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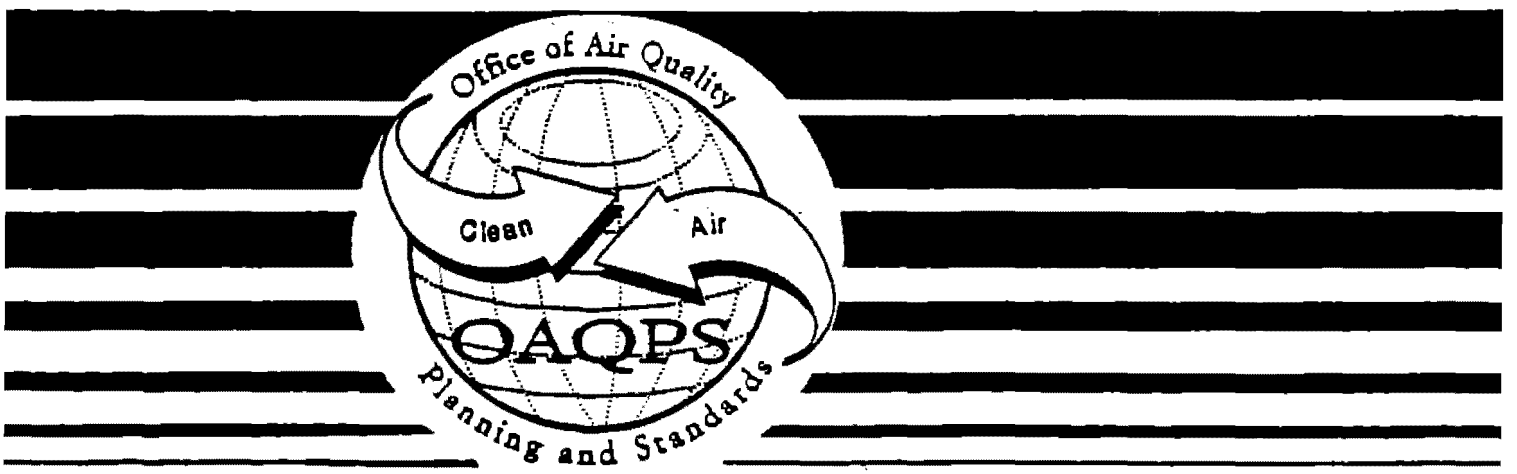
Research Triangle Park, NC 27711

EMB Report 91-ASP-10
September 1991

Air

ASPHALT
Emission Test Report

Mathy Construction Company
La Crosse, Wisconsin



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EMISSION TESTING FOR ASPHALT CONCRETE INDUSTRY

EMISSION TEST REPORT

Mathy Construction Company
Plant 6

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1. INTRODUCTION

Radian Corporation, under contract to the U.S. Environmental Protection Agency's (EPA) Emission Management Branch (EMB), has conducted a testing program to quantify the emissions of criteria and other air pollutants from the Mathy Construction Company's Facility No. 6, a batch-mix asphalt paving plant located in LaCrosse, Wisconsin. The results of this testing will become part of an updated emission factors database, which will be used by the EPA's Emission Inventory Branch (EIB) to update the asphaltic concrete plant section of the *Compilation of Air Pollutant Emission Factors*, an EPA document commonly referred to as AP-42.

The specific pollutants of interest in the testing program were the following air pollutants: particulate matter (PM); PM less than 10 μm (PM_{10}); condensible PM (CPM); sulfur dioxide (SO_2); nitrogen oxides (NO_x); carbon monoxide (CO); total hydrocarbons (THC); polynuclear aromatic hydrocarbons (PAH), excluding aldehydes and ketones; and trace metals, excluding mercury (Hg). Mathy's Facility No. 6 was selected as one of the two asphaltic concrete plants studied for the revision of AP-42 because it was judged by EMB and the National Asphalt Paving Association (NAPA) to be representative of the processes, equipment configuration, and production rate of batch-mix asphalt paving plants currently in use in the United States.

Testing was performed on September 19 through 20, 1991, and the principal objectives of testing were the following:

- Determine levels of CO, SO_2, NO_x, and THC emitted from the plant stack.
- Determine the levels of toxic metals being emitted from the stack including lead (Pb), chromium (Cr), cadmium (Cd), beryllium (Be), thallium (Tl), arsenic (As), nickel (Ni), antimony (Sb), barium (Ba), silver (Ag), zinc (Zn), phosphorus (P), copper (Cu), manganese (Mn), and selenium (Se). The Hg levels were not analyzed because it was not expected to be present in the process stream.
- Determine the filterable PM_{10} and CPM fractions emitted from the stack.
- Determine the levels of PAHs emitted from the stack.

- Determine the quantities of benzene, toluene, xylene, and methane present in the stack exhaust gas.

- Monitor the process operating conditions including aggregate flow rate, moisture, and ambient moisture. Also, determine the fuel high-heating value, and ultimate analysis.

