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Office of Air Quality
Planning and Standards
Research Triangle Park, NC 27711

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Air

LIME MANUFACTURING EMISSIONS TEST REPORT (FOURIER TRANSFORM INFRARED SPECTROSCOPY)

FINAL REPORT

Chemical Lime Company
(Formerly APG Lime Company)
Ripplemead, Virginia

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Ripplemead, Virginia

Prepared for

Emission Measurement Center
United States Environmental Protection Agency
Research Triangle Park, North Carolina 27711

Attn: Michael L. Toney

EPA Contract No. 68-D-98-027
Work Assignment 2-11
MRI Project No. 104951-1-015-05

September 30, 1999

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. 2-11. Mr. Micheal Toney is the EPA Work Assignment Manager (WAM). Dr. Thomas Geyer is the MRI Work Assignment Leader (WAL). The field test was performed, and draft and revised test reports were submitted under EPA Contract No. 68-D2-0165, Work Assignment No. 4-01. Mr. Dennis Holzschuh and Michael Toney were the EPA WAMs for the Emission Measurement Center (EMC) under Work Assignemnt 4-01.

This report presents the procedures, schedule, and test results for an emissions test performed at APG Lime Company in Ripplemead Virginia. The field test was conducted in October, 1996. The draft and revised test reports were submitted in January and September 1997, respectively. MRI performed FTIR emissions measurements at the inlet and outlet of a baghouse control device and at the cooling stack using EPA Method 320. Method 320 has since been promulgated in the Federal Register on May 19, 1999. The report has been updated, but no changes have been made to the results since the draft reports.

This report consists of one volume (250 pages) with six sections and five appendices.

Midwest Research Institute

for Andrew Trenholm
Deputy Program Manager

Approved:

for Jeff Shular
Director, Environmental Engineering Division

September 30, 1999

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1.0 INTRODUCTION

1.1 BACKGROUND

The Emission Measurement Center (EMC) of the U. S. EPA received a request from the Minerals and Inorganic Chemicals Group (MICG) of the U.S. EPA to perform emissions testing at coal-fired lime kilns. In partial fulfillment of the test request EMC issued Work Assignments 2-11 and 4-01 under EPA Contract Nos. 68-D-98-027 and 68-D2-0165, respectively, to Midwest Research Institute (MRI). The purpose of this project was to measure organic and inorganic hazardous air pollutants (HAPs) using a test method based on Fourier transform infrared (FTIR) spectroscopy. This report describes the test procedures and presents results of the testing at the Chemical Lime Company's (formerly APG Lime Company) plant in Ripplemead, Virginia.

1.2 PROJECT SCOPE

Three locations were tested at APG: the inlet and outlet of a baghouse controlling particulate emissions off of the kiln, and the stack from the cooler where the product is cooled.

The procedures followed in this test are described in EPA FTIR Method 320 and the EPA FTIR Protocol (Appendix D and references 1 and 2). The objectives of the field test were to: (1) screen for hazardous air pollutants (HAPs) regulated in Title III of the 1990 Clean Air Act Amendments, (2) measure, if detected, compounds that have been previously measured at cement kilns (e.g., formaldehyde, naphthalene, p-xylene), and (3) measure other pollutants such as SO_2 and NO_x.

The test request specifically identified HCl as a target analyte. This facility used coal as the fuel to fire the kiln, and HCl has been measured with FTIR methods at other coal-burning

facilities. Method 320[1] uses an analyte spiking procedure for quality assurance or Method 301 validation to verify that the sampling system is suitable for measuring target analyte(s) at the expected concentration. In this test, analyte spiking was performed using an HCl cylinder standard from Scott Specialty Gases.

In the FTIR screening procedure, spectra of gas samples contained in a leak tight infrared gas cell are recorded at regular intervals over a sampling run. Typically, 8 to 10 sample spectra are recorded per hour. These spectra are then analyzed using reference spectra in the EPA FTIR spectral library to detect and measure any HAPs in the sample. Unidentified spectral features are analyzed to check for the presence of other compounds for which there may currently be no reference spectra.

1.3 PROJECT PERSONNEL

This project was administered by the Emission Measurement Center (EMC) of the U.S. EPA. The Test Request was initiated by the Minerals and Inorganic Chemicals Group (MICG) of the Office of Air Quality and Standards (OAQPS). Key project personnel are listed in Table 1-1. Environmental Testing Services, Inc. (ETS) and Envirostaff, Inc., assisted assist MRI in the field test. Dr. Grant Plummer of Rho Squared assisted in analyzing the data.

TABLE 1-1. PROJECT CONTACTS

APG Lime Co.	Ronald E. Bonnema	(540) 626-7163
EMC Work Assignment Managers	Michael Toney	(919) 541-5247
MRI WAL	Thomas Geyer	(919) 851-8181 Ex. 3120

2.0 TEST LOCATIONS

The information in this Section is summarized from material provided by Pacific Environmental Services, Inc. (PES). Figures 2-1 through 2-3 were also prepared by PES.

APG Lime Company has three coal-fired rotary kilns. The emissions from each kiln are controlled by a fabric filter baghouse. Each kiln has a cooler exhaust stack that is uncontrolled. Testing was performed at the No. 2 cooler stack and at the No. 1 kiln baghouse inlet and outlet.

2.1 NO. 2 COOLER STACK

The sampling ports were in a (71.25-in. ID) round, vertical stack, 192 in. upstream and 372 in. downstream of the nearest flow disturbances. The sampling location was indoors just above floor level. The test location was within 100-ft of the where the FTIR trailer was parked. The FTIR sampling ports were about 2-ft below the manual test ports shown in Figure 2-1.

2.2 No. 1 KILN BAGHOUSE INLET

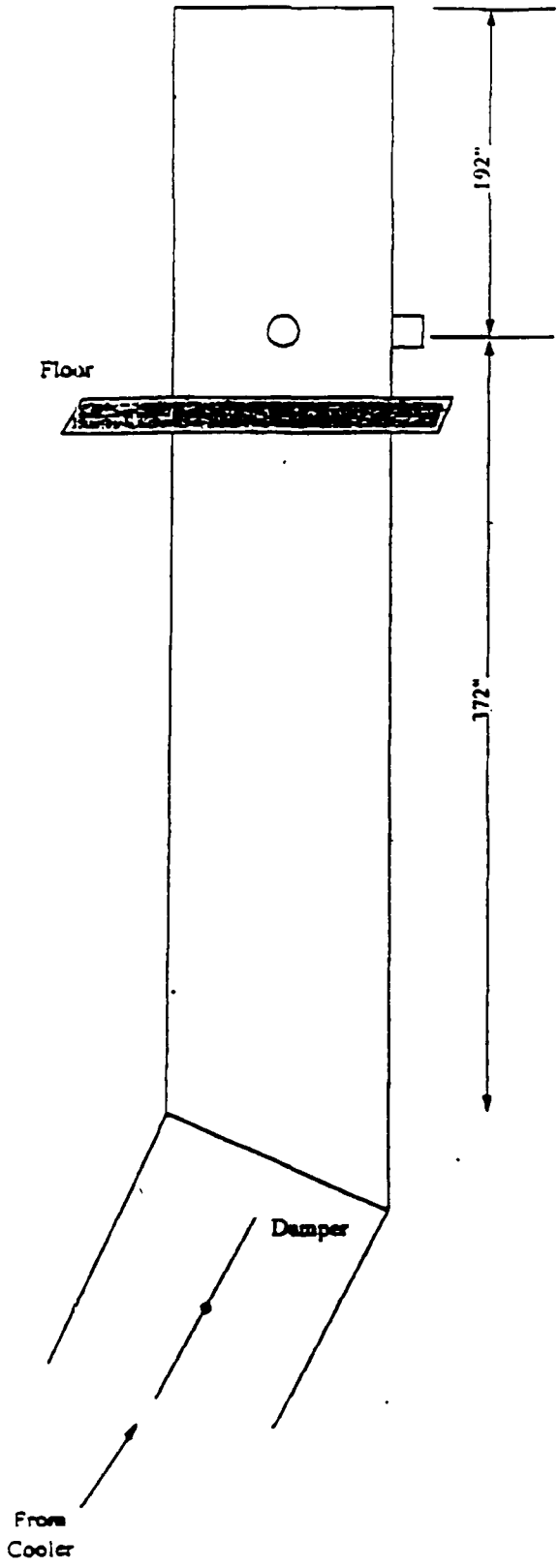
The inlet sampling ports were in a round horizontal duct (72 in ID) 47 ft downstream of the nearest flow disturbance and about 9 ft upstream of the nearest flow disturbance. The FTIR sampling ports were about 5 ft downstream of the manual test ports shown in Figure 2-2. Access was provided by scaffolding that was erected prior to the test.

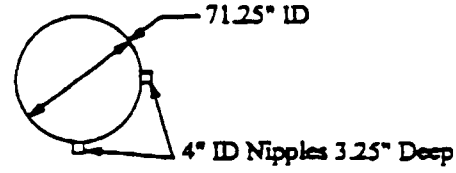
The baghouse inlet location was within 100 ft of the FTIR trailer location.

2.3 No. 1 KILN BAGHOUSE OUTLET

The outlet sampling ports were in a 72.25 in. ID round vertical stack, 390 in. downstream of the nearest flow disturbance and 390 in. before the stack exit. The FTIR sampling port was one of the four (A, B, C, and D) shown in Figure 2-3 (in the same plane and 90° apart).

The sampling location was within 100 ft of the FTIR trailer location.



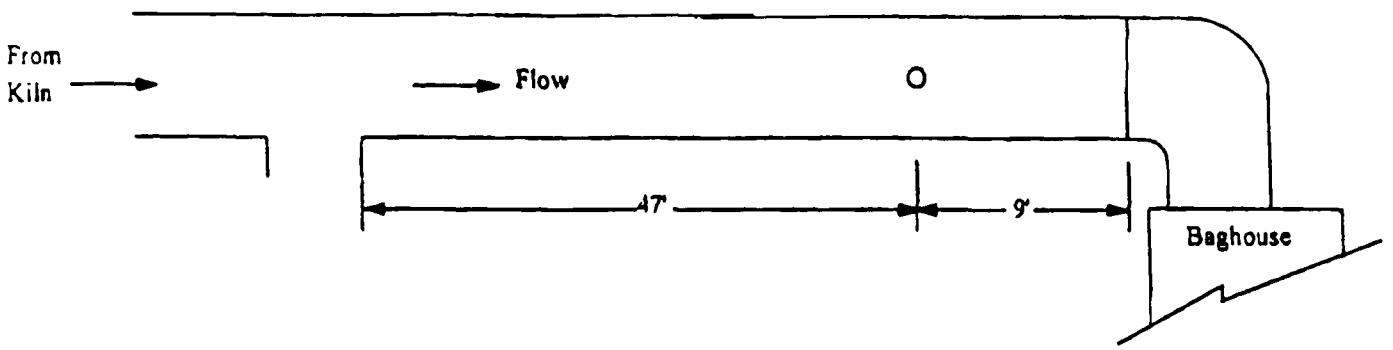


Cross Section

SAMPLE TRAVERSE POINT LOCATIONS

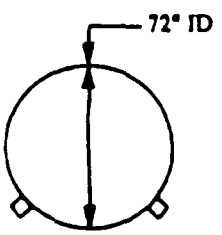
Point Number	Fraction of Stack ID	Distance Inches	Port Depth Inches	Port Location Inches
1	.021	1.50	3.25	4.75
2	.067	4.75	3.25	8.00
3	.118	8.44	3.25	11.69
4	.177	12.63	3.25	15.88
5	.250	17.81	3.25	21.06
6	.356	25.38	3.25	28.63
7	.644	45.88	3.25	49.13
8	.750	53.44	3.25	56.69
9	.823	58.63	3.25	61.88
10	.882	62.81	3.25	66.06
11	.933	66.50	3.25	69.75
12	.979	69.75	3.25	73.00

Figure 2-1. No. 2 kiln cooling stack location.



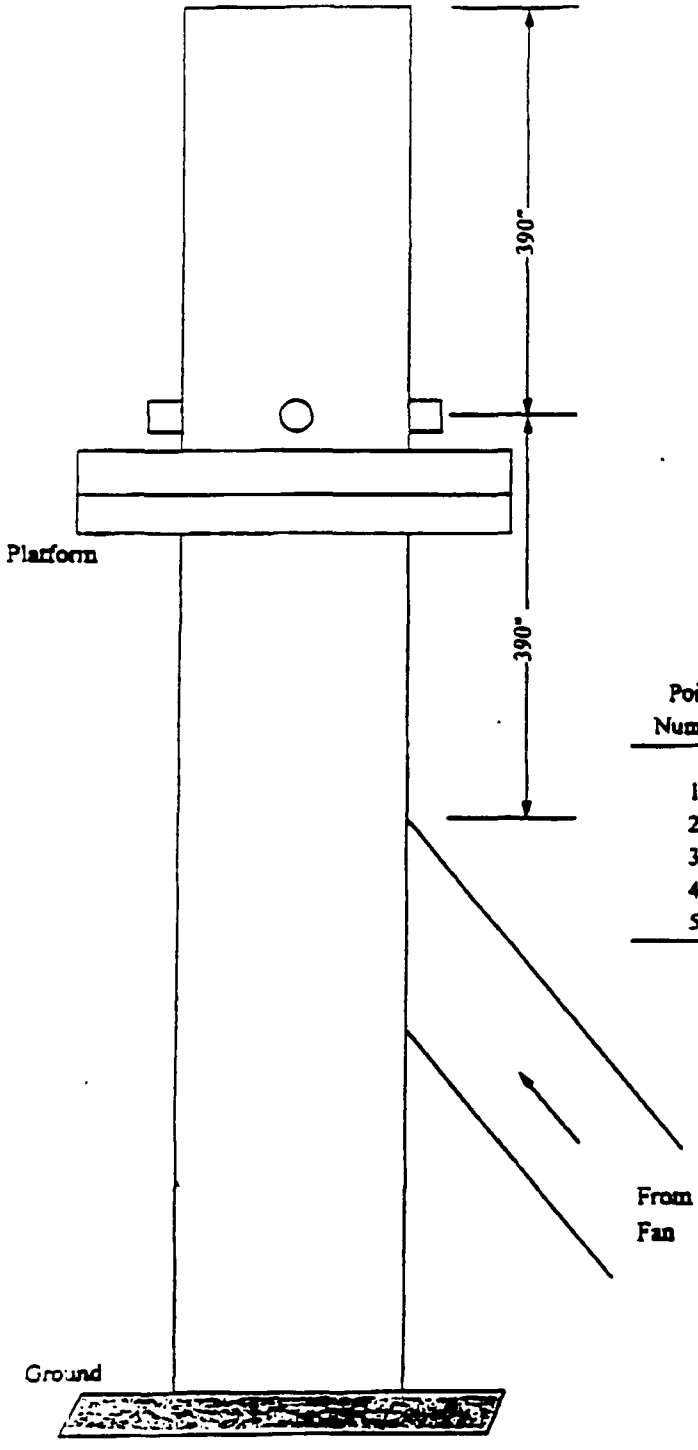
SAMPLE TRAVERSE POINT LOCATIONS

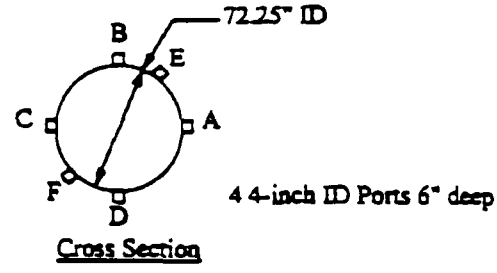
Point Number	Fraction of Stack ID	Distance Inches
1	.026	1.9
2	.082	5.9
3	.146	10.5
4	.226	16.3
5	.342	24.6
6	.658	47.4
7	.774	55.7
8	.854	61.5
9	.918	66.1
10	.974	70.1



Cross-Section

Figure 2-2. Kiln No. 1 baghouse inlet location.





SAMPLE TRAVERSE POINT LOCATIONS

Point Number	Fraction of Stack ID	Distance Inches	Port Depth Inches	Port Location Inches
1	.026	1.88	6.00	7.88
2	.082	5.94	6.00	11.94
3	.146	10.56	6.00	16.56
4	.226	16.31	6.00	22.31
5	.342	24.69	6.00	30.69

Figure 2-3. Kiln No. 1 baghouse outlet location.

3.0 RESULTS

3.1 TEST SCHEDULE

A summary of the test schedule is given in Table 3-1 at the back of this section. The FTIR test runs were coordinated with manual and Method 25A testing conducted by Pacific Environmental Services (PES). Process conditions were monitored by Research Triangle Institute (RTI). The schedule also shows the test runs where analyte spiking was performed. A complete record of the FTIR sampling is in Appendix B.

3.2 COOLING STACK

Table 3-2 shows the analytical results for HCl, SF_6, and CO and the deviation from 100 percent recovery of the HCl spike.

3.3 BAGHOUSE INLET

The primary emissions from the baghouse inlet were water vapor, CO_2, HCl, SO_2, and CO. The spectra were also analyzed for NO and NO_2. The NO_x compounds were difficult to measure because of moisture interference, except in the samples treated with the condenser. Tables 3-3 to 3-5 present the ppm results for the analysis of HCl, SO_2, SF_6 and CO. Figures 3-1 to 3-6 present the same results graphically.

The sample flow rate during Run 1 was very consistent. But during Run 2 the flow steadily decreased as the run progressed until the sampling rate was only about 1 to 2 LPM for the latter part of the run. This is the reason for the lower HCl concentrations measured at the inlet during Run 2. (It is important to maintain sample flow rate of at least 5 lpm or HCl can be reduced in a moist sampling system.) Additionally, with such a low flow rate, it is difficult to fill the FTIR cell and maintain a positive flow out the manifold vent to prevent air from diluting the sample back through the vent. The particulate filter was replaced during Run 1, but there was not enough particulate collected to cause the severe flow restriction. During Run 3 the flow also

decreased and a block was removed from near the back of the probe just upstream of the particulate filter. After this block was removed the flow was again very consistent for the remainder of Run 3.

3.4 BAGHOUSE OUTLET

Tables 3-6 to 3-8 present the measured concentrations for HCl, SO_2, SF_6, and CO at the baghouse outlet. The results are presented graphically in Figures 3-7 to 3-12. The gas stream composition was very similar to the inlet.

Except for Run 2 there is good agreement between the measured HCl concentrations at the inlet and outlet. The flow restriction at the inlet during Run 2 may have introduced a low bias in the Run 2 HCl concentrations. Sample flow at the outlet was very stable during all three runs.

Additionally, the magnitude and period of the variation in flue gas HCl concentrations was very similar in the spiked and unspiked portions of the test runs.

3.5 ANALYTE SPIKING AND VALIDATION OF HCl

The inlet sample was spiked with the HCl gas standard for part of Run 1. Table 3-9 presents an EPA Method 301 (reference 5) analysis of the spiked and unspiked results. The statistical analysis gave a t-statistic that meets Method 301 criteria for using the method without correction (the calculated correction factor was nearly 1). The spiked and unspiked measurements were not synchronized, but the stack gas HCl concentration was consistent enough to allow the comparison.

Sample from the outlet was spiked with HCl standard during parts of Runs 2 and 3. A Method 301 analysis of the spiked and unspiked HCl measurements is presented in Tables 3-10 and 3-11 for Runs 2 and 3, respectively. The statistical analysis of the Run 2 results gave a t-statistic outside of Method 301 criteria. The calculated correction factor for Run 2 was nearly 1.5, which also exceeds the Method 301 requirement. However, the analysis of the Run 3 spike results gave a t-statistic within Method 301 criteria (the calculated correction factor was nearly 1).

Statistically, the reason for the "failure" of the Run 2 spiked results is that the unspiked concentrations varied over a greater range than the spiked results. This can happen when the

spiked and unspiked measurements are made sequentially and the flue gas analyte concentration varies with time. For most of the testing, however, the HCl concentrations were relatively consistent and the Run 1 inlet and Run 3 outlet results passed the Method 301 criteria.

TABLE 3-1. SCHEDULE OF FTIR TESTING AT APG

Date	Time (Bkg & Cals)	Time (sampling)	Cooling stack	Kiln No 1. baghouse inlet	Kiln No 1. baghouse outlet
10/21/96	951-1159		Background, Calibrations, and leak check		
		1202-1204	Cooling stack APG		
	1209		Background		
		1212-1257	Cooling stack		
	1303		Background		
	1315		started SF6 spike		
		1324-1333	SF6 @ 0.48lpm (Cooling stack)		
		1343	started HCl spike		
		1353-1444	Spiked w/HCl (Cooling stack)		
	1452-1502		HCl 103 ppm flowing through cell		
	1507-1512		SF6 4.01 ppm in cell		
	1515-1853		Background and Calibrations		
10/22/96	840-900		Background and Calibrations		
		1030			Probe in outlet
	1048				Dry N2
		1050		Probe in inlet	
	1051-1056			Calibrations	
		1057-1434		Baghouse inlet	Baghouse outlet
		1438		SF6 spike on	
		1439-1500		SF6 spike	Unspiked
		1504		started HCl spike	
		1504-1739		HCl spike	Unspiked
		1740		HCl spike off	
		1741-1806		SF6 spike	
		1813-1839		ambient sample	ambient sample
	1844-1847				SF6 spike
		1855-1857			ambient air through probe
		1900-1901		ambient air through probe	
	1907-1937			Background and calibrations	

TABLE 3-1. (continued)

Date	Time (Bkg & Cals)	Time (sampling)	Cooling stack	Kiln No 1. baghouse inlet	Kiln No 1. baghouse outlet
10/23/96		850			ambient air sample
	854-1020				Background, calibration, and leak check
		1028-1036			ambient through probe
		1037-1039		ambient air	
		1040			Outlet probe in stack
		1045		Inlet probe in stack	
	1052-1057			Calibrations	
		1059-1301			Outlet H/W
		1307			SF6 spike to outlet
		1310-1316		Inlet flowing sample	
		1317-1327		SF6 spike	
		1330-1336		Inlet flowing	
		1331			HCl spike on to outlet
		1337			Evacuated cell and jumper line
		1339-1534		Spiked	Spiked
		1538			HCl spike off, SF6 spike on
		1539-1602		Inlet flowing	Spiked
		1603			SF6 spike off
		1605-1610		ambient air through inlet probe	
		1612-1613			H/W outlet
		1617			Outlet probe out of stack
	1625-1633				Background and calibrations
		1637-1715		ambient air samples	ambient air samples
10/23/96		1720			started N2 purge through both sample lines

TABLE 3-1. (continued)

Date	Time (Bkg & Cals)	Time (sampling)	Cooling stack	Kiln No 1. baghouse inlet	Kiln No 1. baghouse outlet
10/24/96	838-1106				Background, calibrations, and leak check
		1108-1415		Unspiked	Unspiked
		1416-1437		Unspiked	spiked w/SF6
		1441			started HCl spike
		1448-1619		Unspiked	Spiked w/HCl
		1621			HCl spike off
		1623-1624		Unspiked	
		1628-1629			SF6 spike
		1632-1645		Unspiked	Spiked
		1647			SF6 spike off
		1650-1957		Unspiked	Unspiked
	1959				Background
		2003-2018		ambient air sample	ambient air sample
	2023-2031				Background and calibrations
		2034-2041		ambient air sample	ambient air sample
	2049-2212				Background and calibrations

TABLE 3-2 FTIR RESULTS FROM THE APG COOLING STACK, 10/21/96.

File name [1]	Time	HCl ppm	4 * σ	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr
COOLH001	12:03	0.000	0.493	0.000	0.006	9.6	4.24	2.42E-01
COOLH002	12:11	0.000	0.480	0.009	0.006	10.5	4.71	2.64E-01
COOLC003	12:16	0.000	0.501	0.000	0.007	16.4	6.02	4.13E-01
COOLC004	12:21	0.000	0.525	0.000	0.007	16.4	6.51	4.12E-01
COOLH005	12:25	0.000	0.517	0.000	0.006	11.2	5.67	2.83E-01
COOLH006	12:30	0.000	0.540	0.000	0.006	11.4	5.63	2.87E-01
COOLH007	12:34	0.000	0.515	0.000	0.007	15.9	6.34	4.00E-01
COOLH008	12:40	0.000	0.533	0.000	0.006	18.9	6.01	4.75E-01
COOLH009	12:43	0.000	0.532	0.000	0.006	9.3	5.53	2.35E-01
COOLH010	12:48	0.000	0.545	0.000	0.007	9.4	5.82	2.36E-01
COOLH011	12:51	0.000	0.508	0.000	0.006	9.9	5.87	2.49E-01
COOLH012	12:54	0.000	0.533	0.000	0.006	9.4	5.88	2.36E-01
COOLS013	13:24	0.000	0.578	**0.151**	**0.011**	21.7	6.02	5.70E-01
COOLS014	13:27	0.000	0.663	**0.156**	**0.009**	14.8	6.06	3.87E-01
COOLS015	13:31	0.000	0.825	**0.153**	**0.011**	15.9	6.19	4.16E-01
COOLS016	13:53	**0.000**	**0.503**	0.000	0.005	13.9	5.31	3.64E-01
COOLS017	14:21	**1.447**	**0.529**	0.000	0.006	15.8	6.60	4.13E-01
COOLS019	14:28	**2.643**	**0.570**	0.000	0.007	12.6	7.28	3.31E-01
COOLS020	14:34	**3.148**	**0.553**	0.000	0.007	12.7	7.24	3.32E-01
COOLS021	14:40	**3.572**	**0.561**	0.000	0.007	12.9	7.43	3.39E-01
HCl expected =	3.94			0.153	= SF_6 average concentration			3.44E-01
HCl spike averag	3.12			4.01	= SF_6 standard concentration			
percent deviatior	-20.71%			26.17	= dilution			

1 - File name: "COOL" cooling stack; "H" untreated; "S" spiked (bold indicates with HCl or with SF_6);
"4*sigma" - estimated uncertainty.

TABLE 3-3 FTIR RESULTS FROM THE APG BAGHOUSE INLET, RUN 1, 10/22/96.

File name[1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGIH001	11:08	192.2	5.2	102.9	0.000	0.028	1198.8	284.9	280.8	6.4	0.9	2.0
BAGIH002	11:13	203.2	5.3	108.8	0.000	0.028	1266.2	274.5	296.5	7.3	0.9	2.3
BAGIH003	11:28	214.6	5.6	114.9	0.000	0.030	1263.0	287.2	295.8	26.0	1.4	8.0
BAGIH004	11:31	214.2	5.5	114.7	0.000	0.029	1241.1	283.9	290.6	26.8	1.4	8.2
BAGIH005	11:46	218.7	5.6	117.1	0.000	0.030	1194.5	312.0	279.7	25.0	1.3	7.7
BAGIH006	11:49	221.7	5.7	118.7	0.000	0.030	1279.9	278.8	299.7	28.8	1.5	8.9
BAGIH007	12:14	221.9	5.8	118.8	0.000	0.031	1260.3	286.1	295.2	24.7	1.3	7.6
BAGIH008	12:19	221.3	6.0	118.5	0.000	0.032	1363.9	305.7	319.4	30.3	1.4	9.3
BAGIC009	12:35	160.6	4.0	86.0	0.000	0.021	1421.7	330.3	333.0	2.7	0.5	
BAGIC010	12:39	158.1	4.0	84.6	0.000	0.021	1294.3	255.9	303.1	1.6	0.5	
BAGIF011	13:10	232.6	6.2	124.5	0.000	0.033	1324.9	276.7	310.3	38.1	1.6	11.7
BAGIF012	13:17	238.0	6.3	127.4	0.000	0.034	1302.5	265.7	305.0	38.8	1.7	11.9
BAGIF013	14:05	243.3	6.5	130.3	0.000	0.034	1323.8	263.8	310.0	42.8	1.7	13.2
BAGIH014	14:10	240.8	6.3	128.9	0.000	0.033	1452.4	371.2	340.1	36.8	1.5	11.3
BAGIF015	14:27	213.5	6.2	114.3	0.000	0.033	798.0	135.8	186.9	41.1	1.6	12.6
BAGIH016	14:33	163.6	5.8	87.6	0.000	0.031	282.7	51.0	66.2	40.5	1.5	12.4
BAGIS017	14:51	81.0	4.8	54.4	**0.814**	**0.026**	39.1	16.9	11.5	27.5	1.1	10.6
BAGIS018	14:54	86.9	5.0	58.4	**0.831**	**0.026**	22.5	17.6	6.6	27.8	1.1	10.7
BAGIS019	14:59	190.0	5.4	127.7	**0.818**	**0.029**	1149.7	274.2	338.2	30.2	1.2	11.7
BAGIS020	15:17	175.6	5.4	118.1	0.029	0.029	1221.5	256.1	359.3	**49.8**	**1.7**	
BAGIS021	15:20	174.7	5.3	117.4	0.000	0.028	1240.4	263.6	364.8	**49.8**	**1.7**	
BAGIS022	15:25	175.8	5.5	118.2	0.000	0.029	1027.6	232.0	302.3	**53.9**	**1.8**	
BAGISF23	15:32	177.5	5.6	119.3	0.000	0.030	1048.4	237.3	308.4	**58.1**	**2.0**	
BAGISF24	15:39	163.1	5.5	109.6	0.000	0.029	762.1	139.3	224.2	**57.5**	**2.0**	
BAGISF25	15:45	167.5	5.5	112.6	0.000	0.029	966.5	225.9	284.3	**58.6**	**2.0**	

1 - File name: "BAGI" baghouse inlet; "H" untreated; "F" flowing; "S" spiked (bold indicates SF_6 or HCl); "A" ambient air through the sampling system; "C" condenser sample. "4*sigma" - estimated uncertainty.

TABLE 3-3 CONTINUED

File name[1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGIS026	15:54	166.3	5.5	111.8	0.000	0.029	1363.7	295.8	401.1	**46.7**	**1.5**	
BAGIS027	16:04	164.2	5.6	110.4	0.000	0.030	1634.6	437.3	480 8	**49.0**	**1.7**	
BAGIS028	16:14	141.4	5.4	95.1	0.000	0.029	432.1	76.7	127 1	**53.4**	**1.8**	
BAGIS029	16:24	199.1	5.5	133.9	0.000	0.029	1078.6	249.6	317.3	**56.1**	**1.9**	
BAGIS030	16:33	79.6	5.1	53.5	0.000	0.027	53.4	20.1	15.7	**54.9**	**1.9**	
BAGIS031	16:42	109.0	5.1	73.3	0.000	0.027	28.6	19.2	8.4	**58.4**	**2.0**	
BAGIS032	16:51	191.5	5.5	128.8	0.000	0.029	1199.7	284.1	352.9	**57.8**	**2.0**	
BAGIS033	17:01	185.5	5.5	124.7	0.000	0.029	1288.7	284.0	379.1	**54.4**	**1.8**	
BAGIS034	17:10	138.7	5.3	93.2	0.000	0.028	245.1	48.4	72.1	**57.9**	**2.0**	
BAGIS035	17:18	138.0	5.5	92.8	0.000	0.029	423.7	77.0	124.6	**55.5**	**1.9**	
BAGIS036	17:27	211.2	5.7	142.0	0.000	0.030	1216.6	276.8	357.9	**58.0**	**2.0**	
BAGIS037	17:38	126.8	5.4	85.3	0.000	0.028	140.2	36.5	41.2	**55.1**	**1.8**	
BAGIS038	17:55	182.7	5.2	122.9	**0.814**	**0.028**	1028.5	221.0	302.5	28.7	1.0	11.1
BAGIS039	18:00	183.2	5.5	123.2	**0.807**	**0.029**	1235.5	274.1	363.4	31.7	1.1	12.2
BAGIS040	18:04	171.0	5.6	114.9	**0.820**	**0.030**	1616.4	446.4	475.4	29.3	1.1	11.3
BAGIA041	18:14	0.0	1.8		0.015	0.009	62.4	13.9		5.8	0.5	
BAGIA042	18:18	0.0	1.8		0.014	0.009	0.0	8.2		3.7	0.5	
BAGIA043	18:28	0.0	1.9		0.017	0.010	0.0	8.7		2.5	0.5	
BAGIA044	18:37	0.0	2.2		0.017	0.012	0.0	9.1		1.6	0.5	
BAGIA045	19:00	0.0	2.7		0.022	0.014	0.0	8.9		0.7	0.5	
Average lbs per hour =				108.5					263.3			9.7
		Average SF_6 =			0.817	0.028	3.43%					
		Standard SF_6 =			4.01				DSCFM =	51576		
		dilution =			4.906				% moisture =	3.9		

1 - File name: "BAGI" baghouse inlet; "H" untreated; "F" flowing; "S" spiked (bold indicates SF_6 or HCl); "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-4 FTIR RESULTS FROM THE APG BAGHOUSE INLET, RUN 2, 10/23/96.

File name [1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGIA201	10:38	0.0	14.4		11.2	8.7		1.95	0.44	
BAGIH202	11:06	262.8	6.2	148	1227.7	231.1	302.1	8.84	0.93	2.86
BAGIH203	11:16	265.9	6.1	150	1181.9	241.0	290.9	11.09	1.02	3.58
BAGIH204	11:29	271.8	5.9	153	1187.9	233.1	292.3	13.46	1.04	4.35
BAGIH205	11:41	266.9	5.8	150	1084.5	210.3	266.9	18.58	1.13	6.00
BAGIH206	12:10	255.9	5.7	144	938.8	150.3	231.0	11.49	0.89	3.71
BAGIH207	12:25	261.8	6.0	147	1093.0	224.0	269.0	18.25	1.07	5.90
BAGIF208	13:02	276.6	6.3	156	1128.5	251.6	277.7	12.10	0.95	3.91
BAGIF209	13:13	268.9	6.2	151	1244.5	333.8	306.3	6.57	0.81	2.12
BAGIF210	13:33	240.7	5.8	135	1047.3	204.2	257.7	10.56	0.90	3.41
BAGIF211	13:42	265.7	6.1	149	1091.4	238.6	268.6	6.35	0.87	2.05
BAGIF212	13:57	259.3	5.8	146	948.1	158.9	233.3	9.47	0.84	3.06
BAGIF213	14:17	246.6	5.8	139	579.4	84.3	142.6	16.46	0.94	5.32
BAGIF214	14:31	209.8	5.5	118	315.7	48.7	77.7	17.14	0.93	5.54
BAGIF215	14:46	208.9	5.4	118	324.2	48.6	79.8	20.59	1.07	6.65
BAGIF216	15:05	209.9	5.4	118	236.6	40.8	58.2	26.00	1.19	8.40
BAGIF217	15:47	270.9	5.8	152	802.2	125.3	197.4	30.31	1.30	9.79
BAGIA218	16:08	0.0	1.5		38.5	7.4		7.28	0.46	
BAGIA219	16:44	0.0	0.9		23.0	4.6		2.95	0.34	
BAGIA220	17:10	0.0	0.8		20.0	4.4		1.54	0.38	
average lbs/hour =				142.1			222.0			4.79
		DSCFM =	54368							
		% moisture =	3.6							

1 - File name: "BAGI" baghouse inlet sample; "H" untreated; "F" flowing; "A" ambient air through the sampling system.
"4*sigma" - estimated uncertainty.

TABLE 3-5 FTIR RESULTS FROM THE APG BAGHOUSE INLET, RUN 3, 10/24/96.

File name	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGIA301	10:40	0.0	4.1		0.0	7.1		1.53	1.45	
BAGIH302	11:14	159.1	7.7	92.4	62.2	18.2	23.5	57.07	4.11	19.0
BAGIH303	11:21	126.9	8.0	73.7	26.8	17.2	18.7	66.44	4.74	22.1
BAGIH304	11:29	127.1	8.2	73.8	117.2	21.6	18.7	63.51	4.50	21.2
BAGIH305	11:37	149.7	8.5	86.9	145.0	24.2	22.1	64.50	4.45	21.5
BAGIH306	11:46	132.9	8.8	77.1	124.1	22.9	19.6	63.61	4.35	21.2
BAGIH307	12:03	150.3	7.0	87.3	97.5	21.4	22.2	60.67	3.99	20.2
BAGIH308	12:14	162.0	7.1	94.0	73.5	19.6	23.9	55.71	3.72	18.6
BAGIH309	12:26	155.3	7.1	90.2	97.6	22.0	22.9	62.64	3.84	20.9
BAGIH310	12:39	135.1	7.0	78.4	91.1	21.1	19.9	60.58	3.76	20.2
BAGIH311	12:51	141.2	7.2	82.0	52.5	20.0	20.8	59.23	3.60	19.7
BAGIH312	13:06	169.1	6.9	98.2	300.2	45.9	24.9	67.14	3.80	22.4
BAGIH313	13:15	157.5	7.2	91.4	181.6	37.9	23.2	65.87	3.75	22.0
BAGIH314	13:41	158.8	6.6	92.2	374.7	53.5	23.4	55.31	3.12	18.4
BAGIC315	13:52	108.7	4.9	63.1	200.7	33.2	16.0	1.73	1.68	
BAGIC316	13:57	113.1	4.8	65.7	445.9	56.2	16.7	0.00	1.80	
BAGIH317	14:18	149.7	6.7	86.9	0.0	18.7	22.1	43.88	2.56	14.6
BAGIH318	14:31	225.8	7.1	131.1	781.3	101.4	33.3	65.07	3.35	21.7
BAGIH319	14:48	235.3	7.2	136.6	1067.0	143.2	34.7	57.54	3.05	19.2
BAGIH320	15:00	233.4	7.6	135.5	1033.0	142.2	34.4	60.63	3.17	20.2
BAGIH321	15:14	239.7	7.6	139.1	992.9	138.5	35.3	70.25	3.41	23.4
BAGIH322	15:23	227.5	7.5	132.0	812.9	112.6	33.5	68.24	3.27	22.7
BAGIH323	15:33	245.4	8.1	142.5	1739.3	325.7	36.2	62.13	3.11	20.7
BAGIH324	15:41	233.7	7.7	135.7	1349.1	203.8	34.5	64.67	3.25	21.6
BAGIH325	15:50	240.2	7.7	139.4	1271.4	189.3	35.4	62.19	3.07	20.7
BAGIH326	16:06	192.1	7.2	111.5	431.4	67.0	28.3	67.74	3.32	22.6
BAGIH327	16:23	223.1	7.5	129.5	960.0	130.1	32.9	68.75	3.37	22.9
BAGIH328	16:32	235.7	7.6	136.8	1311.8	201.8	34.7	66.69	3.22	22.2

1 - File name: "BAGI" baghouse inlet; "H" untreated; "F" flowing; "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-5 CONTINUED

File name	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGIH329	16:39	248.8	7.6	144.4	1448.3	272.4	36.7	65.96	3.21	22.0
BAGIH330	16:50	246.3	7.6	143.0	1412.5	253.6	36.3	66.05	3.35	22.0
BAGIH331	17:15	251.6	7.9	146.1	1609.5	298.1	37.1	62.75	3.20	20.9
BAGIH332	17:22	243.8	7.8	141.5	1178.1	168.7	35.9	66.17	3.25	22.1
BAGIH333	17:30	249.2	8.1	144.7	1718.7	310.9	36.7	67.01	3.36	22.3
BAGIC334	17:40	126.3	4.4	73.3	384.4	51.9	18.6	2.92	1.79	
BAGIC335	17:48	120.7	4.4	70.1	234.7	38.1	17.8	3.85	1.74	
BAGIH336	18:01	226.5	7.3	131.5	720.6	95.3	33.4	64.39	3.35	21.5
BAGIH337	18:17	208.1	7.4	120.8	663.9	91.1	30.7	65.85	3.35	21.9
BAGIH338	18:30	206.2	7.5	119.7	515.0	76.4	30.4	70.94	3.55	23.6
BAGIH339	18:46	202.1	7.5	117.3	706.0	96.8	29.8	60.85	3.26	20.3
BAGIH340	18:54	199.5	7.6	115.8	379.4	60.8	29.4	62.28	3.36	20.8
BAGIH341	19:03	207.0	7.7	120.1	603.7	84.1	30.5	60.52	3.29	20.2
BAGIH342	19:12	225.0	7.8	130.6	918.5	124.8	33.2	56.99	3.14	19.0
BAGIH343	19:20	226.4	7.9	131.4	913.2	122.1	33.4	58.75	3.24	19.6
BAGIH344	19:28	237.6	8.1	137.9	1269.1	188.4	35.0	60.29	3.22	20.1
BAGIH345	19:35	228.2	7.7	132.5	968.9	132.4	33.6	57.60	3.13	19.2
BAGIH346	19:42	239.4	8.2	139.0	1577.4	288.8	35.3	56.37	3.16	18.8
BAGIA347	20:03	0.0	2.3		12.5	6.6		3.29	1.56	
BAGIA348	20:12	0.0	2.4		0.0	6.6		2.84	1.57	
BAGIA349	20:42	0.0	2.7		0.0	6.8		2.21	1.62	
average lbs/hour =				86.0			21.8			20.6
		DSCFM =	55870							
		% moisture =	4							

1 - File name: "BAGI" baghouse inlet; "H" untreated; "F" flowing; "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-6. FTIR RESULTS FROM THE APG BAGHOUSE OUTLET, RUN 1, 10/22/96.

File name[1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOH001	11:00	48.86	0.28	26.2	1309.5	65.9	307.3	21.2	1.20	6.5
BAGOH002	11:05	47.05	0.32	25.2	1351.1	64.4	317.0	26.4	1.41	8.1
BAGOH003	11:18	47.82	0.33	25.6	1450.9	69.1	340.4	22.9	1.26	7.0
BAGOH004	11:22	47.39	0.34	25.4	1545.6	72.8	362.7	26.1	1.39	8.1
BAGOC005	11:38	29.03	0.21	15.6	1632.8	133.0	383.1	2.3	0.45	0.7
BAGOC006	11:41	33.70	0.23	18.1	1816.5	227.3	426.2	1.2	0.46	0.4
BAGOH007	11:54	47.85	0.33	25.7	1364.7	67.4	320.2	28.0	1.39	8.6
BAGOH008	12:00	47.60	0.34	25.5	1301.4	68.4	305.3	28.8	1.43	8.9
BAGOH009	12:25	48.54	0.33	26.0	1187.4	64.8	278.6	28.4	1.36	8.8
BAGOH010	12:30	46.95	0.34	25.2	1177.5	66.7	276.3	29.6	1.39	9.1
BAGOF011	12:49	49.11	0.35	26.3	1250.4	66.7	293.4	32.5	1.48	10.0
BAGOF012	12:58	50.88	0.35	27.3	1232.3	62.6	289.1	35.1	1.55	10.8
BAGOF013	13:30	53.18	0.36	28.5	1431.5	86.0	335.9	38.6	1.59	11.9
BAGOH014	13:57	52.23	0.34	28.0	1831.9	222.4	429.8	30.6	1.36	9.4
BAGOF015	14:16	49.78	0.36	26.7	1480.6	75.3	347.4	38.5	1.52	11.9
BAGOH016	14:21	49.87	0.35	26.7	1319.2	74.9	309.5	34.6	1.36	10.7
BAGOH017	14:40	47.45	0.34	25.4	1909.9	222.3	448.1	34.4	1.32	10.6
BAGOF018	14:46	26.10	0.32	14.0	194.9	10.3	45.7	35.8	1.34	11.0
BAGOH020	15:05	46.44	0.34	24.9	1095.4	65.2	257.0	37.0	1.33	11.4
BAGOF020	15:12	46.36	0.36	24.9	1159.5	67.9	272.0	39.9	1.45	12.3
BAGOH021	15:51	45.08	0.35	24.2	1259.1	77.2	295.4	36.9	1.28	11.4
BAGOH022	16:00	41.29	0.34	22.1	1270.6	82.7	298.1	37.5	1.31	11.6
BAGOH023	16:09	45.25	0.35	24.3	1357.5	77.3	318.5	34.4	1.20	10.6
BAGOH024	16:20	20.02	0.31	10.7	28.55	4.9	6.7	35.3	1.24	10.9
BAGOH025	16:28	47.76	0.36	25.6	1306.1	78.9	306.5	38.9	1.31	12.0

1 - File name: "BAGO" baghouse outlet; "H" untreated; "F" flowing; "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-6. CONTINUED

File name[1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOH026	16:38	20.81	0.32	11.2	24.5	5.05	5.7	37.1	1.30	11.4
BAGOH027	16:46	50.58	0.35	27.1	1009.5	58.5	236.8	44.7	1.49	13.8
BAGOH028	16:55	49.29	0.35	26.4	1228.9	72.8	288.3	40.7	1.39	12.5
BAGOH029	17:06	49.23	0.37	26.4	2228.0	305.4	522.8	40.9	1.38	12.6
BAGOH030	17:14	47.48	0.36	25.5	1315.0	72.8	308.5	41.3	1.40	12.7
BAGOH031	17:23	21.99	0.32	11.8	65.7	5.43	15.4	37.5	1.33	11.5
BAGOH032	17:32	48.41	0.36	26.0	1236.8	67.6	290.2	40.5	1.39	12.5
BAGOA033	18:24	0.00	0.12		28.45	2.73		3.7	0.46	
BAGOA034	18:33	0.00	0.12		13.98	2.33		2.3	0.47	
BAGOA035	18:56	0.00	0.11		18.16	2.22		1.0	0.47	
average lbs/hour =				23.5			288.7			10.0
		DSCFM =	51888							
		% moisture =	3.5							

1 - File name: "BAGO" baghouse outlet; "H" untreated; "F" flowing; "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-7 FTIR RESULTS FROM THE APG BAGHOUSE OUTLET, RUN 2, 10/23/96.

File name	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOA201	10:29	0.00	0.15		0.0	0.0	50.1	2.5		4.89	0.46	
BAGOA202	10:34	0.00	0.19		0.0	0.0	91.6	3.3		5.84	0.50	
BAGOH203	10:59	58.46	0.33	31.3	0.0	0.0	1376.1	66.6	322.2	42.18	2.12	13.0
BAGOH204	11:10	57.63	0.33	30.8	0.0	0.0	1320.8	66.4	309.3	45.29	2.26	13.9
BAGOH205	11:21	57.93	0.34	31.0	0.0	0.0	1280.1	65.7	299.7	45.01	2.19	13.8
BAGOH206	11:33	54.57	0.34	29.2	0.0	0.0	913.3	35.6	213.8	46.22	2.16	14.2
BAGOH207	11:47	56.62	0.34	30.3	0.0	0.0	1010.6	41.6	236.6	47.40	2.17	14.6
BAGOH208	12:18	56.90	0.35	30.5	0.0	0.0	962.0	40.0	225.3	48.19	2.08	14.8
BAGOH209	12:30	57.28	0.34	30.7	0.0	0.0	917.7	37.2	214.9	48.61	2.07	14.9
BAGOH210	12:37	56.81	0.34	30.4	0.0	0.0	922.1	38.8	215.9	50.93	2.13	15.7
BAGOH211	12:42	56.71	0.35	30.4	0.0	0.0	908.6	38.1	212.8	53.24	2.24	16.4
BAGOH212	12:46	55.72	0.35	29.8	0.0	0.0	835.9	33.7	195.7	52.61	2.22	16.2
BAGOH213	12:51	57.76	0.35	30.9	0.0	0.0	1035.8	53.2	242.5	52.32	2.18	16.1
BAGOH214	12:55	57.95	0.35	31.0	0.0	0.0	989.0	44.4	231.6	53.34	2.19	16.4
BAGOS215	13:17	52.34	0.33	30.5	**0.327**	**0.008**	949.7	38.9	242.2	43.23	1.73	14.5
BAGOS216	13:22	53.14	0.32	31.0	**0.321**	**0.008**	1015.5	49.3	259.0	46.91	1.90	15.7
BAGOS217	13:25	53.48	0.34	31.2	**0.326**	**0.008**	1339.5	81.4	341.6	48.76	1.93	16.3
BAGOS218	13:48	50.75	0.67	29.6	0.0	0.0	892.0	34.1	227.5	43.75	1.78	
BAGOS219	14:00	52.36	0.35	30.5	0.0	0.0	885.0	35.6	225.7	50.25	1.99	
BAGOS220	14:05	50.85	0.42	29.6	0.0	0.0	805.9	31.0	205.5	**53.05**	**2.16**	
BAGOS221	14:10	49.52	0.43	28.9	0.0	0.0	744.4	27.7	189.9	**54.16**	**2.20**	
BAGOS222	14:21	43.75	0.31	25.5	0.0	0.0	245.7	10.4	62.7	**52.69**	**2.05**	
BAGOS223	14:36	39.31	0.30	22.9	0.0	0.0	106.5	4.9	27.2	**53.51**	**2.16**	
BAGOS224	14:57	25.98	0.28	15.1	0.0	0.0	27.6	3.7	7.0	**52.49**	**2.11**	
BAGOS225	15:10	46.68	0.31	27.2	0.0	0.0	383.3	13.5	97.8	**54.90**	**2.22**	
BAGOS226	15:14	47.89	0.32	27.9	0.0	0.0	476.1	16.5	121.4	**56.92**	**2.34**	
BAGOS227	15:18	41.02	0.31	23.9	0.0	0.0	212.4	9.3	54.2	**56.61**	**2.37**	

1 - File name: "BAGO" baghouseoutlet, "H" untreated, "F" flowing; "S" spiked (bold in box indicates SF_6 or HCl), "A" ambient air through the sampling system, "C" condenser sample.
"4*sigma" - estimated uncertainty

TABLE 3-7 CONTINUED

File name	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOS228	15:22	38.92	0.30	22.7	0.0	0.0	217.5	9.3	55.5	**55.49**	**2.29**	
BAGOS229	15:25	35.61	0.30	20.8	0.0	0.0	47.2	3.9	12.0	**56.24**	**2.35**	
BAGOS230	15:29	23.29	0.28	13.6	0.0	0.0	28.8	3.5	7.3	**53.86**	**2.21**	
BAGOS231	15:34	26.37	0.29	15.4	0.0	0.0	32.8	3.6	8.4	**54.71**	**2.26**	
BAGOS232	15:52	50.21	0.32	29.3	**0.329**	**0.007**	704.6	23.9	179.7	48.13	2.02	14.8
BAGOS233	15:56	47.51	0.32	27.7	**0.332**	**0.007**	585.6	20.1	149.4	49.29	2.02	15.1
BAGOS234	16:00	41.31	0.31	24.1	**0.333**	**0.007**	310.5	11.3	79.2	48.24	2.01	14.8
BAGOH235	16:11	50.46	0.32	27.0	0.0	0.0	563.4	19.0	131.9	50.65	2.14	15.6
BAGOA236	16:37	0.00	0.10		0.0	0.0	42.0	1.6		5.95	0.35	
BAGOA237	17:00	0.00	0.16		0.0	0.0	10.5	0.9		3.39	0.76	
BAGOA238	17:14	0.00	0.10		0.0	0.0	24.6	1.3		3.45	0.37	
average lbs/hour =				26.5					164.9			14.3
		Average SF_6 concentration =			0.328							
		SF_6 standard =			4.01					DSCFM =	51890	
		dilution =			12.21					% moisture =	3.3	

1 - File name: "BAGO" baghouseoutlet; "H" untreated; "F" flowing, "S" spiked (bold in box indicates SF_6 or HCl); "A" ambient air through the sampling system, "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-8 FTIR RESULTS FROM THE APG BAGHOUSE OUTLET, RUN 3, 10/24/96.

