GAS
CONC MOLES

20.

PPM

BALANCE

ANALYSIS
(MOLES)

20.01

PPM

BALANCE

C E R T I F I E D

ANALYST:

G. Debit
GENYA KOGUT



813-686-2184
FAX 813-686-2082

LIQUID CARBONIC

CYLINDER GAS PRODUCTS

5700 SOUTH ALAMEDA STREET • LOS ANGELES, CA 90008

CERTIFICATE OF ANALYSIS / EPA PROTOCOL GAS

CUSTOMER ENV. & INDUST. DIST.

P.O. NUMBER 84110932 RE:032295-1

REFERENCE STANDARD

COMPONENT	GH18	NIST SRM NO.	CYLINDER NO.	CONCENTRATION
PROPANE		vs 2649	SA 7651	5019 ppm

ANALYSIS READINGS

R=REFERENCE STANDARD

Z=ZERO GAS

C=GAS CANDIDATE

1. COMPONENT	PROPANE	GH18	ANALYZER MAKE-MODEL-SN	HP 5890 SERIES II	S/N 3310A48533
ANALYTICAL PRINCIPLE	GC/ PLAME IONIZATION			LAST CALIBRATION DATE	03/28/95
FIRST ANALYSIS DATE	04/07/95			SECOND ANALYSIS DATE	
Z 0	R 2607332	C 2744925	CONC. 5283 ppm	Z	R C CONC.
R 2612210	Z 0	C 2745146	CONC. 5274 ppm	R	Z C CONC.
Z 0	C 2737157	R 2602984	CONC. 5278 ppm	Z	C R CONC.
U/M W-6		MEAN TEST ASSAY	5278 ppm	U/M W-6	MEAN TEST ASSAY

Values not valid below 150 psig

THIS CYLINDER NO.	SA 8458	CERTIFIED CONCENTRATION
MAS BEEN CERTIFIED ACCORDING TO SECTION	EPA-600/R93/224	PROPANE 5278 ppm
OF TRACEABILITY PROTOCOL NO.	Rev. 9/93	NITROGEN BALANCE
PROCEDURE	61	
CERTIFIED ACCURACY	± 1 % NIST TRACEABLE	
CYLINDER PRESSURE	2000 PSIG	
CERTIFICATION DATE	04/07/95	
EXPIRATION DATE	04/07/98	TERM 36 MONTHS

ANALYZED BY

[Signature]

CERTIFIED BY

[Signature] JIM T. YOUNG

C-2 ENVIRONICS MASS FLOW METER CALIBRATIONS

ENVIRONICS FLOW CONTROLLER CALIBRATION SHEET

Mf #: 4, Description: AIR, Size: 100.0 SCCM, K-factor: 1.0

SERIAL # AW9612049

This flow controller was calibrated using a Sierra Cal Bench(TM), a traceable Primary Flow Standard Calibration System. This calibration is referenced to dry air at a temperature of 32F (C) and a pressure of 29.92 in.Hg (760Torr).

	Set Flow	True Flow
5 %	5.0 CCM	5.236 CCM
10 %	10.0 CCM	10.269 CCM
20 %	20.0 CCM	20.434 CCM
30 %	30.0 CCM	30.524 CCM
40 %	40.0 CCM	40.606 CCM
50 %	50.0 CCM	50.636 CCM
60 %	60.0 CCM	60.683 CCM
70 %	70.0 CCM	70.779 CCM
80 %	80.0 CCM	80.917 CCM
90 %	90.0 CCM	91.035 CCM
100%	100.0 CCM	101.12 CCM

Calibration data was last saved on Friday 03 January 97 at 19:11:00

Verified by: Paul Senturia Date: 1 - 3 - 97

ENVIRONICS FLOW CONTROLLER CALIBRATION SHEET

Mf #: 3, Description: AIR, Size: 1000.0 SCCM, K-factor: 1.0

SERIAL # AW9502153

This flow controller was calibrated using a Sierra Cal Bench(TM), a traceable Primary Flow Standard Calibration System. This calibration is referenced to dry air at a temperature of 32F (C) and a pressure of 29.92 in.Hg (760Torr).

	Set Flow		True Flow
5 %	50.0	CCM	50.515 CCM
10 %	100.0	CCM	101.84 CCM
20 %	200.0	CCM	204.84 CCM
30 %	300.0	CCM	306.67 CCM
40 %	400.0	CCM	408.82 CCM
50 %	500.0	CCM	510.43 CCM
60 %	600.0	CCM	611.44 CCM
70 %	700.0	CCM	713.59 CCM
80 %	800.0	CCM	816.61 CCM
90 %	900.0	CCM	918.19 CCM
100%	1000.0	CCM	1021.3 CCM

Calibration data was last saved on Friday 03 January 97 at 17:55:00

Verified by: Mark Santinny Date: 1 - 3 - 97

ENVIRONICS FLOW CONTROLLER CALIBRATION SHEET

Mf #: 2, Description: AIR , Size: 10000. SCCM, K-factor: 1.0

SERIAL # AW9502157

This flow controller was calibrated using a Sierra Cal Bench(TM), a traceable Primary Flow Standard Calibration System. This calibration is referenced to dry air at a temperature of 32F (___C) and a pressure of 29.92 in.Hg (760Torr).

	Set Flow	True Flow
5 %	500.0 CCM	510.51 CCM
10 %	1000.0 CCM	1021.4 CCM
20 %	2000.0 CCM	2046.9 CCM
30 %	3000.0 CCM	3074.8 CCM
40 %	4000.0 CCM	4103.8 CCM
50 %	5000.0 CCM	5135.6 CCM
60 %	6000.0 CCM	6156.8 CCM
70 %	7000.0 CCM	7182.5 CCM
80 %	8000.0 CCM	8203.3 CCM
90 %	9000.0 CCM	9219.5 CCM
100%	10000. CCM	10233. CCM

Calibration data was last saved on Friday 03 January 97 at 17:09:00

Verified by: Neal Senturia Date: 1 - 3 - 97

5/12073

ENVIRONICS FLOW CONTROLLER CALIBRATION SHEET

Mf #: 1, Description: AIR, Size: 10000. SCCM, K-factor: 1.0

SERIAL # AW9502156

This flow controller was calibrated using a Sierra Cal Bench(TM), a traceable Primary Flow Standard Calibration System. This calibration is referenced to dry air at a temperature of 22F (C) and a pressure of 29.92 in.Hg (760Torr).