In order to ensure repeatability of results, the measurements above were repeated in triplicate at near-design operating conditions while the plant was operating on natural gas.

The concentrations of CO_2, O_2, NO_x, SO_2, and CO in the flue gases were determined using continuous emissions monitoring (CEM) systems designed in accordance with EPA Methods 3A, 7E, 6C, and 10, respectively. Emissions of THC were determined by CEM following EPA Method 25A. The EPA's Method 18 was followed in the gas chromatography (GC) analysis for flue gas concentrations of benzene, toluene, xylene, and methane. Samples of PM and metals were collected during three sampling train runs, performed according to EPA Method 5/Combined Train SW 846 Test Method 0031. The EPA SW 846 Test Method 0010 and 0011 were used in the collection of PAHs and speciated THCs, respectively. PM_{10} and CPM were sampled by means of three test runs following a combination of protocols outlined in EPA Methods 201A and 202, respectively. All of the manual method flue gas samples were analyzed in Radian's Perimeter Park laboratory facility in Morrisville, North Carolina.

1.1 BACKGROUND

The EIB is the branch of the EPA's Office of Air Quality Planning and Standards (OAQPS) that is responsible for developing and maintaining air pollution emission factors for a variety of industrial processes. The EPA publishes these emission factor data for industrial processes--including asphaltic concrete production--in AP-42, the *Compilation of Air Pollutant Emission Factors*. This testing program is a part of ongoing efforts to keep AP-42 up to date.

The part of AP-42 that deals with asphaltic concrete plants was last revised in October of 1986. It divided conventional asphaltic concrete plants into two major types, based on the procedure used to mix the asphaltic concrete. One type is the

continuous-mix plant and the other is the batch-mix plant. Following this precedent and the recommendation of NAPA, EPA instructed Radian to direct its testing efforts at these two types of plants. The testing program described in this report is one of two testing programs being conducted by Radian in support of efforts to revise AP-42's asphaltic concrete section. The other emissions tests were conducted on Mathy Construction's Facility No. 26, a continuous-mix asphalt paving plant. Mathy Facility No. 6, the plant discussed in this report, is a batch-mix plant. The plants owned and operated by Mathy Construction Company have been recommended by NAPA to be representative of both types of asphalt paving plants in the United States.

1.2 BRIEF PROCESS AND SITE DESCRIPTION

In general, batch-mix plants operate in the following manner. The cold feed materials, known as aggregate, are sorted by size and stored in a series of bins that each feed aggregate to a common conveyor belt. The aggregate is then dispensed from the bins in a mix that achieves the desired aggregate size distribution and weight for the batch of asphaltic concrete. The conveyor carries the aggregate to a rotating drum, where it is mixed and dried at approximately 300°F. The dried aggregate is then carried to a gradation control unit, which separates and stores the aggregate by size. The necessary amount of each size of aggregate is then dropped into a weigh hopper and then into a pug mill, where is it thoroughly mixed with hot liquid asphalt. The hot mix is then transferred to storage silos, from which it is dispensed into paving trucks. Figure 1-1 is a diagram of a generic batch-mix asphalt plant.

The burner in the drum dryer is natural gas fired. The air from this process is drawn into the system by an exhaust fan located on the baghouse. After it passes through the burner and mixing drum, the air passes through the baghouse. The air is discharged to the atmosphere from a stack connected to the baghouse.

The stationary batch plant at Mathy Facility No. 6 has been rated at a 300 ton-per-hour production rate. Several factors affect this production rate. As a stationary plant, Facility No. 6 operates only when there is a demand for asphalt. If paving is stalled or slowed, the plant production must respond accordingly. Paving and plant operation are also shut down during rainy periods. In addition, the burner for