File name [1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOA301	10:34	0.00	0.15		0.033	0.004	0.00	1.55		0.00	1.48	
BAGOH302	11:09	31.44	0.42	18.2	0.071	0.010	149.94	5.54	38 0	45.00	3.42	15.0
BAGOH303	11:17	30.00	0.44	17.4	0.066	0.010	34.69	4.07	8.8	56.99	4.11	19.0
BAGOH304	11:25	28.96	0.46	16.8	0.072	0.011	91.77	4.92	23.3	59.39	4.23	19.8
BAGOH305	11:33	29.75	0.48	17.2	0.077	0.011	125.71	5.52	31.9	60.11	4.16	20.0
BAGOH306	11:41	34.79	0.50	20.2	0.083	0.012	149.66	6.08	38.0	60.36	4.14	20.1
BAGOH307	11:58	29.17	0.37	16.9	0.035	0.009	22.36	4.02	5.7	53.66	3.73	17.9
BAGOH308	12:08	28.74	0.40	16.7	0.039	0.009	21.32	4.29	5.4	60.12	3.96	20.0
BAGOH309	12:21	29.44	0.39	17.1	0.036	0.009	27.60	4.15	7.0	54.75	3.59	18.2
BAGOH310	12:32	24.94	0.39	14.5	0.040	0.009	20.12	4.34	5.1	56.18	3.60	18.7
BAGOH311	12:44	23.05	0.39	13.4	0.037	0.009	19.09	4.37	4.8	54.10	3.38	18.0
BAGOH312	13:00	28.48	0.39	16.5	0.036	0.009	85.00	5.29	21.6	54.81	3.38	18.2
BAGOH313	13:09	32.99	0.40	19.1	0.000	0.009	176.24	8.82	44.7	58.61	3.50	19.5
BAGOH314	13:25	34.59	0.39	20.0	0.000	0.009	343.48	12.61	87.1	57.03	3.29	19.0
BAGOC315	13:29	27.88	0.29	16.2	0.000	0.007	283.56	9.69	71.9	3.16	1.76	
BAGOC316	13:33	24.34	0.29	14.1	0.000	0.007	61.88	3.49	15.7	0.00	1.75	
BAGOH317	14:08	49.64	0.40	28 8	0.039	0.009	785.68	24.44	199.2	52.78	2.88	17.6
BAGOS318	14:21	44.93	0.39	27.4	**0.197**	**0.009**	797.92	24.86	212.8	60.85	3.17	21.3
BAGOS319	14:27	37.68	0.38	23.0	**0.192**	**0.009**	437.71	15.54	116.7	56.90	2.97	19.9
BAGOS320	14:35	45.77	0.38	27.9	**0.201**	**0.009**	661.86	21.06	176.5	60.98	3.13	21.3
BAGOS321	14:54	44.54	0.39	27.1	0.000	0.009	683.66	22.20	182.3	**58.50**	**3.02**	
BAGOS322	15:05	41.86	0.40	25.5	0.000	0.009	418.19	15.61	111.5	**64.23**	**3.21**	
BAGOS323	15:09	46.08	0.38	28.1	0.036	0.009	742.92	23.77	198.1	**65.29**	**3.18**	
BAGOS324	15:18	47.46	0.39	28.9	0.038	0.009	1069.51	36.56	285.2	**62.23**	**3.08**	
BAGOS325	15:29	47.82	0.40	29.1	0.037	0.009	1101.91	38.66	293.8	**61.02**	**3.00**	
BAGOS326	15:37	46.32	0.41	28.2	0.000	0.009	1252.09	46.07	333.9	**60.49**	**2.99**	
BAGOS327	15:45	47.99	0.41	29.3	0.000	0.009	1067.77	37.42	284.7	**61.41**	**3.02**	

1 - File name: "BAGO" baghouse outlet, "H" untreated, "F" flowing, "S" spiked (bold, in box, indicates SF_6 or HCl) "A" ambient air through the sampling system, "C" condenser sample.
"4*sigma" - estimated uncertainty

TABLE 3-8 . CONTINUED

File name [1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOS328	15:53	47.17	0.40	28.8	0.038	0.009	981.93	33.15	261.8	**60.21**	**3.06**	
BAGOS329	15:58	45.97	0.40	28.0	0.000	0.009	1141.39	40.30	304.4	**58.34**	**2.96**	
BAGOS330	16:02	43.94	0.39	26.8	0.000	0.009	780.38	26.11	208.1	**60.61**	**3.03**	
BAGOS331	16:09	43.48	0.37	26.5	0.036	0.009	726.81	25.06	193.8	**64.47**	**3.04**	
BAGOS332	16:13	40.31	0.37	24.6	0.036	0.009	573.27	19.54	152.9	**62.73**	**3.08**	
BAGOS333	16:18	37.25	0.38	22.7	0.000	0.009	356.38	14.73	95.0	**64.70**	**3.13**	
BAGOS334	16:28	45.43	0.38	27.7	**0.192**	**0.009**	949.85	33.02	253.3	58.27	2.84	20.4
BAGOS335	16:36	46.82	0.39	28.5	**0.200**	**0.009**	1089.77	38.78	290.6	57.39	2.92	20.1
BAGOS336	16:43	43.73	0.37	26.7	**0.198**	**0.009**	784.15	26.46	209.1	55.74	2.84	19.5
BAGOH337	17:07	53.33	0.40	30.9	0.037	0.009	1454.58	61.65	368.9	55.52	2.90	18.5
BAGOH338	17:18	48.71	0.40	28.2	0.039	0.009	846.33	28.14	214.6	59.73	3.08	19.9
BAGOH339	17:27	50.62	0.42	29.3	0.040	0.010	1664.03	73.69	422.0	58.73	2.98	19.5
BAGOH340	17:34	50.68	0.42	29.4	0.042	0.010	886.12	29.86	224.7	59.66	3.08	19.9
BAGOH341	17:44	47.43	0.39	27.5	0.040	0.009	727.87	24.13	184.6	58.40	3.00	19.4
BAGOH342	17:52	45.43	0.40	26.3	0.041	0.009	626.95	20.88	159.0	60.38	3.14	20.1
BAGOC343	18:12	28.25	0.25	16.4	0.000	0.006	374.72	12.83	95.0	0.00	1.82	
BAGOC344	18:20	29.26	0.27	17.0	0.000	0.006	432.92	14.44	109.8	3.00	1.88	
BAGOH345	18:26	44.58	0.39	25.8	0.040	0.009	639.27	21.02	162.1	59.74	3.05	19.9
BAGOH346	18:34	44.92	0.40	26.0	0.040	0.009	659.12	22.29	167.1	62.87	3.28	20.9
BAGOH347	18:42	43.15	0.39	25.0	0.039	0.009	550.73	18.53	139.7	57.99	3.08	19.3
BAGOH348	18:50	45.70	0.40	26.5	0.041	0.009	782.79	25.41	198.5	59.78	3.13	19.9
BAGOH349	18:59	45.97	0.42	26.6	0.044	0.010	667.00	22.32	169.1	63.29	3.28	21.1
BAGOH350	19:07	45.52	0.41	26.4	0.043	0.009	742.26	24.11	188.2	60.63	3.17	20.2
BAGOH351	19:16	48.46	0.42	28.1	0.045	0.010	1028.54	33.61	260.8	61.15	3.21	20.4
BAGOH352	19:23	47.28	0.41	27.4	0.043	0.009	996.61	31.25	252.7	61.49	3.30	20.5
BAGOH353	19:32	48.27	0.41	28.0	0.041	0.010	902.44	28.20	228.8	59.48	3.23	19.8
BAGOH354	19:39	48.10	0.41	27.9	0.042	0.010	1023.76	33.03	259.6	60.42	3.25	20.1

1 - File name: "BAGO" baghouse outlet; "H" untreated; "F" flowing, "S" spiked (bold, in box, indicates SF_6 or HCl) "A" ambient air through the sampling system; "C" condenser sample.
"4*sigma" - estimated uncertainty.

TABLE 3-8 CONTINUED

File name [1]	Time	SO_2 ppm	4 * σ	SO_2 lbs/hr	SF_6 ppm	4 * σ	CO ppm	4 * σ	CO lbs/hr	HCl ppm	4 * σ	HCl lbs/hr
BAGOH355	19:47	50.20	0.43	29.1	0.042	0.010	1453.50	64.58	368.6	60.53	3.30	20.1
BAGOH356	19:51	51.23	0.44	29.7	0.045	0.010	1400.77	61.59	355.2	58.46	3.22	19.5
BAGOA357	20:07	0.00	0.14		0.000	0.003	0.00	1.72		8.14	1.67	
BAGOA358	20:17	0.00	0.16		0.000	0.004	0.00	1.69		7.72	1.67	
BAGOA359	20:35	0.00	0.15		0.000	0.003	0.00	1.56		3.90	1.62	
BAGOA360	20:39	0.00	0.17		0.000	0.004	0.00	1.66		5.17	1.68	
Average lbs/hour =				24.2					169.1			19.5
			SF_6 spike =		0.197	0.009				DSCFM =	55904	
			SF_6 standard =		4.01	4.50%				% moisture =	3.8	
			dilution =		20.38							

1 - File name: "BAGO" baghouse outlet, "H" untreated, "F" flowing, "S" spiked (bold, in box, indicates SF_6 or HCl) "A" ambient air through the sampling system, "C" condenser sample.
"4*sigma" - estimated uncertainty

TABLE 3-9. RESULTS OF HCl SPIKING AT APG BAGHOUSE INLET; RUN 1, 10/22/96.

HCl spiking

Time	Unpiked (ppm)	d_i	$(d_i)^2$	Time	Spiked	d_i	$(d_i)^2$
11:28	26.02			15:17	49.79		
11:31	26.83	0.81	0.650	15:20	49.83	0.04	0.001
11:46	24.97			15:25	53.91		
11:49	28.79	3.83	14.648	15:32	58.06	4.14	17.171
12:14	24.66			15:39	57.49		
12:19	30.26	5.61	31.434	15:45	58.60	1.11	1.238
13:10	38.06			15:54	46.67		
13:17	38.82	0.76	0.581	16:04	49.04	2.37	5.594
14:05	42.82			16:14	53.40		
14:10	36.77	-6.05	36.599	16:24	56.08	2.68	7.185
14:27	41.10			16:33	54.93		
14:33	40.45	-0.65	0.422	16:42	58.44	3.51	12.317
	33.30	4.30	84.334		53.85	13.85	43.506
	26.51						
	SD =	2.651			SD =	1.904	
	F =	0.516			SD_{pooled} =	2.308	
	RSD=	8.0					
	Bias =	**-0.439**			**Exp Conc =**	**20.99**	
	t =	**0.47**			**CF =**	**1.02**	

SF_6 spike =	0.817	0.007	0.91%	Results from Table 3-3
SF_6 standard :	4.01 measured			
dilution =	4.906 =..8173/4.01			
HCl standard	103	ppm		
Exp con =	20.994	= 103 ppm/dilution		

Statistical quantities are defined in EPA Method 301, Section 6.3.

TABLE 3-10. RESULTS OF HCl SPIKING AT BAGHOUSE OUTLET; RUN 2, 10/23/96.

HCl spiking

Time	Unspiked (ppm)	d_i	$(d_i)^2$	Time	Spiked	d_i	$(d_i)^2$
10:59	42.2			14:05	53.0		
11:10	45.3	3.11	9.67	14:10	54.2	1.11	1.24
11:21	45.0			14:21	52.7		
11:33	46.2	1.21	1.47	14:36	53.5	0.81	0.66
11:47	47.4			14:57	52.5		
12:18	48.2	0.79	0.62	15:10	54.9	2.42	5.83
12:30	48.6			15:14	56.9		
12:37	50.9	2.32	5.37	15:18	56.6	-0.31	0.10
12:42	53.2			15:22	55.5		
12:46	52.6	-0.63	0.40	15:25	56.2	0.75	0.56
12:51	52.3			15:29	53.9		
12:55	53.3	1.02	1.04	15:34	54.7	0.86	0.73
	48.8	7.81	18.57		54.6	5.63	9.12
	44.8						
	SD =	1.244			SD =	0.872	
	F =	0.491			SD_{pooled} =	1.074	
	RSD=	2.6					
	Bias =	**-2.66**	**1.33**		**Exp Conc =**	**8.44**	
	t =	**6.07**	**3.04**		**CF =**	**1.46**	**0.86**

SF_6 spike =	0.328	0.007	2.26%	Results from Table 3-7
SF_6 standard :	4.01	measured		
dilution =	12.21	=.3285/4.01		
HCl standard	103	ppm		
Exp con =	8.44	= 103 ppm/dilution		

Statistical quantities are defined in EPA Method 301, Section 6.3.

TABLE 3-11. RESULTS OF HCl SPIKING AT APG BAGHOUSE OUTLET; RUN 3, 10/24/96.

HCl spiking

Time	Unspiked (ppm)	d_i	$(d_i)^2$	Time	Spiked (ppm)	d_i	$(d_i)^2$
11:17	56.99			15:05	64.23		
11:25	59.39	2.41	5.804	15:09	65.29	1.06	1.121
11:33	60.11			15:18	62.23		
11:41	60.36	0.25	0.062	15:29	61.02	-1.21	1.469
11:58	53.66			15:37	60.49		
12:08	60.12	6.46	41.672	15:45	61.41	0.93	0.861
12:21	54.75			15:53	60.21		
12:32	56.18	1.43	2.032	15:58	58 34	-1.87	3.504
12:44	54.10			16:02	60.61		
13:00	54.81	0.71	0.507	16:09	64.47	3.87	14.957
13:09	58.61			16:13	62.73		
13:25	57.03	-1.58	2.483	16:18	64.70	1.97	3.871
	57.18	9.68	52.561		62.14	4.74	25.784
	54.37						
	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**	

SF_6 spike =	0.197	0.009	Results from Table 3-8
SF_6 standard	4.01 measured		
dilution =	20.377 =.1968/4.01		
HCl standard	103 ppm		
Exp con =	5.055 = 103 ppm/dilution		

Statistical quantities are defined in EPA Method 301, Section 6.3.

TABLE 3-12. APG BAGHOUSE INLET HAP ESTIMATED UNCERTAINTIES (ppm)

Compound	Baghouse Inlet (Runs 1 and 2				Baghouse Inlet (Run 3			
	Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Acetonitrile	3038.97	3042.42	2.37E-03	26.6	3038.97	3042.42	1.48E-03	29.4
Acrolein	2636.11	2875.59	2.27E-03	3.2	2636.11	2875.59	2.14E-03	5.3
Acrylonitrile	968.58	974.19	6.81E-03	3.5	968.58	974.19	4.77E-03	4.3
Allyl Chloride	899.55	965.72	8.61E-03	5.9	899.55	965.72	6.31E-03	7.6
Benzene	3036.88	3063.07	1.45E-02	7.3	3036.88	3063.07	9.77E-03	8.7
Bromoform	1135.9	1154.2	8.17E-03	1.6	1135.9	1154.2	6.02E-03	2.1
1,3-Butadiene	895.91	919.75	2.96E-03	1.0	895.91	919.75	2.15E-03	1.2
Carbonyl Sulfide	2026.14	2085.23	1.77E-01	6.2	2026.14	2085.23	4.18E-02	2.6
Chlorobenzene	1069.86	1103.34	7.19E-03	3.8	1069.86	1103.34	5.21E-03	4.8
Ethyl Benzene	2850.71	2959.43	3.93E-03	5.2	2959.43	2997.25	4.81E-03	4.7
Ethyl Chloride	2916.56	3041.03	1.24E-02	10.0	2916.56	3041.03	8.34E-03	11.8
Ethylene Dibromide	1167.96	1208.92	7.52E-03	5.8	1167.96	1208.92	7.15E-03	9.7
n-Hexane	2835.27	3005.43	5.77E-03	0.9	2835.27	3005.43	3.73E-03	1.0
Methyl Bromide	2948.11	2972.53	6.91E-03	8.5	2948.11	2972.53	4.21E-03	9.2
Methyl Chloride	1017.96	1020.72	2.01E-03	7.1	1017.96	1020.72	1.23E-03	7.7
Methyl Ethyl Ketone	1140.7	1222.63	8.33E-03	6.6	1140.7	1222.63	7.95E-03	11.2
Methyl Isobutyl Ketone	2872.05	2994.95	6.24E-03	2.8	2872.05	2994.95	3.89E-03	3.1
Methyl Methacrylate	1137 5	1232.04	1.00E-02	1.2	1137.5	1232.04	8.99E-03	1.9
Methylene Chloride	1252.28	1283.79	2.88E-02	11.5	1252.28	1283.79	3.33E-02	23.6
2-Nitropropane	831.47	868.5	4.55E-03	5.4	831.47	868.5	3.43E-03	7.2
Propylene Dichloride	996.86	1038	5.51E-03	7.0	996.86	1038	4.50E-03	10.1
Styrene	886.69	920.72	2.86E-03	2.2	886.69	920.72	2.23E-03	3.1
Tetrachloroethylene	899.2	925.2	3.55E-03	0.3	899.2	925.2	2.22E-03	0.4
Toluene	2862	2924	2.70E-03	4.6	2862	2924	2.43E-03	7.3
1,1,2-Trichloroethane	916.98	956.37	1.00E-02	7.6	916.98	956.37	7.12E-03	9.6
Trichloroethylene	826.25	860.91	4.71E-03	0.8	826.25	860.91	3.47E-03	1.0
2,2,4-Trimethylpentane	2861.57	3009.23	6.21E-03	0.8	2861.57	3009.23	3.90E-03	0.9
Vinyl Acetate	1201.77	1242.73	1.26E-02	0.5	1201.77	1242.73	1.13E-02	0.8
Vinyl Bromide	899.81	904.54	1.26E-03	0.6	899.81	904.54	1.65E-03	1.3
Vinyl Chloride	894.43	899.25	1.18E-03	2.2	894.43	899.25	1.00E-03	3.3
Vinylidene Chloride	834.13	898.73	3.76E-03	2.6	1059.44	1113.01	6.53E-03	3.3
O-xylene	2859.84	3095.04	1.23E-02	8.4	2859.84	3095.04	8.42E-03	10.2
P-xylene	2854.43	3083.14	1.22E-02	7.2	2854.43	3083.14	8.48E-03	8.8
Carbon Disulfide	2171.64	2198.03	1.95E-02	20.5	2171.64	2198.03	3.26E-03	6.1
Carbon Tetrachloride	793.89	800.58	2.31E-02	0.5	793.89	800.58	2.11E-02	0.8
Chloroform	758.21	781.25	4.91E-02	2.7	758.21	781.25	5.10E-02	4.9
Cumene	2951.21	2998.48	8.98E-03	2.5	2951.21	2998.48	4.91E-03	2.4
1,2-Epoxy Butane	902.37	919.7	3.14E-03	2.5	902.37	919.7	2.22E-03	3.1
Ethylene Oxide	866.9	875	1.68E-03	0.4	866.9	875	2.11E-03	1.0
Methanol	2807.91	3029.4	8.20E-03	7.1	2807.91	3029.4	5.43E-03	8.3
Methyl Chloroform	1057.95	1105.3	9.79E-03	1.8	1057.95	1105.3	6.88E-03	2.3

See Section 4.8. Screening For HAPs.

TABLE 3-12. CONTINUED

Compound	Baghouse Inlet (Runs 1 and 2 Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Baghouse Inlet (Run 3 Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Methyl Iodide	1250.18	1253.53	2.13E-03	2.1	1250.18	1253.53	1.35E-03	2.3
Methyl t-Butyl Ether	1195	1210	5.29E-03	0.9	1195	1210	4.21E-03	1.3
Propylene Oxide	2875.59	3097.75	1.32E-02	9.0	2875.59	3097.75	8.96E-03	10.8
M-xylene	2910.25	2952.78	4.67E-03	3.1	2910.25	2952.78	3.59E-03	4.2
Acetone	1182	1255.03	1.13E-02	5.3	1182	1255.03	1.01E-02	8.4
Acetaldehyde	2685.41	2744.4	1.69E-03	3.0	2685.41	2744.4	1.62E-03	5.1
Acetophenone	1140.4	1286.06	1.60E-02	2.2	1140.4	1286.06	1.75E-02	4.3
Acrylic Acid	1104.89	1164.68	7.28E-03	1.0	1104.89	1164.68	5.24E-03	1.3
Aniline	1102.9	1123.63	3.73E-03	1.3	1102.9	1123.63	2.97E-03	1.8
Benzotrichloride	866.5	877.9	2.46E-03	0.5	866.5	877.9	2.27E-03	0.8
Benzyl Chloride	3027.52	3109.06	1.93E-02	16.2	3027.52	3109.06	1.31E-02	19.5
Bis(chloromethyl)ether	1068.78	1154.25	8.92E-03	1.0	1068.78	1154.25	6.54E-03	1.3
Chloroacetic acid	1094.97	1124.12	4.08E-03	0.9	1094.97	1124.12	3.01E-03	1.2
2-Chloroacteophenone	1274.39	1285.42	1.17E-02	2.3	1274.39	1285.42	1.22E-02	4.3
Chloromethyl methyl ether	1111.02	1146.08	8.52E-03	1.3	1111.02	1146.08	5.96E-03	1.6
Chloroprene	875.9	878.8	3.01E-03	0.6	875.9	878.8	1.58E-03	0.5
o-Cresol	1092.8	1114.07	3.56E-03	1.6	1092.8	1114.07	2.50E-03	2.0
m-Cresol	1139.68	1172.77	4.72E-03	0.9	1139.68	1172.77	3.44E-03	1.2
p-Cresol	1159.1	1185.5	5.22E-03	0.6	1159.1	1185.5	5.67E-03	1.2
1,2-Dibromo-3-chloropropane	1134.26	1175.42	7.75E-03	9.3	1134.26	1175.42	6.54E-03	13.9
1,4-Dichlorobenzene	995.96	1031.06	3.41E-03	1.6	995.96	1031.06	2.41E-03	2.0
Dichloroethyl ether	1109.35	1155.04	7.87E-03	0.9	1109.35	1155.04	5.67E-03	1.2
1,3-Dichloropropene	768	791	2.77E-02	5.9	768	791	1.95E-02	7.4
Dichlorvos	835.77	876.95	4.47E-03	0.5	835.77	876.95	3.38E-03	0.7
N,N-Diethyl aniline	2655.32	3156.07	1.16E-02	7.1	2655.32	3156.07	7.91E-03	8.6
Dimethyl carbamoyl chloride	889.55	917.52	2.87E-03	0.8	889.55	917.52	2.09E-03	1.0
Dimethyl formamide	2824.8	2873.6	2.48E-03	1.7	2824.8	2873.6	2.29E-03	2.7
1,1-Dimethyl hydrazine	2740.77	2914.08	2.49E-03	1.2	2740.77	2914.08	2.49E-03	2.2
Dimethyl phthalate	1157.86	1254.16	1.03E-02	5.8	1157.86	1254.16	9.30E-03	9.2
1,4-Dioxane	2861.1	2864.8	1.78E-03	0.3	2919.4	2921.3	2.43E-04	0.1
Epichlorohydrin	943.52	981.73	1.01E-02	8.7	943.52	981.73	7.77E-03	11.9
Ethyl Acrylate	1181.93	1210	8.14E-03	0.4	1181.93	1210	7.03E-03	0.7
Ethylene Dichloride	1227.88	1241.5	4.28E-03	2.5	1227.88	1241.5	3.11E-03	3.3
Ethylidene dichloride	1041.11	1080.5	1.32E-02	6.0	1041.11	1080.5	9.82E-03	7.8
Formaldehyde	2788.33	2842.2	2.56E-03	2.7	2788.33	2842.2	2.45E-03	4.6
Hexachlorobutadiene	847.5	864.5	5.58E-03	0.5	847.5	864.5	3.77E-03	0.6
Hexachlorocylcopentadiene	1227.02	1240.42	4.42E-03	0.4	1227.02	1240.42	3.27E-03	0.5
Hexachloroethane	779.26	797.38	2.43E-01	5.5	779.26	797.38	9.09E-02	3.6
Hexamethylphosphoramide	949.42	1019.53	6.80E-03	1.0	949.42	1019.53	5.26E-03	1.3
Isophorone	2681.2	3130.6	1.18E-02	7.9	2681.2	3130.6	7.99E-03	9.5
Maleic Anhydride	885.27	905.56	1.86E-03	0.2	838.45	841.3	1.92E-03	0.4
Methyl hydrazine	2683	3061.78	8.49E-03	9.8	2683	3061.78	5.96E-03	12.2

See Section 4.8, Screening For HAPs.

TABLE 3-12. CONTINUED

Compound	Baghouse Inlet (Runs 1 and 2				Baghouse Inlet (Run 3			
	Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Naphthalene	779.31	783.55	3.82E-03	0.4	779.31	783.55	5.44E-03	1.0
Nitrobenzene	841.7	861.39	5.14E-03	2.1	841.7	861.39	3.53E-03	2.6
N-Nitrosodimethylene	928	1085.28	1.01E-02	3.3	928	1085.28	7.72E-03	4.5
N-Nitrosomorpholine	892.23	1024.64	7.65E-03	3.2	892.23	1024.64	5.82E-03	4.4
Phenol	1162.67	1195.76	8.00E-03	1.3	998.4	999.9	1.85E-04	1.7
beta-Propiolactone	860.13	957.64	7.19E-03	1.6	860.13	957.64	5.32E-03	2.1
Propionaldehyde	2546.18	3114.35	9.40E-03	10.6	2546.18	3114.35	6.38E-03	12.8
1,2-Propylenimine	817.57	821.31	3.25E-03	1.5	817.57	821.31	2.13E-03	1.7
Quinoline	800.19	803.73	1.21E-02	1.9	800.19	803.73	7.43E-03	2.1
Styrene Oxide	861.39	903.93	2.45E-03	1.8	861.39	903.93	2.60E-03	3.4
1,1,2,2-Tetrachloroethane	794.92	824.07	1.30E-02	3.1	794.92	824.07	1.14E-02	4.8
2,4-Toluene diisocyanate	2254.7	2301.18	2.74E-02	0.4	881.58	911.51	2.44E-03	3.4
o_Toluidine	2858.5	2951.85	3.60E-03	3.1	2858.5	2951.85	2.96E-03	4.6
1,2,4-Trichlorobenzene	1086.21	1114.37	4.22E-03	1.1	1086.21	1114.37	3.04E-03	1.4
2,4,5-Trichlorophenol	1178.04	1204.16	8.38E-03	2.7	1178.04	1204.16	7.20E-03	4.1
2,4,6-Trichlorophenol	856.27	863.36	2.67E-03	0.7	856.27	863.36	2.42E-03	1.1
Triethylamine	2756.62	2839.34	2.38E-03	1.1	2756.62	2839.34	2.45E-03	1.9
Ammonia	893.1 -	926	3.36E-03	2.2	893.1 -	926	2.08E-03	2.4

See Section 4.8, Screening For HAPs.

TABLE 3-13. APG BAGHOUSE OUTLET. HAP ESTIMATED UNCERTAINTIES

Compound	Baghouse Outlet, Runs 1 and 2 Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Run 3 RMSD	Estimated Uncertainty (ppm)
Acetonitrile	3038.97 -	3042.42	1.2E-03	13.4	1.7E-03	32.9
Acrolein	2636.11	2875.59	2.0E-03	2.9	1.9E-03	4.7
Acrylonitrile	968.58	974.19	6.7E-03	3.4	4.1E-03	3.7
Allyl Chloride	899.55 -	965.72	8.4E-03	5.7	6.2E-03	7.5
Benzene	3036.88	3063.07	1.9E-02	9.8	8.9E-03	7.9
Bromoform	1135.9	1154.2	6.4E-03	1.3	5.3E-03	1.9
1,3-Butadiene	895.91	919.75	2.7E-03	0.9	2.4E-03	1.4
Carbonyl Sulfide	2026.14	2085.23	6.4E-02	2.2	3.9E-02	2.4
Chlorobenzene	1069.86	1103.34	6.8E-03	3.6	4.7E-03	4.4
Ethyl Benzene	2850.71	2959.43	4.2E-03	5.5	4.3E-03	4.2
Ethyl Chloride	2916.56 -	3041.03	1.5E-02	12.2	7.7E-03	10.9
Ethylene Dibromide	1167.96 -	1208.92	9.3E-03	7.1	5.7E-03	7.7
n-Hexane	2835.27	3005.43	7.5E-03	1.2	3.3E-03	0.9
Methyl Bromide	2948.11	2972.53	8.0E-03	9.8	3.8E-03	8.2
Methyl Chloride	1017.96	1020.72	2.2E-03	7.6	8.4E-04	5.2
Methyl Ethyl Ketone	1140.7	1222.63	9.8E-03	7.8	6.6E-03	9.3
Methyl Isobutyl Ketone	2872.05	2994.95	8.1E-03	3.7	3.4E-03	2.8
Methyl Methacrylate	1137.5	1232.04	1.2E-02	1.4	7.3E-03	1.5
Methylene Chloride	743.96	769.17	1.2E-01	12.1	2.9E-02	20.7
2-Nitropropane	831.47	868.5	5.0E-03	5.9	3.0E-03	6.3
Propylene Dichloride	996.86	1038	6.0E-03	7.6	3.8E-03	8.4
Styrene	886.69	920.72	2.5E-03	2.0	2.3E-03	3.2
Tetrachloroethylene	899.2	925.2	3.0E-03	0.3	2.4E-03	0.4
Toluene	2862	2924	2.5E-03	4.3	2.3E-03	6.8
1,1,2-Trichloroethane	916.98	956.37	9.9E-03	7.5	7.0E-03	9.4
Trichloroethylene	826.25 -	860.91	5.3E-03	0.9	2.9E-03	0.9
2,2,4-Trimethylpentane	2861.57	3009.23	8.2E-03	1.1	3.5E-03	0.8
Vinyl Acetate	1201.77	1242.73	1.6E-02	0.6	9.1E-03	0.6
Vinyl Bromide	899.81	904.54	1.0E-03	0.5	1.6E-03	1.2
Vinyl Chloride	894.43 -	899.25	9.5E-04	1.7	1.2E-03	3.8
Vinylidene Chloride	1059.44	1113.01	8.8E-03	2.5	5.8E-03	3.0
O-xylene	2859.84	3095.04	1.5E-02	10.5	7.8E-03	9.4
P-xylene	2854.43 -	3083.14	1.5E-02	9.1	7.8E-03	8.2
Carbon Disulfide	2171.64	2198.03	6.9E-03	7.3	7.4E-03	13.7

See Section 4.8, Screening For HAPs.

TABLE 3-13. CONTINUED

Compound	Baghouse Outlet, Runs 1 and 2 Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Run 3 RMSD	Estimated Uncertainty (ppm)
Carbon Tetrachloride	793.89	800.58	2.6E-02	0.5	1.6E-02	0.6
Chloroform	758.21	781.25	4.9E-02	2.7	4.7E-02	4.5
Cumene	2951.21	2998.48	1.2E-02	3.3	4.4E-03	2.1
1,2-Epoxy Butane	902.37	919.7	2.8E-03	2.2	2.5E-03	3.5
Ethylene Oxide	866.9	875	1.3E-03	0.3	1.9E-03	0.9
Methanol	2807.91	3029.4	9.7E-03	8.4	5.0E-03	7.6
Methyl Chloroform	1057.95	1105.3	9.2E-03	1.7	6.1E-03	2.1
Methyl Iodide	1250.18	1253.53	6.7E-04	0.7	1.3E-03	2.2
Methyl t-Butyl Ether	1195	1210	6.2E-03	1.1	3.2E-03	1.0
Propylene Oxide	2875.59	3097.75	1.6E-02	11.2	8.2E-03	9.9
M-xylene	2910.25	2952.78	5.1E-03	3.3	3.2E-03	3.7
Acetone	1182	1255.03	1.4E-02	6.5	8.2E-03	6.8
Acetaldehyde	2685.41	2744.4	1.3E-03	2.3	1.7E-03	5.2
Acetophenone	1140.4	1286.06	1.9E-02	2.6	1.5E-02	3.7
Acrylic Acid	1104.89	1164.68	6.2E-03	0.8	4.4E-03	1.0
Aniline	1102.9	1123.63	4.8E-03	1.6	2.3E-03	1.4
Benzotrichloride	866.5	877.9	1.6E-03	0.3	1.8E-03	0.6
Benzyl Chloride	3027.52	3109.06	2.5E-02	20.8	1.2E-02	17.7
Bis(chloromethyl)ether	1068.78	1154.25	8.2E-03	0.9	5.7E-03	1.2
Chloroacetic acid	1094.97	1124.12	4.4E-03	1.0	2.7E-03	1.1
2-Chloroacteophenone	1274.39	1285.42	1.4E-02	2.8	1.1E-02	3.9
Chloromethyl methyl ether	1111.02	1146.08	7.1E-03	1.1	4.9E-03	1.3
Chloroprene	875.9	878.8	2.6E-03	0.5	1.2E-03	0.4
o-Cresol	1092.8	1114.07	3.6E-03	1.6	2.2E-03	1.8
m-Cresol	1139.68	1172.77	3.6E-03	0.7	3.3E-03	1.1
p-Cresol	1159.1	1185.5	6.0E-03	0.7	4.7E-03	1.0
1,2-Dibromo-3-chloropropane	1134.26	1175.42	7.6E-03	9.1	5.5E-03	11.8
1,4-Dichlorobenzene	995.96	1031.06	3.3E-03	1.5	2.2E-03	1.8
Dichloroethyl ether	1109.35	1155.04	6.7E-03	0.8	4.8E-03	1.0
1,3-Dichloropropene	768	791	2.7E-02	5.9	1.7E-02	6.5
Dichlorvos	835.77	876.95	4.7E-03	0.6	3.0E-03	0.7
N,N-Diethyl aniline	2655.32	3156.07	1.5E-02	9.1	7.2E-03	7.8
Dimethyl carbamoyl chloride	889.55	917.52	2.4E-03	0.6	2.3E-03	1.1
Dimethyl formamide	2824.8	2873.6	2.2E-03	1.5	1.9E-03	2.2
1,1-Dimethyl hydrazine	2740.77	2914.08	2.4E-03	1.2	2.2E-03	1.9

See Section 4.8, Screening For HAPs.

TABLE 3-13. CONTINUED

Compound	Baghouse Outlet, Runs 1 and 2 Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)	Run 3 RMSD	Estimated Uncertainty (ppm)
Dimethyl phthalate	1157.86	1254.16	1.3E-02	7.0	7.5E-03	7.5
1,4-Dioxane	2919.4	2921.3	4.7E-04	0.1	2.5E-04	0.1
Epichlorohydrin	943.52	981.73	9.8E-03	8.5	7.5E-03	11.5
Ethyl Acrylate	1181.93	1210	9.8E-03	0.5	5.4E-03	0.5
Ethylene Dichloride	1227.88	1241.5	5.2E-03	3.1	2.5E-03	2.6
Ethylidene dichloride	1041.11	1080.5	1.3E-02	5.8	8.2E-03	6.6
Formaldehyde	2788.33	2842.2	2.5E-03	2.6	2.2E-03	4.1
Hexachlorobutadiene	847.5	- 864.5	6.4E-03	0.6	3.1E-03	0.5
Hexachlorocylcopentadiene	1227.02	1240.42	5.4E-03	0.4	2.6E-03	0.4
Hexachloroethane	779.26	797.38	1.6E-01	3.7	7.4E-02	2.9
Hexamethylphosphoramide	949.42	1019.53	6.6E-03	0.9	5.2E-03	1.3
Maleic Anhydride	885.27	905.56	1.8E-03	0.2	3.0E-03	0.4
Methyl hydrazine	2683	3061.78	1.1E-02	12.2	7.3E-03	11.2
Naphthalene	779.31	783.55	5.7E-03	0.6	2.2E-03	0.8
Nitrobenzene	841.7	861.39	5.9E-03	2.5	5.5E-03	2.1
N-Nitrosodimethylene	928	- 1085.28	1.0E-02	3.3	4.2E-03	3.9
N-Nitrosomorpholine	892.23	1024.64	7.5E-03	3.2	2.8E-03	4.1
Phenol	1162.67	1195.76	9.6E-03	1.6	6.7E-03	1.7
beta-Propiolactone	860.13	957.64	6.9E-03	1.5	5.4E-03	2.0
Propionaldehyde	2546.18	3114.35	1.2E-02	13.3	6.1E-03	11.8
1,2-Propylenimine	817.57	821.31	2.8E-03	1.3	5.2E-03	1.2
Quinoline	800.19	803.73	1.5E-02	2.3	5.9E-03	1.2
Styrene Oxide	861.39	903.93	2.0E-03	1.4	1.5E-03	3.2
1,1,2,2-Tetrachloroethane	794.92	824.07	1.4E-02	3.4	4.3E-03	3.7
2,4-Toluene diisocyanate	881.58	911.51	2.3E-03	1.9	2.4E-03	3.6
o_Toluidine	2858.5	2951.85	3.8E-03	3.3	8.7E-03	4.1
1,2,4-Trichlorobenzene	1086.21	1114.37	4.2E-03	1.1	2.6E-03	1.2
2,4,5-Trichlorophenol	1178.04	1204.16	1.0E-02	3.3	2.7E-03	3.2
2,4,6-Trichlorophenol	856.27	863.36	1.9E-03	0.5	2.7E-03	1.0
Triethylamine	2756.62	2839.34	2.3E-03	1.0	5.5E-03	1.7
Ammonia	893.1	926	2.8E-03	1.8	2.2E-03	2.6

See Section 4.8, Screening For HAPs.

TABLE 3-14. APG COOLING STACK.

Compound	Cooling Stack Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Acetonitrile	1041.4	1042.88	3.3E-04	1.84
Acrolein	913.7	1000.35	8.3E-04	0.57
Acrylonitrile	968.58	974.19	3.0E-04	0.16
Allyl Chloride	899.55	965.72	9.7E-04	0.66
Benzene	3036.88	3063.07	3.0E-03	1.51
Bromoform	1135.9	1154.2	1.1E-03	0.22
1,3-Butadiene	895.91	919.75	9.9E-04	0.32
Carbonyl Sulfide	2026.14 -	2085.23	3.7E-03	0.13
Chlorobenzene	1069.86	1103.34	6.9E-04	0.36
Ethyl Benzene	2959.43	2997.25	2.0E-03	1.13
Ethyl Chloride	943.43	1000.16	5.7E-04	0.86
Ethylene Dibromide	1167.96	1208.92	1.9E-03	1.48
n-Hexane	2835.27	3005.43	1.9E-03	0.29
Methyl Bromide	2948.11	2972.53	2.2E-03	2.72
Methyl Chloride	1017.96	1020.72	2.7E-04	0.95
Methyl Ethyl Ketone	1140.7	1222.63	2.0E-03	1.57
Methyl Isobutyl Ketone	2872.05	2994.95	2.1E-03	0.93
Methyl Methacrylate	1137.5	1232.04	2.4E-03	0.29
Methylene Chloride	743.96	769.17	2.8E-03	0.29
2-Nitropropane	831.47	868.5	1.4E-03	1.71
Propylene Dichloride	996.86	1038	8.7E-04	1.10
Styrene	974.29	1006.59	4.6E-04	0.62
Tetrachloroethylene	899.2	925.2	9.9E-04	0.10
Toluene	3018.19	3054.7	3.7E-03	1.87
1,1,2-Trichloroethane	916.98	956.37	9.2E-04	0.70
Trichloroethylene	919.7 -	959.88	8.6E-04	0.18
2,2,4-Trimethylpentane	2861.57	3009.23	2.0E-03	0.25
Vinyl Acetate	1132.83	1161.19	1.0E-03	0.09
Vinyl Bromide	939.59	944.72	2.8E-04	0.10
Vinyl Chloride	894.43	899.25	2.1E-04	0.39
Vinylidene Chloride	1059.44 -	1113.01	8.4E-04	0.24
O-xylene	2859.84	3095.04	3.1E-03	2.12
P-xylene	770.61 -	819.06	2.3E-03	1.39
Carbon Disulfide	2171.64 -	2198.03	1.1E-03	1.11

See Section 4.8, Screening For HAPs.

TABLE 3-14. CONTINUED

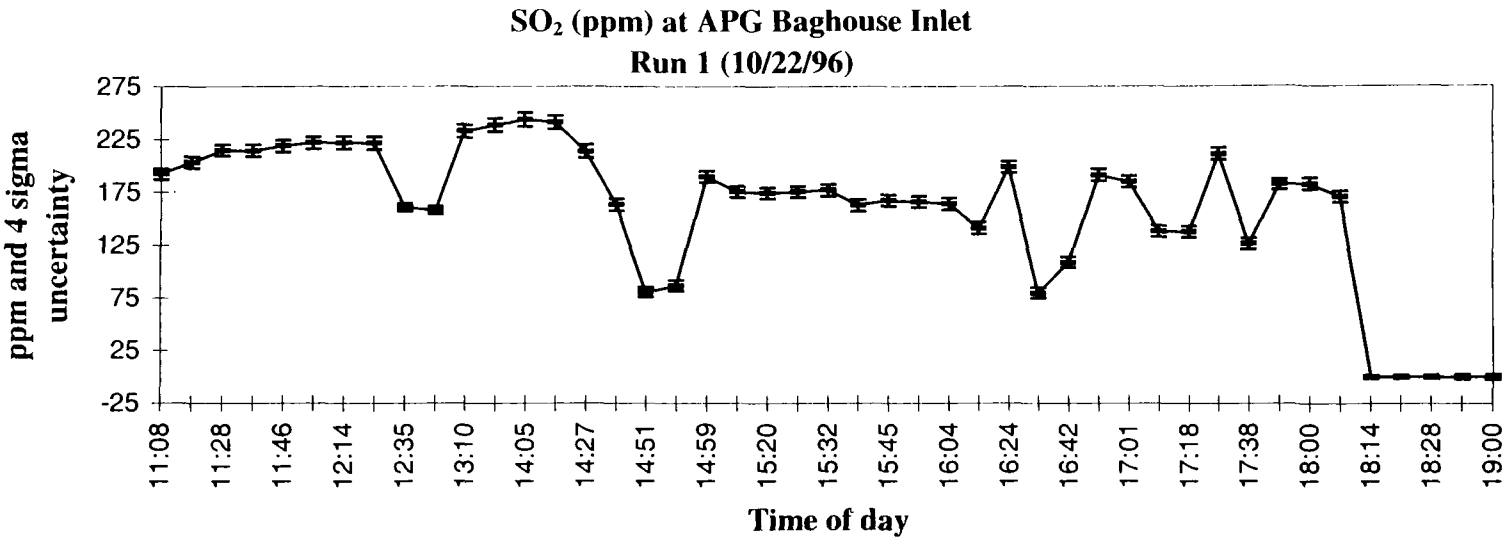
Compound	Cooling Stack Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Carbon Tetrachloride	793.89	800.58	3.3E-03	0.07
Chloroform	758.21 -	781.25	1.5E-03	0.08
Cumene	2951.21	2998.48	2.2E-03	0.60
1.2-Epoxy Butane	902.37	919.7	9.9E-04	0.78
Ethylene Oxide	866.9	875	4.5E-04	0.12
Methanol	2807.91	3029.4	2.4E-03	2.07
Methyl Chloroform	1057.95	1105.3	7.5E-04	0.14
Methyl Iodide	1250.18	1253.53	5.4E-04	0.53
Methyl t-Butyl Ether	1070.6	1109	7.7E-04	0.14
Propylene Oxide	2875.59	3097.75	3.3E-03	2.26
M-xylene	2910.25	2952.78	2.1E-03	1.39
Acetone	1182	1255.03	3.2E-03	1.52
Acetaldehyde	1092.5	1141	1.1E-03	1.65
Acetophenone	1140.4 -	1286.06	4.2E-03	0.59
Acrylic Acid	1104.89	1164.68	1.0E-03	0.14
Aniline	1102.9	1123.63	1.0E-03	0.36
Benzotrichloride	866.5	877.9	4.9E-04	0.10
Benzyl Chloride	3027.52	3109.06	4.3E-03	3.64
Bis(chloromethyl)ether	1068.78	1154.25	9.3E-04	0.11
Chloroacetic acid	1094.97	1124.12	1.0E-03	0.23
2-Chloroacteophenone	1274.39	1285.42	4.0E-03	0.79
Chloromethyl methyl ether	1111.02	1146.08	1.1E-03	0.17
Chloroprene	875.9	878.8	9.1E-05	0.02
o-Cresol	1092.8	1114.07	7.7E-04	0.35
m-Cresol	1139.68	1172.77	9.6E-04	0.18
p-Cresol	1159.1	1185.5	1.5E-03	0.17
1.2-Dibromo-3-chloropropane	1134.26	1175.42	1.7E-03	2.01
1.4-Dichlorobenzene	995.96	1031.06	7.2E-04	0.33
Dichloroethyl ether	1109.35	1155.04	1.1E-03	0.13
1.3-Dichloropropene	768 -	791	1.4E-03	0.31
Dichlorvos	967.79 -	1000.25	4.4E-04	0.07
N.N-Diethyl aniline	2655.32	3156.07	3.5E-03	2.12
Dimethyl carbamoyl chloride	1068.78	1114.47	7.5E-04	0.11
Dimethyl formamide	1057.8	1103.9	7.5E-04	0.18
1,1-Dimethyl hydrazine	885.12	936.33	1.2E-03	0.56

See Section 4.8, Screening For HAPs.

TABLE 3-14. CONTINUED

Compound	Cooling Stack Analytical Region (cm^{-1})		RMSD	Estimated Uncertainty (ppm)
Dimethyl phthalate	1157.86	1254.16	3.1E-03	1.75
1,4-Dioxane	2861.1	2864.8	6.9E-04	0.11
Epichlorohydrin	943.52	981.73	5.7E-04	0.49
Ethyl Acrylate	1181.93	1210	1.9E-03	0.10
Ethylene Dichloride	1227.88	1241.5	2.0E-03	1.16
Ethylidene dichloride	1041.11	1080.5	9.9E-04	0.44
Formaldehyde	2788.33 -	2842.2	1.0E-03	1.06
Hexachlorobutadiene	976.9	997.7	4.5E-04	0.07
Hexachlorocylcopentadiene	1136.92	1148.34	6.9E-04	0.13
Hexachloroethane	779.26	797.38	1.9E-03	0.04
Hexamethylphosphoramide	949.42	1019.53	5.4E-04	0.08
Hydrochloric Acid	2817.35	2823.26	4.5E-04	0.47
Isophorone	2681.2	3130.6	3.3E-03	2.23
Maleic Anhydride	838.45 -	841.3	4.3E-04	0.04
Methyl hydrazine	2683	3061.78	2.7E-03	3.09
Naphthalene	779.31	783.55	6.9E-04	0.07
Nitrobenzene	841.7	861.39	1.7E-03	0.72
N-Nitrosodimethylene	928	1085.28	1.4E-03	0.45
N-Nitrosomorpholine	892.23	1024.64	1.0E-03	0.43
Phenol	1162.67	1195.76	2.1E-03	0.35
beta-Propiolactone	1034.68	1152.25	1.2E-03	0.23
Propionaldehyde	2546.18	3114.35	3.7E-03	4.22
1,2-Propylenimine	817.57	821.31	5.0E-04	0.23
Quinoline	800.19	803.73	1.0E-03	0.16
Styrene Oxide	861.39	903.93	7.1E-04	0.53
1,1,2,2-Tetrachloroethane	794.92	824.07	2.2E-03	0.52
2,4-Toluene diisocyanate	1066.71	1086.8	5.4E-04	0.45
o_Toluidine	2858.5	2951.85	1.7E-03	1.52
1,2,4-Trichlorobenzene	1086.21	1114.37	8.0E-04	0.21
2,4,5-Trichlorophenol	1178.04	1204.16	1.9E-03	0.60
2,4,6-Trichlorophenol	856.27	863.36	5.8E-04	0.15
Triethylamine	2756.62	2839.34	1.1E-03	0.50
Ammonia	893.1 -	926	1.0E-03	0.66

See Section 4.8, Screening For HAPs.