	Set Flow	True Flow
5 %	500.0 CCM	498.79 CCM
10 %	1000.0 CCM	1009.0 CCM
20 %	2000.0 CCM	2029.8 CCM
30 %	3000.0 CCM	3058.2 CCM
40 %	4000.0 CCM	4088.8 CCM
50 %	5000.0 CCM	5121.9 CCM
60 %	6000.0 CCM	6143.3 CCM
70 %	7000.0 CCM	7178.3 CCM
80 %	8000.0 CCM	8206.3 CCM
90 %	9000.0 CCM	9224.6 CCM
100%	10000. CCM	10252. CCM

Calibration data was last saved on Friday 03 January 97 at 16:22:00

Verified by: Harl Sentiary Date: 1 - 3 - 97

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

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_1) and analytical uncertainty (AU_1) 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.

$$A_i = a_i b c_i \quad (1)$$

where:

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

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

b = path length of the cell.

c_i = concentration of the i th 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 discrete, 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 bypass 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 bypass 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 $\pm 2^{\circ}\text{C}$.

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 (C_{MAX_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, C_{MAX_i} . The expected measurement range is fixed by DL_i and C_{MAX_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 $< \text{AU}$. 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 , 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_o 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:

$$\%V_L = 50 t_{ss} \frac{\Delta P_{max}}{P_{ss}} \quad (2)$$

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:

$$DF = \frac{SF_{6(sp)}}{SF_{6(dir)}} \quad (3)$$

where:

$$CS = DF * Spike_{dir} + Unspike(1-DF) \quad (4)$$

- 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.
- $SF_{6(sp)}$ = Diluted SF_6 (or tracer gas) concentration measured in a spiked sample.

S_{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 ≤ 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:

$$V_{ss} = \frac{V_m \frac{P_m}{T_m}}{\left[\frac{P_f}{T_f} - \frac{P_i}{T_i} \right]} \quad (5)$$

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,

$$C_{corr} = \left(\frac{L_r}{L_s} \right) \left(\frac{T_s}{T_r} \right) \left(\frac{P_r}{P_s} \right) C_{calc} \quad (6)$$

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:

$$B = S_m - CS \quad (7)$$

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 it is determined that the bias is significant and $CF > \pm 30$ 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 1.6×10^{-4} to 3.2×10^{-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

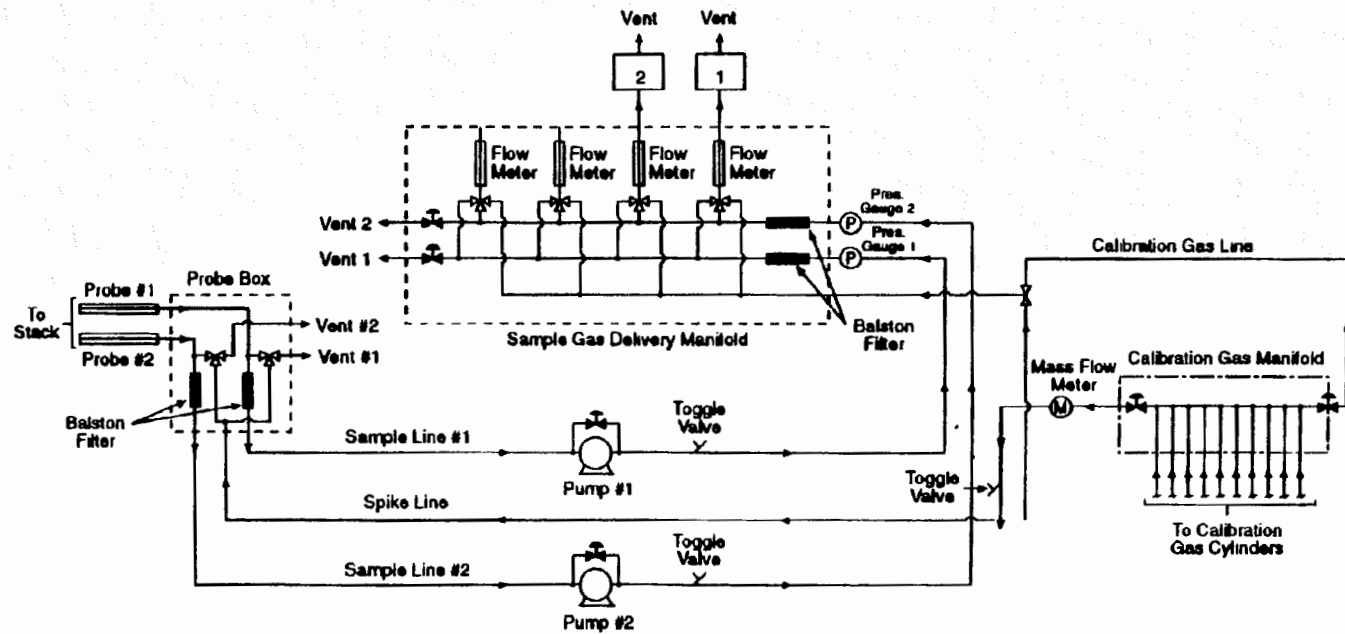


Figure 1. Extractive FTIR sampling system.