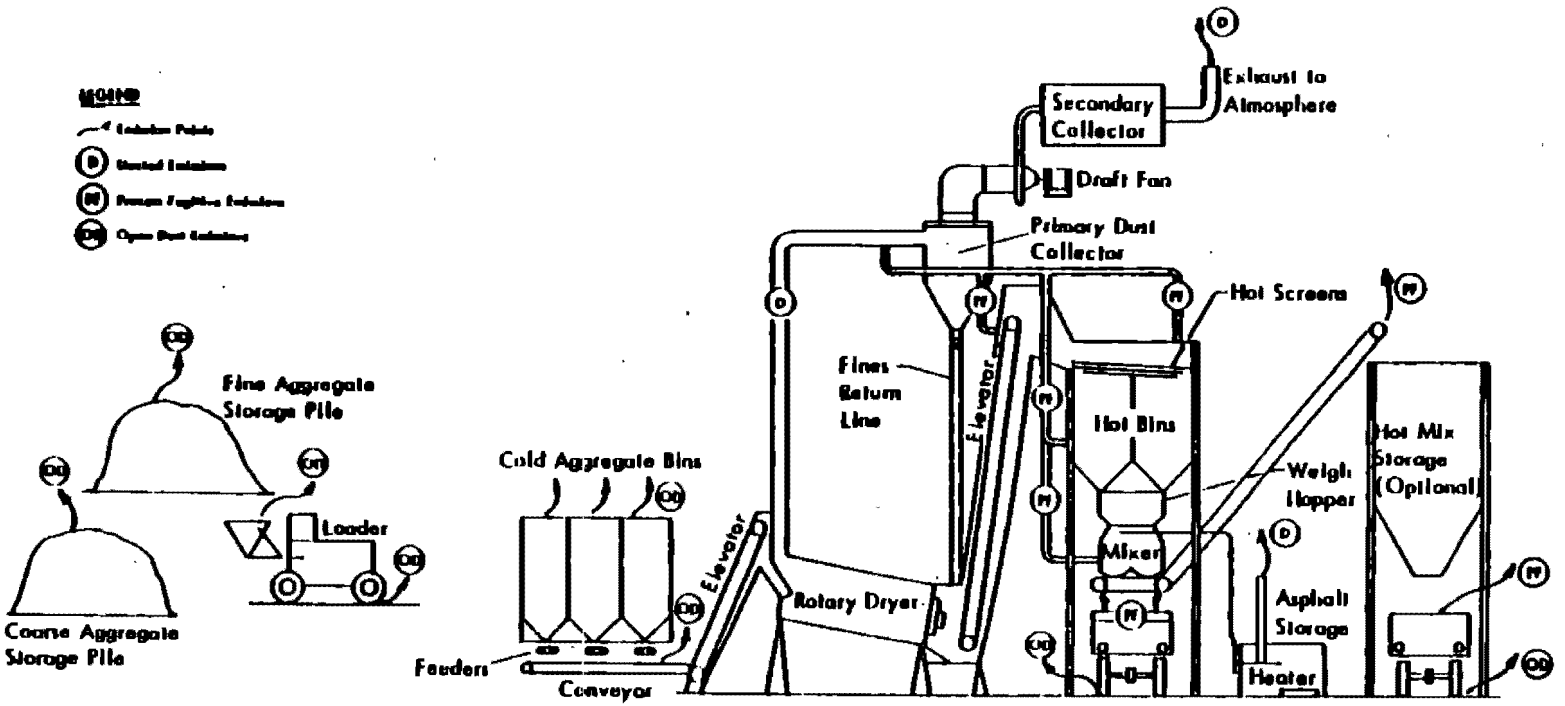

Figure 1-1. General Process Flow Diagram for Batch-Mix Asphalt Plants

drying the aggregate must be fired at a level suited to the moisture content and composition of the aggregate being used.

The rest of this report is structured as follows. Section 2 contains a summary of the test results. The process is discussed in Section 3, and the sample locations and sampling and analytical procedures are presented in Sections 4 and 5, respectively. Section 6 presents results of implementing the the quality assurance/quality control (QA/QC) procedures followed in the test program. Section 7 contains references used in developing this report. Appendices to this report include detailed methods and procedures, field and laboratory data, and complete calculations used in deriving the results presented here.

--	Benzaldehyde	=	1.41×10^{-4} lb/ton of product;
--	Butyaldehyde/ Isobutyraldehyde	=	0.428×10^{-4} lb/ton of product;
--	Crotonaldehyde	=	0.358×10^{-4} lb/ton of product;
--	Formaldehyde	=	19.4×10^{-4} lb/ton of product;
--	Hexanal	=	0.201×10^{-4} lb/ton of product;
--	Methyl ethyl ketone	=	0.701×10^{-4} lb/ton of product;
--	Propionaldehyde	=	0.710×10^{-4} lb/ton of product;
--	Quinone	=	4.05×10^{-4} lb/ton of product;
--	Valeraldehyde	=	0.427×10^{-4} lb/ton of product.

The following sections present more detailed summaries of the results of this test program.

2.1 EMISSIONS TEST LOG

Emissions testing was conducted over a two-day period from September 19 to September 20, 1991. Table 2-1 shows the emissions test log. This table shows the test date, sample location, run number, test type, run times, and average production rate during testing. Testing was performed using EPA manual test methods for six different types of substances. Testing was conducted in triplicate for each type of analyte.

Particulate matter and metals were sampled in the same sampling train by employing a combination of EPA Method 5 and EPA SW 846 Test Method 0031. Particulate matter was determined gravimetrically from the front half filter catch then combined with the back half for total metals analysis.

The PAHs was sampled concurrently with the PM and metals by EPA SW 846 Test Method 0010 using a dual probe arrangement. The dual probe arrangement allowed both trains to operate side-by-side with their nozzles in approximately the same sample location.

Sampling for aldehydes was conducted in a separate train using EPA SW 846 Test Method 0011.

Testing for PM_{10} and CPM was performed in a single train employing a combination of EPA Method 201A and EPA Method 202. An in-stack cyclone with a backup filter composed the front half of this train. The cyclone captured PM greater than 10 microns. The backup filter caught PM of 10 microns or less. The CPM was caught in the back half impingers.