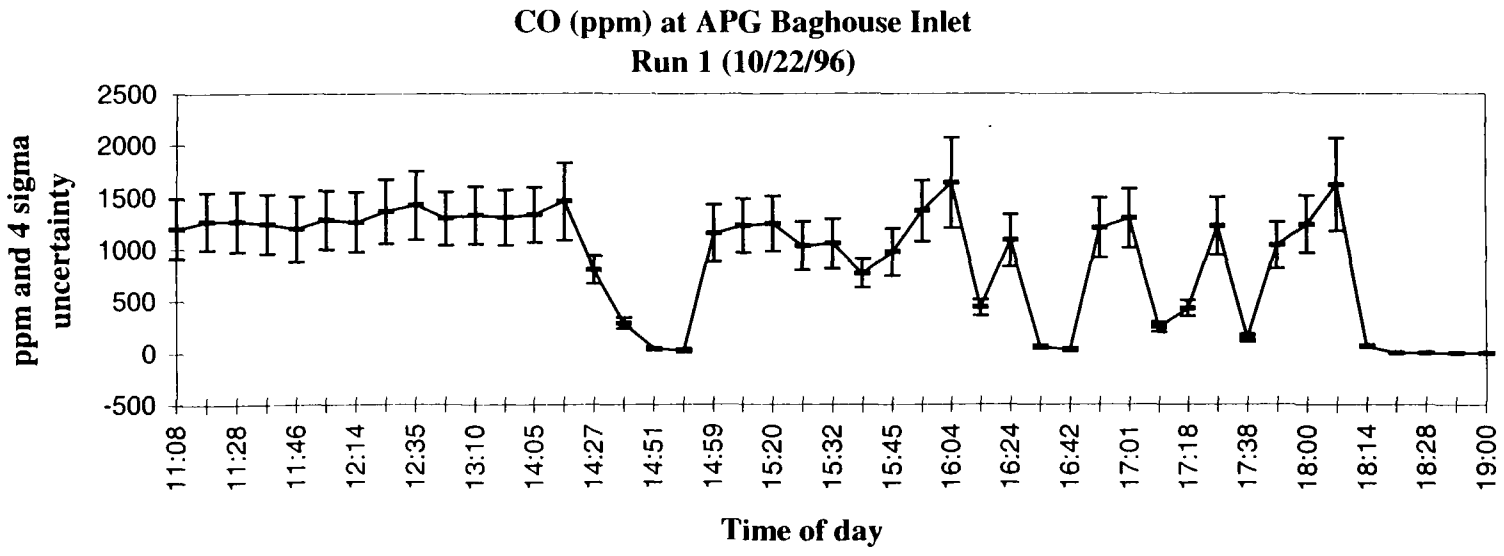


Figure 3-1. Concentrations at APG baghouse inlet. Run 1, 10/22/96

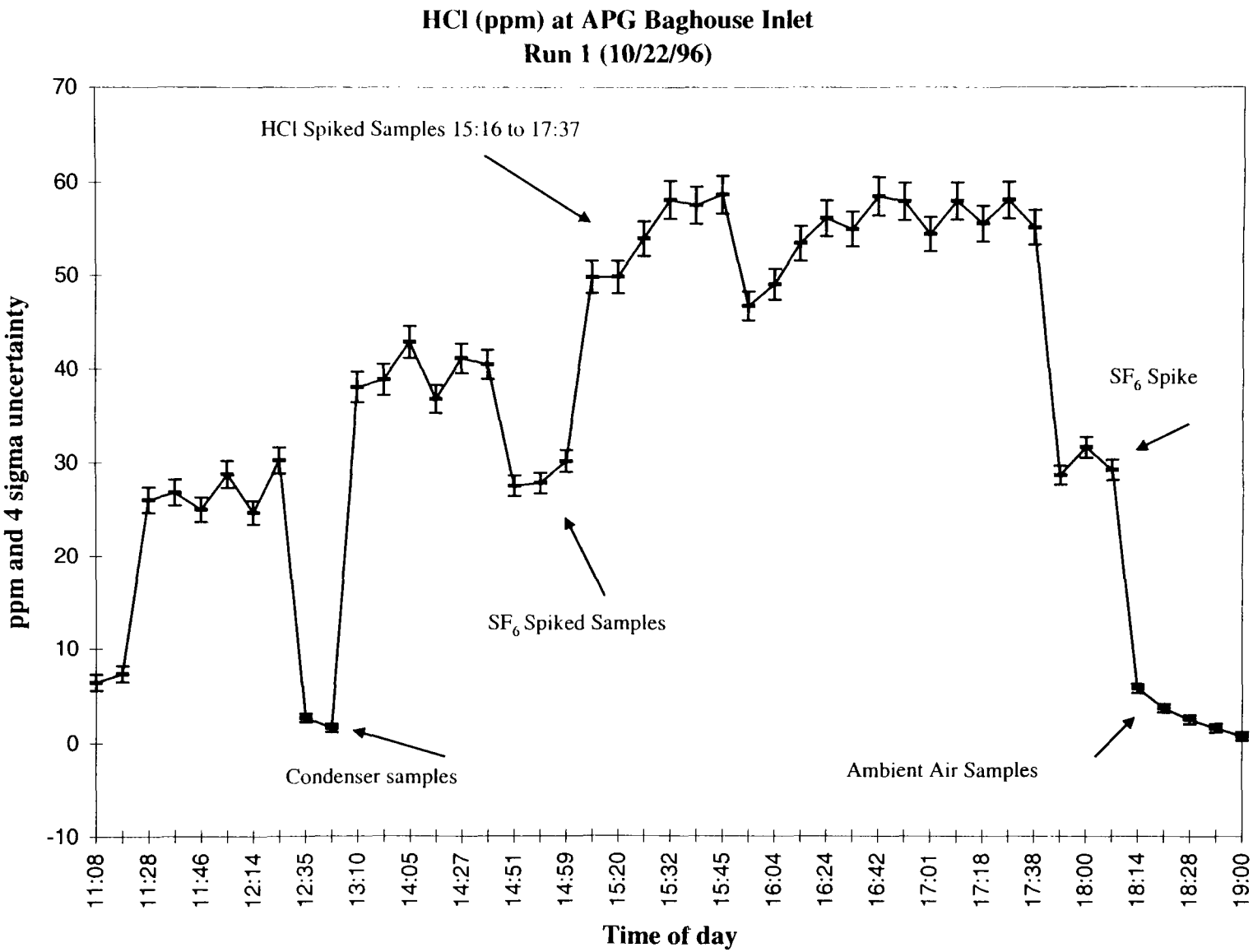
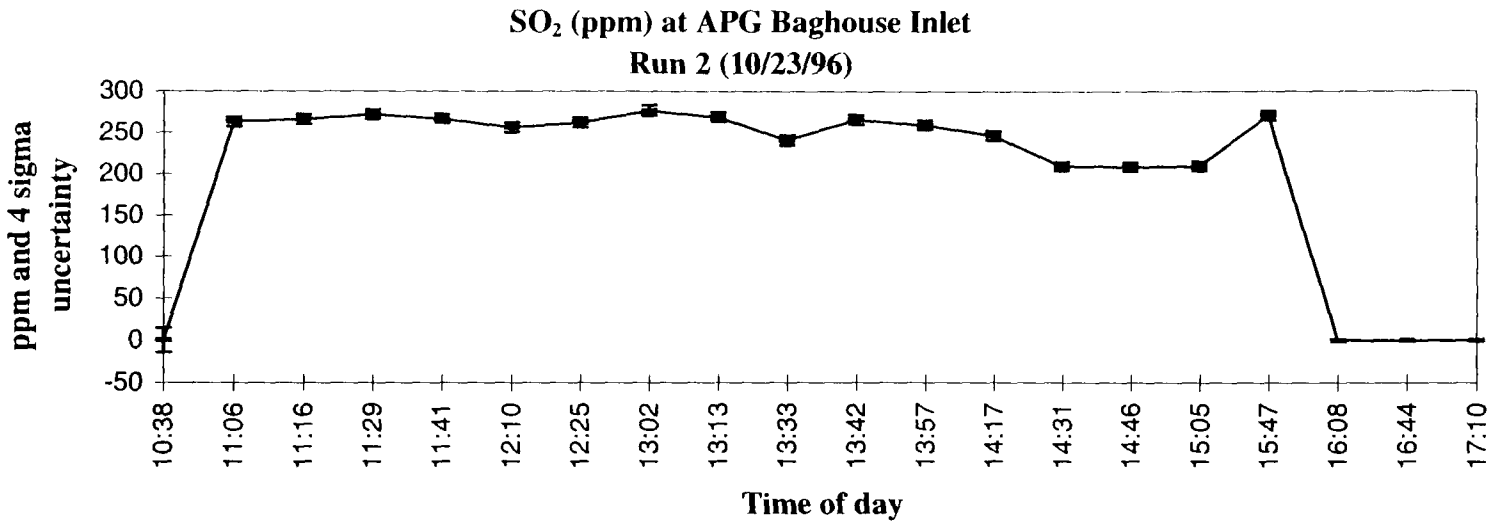


Figure 3-2. Concentrations at APG baghouse inlet. Run 1, 10/22/96



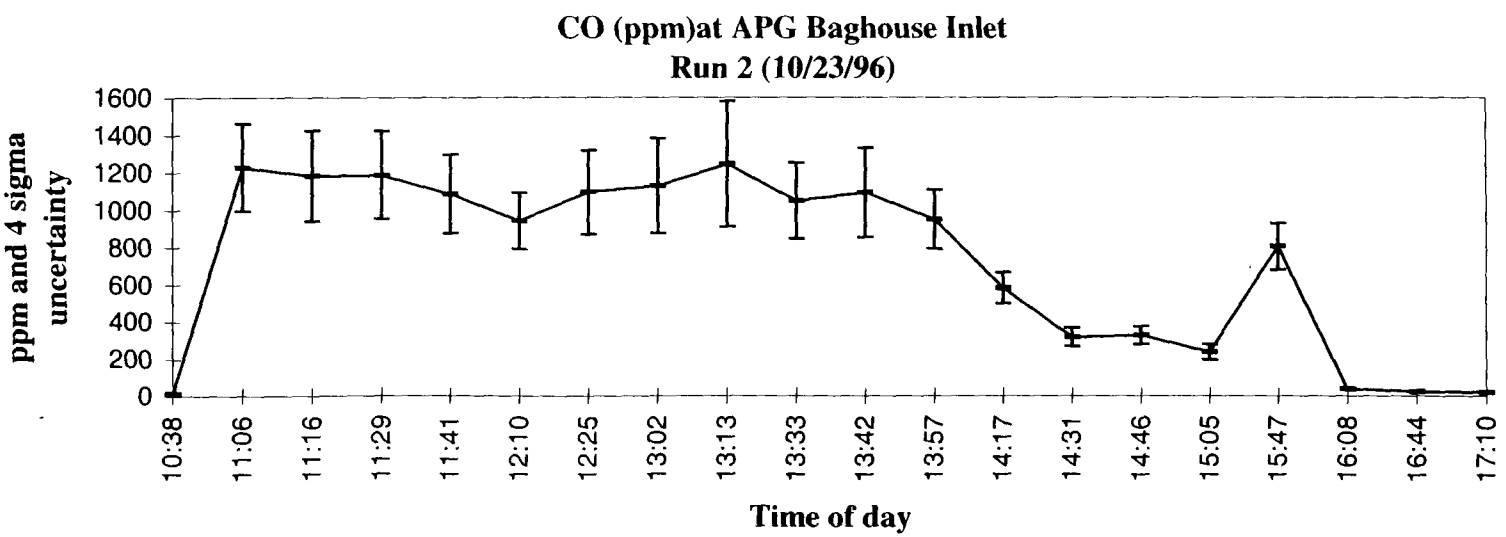


Figure 3-3. Concentrations at APG baghouse inlet. Run 2, 10/23/96

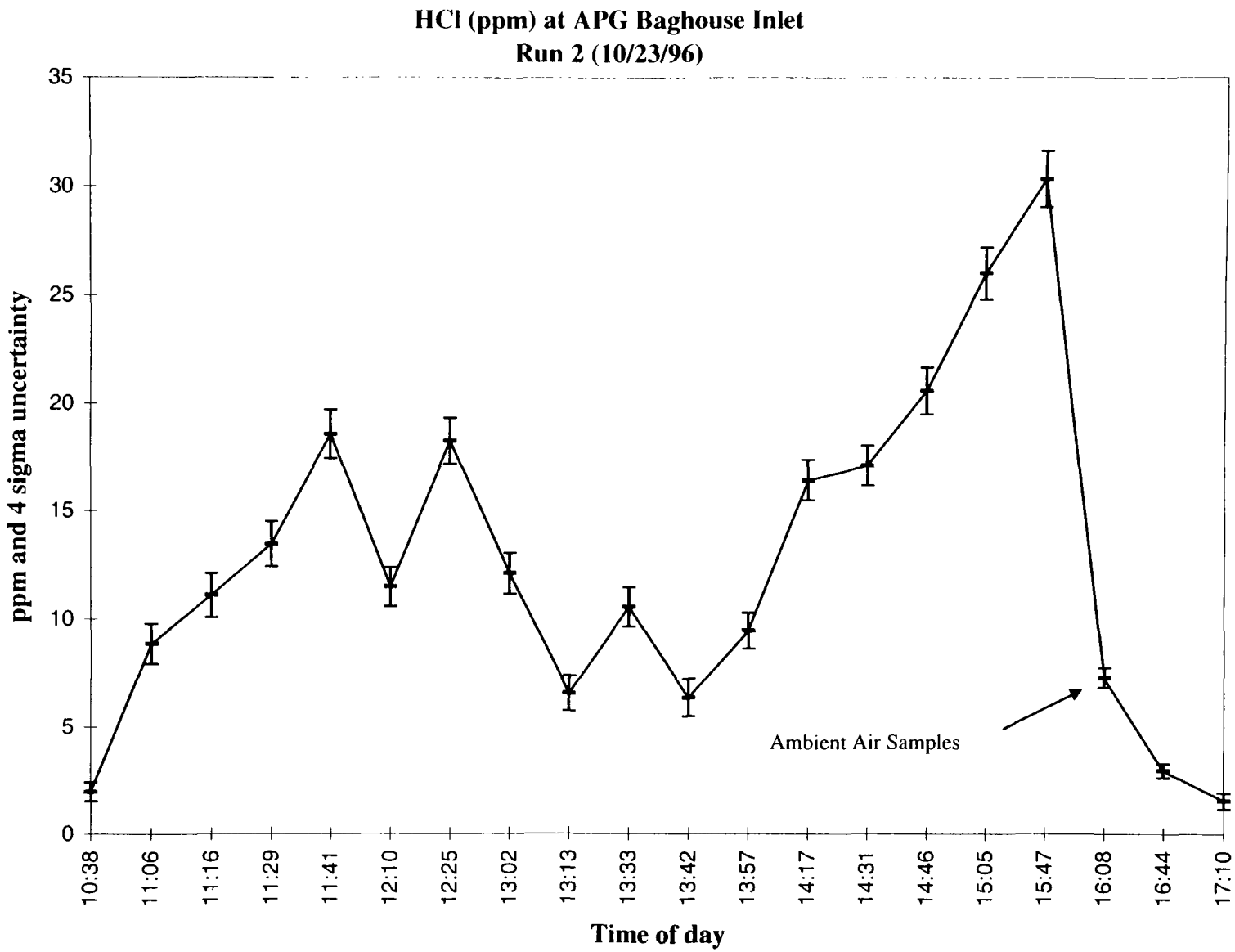
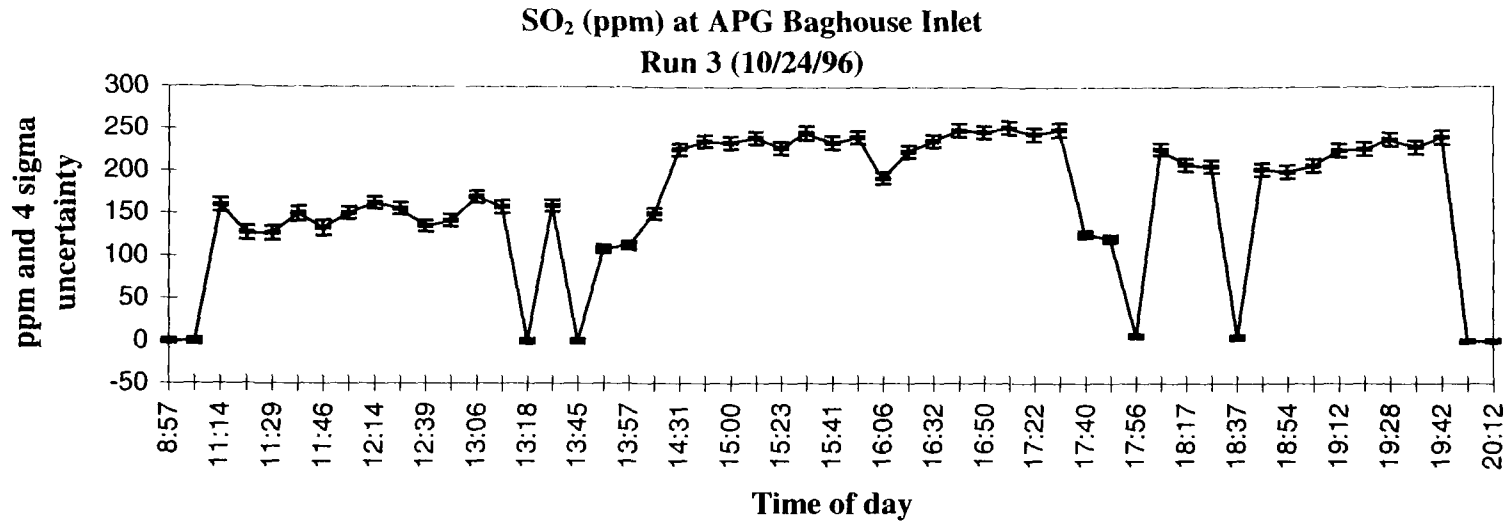


Figure 3-4. HCl concentrations at APG baghouse inlet. Run 2, 10/23/96



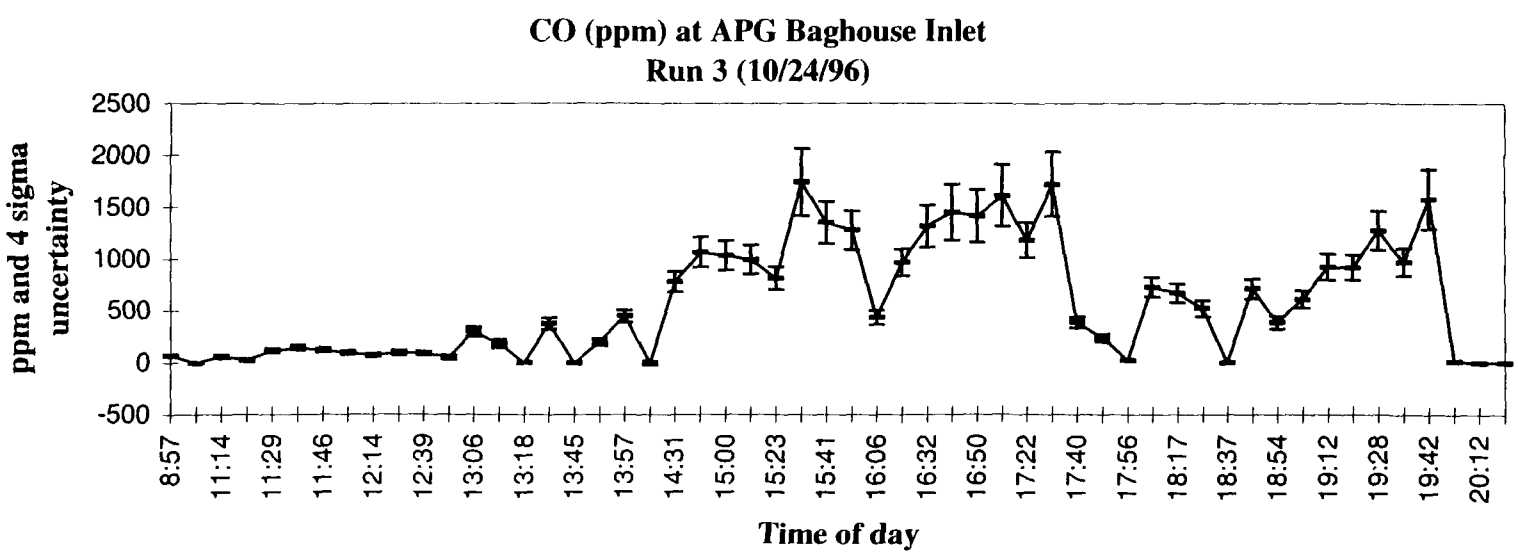


Figure 3-5. Concentrations at APG baghouse inlet. Run 3, 10/24/96

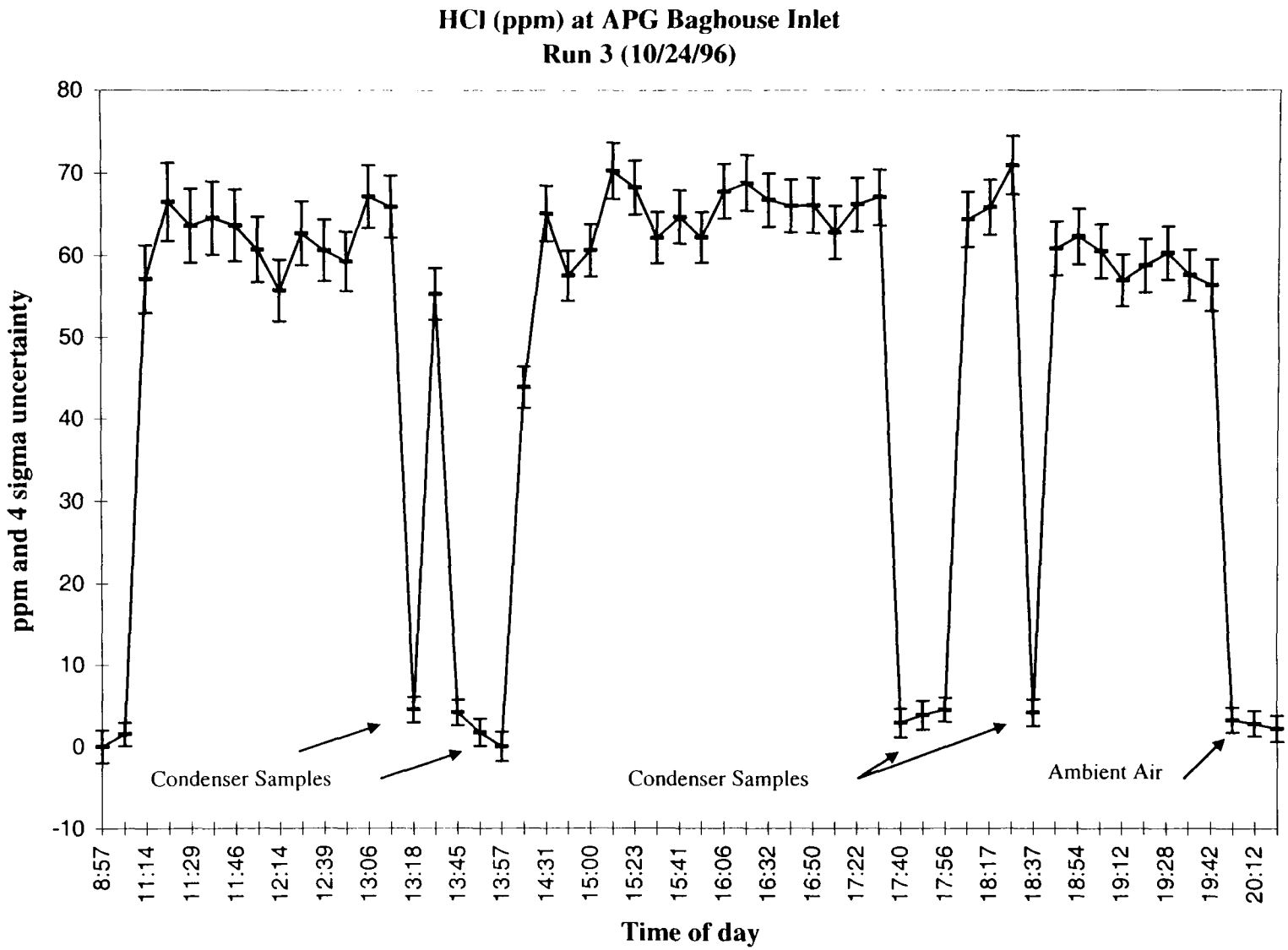
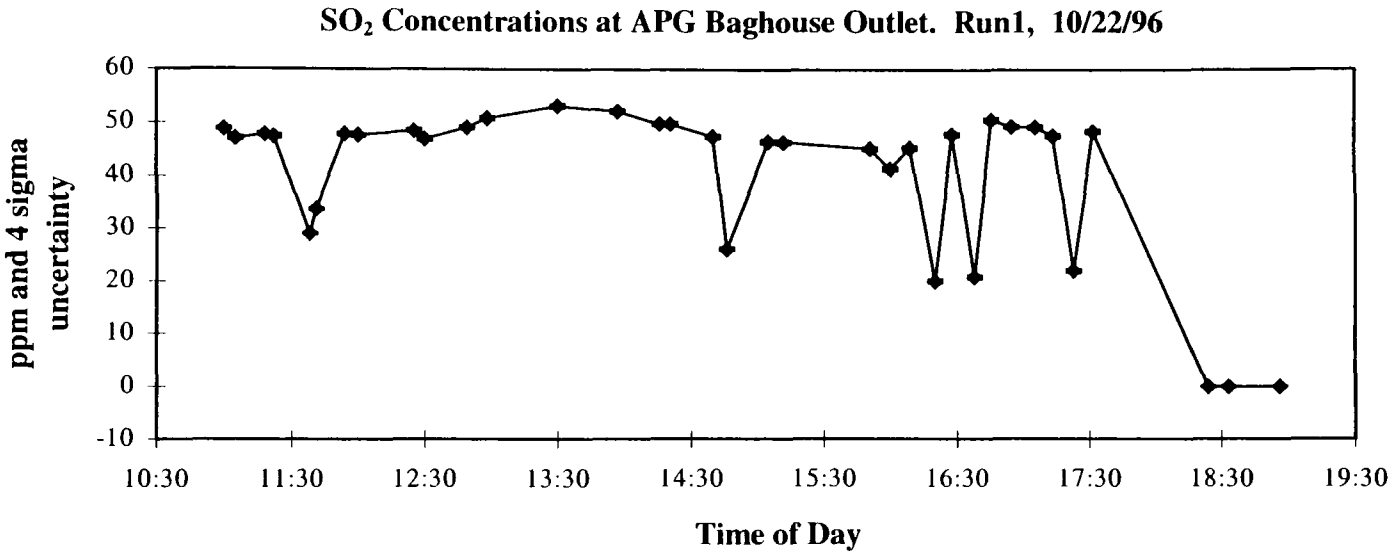


Figure 3-6. HCl concentrations at APG baghouse inlet. Run 3, 10/24/96



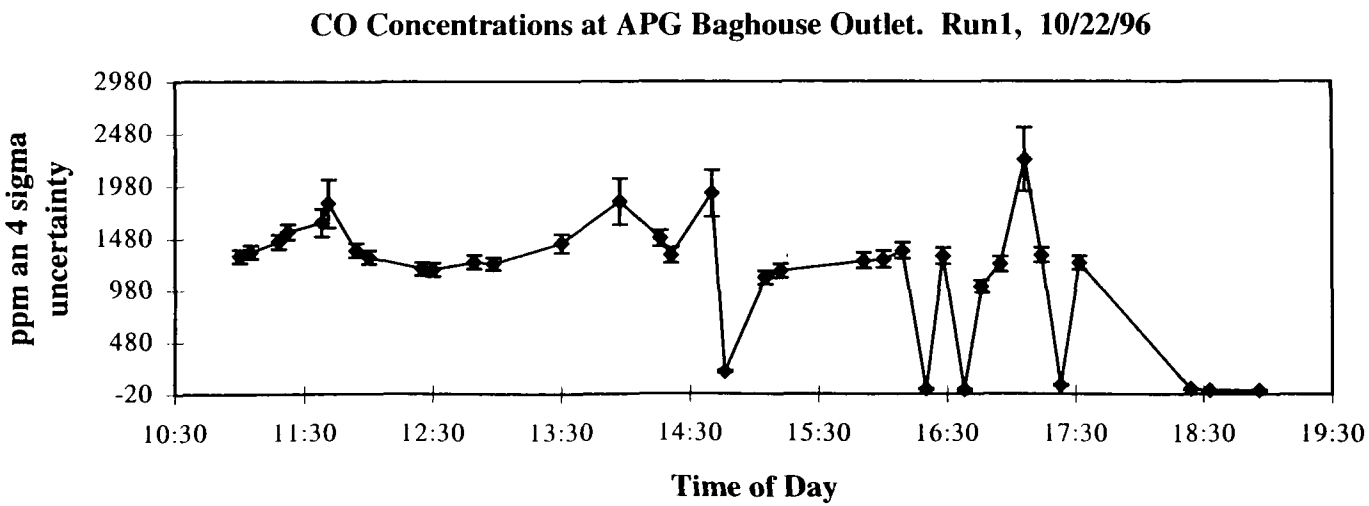


Figure 3-7. Concentrations at APG baghouse outlet. Run 1, 10/22/96

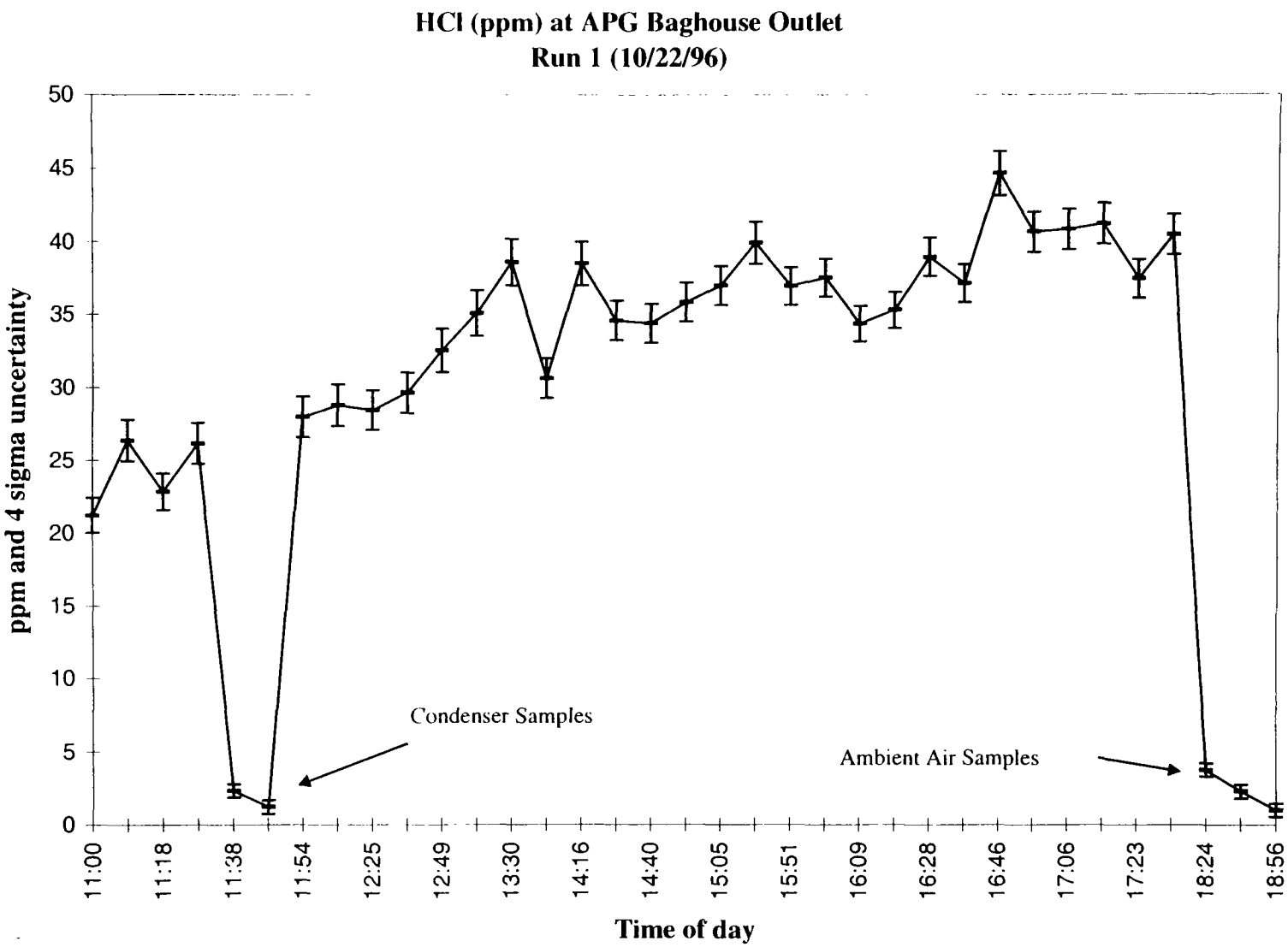
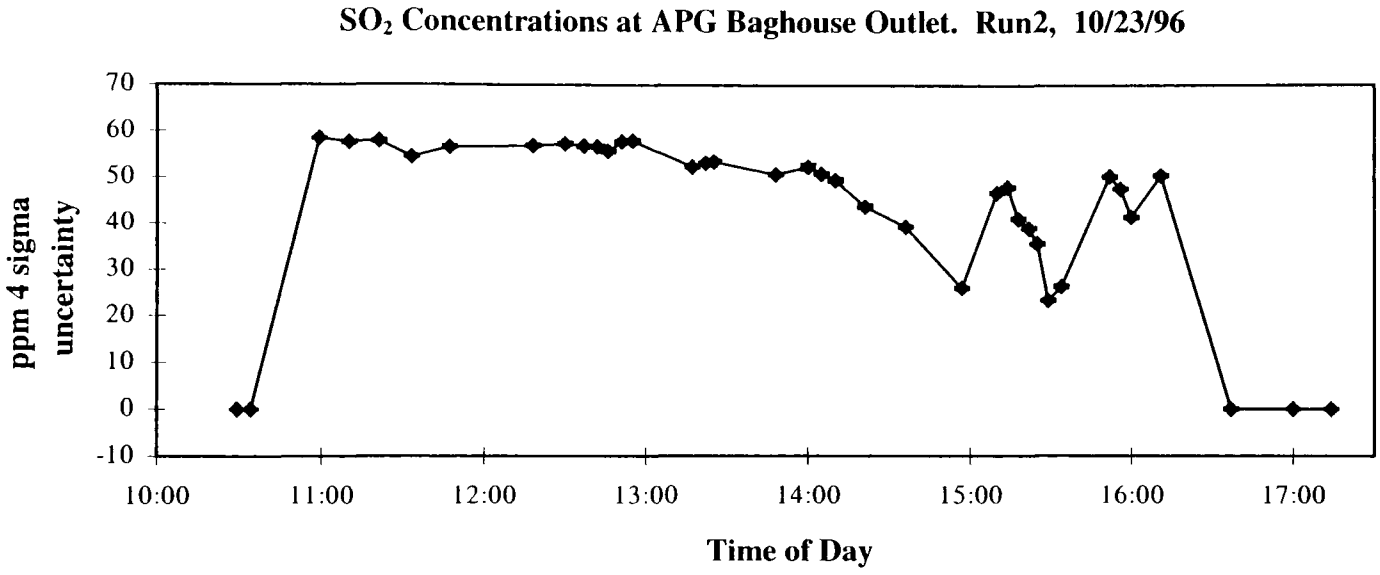


Figure 3-8. HCl concentrations at APG baghouse outlet. Run 1, 10/22/96



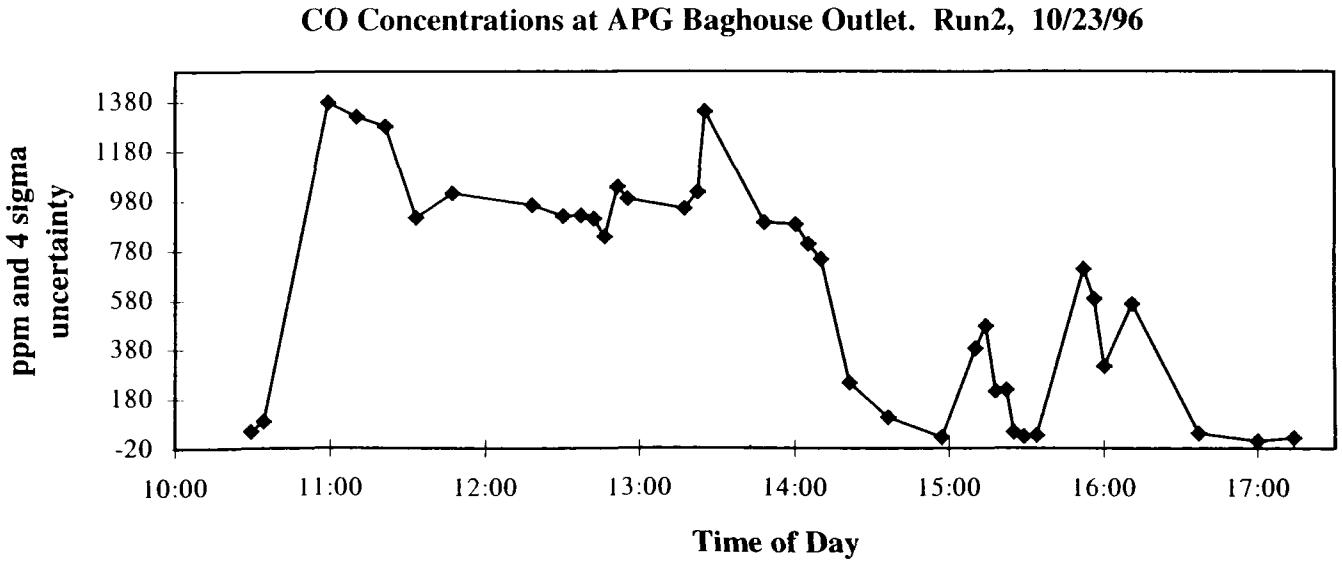


Figure 3-9. Concentrations at APG baghouse outlet. Run 2, 10/23/96

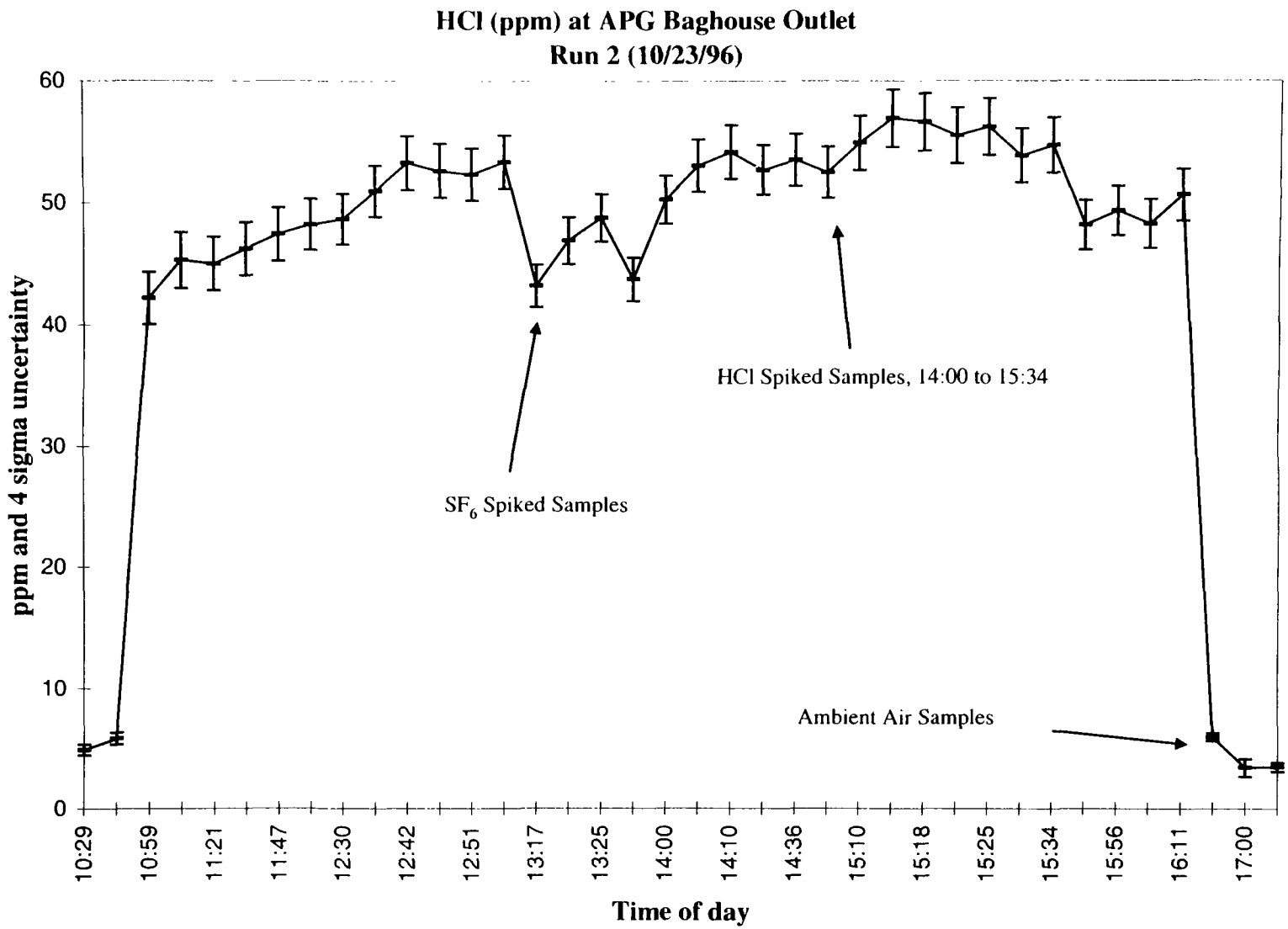
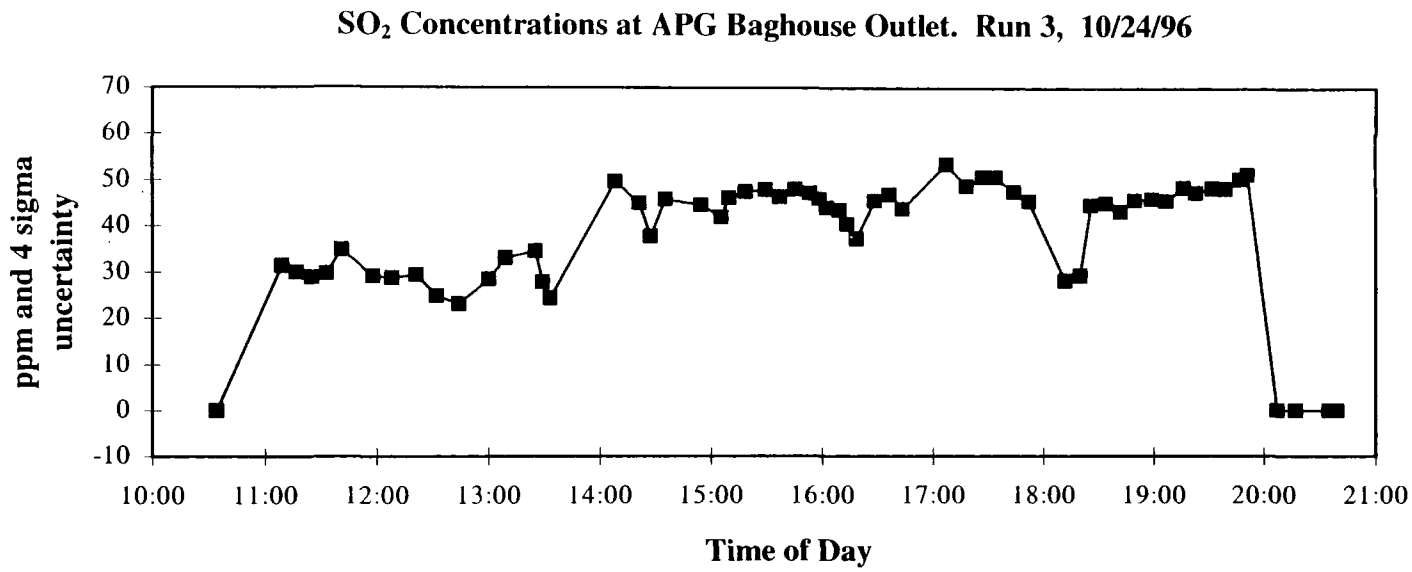


Figure 3-10. HCl concentrations at APG baghouse outlet. Run 2, 10/23/96



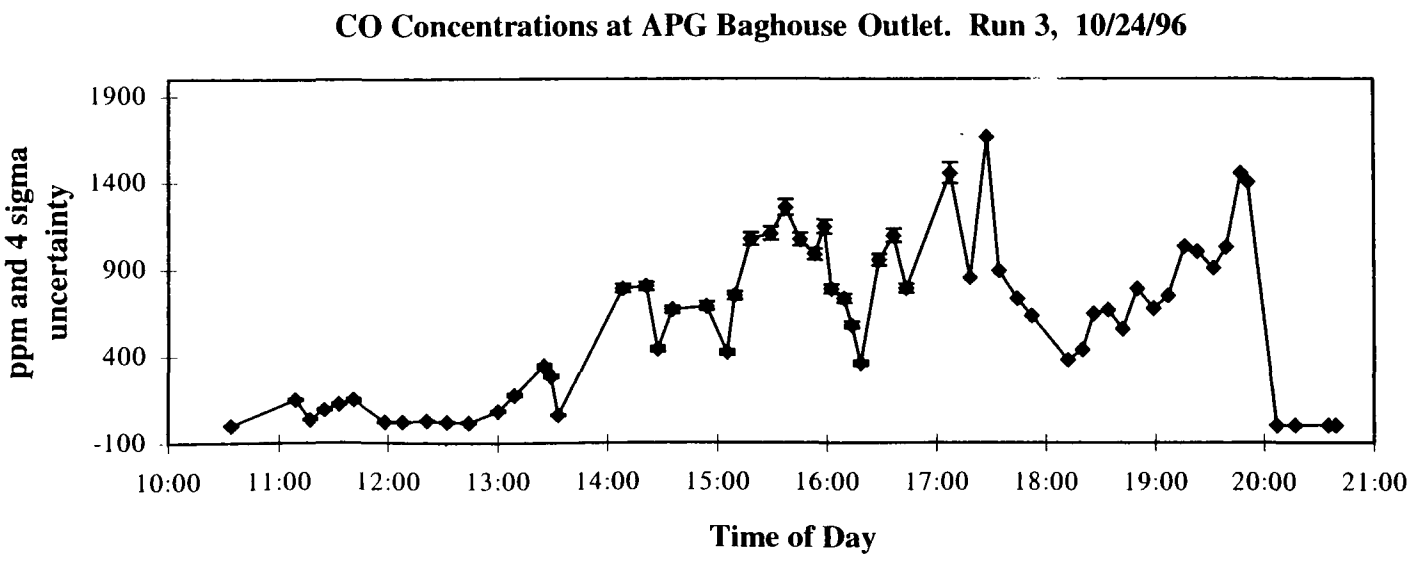


Figure 3-11. Concentrations at APG baghouse outlet. Run 3, 10/24/96

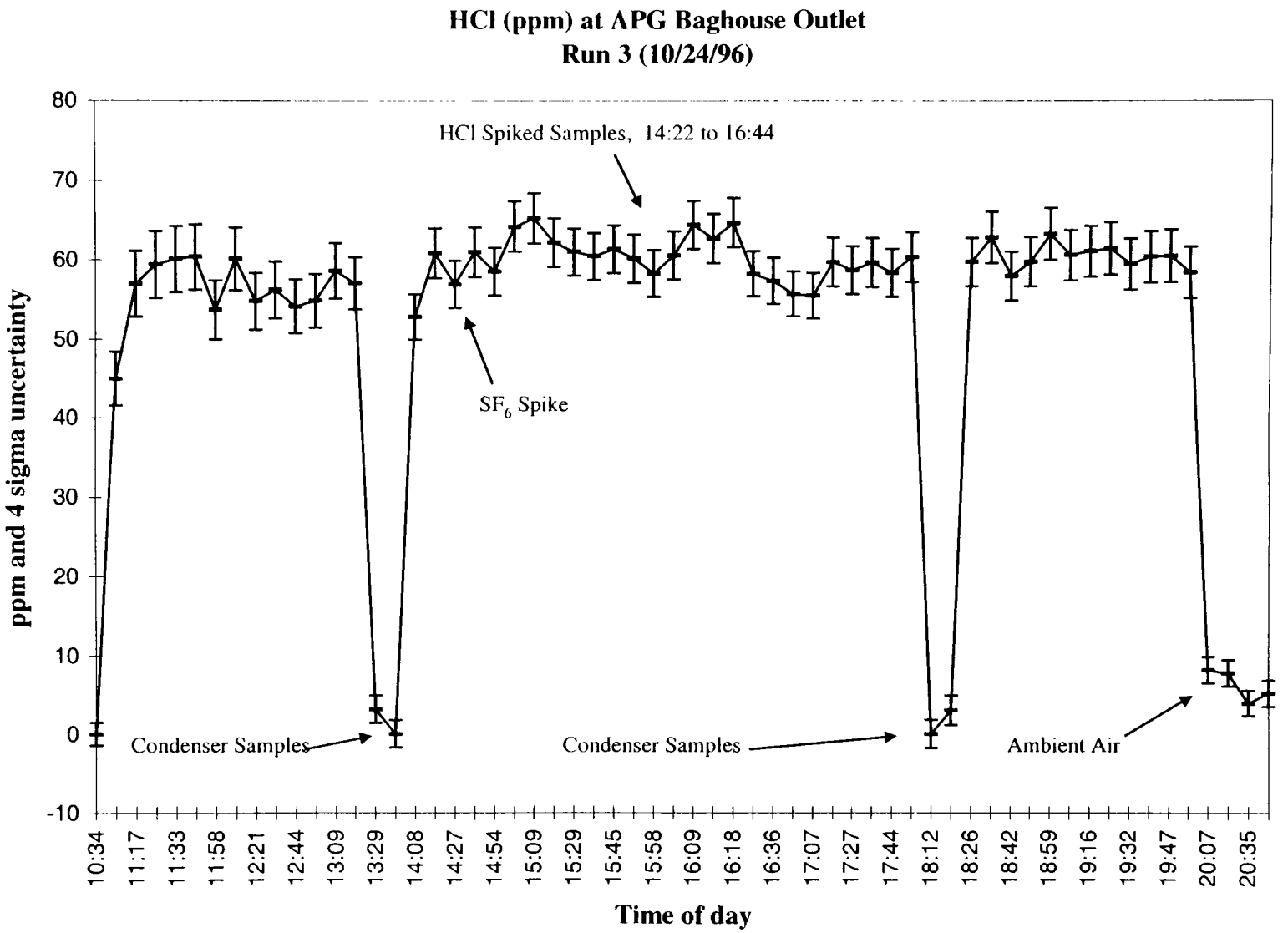


Figure 3-12. HCl concentrations at APG baghouse outlet. Run 3, 10/24/96

4.0 FTIR TEST PROCEDURES

A heated sample delivery system (Figure 4-1) was used to extract flue gas through a stainless steel probe and transport the flowing sample gas through a heated teflon sampling line to a heated gas distribution manifold. Valves in the manifold were used to direct the sample flow (or a calibration standard) to the FTIR gas cell.

4.1 SAMPLING SYSTEM DESCRIPTION

This description refers to Figure 4-1.

4.1.1 Sampling System Components

The sample was extracted through a single port using 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 sample pump outlet was connected to the sample manifold. The sample stream passed through a secondary Balston particulate filter immediately after entering the manifold box. The manifold is constructed of stainless steel 3/8-in tubing and contains 4-way valves and heated rotameters (0 to 20 LPM) to allow the operator to control sample flow to the FTIR cell. A heated 1/4-in diameter 20-ft long Teflon jumper line connected the manifold to the inlet of the FTIR gas cell. The manifold was maintained at 300° to 310°F.

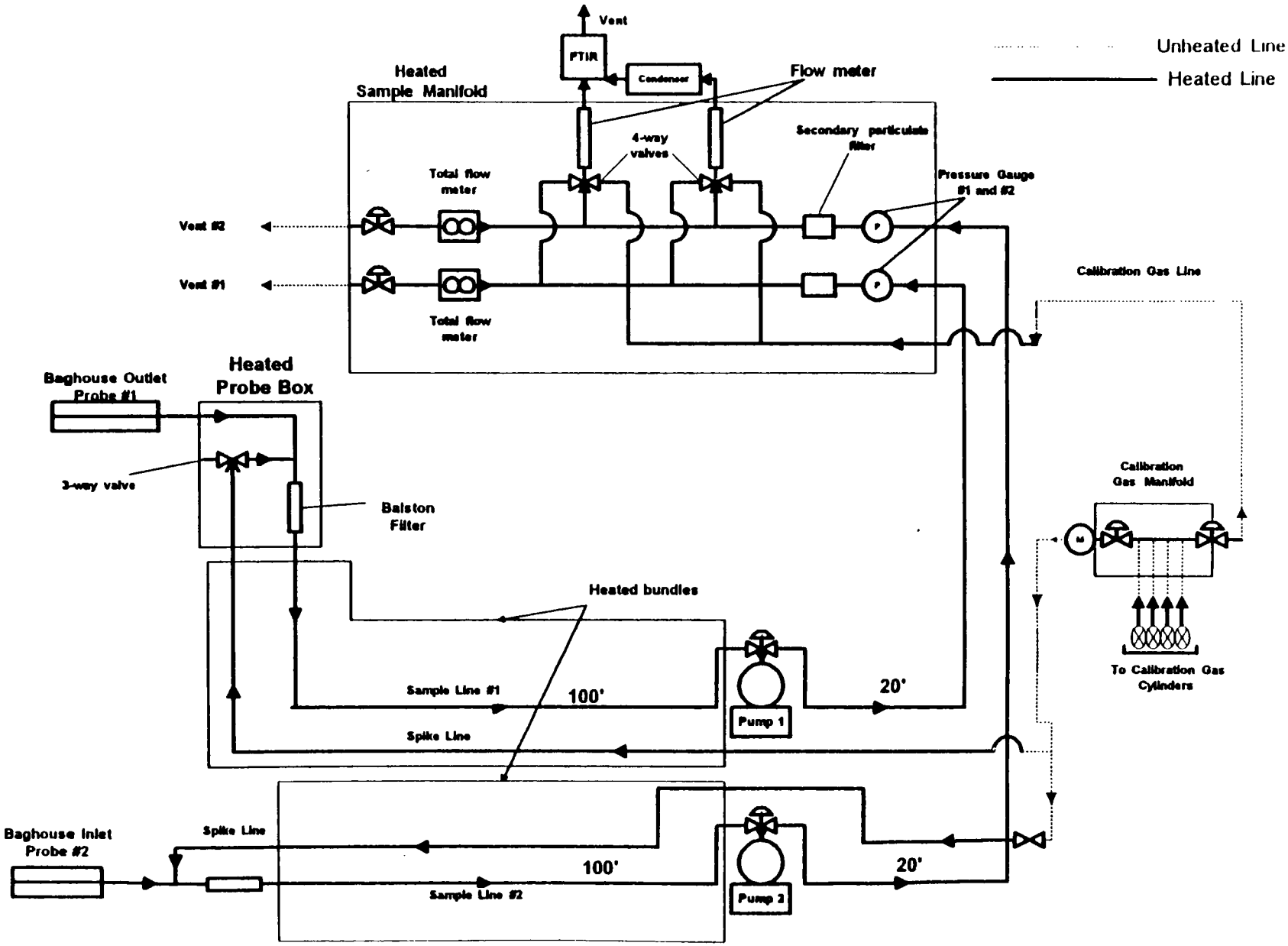


Figure 4-1. FTIR sampling system configuration for test at APG Lime Plant.