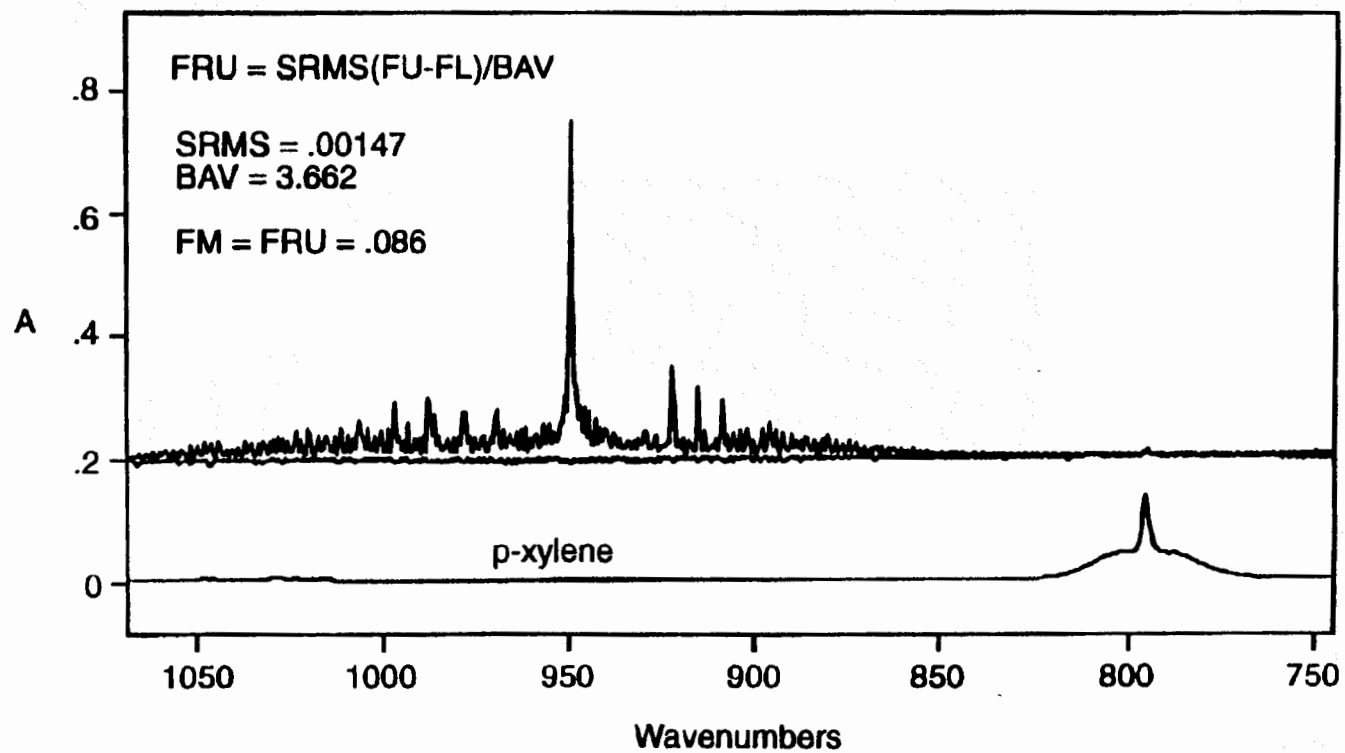


Figure 2. Fractional Reproducibility. Top: average of cts1031a and cts1101b. Bottom: Reference spectrum of p-xylene.

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 i^{th} 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_1 (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.

Note: 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 $\pm t_{SR}$. 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_1 , 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_{GST} , 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 $R_{LPS} = (L_R P_{RTS}) / (L_S P_{STR})$. 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 44, 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, $\bar{\nu}$ - 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).

$$A = \log_{10} \left(\frac{1}{T} \right) = -\log_{10} T \quad (1)$$

AAI_{im} - band area of the i^{th} analyte in the m^{th} 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 i^{th} analyte in the m^{th} 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 i^{th} analyte concentration, expressed as a fraction of the analyte concentration determined in the analysis.

AVT_m - average estimated total absorbance in the m^{th} analytical region.

CKWN_k - estimated concentration of the k^{th} known interferant.

CMAX_i - estimated maximum concentration of the i^{th} analyte.

- $CPOT_j$ - estimated concentration of the j^{th} potential interferant.
- DL_i , **required detection limit** - for the i^{th} 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 m^{th} analytical region.
- FAU_i , **fractional analytical uncertainty** - calculated uncertainty in the measured concentration of the i^{th} 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 i^{th} 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 m^{th} analytical region.
- FFU_m - upper wavenumber position of the CTS absorption band associated with the m^{th} analytical region.
- FL_m - lower wavenumber position of the m^{th} analytical region.
- FMU_i , **fractional model uncertainty** - calculated uncertainty in the measured concentration of the i^{th} 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 i^{th} analyte because of errors in the reproducibility of spectra from the FTIR system.
- FU_m - upper wavenumber position of the m^{th} analytical region.
- IAI_{jm} - band area of the j^{th} potential interferant in the m^{th} analytical region, at its expected concentration ($CPOT_j$).
- IAV_{im} - average absorbance of the i^{th} analyte in the m^{th} analytical region, at its expected concentration ($CPOT_j$).

ISC_i or k, indicated standard concentration - the concentration from the computerized analytical program for a single-compound reference spectrum for the *i*th 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 *i*th analyte, based on spectral data in the *m*th 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 *j*th interferant, based on spectral data in the *m*th analytical region, can be maintained.

MIL, minimum instrumental linewidth - the minimum linewidth from the FTIR system, in wavenumbers.

Note: 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 m^{th} analytical region.

RMSD, root mean square difference - a measure of accuracy determined by the following equation:

$$RMSD = \sqrt{\left(\frac{1}{n}\right) \sum_{i=1}^n e_i^2} \quad (2)$$

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

$$RMSD = \sqrt{\left(\frac{1}{n}\right) \sum_{i=1}^n (A_i - A_M)^2} \quad (3)$$

RSA_i - the (calculated) final concentration of the i^{th} analyte.

RSI_k - the (calculated) final concentration of the k^{th} 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 m^{th} 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 m^{th} 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 m^{th} 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

Analyte Labels	Analytical Regions				
	1	.	.	.	M
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

Analyte Labels	Analytical Regions				
	1	.	.	.	M
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, under those conditions necessary to yield the specified minimum instrumental linewidth, 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:

$$RMS_{EST} = RMS_{MAN} \cdot TP \cdot \sqrt{\frac{t_{SS}}{t_{MAN}}} \quad (4)$$

APPENDIX D

ESTIMATING MINIMUM CONCENTRATION MEASUREMENT UNCERTAINTIES (MAU and MIU)

D.1 General

Estimate the minimum concentration measurement uncertainties for the i^{th} analyte (MAU_i) and j^{th} interferant (MIU_j) based on the spectral data in the m^{th} 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 $\text{RMS} = \text{RMS}_{\text{Sm}}$ if measured (Appendix G), or set $\text{RMS} = \text{RMS}_{\text{EST}}$ if estimated (Appendix C).

D.2.2 For each analyte associated with the analytical region, calculate

$$\text{MAU}_{im} = (\text{RMS}) (\text{DL}_i) (\text{AU}_i) \frac{(\text{FU}_m - \text{FL}_m)}{\text{AAI}_{im}} \quad (5)$$

D.2.3 If only the m^{th} analytical region is used to calculate the concentration of the i^{th} analyte, set $\text{MAU}_i = \text{MAU}_{im}$.