Table 2-1. Emissions Test Log

Date	Location	Run Number	Test Type	Run Time	Average Production Rate (tph)
09/19/91	Stack[a]	1	PAH	07:12-09:23	245
09/19/91	Stack	2		13:28-15:40	244
09/20/91	Stack	3		09:45-12:03	222
09/19/91	Stack	1	PM	07:12-09:23	245
09/19/91	Stack	2		13:28-15:40	244
09/20/91	Stack	3		09:45-12:03	222
09/19/91	Stack	1	Metals	07:12-09:23	245
09/19/91	Stack	2		13:28-15:40	244
09/20/91	Stack	3		09:45-12:03	222
09/19/91	Stack	1	PM_{10}/CPM	10:36-11:55	224
09/20/91	Stack	2		11:27-12:52	213
09/20/91	Stack	3		14:13-15:24	215
09/19/91	Stack	1	Aldehydes	09:06-09:32 10:35-11:17	229
09/20/91	Stack	2		07:45-08:25 09:38-10:05	284
09/20/91	Stack	3		13:37-13:41 14:13-15:24	194
09/19/91	Stack	M-1	O_2/CO_2, SO_2, NO_x, THC	08:15-09:23	245
09/19/91	Stack	M-2		13:41-15:40	244
09/20/91	Stack	M-3		10:11-12:03	222
09/19/91	Stack	P-1		11:25-11:55	224
09/20/91	Stack	P-2		11:27-12:52	213
09/20/91	Stack	P-3		14:15-15:24	215

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Table 2-1, continued

Date	Location	Run Number	Test Type	Run Time	Average Production Rate (tph)
09/19/91	Stack	A-1	O_2/CO_2, SO_2, NO_x, THC	09:06-09:23	229
09/20/91	Stack	A-2		07:45-08:10 08:48-10:05	284
09/20/91	Stack	A-3		14:15-1:24	194
09/19/91	Stack	1	Benzene, Toluene, Ethylbenzene, Xylene, Methane	08:36[b]	245
09/19/91	Stack	2		13:41	244
09/19/91	Stack	3		14:25	244
09/19/91	Stack	4		15:05	244
09/19/91	Stack	5		15:36	244
09/20/91	Stack	6		08:01	222
09/20/91	Stack	7		08:48	222
09/20/91	Stack	8		09:52	222
09/20/91	Stack	9		10:25	222
09/20/91	Stack	10		10:55	222
09/20/91	Stack	11		11:51	222
09/20/91	Stack	12		12:46	213
09/20/91	Stack	13		14:21	215
09/20/91	Stack	14		14:51	215
09/20/91	Stack	15		15:20	215

[a]After baghouse exit and ID fan.
[b]Exact times GC samples were shot. Individual results were averaged over the time period during which manual sampling was conducted.

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2. SUMMARY OF RESULTS

This section provides results of the emission test program conducted at Mathy Construction Company's Plant 6 from September 19 to September 20, 1991. Included in this section are results of manual tests conducted for trace metals excluding Hg, PM, PM_{10}, CPM, aldehydes and ketones, and PAHs. This section also contains the results of the continuous emissions monitoring for CO_2/O_2, CO, SO_2, NO_x, and THC gases as well as gas chromatography (GC) results for benzene, toluene, xylene, and methane.

Stated briefly, the significant emissions from Mathy Construction's Plant 6 are as follows:

- Of the 15 metals analyzed, 9 were found in detectable quantities. Their emission rates are:

  -- Ba = 1.23×10^{-6} lb/ton of product;
  -- Cd = 0.351×10^{-6} lb/ton of product;
  -- Cr = 0.810×10^{-6} lb/ton of product;
  -- Cu = 1.86×10^{-6} lb/ton of product;
  -- Pb = 1.02×10^{-6} lb/ton of product;
  -- Mn = 11.8×10^{-6} lb/ton of product;
  -- Ni = 6.39×10^{-6} lb/ton of product;
  -- Ag = 0.621×10^{-6} lb/ton of product;
  -- Zn = 6.29×10^{-6} lb/ton of product.

  These averages include Run 2. (Run 2 void)

- Of the 19 PAHs analyzed, 2 were found in detectable levels. Their average emission rates are:

  -- 2-Methylnaphthalene = 0.117×10^{-4} lb/ton of product;
  -- Naphthalene = 0.320×10^{-4} lb/ton of product.

- For PM and PM_{10} (actually PM_8; see explanation in Section 2.4.1), the average emission rates are:

  -- PM = 0.005 lb/ton of product;
  -- PM_{10} = 0.008 lb/ton of product.

- Of the 18 aldehydes analyzed, 11 were found in detectable quantities. Their average emission rates are:

  -- Acetaldehyde = 6.92×10^{-4} lb/ton of product;
  -- Acetone = 105×10^{-4} lb/ton of product;

Eleven other analytes were sampled for by a combination of CEM and GC instruments. These tests were conducted concurrent with the manual method tests to the extent possible. Continuous emissions monitoring was operated continuously and results were averaged over the manual test period in which they were performed. Gas chromatography measurements were taken on a semicontinuous basis and multiple readings were averaged over the manual test period in which they were performed.