4.1.2 Sample Conditioning

Some samples were passed through a chilled condenser system to remove moisture before going to the FTIR cell. The condenser inlet was connected to a second outlet of the gas distribution manifold through a another heated 1/4-in Teflon. A 4-way valve on the manifold controlled sample flow to the condenser. The condenser outlet was connected by a Teflon line to the inlet of the FTIR gas cell.

Since the condenser is not effective for measuring HCl or other water-soluble compounds, it was not used extensively. The primary benefit of the condenser is in lowering moisture to better reveal spectral features of gas phase compounds, such NO_x and SO_2.

4.2 SAMPLING PROCEDURE

This test required two sampling configurations.

4.2.1 Testing Two Locations Simultaneously

The inlet and outlet to the baghouse were sampled with the configuration shown in Figure 4-1. Two separate sample lines, probes, and pumps were used, one for each location. Both sampling lines were connected to the common sampling manifold. Each line had a pressure gauge at the manifold inlet and a rotameter at the manifold outlet. A vent turn-valve was used to independently control and monitor the total sample flow through either sample line. The 4-way valves, at the manifold outlet leading to the FTIR cell and leading to the condenser, could be turned to select gas from sample line 1, gas from sample line 2, calibration standard gas, or could be closed.

The baghouse outlet sample probe was connected to a heated box that contained the initial Balston particulate filter. The initial particulate filter at the baghouse inlet probe was wrapped in heat tape and insulated. The gas temperature at the inlet was about 500°F so it was necessary to let the gas cool in the unheated probe to about 350°F before reaching the Teflon line. Each location was within 100-ft of the FTIR trailer position.

A 100-ft spike line went to each location and connected to the sample line at a "tee" just upstream of the particulate filter. Spike flow to either location was controlled by the mass flow meter at the outlet to the calibration manifold. Spike flow to the baghouse inlet could be turned on or off with a 2-way valve in the FTIR trailer. Spike flow to the baghouse outlet sample line was turned on or off with a 3-way valve in the heated probe box. For each location the spike and

sample lines were contained in the same heated bundle and the temperature was regulated by the same heat controller. Either sample line could be spiked with the pre-heated HCl standard at a controlled dilution ratio.

4.2.2 Testing a Single Location

The cooling stack was sampled alone. This configuration was the same as that shown in Figure 4-1 for the baghouse outlet location. The same spike line and valve configuration were also used.

4.3 SAMPLING PROCEDURES

Figure 4-2 is a schematic showing the FTIR instrument configuration and connections with the sample manifold and condenser.

Because HCl was prominent in the emissions, most of the measurements were performed using a batch sampling procedure to collect a spectrum of a static sample. Some measurements were performed with the sample flowing through the cell. Some samples were diluted in the cell with dry nitrogen and some were passed through a condenser.

4.3.1 Batch Sampling

The batch sampling procedure was used to collect samples in the FTIR cell (Section 8.7.1 in EPA Method 320 in Appendix D and reference 1). Sample gas continuously flowed from the probe through the sample line, through the manifold and out a manifold vent (Figure 4-1). The 4-way valve on the manifold was turned to fill the FTIR cell to ambient pressure, which was about 720 mm Hg. The total flow meter before the vent was monitored to ensure that a positive flow was always directed out the vent during sampling. The cell inlet valve was then closed to isolate the sample and the sample spectrum was recorded. Then the cell was evacuated for the next sample.

4.3.2 Flow Through Measurements

The cell was filled as in the batch sampling procedure. The sample inlet valve was kept open and the cell outlet valve was also opened to allow gas to pass through the cell. The sample was maintained at ambient pressure by having the outlet valve partially open to the vacuum pump. The inlet sample flow valve was adjusted until the pressure gauge was stable at ambient pressure. The spectrum of the sample was recorded, and then the cell was evacuated for the next sample.

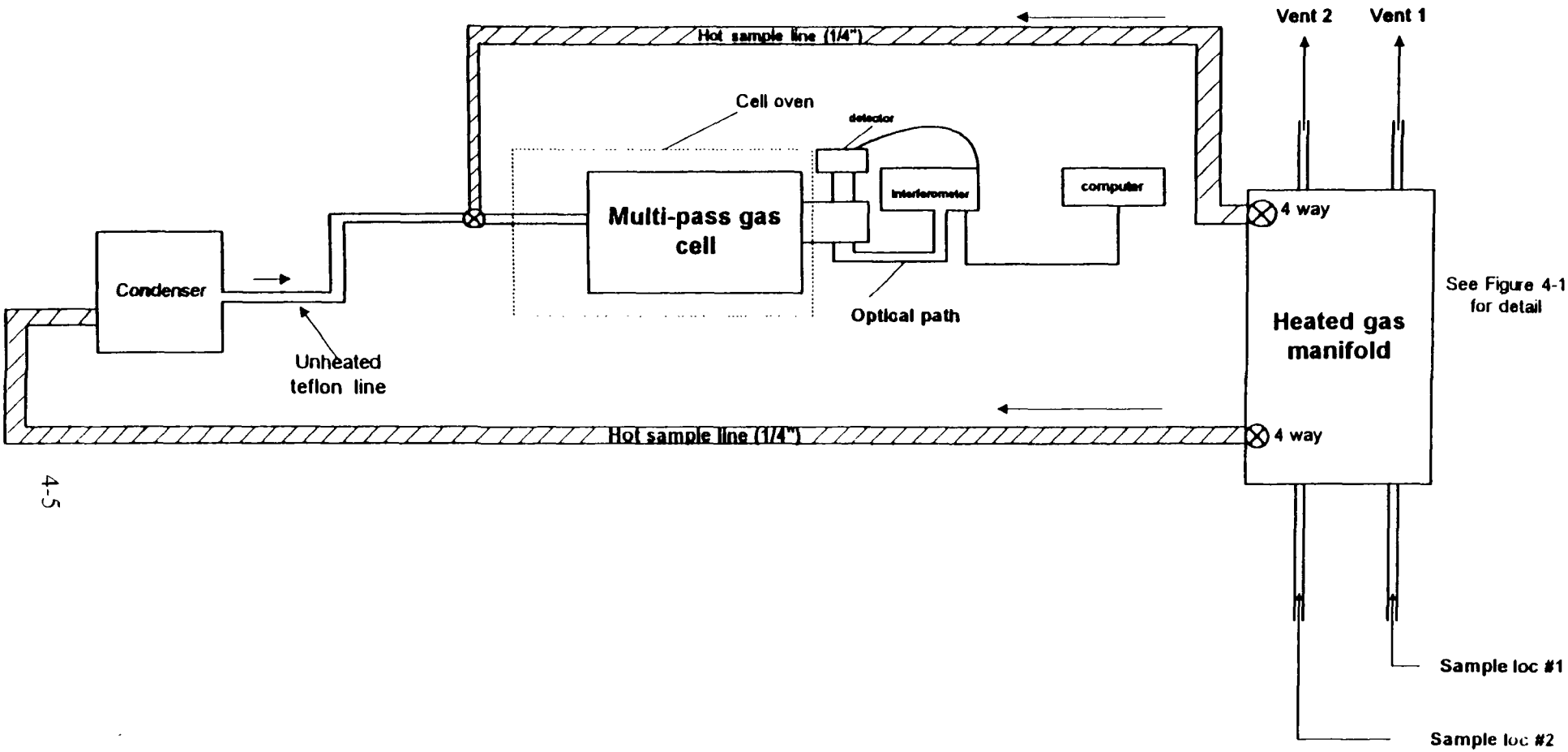


Figure 4-2. FTIR instrument and sampling configuration.

4.3.3 Dilution Samples

Diluting the sample is a procedure for reducing spectral interference from moisture or CO_2. This procedure is only effective if the target analyte is present at a high enough concentration to be detected after the dilution. The objective was to dilute the sample to 1/2 to 1/4 its original concentration.

The cell was partially filled with dry nitrogen and the cell pressure was recorded. Then the cell was filled to ambient pressure with sample gas. The final pressure was recorded, the spectrum of the static sample was measured and the cell was evacuated for the next sample.

4.3.4 Condenser Samples

The condenser was not used extensively at the baghouse locations because it is not effective for measuring HCl. Directing the sample through a condenser can remove much of the moisture and improve the measurement sensitivity for analytes that pass through the condenser. Analytes that are water soluble, such as HCl, or have low vapor pressures at 32°F cannot be measured using a condenser system.

Sample was diverted to the condenser through a second 4-way valve on the main manifold (Figure 4-2). The valve was turned to direct sample from either location through the condenser. This could be done while untreated sample was sent to the FTIR cell through the other 4-way valve. After flow passed through the condenser for about 10 minutes, a 3-way valve at the cell inlet was turned to allow the condenser sample into the cell. The cell was filled to ambient pressure and the spectrum recorded using the batch sampling procedure.

Before and after sampling a location dry nitrogen was passed through the condenser and into the FTIR cell and a spectrum of the nitrogen was recorded. This was to verify that the condenser was not contaminating the samples.

4.4 ANALYTICAL PROCEDURES

Analytical procedures in the EPA FTIR Protocol[2] (Appendix D) were followed. Analytical programs were prepared after the field test was completed. The programs employed an automated routine to analyze the spectra using mathematical techniques based on a K-matrix analysis to determine analyte concentrations. The calculated concentrations were used to scale reference spectra, which were sequentially subtracted from the sample spectra. The subtracted residual baseline spectra were analyzed to estimate uncertainties in the reported concentrations.

K-matrix, and other quantitative methods, are described in references 3 and 4. Additional detail on the quantitative method is given in Appendix C.

4.5 FTIR SYSTEM

The FTIR system was a KVB/Analect RFX-40 interferometer. A heated variable path (D-22H) gas cell from Infrared Analysis, Inc contained the samples.

Two path lengths were used in this test The cell was adjusted to 40 beam passes for testing at the cooling stack and Runs 1 and 2 at the baghouse locations. The path length was decreased to 20 beam passes for Run 3 at the baghouse locations. The number of beams passes was measured using a helium-neon laser. 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. More detail on the path length determinations is given in Appendix C.

A mercury/cadmium/telluride (MCT) liquid nitrogen detector will be used. Spectra were collected at 1.0 cm^{-1}, the highest resolution of the RFX-40 system.

4.6 ANALYTE SPIKING

Hydrogen chloride was an important target analyte. It is reactive and water soluble. Sample flow and temperature influence whether HCl can quantitatively pass through the sampling system to the analyzer. An FTIR instrument is ideally suited to measure spiked samples because many analytes have very distinct infrared spectra and this is especially true of HCl.

The purpose of this procedure is to measure a gas standard directly with the analyzer and compare that measurement to one in a sample that has been spiked with a known concentration of the analyte. Ideally, the spike will comprise about 1/10 or less of the spiked sample.

The spike procedure follows Section 9.2 of EPA Method 320[1] (Appendix D). The SF_6 tracer gas was not contained in the same cylinder as the HCl standard. The tracer gas was first spiked from a cylinder standard of 4 ppm SF_6 in nitrogen. The total sample flow and the spike flow were continuously monitored and recorded while 3 separate spiked batch samples were collected and their spectra recorded. The SF_6 spike was then turned off and the HCl spike was turned on. The HCl spike flow was set at the same value as the SF_6 flow. At least three batch

samples spiked with HCl were collected and their spectra recorded while the total sample flow was continuously monitored and recorded. The HCl spike was then turned off and the procedure was repeated with the SF_6 standard to collect three more samples.

Three samples from the cooling stack were spiked with HCl. The baghouse inlet was spiked during Run 1 and the outlet was spiked during Runs 2 and 3. The sample flow was very consistent during Run 1 at the baghouse inlet and Runs 2 and 3 at the outlet. Since the spike flow rate was kept constant, it can be assumed the spike dilution ratio was also consistent. For this test the procedure of spiking the analyte and the tracer gas separately was effective. This is supported by the results of the SF_6 spike measurements before and after each spike run. The SF_6 results were consistent. The HCl flue gas concentrations also appeared to be consistent so even the sequential sampling procedure (Section 13.1.2 in EPA Method 320 in Appendix D) appears to have been effective for spiking with HCl in this test.

4.7 ANALYSIS OF SPIKED RESULTS

The statistical procedures in Section 6.3 of EPA Method 301 were followed to analyze the spiked and unspiked results. The application of these procedures to FTIR test data is described in Section 13 of EPA Method 320 in Appendix D.

4.8 SCREENING FOR HAPs

Estimated uncertainties for undetected compounds are presented in Tables 3-12 to 3-14.

After analysis, the residual sample spectra were screened for absorbances due to hazardous air pollutants in the EPA FTIR spectral library.

The residual spectra were produced by sequential subtractions of scaled reference spectra. Reference spectra were scaled by a factor equal to the ratio of the calculated sample concentration divided by the reference spectrum concentration (corrected for path length and temperature). The estimated uncertainty is determined primarily by the moisture in the sample gas. Higher moisture results in a higher calculated uncertainty.

The noise level in each analytical region of the residual spectra was taken as the root mean square deviation (RMSD) of the baseline. The RMSD was multiplied by the width (in cm^{-1}) of the analytical region. This value was compared to the integrated area in the same region of a reference spectrum of the compound. The noise was calculated from the equation:

$$RMSD = \left[\left(\frac{1}{n}\right)\Sigma_{i=1}^{n}(A_i - A_M)^2\right]^{\frac{1}{2}} \quad (1)$$

where:

RMSD	=	Root mean square deviation in the absorbance values within a region.
n	=	Number of absorbance values in the region.
A_i	=	Absorbance value of the ith data point in the analytical region.
A_M	=	Mean of all the absorbance values in the region.

The estimated uncertainty for a non-detect is given by:

$$U_{ppm} = \frac{RMSD \ x \ (x_2 - x_1)}{Area_R} \ x \ CON_R \quad (2)$$

where:

Uppm	=	Noise related uncertainty in ppm.
X_2	=	Upper limit, in cm^{-1}, of the analytical region.
X_1	=	Lower limit, in cm^{-1}, of the analytical region.
$Area_R$	=	Total band area (corrected for path length, temperature, and pressure) in analytical region of reference spectrum.
Con_R	=	Reference spectrum concentration.

This procedure for estimating the uncertainty for an undetected compound usually yields a number that is higher than the actual quantitation limit. This is because no attempt is made to optimize the analytical regions for each compound, nor is the spectral subtraction optimized. (All spectral subtractions are performed, even subtractions that are unnecessary for detecting a particular compound, before the RMSD calculations are performed.) Additionally, band area calculations give a conservative estimate of analyte quantitation limits because the analytical program can usually detect analyte absorbances at lower concentrations than the band area calculations indicate.

5.0 SUMMARY OF FTIR QA/QC PROCEDURES

5.1 SAMPLING AND TEST CONDITIONS

Sample lines were cleaned by purging with moist air (250° F). Following this, the lines were checked with nitrogen. This was done by heating the sampling lines to 250°F and then 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 (99.99 percent) that was taken directly from a cylinder. The lines were checked again on site before sampling, after each change of location, and after spiking.

Each FTIR run was performed for the full duration of the manual sampling. Most of the batch samples were untreated or untreated samples spiked with the HCl standard. Since HCl was the primary pollutant, only a few condenser samples were measured. HCl spiked and unspiked results were analyzed as a QA check on the suitability of the sampling system for transporting HCl.

Several QA measures were followed to avoid contamination of the sampling system with the HCl spike. At least 10 unspiked samples were measured at the start of each run before any spiking was performed. No spiking was performed during Run 1 at the baghouse outlet or Runs 2 and 3 at the inlet. At both the baghouse inlet and outlet, ambient air was sampled through the lines after each run and before Runs 2 and 3.

Each spectrum was assigned a unique file name and written to the hard disk and a backup disk under that file name. Each interferogram also received a file name that identifies it with its corresponding absorbance spectrum. All background spectra and calibration spectra were stored on disks with their corresponding interferograms. A backup copy of all spectra was given to EPA on-site immediately after the test was completed.

A hard copy data sheet showing a record of every sample, background and calibration is included in Appendix B of this report. A copy of these records was given to EPA immediately after the test was completed. A transcribed record is also included in the report. Several minor errors in the original written record were corrected in the transcribed version.

Below are listed some important sampling and instrument parameters that were constantly monitored and some were periodically recorded.

Sampling Conditions

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

Instrument Configuration

- 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

Effluent was allowed to flow through the entire sampling system for at least 5 minutes before a the start of a sampling run or after changing to a different conditioning system, or test location. Single beam background spectra were periodically collected to try and maintain a baseline within ±5 percent of 100 percent transmittance.

The samples from two locations passed through separate sampling systems, except for a 20-ft section from the manifold to the cell. This section was evacuated each time sampling was switched from one location to the other. Background spectra and spectra of the empty cell (with the cell valve open) served as a check that the 20-ft connection line did not become contaminated.

When the condenser was in use, sample was kept constantly flowing through it before a sample was measured. Before switching to the other location, nitrogen was passed through the condenser and a sample of the nitrogen was measured in the FTIR cell.

5.2 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" (Appendix D).[2] A spectrum of the calibration transfer standard (CTS) was recorded at the beginning and end of each test run. Vacuum and positive pressure leak checks of the FTIR cell through the sample manifold were performed according to the procedures in the EPA Protocol and Method 320. Leak check results are recorded in Appendix B. Two CTS gases were used; 99.4 ppm ethylene in nitrogen and 20.0 ppm ethylene in nitrogen. Both CTS gases were measured at least once each time the path length was changed. The 99.4 standard was used primarily for the shorter path lengths and the 20.0 ppm standard was used primarily for the longer path lengths. The CTS spectrum provided a check on the operating conditions of the FTIR instrumentation, e.g. spectral resolution and cell path length. Ambient pressure was recorded whenever a CTS spectrum was collected and frequently during sampling. MRI also obtained independent pressure recordings from PES (Appendix A). Ambient pressure was consistently about 720 mm Hg (about 28.4 in. Hg).

Multiple copies of all interferograms, processed backgrounds, sample spectra, and the CTS were stored on separate computer cartridges.

5.3 CORRECTIVE ACTIONS

During Run 2 at the baghouse inlet the particulate filter was changed to increase the sample flow. This improved the flow, but it decreased as the run continued. Eventually, a clog was removed from the back of the probe and the flow became consistent again as it had been in Run 1.

6.0 REFERENCES

1. "Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy," EPA Contract No. 68-D2-0165, Work Assignment 3-08, July, 1996.

2. "Protocol For The Use of FTIR Spectrometry to Perform Extractive Emissions Testing at Industrial Sources," EPA Contract No. 68-D2-0165, Work Assignment 3-12, EMTIC Bulletin Board, September, 1996.

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

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

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

APPENDIX A.

ADDITIONAL DATA AND CALCULATIONS

This appendix presents measurements and results from PES. Included are Method 25A results and stack gas measurements conducted during the testing.

Summary of Stack Gas Parameters and Test Results
Lime Manufacturing Emission Test - APG Lime Company
US EPA Test Method 29 - Metals and Particulate Matter
Kiln No. 2 - Cooler Stack
Page 1 of 3

	RUN NUMBER	M29-10	M29-11	M29-12	
	RUN DATE	10/21/96	10/21/96	10/21/96	Average
	RUN TIME	1125-1334	1404-1607	1625-1828	
	MEASURED DATA				
(Y)	Meter Box Correction Factor, Y	1.008	1.008	1.008	1.008
(dH)	Avg Meter Orifice Pressure, in. H2O	1.542	1.378	1.464	1.461
(Pbar)	Barometric Pressure, in. Hg	28.31	28.31	28.31	28.31
(Vm)	Sample Volume, ft^3	86.971	83.919	86.680	85.857
(Tm)	Average Meter Temperature, °F	101	106	109	105
(Pg)	Stack Static Pressure, in. H2O	0.00	0.00	0.00	0.00
(Ts)	Average Stack Temperature, °F	201	226	207	211
(Vlc)	Condensate Collected, ml	16.0	22.8	15.0	17.9
(%CO2)	Carbon Dioxide content, % by volume	0.0	0.0	0.0	0.0
(%O2)	Oxygen content, % by volume	20.9	20.9	20.9	20.9
(%N2)	Nitrogen content, % by volume	79.1	79.1	79.1	79.1
(Cp)	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
(dP)	Avg Sqrt Delta P, (in. H2O)½	0.0731	0.0708	0.0708	0.0716
(Theta)	Sample Time, min	120	120	120	120
(Dn)	Nozzle Diameter, in.	0.748	0.748	0.748	0.748
	CALCULATED DATA				
(An)	Nozzle Area, square feet	0.00305	0.00305	0.00305	0.00305
(Vmstd, cf)	Standard Meter Volume, ft^3	78.328	74.946	76.976	76.750
(Vmstd, cm)	Standard Meter Volume, m^3	2.218	2.122	2.180	2.173
(Qm)	Average Sampling Rate, dscfm	0.653	0.625	0.641	0.640
(Ps)	Stack Pressure, inches Hg	28.31	28.31	28.31	28.31
(%H2O)	Moisture, %	1.0	1.4	0.9	1.1
(%H2Osat)	Moisture (at saturation), %	83.8	139.8	95.8	106.5
(Vwstd)	Standard Water Vapor Volume, ft^3	0.753	1.073	0.706	0.844
(Mfd)	Dry Mole Fraction	0.990	0.986	0.991	0.989
(Md)	Molecular Weight-dry, lb/lb-mole	28.84	28.84	28.84	28.84
(Ms)	Molecular Weight-wet, lb/lb-mole	28.73	28.68	28.74	28.72
(Vs)	Velocity, ft/s	4.7	4.7	4.6	4.7
(A)	Stack Area, ft^2	27.69	27.69	27.69	27.69
(Qa)	Stack Gas Volumetric flow, acfm	7,862	7,767	7,653	7,760
(Qs, cfm)	Stack Gas Volumetric flow, dscfm	5,887	5,571	5,677	5,711
(Qs, cmm)	Stack Gas Volumetric flow, dscmm	167	158	161	162
(I)	Isokinetic Rate, %	100.6	101.7	102.5	101.6

Summary of Stack Gas Parameters and Test Results
Lime Manufacturing Emission Test - APG Lime Company
US EPA Test Method 23 - CDD/CDF
Kiln No. 1 - Baghouse Inlet
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	RUN NUMBER	M23-6-I	M23-7-I	M23-8-I	
	RUN DATE	10/22/96	10/23/96	10/24/96	Average
	RUN TIME	1130-1743	1040-1540	1054-1715	
	MEASURED DATA				
(Y)	Meter Box Correction Factor, Y	1.009	1.009	1.009	1.009
(dH)	Avg Meter Orifice Pressure, in. H2O	0.537	0.603	0.561	0.567
(Pbar)	Barometric Pressure, in. Hg	28.38	28.17	28.30	28.283
(Vm)	Sample Volume, ft^3	99.554	105.205	101.289	102.016
(Tm)	Average Meter Temperature, °F	77	70	69	71.931
(Pg)	Stack Static Pressure, in. H2O	-2.80	-2.80	-2.80	-2.80
(Ts)	Average Stack Temperature, °F	497	498	497	497.472
(Vlc)	Condensate Collected, ml	79.9	80.0	85.8	81.9
(%CO2)	Carbon Dioxide content, % by volume	10.7	10.3	10.1	10.4
(%O2)	Oxygen content, % by volume	13.2	13.9	14.0	13.7
(%N2)	Nitrogen content, % by volume	76.1	75.8	75.9	75.9
(Cp)	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
(dP)	Avg Sqrt Delta P, (in. H2O)½	0.788	0.833	0.856	0.825
(Theta)	Sample Time, min	240	240	240	240
(Dn)	Nozzle Diameter, in.	0.197	0.197	0.191	0.195
	CALCULATED DATA				
(An)	Nozzle Area, ft^2	0.000212	0.000212	0.000199	0.000207
(Vmstd, cf)	Standard Meter Volume, dscf	93.834	99.690	96.556	96.693
(Vmstd, cm)	Standard Meter Volume, dscm	2.643	2.808	2.720	2.724
(Qm)	Average Sampling Rate, dscfm	0.391	0.415	0.402	0.403
(Ps)	Stack Pressure, in. Hg	28.17	27.96	28.09	28.08
(%H2O)	Moisture, % by volume	3.9	3.6	4.0	3.8
(%H2Osat)	Moisture (at saturation), %	5012.5	5102.5	5040.2	5051.7
(Vwstd)	Standard Water Vapor Volume, ft^3	3.761	3.766	4.039	3.855
(Mfd)	Dry Mole Fraction	0.96	0.96	0.96	0.962
(Md)	Molecular Weight-dry, lb/lb-mole	30.24	30.20	30.18	30.21
(Ms)	Molecular Weight-wet, lb/lb-mole	29.77	29.76	29.69	29.74
(Vs)	Stack Gas Velocity, ft/s	60.5	64.2	65.8	63.5
(A)	Stack Area, ft^2	28.47	28.47	28.47	28.47
(Qa)	Stack Gas Volumetric flow, acfm	103,295	109,590	112,436	108440
(Qs,cmm)	Stack Gas Volumetric flow, dscfm	51,576	54,368	55,870	53,938
(Qs,cfm)	Stack Gas Volumetric flow, dscmm	1,460.5	1,539.5	1,582.1	1,527.4
(I)	Isokinetic Sampling Ratio, %	102.0	102.8	103.1	102.6

Summary of Stack Gas Parameters and Test Results
Lime Manufacturing Emission Test - APG Lime Company
US EPA Test Method 29 - Metals and Particulate Matter
Kiln No. 1 - Baghouse Inlet
Page 1 of 3

	RUN NUMBER	M29-13-I	M29-14-I	M29-14-I	
	RUN DATE	10/22/96	10/23/96	10/24/96	Average
	RUN TIME	1130-1743	1040-1540	1054-1715	
	MEASURED DATA				
(Y)	Meter Box Correction Factor, Y	0.978	0.978	0.978	
(dH)	Avg Meter Orifice Pressure, in. H2O	0.54	0.48	0.54	0.52
(Pbar)	Barometric Pressure, in. Hg	28.38	28.17	28.30	28.28
(Vm)	Sample Volume, ft^3	100.071	94.188	100.000	98.086
(Tm)	Average Meter Temperature, °F	86	105	78	89
(Pg)	Stack Static Pressure, in. H2O	-2.80	-3.00	-3.50	-3.10
(Ts)	Average Stack Temperature, °F	500	504	504	503
(Vlc)	Condensate Collected, ml	79.9	80.0	89.6	83.2
(%CO2)	Carbon Dioxide content, % by volume	10.7	10.3	9.9	10.3
(%O2)	Oxygen content, % by volume	13.2	13.9	14.0	13.7
(%N2)	Nitrogen content, % by volume	76.1	75.8	76.1	76.0
(Cp)	Pitot Tube Coefficient	0.84	0.84	0.84	
(dP)	Avg Sqrt Delta P, (in. H2O)½	0.786	0.800	0.855	0.814
(Theta)	Sample Time, min	240.0	240.0	240.0	
(Dn)	Nozzle Diameter, in.	0.197	0.188	0.188	
	CALCULATED DATA				
(An)	Nozzle Area, ft^2	0.000212	0.000193	0.000193	0.000199
(Vmstd)	Standard Meter Volume, dscf	89.903	81.105	90.953	87.320
(Ps)	Stack Pressure, in. Hg	28.17	27.95	28.04	28.06
(%H2O)	Moisture, % by volume	4.0	4.4	4.4	4.3
(Vwstd)	Standard Water Vapor Volume, ft^3	3.761	3.766	4.217	3.915
(Mfd)	Dry Mole Fraction	0.96	0.96	0.96	0.957
(Md)	Molecular Weight-dry, lb/lb-mole	30.24	30.20	30.14	30.20
(Ms)	Molecular Weight-wet, lb/lb-mole	29.75	28.86	28.81	29.14
(Vs)	Stack Gas Velocity, ft/s	60.4	62.8	67.0	63.4
(A)	Stack Area, ft^2	27.69	27.69	27.69	27.69
(Qa)	Stack Gas Volumetric flow, acfm	100,343	104,372	111,388	105,368
(Qs)	Stack Gas Volumetric flow, dscfm	49,838	51,027	54,621	51,829
(I)	Isokinetic Sampling Ratio, %	98.3	95.1	99.7	97.7

APG Lime Corporation

Kiln No. 1 Inlet to Baghouse

10:33	0.00	0.00%				
	50.00	1.60%				
10:54-11:10	2.6		Corrected	2.5	ppm THC	(wet basis)
11:10-11:25	1.8			1.5		
11:25-11:40	1.8			1.5		
11:40-11:55	1.8			1.5		
11:55-12:10	1.7			1.4		
12:10-12:25	1.8			1.2		
12:25-12:40	1.5			1.1		
	Post Cal	Drift				
12:43	0.40	0.40%				
	50.80	2.40%				
12:54-13:10	1.5		Corrected	1.1	ppm THC	(wet basis)
13:10-13:25	1.7			1.4		
13:25-13:40	1.8			1.5		
13:40-13:55	1.8			1.5		
13:55-14:10	1.9			1.6		
14:10-14:25	1.9			1.6		
14:25-14:40	2.1			1.8		
	Post Cal	Drift				
14:44	0.40	0.40%				
	50.80	2.40%				

Calibration Gases	System Calibration
0.0 ppm	0.40
30.04 ppm	30.80
48.72 ppm	50.80
87.88 ppm	88.00
	Calibration Error
	0.44%
	1.02%
	Correlation 0.9999409
	Slope 0.9973203
	Intercept 0.7072909

15:00-15:10	2.6		Corrected	1.9	ppm THC	(wet basis)
15:10-15:25	2.6			1.9		
15:25-15:40	3.5			2.8		
15:40-15:55	3.8			3.1		
15:55-16:10	3.2			2.5		
16:10-16:25	2.5			1.8		
16:25-16:40	3.3			2.6		
16:40-16:51	3.3			2.6		
	Post Cal	Drift				
16:52	0.40	0.00%				
	52.00	1.20%				
17:10-17:25	3.9		Corrected	3.2	ppm THC	(wet basis)
17:25-17:40	3.9			3.2		
17:40-17:55	2.9			2.2		
17:55-18:10	2.8			2.1		
18:10-18:25	2.4			1.7		
18:25-18:40	2.4			1.7		
18:40-18:55	2.5			1.8		
18:55-19:10	2.6			1.9		
19:10-19:25	2.9			2.2		
19:25-19:40	2.7			2.0		
19:40-19:45	3.2			2.5		
	Post Cal	Drift				
19:52	0.00	0.40%				
	48.80	2.00%				

Summary of Stack Gas Parameters and Test Results
Lime Manufacturing Emission Test - APG Lime Company
US EPA Test Method 23 - CDD/CDF
Kiln No. 1 - Baghouse Outlet
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	RUN NUMBER	M23-6-O	M23-7-O	M23-8-O	
	RUN DATE	10/22/96	10/23/96	10/24/96	Average
	RUN TIME	1130-1749	1041-1542	1121-1731	
	MEASURED DATA				
(Y)	Meter Box Correction Factor, Y	1.003	1.003	1.003	1.003
(dH)	Avg Meter Orifice Pressure, in. H2O	1.458	1.557	1.732	1.582
(Pbar)	Barometric Pressure, in. Hg	28.38	28.17	28.30	28.283
(Vm)	Sample Volume, ft^3	160.585	171.734	181.806	171.375
(Tm)	Average Meter Temperature, °F	88	84	80	83.979
(Pg)	Stack Static Pressure, in. H2O	-0.40	-0.43	-0.45	-0.43
(Ts)	Average Stack Temperature, °F	400	401	410	403.617
(Vlc)	Condensate Collected, ml	114.4	115.7	143.6	124.6
(%CO2)	Carbon Dioxide content, % by volume	8.5	9.6	8.8	9.0
(%O2)	Oxygen content, % by volume	14.9	14.2	14.7	14.6
(%N2)	Nitrogen content, % by volume	76.6	76.2	76.5	76.4
(Cp)	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
(dP)	Avg Sqrt Delta P, (in. H2O)½	0.744	0.748	0.810	0.767
(Theta)	Sample Time, min	240	240	240	240
(Dn)	Nozzle Diameter, in.	0.250	0.254	0.254	0.253
	CALCULATED DATA				
(An)	Nozzle Area, ft^2	0.000341	0.000352	0.000352	0.000348
(Vmstd)	Standard Meter Volume, dscf	147.758	158.021	169.242	158.341
(Vmstd, cm)	Standard Meter Volume, dscm	4.162	4.451	4.767	4.460
(Qm)	Average Sampling Rate, dscfm	0.616	0.658	0.705	0.660
(Ps)	Stack Pressure, in. Hg	28.35	28.14	28.27	28.25
(%H2O)	Moisture, % by volume	3.5	3.3	3.8	3.6
(%H2Osat)	Moisture (at saturation), %	1829.2	1872.0	2055.7	1919.0
(Vwstd)	Standard Water Vapor Volume, ft^3	5.385	5.446	6.759	5.863
(Mfd)	Dry Mole Fraction	0.96	0.97	0.96	0.964
(Md)	Molecular Weight-dry, lb/lb-mole	29.96	30.10	30.00	30.02
(Ms)	Molecular Weight-wet, lb/lb-mole	29.54	29.70	29.54	29.59
(Vs)	Stack Gas Velocity, ft/s	54.1	54.5	59.4	56.0
(A)	Stack Area, ft^2	28.47	28.47	28.47	28.47
(Qa)	Stack Gas Volumetric flow, acfm	92,465	93,134	101,413	95671
(Qs,cmm)	Stack Gas Volumetric flow, dscfm	51,888	51,890	55,904	53,228
(Qs,cfm)	Stack Gas Volumetric flow, dscmm	1,469	1,469	1,583	1,507
(I)	Isokinetic Sampling Ratio, %	99.1	102.7	102.1	101.3

Summary of Stack Gas Parameters and Test Results
Lime Manufacturing Emission Test - APG Lime Company
US EPA Test Method 29 - Metals and Particulate Matter
Kiln No. 1 - Baghouse Outlet
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	RUN NUMBER	M29-13-O	M29-13-O	M29-13-O	
	RUN DATE	10/22/96	10/23/96	10/24/96	Average
	RUN TIME	1130-1749	1041-1542	1121-1731	
	MEASURED DATA				
(Y)	Meter Box Correction Factor, Y	0.985	0.985	0.985	0.985
(dH)	Avg Meter Orifice Pressure, in. H2O	1.43	1.47	1.59	1.50
(Pbar)	Barometric Pressure, in. Hg	28.38	28.17	28.30	28.28
(Vm)	Sample Volume, ft^3	173.582	177.496	186.444	179.174
(Tm)	Average Meter Temperature, °F	90	84	81	85
(Pg)	Stack Static Pressure, in. H2O	-0.40	-0.43	-0.45	-0.43
(Ts)	Average Stack Temperature, °F	411	406	413	410
(Vlc)	Condensate Collected, ml	123.7	121.9	143.0	129.5
(%CO2)	Carbon Dioxide content, % by volume	8.5	9.6	8.8	9.0
(%O2)	Oxygen content, % by volume	14.9	14.2	14.7	14.6
(%N2)	Nitrogen content, % by volume	76.6	76.2	76.5	76.4
(Cp)	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
(dP)	Avg Sqrt Delta P, (in. H2O)½	0.768	0.767	0.802	0.779
(Theta)	Sample Time, min	240.0	240.0	240.0	240.0
(Dn)	Nozzle Diameter, in.	0.255	0.250	0.250	0.252
	CALCULATED DATA				
(An)	Nozzle Area, ft^2	0.000355	0.000341	0.000341	0.000345
(Vmstd)	Standard Meter Volume, dscf	156.211	160.256	170.099	162.189
(Ps)	Stack Pressure, in. Hg	28.35	28.14	28.27	28.25
(%H2O)	Moisture, % by volume	3.6	3.5	3.8	3.6
(Vwstd)	Standard Water Vapor Volume, ft^3	5.823	5.738	6.731	6.097
(Mfd)	Dry Mole Fraction	0.96	0.97	0.96	0.96
(Md)	Molecular Weight-dry, lb/lb-mole	29.96	30.10	30.00	30.02
(Ms)	Molecular Weight-wet, lb/lb-mole	29.53	29.69	29.54	29.58
(Vs)	Stack Gas Velocity, ft/s	56.2	56.1	58.9	57.1
(A)	Stack Area, ft^2	28.47	28.47	28.47	28.47
(Qa)	Stack Gas Volumetric flow, acfm	96,040	95,852	100,604	97,498
(Qs)	Stack Gas Volumetric flow, dscfm	53,162	53,040	55,265	53,822
(I)	Isokinetic Sampling Ratio, %	98.3	105.2	107.1	103.5

APG Lime Corporation

Kiln No. 1 Outlet of Baghouse

October 22, 1996

Calibration Gases	System Calibration
0.0 ppm	0.80
30.04 ppm	30.00
49.72 ppm	49.20
87.86 ppm	84.00

Correlation	0.9998316
Slope	0.9472941
Intercept	1.3036408

Sampling System Bias
0.80%
0.40%
1.60%
3.60%

Direct Calibration
0.00
30.40
50.80
87.80

Correlation	0.999877
Slope	0.999068
Intercept	0.375103

Calibration Error
1.20%
2.17%

10:34

Calibration Gases	System Calibration
0.0 ppm	0.00
30.04 ppm	29.80
49.72 ppm	49.20
87.86 ppm	85.20

Calibration Error
0.38%
1.28%

Correlation	0.9999137
Slope	0.9702479
Intercept	0.3417826

	Post Cal	Drift
11:22	0.00	0.00%
	48.80	0.40%

11:30-11:45	2.1	Corrected	1.8
11:45-12:00	2.1		1.8
12:00-12:15	2.3		2.0
12:15-12:30	2.2		1.9
12:30-12:45	2.2		1.9
12:45-13:00	2.3		2.0
13:00-13:15	2.2		1.9
13:15-13:30	2.3		2.0
13:30-13:45	2.3		2.0

ppm THC (wet basis)

	Post Cal	Drift
13:50	0.40	0.40%
	48.80	0.40%

14:00-14:15	2.3	Corrected	2.0
14:15-14:30	2.3		2.0
14:30-14:45	2.0		1.7
14:45-15:00	1.4		1.1
15:00-15:15	1.9		1.6
15:15-15:30	2.1		1.8
15:30-15:45	1.9		1.6

ppm THC (wet basis)

	Post Cal	Drift
15:50	0.80	0.80%
	48.80	0.40%

16:00-16:15	2.3	Corrected	2.0
16:15-16:30	1.6		1.3
16:30-16:45	1.5		1.2
16:45-17:00	1.9		1.6
17:00-17:15	1.9		1.6
17:15-17:30	1.7		1.4
17:30-17:45	1.9		1.6
17:45-18:00	2.0		1.7

ppm THC (wet basis)

APG Lime Corporation
Kiln No. 1 Outlet of Baghouse

	Post Cal	Drift
18:04	0.80	0.80%
	48.80	0.40%

October 23, 1996

Calibration Gases	System Calibration
0.0 ppm	0.80
30.04 ppm	30.00
49.72 ppm	49.60
87.86 ppm	84.80

Correlation	0.9998573
Slope	0.9570957
Intercept	1.1929047

Sampling System Bias
0.80%
0.80%
1.20%
3.20%

Direct Calibration
0.00
30.80
50.80
88.00

Correlation	0.999905
Slope	1.001439
Intercept	0.434587

Calibration Error
2.53%
2.17%

10:40-10:55	3.9	Corrected	2.8	ppm THC	(wet basis)
10:55-11:10	4.1		3.0		
11:10-11:25	4.6		3.5		

	Post Cal	Drift
11:32	0.80	0.00%
	49.20	0.40%

11:40-11:55	4.5	Corrected	3.5	ppm THC	(wet basis)
11:55-12:10	4.3		3.2		
12:10-12:25	4.5		3.5		
12:25-12:40	4.5		3.5		
12:40-12:55	4.4		3.4		
12:55-13:10	4.4		3.4		
13:10-13:23	4.7		3.7		

	Post Cal	Drift
13:36	0.80	0.00%
	49.20	0.40%

13:45-14:00	4.4	Corrected	3.4	ppm THC	(wet basis)
14:00-14:15	4.2		3.1		
14:15-14:30	3.2		2.1		
14:30-14:45	2.7		1.6		
14:45-15:00	2.4		1.3		
15:00-15:15	2.6		1.5		
15:15-15:30	2.7		1.6		
15:30-15:45	2.7		1.6		
15:45-16:00	3.8		2.7		

	Post Cal	Drift
16:09	0.80	0.00%
	49.60	0.00%

October 24, 1996

Calibration Gases	System Calibration
0.0 ppm	0.40
30.04 ppm	30.00
49.72 ppm	49.20
87.86 ppm	84.40

Correlation	0.9998356

Direct Calibration
0.00
30.80
50.80
88.00

Correlation	0.999906

APG Lime Corporation

Kiln No. 1 Outlet of Baghouse

Slope	0.9559295
Intercept	0.941773

Sampling System Bias
0.40%
0.80%
1.60%
3.60%

Slope	1.001439
Intercept	0.434697

Calibration Error
2.53%
2.17%

	Post Cal	Drift
10:37	0.40	0.00%
	49.20	0.00%

10:58-11:10	2.9		Corrected	2.0	ppm THC (wet basis)
11:10-11:25	2.0			1.1	
11:25-11:40	2.0			1.1	
11:40-11:55	2.1			1.2	
11:55-12:10	1.9			1.0	
12:10-12:25	1.8			0.9	
12:25-12:40	1.8			0.9	
12:40-12:48	1.7			0.8	

	Post Cal	Drift
12:51	0.80	0.40%
	49.60	0.40%

12:58-13:10	1.7		Corrected	0.8	ppm THC (wet basis)
13:10-13:25	1.8			0.9	
13:25-13:40	2.0			1.1	
13:40-13:55	2.0			1.1	
13:55-14:10	2.1			1.2	
14:10-14:25	2.0			1.1	
14:25-14:40	2.4			1.5	
14:40-14:55	2.6			1.7	

	Post Cal	Drift
14:57	1.20	0.80%
	50.00	0.80%

15:10-15:25	2.7		Corrected	1.8	ppm THC (wet basis)
15:25-15:40	3.6			2.8	
15:40-15:55	3.8			3.0	
15:55-16:10	3.3			2.5	
16:10-16:25	2.6			1.7	
16:25-16:40	3.3			2.5	
16:40-16:53	3.4			2.6	

	Post Cal	Drift
16:57	0.80	0.40%
	50.00	0.80%

17:10-17:25	4.1		Corrected	3.3	ppm THC (wet basis)
17:25-17:40	4.1			3.3	
17:40-17:55	3.1			2.3	
17:55-18:10	3.1			2.3	
18:10-18:25	2.6			1.9	
18:25-18:40	2.6			1.9	
18:40-18:55	2.9			2.0	
18:55-19:10	3.0			2.2	
19:10-19:25	3.4			2.6	
19:25-19:40	3.2			2.4	
19:40-19:45	3.6			2.8	

	Post Cal	Drift
16:57	0.80	0.40%
	49.20	0.00%

APPENDIX B.