D.2.4 If a number of analytical regions are used to calculate the concentration of the i^{th} 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

$$MAU_i = \sum_{k \in \{m'\}} W_{ik} MAU_{ik} \quad (6)$$

where the weight W_{ik} is defined for each term in the sum as

$$W_{ik} = (FM_k - FL_k) \left(\sum_{p \in \{m'\}} [FM_p - FL_p] \right)^{-1} \quad (7)$$

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 m^{th} analytical region is used to calculate the concentration of the i^{th} 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 i^{th} 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

$$FRU_i = \sum_{k \in \{m'\}} W_{ik} FM_k \quad (8)$$

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 i^{th} 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			Interferants		
			i=1.....I			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 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 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 $A_S = rA_R + E$, determine the least-squares estimate of r' , the value of r which minimizes the square error 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 A_S and A_R as described in Section H.2.1, using values from {R1} to form A_R , and values from {R4} to form A_S . Calculate the values

$$\text{NRMS}_E = \sqrt{\sum_{i=1}^n \left[A_{Si} - \left(\frac{T_R}{T_S} \right) \left(\frac{L_S}{L_R} \right) \left(\frac{P_S}{P_R} \right) A_{Ri} \right]^2} \quad (9)$$

and

$$\text{IA}_{AV} = \frac{1}{2} \sum_{i=1}^n \left[A_{Si} + \left(\frac{T_R}{T_S} \right) \left(\frac{L_S}{L_R} \right) \left(\frac{P_S}{P_R} \right) A_{Ri} \right] \quad (10)$$

The fractional analytical uncertainty is defined as

$$\text{FAU} = \frac{\text{NRMS}_E}{\text{IA}_{AV}} \quad (11)$$

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

$$RA_i = \frac{T_R L_S P_S RSA_i}{T_S L_R P_R ISC_i} \quad (12)$$

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

$$RI_k = \frac{T_R L_S P_S RSI_k}{T_S L_R P_R ISC_k} \quad (13)$$

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

$$FM_m = \frac{RMSS_m (FFU_m - FFL_m) AU_i DL_i}{AAI_i RSA_i} \quad (14)$$

for each analytical region associated with the analyte.

I.2.6 If only the m^{th} analytical region is used to calculate the concentration of the i^{th} analyte, set $FMU_i = FM_m$.

I.2.7 If a number of analytical regions are used to calculate the concentration of the i^{th} 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

$$FMU_i = \sum_{k \in \{m'\}} W_{ik} FM_k \quad (15)$$

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 $OFU_i = \text{MAX}\{FRU_i, FCU_i, FAU_i, FMU_i\}$ and $OCU_i = \text{MAX}\{RSA_i * OFU_i, MAU_i\}$.

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 procedure using another software package 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 GramsTM Software Procedure - GramsTM is a software package that displays and manipulates spectra from a variety of instrument manufacturers. This procedure assumes familiarity with basic functions of GramsTM.

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 P h a s e :
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).

$$\frac{RMSS_i \times n(FFU_i - FFL_i)}{A_{CTS-test}} \leq .05 \quad (16)$$

RMSS=RMSD in the i^{th} analytical region in subtracted result, test CTS minus CTS standard.

n =number of data points per cm^{-1} . Exclude zero filled points.

FFU_i &= FFL_i =The upper and lower limits (cm^{-1}), respectively, of the i^{th} analytical region.

$A_{test-CTS}$ =band area in the i^{th} analytical region of the test CTS.

D-3 EPA METHOD 25A

**EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER
NSPS TEST METHOD**

**METHOD 25A-DETERMINATION OF TOTAL GASEOUS ORGANIC
CONCENTRATION USING A FLAME IONIZATION ANALYZER**

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

1.2 Principle. A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

2. Definitions

2.1 Measurement Systems. The total equipment required for the determination of the gas concentration. The system consists of the following major subsystems:

2.1.1 Sample Interface. That portion of the system that is used for one or more of the following: sample acquisition, sample transportation, sample conditioning, or protection of the analyzer from the effects of the stack effluent.

2.1.2 Organic Analyzer. That portion of the system that senses organic concentration and generates an output proportional to the gas concentration.

2.2 Span Value. The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations. The span value is established in the applicable regulation and is usually 1.5 to 2.5 times the applicable emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100 percent of the recorder scale.

2.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

2.4 Zero Drift. The difference in the measurement system response to a zero level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

2.5 Calibration drift. The difference in the measurement system response to a midlevel calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair or adjustment took place.

2.6 Response Time. The time interval from a step change in pollutant

concentration at the inlet to the emission measurement system to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.

2.7 Calibration Error. The difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.

3. Apparatus.

A schematic of an acceptable measurement system is shown in Figure 25A-1. The essential components of the measurement system are described below:

3.1 Organic Concentration Analyzer. A flame ionization analyzer (FIA) capable of meeting or exceeding the specifications in this method.

3.2 Sample Probe. Stainless steel, or equivalent, three-hole rake type. Sample holes shall be 4 mm in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

3.3 Sample Line. Stainless steel or Teflon * tubing to transport the sample gas to the analyzer. The sample line should be heated, if necessary, to prevent condensation in the line.

3.4 Calibration Valve Assembly. A three way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are applicable.

3.5 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter should be heated to prevent any condensation.

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

3.6 Recorder. A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute, Note: This method is often applied in highly explosive areas. Caution and care should be exercised in choice of equipment and installation.

4. Calibration and Other Gases.

Gases used for calibrations, fuel, and combustion air (if required) are contained in compressed gas cylinders. Preparation of calibration gases shall be done according to the procedure in Protocol No. 1, listed in Citation 2 of Bibliography. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ± 2 percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval of the Administrator.

Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value. Organic compounds other than propane can

be used following the above guidelines and making the appropriate corrections for response factor.

4.1 Fuel. A 40 percent H₂/60 percent N₂ gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

4.2 Zero Gas. High purity air with less than 0.1 parts per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

4.3 Low-level Calibration Gas. An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value.

4.4 Mid-level Calibration Gas. An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value.

4.5 High-level Calibration Gas. An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value.

5. Measurement System Performance Specifications

5.1 Zero Drift. Less than ± 3 percent of the span value.

5.2 Calibration Drift. Less than ± 3 percent of span value.

5.3 Calibration Error. Less than ± 5 percent of the calibration gas value.

6. Pretest Preparations

6.1 Selection of Sampling Site. The location of the sampling site is generally specified by the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. The sample port shall be located at least 1.5 meters or 2 equivalent diameters upstream of the gas discharge to the atmosphere.

6.2 Location of Sample Probe. Install the sample probe so that the probe is centrally located in the stack, pipe, or duct and is sealed tightly at the stack port connection.

6.3 Measurement System Preparation. Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing the sample interface and the organic analyzer. Make the system operable.

FIA equipment can be calibrated for almost any range of total organics concentrations. For high concentrations of organics (>1.0 percent by volume as propane) modifications to most commonly available analyzers are necessary. One accepted method of equipment modification is to decrease the size of the sample to the analyzer through the use of a smaller diameter sample capillary. Direct and continuous measurement of organic concentration is a necessary consideration when determining any modification design.