2.2 METAL AND POLYNUCLEAR AROMATIC HYDROCARBON RESULTS

2.2.1 Overview

The PM/metals and PAH manual sampling trains shared a dual-probe arrangement, which allowed testing to be conducted simultaneously in separate trains at the same port location. The PM/metals sampling train was used to determine emission rates of 15 metals (Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, P, Se, Ag, Tl, and Zn) and PM. The PAH sampling train was used to quantify emission rates of 19 PAHs (acenaphthylene, ancenaphthene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perlyene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, dibenzofuran, 7.12-dimethylbenz(a)anthracene, fluoranthene, florene, indeno(1,2,3-cd)pyrene, 2-methylnaphthalene, naphthalene, phenanthrene, and pyrene).

Three manual sampling runs for PM/metals and PAHs were performed to ensure representative test results. The back half sample bottle from Run 2 was broken during shipment, so only the front half was analyzed for PM and metal content.

Section 2.2 presents process operations. The average emission rates in μg/dscm, corrected to 7 percent O_2, and g/hr are summarized in Section 2.2.3. The metals-to-PM ratios are presented in Section 2.2.4, and flue gas-by-sample fractions are presented in Section 2.2.5.

2.2.2 Process Operation

Table 2-2 summarizes the metals and PAH emission factor results with the process operating data. Sampling was performed in two separate trains simultaneously using a unique dual-probe arrangement. Total asphaltic concrete production varied between 222 tons/hr and 245 tons/hr (73 to 82 percent of capacity), while natural gas

Table 2-2

SUMMARY OF METALS/PM AND PAH EMISSION FACTORS AND PROCESS OPERATING DATA

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run Number		1	2	3	Average
Date		09/19/91	09/19/91	09/20/91	
Production Rate	(tons/hr)	245	244	222	237
Virgin Asphalt Rate	(tons/hr)	230.8	229.8	209.1	223.2
Recycled Asphalt Rate	(tons/hr)	0	0	0	0
Asphalt Cement Rate	(tons/hr)	14.2	14.2	12.9	13.8
Percent of Rated Capacity	(%)	82	81	73	79
Aggregate Moisture	(%)	4.3	4.3	4.4	4.3
Fuel Flow	(cubic foot/hr)	5250	5280	5190	5240
Heat Imput Rate	(1000 cubic foot/hr)	5.25	5.28	5.19	5.24
Burner Setting - Flame Meter	(%)	78	80	81	80
Ambient Temperature	(degree F)	43	51	53	49
Ambient Humidity	(%)	85	54	70	70
Kiln Exit Temperature	(degree F)	339	341	349	343
Stack Flow Rate	(dscfm)	34280	32660	31570	32840
Stack Flow Rate	(dscf/ton of product)	8390	8030	8530	8320
Stack Temperatrue	(degree F)	240	236	244	240
Stack Moisture	(% volume)	27.5	28.4	28.1	28.0
Stack CO2	(volume % dry)	5.7	5.8	5.2	5.6
Stack O2	(volume % dry)	14.1	12.5	14.2	13.6
Stack CO	(ppmV)	> 1000	> 1000	1362	1362
Control Device		Baghouse	Baghouse	Baghouse	
Barium	(lbs x 10(-6)/ton of product) *	1.27	0.785	1.64	1.23
Cadmium	(lbs x 10(-6)/ton of product) *	0.477	ND	0.225	0.351
Chromium	(lbs x 10(-6)/ton of product) *	1.47	ND	0.151	0.810
Copper	(lbs x 10(-6)/ton of product) *	1.82	ND	1.90	1.86
Lead	(lbs x 10(-6)/ton of product) *	0.150	ND	1.90	1.02
Manganese	(lbs x 10(-6)/ton of product) *	16.0	7.05	12.4	11.8
Nickel	(lbs x 10(-6)/ton of product) *	12.5	ND	0.246	6.39
Silver	(lbs x 10(-6)/ton of product) *	0.862	0.381	ND	0.621
Zinc	(lbs x 10(-6)/ton of product) *	4.69	ND	7.90	6.29
Particulate Matter	(lbs/ton of product)	0.00690	0.00386	0.00500	0.00595
2-Methylnaphthalene	(lbs x 10(-4)/ton of product) **	0.116	0.100	0.133	0.117
	(lbs x 10 (-4)/1000 cubic foot) **	5.42	4.63	5.69	5.25
Naphthalene	(lbs x 10(-4)/ton of product) **	ND	ND	0.320	0.320
	(lbs x 10 (-4)/1000 cubic foot) **	ND	ND	13.7	13.7

ND = Not Detected

* = 1 x 10(-6) or (0.000001)

** = 1 x 10(-4) or (0.0001)

NOTE = Run averages were calculated from readings taken periodically throughout the duration of the emission test run.