FIELD DATA RECORDS

Data Sheet: FTIR Background and Calibration Spectra: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Time	File Name	Path	Location/Notes	#scans	Res (cm-1)	Cell temp (F)	Press.	BKG	Apod
10/21/96	9:51	BKG1021A	16 passes	cold cell located at cooling stack	100	2.0	ambient	719.6		NB/med
	10:14	CTS1021A	16 passes	cold cell 99.4 ppm ethylene	50/100	2.0	ambient	719.6		NB/med
	10:22	CTS1021B	16 passes	cold cell 99.4 ppm ethylene 2nd fill after evacuating	50/100	2.0	ambient	718.1		NB/med
	10:33	CTS1021C	16 passes	cold cell 99.4 ppm ethylene 3rd fill	50/100	2.0	ambient	718.1		NB/med
				*Cell @ 120C Positive pressure leak check (time =0, P=823.1), (time=2, P=825)						
				*Vacuum leak check (time=0, P=5.3torr), (time=2min, P=7.0 torr)						
10/21/96	11:35	BKG1021B	40 passes	N2 background	100	1.0	119C	718.5		NB/med
	11:40	CTS1021D	40 passes	99.4 ppm ethylene	50/100	1.0	119C	718.5	B	NB/med
	11:45	CTS1021E	40 passes	99.4 ppm ethylene 2nd fill	50/100	1.0	119C	718.5	B	NB/med
	11:49	BKG1021C	40 passes	Dryer than B	100	1.0	120C	718.5	B	NB/med
	11:55	CTS1021F	40 passes	20.0 ppm ethylene	50/100	1.0	120C	718.8	C	NB/med
	11:59	CTS1021G	40 passes	20.0 ppm ethylene	50/100	1.0	120C	718.5	C	NB/med
	12:09	BKG1021D	40 passes		100	1.0	120C	718.5		NB/med
	13:03	BKG1021E	40 passes		100	1.0	120C	718		NB/med
	13:15			*started SF6 spike @ 0.48 lpm and 4.01 ppm	50/100					
	14:52	HClAPG01	40 passes	HCl 103 ppm flowing through cell @ 120 on meter #2		1.0	120C	718.5	E	NB/med
	15:02	HClAPG02	40 passes	HCl 103 ppm flow through cell @ 95 on meter #2		1.0	120C	716	E	NB/med
	15:07	SF6APG01	40 passes	SF6 4.01 ppm static in cell		1.0	120C	717.5	E	NB/med
	15:12	SF6APG02	40 passes	SF6 4.01 ppm new fill		1.0	120C	717.5	E	NB/med
	15:15	BKG1021F	40 passes		100	1.0	120C			NB/med
	15:23	CTS1021H	40 passes	20.0 ppm ethylene	50/100	1.0	120C	717.1	F	NB/med
	15:28	CTS1021I	40 passes	20.0 ppm ethylene 2nd fill	50/100	1.0	120C		F	NB/med
	18:39	BKG1021G	40 passes	Baghouse	100	1.0	120C	717.9		NB/med
	18:45	CTS1021J	40 passes	20.0 ppm ethylene	50/100	1.0	120C	718	G	NB/med
	18:53	CTS1021K	40 passes	20.0 ppm ethylene	50/100	1.0	120C		G	NB/med
10/22/96	8:40	BKG1022A	40 passes	N2 background in cold cell	100	2.0	ambient	719.1		NB/med
	8:48	CTS1022A	40 passes	20.0 ppm ethylene	50/100	2.0	ambient	719.1	A	NB/med
	8:55	CTS1022B	40 passes	20.0 ppm ethylene 2nd fill	50/100	2.0	ambient	719	A	NB/med
	9:00	CTS1022C	40 passes	20.0 ppm ethylene 3rd fill	50/100	2.0	ambient	719	A	NB/med
				*Vacuum leak check (time=0, P=6.8), (time=2min, P=12.0)						
10/22/96				*Positive pressure leak check (time=0, P=769.1), (time=1min, P=768.9)						
	10:48	BKG1022B	40 passes	Dry N2	200	1.0	122C	719.3		NB/med
	10:51	CTS1022D	40 passes	20.0 ppm ethylene	50/200	1.0	122C	719.3	B	NB/med
	10;56	CTS1022E	40 passes	20.0 ppm ethylene 2nd fill	50/200	1.0	122C	719.3	B	NB/med
				*Some CO coming through manifold into BKG spectrum RMSD 2700-2900cm-1 = 1.4 X 10-3						
				*Sample line leak checks completed @ 10:30 and both lines purging since before 10:00 am						

Data Sheet: FTIR Background and Calibration Spectra: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Time	File Name	Path	Location/Notes	#scans	Res (cm-1)	Cell temp (F)	Press.	BKG	Apod
10/22/96		BKG1022C	40 passes	New N2 background to get rid of ice band	200	1.0	122C	717.9		NB/med
	18:44	SF6APG00	40 passes	SF6 4.01ppm	50/200	1.0	122C	716.9	C	NB/med
	18:47	SF6APG04	40 passes	SF6 4.01ppm	50/200	1.0	122C	716	C	NB/med
	19:07	BKG1022D	40 passes	New background w/N2	200	1.0	122C	717.1		NB/med
	19:15	HCIAPG03	40 passes	HCl 103ppm flow through cell P=717.1-718.5	50/200				D	NB/med
	19:20	HCIAPG04	40 passes	HCl 103ppm flow through cell P=717.1-718.5	50/200	1.0	122C	718.5	D	NB/med
	19:28	BKG1022E	40 passes	N2 in cell	50/200	1.0	122C	717		NB/med
	19:33	CTS1022F	40 passes	20.0 ppm ethylene	50/200	1.0	122C	716.8	D	NB/med
	19:37	CTS1022G	40 passes	20.0 ppm ethylene	50/200	1.0	122C	716.8	D	NB/med
10/23/96	8:54	BKG1023A	40 passes	N2 background	100	2.0	25C	713.6		NB/med
	9:02	CTS1023A	40 passes	20.0 ppm ethylene	50/100	2.0	25C	713.4	A	NB/med
	9:05	CTS1023B	40 passes	20.0 ppm ethylene 2nd fill	50/100	2.0	25C	713.4	A	NB/med
	9:11	CTS1023C	40 passes	20.0 ppm ethylene 3rd fill	50/100	2.0	25C	713.4	A	NB/med
				*Positive pressure leak check through manifold (time=0, P=858.1), (time=1min, P=861.6)						
	10:20	BKG1023B	40 passes	N2 in cell static	100	1.0	121C	713.6		NB/med
	10:52	CTS1023D	40 passes	20.0 ppm ethylene	50/100	1.0	121C	713.6	B	NB/med
	10:57	CTS1023E	40 passes	20.0 ppm ethylene	50/100	1.0	121C	713.4	B	NB/med
				*Vacuum leak check (time=0, P=6.7), (time=1min, P=9.4)						
	16:25	BKG1023C	40 passes	N2 background	100	1.0	122C	713.1		NB/med
	16:29	CTS1023F	40 passes	20.0 ppm ethylene	50/100	1.0	122C	712.7	C	NB/med
	16:33	CTS1023G	40 passes	20.0 ppm ethylene 2nd fill	50/100	1.0	122C	712.7	C	NB/med
10/24/96	8:38	BKG1024A	40 passes	N2 cold	100	1.0	19.5C	717.1		NB/med
	8:42	BKG1024B	40 passes	N2 cold same fill	100	2.0	19.5C	717.1		NB/med
				*Purged flow meter & sample lines w/N2 overnight						
	8:50	BAGON201	40 passes	N2 purge through outlet line	50/100	1.0	19.5C	717.1	A	NB/med
		BAGIN201	40 passes	N2 purge through inlet	50/100	1.0	19.5C	719.2	A	NB/med
	9:07	CTS1024A	40 passes	20.0 ppm ethylene	50/100	2.0	19.5C	717.6	B	NB/med
		CTS1024B	40 passes	20.0 ppm ethylene 2nd fill	50/100	2.0	19.5C	717.8	B	NB/med
	9:10			*good leak @ inlet new filter, no glass wool plug						
	9:50	BKG1024C	20 passes	decreased path length	100	2.0	23.2C	718.2		NB/med
	9:58	CTS1024C	20 passes	20.0 ppm ethylene	50/100	2.0	23.2C	718.2	C	NB/med
	10:01	CTS1024D	20 passes	20.0 ppm ethylene 2nd fill	50/100	2.0	23.2C	718.2	C	NB/med
	10:09	CTS1024E	20 passes	99.4 ppm ethylene	50/100	2.0	23.2C	718.2	C	NB/med
	10:12	CTS1024F	20 passes	99.4 ppm ethylene 2nd fill	50/100	2.0	23.2C	718.2	C	NB/med
	10:31	BKG1024D	20 passes	N2 background	100	1.0	111C	718.5		NB/med
	10:34-10:36	BAGOA301	20 passes	ambient sample from outlet flow through @ 725.8 t	50	1.0	113C	718.5	D	NB/med
	10:39	BAGIA301	20 passes	ambient sample from intlet flow through @ 725.7 to	50	1.0	116C	718.5	D	NB/med

Data Sheet: FTIR Background and Calibration Spectra: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Time	File Name	Path	Location/Notes	#scans	Res (cm-1)	Cell temp (F)	Press.	BKG	Apod
10/24/96	10:57	BKG1024E	20 passes	N2 background probes in stack	100	1.0	119	718.6		NB/med
	11:03	CTS1024G	20 passes	99.4 ppm ethylene	100	1.0	120	718.6	E	NB/med
	11:06	CTS1024H	20 passes	99.4 ppm ethylene 2nd fill	100	1.0	120	718.6	E	NB/med
	10:47			*Inlet in stack						
	10:53			*Outlet in stack						
	11:54	BKG1024F	20 passes	N2 in cell	100	1.0	121C	718.5		NB/med
	19:59	BKG1024G	20 passes	N2 in cell	100	1.0	122C	719.3		NB/med
	20:23	CTS1024I	20 passes	99.4 ppm ethylene	50/100	1.0	122C	718.7	G	NB/med
	20:28	CTS1024J	20 passes	99.4 ppm ethylene 2nd fill	50/100	1.0	122C	718.8	G	NB/med
	20:31	CTS1024K	20 passes	99.4 ppm ethylene 3rd fill	50/100	1.0	122C	718.7	G	NB/med
	20:49	BKG1024H	20 passes	N2 in cell	100	1.0	122C	718.8		NB/med
	20:53	SF6AP301	20 passes	SF6 4.01 ppm	50/100	1.0	122C	718.9	H	NB/med
	20:58	SF6AP302	20 passes	SF6 4.01 ppm 2nd fill	50/100	1.0	122C	718.9	H	NB/med
	21:02	SF6AP303	20 passes	SF6 4.01 ppm 3rd fill	50/100	1.0	122C	719	H	NB/med
10/24/96	21:08	BKG1024I	20 passes	N2 fill -- background	100	1.0	122C	719.1		NB/med
	21:18	HClAA301	20 passes	HCl dried from cylinder -- flowing through cell	50/100	1.0	122C	718.2	I	NB/med
		EMPAP30	20 passes	evacuated cell	50/100	1.0	122C	2.8	I	NB/med
	21:24	HClAP302	20 passes	103 ppm HCl static sample 2nd fill	50/100	1.0	122C	719.3	I	NB/med
	21:28	HClAP303	20 passes	103 ppm HCl static sample 3rd fill	50/100	1.0	122C	719.4	I	NB/med
	21:34	HClAP304	20 passes	103 ppm HCl static sample 4th fill	50/100	1.0	122C	18.8 to 719	I	NB/med
	21:31	EMPAP302	20 passes	evacuated cell before HClAP304	50/100	1.0	122C	3.3	I	NB/med
	21:54	BKG1024J	20 passes	N2 in cell	100	1.0	122C	719.5		NB/med
	22:00	CTS1024L	20 passes	99.4 ppm ethylene	50/100	1.0	122C	719.2	J	NB/med
	22:12	CTS1024M	20 passes	99.4 ppm ethylene 2nd fill	50/100	1.0	122C	719.4	J	NB/med

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/21/96	12:02-12:04	COOLH001	40 passes	Cooling stack APG	50/100	1.0	120C	U	H/W	90	C
	12:12-12:14	COOLH002	40 passes	Cooling stack APG	50/100	1.0	120C	U	H/W	90	D
	12:16-12:18	COOLC003		Cooling stack	50/100	1.0	120C	U	Cond.	90	D
	12:21-12:23	COOLC004		Cooling stack	50/100	1.0	120C	U	Cond.		D
	12:26-12:28	COOLH005		Cooling stack	50/100	1.0	120C	U	H/W		D
	12:29-12:32	COOLH006		Cooling stack	50/100	1.0	120C	U	H/W		D
	12:34-12:36	COOLH007		Cooling stack	50/100	1.0	120C	U	H/W		D
	12:41-12:42	COOLH008		Cooling stack	50/100	1.0	120C	U	H/W	90	D
	12:45-12:46	COOLH009		Cooling stack	50/100	1.0	120C	U	H/W	90	D
	12:48-12:49	COOLH010		Pressure = 718.1	50/100	1.0	120C	U	H/W	90	D
	12:52-12:53	COOLH011		Cooling stack	50/100	1.0	120C	U	H/W	90	D
	12:56-12:57	COOLH012		Cooling stack	50/100	1.0	120C	U	H/W	90	D
10/21/96	13:15			turned on SF6 spike 0.48lpm						90	
	13:24-13:26	COOLS013		SF6 @ 0.48 lpm	50/100	1.0	120C	S(SF6)	H/W	90	E
	13:28-13:29	COOLS014		SF6 @ 0.48 lpm	50/100	1.0	120C	S(SF6)	H/W	90	E
	13:32-13:33	COOLS015		SF6 @ 0.48 lpm	50/100	1.0	120C	S(SF6)	H/W	90	E
	13:43			started HCl spike @ 0.52 lpm							
	13:53-13:55	COOLS016		spike w/HCl 0.46 lpm							
	14:10-14:23	COOLS017		spike w/HCl @ 0.48 lpm		static				total = 90	E
	14:26-14:32	COOLS019		spiked w/HCl @ 0.47 lpm, flow through cell @ 70, P=720.5 torr						total = 90	E
	14:34-14:38	COOLS020		spiked w/HCl @ 0.48 lpm, flow through cell @ 70, P=720.6 torr							
	14:40-14:44	COOLS021		spiked w/HCl @ 0.48 lpm, flow through cell @ 70, P=720.5 torr							
10/22/96	10:30			Probe in outlet stack						just below 120	
	10:50			Probe in inlet stack						150	
	10:57			Outlet flow = 115, Inlet flow = 150							
	10:59-11:01	BAGOH001	40 passes	Baghouse Outlet	50	1.0	122C	U	H/W	115	B
	11:05-11:07	BAGOH002		Baghouse Outlet, Pressure = 719.1							
	11:09-11:11	BAGIH001		Baghouse Inlet	50	1.0	122C	U	H/W	115	B
	11:13-11:15	BAGIH002		Baghouse Inlet	50	1.0	122C	U	H/W	115	B
	11:17-11:19	BAGOH003		Baghouse Outlet	50	1.0	122C	U	H/W	115	B
	11:22-11:24	BAGOH004		Baghouse Outlet							
	11:27-11:28	BAGIH003		Baghouse Inlet	50	1.0	122C	U	H/W	115	B
	11:32-11:34	BAGIH004		Baghouse Inlet	50	1.0	122C	U	H/W	115	B
	11:38-11:40	BAGOC005		Baghouse Outlet	50/200	1.0	122C	U	Cond.	Total = 115	B
	11:43-11:44	BAGOC006		Baghouse Outlet	50/200	1.0	122C	U	Cond.	Total = 115	B
	11:44			started nitrogen through condenser							
	11:46-11:47	BAGIH005		Baghouse Inlet	50/200	1.0	122C	U	H/W	150	B
	11:50-11:52	BAGIH006		Baghouse Inlet	50/200	1.0	122C	U	H/W	150	B
	11:55-11:57	BAGOH007		Baghouse Outlet	50/200	1.0	122C	U	H/W	110	B
	12:00-12:02	BAGOH008	40 passes	Baghouse Outlet	50/200	1.0	122C	U	H/W	110	B

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/22/96	12:05-12:06	NITCON01		Nitrogen through condenser, saved in CTS d	50/200	1.0	122C	U	Cond.		B
	12:10-12:12	NITCON02		Nitrogen through condenser	50/200	1.0	122C	U	Cond.		B
	12:14-12:16	BAGIH007		Baghouse Inlet	50/200	1.0	122C	U	H/W	145	B
	12:17			started inlet sample through the condenser, flow through the condenser approx. 35							
	12:18-12:20	BAGIH008		Baghouse Inlet	50/200	1.0	122C	U	H/W	145	B
	12:24-12:27	BAGOH009		Baghouse Outlet	50/200	1.0	122C	U	H/W	110	B
	12:30-12:32	BAGOH010		Baghouse Outlet	50/200	1.0	122C	U	H/W	110	B
	12:35-12:37	BAGIC009		Baghouse Inlet (Condenser)	50/200	1.0	121C	U	Cond.	prox 30 to cond. , total=1	B
	12:39-12:41	BAGIC010		Baghouse Inlet (Condenser)	50/200	1.0	121C	U	Cond.	prox 30 to cond. , total=1	B
	12:45-12:52	BAGOF011		Baghouse Outlet P=720	50/200	1.0	121C	U	H/W	total=110	B
	12:46			started N2 through the condenser							
	12:54-13:01	BAGOF012		Baghouse outlet flowing through cell @ 55, P	50/200	1.0	121C	U	H/W	total=110	B
	13:07-13:14	BAGIF011		Baghouse inlet flowing through cell, P=719.3	50/200	1.0	121C	U	H/W	145, w/sample through ce	B
	13:05	empty001		Evacuated cell and jumper line to manifold--- In Bagout directory							
	13:15-13:21	BAGIF012		Baghouse Inlet flowing through cell P=719.6	50/200	1.0	121C	U	H/W	total=145	B
10/22/96	13:27-13:33	BAGOF013	40 passes	Outlet flowing through cell, P=719.9	50/200	1.0	122C	U	H/W	total=115	B
	13:35-13:36	NITCON03	40 passes	Nitrogen through condenser, P=718	50/200	1.0	122C	U	H/W	total=115	B
	13:56-13:58	BAGOH014	40 passes	static sample, outlet	50/200	1.0	122C	U	H/W	total=110	C
	14:02-14:08	BAGIF013	40 passes	Inlet flowing sample, P=718.2	50/200	1.0	122C	U	H/W	45, w/sample through ce	C
	14:10-14:12	BAGIH014	40 passes	Inlet, static, P=717.6	50/200	1.0	122C	U	H/W	total=145	C
	14:14-14:18	BAGOF015	40 passes	Outlet flowing, P=717.9	50/200	1.0	122C	U	H/W	10, w/sample through ce	C
	14:21-14:22	BAGOH016	40 passes	Outlet static, P=717.5	50/200	1.0	122C	U	H/W	total=110	C
	14:26-14:30	BAGIF015	40 passes	Inlet, flowing sample	50/200	1.0	122C	U	H/W	45, w/sample through ce	C
	14:33-14:34	BAGIH016	40 passes	Inlet, static sample P=717.4	50/200	1.0	122C	U	H/W	total=145	C
	14:39-14:41	BAGOH017		Outlet, static sample	50/200	1.0	122C	U	H/W	total=110	C
	14:38			SF6 spike on to inlet @ 2.00 lpm							
	14:44-14:49	BAGOF018		Outlet flowing, P =717.2	50/200	1.0	122C	U	H/W	total=110	C
	14:51-14:52	BAGIS017	40 passes	SF6 spike @ 2.00 lpm, Inlet: static sample	50/200	1.0	122C	S(SF6)	H/W	145	C
	14:55-14:56	BAGIS018	40 passes	SF6 spike @ 1.98 lpm, Inlet: static sample	50/200	1.0	122C	S(SF6)	H/W	145	C
	14:58-15:00	BAGIS019	40 passes	SF6 spike @ 1.98 lpm, Inlet: static sample	50/200	1.0	122C	S(SF6)	H/W	145	C
	15:04			started HCl spike to inlet @ 2.03 lpm							
	15:04-15:06	BAGOH020	40 passes	Outlet static sample	50/200	1.0	122C	U	H/W	110	C
	15:09-15:14	BAGOF020	40 passes	Outlet flowing sample, P=718.7	50/200	1.0	122C	U	H/W	total=110, w/sample =45	C
	15:16-15:18	BAGIS020	40 passes	Inlet static, HCl spike @ 2.00 lpm, P=716.8	50/200	1.0	122C	S(HCl)	H/W	145	C
	15:20-15:22	BAGIS021	40 passes	Inlet static, HCl spike @ 2.00 lpm, P=716.8	50/200	1.0	122C	S(HCl)	H/W	145	C
	15:24-15:27	BAGIS022	40 passes	Inlet static, HCl spike @ 2.05 lpm, P=716.8	50/200	1.0	122C	S(HCl)	H/W	145	C
	15:30-15:36	BAGISF23	40 passes	Inlet spiked and flowing HCl @ 2.05 lpm, P=	50/200	1.0	122C	S(HCl)	H/W	total=145, w/sample=110	C
	15:38-15:43	BAGISF24	40 passes	Inlet spiked and flowing HCl @ 2.05 lpm, P=	50/200	1.0	122C	S(HCl)	H/W	total=145, w/sample=50	C
	15:44-15:47	BAGISF25	40 passes	Inlet spiked and flowing HCl @ 2.00 lpm, P=	50/200	1.0	122C	S(HCl)	H/W	total=145	C
	15:49-15:51	BAGOH021	40 passes	Outlet: static	50/200	1.0	122C	U	H/W	110	C
	15:54-15:56	BAGIS026	40 passes	Inlet: static HCl 2.05 lpm	50/200	1.0	122C	S	H/W	145	C
	15:58-16:00	BAGOH022	40 passes	Outlet unspiked	50/200	1.0	122C	U	H/W	115	C
	16:03-16:05	BAGIS027	40 passes	Inlet spiked HCl spike @ 2.00 lpm	50/200	1.0	122C	S	H/W	145	C

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/22/96	16:08-16:11	BAGOH023	40 passes	Outlet H/W	50/200	1.0	122C	U	H/W	110	C
	16:13-16:16	BAGIS028	40 passes	Inlet HCl spike @ 1.97 lpm	50/200	1.0	122C	S	H/W	145	C
	16:19-16:21	BAGOH024	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	16:24-16:25	BAGIS029	40 passes	Inlet spike HCl spike @ 1.97 lpm	50/200	1.0	122C	S	H/W	145	C
	16:28-16:30	BAGOH025	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	16:33-16:34	BAGISO30	40 passes	Inlet spike HCl spike @ 1.96 lpm	50/200	1.0	122C	S	H/W	145	C
	16:37-16:39	BAGOH026	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	16:42-16:43	BAGIS031	40 passes	Inlet spike HCl spike @ 1.99 lpm	50/200	1.0	122C	S	H/W	145	C
	16:46-16:48	BAGOH027	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	16:51-16:53	BAGIS032	40 passes	Inlet spike HCl spike @ 1.98 lpm	50/200	1.0	122C	S	H/W	145	C
	16:55-16:57	BAGOH028	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	17:02-17:03	BAGIS033	40 passes	Inlet spike HCl spike @ 1.98 lpm	50/200	1.0	122C	S	H/W	145	C
	17:05-17:07	BAGOH029	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	17:10-17:11	BAGIS034	40 passes	Inlet spike HCl spike @ 1.98 lpm	50/200	1.0	122C	S	H/W	145	C
	17:14-17:16	BAGOH030	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	17:18-17:20	BAGIS035	40 passes	Inlet spike HCl spike @ 1.97-2.00 lpm	50/200	1.0	122C	S	H/W	145	C
	17:23-17:24	BAGOH031	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	17:27-17:28	BAGIS036	40 passes	Inlet spike HCl spike @ 2.00 lpm	50/200	1.0	122C	S	H/W	145	C
	17:31-17:33	BAGOH032	40 passes	outlet H/W	50/200	1.0	122C	U	H/W	110	C
	17:37-17:39	BAGIS037	40 passes	Inlet spike HCl spike @ 2.00 lpm	50/200	1.0	122C	S	H/W	145	C
	17:40			HCl spike off							
	17:41			SF6 spike on @ 1.98 lpm							
	17:55-17:57	BAGIS038	40 passes	Inlet spiked w/SF6 @ 1.97- 2.00 lpm	50/200	1.0	122C	S	H/W	145	C
	18:00-18:01	BAGISO39	40 passes	Inlet spiked w/SF6 @ 1.96 lpm	50/200	1.0	122C	S	H/W	145	C
	18:04-18:06	BAGIS040	40 passes	Inlet spiked w/SF6 @ 1.95 lpm	50/200	1.0	122C	S	H/W	145	C
				Probes out of stack @ 16:12 @ Inlet							
	18:13-18:15	BAGIA041	40 passes	ambient sample through inlet line	50/200	1.0	122C	S	H/W	150	C
	18:17-18:19	BAGIA042	40 passes	ambient sample through inlet line							
	18:19			Outlet probe out of stack							
	18:23-18:25	BAGOA033	40 passes	Outlet ambient air through probe	50/200	1.0	122C	S	H/W	115	C
	18:28-18:29	BAGIA043	40 passes	Inlet ambient air through probe	50/200	1.0	122C	S	H/W	150	C
	18:32-18:34	BAGOA034	40 passes	Outlet ambient air through probe	50/200	1.0	122C	S	H/W	115	C
	18:37-18:39	BAGIA044	40 passes	Inlet ambient air through probe	50/200	1.0	122C	S	H/W	150	C
	18:55-18:57	BAGOA035	40 passes	Outlet ambient air through probe	50/200	1.0	122C	S	H/W	115	C
	19:00-19:01	BAGIA045	40 passes	Inlet ambient air through probe	50/200	1.0	122C	S	H/W	150	C
)/23/96	8:50			Outlet probe had been in stack about 5 minutes. Removed from stack to collect ambient air sample.							
	10:28-10:30	BAGOA201	40 passes	Baghouse outlet ambient through probe (P=7	50/100	1.0	122C	U	H/W	115	B
	10:34-10:36	BAGOA202	40 passes	Baghouse outlet ambient through probe	50/100	1.0	122C	U	H/W	115	B
	10:37-10:39	BAGIA201	40 passes	Inlet ambient air	50/100	1.0	122C	U	H/W	115	B
	10:40		40 passes	Outlet probe in stack							
	10:45		40 passes	Inlet probe in stack							

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	10:59-11:01	BAGOH203	40 passes	Outlet H/W (P=713.3)	50/100	1.0	122C	U	H/W	105	B
	11:05-11:07	BAGIH202	40 passes	Inlet H/W (diluted by an unknown factor b/c I	50/100	1.0	122C	U	H/W	100	B
	11:11-11:12	BAGOH204	40 passes	Outlet H/W	50/100	1.0	122C	U	H/W	105	B
	11:15-11:17	BAGIH203	40 passes	Inlet H/W (diluted by an unknown factor b/c I	50/100	1.0	122C	U	H/W	95	B
	11:21-11:23	BAGOH205	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	11:27-11:30	BAGIH204	40 passes	Inlet	50/100	1.0	122C	U	H/W	85	B
	11:33-11:34	BAGOH206	40 passes	Outlet	50/100	1.0	122C	U	H/W	100	B
	11:40-11:43	BAGIH205	40 passes	Inlet	50/100	1.0	122C	U	H/W	85	B
	11:45		40 passes	Removing inlet probe to take out some of the glass wool to try and increase the flow. flow is even less with probe back in. Will remove probe to see if							
	11:46-11:47	BAGOH207	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	12:07-12:11	BAGIH206	40 passes	Inlet-- flow was high with probe off	50/100	1.0	122C	U	H/W	65	B
	12:17-12:18	BAGOH208	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	12:22-12:26	BAGIH207	40 passes	Inlet	50/100	1.0	122C	U	H/W	55	B
10/23/96	12:29			low flow @ inlet, remove probe again and change filter to make sure it is not clogged. Continue testing at outlet.							
	12:30-12:32	BAGOH209	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	12:37-12:38	BAGOH210	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	12:41-12:44	BAGOH211	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
	12:47-12:48	BAGOH212	40 passes	Outlet, P=714.2	50/100	1.0	122C	U	H/W	105	B
	12:51-12:52	BAGOH213	40 passes	Outlet, P=713.9	50/100	1.0	122C	U	H/W	105	B
	12:55-12:57	BAGOH214	40 passes	Outlet, P=714.6	50/100	1.0	122C	U	H/W	105	B
	13:01	BAGIF208	40 passes	Inlet (new filter) flowing through cell @ P=71	50/100	1.0	122C	U	H/W	60, w/sample = 45	B
	13:07			started SF6 spike to outlet @ 1.00 lpm							
	13:10-13:16	BAGIF209		Inlet flowing sample	50/100	1.0	122C	U	H/W	total = 55	
	13:17-13:18	BAGOS215	40 passes	SF6 spike @ 1.00 lpm (P=713.5)	50/100	1.0	122C	S(SF6)	H/W	total = 95	B
	13:21-13:23	BAGOS216		SF6 spike @ 1.00 lpm (P=713.5)	50/100	1.0	122C	S(SF6)	H/W	95	B
	13:25-13:27	BAGOS217	40 passes	SF6 spike @ 1.00 lpm (P=713.5)	50/100	1.0	122C	S(SF6)	H/W	95	B
	13:30-13:36	BAGIF210		Inlet flowing	50/100	1.0	122C	U	H/W	55 w/sample, total =95	
	13:31			HCl spike on to outlet @ 1.00 lpm							
	13:37	EMPTY 201		Evacuated cell and jumper line (in BAGOUT directory)						95	
	13:39-13:45	BAGIF211		Inlet flowing	50/100	1.0	122C	U	H/W	55	
	13:48-13:50	BAGOS218	40 passes	spike had cut off, reset spike flow @ 13:50, unknown in spike level				S	H/W	95	B
	13:54-13:59	BAGIF212	40 passes	flowing through cell	50/100	1.0	122C	U	H/W	55	B
	14:00-14:02	BAGOS219	40 passes	outlet HCl @ 1.00 lpm	50/100	1.0	122C	S	H/W	95	B
	14:05-14:07	BAGOS220	40 passes	outlet HCl @ 1.00 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B
	14:09-14:11	BAGOS221	40 passes	outlet HCl @ 1.00 lpm (P=713.5)	50/100	1.0	122C	S(HCl)	H/W	95	B
	14:14-14:20	BAGIF213	40 passes	Inlet	50/100	1.0	122C	U	H/W	50	B
	14:22-14:23	BAGOS222	40 passes	Outlet HCl spike @ 1.00 lpm dropped 100.6	50/100	1.0	122C	S(HCl)	H/W	95	B
	14:27-14:34	BAGIF214	40 passes	Inlet flowing	50/100	1.0	122C	U	H/W	50	B
	14:36-14:37	BAGOS223	40 passes	HCl @ 1.00 lpm	50/100	1.0	122C	S	H/W	95	B
	14:42-14:47	BAGIF215	40 passes	Inlet flowing	50/100	1.0	122C	U	H/W	48	B
	14:56-14:57	BAGOS224	40 passes	Outlet HCl spike @ 1.04 (P=713)	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:01-15:07	BAGIF216	40 passes	Inlet flowing	50/100	1.0	122C	U	H/W	45	B
	15:11-15:12	BAGOS225	40 passes	Outlet HCl spike @ 1.03 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	15:14-15:15	BAGOS226	40 passes	Outlet HCl spike @ 1.03 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:18-15:19	BAGOS227	40 passes	Outlet HCl spike @ 1.00 lpm (P=712.4)	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:22-15:23	BAGOS228	40 passes	Outlet HCl spike @ 1.00 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:26-15:27	BAGOS229	40 passes	Outlet HCl spike @ 1.00 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:29-15:31	BAGOS230	40 passes	Baghouse outlet HCl spike @ 1.00 lpm	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:33-15:34	BAGOS231	40 passes	Baghouse outlet HCl spike @ 1.00 lpm, P=71	50/100	1.0	122C	S(HCl)	H/W	95	B
	15:38			HCl spike off SF6 spike on @ 1.00 lpm							
	15:39-15:49	BAGIF217	40 passes	Inlet flowing	50/100	1.0	122C	U	H/W	45	B
	15:53-15:54	BAGOS232		SF6 @ 0.98 lpm. P=712.3	50/100	1.0	122C	S(SF6)	H/W	95	B
	15:54			Inlet probe out of stack							
	15:56-15:58	BAGOS233	40 passes	Outlet SF6 @ 0.99 lpm	50/100	1.0	122C	S(SF6)	H/W	95	B
	16:00-16:02	BAGOS234	40 passes	Outlet SF6 @ 0.98 lpm	50/100	1.0	122C	S(SF6)	H/W	95	B
	16:03			SF6 spike off							
	16:05-16:10	BAGIA218		ambient air through inlet probe	50/100	1.0	122C	U	H/W	55	B
	16:12-16:13	BAGOH235		H/W outlet	50/100	1.0	122C	U	H/W	95	B
	16:17			Outlet probe out of stack							
	16:37	BAGOA236	40 passes	ambient through outlet probe	50/100	1.0	122C	U	H/W	95	C
	16:41-16:46	BAGIA219	40 passes	ambient through inlet probe	50/100	1.0	122C	U	H/W	55	C
	17:00-17:01	BAGOA237	40 passes	ambient outlet mixed w/3 lpm N2 (P=712.6)	50/100	1.0	122C	S(N2)	H/W	100	C
	17:08-17:12	BAGIA220		ambient through inlet probe	50/100	1.0	122C	U	H/W	50	C
	17:14-17:15	BAGOA238	40 passes	ambient through outlet, some residual HCl re	50/100	1.0	122C	U	H/W	95	C
	17:20			started N2 purge through both sample lines total = 3.0 lpm							
10/24/96	11:08-11:10	BAGOH302	20 passes	Baghouse Outlet	50/100	1.0	121C	U	H/W	95	E
	11:13-11:14	BAGIH302	20 passes	Baghouse Inlet	50/100	1.0	121C	U	H/W	125	E
	11:17-11:19	BAGOH303	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	E
	11:21-11:22	BAGIH303	20 passes	Inlet	50/100	1.0	121C	U	H/W	125	E
	11:25-11:26	BAGOH304	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	E
	11:29-11:30	BAGIH304	20 passes	Inlet, P=718.3	50/100	1.0	121C	U	H/W	125	E
	11:33-11:34	BAGOH305	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	E
	11:37-11:38	BAGIH305	20 passes	Inlet	50/100	1.0	121C	U	H/W	125	E
	11:42-11:44	BAGOH306	20 passes	Outlet, P=718.2	50/100	1.0	121C	U	H/W	95	E
	11:46-11:47	BAGIH306	20 passes	Inlet	50/100	1.0	121C	U	H/W	125	E
	11:54			new background with N2							F
	11:58-12:00	BAGOH307	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	F
	12:02-12:05	BAGIH307	20 passes	Inlet	50/100	1.0	121C	U	H/W	115	F
	12:08-12:10	BAGOH308	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	F
		EMPTY 301		Evacuated cell and jumper line	50/100	1.0	121C	U	H/W		F
	12:14-12:16	BAGIH305	20 passes	Inlet	50/100	1.0	121C	U	H/W	110	F
	12:21-12:22	BAGOH309	20 passes	Baghouse Outlet	50/100	1.0	121C	U	H/W	95	F
	12:25-12:28	BAGIH309	20 passes	Inlet	50/100	1.0	121C	U	H/W	95	F
	12:32-12:34	BAGOH310	20 passes	Outlet	50/100	1.0	121C	U	H/W	95	F
	12:33-12:37	BAGIH310	20 passes	Baghouse Inlet	50/100	1.0	122C	U	H/W	88	F
	12:44-12:46	BAGOH311	20 passes	Baghouse Outlet	50/100	1.0	122C	U	H/W	95	F

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/24/96	12:48-12:52	BAGIH311	20 passes	Baghouse Inlet	50/100	1.0	122C	U	H/W	80	F
	12:59-13:01	BAGOH312	20 passes	Outlet	50/100	1.0	122C	U	H/W	95	F
	13:04-13:07	BAGIH312	20 passes	Inlet, P=717.6	50/100	1.0	122C	U	H/W	78	F
	13:09-13:11	BAGOH313	20 passes	Outlet	50/100	1.0	122C	U	H/W	95	F
	13:13-13:16	BAGIH313	20 passes	Inlet	50/100	1.0	122C	U	H/W	68	F
	13:18-13:20	CONN2301		Nitrogen through condenser	50/100	1.0	122C	U	Cond.	P=718.1	F
	13:23			started outlet through the condenser							
	13:24-13:26	BAGOH314		Outlet, H/W	50/100	1.0	122C	U	H/W	total=95	F
	13:29-13:31	BAGOC315	20 passes	Outlet condenser Sample	50/100	1.0	122C	U	Cond.	95	F
	13:33-13:35	BAGOC316	20 passes	Outlet condenser Sample	50/100	1.0	122C	U	Cond.	95	F
	13:36			started nitrogen through condenser							
	13:38-13:42	BAGIH314	20 passes	Inlet Hot/wet	50/100	1.0	122C	U	H/W	58	F
	13:45-13:47	CONN2302		Nitrogen through condenser, P=717.4	50/100	1.0	122C	U	H/W		F
	13:48			Started Inlet sample through condenser							
	13:50-13:53	BAGIC315		Inlet sample through condenser	50/100	1.0	122C	U	H/W	50	F
	13:55-13:58	BAGIC316		Inlet sample through condenser	50/100	1.0	122C	U	H/W	43	F
10/24/96	14:07-14:09	BAGOH317	20 passes	Outlet, P=717.1	50/100	1.0	122C	U	H/W	95	F
	14:10			Removed a clog. Flow now improved at Inlet							
	14:15			Flowmeter reading @ 0 flow = -0.08							
	14:16			started SF6 spike to outlet @ 0.49 lpm							
	14:18-14:20	BAGIH317	20 passes	Inlet H/W	50/100	1.0	122C	U	H/W	145	F
	14:22-14:24	BAGOS318	20 passes	spiked w/SF6 @ 0.48 lpm	50/100	1.0	122C	S	H/W	94	F
	14:27-14:28	BAGOS319	20 passes	spiked w/SF6 @ 0.47 lpm	50/100	1.0	122C	S	H/W	94	F
	14:31-14:32	BAGIH318	20 passes	Inlet H/W, P=717.3	50/100	1.0	122C	S	H/W	140	F
	14:35-14:37	BAGOS320	20 passes	Outlet H/W spiked w/SF6 @ 0.49lpm	50/100	1.0	122C	S	H/W	94	F
	14:41			started HCl spike @ 0.52 lpm							
	14:48-14:49	BAGIH319	20 passes	Inlet H/W, P=717.1	50/100	1.0	122C	U	H/W	145	F
	14:53-14:54	BAGOS321	20 passes	spiked HCl @ 0.52 lpm	50/100	1.0	122C	S	H/W	94	F
	15:00-15:02	BAGIH320	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	15:05-15:07	BAGOS322	20 passes	Outlet spike HCl @ 0.52 lpm, P=724.7	50/100	1.0	122C	S	H/W	94	F
	15:10-15:11	BAGOS323	20 passes	Outlet spike HCl @ 0.51 lpm	50/100	1.0	122C	S	H/W	94	F
	15:13-15:14	BAGIH321	20 passes	Inlet, P=717.5	50/100	1.0	122C	U	H/W	140	F
	15:18-15:19	BAGOS324	20 passes	Outlet, HCl spike @ 0.50 lpm	50/100	1.0	122C	S	H/W	93	F
	15:24-15:25	BAGIH322	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	15:28-15:29	BAGOS325	20 passes	Outlet, HCl spike @ 0.49 lpm	50/100	1.0	122C	S	H/W	93	F
	15:33-15:34	BAGIH323	20 passes	Inlet. P=717.5	50/100	1.0	122C	U	H/W	140	F
	15:37-15:38	BAGOS326	20 passes	Outlet, HCl spike @ 0.48 lpm	50/100	1.0	122C	S	H/W	93	F
	15:41-15:42	BAGIH324	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	15:44-15:46	BAGOS327	20 passes	Outlet, HCl spike @ 0.48 lpm	50/100	1.0	122C	S	H/W	93	F
	15:50-15:51	BAGIH325	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	15:53-15:55	BAGOS328	20 passes	Outlet, HCl spike @ 0.48 lpm	50/100	1.0	122C	S	H/W	92	F
	15:58-16:00	BAGOS329	20 passes	Outlet, HCl spike @ 0.49 lpm	50/100	1.0	122C	S	H/W	92	F
	16:02-16:04	BAGOS330	20 passes	Outlet, HCl spike @ 0.49 lpm	50/100	1.0	122C	S	H/W	92	F

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
10/24/96	16:06-16:07	BAGIH326	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	16:09-16:11	BAGOS331	20 passes	Outlet HCl @ 0.51 lpm	50/100	1.0	122C	S	H/W	92	F
	16:14-16:15	BAGOS332	20 passes	Outlet HCl @ 0.52 lpm	50/100	1.0	122C	S	H/W	92	F
	16:17-16:19	BAGOS333	20 passes	Outlet HCl @ 0.52 lpm	50/100	1.0	122C	S	H/W	92	F
	16:21			HCl spike off, SF6 spike on @ 0.49 lpm							
	16:23-16:24	BAGIH327	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	16:28-16:29	BAGOS334	20 passes	SF6 spike @ 0.47 lpm outlet	50/100	1.0	122C	S	H/W	92	F
	16:32-16:34	BAGIH328	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
10/24/96	16:36-16:37	BAGOS335	20 passes	Outlet spiked w/SF6 @ 0.49 lpm, P=716.9	50/100	1.0	122C	S	H/W	92	F
	16:40-16:41	BAGIH329	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	16:44-16:45	BAGOS336	20 passes	Outlet spiked w/SF6 @ 0.48 lpm	50/100	1.0	122C	S	H/W	92	F
	16:47			SF6 spike off							
	16:50-16:52	BAGIH330	20 passes	Inlet H/W	50/100	1.0	122C	U	H/W	140	F
	17:06-17:08	BAGOH337	20 passes	Outlet Unspiked	50/100	1.0	122C	U	H/W	92	F
	17:15-17:16	BAGIH331	20 passes	Inlet Unspiked, P=717.2, pumped out and r	50/100	1.0	122C	U	H/W	140	F
	17:19-17:20	BAGOH338	20 passes	Outlet Unspiked	50/100	1.0	122C	U	H/W	92	F
	17:22-17:24	BAGIH332	20 passes	Inlet, P=717.3	50/100	1.0	122C	U	H/W	140	F
	17:26-17:27	BAGOH339	20 passes	Outlet	50/100	1.0	122C	U	H/W	92	F
	17:31-17:33	BAGIH333	20 passes	Inlet, started Inlet to Cond.	50/100	1.0	122C	U	H/W	140	F
	17:34-17:36	BAGOH340	20 passes	Outlet	50/100	1.0	122C	U	H/W	92	F
	17:40-17:41	BAGIC334	20 passes	Inlet	50/100	1.0	122C	U	Cond.	140	F
	17:44-17:45	BAGOH341	20 passes	Outlet	50/100	1.0	122C	U	H/W	92	F
	17:48-17:49	BAGIC335	20 passes	Inlet/Condenser	50/100	1.0	122C	U	Cond.	140	F
	17:51			started nitrogen to condenser							
	17:52-17:54	BAGOH342	20 passes	Outlet	50/100	1.0	122C	U	H/W	92	F
	17:56-17:57	CONN23O3	20 passes	nitrogen through condenser, (In BAGIN)	50/100	1.0	122C	U	H/W		F
	18:01-18:02	BAGIH336	20 passes	Inlet to baghouse	50/100	1.0	122C	U	H/W	140	F
	18:01			Outlet sample to condenser							
	18:12-18:13	BAGOC343	20 passes	Outlet through condenser	50/100	1.0	122C	U	Cond.	90	F
	18:17-18:18	BAGIH337	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	18:21-18:22	BAGOC344	20 passes	Outlet through condenser	50/100	1.0	122C	U	Cond.	90	F
	18:23			Nitrogen to condenser							
	18:26-18:27	BAGOH345	20 passes	Outlet, P=717.7	50/100	1.0	122C	U	H/W	90	F
	18:30-18:31	BAGIH338	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	18:34-18:36	BAGOH346	20 passes	Outlet	50/100	1.0	122C	U	H/W	90	F
	18:38-18:39	CONN2304	20 passes	Nitrogen through condenser, (In BAGIN)	50/100	1.0	122C	U	H/W		F
	18:42-18:43	BAGOH347	20 passes	Outlet	50/100	1.0	122C	U	H/W	90	F
	18:46-18:47	BAGIH339	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	18:50-18:52	BAGOH348	20 passes	Outlet	50/100	1.0	122C	U	H/W	90	F
	18:55-18:56	BAGIH340	20 passes	Inlet, P=718.2	50/100	1.0	122C	U	H/W	140	F
	18:59-19:01	BAGOH349	20 passes	Outlet	50/100	1.0	122C	U	H/W	90	F
	19:03-19:04	BAGIH341	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	19:07-19:08	BAGOH350	20 passes	Outlet	50/100	1.0	122C	U	H/W	90	F

Data Sheet: FTIR Batch Samples: APG Lime Kiln. EPA Work Assignment 4-01.

Date	Sample Time	File name	Path	Location/Notes	#scans	Res (cm-1)	Cell Temp (F)	Spk/Unsp	Sample Cond.	Sample Flow	BKG
	19:12-19:14	BAGIH342	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
	19:15-19:17	BAGOH351	20 passes	Outlet, P=718.5	50/100	1.0	122C	U	H/W	90	F
	19:20-19:21	BAGIH343	20 passes	Inlet	50/100	1.0	122C	U	H/W	140	F
10/24/96	19:24-19:25	BAGOH352	20 passes	Baghouse Outlet	50/100	1.0	122F	U	H/W	90	F
	19:28-19:29	BAGIH344	20 passes	Inlet	50/100	1.0	122F	U	H/W	140	F
	19:32-19:34	BAGOH353	20 passes	Outlet	50/100	1.0	122F	U	H/W	90	F
	19:36-19:37	BAGIH345	20 passes	Inlet	50/100	1.0	122F	U	H/W	140	F
	19:39-19:41	BAGOH354	20 passes	Outlet	50/100	1.0	122F	U	H/W	90	F
	19:43-19:44	BAGIH346	20 passes	Inlet	50/100	1.0	122F	U	H/W	139	F
	19:46-19:48	BAGOH355	20 passes	Outlet	50/100	1.0	122F	U	H/W	90	F
	19:50-19:52	BAGOH356	20 passes	Outlet and sample, lost sample---manual run	50/100	1.0	122F	U	H/W	90	F
	19:52			Inlet probe out of stack							
	19:57			Outlet probe out of stack							
	20:03-20:04	BAGIA347	20 passes	Inlet ambient sample	50/100	1.0	122F	U	H/W	140	G
	20:07-20:08	BAGOA357	20 passes	Outlet ambient air through probe	50/100	1.0	122F	U	H/W	90	G
	20:12-20:13	BAGIA348	20 passes	Inlet ambient air through probe	50/100	1.0	122F	U	H/W	140	G
	20:16-20:18	BAGOA358	20 passes	Outlet, ambient air through probe	50/100	1.0	122F	U	H/W	90	G
	20:34-20:36	BAGOA359	20 passes	Outlet, ambient air through probe	50/100	1.0	122F	U	H/W	90	G
	20:39-20:41	BAGOA360	20 passes	Outlet, ambient air through probe	50/100	1.0	122F	U	H/W	90	G
	20:42-20:43	BAGIA349	20 passes	Inlet, ambient air through probe	50/100	1.0	122F	U	H/W	140	G

APG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date 10/21/96	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/21/96	9:51	BKG1021A	16 passes	cold cell. Located @ cooling stack.	100	2	ambient	719.6		NB/Med
	10:14	CTS1021A	"	" 99.4 ppm ethylene	50/100	2	"	"		"
	10:22	CTS1021B	"	" second fill after evacuating				718.1		
	10:33	CTS1021C	"	" third fill						
	cell @	Positive	Pressure	leak check time = 0	P =	823.1				
	120 C			time = 2 min	P =	825				
		Vacuum	leak	check time = 0	P =	5.3 torr				
				time = 2 min	P =	7.0				
10/21/96	11:35	BKG1021B	40 passes	N_2 Background.	100	1 cm^{-1}	119C	718.5		NB/Med
				turned path length back to	40 passes.					
	11:40	CTS1021D	40 passes	99.4 ppm Ethylene	50/100				B	
	11:45	CTS1021E		" " 2nd fill					B	
	11:49	BKG1021C		Dryer Chan B	100		120C	718.5		
	11:55	CTS1021F		20.0 ppm Ethylene	50/100	1.0	120C	718.8	C	NB/Med
	11:59	CTS1021G						718.5		

same setup as 10/9/96

APG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/21/96	12:09	BKG10211	40p		100	1.0	120C	718.5		DB/Mod
	1303	BKG10211	"					718.0		
	1305			Started SF6 spike @ ~~0.50~~ 0.48 lpm 50/100 4.01 ppm						
	14:52	HCLAPG01		HCl 103 ppm flowing through cell @ 120 on meter #2		1.0 cm⁻¹		718.5	E	E DB/M
	1502	HCLAPG02		HCl 103 ppm flow through cell @ 95 on meter #2				716.0	E	E
	1504	SF6APG01		SF6 4.01 ppm static in cell				717.5	E	E
	1512	SF6APG02		" " new fill.				"	E	"
	1515	BKG1021F			100	1.0	120C			DB/mod
	1523	CTS1021H		20.0 ppm Ethylene	50/100			717.1	F	"
	1528	CTS1021I		20.0 " 2nd fill						

APG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/21/96	18:39	BKG1021G	40 psi	Baghouse	100	1.0	120C	717.9		NB/med
	18:45	CTS1021J	"	20.0 ppm Ethylene	50/100	1.0	"	718.0	G	"
	18:53	CTS1021K	"	" "	"	"	"		→	
10/22/96	8:40	BKG1022A	40 psi	N_2 Background in cold cell	100	2.0	Ambient	719.1		NB/med
	8:48	CTS1022A	"	20.0 ppm Ethylene	50/100	→		→	A	→
	8:55	CTS1022B	"	" " 2nd fill		→		719.0	→	→
	9:00	CTS1022C		" " 3rd fill	→	→	→		→	→
	Vacuum leak check			Time = 0 P = 6.8 Time = 2 min P = 12.0						
	Pos Pressure " "			Time = 0 P = 769.1 time = 1 min P = 768.9						
Some CO flowing from manifold into BKG spectra	10:48	BKG1022B	40 psi	Dry N_2	200	1.0	120C	719.3		NB/med
	10:51	CTS1022D	"	20.0 ppm Ethylene	50/200	"	"	"	B	"
	10:56	CTS1022E		" " 2nd fill	"	"	"	719.3	B	"
	RMS	2700 -	2900	cm^{-1} = 1.4×10^{-3}						

Sample line leak checks completed @ 10:30 and both lines purging since

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/22/96		BK61022C	40 passes	New N2 Background to get rid of ice Band.	200	1.0	122C	717.9		NB/med
	1844	SF6APG00	"	SF6 4.01 ppm	50/200	1.0	"	716.9	C	"
	1847	SF6APG04	"	" "	"	"	"	716.	C	"
		~~HClAPG03~~	~~"~~	~~HCl 103 ppm~~	~~"~~	~~"~~	~~"~~		~~C~~	~~"~~
1907	~~1907~~	BK61022D	40	N2 Background w/ N2	200	"	"	717.1		NB/med
	1915	HClAPG03	"	HCl 103 ppm P=717.1 - 718.5 flow through cell	50/200			~~718.5~~	D	"
	1920	HClAPG04	"		"	"	"	718.5	D	"
	1928	BK61022E	40 passes	N2 in Cell	"	"	"	717.0		NB/med
	1933	CTS1022F	"	20.0 ppm Ethylene	"	"	"	716.8	D	"
	1937	CTS1022G	"	" "	"	"	"	716.8	D	"
	~~1620~~	~~BK61025~~								

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/23/96	854	BKG 1023A	40 passes	N_2 Background	100	2.0	25C	713.6	—	NB/mod
	9:02	CTS 1023 A	"	20.0 ppm Ethylene	50/100	2.0	"	713.4	A	"
	9:05	CTS 1023 B	"	" " 2nd fill	"	"	"	-	A	"
	9:11	CTS 1023 C	"	" 3rd fill	"	"	"	"	A	"
	Positive Pressure leak check through manifold. time = 0 P = 858.1 time = 1 min P = 861.6									
	1020	BKG 1023B	"	N_2 in cell static	100	1.0	121C	713.6	—	NB/mod
	10:52	CTS 1023 D	↓	20.0 ppm Ethylene	50/100				B	—
	10:57	CTS 1023 E		" "	"	"	"	713.4 "	B	"
	Vacuum leak check Time = 0 P = 6.7 Time = 1 min P = 9.4									
	1625	BKG 1023C	"	N_2 Background	100	1.0	122C	713.1	—	NB/mod
	1629	CTS 1023 F	"	20.0 ppm Ethylene	50/100	"	"	712.7	C	"
	1633	CTS 1023 G	"	" " 2nd fill	"	"	"		"	"

ABG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F (67.7 F)	Press.	BKG	Apod
10/24/96	838	BKG1024A	40	N_2 cold	100	1.0	19.5C	717.01	–	NB/mod
	842	BKG1024B	"	" " same fill	"	2.0				
		Purged flow meter + sample line w/ N_2 overnight								
Contaminated from melted tube →	850	Bag0N201	"	N_2 purge through and line	50/100	1.0	"	"	A	"
		Bag1N201	"	" " " outlet				719.3		
	9:07	CTS1024A	40 passes	20.0 ppm Ethylene	50/100	2.0	19.5C	717.6	B	NB/mod
for longpath aperture in 1.5 mm.		CTS1024B		" " " 2nd fill	"	"	-	717.8	B	"
	9:10		good	leak @ inlet new filter, no glass wool plug.						
				realigned cell to 20 passes & closed aperture to minimum (0.75 mm)						
	9:50	BKG1024C	20 passes	decreased path length	100	2.0	23.2C	718.2	–	NB/mod
	9:58	CTS1024C		200 ppm Ethylene	50/100	2.0	"	"	C	"
	10:01	CTS1024D		" " " 2nd fill	"	2.0	"	"	C	"
	10:09	CTS1024E		99.4 ppm Ethylene	50/100	2.0	"	"	C	"
	10:12	CTS1024F		" " " 2nd fill	"	2.0	"	"	"	"
	1031	BKG1024D	20 passes	N_2 Background	100	1.0	111C	718.1	–	"
	1034-1036	Bag0A301	"	ambient sample from outlet flow through @ 725.8 mm / flow = 95 → 116C →	50	"	113C	"	D	"
	1038-	Bag1A301	"	ambient from outlet flow through 90-						

APG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time 1057	File Name	Path M	Location/ Notes N2 Background	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/24/96		BKG 1024E	20 passes	Probes in Stack.	100	1.0	119	718.6	—	NB/med
	11:03	CTS1024G	"	99.4 Ethylene	100	1.0	120	"	E	"
	11:06	CTS1024H		" " 2nd fill						"
	1047	Outlet in Stack								
	1053	Outlet in Stack								
	1154	BKG1024F	20 passes	N2 in cell	100	1.0	127C	718.5	—	NB/med
	1959	BKG1024G	20 passes	N2 in Cell	100	1.0	122C	719.3	—	NB/med
	2023	CTS1024 I	20 passes	99.4 ppm Ethylene	50/100	"	"	718.7	G	"
	2028	CTS1024 J	"	" " " 2nd fill	"	"	"	718.8	G	"
	2031	CTS1024 K	"	" " " 3rd fill	"	"	"	718.7	G	"
	2049	BKG1024H	"	N2 in Cell	100	1.0	122C	718.8	-	"
	2053	SF6AP301		SF6 4.01 ppm	50/100	"	"	718.9	H	"
	2058	SF6AP302		" " 2nd Fill	"	"	"	"	H	"
	2102	SF6AP303		" " 3rd Fill	"	"	"	719.0	H	"

APG Lime

Data Sheet: FTIR CTS and Background Spectra. Lime Kilns. EPA W.A. 3804-01.