6.4 Calibration Error Test. Immediately prior to the test series, (within 2 hours of the start of the test) introduce zero gas and high-level calibration gas at the calibration valve assembly. Adjust the analyzer output to the appropriate levels, if necessary. Calculate the predicted response for the low-level and

mid-level gases based on a linear response line between the zero and high-level responses. Then introduce low-level and mid-level calibration gases successively to the measurement system. Record the analyzer responses for low-level and mid-level calibration gases and determine the differences between the measurement system responses and the predicted responses. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and must be replaced or repaired prior to testing. No adjustments to the measurement system shall be conducted after the calibration and before the drift check (Section 7.3). If adjustments are necessary before the completion of the test series, perform the drift checks prior to the required adjustments and repeat the calibration following the adjustments. If multiple electronic ranges are to be used, each additional range must be checked with a mid-level calibration gas to verify the multiplication factor.

6.5 Response Time Test. Introduce Zero gas into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. Record the time from the concentration change to the measurement system response equivalent to 95 percent of the step change. Repeat the test three times and average the results.

7. Emission Measurement Test Procedure

7.1 Organic Measurement. Begin sampling at the start of the test period, recording time and any required process information as appropriate. In particular, note on the recording chart periods of process interruption or cyclic operation.

7.2 Drift Determination. Immediately following the completion of the test period and hourly during the test period, reintroduce the zero and mid-level calibration gases, one at a time, to the measurement system at the calibration valve assembly. (Make no adjustments to the measurement system until after both the zero and calibration drift checks are made.) Record the analyzer response. If the drift values exceed the specified limits, invalidate the test results preceding the check and repeat the test following corrections to the measurement system. Alternatively, recalibrate the test measurement system as in Section 6.4 and report the results using both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

8. Organic Concentration calculations

Determine the average organic concentration in terms of ppmv as propane or other calibration gas. The average shall be determined by the integration of the output recording over the period specified in the applicable regulation. If results are required in terms of ppmv as carbon, adjust measured concentrations using Equation 25A-1.

$$C_c = KC_{meas} \quad \text{Eq. 25A-1}$$

Where:

- C_c = Organic concentration as carbon, ppmv.
 C_{meas} = Organic concentration as measured, ppmv.
 K = Carbon equivalent correction factor.

- K = 2 for ethane.
- K = 3 for propane.
- K = 4 for butane.
- K = Appropriate response factor for other organic calibration gases.

9. Bibliography

1. Measurement of Volatile Organic Compounds-Guideline Series. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. EPA-450/2-78-041. June 1978. p. 46-54.
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3. Gasoline Vapor Emission Laboratory Evaluation-Part 2. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. EMB Report No. 75-GAS-6. August 1975.

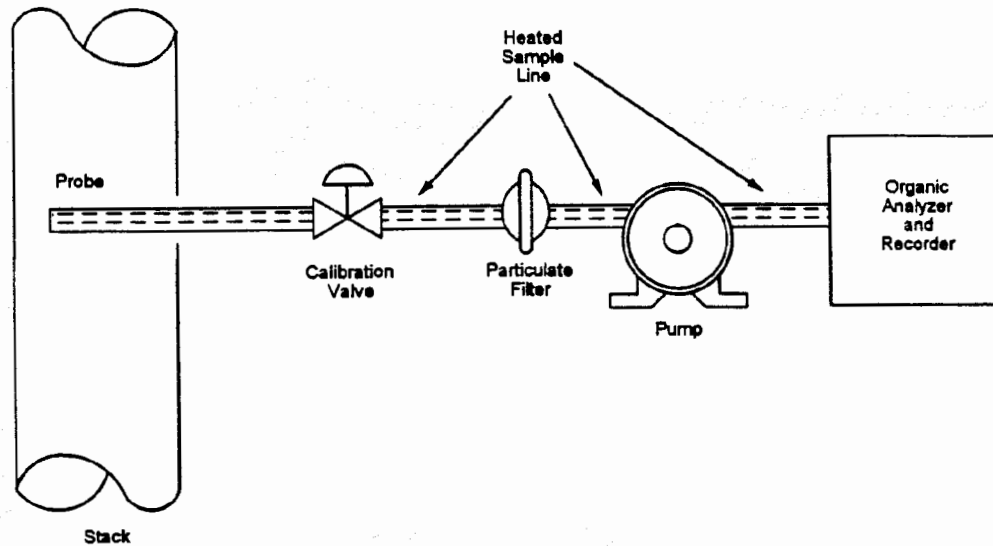


Figure 25A-1. Organic Concentration Measurement System.

D-4 EPA DRAFT METHOD 205

**EMISSION MEASUREMENT TECHNICAL INFORMATION CENTER
TEST METHOD**

DRAFT--DO NOT CITE OR QUOTE

The EPA proposes to amend Title 40, Chapter I, Part 51 of the Code of Federal Regulations as follows:

1. The authority citation for Part 51 continues to read as follows:
Authority: Section 110 of the Clean Air Act as amended. 42 U.S.C. 7410.
2. Appendix M, Table of Contents is amended by adding an entry to read as follows:

Method 205--Verification of Gas Dilution Systems for Field Instrument Calibrations

3. By adding Method 205 to read as follows:

**Method 205 - Verification of Gas Dilution Systems
for Field Instrument Calibrations**

1. INTRODUCTION

1.1 Applicability. A gas dilution system can provide known values of calibration gases through controlled dilution of high-level calibration gases with an appropriate dilution gas. The instrumental test methods in 40 CFR Part 60 -- e.g., Methods 3A, 6C, 7E, 10, 15, 16, 20, 25A and 25B -- require on-site, multi-point calibration using gases of known concentrations. A gas dilution system that produces known low-level calibration gases from high-level calibration gases, with a degree of confidence similar to that for Protocol¹ gases, may be used for compliance tests in lieu of multiple calibration gases when the gas dilution system is demonstrated to meet the requirements of this method. The Administrator may also use a gas dilution system in order to produce a wide range of Cylinder Gas Audit concentrations when conducting performance specifications according to Appendix F, 40 CFR Part 60. As long as the acceptance criteria of this method are met, this method is applicable to gas dilution systems using any type of dilution technology, not solely the ones mentioned in this method.

1.2 Principle. The gas dilution system shall be evaluated on one analyzer once during each field test. A precalibrated analyzer is chosen, at the discretion of the source owner or operator, to demonstrate that the gas dilution system produces predictable gas concentrations spanning a range of concentrations. After meeting the requirements of this method, the remaining analyzers may be calibrated with the dilution system in accordance to the requirements of the applicable method for the duration of the field test. In Methods 15 and 16, 40 CFR Part 60, Appendix A, reactive compounds may be lost in the gas dilution system. Also, in Methods 25A and 25B, 40 CFR Part 60, Appendix A, calibration with target compounds other than propane is allowed. In these cases, a laboratory evaluation is required once per year in order to assure the Administrator that the system will dilute these reactive gases without significant loss. **Note:** The laboratory evaluation is required only if the source owner or operator plans to utilize the dilution system to prepare gases mentioned above as being reactive.