See Table 3-1 and 3-2 for the individual readings.

Metals and PAH compounds analyzed, but not detected, are not included in this table.

Metals/PAH concentrations have been blank corrected.

consumption ranged from 5190 ft^3/hr to 5280 ft^3/hr. Metals emissions from this facility are generally associated with aggregate processing because natural gas is used exclusively in the kiln. Facilities that alternate fuels may realize different or increased metal emission factors because of the chemical composition of fuel oils, which can be burned in similar kilns. Therefore, these results are only applicable to natural gas-fired kilns and not fuel oil systems.

The PAH emissions from asphaltic concrete plants may originate from fuel combustion; the volatile fraction of the asphalt cement, if any; and organic residues commonly found in recycled asphalt (i.e., gasoline, engine oils). No recycled asphalt was processed during these tests, nor were data found in the literature indicating the contribution of PAH from asphalt cement. Therefore, the emission factors presented are expressed in lb/ton of product and lb/ft^3 of natural gas consumed. The former units allow for an unknown volatile fraction to be present in the asphalt cement, whereas the latter units assume no volatile fraction in the asphalt cement and that all PAHs result from fuel combustion in the kiln. In either case, the emission factors do not apply to product containing recycled asphalt. During the emission tests, the plant was operating at the normal plant capacity, with Run 1 at 82 percent, Run 2 at 81 percent, and Run 3 at 73 percent. This allows a comparison of the emission tests during the three runs. The production rates were 245 tons/hr, 244 ton/hr, and 222 tons/hr for Runs 1, 2, and 3, respectively.

Only the metals and PAHs detected are given in Table 2-2. The other metals and PAHs were analyzed, but they were not collected in detectable amounts. Nine metals of the 15 analyzed were detected (Ba, Cd, Cr, Cu, Pb, Mn, Ni, Ag, and Zn). Only 2 of the 22 PAHs analyzed were detected. These were 2-methylnaphthalene and naphthalene. The PAHs detected were blank corrected and reported as shown.

2.2.3 Emissions

Metals

Table 2-3 presents the metals emissions results for the test conditions. Also shown for each run are the date, metered volume (in dscm), O_2 concentration, and flue gas flow rate. Flue gas concentrations are given in terms of μg/dscm and μg/dscm

Table 2-3

METALS CONCENTRATION EMISSION RATES

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/19/91	09/19/91	09/20/91	
Metered Volume (dscm)		1.752	1.677	1.631	
O2 Concentration (%V)		14.15	12.53	14.27	
Volumetric Flow Rate (dscm/m)		947.928	985.721	880.342	AVERAGE
Barium	(ug/dscm)	2.48	1.60	3.13	2.40
	(ug/dscm @ 7% O2)	5.11	2.65	6.57	4.78
	(g/hr)	0.141	0.0868	0.165	0.131
Cadmium	(ug/dscm)	0.932	ND	0.429	0.680
	(ug/dscm @ 7% O2)	1.92	ND	0.900	1.41
	(g/hr)	0.0530	ND	0.0227	0.0378
Chromium	(ug/dscm)	2.87	ND	0.288	1.58
	(ug/dscm @ 7% O2)	5.91	ND	0.603	3.26
	(g/hr)	0.163	ND	0.0152	0.089
Copper	(ug/dscm)	3.56	ND	3.63	3.59
	(ug/dscm @ 7% O2)	7.32	ND	7.61	7.47
	(g/hr)	0.202	ND	0.192	0.197
Lead	(ug/dscm)	0.293	ND	3.62	1.95
	(ug/dscm @ 7% O2)	0.603	ND	7.58	4.09
	(g/hr)	0.0167	ND	0.191	0.104
Manganese	(ug/dscm)	31.2	14.4	23.7	23.1
	(ug/dscm @ 7% O2)	64.2	23.8	49.7	45.9
	(g/hr)	1.77	0.780	1.25	1.27
Nickel	(ug/dscm)	24.5	ND	0.469	12.5
	(ug/dscm @ 7% O2)	50.4	ND	0.983	25.7
	(g/hr)	1.39	ND	0.0248	0.709
Silver	(ug/dscm)	1.68	0.775	ND	1.23
	(ug/dscm @ 7% O2)	3.47	1.29	ND	2.38
	(g/hr)	0.0958	0.0421	ND	0.0689
Zinc	(ug/dscm)	9.17	ND	15.1	12.1
	(ug/dscm @ 7% O2)	18.9	ND	31.6	25.2
	(g/hr)	0.521	ND	0.795	0.658

ND = Not Detected

NOTE: Run 2 impinger sample bottle broke during shipment. Therefore, only the front half is recorded. The average is based on Runs 1 and 3.

Metals analyzed, but not detected, are not included in this table.

corrected to 7 percent O_2. Oxygen concentrations were determined from CEM data (see Section 2.6). It should be noted that Run 2 results are from the analysis of the front half only because of sample breakage. Therefore, Run 2 was not used in averaging the runs.