Date	Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Cell Temp. °F	Press.	BKG	Apod
10/24/92	2108	BKG1024I	2 passes	N_2 fill - Background	100	1.0	122C	719.1	–	NB/mod
	2118	HCIAP301	"	HCl direct from cylinder - flowing	50/100	"	"	718.2	I	"
		EMPAP30	evacuated cell	through cell			→	2.8	I	"
	2124	HClAP302	"	103 ppm HCl static sample	"	"	"	719.3	I	"
	2128	HClAP303	"	" " HCl static 3rd fill 2nd fill	"	"	"	719.4	I	"
	2134	HClAP304	"	103 ppm HCl flowing through cell 4th fill	"	"	"	718.8 to 719.9	I	"
	2131	EMPAP302		Evacuated Cell (before HCl AP304)	"		"	3.3	I	"
	2154	BKG1024J	"	N_2 in Cell	100	1.0	122C	719.5	–	NB/mod
	2200	CTS1024Q		99.4 ppm Ethylene	50/100	1.0	"	719.2	I	NB/mod
	2212	CTS1024M		" " " 2nd fill	"	"	"	719.4	I	"

APG Lime

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01,

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/21/96	12:02-12:04	Cool H001	40 pass	cooling stack APG	50/100	1.0	120C	U	H/W	90	C
	1212-1214	Cool H002	"							>90	D
	1216-1218	Cool C003		cooling stack.					Cond.	70 to Cond 90	D
	1221-1223	Cool C004									
	1226 1228	Cool H005							H/W		D
	12:29 12:32	Cool H006							H/W		D
	12:34 1236	Cool H007							H/W		+
	12:41-1242	Cool H008							H/W	90	D
	1245-1246	Cool H009									
	1248-1249	Cool H010		Pressure = 718.1						90	D
	1252 1253	Cool H011		Cooling stack				U			
	1255 1257	Cool H012						U			
10/21/96	13:15	turned on	SF_6	spike 0.48 lpm						90	
	13:24 1326	Cool S013		SF_6 @ 0.48 lpm	50/100	1.0	120C	S(SF_6)	H/W	90	E
	1328-1329	Cool S014		"	"	"	"	"	"	"	"
	1332-1333	Cool S015		" 0.48 lpm	"	"	"	"	"	"	"

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01,

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/21/96	13:43			Started HCl spike @ 0.52 lpm							
	1353-1355	Coo/S016		Spike w/ HCl 0.46 lpm							
	1400-1423	Cool S017		" 0.48 lpm		Static				Total=93	E
1426	1432	Cool S019		Spiked w/HCl @ 0.41 lpm			flow through cell @ 70		P = 720.5	Total=9	E
	14:34-1438	Cool S020		" 0.48 lpm		flow through		as in	S019	P = 720.	
	14:40-1444	Cool S021		Same as 020 & 021		HCl @ 0.48 lpm.				P = 720.5	

HCl spike is visible. more so with flow-through

HPG Lime

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/22/96	1030			Probe in Outlet Stack						just below 120	
	1050			Inlet						150	
	1057			outlet flow = 115							
				Inlet flow = 150							
10/22/96	10:59-11:01	Bag0H001	40 passes	Baghouse Outlet	50	1.0	122C	U	H/W	115	B
	11:05-11:07	Bag0H002		" "					P=719.1		
	11:09-11:11	BagIH001		Baghouse Inlet							
	11:13-11:15	BagIH002		" "							
	11:17-11:19	Bag0H003		outlet							
	11:22-11:24	Bag0H004		" outlet							
	11:27-11:29	BagIH003		" Inlet							
	11:32-11:34	BagIH004		" "							
	11:38-11:40	Bag0C005		Baghouse Outlet.	50/200	1.0cm⁻¹	122C	U	Condensed	Total ~115 1804 Cond	
	11:43-11:44	Bag0C006		" "							
	11:44	Started nitrogen through Condenser.									
	11:46-11:47	BagIH005		Baghouse Inlet	50/200	1.0	122C	U	H/W	150	B
	11:50-11:52	BagIH006		" "							
	11:55-11:57	Bag0H007		Baghouse Outlet					H/W	110	

~4% moisture

APG Line:

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F (FTIR Cell)	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/22/96	12:00-12:02	BagOH008	40 passes	Baghouse Outlet	50/200	1.0	122C	U	H/w	110	B
	12:05-12:06	NitCON01		Nitrogen through Condenser →					→ Cond		B
		↳ saved in ~~Bagout~~ CTS directory.							Cond		
	12:10-12:12	NitCon02		Nitrogen through Condenser						→	
	12:14-1216	BagIH007		Baghouse Inlet	50/200		→		H/w	145	B
	12:17	Started Inlet		sample through the Condenser.					flow through Condenser =	35	
	12:18-12:20	BagIH008		Baghouse Inlet	50/200	1.0	122C	U	H/w	145 →	
	12:24-12:27	BagOH009		" Outlet						110 →	B
	12:30-12:32	BagOH010		" "						→	
	12:35-12:37	BagIC009		Baghouse Inlet (Condenser)	50/200	1.0	121C	U	Condenser	~30% Cond. 145 total	B
Pressure = 718.6	12:39-12:40	BagIC010		" " "					→	→	
	12:45-12:52	BagOF011		Baghouse outlet. Sample flowing through cell @	50/200	1.0 z	121C	U	H/w	Total = 110 w/sample through cell =	B
						P= 720.0					
	12:46	Started N_2 through Condenser									
	1254-13:01	BagOF012		Baghouse outlet flowing through cell P= 718.9	50/200	1.0	121C	U	H/w flow w/sample through cell	Total = 110 = 55	B
	13:07-13:14	BagIF011		Baghouse Inlet flowing through cell P= 719.3	"	"	"	U	H/w w/sample through cell	Total = 145 = 60	
		~~Empty~~									
	1305	empty001		Evacuated cell a jumpline to manifold.		- Sm		Bagout directory.			
	1315-13:21	BagIF012		Baghouse Inlet flowing through cell P= 719.6	50/200	1.0	121C	U	H/w w/sample flow	Total = 145 =	B

Sample Flow = flow reading with Sample flowing through cell

Sample Manifold @ 300 F

FTIR cell

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/22/96	13:27-13:33	BagOF013	40 psig	Outlet flowing through cell P= 719.9	50/200	1.0	122 C	U	H/W	total = 115	B
	1335-1336	NitCon#3		Nitrogen through Cadenza. P = 718.0 in outlet ductwork							
											E
1356-1358	~~1355~~	BagOH014	40 psig	Static sample; outlet	50/200	1.0	122C	U	H/W	Total = 110 ~~105~~	C
	1402-14:08	BagIF013		Inlet flowing sample P= 718.2	50/200	1.0	122C	U	H/W	Total = 145 w/sample through cell = 75	C
	14:10-14:12	BagIH014		Inlet, static P=717.6					H/W	145	C
Flow meter = .008@ 9/bar	14:14-14:18	BagOF015		outlet flowing P= 717.9	50/200	1.0	122C	U	H/W	Total = 110 w/sam = 60	C
	14:21-14:22	BagOH016		outlet static P = 717.5	50/200	1.0	122C	U	H/W	110	C
	14:26-1430	BagIF015		Inlet; flowing sample						Total = 145 w/samp = 30	C
	1433-1434	BagIH016		Inlet; static sample P = 717.4						145	C
	1439-1441	BagOH017		outlet; static sample						110	C
	~~1444~~										
	14:98	SF6 spike on		to Inlet @ 200 lpm							
	1444-1449	BagOF018		outlet flowing P= 717.2						total = 110 w/samp.	C

APG Lime

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/22/90	1451-1452	BagIS017	40 passes	SF_6 spike @ 2.00 lpm inlet; static sample	60/200	1.0	122C	S (SF_6)	H/W	145	C
	1455-1456	BagIS018	↓	SF_6 spike @ 1.98 lpm inlet static							
	1458-1500	BagIS019	↓	" spike @							
	1504	started HCl		spike to inlet @ 2.03 lpm							
	1504-1506	BagOH020	40 passes	outlet static sample	50/200	1.0	122C	U	H/W	110	C
	1509-1511	BagOF020	↓	outlet flowing sample P = 718.7						Total = 110 w/sample = 45	C
	1516-1518	BagIS020		Inlet static HCl spike @ 2.00 lpm P = 716.8		1.0	122C	S (HCl)	H/W	145	C
	1520-1522	BagIS021		"							
1524-1527	1524-1527	BagIS022		" HCl @ 2.05 lpm							
	1530-1536	BagISF23		Inlet spike + flowing HCl @ 2.05 lpm P = 718.5	50/200	1.0	122C	S (HCl) + magic cell	H/W w/sample	Total = 145 = 110	C
	1538-1543	BagISF24	↓	" P = 718.0					w/samp	Total = 145 = 50	C
	1544-1547	BagISF25	↓	" P = 716.3 HCl @ 2.00 lpm					w/samp	total = 145 =	C
	1549-1551	BagOH021	↓	outlet; static	50/200	1.0	122C	U	H/W	110	C
	1554-1556	BagIS026		Inlet " HCl 2.05 lpm				S		145	C
	1558-1600	BagOH022		Outlet unspiked	50/200	1.0	"	U	H/W	105	C
	1603-1605	BagIS027	↓	Inlet spiked HCl spike @ 2.00 lpm	"	"	"	S	"	145	C

Sample Manifold 300 P

P = 716.4

AP6 Lime

Data Sheet: FTIR Batch Samples: Lime Kilns. EPA WA. 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/27/96	1608-1611	Bag0H023	40 passes	outlet H/W	59/200	1.0	122 C	U	H/W	110	C
	1613-1616	BagIS028	↓	Inlet HCl spike @ 1.97 lpm	"	"	"	S	"	145	C
	1619-1621	Bag0H024	↓	outlet H/W	"	"	"	U	H/W	110	C
1624-1625		BagIS029	"	Inlet HCl spike @ 1.97 lpm	"	"	"	S	"	145	C
1628-1630		Bag0H025	"	Outlet H/W	"	"	"	U	"	110	C
1633-1634		BagIS030		Inlet HCl spike @ 1.96 lpm	"	"	"	S	"	145	C
1637-1639		Bag0H026	"	outlet ; H/W	"	"	"	U	"	110	C
1642-1643		BagIS031	"	Inlet HCl spike @ 1.99 lpm	"	"	"	S	"	145	C
1646-1648		Bag0H027	"	outlet, H/W	"	"	"	U	"	110	C
1651-1653		BagIS032	"	Inlet Spiked HCl @ 1.98 lpm	"	"	"	S	"	145	C
1655-1657		Bag0H028	"	outlet H/W	"	"	"	U	"	110	C
1702-1703		BagIS033	"	Inlet spiked @ 1.98 lpm	"	"	"	S	"	145	C
1705-1707		Bag0H029	"	Outlet H/W	"	"	"	U	"	110	C
1710-1711		BagIS034	"	Inlet spike @ 1.98 lpm	"	"	"	S	"	145	C
1714-1716		Bag0H030	"	Outlet H/W	"	"	"	U	"	110	C
1718-1720		BagIS035	"	Inlet Spiked @ 2.00 1.97 lpm	"	"	"	S	"	145	C
1723-1724		Bag0H031	"	Outlet H/W	"	"	"	U	"	110	C
1727-1728		BagIS036	"	Inlet Spiked @ 2.00	"	"	"	S	"	145	C
1731-1733		Bag0H032	"	Outlet H/W	"	"	"	U	"	110	C
1737-1739		BagIS037	"	Inlet Spiked @ 2.00 lpm	"	"	"	S	"	145	C

1700 - empty 02 evacuated cell in bag out subtracting P = 716.2

1740 HCl spike off

1741 [illegible]

APG Line

manifold = 300 F

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

P = 716.7

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/27/96	1755-1757	BagIS038	40 passes	Inlet spiked w/ SF_6 @ 1.97 lpm 50/50 2.00 lpm		1.0	122C	S	H/W	145	C
1800-1801		BagIS039	"	" SF_6 @ 1.96 lpm	"	"	"	"	"	"	"
1804-1806		BagIS040	"	" SF_6 @ 1.95 lpm	"	"	"	"	"	"	"
Probes out of stack @ 1612 @ ~~outlet~~ Inlet.											
1813-1815		BagIA041		ambient sample through inlet line	"	"	"	"	"	150	"
1817-1819		BagIA042		"							
	1817	~~out~~		outlet probe out of stack							
	1823-1825	BagOA033	"	Outlet ambient air through "Probe	"	"	"	"	"	115	C
	1828-1829	BagIA043	"	Inlet " "	"	"	"	"	"	150	C
	1832-1834	BagOA034	"	Outlet " "	"	"	"	"	"	115	C
	1837-1839	BagIA044	"	Inlet " "	"	"	"	"	"	150	C
1855-1857		BagOA035	"	Outlet " "	"	"	"	"	"	115	C
1900-1901		BagIA045	"	Inlet " "	"	"	"	"	"	150	C

APG Lime

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA. 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	850			outlet Probe had been in stack about 5 minutes. Removed from stack to							
				Collect ambient air sample.							

manifold Temp = 300 F

APG Lime

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
P = 713.3	10/23/96	1028-1030	BAG0A201	40 pass	Baghouse outlet ambient air through probe	50/100	1.0	122C	U	H/W	115	B
		1034-1036	Bag0A202	"	"	"	"	"	"	"	"	B
		1037-1039	BagIA201	"	Inlet ambient air	"	"	"	"	"	"	"
		1040	Outlet Probe		in Stack							
		1045	Inlet Probe		in Stack							
P = 713.3	10/23/96	1059-1101	Bag0H203	"	outlet H/W	50/100	1.0	122C	U	H/W	105	B
	→	1105-1107	BagIH202	"	Inlet H/W	"	"	"	U	"	100	C
		1111-1112	Bag0H204	"	outlet H/W	"	"	"	U	"	105	C
		1115-1117	BagIH203	"	Inlet	"	"	"	U	H/W	95	C
		1121-1123	Bag0H205	"	Outlet	"	"	"	U	"	105	C
		1127-1130	BagIH204	"	Inlet	"	"	"	U	"	85	C
		1133-1134	Bag0H206	"	Outlet	"	"	"	U	"	100	C
		1140-1143	BagIH205	"	Inlet	"	"	"	U	"	85	C
		1145			Removing Inlet Probe to take out some of the glass wool to try and increase the flow. Flow is even less with probe back in. Will remove probe to see if wool plug is lodged in probe.							B
		1146-1147	Bag0H207	"	outlet	"	"	"	U	"	105	C
		1207-1211	BagIH206	"	Inlet - Flow was high with probe off	50/100	"	"	U	"	65	C
		1217-1218	Bag0H208	"	Outlet	"	"	"	U	"	105	C

These are dilated by an unknown factor because I opened the valve too far and pulled air into the cell (i.e., a negative flow)

Total flow is lower today because the glass wool plug came out yesterday

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	1222 1226	BagIH207	40 passes	Inlet	50/100	1.0	122C	U	H/W	55	B
	1224	low flow @ inlet. Remove probe again and change filter to make sure it is not clogged. Continue taking at outlet.									
1230	1232	Bag0H209	40 passes	Outlet	50/100	1.0	122C	U	H/W	105	B
1237	1238	Bag0H210	"	"							
1241	1244 ~~1243~~	Bag0H211	"	Outlet ~~left probe open~~ P=							
1247	1248	Bag0H212	"	Outlet P= 714.2							
1251	1252	Bag0H213	"	" P= 713.9							
1255	1259	Bag0H214	"	Outlet P= 714.6							
1301		BagIF208	"	Inlet (new filter) flowing through cell @ P= 715.0					w/samp	60 = 45	B
1307		Started SF_6	spike	to outlet @ 1.00 lpm							
1310	1316	BagIF209		Inlet flowing sample	50/100	1.0	122	U	w/samp =	total=55	
1317	1318	Bag0S215	40 passes	SF_6 spike @ 1.00 lpm	"	"	"	S(SF_6)	H/W	total = 95	B
1321	1323	Bag0S216		" "	"	"	"	"	"	95	B
1325	1327	Bag0S217	"	" "	"	"	"	"	"	95	B
1330	1336	BagIF210		Inlet flowing	"	"	"	U	H/W	55 (w/samp total)	
	1341	HCl spike		into Outlet @ 1.00 lpm						total = 95	
	1337	EMPTY201		Evacuated Cell & passing line in " Bag out line bag							
1339	1345	BagIF211		Inlet flowing	"	"	"	U	H/W	55	

P= 713.5
"

w/samp =

Pressure from Outlet if sample flows through cell is = 737 torr.

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/23	1348-1350	Bag0S 218	40 passes	Spike had cut off	unknown spike level			S	H/w	95	B
				reset spike flow @ 1350							
	1354-1359	Bag IF212	"	flowing through cell	"	"	122C	U	H/w	55	B
	1400-1402	Bag 0S219	"	Outlet HCl @ 1.00 lpm	50/100	1.0	122C	S	H/w	95	B
	1405-1407	Bag0S220	"	" " 1.00 lpm	"	1.0	122C	S(HCl)	H/w	95	B
P=713.5	1409-1411	Bag0S221	"	" 1.00 lpm	"	"	"	"	"	95	B
	1414-1420	Bag IF213	"	Inlet	"	"	"	U	"	50	B
	1422-1423	Bag 0S222	"	Outlet HCl spike @ 1.00 lpm	"	"	"	S(HCl)	"	95	B
		~~Bag IF214~~		dropped to 0.6 during spectrum.							
	1427 1434	Bag IF214	"	Inlet flowing	"	"	"	U	"	50	B
	1436-1439	Bag 0S223	"	HCl @ 1.00 lpm	"	"	"	S	"	95	B
	1442-1447	Bag IF215	"	Inlet flowing	"	1.0	122C	U	"	48	B
P=713.0	1456-1457	Bag0S224	"	Outlet HCl spike @ 1.04	"	"	"	S(HCl)	"	95	B.
	1501-1507	Bag IF216	"	Inlet flowing	"	"	"	U	"	45	B
(6)	1511-1512	Bag0S225	"	Outlet HCl spike @ 1.03 lpm	"	"	"	S(HCl)	"	95	B
	1514-1515	Bag0S226	"	" " " 1.03 lpm	"	"	"	"	"	"	B
P=712.4	1518-1519	Bag 0S227	"	" " " " 1.00 lpm	"	"	"	"	"	95	B
	1522 1523	Bag0S228	"	" " " " 1.00	"	"	"	"	"	95	B
	1526 1527	Bag0S229	"	" " " " 1.00 lpm	"	"	"	"	"	95	B

with sample flowing through cell from outlet - cell pressure is 732 torr

AP6 Line

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes 1.00 lpm	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	1529-1531	Bag0S230	40 passes	Baghouse Outlet HCl spike	50/100	1.0	122C	S(HCl)	H/w	95	B
1533-1534		Bag0S231	"	" HCl spike @ 1.00 lpm	"	"	"	"	"	95	B
P= 712.8											
	1538	HCl spike off		SF6 spike on @ 1.00 lpm.							
1539-1549		Bag1F217	"	Inlet flowing	50/100	"	"	U	H/w	95	B
P= 712.3	1553-1554	Bag0S232		SF6 @ 0.98 lpm	50/100	"	"	S(SF6)	H/w	95	B
	1534	Inlet Probe out of Stack									
1556-1558		Bag0S233	"	outlet SF6 @ 0.99 lpm	—	—	—	—	—	95	B
1600-1602		Bag0S234	"	" SF6 @ 0.98 lpm	"	"	"	"	"	95	B
	1603	SF6 Spike off									
1605-1610		Bag1A218		ambient air through inlet probe	50/100	"	"	U	H/w	55	B
1612-1613		Bag0H235		H/w outlet	"	"	"	U	"	95	B
	1617	outlet Probe out of Stack									

APG Lime

Data Sheet; FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01,

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/23/96	1637	Bag0A236	49 pass	Ambient through outlet Probe	50/100	1.0	122C	U	H/W	95	C
	1641-1646	Bag~~I~~A219	"	Ambient through outlet Probe	"	"	"	U	"	55	C
P: 712.6	1700-1701	Bag0A237	"	Ambient outlet mixed w/ 3 lpm N2	"	"	"	S(N2)	"	100	
		~~Bag IA~~									
		refilled	MCT Dewar								
HCl nearly non-detected.	1708-1712	Bag IA220		ambient through inlet Probe	"	"	"	U	"	50	C
same results IR uncorrected	1714-1715	Bag0A238	"	ambient through outlet	"	"	"	U	"	95	C
	1720	started	N2	purge through both Sample Lines						3.0 lpm	

new sample line for outlet + dary.

APG Line

Manifold @ 300 F

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/24/96	1108-1110	Bag OH 302	20 passes	Baghouse Outlet	50/100	1.0	121C	U	H/W	95	E
	1113-1114	Bag I H 302	"	Inlet	→	→	→	→	→	125	E
	1117-1119	Bag OH 303	"	Outlet	→	→	→	→	→	95	E
	1121-1122	Bag I H 303	"	Inlet	→	→	→	→	→	125	E
	1125-1126	Bag O H 304	"	Outlet	→	→	→	→	→	95	E
P=718.3	1129-1130	Bag I H 304	"	Inlet	→	→	→	→	→	125	E
	1133-1134	Bag OH 305	"	Outlet	→	→	→	→	→	95	E
	1137-1139	Bag I H 305	"	Inlet	→	→	→	→	→	125	E
P=718.2	1143-1144	Bag OH 306	"	Outlet	→	→	→	→	→	95	E
	1146-1147	Bag I H 306	"	Inlet	→	→	→	→	→	125	E
	1154	new Background w/ N2									F
	1158-1200	Bag OH 307	"	Outlet	50/100	1.0	121	U	H/W	95	F
	1202-1205	Bag I H 307	"	Inlet	"	"	"	U	"	115	F
	1208-1210	Bag O H 308	"	Outlet	"	"	"	"	"	95	F
in "Bag 0"	→	EMPTY 301		Evacuated cell & jumper line	"	"	"	—	—	—	F
	1214-1216	Bag I H 308	"	Inlet	"	"	"	U	"	110	F
	1221-1222	Bag OH 309	"	Baghouse Outlet	"	"	"	U	"	95	F
	1225-1228	Bag I H 309	"	Inlet	"	"	"	"	"	95	F
10/24/96	1232-1234	Bag OH 310	"	Outlet	"	"	"	"	"	95	F

AP6 Lime

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
10/24/96	1233-1237	BagIH 310	20 passes	Bag house Inlet	50/100	1.0	122C	U	H/W	88	F
	1244-1246	BagOH 311	"	" Outlet	"	"	"	U	"	95	F
	1248-1252	BagIH 311	"	" Inlet	"	"	"	"	"	80	F
Pump pad out 1259-1301 P=717.6	1255-1257	BagOH 312	"	Outlet	"	"	"	"	"	95	F
	1304-1307	BagIH 312	"	Inlet	"	"	"	"	"	78	F
1309	1311	BagOH 313	"	Outlet	"	"	"	"	"	95	F
1313	1316	BagIH 313	"	Inlet	"	"	"	"	"	68	F
		~~Bag~~									
in "Pump" 1318	1320	CONN2 301		Nitrogen through Condenser	"	"	"	U	Cond	P=718.1	F
	1323	Started outlet through the Condenser									
Valve was open from sample. Started P.	1324	1326 BagOH 314		Outlet ; H/W	50/100	"	"	U	H/W	Total 95	F
1329	1331	BagOC 315	"	Outlet Condenser Sample.	"	"	"	"	Cond.	95	F
1333	1335	BagOC 316	"	" " "	"	"	"	"	"	"	"
	1336	Started	Nitrogen through Condenser								
1338	1342	BagIH 314	"	Inlet H/wet	"	"	"	"	H/W	58	F
1345	1347	CONN2 302		Nitrogen through Condenser	"	"	" P=	717.4	"		F
	1348	Started	Inlet sample through Condenser.								
1350	1353	BagIC 315		Inlet sample through Condenser	"	"	"	"	" total	50	F
Total flow was 48 liters	1355	1358 BagIC 316		" " "	"	"	"	"	"	43	F

Data Sheet: FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01.

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
P=717.1	10/24/96	1401-1409	Bag0H317	20 passes	Outlet	50/100	1.0	122C	U	H/W	95	F
		1410	change filter		on inlet @ back of probe							
					We did not change filter, but removed a clog. Flow now improved at inlet.							
1415 flow meter reading @ 1.0 lpm												
		1416	Started	SF_6	Spike to outlet @ 0.49 lpm.							
	1318 - 1320		Bag I H317	20 passes	Inlet H/W	50/100	1.0	122C	U	H/W	145	F
	1322 - 1324		Bag O S318	↓	spiked w/ SF_6 @ 0.48 lpm	"	"	"	S	"	94	F
	1327 - 1328		Bag O S319	↓	" " 0.47 "	"	"	"	S	"	94	F
P=717.3	1330 - 1332		Bag I H318	↓	Inlet H/W	"	"	"	"	"	140	F
	1435 - 1437		Bag O S320	↓	Outlet H/W spiked w/ SF_6 @ 0.49 lpm	"	"	"	"	"	94	F
		1441	Started	HCl	Spike @ 0.52 lpm							
P=717.1		1448	1449 Bag I H319	20 passes	Inlet H/W	50/100	1.0	122C	U	H/W	145	F
did not start spectrum 1457		1453 - 1454	Bag O S321	"	Spiked HCl @ 0.52 lpm	"	"	"	S	"	94	F
		1500 - 1502	Bag I H320	"	Inlet	"	"	"	U	"	140	F
P=724.2		1505 - 1507	Bag O S322	"	Outlet Spike HCl @ 0.52 lpm	"	"	"	S	"	94	F
		1510 - 1511	Bag O S323	"	~~Inlet~~ " " @ 0.51 lpm	"	"	"	S	"	94	F

APG Lime

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01.

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
P=717.5	10/24/96	1513-1514	Bag IH 321	20 passes	Inlet	50/100	1.0	122C	U	11/W	140	F
		1518-1519	Bag OS 324	"	Outlet, HCl spike @ 0.50 lpm	"	"	"	S	"	93	F
	1524	-1525	Bag IH 322	"	Inlet	"	"	"	U	"	140	F
	1528	-1529	Bag OS 325	"	Outlet HCl spike @ 0.49 lpm	"	"	"	S	"	93	F
P=717.5	1533	-1534	Bag IH 323	"	Inlet	"	"	"	U	"	140	F
sample inlet P=729	1537	-1538	Bag OS 326	"	Outlet HCl spike @ 0.48 lpm	"	"	"	S	"	93	F
	1541	-1542	Bag IH 324	"	Inlet	"	"	"	U	"	140	F
	1544	-1546	Bag OS 327	"	Outlet HCl spike @ 0.48 lpm	"	"	"	S	"	93	F
P=717.2	1550	-1551	Bag IH 325	"	Inlet	"	"	"	U	"	140	F
	1553	-1555	Bag OS 328	"	Outlet HCl spike @ 0.48 lpm	"	"	"	S	"	92	F
	1558	-1600	Bag OS 329	"	Outlet " " " 0.49 lpm	"	"	"	S	"	92	F
	1602	-1604	Bag OS 330	"	" " " " " 0.49 lpm	"	"	"	S	"	92	F
	1606	-1607	Bag IH 326	"	Inlet	"	"	"	U	"	140	F
	1609	-1611	Bag OS 331	"	Outlet HCl @ 0.51 lpm	"	"	"	S	"	92	F
	1614	-1615	Bag OS 332	"	Outlet " " 0.52 lpm	"	"	"	S	"	92	F
	1617	-1619	Bag OS 333	"	" " " 0.52 lpm	"	"	"	S	"	92	F
		1621	HCl spike off		SF6 spike on @ 0.49 lpm							
	1623	-1624	Bag IH 327	"	Inlet	"	"	"	U	"	140	F
P=717.1	1628	1629	Bag OS 334	"	SF6 spike @ 0.47 lpm	outlet "	"	"	S	"	92	F
	1632	-1634	Bag IH 328	"	Inlet	"	"	"	U	"	140	F

APG Lime

Data Sheet: FTIR Batch Samples: Lime Kilns, EPA WA, 3804-01.

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
P=716.9	10/24/96	1636-1637	Bag OS335	20 passes	Outlet spiked w/ SF_6 @ 0.49 lpm	50/10	1.0	1220	S	H/W	92	F
		1640-1641	Bag IH329	"	Inlet	"	"	"	U	"	140	F
		1644-1645	Bag OS336	"	Outlet SF_6 spike @ 0.48 lpm	"	"	"	S	"	92	F
		1647	SF_6 Spike off									
		1650-1652	Bag IH330	20 passes	Inlet H/W	"	"	"	U	"	140	F
		1706-1708	Bag OH337	20 passes	outlet unspiked	"	"	"	U	"	92	F
Pumped out + refilled		1715-1716 ~~1712-1713~~	Bag IH331	"	Inlet " P=717.2	"	"	"	U	"	140	F
		1719-1720	Bag OH338	"	Outlet "	"	"	"	"	"	92	F
P=717.3		1722-1724	Bag IH332	"	Inlet	"	"	"	"	"	140	F
		1726-1727	Bag OH339	"	Outlet	"	"	"	"	"	92	F
started inlet to Cond.		1731-1733	Bag IH333	"	Inlet	"	"	"	"	"	140	F
		1734-1736	Bag OH340	"	Outlet	"	"	"	"	"	92	F
		1740-1741	Bag IC334	"	Inlet	"	"	"	"	Condenser	140	F
		1744-1745	Bag OH341	"	Outlet	"	"	"	"	H/W	92	F
		1748-1749	Bag IC335	"	Inlet / Condenser	"	"	"	"	Cond	140	F
		1751	started	Nitrogen to Condenser								
		1752-1754	Bag OH342	"	Outlet	"	"	"	"	H/W	92	F
re "Bag in"		1756-1757	Con N2 503	"	nitrogen through Condenser	"	"	"	"	"	—	F

APG Lime

Data Sheet; FTIR Batch Samples; Lime Kilns, EPA WA. 3804-01.

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
	10/24/96	1801-1802	BagIH336	20	Inlet to Baghouse	50/100	1.0	122C	U	H/W	140	F
		1801	outlet	sample	to Condenser							
	1812	1817	BagOC343		Outlet through Condenser	"	1.0	122C	U	Cond	90	F
	1817	1818	BagIH337	"	Inlet	"	"	"	"	N/W	140	F
	1821	1822	BagOC344	"	Outlet through Condenser	"	"	"	"	Cond	90	F
		1823	Nitrogen		to Condenser.							
P=717.7	1826	1827	BagOH345	"	Outlet	"	"	"	"	N/W	90	F
	1830	1831	BagIH338	"	Inlet	"	"	"	"	"	140	F
	1834	1836	BagOH346	"	Outlet	"	"	"	"	"	90	F
on "Bag in"	1838	1839	CONN2304	"	Nitrogen through Condenser	"	"	"	"	"	–	F
	1842	1843	BagOH347	"	Outlet	"	"	"	"	"	90	F
	1846	1847	BagIH339	"	Inlet	"	"	"	"	"	140	F
	1850	1852	BagOH348	"	Outlet	"	"	"	"	"	90	F
P=718.2	1855	1856	BagIH340	"	Inlet	"	"	"	"	"	140	F
	1859	1901	BagOH349	"	Outlet	"	"	"	"	"	90	F
	1903	1904	BagIH341	"	Inlet	"	"	"	"	"	140	F
	1907	1908	BagOH350	"	Outlet	"	"	"	"	"	90	F
	1912	1914	BagIH342	"	Inlet	"	"	"	"	"	140	F
P=718.5	1915	1917	BagOH351	"	Outlet	"	"	"	"	"	90	F
	1920	1921	BagIH343	"	Inlet	"	"	"	"	"	140	F

APG Lime

Data Sheet; FTIR Batch Samples; Lime Kilns, EPA WA, 3804-01,

	Date	Sample Time	File Name	Path M	Location/ Notes	# scans	Res. cm^{-1}	Temp. °F	Spk/ Unsp	Sample Cond.	Sample Flow	BKG
	10/24/96	1924-1925	BagOH352	20 passes	Baghouse Outlet	50/100	1.0	122F	U	H/W	90	F
		1928-1929	BagIH344	"	Inlet	"	"	"	"	"	140	F
		1932-1934	BagOH353	"	Outlet	"	"	"	"	"	90	F
		1936-1937	BagIH345	"	Inlet	"	"	"	"	"	140	F
		1939-1941	BagOH354	"	outlet	"	"	"	"	"	90	F
		1943-1944	BagIH346	"	Inlet	"	"	"	"	"	139	F
		1946-1948	BagOH355	"	Outlet	"	"	"	"	"	90	F
			~~BagIH347~~	"	~~Inlet sample~~							
		1950-1952	BagOH356	"	Outlet sample	"	"	"	"	"	90	F
					last sample - Manual run is completed.							
		1952	Inlet Probe out of Stack									
		1957	outlet Probe out of Stack									
		2003-2004	BagIA347	"	Inlet ambient sample	"	"	"	"	"	140	G
		2007-2008	BagOA357	"	Outlet ambient air through probe	"	"	"	"	"	90	G
P=718.7		2012-2013	BagIA348	"	Inlet - ambient air through Probe	"	"	"	"	"	140	G
P=718.5		2016-2018	BagOA358	"	Outlet, ambient air through Probe	"	"	"	"	"	90	G
"		2034-2036	BagOA359	"	" " " "	"	"	"	"	"	90	G
		2039-2041	BagOA360	"	" " " "	"	"	"	"	"	90	G
P=718.6		2042-2043	BagIA349	"	Inlet ambient air through line	"	"	"	"	"	140	G

APPENDIX C.

FTIR ANALYTICAL RESULTS

Draft Report

December 1996

Results of Least Squares Concentration Determinations for
FTIR Spectra Collected at APG Lime Kiln

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Disclaimer

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Data Collection and Analytical Method

Midwest Research Institute performed extractive FTIR source testing in October 1996 at APG lime kiln and provided the spectral data to Rho Squared for preliminary quantitative least squares analysis. Compounds of quantitative interest in the samples, referred to below as analytes and identified in conversations with Dr. Tom Geyer of MRI, are HCl, H_2CO, CO, SO_2, NO, and NO_2. The spectra also contain features from the interferant compound H_2O, and SF_6 was quantified in some spectra as the diluent tracer compound used for dynamic spiking.

References 1 through 5 comprise a thorough description of one technique for analyzing FTIR absorbance spectra. Using the programming language ARRAY BASIC™ (GRAMS,™ Version 3.02, Galactic Industries Corporation, Salem, New Hampshire) Rho Squared has prepared a computer program to perform this technique. The "classical least squares" (CLS) or "K-Matrix" technique and the associated computer program are described in Reference 6. The terminology and basic analytical approach employed in this work are described in the "EPA FTIR Protocol" (Reference 7).

The program allows the analyst to select a number of analytical regions and to specify which of the selected reference spectra will be employed in determining the corresponding compound concentrations. Baseline parameters (linear, and quadratic in some cases) were also determined in the calculations but are not reported here. Reference spectra for the current work were provided by MRI or were taken from the EPA FTIR spectral library of Hazardous Air Pollutants (hereafter, the "EPA library"). Additional information regarding the reference spectra is listed below.

The program calculates the standard 1σ uncertainty in each concentration. However, all uncertainties quoted below are equal to four times the calculated 1σ values. The program also calculates the residual spectra (the difference between the observed and least squares fit absorbance values) for each sample spectrum and analytical region. These data are not presented here but have been submitted to MRI in digital form with this report. The GRAMS™ format residual spectral files have DOS extensions of the form "r*n*", where the integer *n* designates the analytical region label for a particular analytical run. Although this labeling scheme does not uniquely identify the residual spectra, the frequency ranges are unique and make identification of the various spectra straightforward.

For each analytical region, compounds whose reference spectra are employed in the least squares fits are characterized either as analytes or as interferants. Table 1 lists the analytical regions and summarizes the characterizations of the six target compounds (HCl, H_2CO, CO, SO_2, NO, and NO_2). Note that each target compound appears as an analyte in one and only one analytical region. The concentrations and uncertainties reported in this work correspond to the analyte characterizations of Table 1.

TABLE 1. ANALYTICAL REGIONS AND COMPOUND CHARACTERIZATIONS[a,b]

Analytical Region	Lower Bound (cm^{-1})	Upper Bound (cm^{-1})	HCl	H_2CO	CO	SO_2	NO	NO_2	H_2O	CO_2	SF_6
0	900	1200	-	-	-	A	-	-	I	I	A
1	1581.7	1613.3						A	I	-	-
2	1898.6	1904.8	-	-	-	-	A	-	I	-	-
3	2110	2125.5	-	-	A	-	-	-	-	I	-
4	2747	2848	A	A	-	-	-	-	-	-	-

[a]I indicates "interferant," A indicates "analyte," and the hyphen indicates that the compound was not included in the least squares spectral analyses of the analytical region.
[b]Baseline slope and offset for each analytical region were also determined in the least squares concentration analyses (see

MRI provided a total of 279 spectral files for analysis. After determining concentration values and uncertainties for each compound in each analytical region of every sample spectrum, the program rejects compounds from each analytical region if either a) the determined concentration is negative or b) the 4σ uncertainty in the concentration is greater than the (positive) determined concentration. If a compound is rejected from a region for a particular spectrum, the concentration is recorded as exactly zero in the output file along with the related uncertainty from the original fit. Such uncertainty values are extremely conservative upper limits on the uncertainty of the reported zero concentration values. Concentration results and their 4σ uncertainties were recorded in Excel™ spreadsheet files and provided to MRI for inclusion in a comprehensive report to EPA.

Pathlength Determinations

Absorption pathlengths were determined from 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 7 (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 7, 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 2. The CH values were used in analytical region 4; the FP values were used in all other analytical regions.

TABLE 2. PATHLENGTH DETERMINATION RESULTS FOR APG TEST DATA

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
20	293	11.0	2.6	11.3	1.6
20	393	10.2	2.5	14.3	2.2
40	293	19.2	5.5	20.0	1.8
40	393	20.2	2.6	23.4	1.6

Reference Spectra

Reference spectra for the current work were provided by MRI or were taken from the EPA library. Table 3 lists the spectra used in the analyses for each analytical region.

TABLE 3. REFERENCE SPECTRA

Compound	Analytical region				
	0	1	2	3	4
HCl	-			-	097.alf
H_2CO	-				087c1asb.spc [b]
CO				co20829a.spc	
SO_2	198.alf				
NO			199c1bsa.spc	-	
NO_2	-	200c1bse.spc	-	-	-
H_2O	194jsub.spc	194fsub.spc	194fsub.spc		
CO_2	193c1bsa.spc			193c1bsa.spc	
SF_6	a				-

[a]File sf620p_2.alf was used for spectra recorded at (nominal) twenty passes in the infrared absorption cell; file sf640p_1.alf was used for spectra recorded at (nominal) forty passes in the infrared absorption cell.
[b]Results of analyses excluding H_2CO from this analytical region were also supplied to MRI.

For the compound HCl, the FTIR library spectra were de-resolved to 1 cm^{-1} and normalized for absolute temperature, concentration, and absorption pathlength. The resulting files were averaged to provide a "reduced absorptivity" (see Reference 6), 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 4. Similar procedures were followed to

determine the reduced absorptivity and FCU values for the compounds SO_2 and SF_6. For SO_2, 1.0 cm-1 resolution spectra provided by MRI were used; the spectra used for SF_6 were those recorded on the field instrument, at two different absorption pathlengths, during the APG field test.

TABLE 4. FRACTIONAL CALIBRATION UNCERTAINTY (FCU)

Compound	FCU (%)
SO_2	4.6
HCl	8.5
SF_6 (20 passes)	1.5
SF_6 (40 passes)	1.2

References

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4. W.C. Hamilton, Statistics in Physical Science, Ronald Press Co., New York, 1964, Chapter 4.

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

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APPENDIX D.

PROCESS DESCRIPTION AND DATA

RESEARCH TRIANGLE INSTITUTE

Center for Environmental Analysis

MEMORANDUM

TO: Joseph Wood, ESD/MICG (MD-13)
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

FROM: Cybele Brockmann, RTI

DATE: July 31, 1997

SUBJECT: Process Description for APG Lime

REFERENCE: Information Gathering and Analysis for the Lime Manufacturing Industry NESHAP
EPA Contract 68-D1-0118
ESD Project 95/06
RTI Project 6750-017

Attached is the description of processes at APG Lime; processes were monitored during testing at the plant October 21-24, 1997.

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I. Process Description of the APG Plant

Lime (calcium oxide, CaO) is typically produced in the U.S. by crushing and then heating limestone ($CaCO_3$) in an inclined, rotating kiln. The limestone is heated to temperatures of around 2000 degrees Fahrenheit (deg F) which cause it to breakdown chemically into lime and CO_2. Some of the lime produced in the U.S. is hydrated ($Ca(OH)_2$). At APG, lime is sold as CaO and $Ca(OH)_2$.[1]

Limestone at the APG plant is extracted from an underground mine located at the plant. The limestone is milled and screened to yield three sizes of stone: less than 3/8 inch, 3/8 inch to $1^{1/8}$ inches, and $1^{1/8}$ inches to 2 inches.[2]

During testing, emissions were measured at the inlet and outlet of the fabric filter (FF) that cleans the exhaust from the number one kiln and at the stack associated with the cooler of the number two kiln. Process data from the number one kiln were collected during testing of its FF. Process data from the number two kiln were collected during testing of its cooler stack.

The number one and two kilns are inclined rotating kilns with design capacities of 300 tons of lime per day and 265 tons of lime per day, respectively.[3] Both kilns are approximately 300 feet long with tapered diameters (10 feet in diameter at the front end of each kiln and 8 feet in diameter the remaining length of the kilns).[4] The incline of the kilns is ½ inch per foot.[5] Limestone enters at the back end of each kiln (the highest point of incline) and tumbles through the kiln via gravity and the rotating motion of the kilns (typical rotating rates for both kilns are 0.25 to 1.2 revolution per minute).[6] The residence time of the feed material in the kiln is 2.5 hours.[7] Approximately two tons of limestone are consumed to produce one ton of lime.[8]

The combustion of fuel, which consists of pulverized coal suspended in air, occurs at the front end of each kiln (coal samples were obtained during testing). Coal for both kilns is pulverized to the consistency of powder in a single ball mill. Heated air from the cooling process (described below) is pulled into the ball mill to preheat and dry the coal. A fan on the mill blows the air and dry pulverized coal from the mill into each kiln. Typically a third of a ton of coal is consumed per ton of lime.[9]

Lime exiting each kiln is deposited onto the kiln's moving grate cooler. Ambient air is blown upward through the grates to cool the lime. Most of the air that cools the lime is routed to the kilns or to the ball mill to preheat and dry the coal. A

small portion of the air exiting each cooler is released to the atmosphere through its own stack via natural draft. This was the gas stream from the number two cooler that was tested. Lime from each cooler is conveyed to a screener, separated by particle size, and stored in silos.

II. Kiln Emissions Control

Exhaust gases exiting the number one kiln pass through a FF. The FF, manufactured by Amerex, was installed in 1994.[10] The FF has six compartments. The bags within the compartments are made of 22 ounce fiberglass with a teflon finish.[11] The air-to-cloth ratio is 3.4 actual cubic feet per minute per square foot of fabric.[12] The inlet gas temperature for the FF is 495 degrees F, and the pressure drop across the FF is 6 to 8 inches of water.[13] The FF runs continuously; during cleaning, one of the six compartments is taken off-line and cleaned by pulse-jet, while the other five compartments continue to treat kiln exhaust.[14]

Refer to Figure 1 for a diagram of the number two kiln and cooler (the cooler stack is uncontrolled). Refer to Figure 2 for a diagram of the number one kiln, and the FF that cleans the exhaust from the number one kiln. The diagrams indicate the relative locations for each unit operation, direction of flow for material and gas, input and output of materials and gas, and approximate locations where process parameters and gas samples were measured.

III. Process Operation

Data indicating the operation of the number one and two kilns and the FF on the number one kiln are presented in this section. Data for the number two kiln were collected to provide an indication of the operation of the number two cooler since no other cooler operating parameters were monitored by the plant. Process data for the kilns and the FF were manually recorded every 15 minutes during the testing from instrument panel screens in the kiln control room; the recorded data were measured with instruments already in place and used by the plant for process control of the kiln.

Table 1 is a statistical summary of the process data collected during testing. Tables 2a, 2b, and 2c display all of process data collected during testing.

Notes Pertaining to Test Runs

Limestone feed rate

A constant size of calcitic limestone (3/8 inch to $1^{1/8}$ inch) was burned in the number one and number two kilns during testing. The limestone feed rate into the kilns is not directly measured by the plant. During testing, the plant provided an approximate feed rate of limestone into each kiln by weighing a 6 foot section of limestone on the feed belt to each kiln (see "b" on Figures 1 and 2 for location of feed belts). According to the plant, the speed of both feed belts is held constant at 199 feet per minute; however, the quantity of feed on the belts varies with the amount of limestone brought up by the bucket elevators (see Figures 1 and 2 for location of bucket elevators). The amount of limestone in the elevators is a function of the speed of the belt beneath the crushed limestone bin; the speed of this belt is indirectly measured by the %Feed-O-Weight (FOW).[15] The higher the %FOW, the faster the speed of the belt beneath the bin, which in turn leads to more limestone deposited into the bucket elevators, and, more feed deposited onto the feed belt which conveys limestone to the kiln.

The limestone bin conveyor belt speed is varied with the rotating speed of the kiln (indicated by the motor speed 1, 2, 3, or 4). When the rotation of the kiln is decreased, the limestone bin conveyor belt speed is reduced to reduce the amount of feed going into the kiln. When the rotation of the kiln is increased, the limestone bin conveyor belt speed is increased to increase the amount of feed going into the kiln.

Plant personnel weighed a six foot section of limestone on the feed belt for the number two kiln during testing on 10/21/96. The six foot section was weighed while the limestone bin conveyor belt speed was running at 64% (FOW) (the most frequent belt speed for that day - see Table 2a). At 64% FOW, the weight of the limestone on a 6 foot section of the feed belt was 22 lb. This corresponds to a limestone feed rate of 525 tons per day(TPD) and a lime production rate of 262 TPD. This compares with the typical production rate of the number two kiln reported by the plant to be 265 TPD.[16]

Plant personnel weighed two different times a six foot section of limestone from the feed belt of the number one kiln during testing on 10/22/96. One six foot section was weighed while the limestone bin conveyor belt speed was running at 50% FOW (the most frequent speed for that day - see Table 2b); the other section was weighed while the limestone bin conveyor belt

speed was running at 26% FOW. At 50% FOW, the weight of the limestone on a 6 foot section of the feed belt was 20 lb limestone (corresponds to a production rate of 238 TPD of lime). At 26% FOW, the weight of the limestone on a 6 foot section of the feed belt was 14 lb limestone (167 TPD of lime). This compares with the typical production rate of the number one kiln reported by the plant to be 240 TPD of lime.[17]

Plant personnel did not weigh a six foot section of limestone from the feed belt during testing of the number one kiln on 10/23 and 10/24; on these days, the average limestone bin conveyor belt speed was 45% FOW and 44% FOW, respectively. To approximate the limestone feed rates at these settings, the three known limestone bin conveyor belt speeds and their corresponding weights from above (i.e., 64% and 22 lb; 50% and 20 lb; 26% and 14 lb) were fitted to a curve (see Figure 3). The equation for the curve (shown in Figure 3) was used to predict the weight of a six foot section of limestone on the feed belt at the two limestone bin conveyor belt speeds. At 44% FOW, the predicted weight was 18.7 lb (approximately 224 tons of lime per day). At 45%, the predicted weight was 18.9 lb (approximately 226 tons of lime per day).

In summary, the average indirectly measured lime production rates during testing were typical of the production rates reported in the questionnaire.[18]

Coal Feed Rate

During each test run, three samples of pulverized coal were collected upstream of the kiln. The samples were collected at the beginning, middle, and end of each test run. The three samples from each test run were mixed together and a sample of the mixture sent off for an F-factor analysis. The F-factor analysis uses the thermal value of coal, along with air flow measurements, to calculate coal feed rate.

Percent Damper Opening

As indicated in Figures 1 and 2, a single ball mill pulverizes coal for the number one and number two kilns. Heated air from each kiln's lime cooler is pulled into the ball mill to preheat and dry the coal. A fan on the mill blows the air and dry pulverized coal from the mill to each kiln. Dampers control the distribution of air and coal to each kiln. Plant personnel adjust the dampers based on a visual inspection of the FF dust from each kiln. The color of the FF dust serves as a surrogate measure of combustion efficiency. FF dust that is too dark

indicates that coal is passing through the kiln unburned; consequently, the damper setting is increased to increase the flow of air to the kiln to improve combustion efficiency. The plant also has oxygen analyzers at the exhaust end of each kiln to indicate combustion efficiency. Oxygen readings for the number one kiln were not recorded during testing because the kiln operators indicated that the analyzer was not working. It was later learned (several months after testing) that the analyzer was working during testing.

During testing, the percent damper opening to the number one kiln was recorded as a possible indicator of coal feed rate consistency. The damper to the number one kiln is fully open at 74.6%; as shown in Table 1, average settings during kiln 1 testing were 59.6%, 50.9%, and 66.6%.[19]

Kiln Speed

The speed, i.e., the revolutions per minute (rpm) of the number one and number two kilns is controlled by four motor settings. The fourth motor setting is the fastest kiln speed (1.90 rpm), followed by the third motor setting (1.38 rpm), second motor setting (0.95 rpm), and first motor setting (0.69 rpm). The number one and two kilns typically operate in the fourth motor setting, however, if the front end temperature of either kiln drops, the speed of that kiln is slowed down (usually to second or third motor setting) to raise the front end temperature back up. According to the kiln operator, slowing the kiln speed raises the temperature of the feed traveling through the kiln, which in turn transports more heat to the front end of the kiln.[20]

Secondary air and back end temperatures

According to one of the kiln operators, target back end and secondary air temperatures for the number one kiln are 750 deg F and 300 deg F, respectively, when burning medium stone (the size of stone burned during testing).[21] Target back end and secondary air temperatures for the number two kiln are 680 deg F and 550 deg F, respectively, when burning medium stone.[22] Table 1 shows the average values of back end and secondary air temperatures for the number one and two kilns during testing; these temperatures were close to the target temperatures specified by the kiln operator.

FF temperature and pressure drop

The 1995 questionnaire reports that the inlet temperature and pressure drop of the FF that treats the number one kiln are 495 deg F and 6 to 8 inches of water, respectively.[23] As shown in Table 1, the average inlet temperature and pressure drop of the FF were within these reported ranges during testing.