2. SPECIFICATIONS

2.1 Gas Dilution System. The gas dilution system shall produce calibration gases whose measured values are within ± 2 percent of the predicted values. The predicted values are calculated based on the certified concentration of the supply gas (Protocol gases, when available, are recommended for their accuracy) and the gas flow rates (or dilution ratios) through the gas dilution system.

injection shall differ by more than ± 2 percent from the average instrument response for that dilution. **3.2.5** For each level of dilution, calculate the difference between the average concentration output recorded by the analyzer and the predicted concentration calculated in Section 3.2.2. The average concentration output from the analyzer shall be within ± 2 percent of the predicted value.

3.2.6 Introduce the mid-level supply gas directly into the analyzer, bypassing the gas dilution system. Repeat the procedure twice more, for a total of three mid-level supply gas injections. Calculate the average analyzer output concentration for the mid-level supply gas. The difference between the certified concentration of the mid-level supply gas and the average instrument response shall be within ± 2 percent.

3.3 If the gas dilution system meets the criteria listed in Section 3.2, the gas dilution system may be used throughout that field test. If the gas dilution system fails any of the criteria listed in Section 3.2, and the tester corrects the problem with the gas dilution system, the procedure in Section 3.2 must be repeated in its entirety and all the criteria in Section 3.2 must be met in order for the gas dilution system to be utilized in the test.

4. REFERENCES

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D-5 HCI VALIDATION PAPER

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Validation of EPA FTIR Method For Measuring HCl

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Introduction

In 1997 EPA is preparing to publish a sampling method (Draft Method 320)¹ based on the use of Fourier transform infrared (FTIR) spectroscopy to measure emissions of hazardous air pollutants (HAPs). This method establishes sampling procedures for measuring HAPs and employs analytical procedures in the EPA FTIR Protocol.²

In 1996 EPA conducted a field test at a source with HCl emissions. The test goal was to use the FTIR Draft Method 320 to measure vapor phase pollutants at this source. Measurements were conducted on the inlet and outlet of a control device. Hydrogen chloride (HCl) was a target pollutant for this source and, for this reason, some samples were spiked from a cylinder containing a standard concentration of 103 ppm HCl. Results of HCl measurements are presented along with a Method 301³ statistical analysis of spiked and unspiked samples, and a comparison of results obtained using EPA reference spectra and results obtained using spectra of the HCl gas standard to measure the sample concentrations.

Experimental

The source tested in this project was a coal burning process with a relatively low moisture content (3 to 4% by volume). Flue gas temperatures were between 400 and 500°F. The principal components of the gas stream were water vapor, CO₂, SO₂, and NO.

Sampling System

The sampling system is depicted in Figure 1. The sample was extracted through a 4-ft long, 0.5-in diameter stainless steel probe. Sample was transported through heated 3/8-in Teflon line using a KNF Neuberger heated head sample pump (Model NO35 ST.11I). A Balston particulate filter (holder Model Number 30-25, filter element Model Number 100-25-BH, 99 percent removal efficiency at 0.1 µm) was connected in-line at the outlet of the sample probe. The sample line was heat wrapped and insulated. Temperature controllers were used to monitor and regulate the sample line temperature at about 350° F. The stainless steel manifold contained 3/8-in tubing, rotameters and 4-way valves to monitor and control the sample flow to the FTIR gas cell. The manifold temperature was maintained between 300 to 310°F.

The FTIR system included an Analect instruments Model RFX-40 interferometer equipped with a broad band MCT detector. Samples were contained in an Infrared Analysis Model D22H variable path gas cell. The cell temperature was maintained at 250°F.

Sampling Procedure

A series of discreet batch samples was collected by filling the cell above ambient pressure and closing the inlet valve to isolate the sample. An outlet valve was briefly opened to vent the sample to ambient pressure. The spectrum of the static sample was recorded. Then the cell was evacuated for the next sample. Each spectrum consisted of 50 co-added scans. The minimum time between consecutive samples was about 2 minutes. Inlet and outlet runs were conducted at the same time: the two locations were sampled alternately with the one FTIR system. The minimum time between consecutive measurements was about 3 to 5 minutes.

Path Length Determinations

Two path lengths were used in this test. The cell was adjusted to 40 beam passes for the first two test runs and reduced to 20 beam passes for a third test run. The number of beam passes was measured by shining a He/Ne laser through the optical path and observing the number of laser spots on the field mirror. The path lengths in meters were determined by comparing CTS EPA reference spectra to the CTS spectra collected at each path length.

Absorption path lengths were determined from a comparison of the field test CTS spectra and EPA library CTS spectra of ethylene (C_2H_4). For high temperature spectra, the EPA library interferograms cts0115a.aif and bkg0115a.aif were de-resolved to the appropriate spectral resolution (either 1 or 2 cm^{-1}) according to the procedures of reference 2 (Appendix K). The same procedure was used to generate low-temperature spectra from the original interferometric data in the EPA library files cts0829a.aif and bkg0829a.aif. The resulting files were used in least squares fits to the appropriate field CTS spectra (see reference 2, Appendix H) in two regions (the FP, or "fingerprint" region from 790 to 1139 cm^{-1} and the CH, or "CH-stretch region" from 2760 to 3326 cm^{-1}). The fit results for each region, test, and set of test sampling conditions were averaged. They and their average uncertainties are presented in Table 1. The CH values were used in analytical region 4 where HCl was measured.

Analyte Spiking

Draft Method 320¹ contains a procedure for spiking the flue gas with one or more of the target analytes. The spike procedure closely follows Section 6.3 of reference 3. The primary purpose of analyte spiking is to provide a quality assurance check on the sampling system to determine if analyte losses occur in transport to the analyzer. A second purpose is to test the analytical program to verify that the analyte(s) can be measured in the sample matrix. If at least 12 (independent) spiked and 12 (independent) unspiked samples are measured then a Method 301 statistical analysis can be performed on the results to "validate" the method.