During the emission tests, Mn had the highest average mass rate with 1.27 g/hr, followed by Ni with 0.709 g/hr. These emission rates correspond to an average emission factor of 11.8 x 10^{-6} lb/ton of product and 6.39 x 10^{-6} lb/ton of product. After blank correction, Sb, As, Be, P, Se, and Tl were not collected in detectable amounts for any of the runs during these emission tests. Metal values ranged from 1.77 g/hr of Mn in Run 1, to 0.0152 g/hr of Cr in Run 3.

The metal values for the emission tests are not significantly different between Runs 1 and 3, except for Cd, Pb, and Ni. Cadmium fluctuated from 0.0690 to 0.104 g/hr. Lead fluctuated from 0.0911 g/hr to 0.259 g/hr. Nickel fluctuated from 0.169 to 1.53 g/hr.

Polynuclear Aromatic Hydrocarbons

Table 2-4 presents the PAH emission results for three test runs. Also shown for each run are the date, metered volume, O_2 concentration, and flow rate. Flue gas concentrations are given in terms of g/dscm and g/dscm corrected to 7 percent O_2. Oxygen concentrations were collected from CEM data.

During the emission tests, naphthalene had the highest average mass rate with 23.2 g/hr, followed by 2-methylnaphthalene with 12.5 g/hr. These emission rates correspond to average emission factors of 13.7 x 10^{-4} lb/1000 ft^3 of natural gas and 5.25 x 10^{-4} lb/1000 ft^3 of natural gas, respectively. It should be noted that these compounds may be a degradation by-product of the XAD absorbent used in the sample train. However, these results have been blank corrected and are reported as shown. The other compounds listed were not collected in detectable amounts for any of these emission tests. The PAH values ranged from 23.2 g/hr of naphthalene in Run 3 to 11.1 g/hr of 2-methylnaphthalene in Run 2.

The PAH values for the emission tests did not change significantly from Runs 1 to 3, except for naphthalene, which was detected in Run 3, but not in Runs 1 or 2.

Table 2-4

PAH CONCENTRATION EMISSION RATES

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/19/91	09/19/91	09/20/91	
Metered Volume (dscm)		1.840	1.745	1.665	
O2 Concentration (%V)		14.15	12.53	14.27	
Volumetric Flow Rate (dscmm)		970.660	924.776	894.065	AVERAGE
2-Methylnaphthalene	(ug/dscm)	222	199	250	224
	(ug/dscm @ 7% O2	456	331	525	437
	(g/hr)	12.9	11.1	13.4	12.5
Naphthalene	(ug/dscm)	ND	ND	432	432
	(ug/dscm @ 7% O2	ND	ND	905	905
	(g/hr)	ND	ND	23.2	23.2

ND = Not Detected

NOTE: Concentrations given have been blank corrected. PAH compounds analyzed, but not detected, are not included in this table.

2.2.4 Ratios of Flue Gas Metals to Particulate Matter

A summary of the ratios of metals to PM for the emission tests is presented in Table 2-5. Metals-to-PM ratios are given in units of milligrams of metals per gram of PM collected by the sampling train. The values ranged from 0.0217 mg of Pb per gram of PM during Run 1 to 2.49 mg of Mn per gram of PM during Run 3. Manganese had the highest ratio for Run 1 with 2.31 mg metal/gram PM.

2.2.5 Flue Gas Sampling Fraction and Sample Parameters

Metals

Table 2-6 presents the metal amounts in the flue gas samples by fraction for the emission tests. All metals detected were collected in the highest proportions in the front half (filter, nozzle/probe rinse), except for Cu, which was collected in the highest proportions in the back half fraction. Laboratory analytical results for each sample fraction are presented in detail in Appendix E.1.

Sampling and flue gas parameters for the PM/metals runs are shown in Table 2-7. Total sampling times, sample volume, and isokinetic results for each sampling run are presented. Appendix C.1 contains a complete listing of these and additional sampling and flue gas parameters for each run. The field data sheets are contained in Appendix A.1.

Polynuclear Aromatic Hydrocarbons

Table 2-8 presents the PAH amounts in the flue gas sample for the emission tests in total μg for each run. Laboratory analytical results for each sample are presented in detail in Appendix E.4.

Sampling and flue gas parameters for the PAH runs are shown in Table 2-9. Total sampling times, sample volume, and isokinetic results for each sampling run are presented. Appendix A.4 contains a complete listing of these and additional sampling and flue gas parameters for each run along with the field data sheets.