Table 1. Statistical Summary of Process Data Collected at APG

Runs 1,2 & 3, of #2 Cooler Tests
10/21/96; data recorded from 11:32 am to 6:28 pm

Parameters for Kiln #2	mean	std. dev.	min.	max.	n[1]
Kiln back end temperature (deg F)	672	24	632	750	29
% FOW (% of motor capacity of feed belt to bucket elevators)	59	11	32	65	27
Temperature of secondary air to kiln (deg F)	536	45	395	596	29
Gear Setting on kiln speed (1 through 4; 4 is the highest speed)	4	1	2	4	29

Run 1 of Kiln 1 Baghouse Test
10/22/96; data recorded from 11:35 am to 5:47 pm

Parameters for Kiln #1	mean	std. dev.	min.	max.	n
Pressure drop across baghouse (in. of H_2O)	8.3	0.2	7.9	8.6	23
Temperature of gas at inlet to baghouse (deg F)	492	1	490	494	23
Kiln back end temperature (deg F)	751	20	720	791	23
Temperature of secondary air to kiln (deg F)	290	60	30	342	23
Gear Setting on kiln speed (1 through 4; 4 is the highest speed)	4	1	2	4	23
% FOW (% of motor capacity of feed belt to bucket elevators)	45	12	20	50	23
Damper opening for air/coal feeding kiln (%)	59.6	1.9	58.3	62.7	23

Run 2 of Kiln 1 Baghouse Test
10/23/96; data recorded from 10:41 am to 3:39 pm

Parameters for Kiln #1	mean	std. dev.	min.	max.	n
Pressure drop across baghouse (in. of H_2O)	8.0	0.2	7.8	8.3	18
Temperature of gas at inlet to baghouse (deg F)	492	1	491	494	18
Kiln back end temperature (deg F)	751	10	737	768	18
Temperature of secondary air to kiln (deg F)	293	16	262	321	18
Gear Setting on kiln speed (1 through 4; 4 is the highest speed)	4	0	3	4	18
% FOW (% of motor capacity of feed belt to bucket elevators)	45	6	22	48	18
Damper opening for air/coal feeding kiln (%)	50.9	1.0	50.3	54.8	18

Run 3 of Kiln 1 Baghouse Test
10/24/96; data recorded from 11:05 am to 7:52 pm

Parameters for Kiln #1	mean	std. dev.	min.	max.	n
Pressure drop across baghouse (in. of H_2O)	7.9	0.3	7.5	8.3	27
Temperature of gas at inlet to baghouse (deg F)	491	1	489	494	27
Kiln back end temperature (deg F)	768	13	737	791	27
Temperature of secondary air to kiln (deg F)	289	22	255	327	27
Gear Setting on kiln speed (1 through 4; 4 is the highest speed)	4	0	2	4	27
% FOW (% of motor capacity of feed belt to bucket elevators)	44	9	21	49	27
Damper opening for air/coal feeding kiln (%)	66.6	4.6	60.2	74.4	27

[1]n = number of recordings

Table 2a. Process Data

10/21/96; Runs 1, 2, & 3 of Kiln 2 Cooler Tests
Day Kiln Operator = Randy

Time	BET (deg F)	FOW (%)	Sec T (deg F)	Gear Setting
started testing approximately 11:25 am				
11:32 AM	681	65	596	4
11:47 AM	670	65	577	4
12:02 PM	665	65	550	4
after 12:02 PM recording, operator changed FOW to 41% and gear setting to 3				
12:17 PM	678	41	488	3
12:32 PM	683	62	515	4
12:47 PM	654	62	526	4
12:48 PM stopped feed b/c of a hole in the belt (FOW set to 0%); coal was still burning				
12:51 PM FOW turned back on to 62%.				
12:58 PM	731	32	505	2
Feed shut off (FOW set to 0%) to weigh feed samples; process still running				
1:03 PM	750		395	3
1:18 PM	644		474	4
1:32 run 1 completed				
1:40 PM	632	64	570	4
2:09 PM	649	64	573	4
2:25 PM	660	41	553	3
2:40 PM	658	64	500	4
3:00 PM	652	64	570	4
3:15 PM	646	64	565	4
3:30 PM	654	64	586	4
3:45 PM	649	64	585	4
4:00 PM	679	64	582	4
4:25 PM	689	64	554	4
4:36 PM	682	41	541	3
4:51 PM	677	41	491	3
4:52 PM	677	64	489	4
5:07 PM	672	64	543	4
5:22 PM	681	64	553	4
5:37 PM	680	64	540	4
5:48 PM	683	41	581	3
6:03 PM	689	64	476	4
6:18 PM	669	64	529	4
6:28 PM	662	64	541	4

BET (deg F) = back end temperature of kiln
FOW (%) = % of motor capacity of feed belt
Sec T (deg F) = temperature of secondary air to kiln

Table 2b. Process Data

10/22/96; Run 1 of Baghouse Tests
Day Kiln Operator = Tommy
Stone size = 3/8" by 1 and 1/8"

Time	BH delta P	BH T (deg F)	BET (deg F)	Sec T (deg F)	Gear Setting	FOW (%)	Damper (%)
11:30 AM	8.4	494	734	294			62.6
FOW stopped around 11:30 AM to weigh feed sample							
11:35 AM	8.4	493	738	296	4	50	62.7
11:50 AM	8.4	493	738	288	4	50	62.7
12:05 PM	8.4	493	733	296	4	50	62.7
12:25 PM	8.4	492	733	306	4	50	62.6
12:40 PM	8.5	494	726	307	4	50	62.7
12:56 PM	8.6	494	740	311	4	50	58.6
shortly after 1:00 PM, testing halted for filter change							
1:20 PM	8.5	493	724	325	4	50	58.5
1:36 PM	8.6	493	720	342	4	50	58.6
1:50 PM	8.4	493	744	338	4	50	58.5
2:05 PM	8.4	490	746	327	4	50	58.5
2:20 PM	8.2	493	747	312	4	50	58.6
port change around 2:45 PM							
2:55 PM	8.2	494	770	30	2	20	58.5
3:37 PM	8.1	493	774	275	4	50	58.5
3:59 PM	8.2	493	746	298	2	20	58.4
4:05 PM	8.2	491	752	256	4	50	58.5
4:20 PM	8.1	491	791	296	4	50	58.4
4:35 PM	8.1	490	788	304	4	50	58.4
4:36 PM					2	20	
4:51 PM	8.1	491	761	283	4	50	58.4
5:06 PM	8.1	493	762	293	4	50	58.3
5:21 PM	8	491	784	296	4	50	58.4
5:35 PM	8	491	764	302	4	50	58.4
5:47 PM	7.9	491	750	301	2	20	58.4

BH delta P = Pressure drop across baghouse (in. of H_2O)
BH T (deg F) = Temperature of gas at inlet to baghouse (deg F)
BET (deg F) = Back end temperature of kiln
Sec T (deg F) = temperature of secondary air to kiln
FOW (%) = % of motor capacity of feed belt

Table 2c. Process Data

10/23/96; Run 2 of Baghouse Tests
Day Kiln Operator = Randy
Stone size = 3/8" by 1 and 1/8"

Time	BH delta P	BH T (deg F)	BET (deg F)	Sec T (deg F)	Gear Setting	FOW (%)	Damper (%)
10:41 AM	8.1	494	744	262	3	22	50.3
10:56 AM	8.1	493	737	266	4	46	50.3
11:15 AM	8.3	493	741	275	4	46	54.8
11:30 AM	8.1	493	745	287	4	46	50.7
11:51 AM	7.9	491	753	286	4	46	50.7
12:06 PM	7.9	491	749	285	4	46	50.7
12:22 PM	7.8	491	742	288	4	46	50.7
12:37 PM	7.8	491	745	290	4	46	50.7
12:52 PM	7.9	494	747	295	4	46	50.7
1:15 PM	8.2	493	743	295	4	46	50.7
1:40 PM	8.3	493	744	291	4	46	50.7
1:55 PM	8.2	492	746	295	4	46	50.7
2:25 PM	7.9	491	768	302	4	46	50.7
2:35 PM	8	492	760	316	4	46	50.7
2:45 PM	7.8	491	761	320	4	48	50.7
3:07 PM	7.9	493	763	321	4	48	50.7
3:22 PM	7.9	491	762	303	4	48	50.7
3:39 PM	7.8	491	767	296	4	48	50.6

BH delta P = Pressure drop across baghouse (in. of H_2O)
BH T (deg F) = Temperature of gas at inlet to baghouse (deg F)
BET (deg F) = Back end temperature of kiln
Sec T (deg F) = temperature of secondary air to kiln
FOW (%) = % of motor capacity of feed belt

Table 2d. Process Data

10/24/96; Run 3 of Baghouse Tests
Day Kiln Operator = Tommy

Time	BH delta P	BH T (deg F)	BET (deg F)	Sec T (deg F)	Gear Setting	FOW (%)	Damper (%)
Stone size = 3/8" by 1 and 1/8"							
11:05 AM	8.3	490	764	302	4	49	60.2
11:20 AM	8.2	489	775	309	4	49	60.3
11:35 AM	8	490	764	316	4	49	60.2
11:50 AM	8	491	764	315	4	49	60.2
12:07 PM	7.9	491	759	309	4	49	60.2
12:35 PM	8.2	493	769	293	4	49	60.2
12:50 PM	8.1	491	777	269	4	49	62.6
1:05 PM	8.1	490	791	256	3	24	64.3
1:06 PM	8.2	490	791	255	4	48	64.3
1:21 PM	8.1	493	787	259	4	47	64.2
1:36 PM	8.2	490	782	264	4	47	64.3
3:34 PM	8.1	490	764	262	4	47	68.3
3:49 PM	8.2	491	765	266	4	47	68.3
4:05 PM	8	493	772	270	2	22	68.3
4:36 PM	8.1	491	774	281	4	46	68.3
4:54 PM	8.1	491	758	299	4	47	68.3
5:14 PM	7.9	491	754	304	4	47	68.3
5:30 PM	8	491	737	318	4	47	68.3
5:45 PM	7.9	493	763	323	4	47	68.3
6:00 PM	7.9	491	760	327	4	47	68.3
6:17 PM	7.7	493	778	299	3	22	68.3
6:31 PM	7.6	493	785	268	4	48	68.3
6:47 PM	7.5	492	776	283	4	48	68.3
7:07 PM	7.5	491	768	289	4	48	74.4
7:22 PM	7.5	494	767	281	3	21	74.4
7:37 PM	7.5	493	766	288	4	42	74.4
7:52 PM	7.6	490	737	299	4	47	74.4

BH delta P = Pressure drop across baghouse (in. of H_2O)
BH T (deg F) = Temperature of gas at inlet to baghouse (deg F)
BET (deg F) = Back end temperature of kiln
Sec T (deg F) = temperature of secondary air to kiln
FOW (%) = % of motor capacity of feed belt

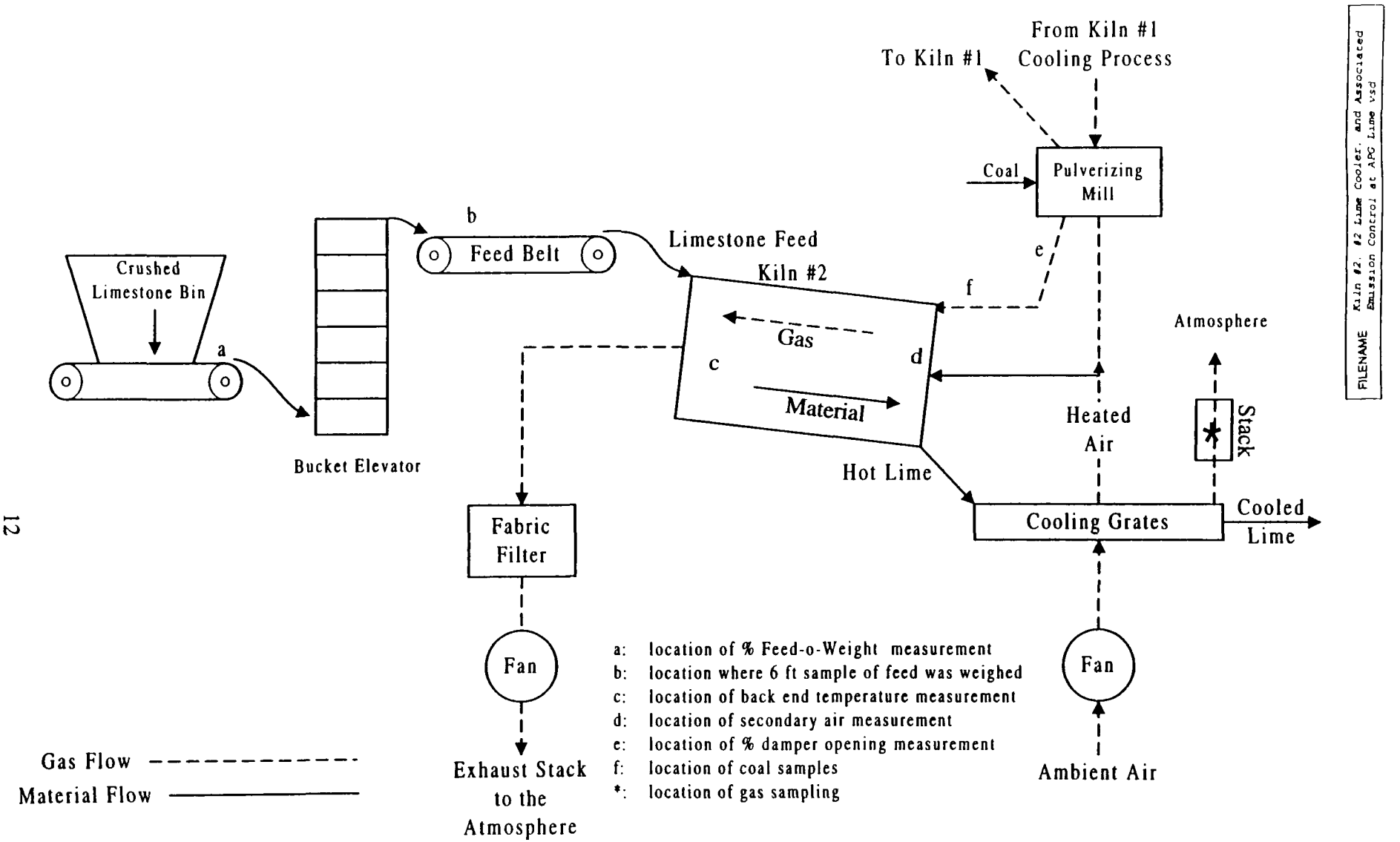

Figure 1. Kiln #2, #2 Lime Cooler, and Associated Emission Control at APG Lime

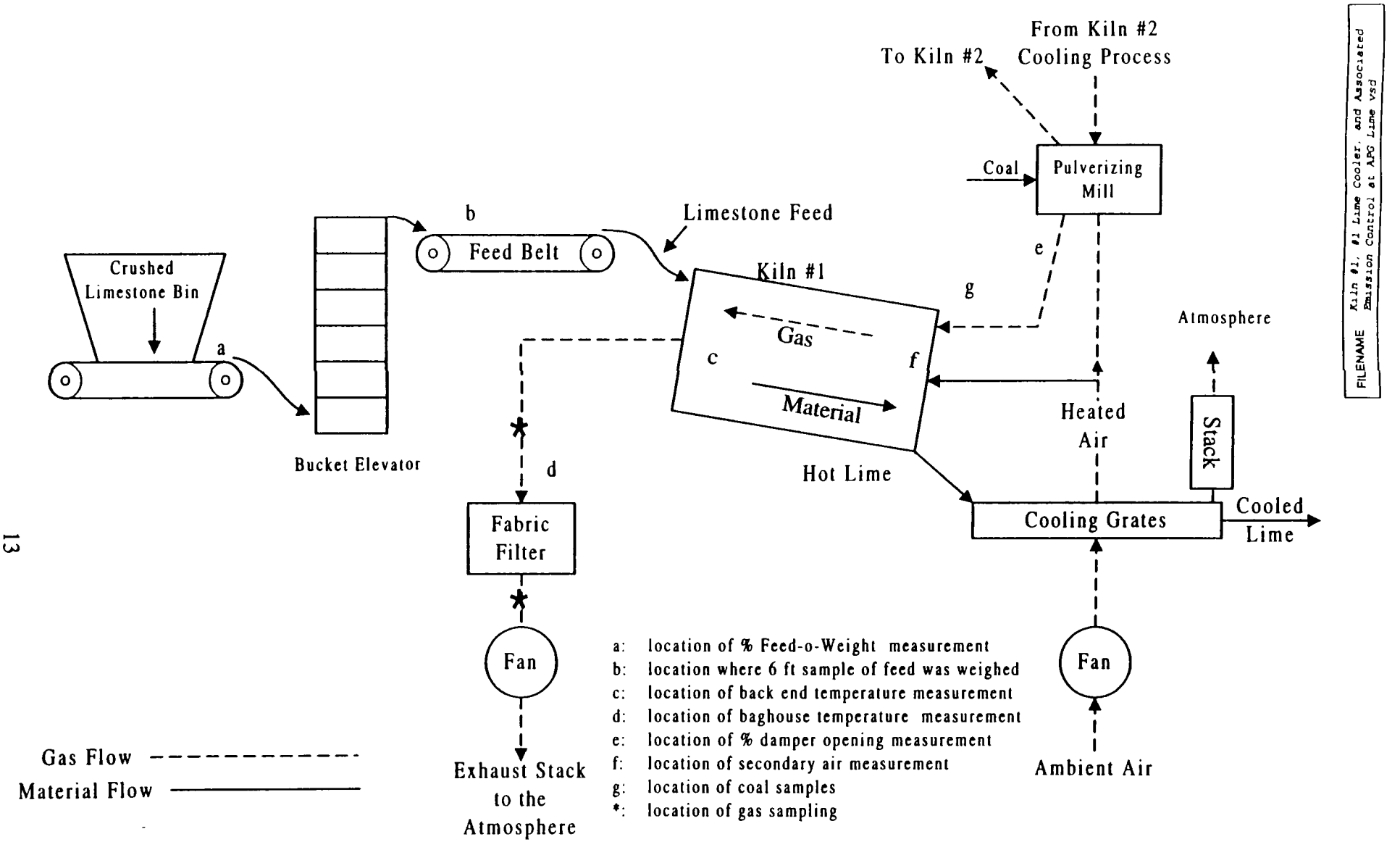

Figure 2. Kiln #1, #1 Lime Cooler, and Associated Emission Control at APG Lime

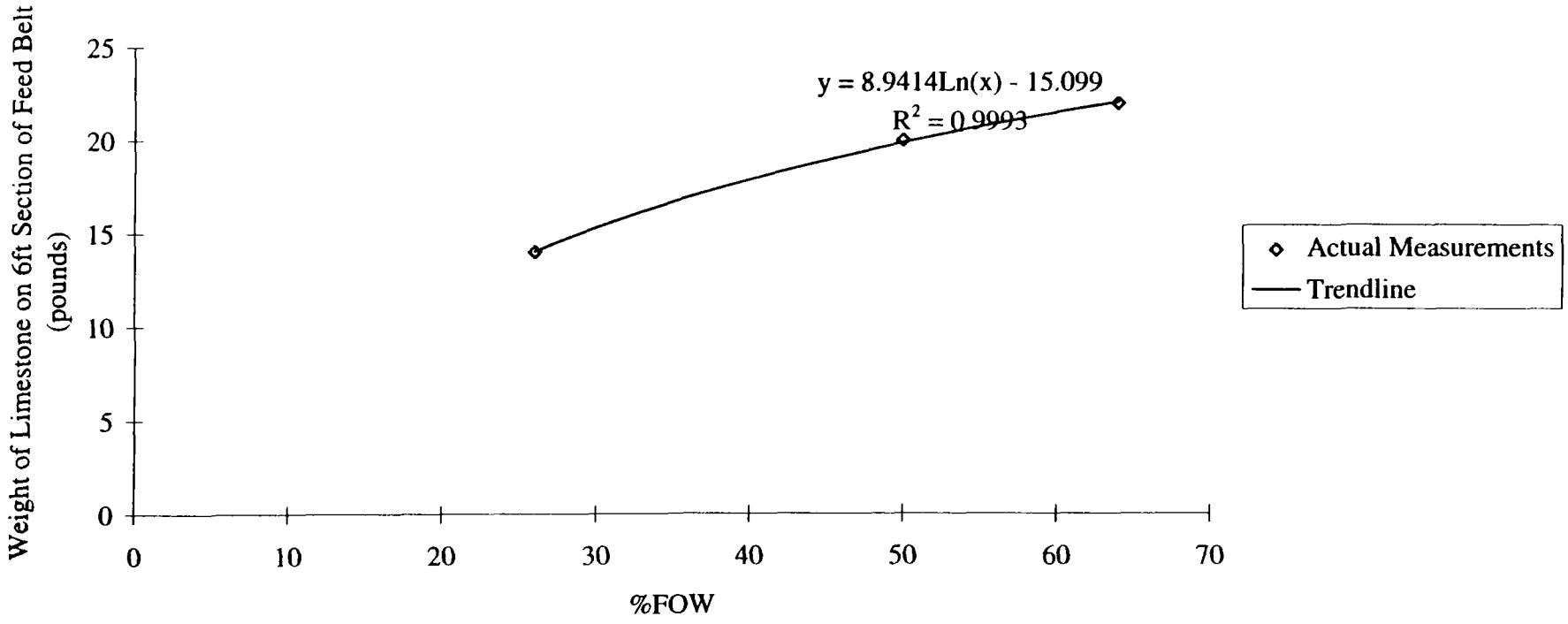

Figure 3. Weight of Limestone on 6 ft Section of Feed Belt vs. % FOW (Feed-O-Weight)

REFERENCES

1. APG response to questionnaire sent out in 1995 by the National Lime Association as part of a voluntary effort with the Environmental Protection Agency to obtain data/information for the MACT program.

2. Heath, Elizabeth, Research Triangle Institute. Site Survey of APG Lime, Inc., Ripplemead, Virginia. February, 1996.

3. Ref 1.

4. Ref 2.

5. Ref 2.

6. Ref 2.

7. Brockmann, Cybele, Research Triangle Institute. Conversation with plant personnel during testing (10/21/96 through 10/24/96).

8. Ref 1.

9. Ref 2.

10. Ref 1.

11. 1.

12. 1.

13. 1.

14. Telecommunication between Cybele Brockmann of Research Triangle Institute and plant personnel on November 25, 1996.

15. Ref 14.

15. 1.

16. 1.

18. Reference 1

APPENDIX E.

EPA Method 320
EPA FTIR PROTOCOL

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_i) and analytical uncertainty (AU_i) for each analyte.

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

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

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

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

2.0 Summary of Method.

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

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

path (background).

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

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

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

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

2.2 Sampling and Analysis. In extractive sampling a probe

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

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

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

where:

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

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

b = path length of the cell.

c_i = concentration of the ith sample component.

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

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

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

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

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

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

3.0 Definitions.

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

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

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

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

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

configurations or sampling temperatures.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4.0 Interferences.

Interferences are divided into two classifications: analytical and sampling.

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

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

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

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

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

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

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

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

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

5.0 Safety.

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

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

6.0 Equipment and Supplies.

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

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

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

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

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

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

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

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

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

analytical system.

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

6.8 Gas Regulators. Appropriate for individual gas standards.

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

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

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

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

6.13 FTIR Analytical System. Spectrometer and detector,

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

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

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

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

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

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

sample.

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

7.0 Reagents and Standards.

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

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

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

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

8.0 Sampling and Analysis Procedure.

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

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

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

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

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

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

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

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

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

8.1.6 Calculate the Minimum Analyte Uncertainty, MAU (section 1.3 of this method discusses MAU and protocol appendix D gives the MAU procedure). The MAU for each analyte, i, and each analytical region, m, depends on the RMS noise.

8.1.7 Analytical Program. See FTIR Protocol, section 4.10. Prepare computer program based on the chosen analytical technique. Use as input reference spectra of all target analytes and expected interferants. Reference spectra of additional compounds shall also be included in the program

if their presence (even if transient) in the samples is considered possible. The program output shall be in ppm (or ppb) and shall be corrected for differences between the reference path length, L_R, temperature, T_R, and pressure, P_R, and the conditions used for collecting the sample spectra. If sampling is performed at ambient pressure, then any pressure correction is usually small relative to corrections for path length and temperature, and may be neglected.

8.2 Leak-check.

8.2.1 Sampling System. A typical FTIR extractive sampling train is shown in Figure 1. Leak check from the probe tip to pump outlet as follows: Connect a 0- to 250-mL/min rate meter (rotameter or bubble meter) to the outlet of the pump. Close off the inlet to the probe, and record the leak rate. The leak rate shall be ≤ 200 mL/min.

8.2.2 Analytical System Leak check. Leak check the FTIR cell under vacuum and under pressure (greater than ambient). Leak check connecting tubing and inlet manifold under pressure.

8.2.2.1 For the evacuated sample technique, close the valve to the FTIR cell, and evacuate the absorption cell to the minimum absolute pressure P_{min}. Close the valve to the pump, and determine the change in pressure ΔP_v after 2 minutes.

8.2.2.2 For both the evacuated sample and purging techniques, pressurize the system to about 100 mmHg above

atmospheric pressure. Isolate the pump and determine the change in pressure ΔP_p after 2 minutes.

8.2.2.3 Measure the barometric pressure, P_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:

$$\%V_L = 50\, t_{SS} \frac{\Delta P_{max}}{P_{SS}} \qquad \textbf{(2)}$$

where 50 = 100% divided by the leak-check time of 2 minutes.

8.2.2.5 Leak volumes in excess of 4 percent of the FTIR system volume V_{SS} are unacceptable.

8.3 Detector Linearity. Once an optical configuration is chosen, use one of the procedures of sections 8.3.1 through 8.3.3 to verify that the detector response is linear. If the detector response is not linear, decrease the aperture, or attenuate the infrared beam. After a change in the instrument configuration perform a linearity check until it is demonstrated that the detector response is linear.

8.3.1 Vary the power incident on the detector by modifying the aperture setting. Measure the background and CTS at three instrument aperture settings: (1) at the aperture setting to be used in the testing, (2) at one half this aperture and (3) at twice the proposed testing aperture.

Compare the three CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system. If test aperture is the maximum aperture, collect CTS spectrum at maximum aperture, then close the aperture to reduce the IR throughput by half. Collect a second background and CTS at the smaller aperture setting and compare the spectra again.

8.3.2 Use neutral density filters to attenuate the infrared beam. Set up the FTIR system as it will be used in the test measurements. Collect a CTS spectrum. Use a neutral density filter to attenuate the infrared beam (either immediately after the source or the interferometer) to approximately 1/2 its original intensity. Collect a second CTS spectrum. Use another filter to attenuate the infrared beam to approximately 1/4 its original intensity. Collect a third background and CTS spectrum. Compare the CTS spectra. CTS band areas shall agree to within the uncertainty of the cylinder standard and the RMSD noise in the system.

8.3.3 Observe the single beam instrument response in a frequency region where the detector response is known to be zero. Verify that the detector response is "flat" and equal to zero in these regions.

8.4 Data Storage Requirements. All field test spectra shall be stored on a computer disk and a second backup copy must stored on a separate disk. The stored information

includes sample interferograms, processed absorbance spectra, background interferograms, CTS sample interferograms and CTS absorbance spectra. Additionally, documentation of all sample conditions, instrument settings, and test records must be recorded on hard copy or on computer medium. Table 1 gives a sample presentation of documentation.

8.5 Background Spectrum. Evacuate the gas cell to ≤ 5 mmHg, and fill with dry nitrogen gas to ambient pressure (or purge the cell with 10 volumes of dry nitrogen). Verify that no significant amounts of absorbing species (for example water vapor and CO_2) are present. Collect a background spectrum, using a signal averaging period equal to or greater than the averaging period for the sample spectra. Assign a unique file name to the background spectrum. Store two copies of the background interferogram and processed single-beam spectrum on separate computer disks (one copy is the back-up).

8.5.1 Interference Spectra. If possible, collect spectra of known and suspected major interferences using the same optical system that will be used in the field measurements. This can be done on-site or earlier. A number of gases, e.g. CO_2, SO_2, CO, NH_3, are readily available from cylinder gas suppliers.

8.5.2 Water vapor spectra can be prepared by the following

procedure. Fill a sample tube with distilled water. Evacuate above the sample and remove dissolved gasses by alternately freezing and thawing the water while evacuating. Allow water vapor into the FTIR cell, then dilute to atmospheric pressure with nitrogen or dry air. If quantitative water spectra are required, follow the reference spectrum procedure for neat samples (protocol, section 4.6). Often, interference spectra need not be quantitative, but for best results the absorbance must be comparable to the interference absorbance in the sample spectra.

8.6 Pre-Test Calibrations

8.6.1 Calibration Transfer Standard. Evacuate the gas cell to ≤ 5 mmHg absolute pressure, and fill the FTIR cell to atmospheric pressure with the CTS gas. Alternatively, purge the cell with 10 cell volumes of CTS gas. (If purge is used, verify that the CTS concentration in the cell is stable by collecting two spectra 2 minutes apart as the CTS gas continues to flow. If the absorbance in the second spectrum is no greater than in the first, within the uncertainty of the gas standard, then this can be used as the CTS spectrum.) Record the spectrum.

8.6.2 QA Spike. This procedure assumes that the method has been validated for at least some of the target analytes at the source. For emissions testing perform a QA spike. Use

a certified standard, if possible, of an analyte, which has been validated at the source. One analyte standard can serve as a QA surrogate for other analytes which are less reactive or less soluble than the standard. Perform the spike procedure of section 9.2 of this method. Record spectra of at least three independent (section 3.22 of this method) spiked samples. Calculate the spiked component of the analyte concentration. If the average spiked concentration is within 0.7 to 1.3 times the expected concentration, then proceed with the testing. If applicable, apply the correction factor from the Method 301 of this appendix validation test (not the result from the QA spike).

8.7 Sampling. If analyte concentrations vary rapidly with time, continuous sampling is preferable using the smallest cell volume, fastest sampling rate and fastest spectra collection rate possible. Continuous sampling requires the least operator intervention even without an automated sampling system. For continuous monitoring at one location over long periods, Continuous sampling is preferred. Batch sampling and continuous static sampling are used for screening and performing test runs of finite duration. Either technique is preferred for sampling several locations in a matter of days. Batch sampling gives reasonably good time resolution and ensures that each spectrum measures a

discreet (and unique) sample volume. Continuous static (and continuous) sampling provide a very stable background over long periods. Like batch sampling, continuous static sampling also ensures that each spectrum measures a unique sample volume. It is essential that the leak check procedure under vacuum (section 8.2 of this method) is passed if the batch sampling procedure is used. It is essential that the leak check procedure under positive pressure is passed if the continuous static or continuous sampling procedures are used. The sampling techniques are described in sections 8.7.1 through 8.7.2 of this method.

8.7.1 Batch Sampling. Evacuate the absorbance cell to ≤ 5 mmHg absolute pressure. Fill the cell with exhaust gas to ambient pressure, isolate the cell, and record the spectrum. Before taking the next sample, evacuate the cell until no spectral evidence of sample absorption remains. Repeat this procedure to collect eight spectra of separate samples in 1 hour.

8.7.2 Continuous Static Sampling. Purge the FTIR cell with 10 cell volumes of sample gas. Isolate the cell, collect the spectrum of the static sample and record the pressure. Before measuring the next sample, purge the cell with 10 more cell volumes of sample gas.

8.8 Sampling QA and Reporting.

8.8.1 Sample integration times shall be sufficient to

achieve the required signal-to-noise ratio. Obtain an absorbance spectrum by filling the cell with N_2. Measure the RMSD in each analytical region in this absorbance spectrum. Verify that the number of scans used is sufficient to achieve the target MAU.

8.8.2 Assign a unique file name to each spectrum.

8.8.3 Store two copies of sample interferograms and processed spectra on separate computer disks.

8.8.4 For each sample spectrum, document the sampling conditions, the sampling time (while the cell was being filled), the time the spectrum was recorded, the instrumental conditions (path length, temperature, pressure, resolution, signal integration time), and the spectral file name. Keep a hard copy of these data sheets.

8.9 Signal Transmittance. While sampling, monitor the signal transmittance. If signal transmittance (relative to the background) changes by 5 percent or more (absorbance = -.02 to .02) in any analytical spectral region, obtain a new background spectrum.

8.10 Post-test CTS. After the sampling run, record another CTS spectrum.

8.11 Post-test QA.

8.11.1 Inspect the sample spectra immediately after the run to verify that the gas matrix composition was close to the expected (assumed) gas matrix.

8.11.2 Verify that the sampling and instrumental parameters were appropriate for the conditions encountered. For example, if the moisture is much greater than anticipated, it may be necessary to use a shorter path length or dilute the sample.

8.11.3 Compare the pre- and post-test CTS spectra. The peak absorbance in pre- and post-test CTS must be ± 5 percent of the mean value. See appendix E of the FTIR Protocol.

9.0 Quality Control.

Use analyte spiking (sections 8.6.2, 9.2 and 13.0 of this method) to verify that the sampling system can transport the analytes from the probe to the FTIR system.

9.1 Spike Materials. Use a certified standard (accurate to ± 2 percent) of the target analyte, if one can be obtained. If a certified standard cannot be obtained, follow the procedures in section 4.6.2.2 of the FTIR Protocol.

9.2 Spiking Procedure. QA spiking (section 8.6.2 of this method) is a calibration procedure used before testing. QA spiking involves following the spike procedure of sections 9.2.1 through 9.2.3 of this method to obtain at least three spiked samples. The analyte concentrations in the spiked samples shall be compared to the expected spike concentration to verify that the sampling/analytical system is working properly. Usually, when QA spiking is used, the

method has already been validated at a similar source for the analyte in question. The QA spike demonstrates that the validated sampling/analytical conditions are being duplicated. If the QA spike fails then the sampling/analytical system shall be repaired before testing proceeds. The method validation procedure (section 13.0 of this method) involves a more extensive use of the analyte spike procedure of sections 9.2.1 through 9.2.3 of this method. Spectra of at least 12 independent spiked and 12 independent unspiked samples are recorded. The concentration results are analyzed statistically to determine if there is a systematic bias in the method for measuring a particular analyte. If there is a systematic bias, within the limits allowed by Method 301 of this appendix, then a correction factor shall be applied to the analytical results. If the systematic bias is greater than the allowed limits, this method is not valid and cannot be used.

9.2.1 Introduce the spike/tracer gas at a constant flow rate of ≤ 10 percent of the total sample flow, when possible. (Note: Use the rotameter at the end of the sampling train to estimate the required spike/tracer gas flow rate.) Use a flow device, e.g., mass flow meter (± 2 percent), to monitor the spike flow rate. Record the spike flow rate every 10 minutes.

9.2.2 Determine the response time (RT) of the system by continuously collecting spectra of the spiked effluent until the spectrum of the spiked component is constant for 5 minutes. The RT is the interval from the first measurement until the spike becomes constant. Wait for twice the duration of the RT, then collect spectra of two independent spiked gas samples. Duplicate analyses of the spiked concentration shall be within 5 percent of the mean of the two measurements.

9.2.3 Calculate the dilution ratio using the tracer gas as follows:

$$DF = \frac{SF_{6(spk)}}{SF_{6(dir)}} \qquad \textbf{(3)}$$

where:

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

DF = Dilution factor of the spike gas; this value shall be ≥10.

$SF_{6(dir)}$ = SF_6 (or tracer gas) concentration measured directly in undiluted spike gas.

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

$Spike_{dir}$ = Concentration of the analyte in the spike standard measured by filling the FTIR cell directly.

CS = Expected concentration of the spiked samples

Unspike = Native concentration of analytes in unspiked samples

10.0 Calibration and Standardization.

10.1 Signal-to-Noise Ratio (S/N). The RMSD in the noise must be less than one tenth of the minimum analyte peak absorbance in each analytical region. For example if the minimum peak absorbance is 0.01 at the required DL, then RMSD measured over the entire analytical region must be $\leq$ 0.001.

10.2 Absorbance Path length. Verify the absorbance path length by comparing reference CTS spectra to test CTS spectra. See appendix E of the FTIR Protocol.

10.3 Instrument Resolution. Measure the line width of appropriate test CTS band(s) to verify instrument resolution. Alternatively, compare CTS spectra to a reference CTS spectrum, if available, measured at the nominal resolution.

10.4 Apodization Function. In transforming the sample interferograms to absorbance spectra use the same

apodization function that was used in transforming the reference spectra.

10.5 FTIR Cell Volume. Evacuate the cell to ≤ 5 mmHg. Measure the initial absolute temperature (T_i) and absolute pressure (P_i). Connect a wet test meter (or a calibrated dry gas meter), and slowly draw room air into the cell. Measure the meter volume (V_m), meter absolute temperature (T_m), and meter absolute pressure (P_m); and the cell final absolute temperature (T_f) and absolute pressure (P_f). Calculate the FTIR cell volume V_{SS}, including that of the connecting tubing, as follows:

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

11.0 Data Analysis and Calculations.

Analyte concentrations shall be measured using reference spectra from the EPA FTIR spectral library. When EPA library spectra are not available, the procedures in section 4.6 of the Protocol shall be followed to prepare reference spectra of all the target analytes.

11.1 Spectral De-resolution. Reference spectra can be converted to lower resolution standard spectra (section 3.3

of this method) by truncating the original reference sample and background interferograms. Appendix K of the FTIR Protocol gives specific deresolution procedures. Deresolved spectra shall be transformed using the same apodization function and level of zero filling as the sample spectra. Additionally, pre-test FTIR protocol calculations (e.g., FRU, MAU, FCU) shall be performed using the de-resolved standard spectra.

11.2 Data Analysis. Various analytical programs are available for relating sample absorbance to a concentration standard. Calculated concentrations shall be verified by analyzing residual baselines after mathematically subtracting scaled reference spectra from the sample spectra. A full description of the data analysis and calculations is contained in the FTIR Protocol (sections 4.0, 5.0, 6.0 and appendices). Correct the calculated concentrations in the sample spectra for differences in absorption path length and temperature between the reference and sample spectra using equation 6,

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

where:

C_{corr} = Concentration, corrected for path length.

C_{calc} = Concentration, initial calculation (output of the analytical program designed for the compound).

L_r = Reference spectra path length.

L_s = Sample spectra path length.

T_s = Absolute temperature of the sample gas, K.

T_r = Absolute gas temperature of reference spectra, K.

P_s = Sample cell pressure.

P_r = Reference spectrum sample pressure.

12.0 Method Performance.

12.1 Spectral Quality. Refer to the FTIR Protocol appendices for analytical requirements, evaluation of data quality, and analysis of uncertainty.

12.2 Sampling QA/QC. The analyte spike procedure of section 9 of this method, the QA spike of section 8.6.2 of this method, and the validation procedure of section 13 of this method are used to evaluate the performance of the sampling system and to quantify sampling system effects, if any, on the measured concentrations. This method is self-validating provided that the results meet the performance requirement of the QA spike in sections 9.0 and 8.6.2 of this method and results from a previous method validation study support the use of this method in the application. Several factors can contribute to uncertainty in the measurement of spiked samples. Factors which can be controlled to provide better accuracy in the spiking procedure are listed in sections 12.2.1 through 12.2.4 of this method.

12.2.1 Flow meter. An accurate mass flow meter is accurate to ± 1 percent of its span. If a flow of 1 L/min is monitored with such a MFM, which is calibrated in the range of 0-5 L/min, the flow measurement has an uncertainty of 5 percent. This may be improved by re-calibrating the meter at the specific flow rate to be used.

12.2.2 Calibration gas. Usually the calibration standard is certified to within ± 2 percent. With reactive analytes, such as HCl, the certified accuracy in a commercially available standard may be no better than ± 5 percent.

12.2.3 Temperature. Temperature measurements of the cell shall be quite accurate. If practical, it is preferable to measure sample temperature directly, by inserting a thermocouple into the cell chamber instead of monitoring the cell outer wall temperature.

12.2.4 Pressure. Accuracy depends on the accuracy of the barometer, but fluctuations in pressure throughout a day may be as much as 2.5 percent due to weather variations.

13.0 Method Validation Procedure.

This validation procedure, which is based on EPA Method 301 (40 CFR part 63, appendix A), may be used to validate this method for the analytes in a gas matrix. Validation at one source may also apply to another type of source, if it can be shown that the exhaust gas characteristics are similar at both sources.

13.1 Section 5.3 of Method 301 (40 CFR part 63, appendix A), the Analyte Spike procedure, is used with these modifications. The statistical analysis of the results follows section 6.3 of EPA Method 301. Section 3 of this method defines terms that are not defined in Method 301.

13.1.1 The analyte spike is performed dynamically. This means the spike flow is continuous and constant as spiked samples are measured.

13.1.2 The spike gas is introduced at the back of the sample probe.

13.1.3 Spiked effluent is carried through all sampling components downstream of the probe.

13.1.4 A single FTIR system (or more) may be used to collect and analyze spectra (not quadruplicate integrated sampling trains).

13.1.5 All of the validation measurements are performed sequentially in a single "run" (section 3.26 of this method).

13.1.6 The measurements analyzed statistically are each independent (section 3.22 of this method).

13.1.7 A validation data set can consist of more than 12 spiked and 12 unspiked measurements.

13.2 Batch Sampling. The procedure in sections 13.2.1 through 13.2.2 may be used for stable processes. If process emissions are highly variable, the procedure in section

13.2.3 shall be used.

13.2.1 With a single FTIR instrument and sampling system, begin by collecting spectra of two unspiked samples. Introduce the spike flow into the sampling system and allow 10 cell volumes to purge the sampling system and FTIR cell. Collect spectra of two spiked samples. Turn off the spike and allow 10 cell volumes of unspiked sample to purge the FTIR cell. Repeat this procedure until the 24 (or more) samples are collected.

13.2.2 In batch sampling, collect spectra of 24 distinct samples. (Each distinct sample consists of filling the cell to ambient pressure after the cell has been evacuated.)

13.2.3 Alternatively, a separate probe assembly, line, and sample pump can be used for spiked sample. Verify and document that sampling conditions are the same in both the spiked and the unspiked sampling systems. This can be done by wrapping both sample lines in the same heated bundle. Keep the same flow rate in both sample lines. Measure samples in sequence in pairs. After two spiked samples are measured, evacuate the FTIR cell, and turn the manifold valve so that spiked sample flows to the FTIR cell. Allow the connecting line from the manifold to the FTIR cell to purge thoroughly (the time depends on the line length and flow rate). Collect a pair of spiked samples. Repeat the procedure until at least 24 measurements are completed.

13.3 Simultaneous Measurements With Two FTIR Systems. If unspiked effluent concentrations of the target analyte(s) vary significantly with time, it may be desirable to perform synchronized measurements of spiked and unspiked sample. Use two FTIR systems, each with its own cell and sampling system to perform simultaneous spiked and unspiked measurements. The optical configurations shall be similar, if possible. The sampling configurations shall be the same. One sampling system and FTIR analyzer shall be used to measure spiked effluent. The other sampling system and FTIR analyzer shall be used to measure unspiked flue gas. Both systems shall use the same sampling procedure (i.e., batch or continuous).

13.3.1 If batch sampling is used, synchronize the cell evacuation, cell filling, and collection of spectra. Fill both cells at the same rate (in cell volumes per unit time).

13.3.2 If continuous sampling is used, adjust the sample flow through each gas cell so that the same number of cell volumes pass through each cell in a given time (i.e. $TC_1 = TC_2$).

13.4 Statistical Treatment. The statistical procedure of EPA Method 301 of this appendix, section 6.3 is used to evaluate the bias and precision. For FTIR testing a validation "run" is defined as spectra of 24 independent samples, 12 of which are spiked with the analyte(s) and 12

of which are not spiked.

13.4.1 Bias. Determine the bias (defined by EPA Method 301 of this appendix, section 6.3.2) using equation 7:

$$B = S_m - CS \tag{7}$$

where:

B = Bias at spike level.

S_m = Mean concentration of the analyte spiked samples.

CS = Expected concentration of the spiked samples.

13.4.2 Correction Factor. Use section 6.3.2.2 of Method 301 of this appendix to evaluate the statistical significance of the bias. If it is determined that the bias is significant, then use section 6.3.3 of Method 301 to calculate a correction factor (CF). Analytical results of the test method are multiplied by the correction factor, if $0.7 \leq CF \leq 1.3$. If is determined that the bias is significant and CF > ± 30 percent, then the test method is considered to "not valid."

13.4.3 If measurements do not pass validation, evaluate the sampling system, instrument configuration, and analytical system to determine if improper set-up or a malfunction was the cause. If so, repair the system and repeat the validation.

14.0 Pollution Prevention.

The extracted sample gas is vented outside the enclosure containing the FTIR system and gas manifold after the analysis. In typical method applications the vented sample volume is a small fraction of the source volumetric flow and its composition is identical to that emitted from the source. When analyte spiking is used, spiked pollutants are vented with the extracted sample gas. Approximately 1.6 x 10^{-4} to 3.2 x 10^{-4} lbs of a single HAP may be vented to the atmosphere in a typical validation run of 3 hours. (This assumes a molar mass of 50 to 100 g, spike rate of 1.0 L/min, and a standard concentration of 100 ppm). Minimize emissions by keeping the spike flow off when not in use.

15.0 Waste Management.

Small volumes of laboratory gas standards can be vented through a laboratory hood. Neat samples must be packed and disposed according to applicable regulations. Surplus materials may be returned to supplier for disposal.

16.0 References.

1. "Field Validation Test Using Fourier Transform Infrared (FTIR) Spectrometry To Measure Formaldehyde, Phenol and Methanol at a Wool Fiberglass Production Facility." Draft. U.S. Environmental Protection Agency Report, EPA Contract No. 68D20163, Work Assignment I-32, September 1994.

2. "FTIR Method Validation at a Coal-Fired Boiler".

Prepared for U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No.: EPA-454/R95-004, NTIS No.: PB95-193199. July, 1993.

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

4. "Molecular Vibrations; The Theory of Infrared and Raman Vibrational Spectra," E. Bright Wilson, J. C. Decius, and P. C. Cross, Dover Publications, Inc., 1980. For a less intensive treatment of molecular rotational-vibrational spectra see, for example, "Physical Chemistry," G. M. Barrow, chapters 12, 13, and 14, McGraw Hill, Inc., 1979.

5. "Fourier Transform Infrared Spectrometry," Peter R. Griffiths and James de Haseth, **Chemical Analysis**, **83**, 16-25,(1986), P. J. Elving, J. D. Winefordner and I. M. Kolthoff (ed.), John Wiley and Sons.

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

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

Table 1. EXAMPLE PRESENTATION OF SAMPLING DOCUMENTATION.

Sample Time	Spectrum File Name	Background File Name	Sample conditioning	Process condition

Sample Time	Spectrum File	Interferogram	Resolution	Scans	Apodization	Gain	CTS Spectrum

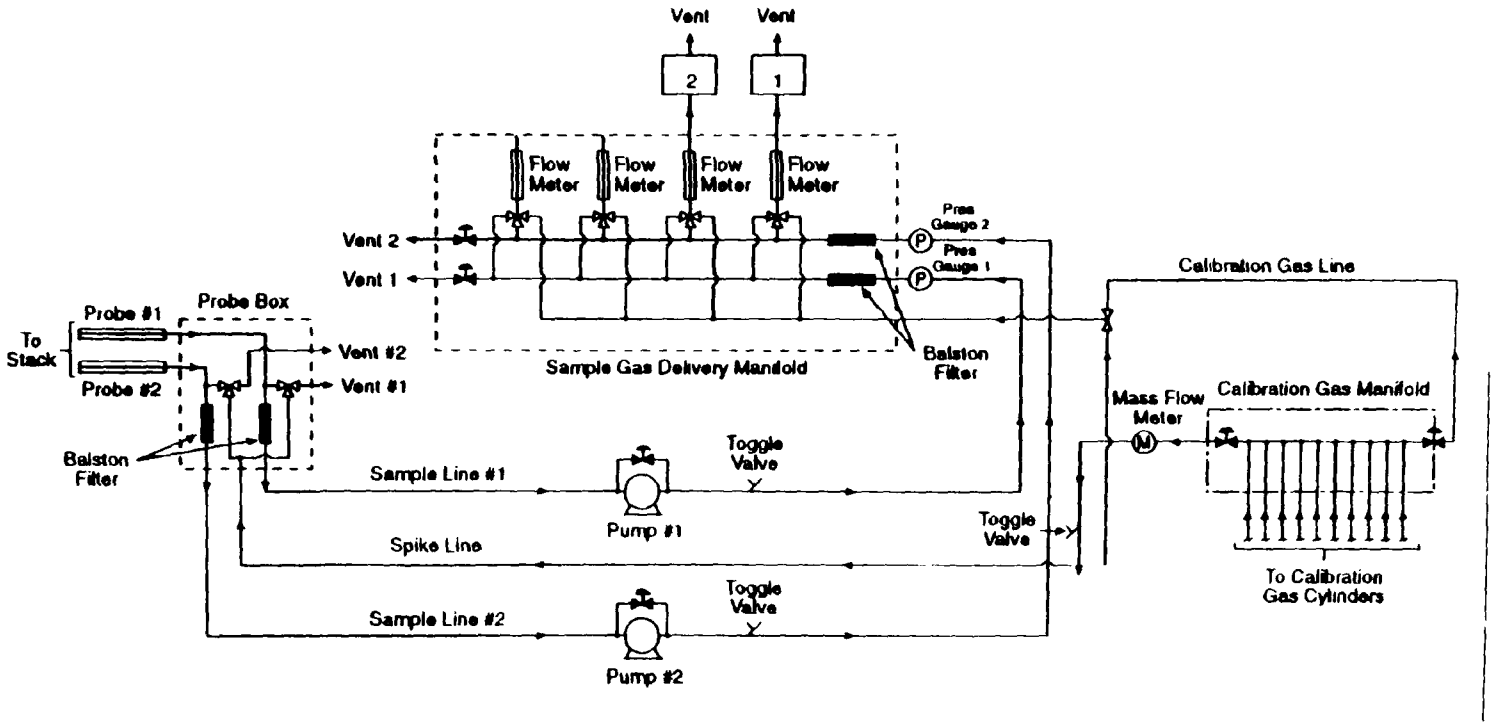

Figure 1. Extractive FTIR sampling system.

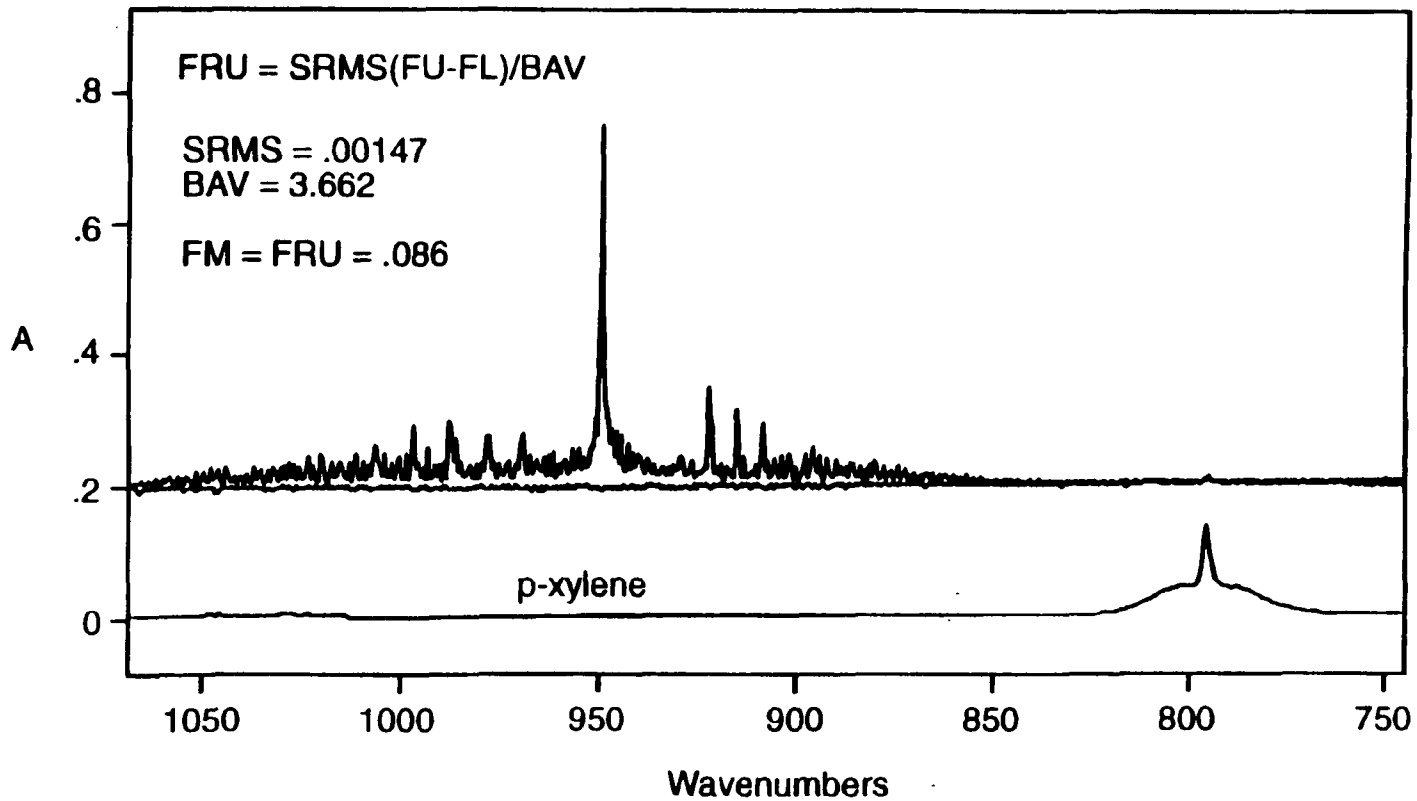

Figure 2. Fractional Reproducibility. Top: average of cts1031a and cts1101b. Bottom: Reference spectrum of p-xylene.

PROTOCOL FOR THE USE OF EXTRACTIVE FOURIER TRANSFORM INFRARED (FTIR) SPECTROMETRY FOR THE ANALYSES OF GASEOUS EMISSIONS FROM STATIONARY SOURCES

INTRODUCTION

The purpose of this document is to set general guidelines for the use of modern FTIR spectroscopic methods for the analysis of gas samples extracted from the effluent of stationary emission sources. This document outlines techniques for developing and evaluating such methods and sets basic requirements for reporting and quality assurance procedures.