Figure 1 shows the sampling configuration used for the analyte spike. This procedure is described in detail elsewhere¹. In this test, a measured flow of the gas standard was preheated to the sample line temperature before being introduced into the extracted flue gas at the back of the probe. The spiked sample then passed through all of the sample components to the gas cell where the spectrum was recorded. A series of unspiked samples was measured, the spike was turned on and then a complete

series of spiked samples was measured. The spike then was turned off to make additional unspiked measurements. Ideally, the spike comprises 1/10 or less of the sample mixture. The dilution is estimated by comparing the spike flow to the total flow, but the actual dilution is determined measuring a tracer (SF_6) concentration in the spiked samples and comparing that to tracer concentration in the undiluted gas standard.¹

Usually the tracer is spiked with the analyte standard. In this test the SF_6 standard and HCl standard were contained in separate cylinders so the SF_6 was spiked first, then the HCl was spiked, and finally the SF_6 was spiked again. The total sample flow stayed constant during the entire sampling period. The spike flow was also held constant to insure that the dilution ratio was the same when the SF_6 was spiked as when the HCl was spiked.

Quantitative Analysis

FTIR analysis is performed in two steps: (1) collecting spectra of samples, and (2) analyzing the spectra to determine concentrations of detected compounds. The quantitative analysis step usually is performed with an automated program that relates sample absorbance intensities to absorbance intensities at known concentrations in reference spectra.² The Protocol² describes procedures for preparing reference spectra and Method 320¹ requires the analyst to use reference spectra prepared with the Protocol procedures. To date, the only existing set of reference spectra for HCl and most Clean Air Act HAPs is in the EPA FTIR spectral library (<http://info.arnold.af.mil/epa/welcome.htm>).

The Calibration Transfer Standard² is the key requirement in using reference spectra for quantitative analysis. CTS spectra help the analyst characterize differences in resolution, path length, temperature, and sample pressure between the instrument system used to collect reference spectra and the system used to collect the sample spectra. Table 1 illustrates how the CTS spectra were used to determine the optical path lengths for the system used in this test. The HCl reference spectra were de-resolved in the same way as the CTS reference spectra before they were used in the quantitative analysis.

References 4 through 8 comprise a thorough description of one technique for analyzing FTIR absorbance spectra. Two different analytical routines were used in this study. The first was prepared by Rho Squared using the programming language ARRAY BASIC™ (GRAMS,™ Version 3.02, Galactic Industries Corporation, Salem, New Hampshire). The "classical least squares" (CLS) or "K-Matrix" technique and the associated computer program "4FIT" are described in Reference 9. The terminology and basic analytical approach employed in this work are described in the "EPA FTIR Protocol" (Reference 2). The second routine used the K-matrix analytical program "Multicomp" version 6.0 (Analect Instruments).

The two analyses were performed independently by different analysts and then compared without modification.

Reference Spectra

The program "4FIT" used as input EPA FTIR library spectra of HCl de-resolved to 1 cm^{-1} and normalized for absolute temperature, concentration, and absorption path length. The resulting files were

averaged to provide a "reduced absorptivity" (see Reference 9), which was stored in the spectral file 097.alf and employed in all subsequent HCl analyses. The HCl analysis was applied to the de-resolved EPA library HCl spectra to determine the fractional calibration uncertainty (FCU), which is presented in Table 2.

During the test MRI recorded spectra of samples taken directly from an HCl cylinder standard (103 ppm HCl in nitrogen, $\pm 5\%$ accuracy from Scott Specialty Gases). Four independent HCl "calibration" spectra were measured at each of the two instrument configurations used to collect the data presented in Figures 2 and 3. The Fractional Calibration Uncertainty for each set of four spectra and the analytical region for the "Multicomp" analysis is presented in Table 2.

Even though the two sets of results are identified by the program names "4FIT" and "Multicomp," it is important to note that the "Multicomp" results were reproduced by the program "4FIT" when the HCl calibration spectra were used as input for "4FIT." Therefore, any differences in the analyses are not attributable to the programs, but to the use of different input spectra.

Results

HCl Concentrations

Table 3 summarizes results from the three test runs at the two locations. The agreement between the "4FIT" and the "Multicomp" analyses is very good except for the third run. This run was conducted after the path length had been decreased from 40 to 20 laser passes.

The two comparisons plotted in Figures 2 and 3 are indicated in Table 3. The Run 2 outlet results (Figure 2) are typical of those obtained for the Run 2 inlet results recorded on the same day and the Run 1 inlet and outlet results recorded a day earlier. The close agreement was typical also for two data sets collected at another field test in one test run. For 3 of the 6 data sets presented in Table 3, the results obtained with program "4FIT," using de-resolved EPA library reference spectra and the CTS-derived absorption path lengths, are nearly identical (within the 4σ uncertainty) to those obtained using "Multicomp," which employed the field HCl calibration standard spectra without an explicit absorption path length determination. The average percent difference of the Run 2 inlet results was slightly higher than the 4σ uncertainty, but this percent difference corresponded to an average difference of 1.7 ppm. The error bars in Figures 2 and 3 correspond to the 4σ statistical uncertainties in the "4FIT" HCl concentrations.

Method 301 Analysis

Tables 4 and 5 present the results of the method 301 statistical analysis of the spiked and unspiked "4FIT" and "Multicomp" Run 3 outlet results, respectively. Note that the nearly constant difference of about 19 percent in the two analyses has almost no effect on the Method 301 statistical analyses, which indicate no significant bias in the HCl measurements. This is because the statistical treatment analyzes differences between spiked and unspiked measurements and compares the differences to an expected value of the spike. Since the same offset is apparent in the "Multicomp" analysis of both the spiked and unspiked results, the calculated bias is not affected.

This is another indication that the difference in the "4FIT" and "Multicomp" run 3 results is not due to a measurement or analytical error. It is likely due either to an anomaly in the Run 3 path length determination for the CH stretch region or to an error associated with using the HCl "calibration spectra as input for the "Multicomp" program. As stated above, the "4FIT" program reproduced the "Multicomp" results when using the HCl "calibration" spectra as input.

Discussion

The uncertainties for the four data sets in Runs 1 and 2 are approximately equal to the small differences between the "4FIT" and "Multicomp" results. The excellent agreement of the two analyses is noteworthy for several reasons. HCl is notoriously difficult both in terms of sampling and data analysis, due (respectively) to the compound's high chemical reactivity and the details of the infrared spectrum which make the analysis susceptible to instrument resolution errors. The results also provide a direct comparison between two fundamentally different analytical approaches, one relying on *in situ* calibration of the instrument using actual calibration gas standards, and the other using the calibration transfer concept.