1.0 NOMENCLATURE

1.1 Appendix A lists definitions of the symbols and terms used in this Protocol, many of which have been taken directly from American Society for Testing and Materials (ASTM) publication E 131-90a, entitled "Terminology Relating to Molecular Spectroscopy."

1.2 Except in the case of background spectra or where otherwise noted, the term "spectrum" refers to a double-beam spectrum in units of absorbance vs. wavenumber (cm^{-1}).

1.3 The term "Study" in this document refers to a publication that has been subjected to EPA- or peer-review.

2.0 APPLICABILITY AND ANALYTICAL PRINCIPLE

2.1 Applicability. This Protocol applies to the determination of compound-specific concentrations in single- and multiple-component gas phase samples using double-beam absorption spectroscopy in the mid-infrared band. It does not specifically address other FTIR applications, such as single-beam spectroscopy, analysis of open-path (non-enclosed) samples, and continuous measurement techniques. If multiple spectrometers, absorption cells, or instrumental linewidths are used in such analyses, each distinct operational configuration of the system must be evaluated separately according to this Protocol.

2.2 Analytical Principle.

2.2.1 In the mid-infrared band, most molecules exhibit characteristic gas phase absorption spectra that may be recorded by FTIR systems. Such systems consist of a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements for the transfer of infrared radiation between components, and gas flow control and measurement components. Adjunct and integral computer systems are used for controlling

the instrument, processing the signal, and for performing both Fourier transforms and quantitative analyses of spectral data.

2.2.2 The absorption spectra of pure gases and of mixtures of gases are described by a linear absorbance theory referred to as Beer's Law. Using this law, modern FTIR systems use computerized analytical programs to quantify compounds by comparing the absorption spectra of known (reference) gas samples to the absorption spectrum of the sample gas. Some standard mathematical techniques used for comparisons are classical least squares, inverse least squares, cross-correlation, factor analysis, and partial least squares. Reference A describes several of these techniques, as well as additional techniques, such as differentiation methods, linear baseline corrections, and non-linear absorbance corrections.

3.0 GENERAL PRINCIPLES OF PROTOCOL REQUIREMENTS

The characteristics that distinguish FTIR systems from gas analyzers used in instrumental gas analysis methods (e.g., EPA Methods 6C and 7E) are: (1) Computers are necessary to obtain and analyze data; (2) chemical concentrations can be quantified using previously recorded infrared reference spectra; and (3) analytical assumptions and results, including possible effects of interfering compounds, can be evaluated after the quantitative analysis. The following general principles and requirements of this Protocol are based on these characteristics.

3.1 Verifiability and Reproducibility of Results. Store all data and document data analysis techniques sufficient to allow an independent agent to reproduce the analytical results from the raw interferometric data.

3.2 Transfer of Reference Spectra. To determine whether reference spectra recorded under one set of conditions (e.g., optical bench, instrumental linewidth, absorption pathlength, detector performance, pressure, and temperature) can be used to analyze sample spectra taken under a different set of conditions, quantitatively compare "calibration transfer standards" (CTS) and reference spectra as described in this Protocol. (Note: The CTS may, but need not, include analytes of interest). To effect this, record the absorption spectra of the CTS (a) immediately before and immediately after recording reference spectra and (b) immediately after recording sample spectra.

3.3 Evaluation of FTIR Analyses. The applicability, accuracy, and precision of FTIR measurements are influenced by a number of interrelated factors, which may be divided into two classes:

3.3.1 Sample-Independent Factors. Examples are system configuration and performance (e.g., detector sensitivity and infrared source output), quality and applicability of reference

absorption spectra, and type of mathematical analyses of the spectra. These factors define the fundamental limitations of FTIR measurements for a given system configuration. These limitations may be estimated from evaluations of the system before samples are available. For example, the detection limit for the absorbing compound under a given set of conditions may be estimated from the system noise level and the strength of a particular absorption band. Similarly, the accuracy of measurements may be estimated from the analysis of the reference spectra.

3.3.2 Sample-Dependent Factors. Examples are spectral interferants (e.g., water vapor and CO_2) or the overlap of spectral features of different compounds and contamination deposits on reflective surfaces or transmitting windows. To maximize the effectiveness of the mathematical techniques used in spectral analysis, identification of interferants (a standard initial step) and analysis of samples (includes effects of other analytical errors) are necessary. Thus, the Protocol requires post-analysis calculation of measurement concentration uncertainties for the detection of these potential sources of measurement error.

4.0 PRE-TEST PREPARATIONS AND EVALUATIONS

Before testing, demonstrate the suitability of FTIR spectrometry for the desired application according to the procedures of this section.

4.1 Identify Test Requirements. Identify and record the test requirements described below in 4.1.1 through 4.1.5. These values set the desired or required goals of the proposed analysis; the description of methods for determining whether these goals are actually met during the analysis comprises the majority of this Protocol.

4.1.1 Analytes (specific chemical species) of interest. Label the analytes from i = 1 to I.

4.1.2 Analytical uncertainty limit (AU_i). The AU_i is the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration in the sample.

4.1.3 Required detection limit for each analyte (DL_i, ppm). The detection limit is the lowest concentration of an analyte for which its overall fractional uncertainty (OFU_i) is required to be less than its analytical uncertainty limit (AU_i).

4.1.4 Maximum expected concentration of each analyte ($CMAX_i$, ppm).

4.2 Identify Potential Interferants. Considering the chemistry of the process or results of previous Studies, identify potential interferants, i.e., the major effluent constituents and any relatively minor effluent constituents that possess either strong absorption characteristics or strong structural similarities to any analyte of interest. Label them 1 through N_j, where the subscript "j" pertains to potential interferants. Estimate the concentrations of these compounds in the effluent ($CPOT_j$, ppm).

4.3 Select and Evaluate the Sampling System. Considering the source, e.g., temperature and pressure profiles, moisture content, analyte characteristics, and particulate concentration), select the equipment for extracting gas samples. Recommended are a particulate filter, heating system to maintain sample temperature above the dew point for all sample constituents at all points within the sampling system (including the filter), and sample conditioning system (e.g., coolers, water-permeable membranes that remove water or other compounds from the sample, and dilution devices) to remove spectral interferants or to protect the sampling and analytical components. Determine the minimum absolute sample system pressure (P_{min}, mmHg) and the infrared absorption cell volume (V_{SS}, liter). Select the techniques and/or equipment for the measurement of sample pressures and temperatures.

4.4 Select Spectroscopic System. Select a spectroscopic configuration for the application. Approximate the absorption pathlength (L_S', meter), sample pressure (P_S', kPa), absolute sample temperature T_S', and signal integration period (t_{SS}, seconds) for the analysis. Specify the nominal minimum instrumental linewidth (MIL) of the system. Verify that the fractional error at the approximate values P_S' and T_S' is less than one half the smallest value AU_i (see Section 4.1.2).

4.5 Select Calibration Transfer Standards (CTS's). Select CTS's that meet the criteria listed in Sections 4.5.1, 4.5.2, and 4.5.3.

> <u>Note</u>: It may be necessary to choose preliminary analytical regions (see Section 4.7), identify the minimum analyte linewidths, or estimate the system noise level (see Section 4.12) before selecting the CTS. More than one compound may be needed to meet the criteria; if so, obtain separate cylinders for each compound.

4.5.1 The central wavenumber position of each analytical region lies within 25 percent of the wavenumber position of at least one CTS absorption band.

4.5.2 The absorption bands in 4.5.1 exhibit peak absorbances greater than ten times the value RMS_{EST} (see Section 4.12) but less than 1.5 absorbance units.

4.5.3 At least one absorption CTS band within the operating range of the FTIR instrument has an instrument-independent linewidth no greater than the narrowest analyte absorption band; perform and document measurements or cite Studies to determine analyte and CTS compound linewidths.

4.5.4 For each analytical region, specify the upper and lower wavenumber positions (FFU_m and FFL_m, respectively) that bracket the CTS absorption band or bands for the associated analytical region. Specify the wavenumber range, FNU to FNL, containing the absorption band that meets the criterion of Section 4.5.3.

4.5.5 Associate, whenever possible, a single set of CTS gas cylinders with a set of reference spectra. Replacement CTS gas cylinders shall contain the same compounds at concentrations within 5 percent of that of the original CTS cylinders; the entire absorption spectra (not individual spectral segments) of the replacement gas shall be scaled by a factor between 0.95 and 1.05 to match the original CTS spectra.

4.6 Prepare Reference Spectra.

Note: Reference spectra are available in a permanent soft copy from the EPA spectral library on the EMTIC (Emission Measurement Technical Information Center) computer bulletin board; they may be used if applicable.

4.6.1 Select the reference absorption pathlength (L_R) of the cell.

4.6.2 Obtain or prepare a set of chemical standards for each analyte, potential and known spectral interferants, and CTS. Select the concentrations of the chemical standards to correspond to the top of the desired range.

4.6.2.1 Commercially-Prepared Chemical Standards. Chemical standards for many compounds may be obtained from independent sources, such as a specialty gas manufacturer, chemical company, or commercial laboratory. These standards (accurate to within ±2 percent) shall be prepared according to EPA Protocol 1 (see Reference D) or shall be traceable to NIST standards. Obtain from the supplier an estimate of the stability of the analyte concentration; obtain and follow all the supplier's recommendations for recertifying the analyte concentration.

4.6.2.2 Self-Prepared Chemical Standards. Chemical standards may be prepared as follows: Dilute certified commercially prepared chemical gases or pure analytes with ultra-pure carrier (UPC) grade nitrogen according to the barometric and volumetric techniques generally described in Reference A, Section A4.6.

4.6.3 Record a set of the absorption spectra of the CTS {R1}, then a set of the reference spectra at two or more concentrations in duplicate over the desired range (the top of the range must be less than 10 times that of the bottom), followed by a second set of CTS spectra {R2}. (If self-prepared standards are used, see Section 4.6.5 before disposing of any of the standards.) The maximum accepted standard concentration-pathlength product (ASCPP) for each compound shall be higher than the maximum estimated concentration-pathlength products for both analytes and known interferants in the effluent gas. For each analyte, the minimum ASCPP shall be no greater than ten times the concentration-pathlength product of that analyte at its required detection limit.

4.6.4 Permanently store the background and interferograms in digitized form. Document details of the mathematical process for generating the spectra from these interferograms. Record the sample pressure (P_R), sample temperature (T_R), reference absorption pathlength (L_R), and interferogram signal integration period (t_{SR}). Signal integration periods for the background interferograms shall be $\geq t_{SR}$. Values of P_R, L_R, and t_{SR} shall not deviate by more than ±1 percent from the time of recording {R1} to that of recording {R2}.

4.6.5 If self-prepared chemical standards are employed and spectra of only two concentrations are recorded for one or more compounds, verify the accuracy of the dilution technique by analyzing the prepared standards for those compounds with a secondary (non-FTIR) technique as follows:

4.6.5.1 Record the response of the secondary technique to each of the four standards prepared.

4.6.5.2 Perform a linear regression of the response values (dependant variable) versus the accepted standard concentration (ASC) values (independent variable), with the regression constrained to pass through the zero-response, zero ASC point.

4.6.5.3 Calculate the average fractional difference between the actual response values and the regression-predicted values (those calculated from the regression line using the four ASC values as the independent variable).

4.6.5.4 If the average fractional difference value calculated in Section 4.6.5.3 is larger for any compound than the corresponding AU_i, the dilution technique is not sufficiently accurate and the reference spectra prepared are not valid for the analysis.

4.7 Select Analytical Regions. Using the general considerations in Section 7 of Reference A and the spectral characteristics of the analytes and interferants, select the analytical regions for the application. Label them m = 1 to M. Specify the lower, center and upper wavenumber positions of each

analytical region (FL_m, FC_m, and FU_m, respectively). Specify the analytes and interferants which exhibit absorption in each region.

4.8 Determine Fractional Reproducibility Uncertainties. Using Appendix E, calculate the fractional reproducibility uncertainty for each analyte (FRU_i) from a comparison of {R1} and {R2}. If $FRU_i > AU_i$ for any analyte, the reference spectra generated in Section 4.6 are not valid for the application.

4.9 Identify Known Interferants. Using Appendix B, determine which potential interferant affects the analyte concentration determinations. If it does, relabel the potential interferant as "known" interferant, and designate these compounds from k = 1 to K. Appendix B also provides criteria for determining whether the selected analytical regions are suitable.

4.10 Prepare Computerized Analytical Programs.

4.10.1 Choose or devise mathematical techniques (e.g, classical least squares, inverse least squares, cross-correlation, and factor analysis) based on Equation 4 of Reference A that are appropriate for analyzing spectral data by comparison with reference spectra.

4.10.2 Following the general recommendations of Reference A, prepare a computer program or set of programs that analyzes all the analytes and known interferants, based on the selected analytical regions (4.7) and the prepared reference spectra (4.6). Specify the baseline correction technique (e.g., determining the slope and intercept of a linear baseline contribution in each analytical region) for each analytical region, including all relevant wavenumber positions.

4.10.3 Use programs that provide as output [at the reference absorption pathlength (L_R), reference gas temperature (T_R), and reference gas pressure (P_R)] the analyte concentrations, the known interferant concentrations, and the baseline slope and intercept values. If the sample absorption pathlength (L_S), sample gas temperature (T_S) or sample gas pressure (P_S) during the actual sample analyses differ from L_R, T_R, and P_R, use a program or set of programs that applies multiplicative corrections to the derived concentrations to account for these variations, and that provides as output both the corrected and uncorrected values. Include in the report of the analysis (see Section 7.0) the details of any transformations applied to the original reference spectra (e.g., differentiation), in such a fashion that all analytical results may be verified by an independent agent from the reference spectra and data spectra alone.

4.11 Determine the Fractional Calibration Uncertainty. Calculate the fractional calibration uncertainty for each analyte (FCU_i) according to Appendix F, and compare these values to the

fractional uncertainty limits (AU_i; see Section 4.1). If $FCU_i > AU_i$), either the reference spectra or analytical programs for that analyte are unsuitable.

4.12 Verify System Configuration Suitability. Using Appendix C, measure or obtain estimates of the noise level (RMS_{EST}, absorbance) of the FTIR system; alternatively, construct the complete spectrometer system and determine the values RMS_{Sm} using Appendix G. Estimate the minimum measurement uncertainty for each analyte (MAU_i, ppm) and known interferant (MIU_k, ppm) using Appendix D. Verify that (a) $MAU_i < (AU_i)(DL_i)$, $FRU_i < AU_i$, and $FCU_i < AU_i$ for each analyte and that (b) the CTS chosen meets the requirements listed in Section 4.5.

5.0 SAMPLING AND ANALYSIS PROCEDURE

5.1 Analysis System Assembly and Leak-Test. Assemble the analysis system. Allow sufficient time for all system components to reach the desired temperature. Then determine the leak-rate (L_R) and leak volume (V_L), where $V_L = L_R\ t_{SS}$. Leak volumes shall be ≤4 percent of V_{SS}.

5.2 Verify Instrumental Performance. Measure the noise level of the system in each analytical region using the procedure of Appendix G. If any noise level is higher than that estimated for the system in Section 4.12, repeat the calculations of Appendix D and verify that the requirements of Section 4.12 are met; if they are not, adjust or repair the instrument and repeat this section.

5.3 Determine the Sample Absorption Pathlength. Record a background spectrum. Then, fill the absorption cell with CTS at the pressure P_R and record a set of CTS spectra {R3}. Store the background and unscaled CTS single beam interferograms and spectra. Using Appendix H, calculate the sample absorption pathlength (L_S) for each analytical region. The values L_S shall not differ from the approximated sample pathlength L_S' (see Section 4.4) by more than 5 percent.

5.4 Record Sample Spectrum. Connect the sample line to the source. Either evacuate the absorption cell to an absolute pressure below 5 mmHg before extracting a sample from the effluent stream into the absorption cell, or pump at least ten cell volumes of sample through the cell before obtaining a sample. Record the sample pressure P_S. Generate the absorbance spectrum of the sample. Store the background and sample single beam interferograms, and document the process by which the absorbance spectra are generated from these data. (If necessary, apply the spectral transformations developed in Section 5.6.2). The resulting sample spectrum is referred to below as S_S.

Note: Multiple sample spectra may be recorded according to the procedures of Section 5.4 before performing Sections 5.5 and 5.6.

5.5 Quantify Analyte Concentrations. Calculate the unscaled analyte concentrations RUA_i and unscaled interferant concentrations RUI_K using the programs developed in Section 4. To correct for pathlength and pressure variations between the reference and sample spectra, calculate the scaling factor $R_{LPS} = (L_R P_R T_S)/(L_S P_S T_R)$. Calculate the final analyte and interferant concentrations $RSA_i = R_{LPS} RUA_i$ and $RSI_k = R_{LPS} RUI_k$.

5.6 Determine Fractional Analysis Uncertainty. Fill the absorption cell with CTS at the pressure P_S. Record a set of CTS spectra {R4}. Store the background and CTS single beam interferograms. Using Appendix H, calculate the fractional analysis uncertainty (FAU) for each analytical region. If the FAU indicated for any analytical region is larger than the required accuracy requirements determined in Section 4.1, then comparisons to previously recorded reference spectra are invalid in that analytical region, and the analyst shall perform one or both of the following procedures:

5.6.1 Perform instrumental checks and adjust the instrument to restore its performance to acceptable levels. If adjustments are made, repeat Sections 5.3, 5.4 (except for the recording of a sample spectrum), and 5.5 to demonstrate that acceptable uncertainties are obtained in all analytical regions.

5.6.2 Apply appropriate mathematical transformations (e.g., frequency shifting, zero-filling, apodization, smoothing) to the spectra (or to the interferograms upon which the spectra are based) generated during the performance of the procedures of Section 5.3. Document these transformations and their reproducibility. Do not apply multiplicative scaling of the spectra, or any set of transformations that is mathematically equivalent to multiplicative scaling. Different transformations may be applied to different analytical regions. Frequency shifts shall be smaller than one-half the minimum instrumental linewidth, and must be applied to all spectral data points in an analytical region. The mathematical transformations may be retained for the analysis if they are also applied to the appropriate analytical regions of all sample spectra recorded, and if all original sample spectra are digitally stored. Repeat Sections 5.3, 5.4 (except the recording of a sample spectrum), and 5.5 to demonstrate that these transformations lead to acceptable calculated concentration uncertainties in all analytical regions.

6.0 POST-ANALYSIS EVALUATIONS

Estimate the overall accuracy of the analyses performed in Section 5 as follows:

6.1 Qualitatively Confirm the Assumed Matrix. Examine each analytical region of the sample spectrum for spectral evidence of unexpected or unidentified interferants. If found, identify the interfering compounds (see Reference C for guidance) and add them to the list of known interferants. Repeat the procedures of Section 4 to include the interferants in the uncertainty calculations and analysis procedures. Verify that the MAU and FCU values do not increase beyond acceptable levels for the application requirements. Re-calculate the analyte concentrations (Section 5.5) in the affected analytical regions.

6.2 Quantitatively Evaluate Fractional Model Uncertainty (FMU). Perform the procedures of either Section 6.2.1 or 6.2.2:

6.2.1 Using Appendix I, determine the fractional model error (FMU) for each analyte.

6.2.2 Provide statistically determined uncertainties FMU for each analyte which are equivalent to two standard deviations at the 95% confidence level. Such determinations, if employed, must be based on mathematical examinations of the pertinent sample spectra (not the reference spectra alone). Include in the report of the analysis (see Section 7.0) a complete description of the determination of the concentration uncertainties.

6.3 Estimate Overall Concentration Uncertainty (OCU). Using Appendix J, determine the overall concentration uncertainty (OCU) for each analyte. If the OCU is larger than the required accuracy for any analyte, repeat Sections 4 and 6.

7.0 REPORTING REQUIREMENTS

[Documentation pertaining to virtually all the procedures of Sections 4, 5, and 6 will be required. Software copies of reference spectra and sample spectra will be retained for some minimum time following the actual testing.]

8.0 REFERENCES

A) Standard Practices for General Techniques of Infrared Quantitative Analysis (American Society for Testing and Materials, Designation E 168-88).

B) The Coblentz Society Specifications for Evaluation of Research Quality Analytical Infrared Reference Spectra (Class II); Anal. Chemistry 47, 945A (1975); **Appl. Spectroscopy 444, pp. 211-215, 1990.**

C) Standard Practices for General Techniques for Qualitative Infrared Analysis, American Society for Testing and Materials, Designation E 1252-88.

D) "Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Emissions Monitors (Protocol Number 1)," June 1978, Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/4-77-027b, August 1977.

APPENDIX A

DEFINITIONS OF TERMS AND SYMBOLS

A.1 Definitions of Terms

absorption band - a contiguous wavenumber region of a spectrum (equivalently, a contiguous set of absorbance spectrum data points) in which the absorbance passes through a maximum or a series of maxima.

absorption pathlength - in a spectrophotometer, the distance, measured in the direction of propagation of the beam of radiant energy, between the surface of the specimen on which the radiant energy is incident and the surface of the specimen from which it is emergent.

analytical region - a contiguous wavenumber region (equivalently, a contiguous set of absorbance spectrum data points) used in the quantitative analysis for one or more analyte.

Note: The quantitative result for a single analyte may be based on data from more than one analytical region.

apodization - modification of the ILS function by multiplying the

interferogram by a weighing function whose magnitude varies with retardation.

background spectrum - the single beam spectrum obtained with all system components without sample present.

baseline - any line drawn on an absorption spectrum to establish a reference point that represents a function of the radiant power incident on a sample at a given wavelength.

Beers's law - the direct proportionality of the absorbance of a compound in a homogeneous sample to its concentration.

calibration transfer standard (CTS) gas - a gas standard of a compound used to achieve and/or demonstrate suitable quantitative agreement between sample spectra and the reference spectra; see Section 4.5.1.

compound - a substance possessing a distinct, unique molecular structure.

concentration (c) - the quantity of a compound contained in a unit quantity of sample. The unit "ppm" (number, or mole, basis) is recommended.

concentration-pathlength product - the mathematical product of concentration of the species and absorption pathlength. For

reference spectra, this is a known quantity; for sample spectra, it is the quantity directly determined from Beer's law. The units "centimeters-ppm" or "meters-ppm" are recommended.

derivative absorption spectrum - a plot of rate of change of absorbance or of any function of absorbance with respect to wavelength or any function of wavelength.

double beam spectrum - a transmission or absorbance spectrum derived by dividing the sample single beam spectrum by the background spectrum.

Note: The term "double-beam" is used elsewhere to denote a spectrum in which the sample and background interferograms are collected simultaneously along physically distinct absorption paths. Here, the term denotes a spectrum in which the sample and background interferograms are collected at different times along the same absorption path.

fast Fourier transform (FFT) - a method of speeding up the computation of a discrete FT by factoring the data into sparse matrices containing mostly zeros.

flyback - interferometer motion during which no data are recorded.

Fourier transform (FT) - the mathematical process for converting an amplitude-time spectrum to an amplitude-frequency spectrum, or vice versa.

Fourier transform infrared (FTIR) spectrometer - an analytical system that employs a source of mid-infrared radiation, an interferometer, an enclosed sample cell of known absorption pathlength, an infrared detector, optical elements that transfer infrared radiation between components, and a computer system. The time-domain detector response (interferogram) is processed by a Fourier transform to yield a representation of the detector response vs. infrared frequency.

Note: When FTIR spectrometers are interfaced with other instruments, a slash should be used to denote the interface; e.g., GC/FTIR; HPCL/FTIR, and the use of FTIR should be explicit; i.e., FTIR not IR.

frequency, ν - the number of cycles per unit time.

infrared - the portion of the electromagnetic spectrum containing wavelengths from approximately 0.78 to 800 microns.

interferogram, $I(\sigma)$ - record of the modulated component of the interference signal measured as a function of retardation by the detector.

interferometer - device that divides a beam of radiant energy into two or more paths, generate an optical path difference between the beams, and recombines them in order to produce repetitive interference maxima and minima as the optical retardation is varied.

linewidth - the full width at half maximum of an absorption band in units of wavenumbers (cm^{-1}).

mid-infrared - the region of the electromagnetic spectrum from approximately 400 to 5000 cm^{-1}.

pathlength - see "absorption pathlength."

reference spectra - absorption spectra of gases with known chemical compositions, recorded at a known absorption pathlength, which are used in the quantitative analysis of gas samples.

retardation, σ - optical path difference between two beams in an interferometer; also known as "optical path difference" or "optical retardation."

scan - digital representation of the detector output obtained during one complete motion of the interferometer's moving assembly or assemblies.

scaling - application of a multiplicative factor to the absorbance values in a spectrum.

single beam spectrum - Fourier-transformed interferogram, representing the detector response vs. wavenumber.

Note: The term "single-beam" is used elsewhere to denote any spectrum in which the sample and background interferograms are recorded on the same physical absorption path; such usage differentiates such spectra from those generated using interferograms recorded along two physically distinct absorption paths (see "double-beam spectrum" above). Here, the term applies (for example) to the two spectra used directly in the calculation of transmission and absorbance spectra of a sample.

standard reference material - a reference material, the composition or properties of which are certified by a recognized standardizing agency or group.

Note: The equivalent ISO term is "certified reference material."

transmittance, T - the ratio of radiant power transmitted by the sample to the radiant power incident on the sample. Estimated in FTIR spectroscopy by forming the ratio of the single-beam sample and background spectra.

wavenumber, $\bar{v}$ - the number of waves per unit length.

Note: The usual unit of wavenumber is the reciprocal centimeter, cm^{-1}. The wavenumber is the reciprocal of the wavelength, λ, when λ is expressed in centimeters.

zero-filling - the addition of zero-valued points to the end of a measured interferogram.

Note: Performing the FT of a zero-filled interferogram results in correctly interpolated points in the computed spectrum.

A.2 Definitions of Mathematical Symbols

A, absorbance - the logarithm to the base 10 of the reciprocal of the transmittance (T).

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

AAI_{im} - band area of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i) .

AAV_{im} - average absorbance of the ith analyte in the mth analytical region, at the concentration (CL_i) corresponding to the product of its required detection limit (DL_i) and analytical uncertainty limit (AU_i) .

ASC, accepted standard concentration - the concentration value assigned to a chemical standard.

ASCPP, accepted standard concentration-pathlength product - for a chemical standard, the product of the ASC and the sample absorption pathlength. The units "centimeters-ppm" or "meters-ppm" are recommended.

AU_i, analytical uncertainty limit - the maximum permissible fractional uncertainty of analysis for the ith analyte concentration, expressed as a fraction of the analyte concentration determined in the analysis.

AVT_m - average estimated total absorbance in the mth analytical region.

$CKWN_k$ - estimated concentration of the kth known interferant.

$CMAX_i$ - estimated maximum concentration of the ith analyte.

$CPOT_j$ - estimated concentration of the jth potential interferant.

DL_i, **required detection limit** - for the ith analyte, the lowest concentration of the analyte for which its overall fractional uncertainty (OFU_i) is required to be less than the analytical uncertainty limit (AU_i).

FC_m - center wavenumber position of the mth analytical region.

FAU_i, **fractional analytical uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the mathematical comparison of reference and sample spectra.

FCU_i, **fractional calibration uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in Beer's law modeling of the reference spectra concentrations.

FFL_m - lower wavenumber position of the CTS absorption band associated with the mth analytical region.

FFU_m - upper wavenumber position of the CTS absorption band associated with the mth analytical region.

FL_m - lower wavenumber position of the mth analytical region.

FMU_i, **fractional model uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the absorption model employed.

FN_L - lower wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

FN_U - upper wavenumber position of the CTS spectrum containing an absorption band at least as narrow as the analyte absorption bands.

FRU_i, **fractional reproducibility uncertainty** - calculated uncertainty in the measured concentration of the ith analyte because of errors in the reproducibility of spectra from the FTIR system.

FU_m - upper wavenumber position of the mth analytical region.

IAI_{jm} - band area of the jth potential interferant in the mth analytical region, at its expected concentration ($CPOT_j$).

IAV_{im} - average absorbance of the ith analyte in the mth analytical region, at its expected concentration ($CPOT_j$).

$ISC_{i\ or\ k}$, indicated standard concentration - the concentration from the computerized analytical program for a single-compound reference spectrum for the ith analyte or k^{th} known interferant.

kPa - kilo-Pascal (see Pascal).

L_S' - estimated sample absorption pathlength.

L_R - reference absorption pathlength.

L_S - actual sample absorption pathlength.

MAU_i - mean of the MAU_{im} over the appropriate analytical regions.

MAU_{im}, minimum analyte uncertainty - the calculated minimum concentration for which the analytical uncertainty limit (AU_i) in the measurement of the ith analyte, based on spectral data in the mth analytical region, can be maintained.

MIU_j - mean of the MIU_{jm} over the appropriate analytical regions.

MIU_{jm}, minimum interferant uncertainty - the calculated minimum concentration for which the analytical uncertainty limit $CPOT_j/20$ in the measurement of the jth interferant, based on spectral data in the mth analytical region, can be maintained.

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

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 mth analytical region.

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

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

where:

n = the number of observations for which the accuracy is determined.

e_i = the difference between a measured value of a property and its mean value over the n observations.

<u>Note</u>: The RMSD value "between a set of n contiguous absorbance values (A_i) and the mean of the values" (A_M) is defined as

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

RSA_i - the (calculated) final concentration of the ith analyte.

RSI_k - the (calculated) final concentration of the kth known interferant.

t_{scan}, **scan time** - time used to acquire a single scan, not including flyback.

t_S, **signal integration period** - the period of time over which an interferogram is averaged by addition and scaling of individual scans. In terms of the number of scans N_{scan} and scan time t_{scan}, $t_S = N_{scan} t_{scan}$.

t_{SR} - signal integration period used in recording reference spectra.

t_{SS} - signal integration period used in recording sample spectra.

T_R - absolute temperature of gases used in recording reference spectra.

T_S - absolute temperature of sample gas as sample spectra are recorded.

TP, Throughput - manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

V_{SS} - *volume of the infrared absorption cell, including parts of* attached tubing.

W_{ik} - weight used to average over analytical regions k for quantities related to the analyte i; see Appendix D.

Note that some terms are missing, e.g., BAV_m, OCU, $RMSS_m$, SUB_S, SIC_i, SAC_i, S_S

APPENDIX B

IDENTIFYING SPECTRAL INTERFERANTS

B.1 General

B.1.1 Assume a fixed absorption pathlength equal to the value L_S'.

B.1.2 Use band area calculations to compare the relative absorption strengths of the analytes and potential interferants. In the mth analytical region (FL_m to FU_m), use either rectangular or trapezoidal approximations to determine the band areas described below (see Reference A, Sections A.3.1 through A.3.3); document any baseline corrections applied to the spectra.

B.1.3 Use the average total absorbance of the analytes and potential interferants in each analytical region to determine whether the analytical region is suitable for analyte concentration determinations.

> Note: The average absorbance in an analytical region is the band area divided by the width of the analytical region in wavenumbers. The average total absorbance in an analytical region is the sum of the average absorbances of all analytes and potential interferants.

B.2 Calculations

B.2.1 Prepare spectral representations of each analyte at the concentration $CL_i = (DL_i)(AU_i)$, where DL_i is the required detection limit and AU_i is the maximum permissible analytical uncertainty. For the mth analytical region, calculate the band area (AAI_{im}) and average absorbance (AAV_{im}) from these scaled analyte spectra.

B.2.2 Prepare spectral representations of each potential interferant at its expected concentration ($CPOT_j$). For the mth analytical region, calculate the band area (IAI_{jm}) and average absorbance (IAV_{jm}) from these scaled potential interferant spectra.

B.2.3 Repeat the calculation for each analytical region, and record the band area results in matrix form as indicated in Figure B.1.

B.2.4 If the band area of any potential interferant in an analytical region is greater than the one-half the band area of any analyte (i.e., $IAI_{jm} > 0.5\ AAI_{im}$ for any pair ij and any m), classify the potential interferant as known interferant. Label the known interferants k = 1 to K. Record the results in matrix form as indicated in Figure B.2.

B.2.5 Calculate the average total absorbance (AVT_m) for each analytical region and record the values in the last row of the matrix described in Figure B.2. Any analytical region where AVT_m >2.0 is unsuitable.

FIGURE B.1 Presentation of Potential Interferant Calculations

	Analytical Regions 1 . . . . . M
Analyte Labels	
1	AAI_{11} . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . AAI_{IM}
Potential Interferant Labels	
1	IAI_{11} . . . IAI_{1M}
.	. .
.	. .
J	IAI_{J1} . . . IAI_{JM}

FIGURE B.2 Presentation of Known Interferant Calculations

	Analytical Regions 1 . . . . . M
Analyte Labels	
1	AAI_{11} . . . . . AAI_{1M}
.	. .
.	. .
I	AAI_{I1} . . . . . AAI_{IM}
Known Interferant Labels	
1	IAI_{11} . . . . . IAI_{1M}
.	. .
.	. .
K	IAI_{K1} . . . . . IAI_{KM}
Total Average Absorbance	AVT_1 AVT_M

APPENDIX C

ESTIMATING NOISE LEVELS

C.1 General

C.1.1 The root-mean-square (RMS) noise level is the standard measure of noise in this Protocol. The RMS noise level of a contiguous segment of a spectrum is defined as the RMS difference (RMSD) between the absorbance values which form the segment and the mean value of that segment (see Appendix A).

C.1.2 The RMS noise value in double-beam absorbance spectra is assumed to be inversely proportional to: (a) the square root of the signal integration period of the sample single beam spectra from which it is formed, and (b) to the total infrared power transmitted through the interferometer and absorption cell.

C.1.3 Practically, the assumption of C.1.2 allow the RMS noise level of a complete system to be estimated from the following four quantities:

(a) RMS_{MAN} - the noise level of the system (in absorbance units), without the absorption cell and transfer optics, <u>under those conditions necessary to yield the specified minimum instrumental linewidth</u>, e.g., Jacquinot stop size.

(b) t_{MAN} - the manufacturer's signal integration time used to determine RMS_{MAN}.

(c) t_{SS} - the signal integration time for the analyses.

(d) TP - the manufacturer's estimate of the fraction of the total infrared power transmitted by the absorption cell and transfer optics from the interferometer to the detector.

C.2 Calculations

C.2.1 Obtain the values of RMS_{MAN}, t_{MAN}, and TP from the manufacturers of the equipment, or determine the noise level by direct measurements with the completely constructed system proposed in Section 4.

C.2.2 Calculate the noise value of the system (RMS_{EST}) as follows:

$$RMS_{EST} = RMS_{MAN} \; TP \sqrt{\frac{t_{ss}}{t_{MAN}}} \qquad (4)$$

APPENDIX D

ESTIMATING MINIMUM CONCENTRATION MEASUREMENT UNCERTAINTIES (MAU and MIU)

D.1 General

Estimate the minimum concentration measurement uncertainties for the ith analyte (MAU_i) and j^{th} interferant (MIU_j) based on the spectral data in the mth analytical region by comparing the analyte band area in the analytical region (AAI_{im}) and estimating or measuring the noise level of the system (RMS_{EST} or RMS_{Sm}).

Note: For a single analytical region, the MAU or MIU value is the concentration of the analyte or interferant for which the band area is equal to the product of the analytical region width (in wavenumbers) and the noise level of the system (in absorbance units). If data from more than one analytical region is used in the determination of an analyte concentration, the MAU or MIU is the mean of the separate MAU or MIU values calculated for each analytical region.

D.2 Calculations

D.2.1 For each analytical region, set RMS = RMS_{Sm} if measured (Appendix G), or set RMS = RMS_{EST} if estimated (Appendix C).

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

$$MAU_{im} = (RMS)(DL_i)(AU_i)\frac{(FU_m - FL_m)}{AAI_{im}} \qquad (5)$$

D.2.3 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $MAU_i = MAU_{im}$.

D.2.4 If a number of analytical regions are used to calculate the concentration of the ith analyte, set MAU_i equal to the weighted mean of the appropriate MAU_{im} values calculated above; the weight for each term in the mean is equal to the fraction of the total wavenumber range used for the calculation represented by each analytical region. Mathematically, if the set of analytical regions employed is {m'}, then the MAU for each analytical region is

$$MAU_i = \sum_{k \in \{m'\}} W_{ik} \; MAU_{ik} \tag{6}$$

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

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

D.2.5 Repeat Sections D.2.1 through D.2.4 to calculate the analogous values MIU_j for the interferants j = 1 to J. Replace the value $(AU_i)(DL_i)$ in the above equations with $CPOT_j/20$; replace the value AAI_{im} in the above equations with IAI_{jm}.

APPENDIX E

DETERMINING FRACTIONAL REPRODUCIBILITY UNCERTAINTIES (FRU)

E.1 General

To estimate the reproducibility of the spectroscopic results of the system, compare the CTS spectra recorded before and after preparing the reference spectra. Compare the difference between the spectra to their average band area. Perform the calculation for each analytical region on the portions of the CTS spectra associated with that analytical region.

E.2 Calculations

E.2.1 The CTS spectra {R1} consist of N spectra, denoted by S_{1i}, i=1, N. Similarly, the CTS spectra {R2} consist of N spectra, denoted by S_{2i}, i=1, N. Each S_{ki} is the spectrum of a single compound, where i denotes the compound and k denotes the set {Rk} of which S_{ki} is a member. Form the spectra S_3 according to $S_{3i} = S_{2i}-S_{1i}$ for each i. Form the spectra S_4 according to $S_{4i} = [S_{2i}+S_{1i}]/2$ for each i.

E.2.2 Each analytical region m is associated with a portion of the CTS spectra S_{2i} and S_{1i}, for a particular i, with lower and upper wavenumber limits FFL_m and FFU_m, respectively.

E.2.3 For each m and the associated i, calculate the band area of S_{4i} in the wavenumber range FFU_m to FFL_m. Follow the guidelines of Section B.1.2 for this band area calculation. Denote the result by BAV_m.

E.2.4 For each m and the associated i, calculate the RMSD of S_{3i} between the absorbance values and their mean in the wavenumber range FFU_m to FFL_m. Denote the result by $SRMS_m$.

E.2.5 For each analytical region m, calculate the quantity

$$FM_m = SRMS_m(FFU_m - FFL_m)/BAV_m$$

E.2.6 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FRU_i = FM_m$.

E.2.7 If a number p_i of analytical regions are used to calculate the concentration of the ith analyte, set FRU_i equal to the weighted mean of the appropriate FM_m values calculated above. Mathematically, if the set of analytical regions employed is {m'}, then

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

where the W_{ik} are calculated as described in Appendix D.

APPENDIX F

DETERMINING FRACTIONAL CALIBRATION UNCERTAINTIES (FCU)

F.1 General

F.1.1 The concentrations yielded by the computerized analytical program applied to each single-compound reference spectrum are defined as the indicated standard concentrations (ISC's). The ISC values for a single compound spectrum should ideally equal the accepted standard concentration (ASC) for one analyte or interferant, and should ideally be zero for all other compounds. Variations from these results are caused by errors in the ASC values, variations from the Beer's law (or modified Beer's law) model used to determine the concentrations, and noise in the spectra. When the first two effects dominate, the systematic nature of the errors is often apparent; take steps to correct them.

F.1.2 When the calibration error appears non-systematic, apply the following method to estimate the fractional calibration uncertainty (FCU) for each compound. The FCU is defined as the mean fractional error between the ASC and the ISC for all reference spectra with non-zero ASC for that compound. The FCU for each compound shall be less than the required fractional uncertainty specified in Section 4.1.

F.1.3 The computerized analytical programs shall also be required to yield acceptably low concentrations for compounds with ISC=0 when applied to the reference spectra. The limits chosen in this Protocol are that the ISC of each reference spectrum for each analyte or interferant shall not exceed that compound's minimum measurement uncertainty (MAU or MIU).

F.2 Calculations

F.2.1 Apply each analytical program to each reference spectrum. Prepare a similar table as that in Figure F.1 to present the ISC and ASC values for each analyte and interferant in each reference spectrum. Maintain the order of reference file names and compounds employed in preparing Figure F.1.

F.2.2 For all reference spectra in Figure F.1, verify that the absolute value of the ISC's are less than the compound's MAU (for analytes) or MIU (for interferants).

F.2.3 For each analyte reference spectrum, calculate the quantity (ASC-ISC)/ASC. For each analyte, calculate the mean of these values (the FCU_i for the ith analyte) over all reference spectra. Prepare a similar table as that in Figure F.2 to present the FCU_i and analytical uncertainty limit (AU_i) for each analyte.

FIGURE F.1

Presentation of Accepted Standard Concentrations (ASC's)
and Indicated Standard Concentrations (ISC's)

Compound Name	Reference Spectrum File Name	ASC (ppm)	ISC (ppm) Analytes i=1			Interferants 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 $\mathbf{A}_R$ containing the absorbance values from all segments of {R1} that are associated with the analytical regions; the members of the array are A_{Ri}, i = 1, n. Form a similar one-dimensional array $\mathbf{A}_S$ from the absorbance values in the spectral set {R3}; the members of the array are A_{Si}, i = 1, n. Based on the model $\mathbf{A}_S = r\mathbf{A}_R + \mathbf{E}$, determine the least-squares estimate of r', the value of r which minimizes the square error $\mathbf{E}^2$. Calculate the sample absorption pathlength $L_S = r'(T_S/T_R)L_R$.

H.2.2 Fractional Analysis Uncertainty. Perform and document separate linear baseline corrections to each analytical region in the spectral sets {R1} and {R4}. Form the arrays $\mathbf{A}_S$ and $\mathbf{A}_R$ as described in Section H.2.1, using values from {R1} to form $\mathbf{A}_R$, and values from {R4} to form $\mathbf{A}_S$. Calculate the values

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

and

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

The fractional analytical uncertainty is defined as

$$FAU = \frac{NRMS_E}{IA_{AV}} \tag{11}$$

APPENDIX I

DETERMINING FRACTIONAL MODEL UNCERTAINTIES (FMU)

I.1 General

To prepare analytical programs for FTIR analyses, the sample constituents must first be assumed; the calculations in this appendix, based upon a simulation of the sample spectrum, verify the appropriateness of these assumptions. The simulated spectra consist of the sum of single compound reference spectra scaled to represent their contributions to the sample absorbance spectrum; scaling factors are based on the indicated standard concentrations (ISC) and measured (sample) analyte and interferant concentrations, the sample and reference absorption pathlengths, and the sample and reference gas pressures. No band-shape correction for differences in the temperature of the sample and reference spectra gases is made; such errors are included in the FMU estimate. The actual and simulated sample spectra are quantitatively compared to determine the fractional model uncertainty; this comparison uses the reference spectra band areas and residuals in the difference spectrum formed from the actual and simulated sample spectra.

I.2 Calculations

I.2.1 For each analyte (with scaled concentration RSA_i), select a reference spectrum SA_i with indicated standard concentration ISC_i. Calculate the scaling factors

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

and form the spectra SAC_i by scaling each SA_i by the factor RA_i.

I.2.2 For each interferant, select a reference spectrum SI_k with indicated standard concentration ISC_k. Calculate the scaling factors

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

and form the spectra SIC_k by scaling each SI_k by the factor RI_k.

I.2.3 For each analytical region, determine by visual inspection which of the spectra SAC_i and SIC_k exhibit absorbance bands within the analytical region. Subtract each spectrum SAC_i

and SIC_k exhibiting absorbance from the sample spectrum S_S to form the spectrum SUB_S. To save analysis time and to avoid the introduction of unwanted noise into the subtracted spectrum, it is recommended that the calculation be made (1) only for those spectral data points within the analytical regions, and (2) for each analytical region separately using the original spectrum S_S.

I.2.4 For each analytical region m, calculate the RMSD of SUB_S between the absorbance values and their mean in the region FFU_m to FFL_m. Denote the result by $RMSS_m$.

I.2.5 For each analyte i, calculate the quantity

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

for each analytical region associated with the analyte.

I.2.6 If only the mth analytical region is used to calculate the concentration of the ith analyte, set $FMU_i = FM_m$.

I.2.7 If a number of analytical regions are used to calculate the concentration of the ith analyte, set FM_i equal to the weighted mean of the appropriate FM_m values calculated above. Mathematically, if the set of analytical regions employed is $\{m'\}$, then

$$FMU_i = \sum_{k \in \{m'\}} W_{ik} FM_k \tag{15}$$

where W_{ik} is calculated as described in Appendix D.

APPENDIX J

DETERMINING OVERALL CONCENTRATION UNCERTAINTIES (OCU)

The calculations in previous sections and appendices estimate the measurement uncertainties for various FTIR measurements. The lowest possible overall concentration uncertainty (OCU) for an analyte is its MAU value, which is an estimate of the absolute concentration uncertainty when spectral noise dominates the measurement error. However, if the product of the largest fractional concentration uncertainty (FRU, FCU, FAU, or FMU) and the measured concentration of an analyte exceeds the MAU for the analyte, then the OCU is this product. In mathematical terms, set $OFU_i = MAX\{FRU_i, FCU_i, FAU_i, FMU_i\}$ and $OCU_i = MAX\{RSA_i*OFU_i, MAU_i\}$.

APPENDIX K

SPECTRAL DE-RESOLUTION PROCEDURES

K.1 General.

High resolution reference spectra can be converted into lower resolution standard spectra for use in quantitative analysis of sample spectra. This is accomplished by truncating the number of data points in the original reference sample and background interferograms.

De-resolved spectra must meet the following requirements to be used in quantitative analysis.

(a) The resolution must match the instrument sampling resolution. This is verified by comparing a de-resolved CTS spectrum to a CTS spectrum measured on the sampling instrument.

(b) The Fourier transformation of truncated interferograms (and their conversion to absorbance spectra) is performed using the same apodization function (and other mathematical corrections) used in converting the sample interferograms into absorbance spectra.

K.2 Procedures

This section details three alternative procedures using two different commercially available software packages. A similar procedures using another software packages is acceptable if it is based on truncation of the original reference interferograms and the results are verified by Section K.3.

K.2.1 KVB/Analect Software Procedure - The following example converts a 0.25 cm^{-1} 100 ppm ethylene spectrum (cts0305a) to 1 cm^{-1} resolution. The 0.25 cm^{-1} CTS spectrum was collected during the EPA reference spectrum program on March 5, 1992. The original data (in this example) are in KVB/Analect FX-70 format.

(i) **decomp cts0305a.aif,0305dres,1,16384,1**

"decomp" converts cts0305a to an ASCII file with name 0305dres. The resulting ASCII interferogram file is truncated to 16384 data points. Convert background interferogram (bkg0305a.aif) to ASCII in the same way.

(ii) **compose 0305dres,0305dres.aif,1**

"Compose" transforms truncated interferograms back to spectral format.

(iii) **IG2SP 0305dres.aif,0305dres.dsf,3,1,low cm^{-1},high cm^{-1}**

"IG2SP" converts interferogram to a single beam spectrum using Norton-Beer medium apodization, 3, and no zero filling, 1. De-resolved interferograms should be transformed using the same apodization and zero filling that will be used to collect sample spectra. Choose the desired low and high frequencies, in cm^{-1}. Transform the background interferogram in the same way.

(iv) **DVDR 0305dres.dsf,bkg0305a.dsf,0305dres.dlf**

"DVDR" ratios the transformed sample spectrum against the background.

(v) **ABSB 0305dres.dlf,0305dres.dlf**

"ABSB" converts the spectrum to absorbance.

The resolution of the resulting spectrum should be verified by comparison to a CTS spectrum collected at the nominal resolution. Refer to Section K.3.

K.2.2 Alternate KVB/Analect Procedure -- In either DOS (FX-70) or Windows version (FX-80) use the "Extract" command directly on the interferogram.

(i) **EXTRACT CTS0305a.aif,0305dres.aif,1,16384**

"Extract" truncates the interferogram to data points from to 16384 (or number of data points for desired nominal resolution). Truncate background interferogram in the same way.

(ii) Complete steps (iii) to (v) in Section K.2.1.

K.2.3 Grams™ Software Procedure - Grams™ is a software package that displays and manipulates spectra from a variety of instrument manufacturers. This procedure assumes familiarity with basic functions of Grams™.

This procedure is specifically for using Grams to truncate and transform reference interferograms that have been imported into Grams from the KVB/Analect format. Table K-1 shows data files and parameter values that are used in the following procedure.

The choice of all parameters in the ICOMPUTE.AB call of step 3 below should be fixed to the shown values, with the exception of the "Apodization" parameter. This parameter should be set (for both background and sample single beam conversions) to the type of apodization function chosen for the de-resolved spectral library.

TABLE K-1. GRAMS DATA FILES AND DE-RESOLUTION PARAMETERS.

Desired Nominal Spectral Resolution (cm^{-1})	Data File Name	Parameter "N" Value
0.25	Z00250.sav	65537
0.50	Z00500.sav	32769
1.0	Z01000.sav	16385
2.0	Z02000.sav	8193

(i) **Import** using "File/Import" the desired *.aif file. Clear all open data slots.

(ii) **Open** the resulting *.spc interferogram as file #1.

(iii) **Xflip** - *If the x-axis is increasing from left to right, and the ZPD burst appears near the left end of the trace, omit this step.*

In the "Arithmetic/Calc" menu item input box, type the text below. Perform the calculation by clicking on "OK" (once only), and, when the calculation is complete, click the "Continue" button to proceed to step (iv). Note the comment in step (iii) regarding the trace orientation.

xflip:#s=#s(#0,#N)+50

(iv) **Run ICOMPUTE.AB** from "Arithmetic/Do Program" menu. Ignore the "subscripting error," if it occurs.

The following menu choices should be made before execution of the program (refer to Table K-1 for the correct choice of "**N**":)

First: **N** Last: **0** Type: **Single Beam**
Zero Fill: **None** Apodization: **(as desired)**
Phasing: **User**
Points: **1024** Interpolation: **Linear** Phase: **Calculate**

(v) **As** in step (iii), in the "**Arithmetic/Calc**" menu item enter and then run the following commands (refer to Table 1 for appropriate "**FILE,**" which may be in a directory other than "c:\mdgrams.")

setffp 7898.8805, 0 : loadspc "c:\mdgrams\ FILE" : #2=#s+#2

(vi) Use "**Page Up**" to activate file #2, and then use the "**File/Save As**" menu item with an appropriate file name to save the result.

K.3 Verification of New Resolution

K.3.1 Obtain interferograms of reference sample and background spectra. Truncate interferograms and convert to absorbance spectra of desired nominal resolution.

K.3.2 Document the apodization function, the level of zero filling, the number of data points, and the nominal resolution of the resulting de-resolved absorbance spectra. Use the identical apodization and level of zero filling when collecting sample spectra.

K.3.3 Perform the same de-resolution procedure on CTS interferograms that correspond with the reference spectra (reference CTS) to obtain de-resolved CTS standard spectra (CTS standards). Collect CTS spectra using the sampling resolution and the FTIR system to be used for the field measurements (test CTS). If practical, use the same pathlength, temperature, and standard concentration that were used for the reference CTS. Verify, by the following procedure that CTS linewidths and intensities are the same for the CTS standards and the test CTS.

K.3.4 After applying necessary temperature and pathlength corrections (document these corrections), subtract the CTS standard from the test CTS spectrum. Measure the RMSD in the resulting subtracted spectrum in the analytical region(s) of the CTS band(s). Use the following equation to compare this RMSD to the test CTS band area. The ratio in equation 7 must be no greater than 5 percent (0.05).

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

RMSS=RMSD in the ith analytical region in subtracted result, test CTS minus CTS standard.

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

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

$A_{test\text{-}CTS}$=band area in the ith analytical region of the test CTS.