This comparison is somewhat clouded by the results depicted in Figure 3, which show the HCl concentration determined during Run 3 at the outlet. These are also typical of the results for another data set recorded on the same day at the inlet. Unlike the Runs 1 and 2 data, the Run 3 data indicate a statistically meaningful difference of approximately 18% between the "4FIT" and "Multicomp" results. We stress that this difference is **not** attributable to errors in the computer programs, which produced reliable results in these and many independent test cases. Rather, the difference seems to be related to an anomaly in the absorption path length determinations presented in Table 1. Note that the CTS-derived absorption path length for (nominally) 20 passes, corresponding to the Run 3 data, are 10.2 meters for the CH-stretch and "fingerprint" (FP) analytical regions. The difference between the CH and FP results is much larger for this particular day of testing than on the other two test days, represented in the table by the 16- and 40- pass results. (It is also anomalous with respect to results obtained using the same instrument in another field test completed within nine days of the testing addressed here.) Moreover, were the average of the CH and FP region values (12.2 meters) used for the HCl concentration values rather than the CH region value of 10.2 meters, the level of agreement between the two sets of analytical results for the Run 3 data would be comparable to that of the Run 1 and 2 data discussed immediately above.

We have attempted to determine the cause of this difference by considering a number of possible operational and instrumental problems. However, no single systematic effect seems sufficient. Because consistent path length determinations were carried out both before and after the HCl measurements in question, a sudden change in instrument performance must be ruled out. Gas pressure and dilution effects cannot cause the type of wavenumber-dependent effects observed in the CTS spectra; subsequent laboratory measurements of C_2H_4 indicated that temperature variations, like pressure and dilution effects, would lead to path length errors in the same direction for the CH and FP regions. Because the same EPA CTS ethylene spectra were used in all the path length determinations and led to excellent statistical results in all cases, potential data processing errors in the deresolution procedure are also insufficient to explain

the anomalous results. However, we note that the observed 18% discrepancy still allows high confidence in the data and the infrared technique, and the discrepancy is obvious mainly because of the overall high quality of the data set and statistical results.

Conclusions

The evaluation presented in this paper demonstrates that the EPA FTIR Protocol analytical procedures based on the use of laboratory reference spectra to determine analyte concentrations in sample spectra give excellent, and verifiable, results. This is true even for HCl, which is difficult to sample, and even when the reference spectra are deresolved to match the sample spectra.

Two independent analyses using different programs and different spectral input data were performed on 6 FTIR data sets collected at a site with HCl emissions. The alternate analyses produced nearly identical results in 4 of the data sets. In two of the data sets the agreement was also good, but the average discrepancy of about 18 percent between results produced by the alternate analyses was larger than the average measurement uncertainty of about 5.5 percent. A preliminary evaluation of this discrepancy has not determined the exact cause, but it is probably attributable to an anomaly in the measurement of the absorption path length for the one test run.

These results also demonstrate the need for careful instrument performance checks and preparation of library reference spectra. Strict QA/QC standard procedures are required to produce accurate measurements. The Method 301 validation results showed no significant bias in the FTIR measurements of HCl at this test, but the validation procedure cannot reveal a constant offset "error" that is applied equally to both spiked and unspiked samples.

Acknowledgments

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References

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Table 1. Pathlength Determination Results.

	CTS Conditions		CH region		FP region	
	# Passes	Temp (K)	Result (m)	% uncert.	Result (m)	% uncert.
	16	293	6.5	2.9	6.7	1.3
Run 3 (Figure 3)	20	293	11.0	2.6	11.3	1.6
	20	393	10.2	2.5	14.3	2.2
Run2 (Figure 2)	40	293	19.2	5.5	20.0	1.8
	40	393	20.2	2.6	23.4	1.6

Table 2. Fractional Calibration Uncertainties (FCU in Reference 2) For the Two Quantitative Analyses.

Compound	FCU (%)	Analytical Region (cm ⁻¹)
HCl "4fit"	4.6	2747 - 2848
HCl "Mcomp"		2569 - 2871
Run 2 *	1.05	
Run 3 *	3.14	

* Spectra of four samples from the cylinder standard (103 ppm HCl in nitrogen) were used in the "Mcomp" analysis. The spectra were measured at the same instrument configuration used in each run.

Table 3. Summary of results comparisons in 4 runs (8 data sets).

Data Set	Average "4FIT" Results		Average "Multicomp" Result		No. of Results ³
	HCl ppm	% 4 * σ ¹	HCl ppm	% Difference ²	
Run 1 Inlet	43.3	3.9	42.1	2.9	36
Run 1 Outlet	34.5	4.1	32.9	4.4	30
Run 2 Inlet	14.8	7.7	13.1	11.8 ⁴	16
Run 2 Outlet (Figure 2)	48.0	4.5	46.4	3.2	33
Run 3 Inlet	62.5	5.6	50.9	18.6	41
Run 3 Outlet (Figure 3)	58.0	5.5	47.3	18.4	52

1 - Average percent uncertainty in the 4FIT results.

2 - Equals (4FIT-Multicomp)/4FIT.

3 - Equals the number of spectra included in the average. Results from condenser and ambient air samples were not included in the averages.

4 - Flow restriction during this run may have caused HCl losses resulting in lower measured concentrations for this run. An average difference of 1.7 ppm corresponded to a relatively large percent difference of 11.8 % on the smaller average concentration for this run.

Table 4. Method 301 statistical analysis of "4FIT" HCl results in Figure 3.

Unspiked				Spiked		
	HCl ppm	d_i	$(d_i)^2$	HCl ppm	d_i	$(d_i)^2$
Run Average =	57.18 *	9.68	52.561	62.14 *	4.74	25.784
Statistical Results	SD =	2.093		SD =	1.466	
	F =	0.491		SD _{pooled} =	1.807	
	RSD=	3.7				
	Bias =	-0.088		Exp Conc =	5.05	
	t =	0.12		CF =	1.02	

* Represents the average result in 12 unspiked or spiked samples. Statistical variables are described in Section 6.3 of EPA Method 301.³ Procedure for determining spiked dilution factor and expected concentration, Exp Conc, is described in reference 10.

Table 5. Summary of Method 301 statistical analysis of "Multicomp" results in Figure 3.

Unspiked				Spiked		
	HCl ppm	d_i	$(d_i)^2$	HCl ppm	d_i	$(d_i)^2$
Run Average =	45.88 *	8.62	34.242	50.86 *	3.51	21.496
Statistical Results	SD =	1.689		SD =	1.338	
	F =	0.628		SD _{pooled} =	1.524	
	RSD=	3.7				
	Bias =	-0.070		Exp Conc =	5.05	
	t =	0.11		CF =	1.01	

* Represents the average result in 12 unspiked or spiked samples. Statistical variables are described in Section 6.3 of EPA Method 301.³ Procedure for determining spiked dilution factor and expected concentration, Exp Conc, is described in reference 10.

Figure 1. Extractive sampling system.