Table 2-5

RATIO OF METALS TO PARTICULATE MATTER MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Metal	Metals to Particulate Matter Ratio (mg metal per gram of particulate)			
	Run 1 (mg/g)	Run 2 (mg/g)	Run 3 (mg/g)	Average (mg/g)
Barium	0.184	0.203	0.329	0.239
Cadmium	0.0692	ND	0.0451	0.0571
Chromium	0.213	ND	0.0302	0.122
Copper	0.264	ND	0.382	0.323
Lead	0.0217	ND	0.380	0.201
Manganese	2.31	1.82	2.49	2.21
Nickel	1.82	ND	0.0493	0.934
Silver	0.125	0.0985	ND	0.112
Zinc	0.681	ND	1.58	1.13

ND = Not Detected

NOTE: Run 2 impinger sample bottle broke during shipment. Therefore, only the front half is recorded. The average is based on Runs 1 and 3.

Metals analyzed, but not detected, are not listed in this table.

Particulate matter is based on the front half ony.

Table 2-6

METAL AMOUNTS IN FLUE GAS SAMPLES BY SAMPLE FRACTION - BLANK CORRECTED

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Metals	Run 1			Run 2	Run 3		
	Front Half (total ug)	Impinger 1,2,3 (total ug)	Total ug	Front Half (total ug)	Front Half (total ug)	Impinger 1,2,3 (total ug)	Total ug
Antimony	ND	ND	ND	ND	ND	ND	ND
Arsenic	ND	ND	ND	ND	ND	ND	ND
Barium	4.35	ND	4.35	2.68	4.70	0.410	5.11
Beryllium	ND	ND	ND	ND	ND	ND	ND
Cadmium	1.35	0.282	1.63	ND	ND	0.700	0.700
Chromium	3.97	1.06	5.03	ND	ND	0.469	0.469
Copper	ND	6.23	6.23	ND	ND	5.92	5.92
Lead	ND	0.513	0.513	ND	4.61	1.29	5.90
Manganese	53.1	1.56	54.6	24.1	36.6	2.09	38.7
Nickel	41.7	1.22	42.9	ND	ND	0.765	0.765
Phosphorus	ND	ND	ND	ND	ND	ND	ND
Selenium	ND	ND	ND	ND	ND	ND	ND
Silver	2.95	ND	2.95	1.30	ND	ND	ND
Thallium	ND	ND	ND	ND	ND	ND	ND
Zinc	2.20	13.9	16.1	ND	13.4	11.2	24.6

NOTE: Run 2 impinger sample bottle broke during shipment. Therefore, only the front half is recorded. The average is based on Runs 1 and 3.

ND = Not Detected

Table 2-7

METALS/PM EMISSIONS SAMPLING AND FLUE GAS PARAMETERS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run No. / Date	Run 1 09/19/91	Run 2 09/19/91	Run 3 09/20/91	Average
Total Sampling Time (min)	125	125	125	NA
Average Sampling Rate (dscfm)	0.490	0.470	0.460	0.473
Metered Volume (dscf)	61.9	59.2	57.6	59.6
Metered Volume (dscm)	1.752	1.677	1.631	1.687
Average Stack Temperature (F)	241	236	245	241
O2 Concentration (%V)	14.2	12.5	14.3	13.7
CO2 Concentration (%V)	5.6	5.8	5.4	5.6
Stack Gas Moisture (%V)	29.7	29.3	29.3	29.4
Volumetric Flow Rate (dscfm)	33350	32000	31100	32200
Volumetric Flow Rate (dscmm)	948	906	880	911
Percent Isokinetic	104	104	104	NA
Particulate Catch (grams)	0.0236	0.0132	0.0155	0.0174

NOTE: Run 2 impinger sample bottle broke during shipment.

NA = Not Applicable

TABLE 2-8

PAH AMOUNTS IN FLUE GAS SAMPLES - BLANK CORRECTED

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Field Blank (total ug)
Acenaphthylene	ND	ND	ND	ND
Acenoaphthene	ND	ND	ND	ND
Anthracene	ND	ND	ND	ND
Benzo(a)anthracene	ND	ND	ND	ND
Benzo(a)pyrene	ND	ND	ND	ND
Benzo(b)fluoranthene	ND	ND	ND	ND
Benzo(e)pyrene	ND	ND	ND	ND
Benzo(g,h,i)perylene	ND	ND	ND	ND
Benzo(k)fluoranthene	ND	ND	ND	ND
2-Chloronapthalene	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND
Dibenz(a,h)anthracene	ND	ND	ND	ND
Dibenzofuran	ND	ND	ND	ND
7,12-Dimethylbenz(a)anthracene	ND	ND	ND	ND
Fluoranthene	ND	ND	ND	ND
Fluorene	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	ND	ND	ND	ND
2-Methylnaphthalene	408	348	417	(2.36)
Naphthalene	ND	ND	719	751
Perylene	ND	ND	ND	ND
Phenanthrene	ND	ND	ND	ND
Pyrene	ND	ND	ND	ND

( ) = Estimated Value

ND = Not Detected

Table 2-9

PAH SAMPLING AND FLUE GAS PARAMETERS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run No. Date	Run 1 09/19/91	Run 2 09/19/91	Run 3 09/20/91	Average
Total Sampling Time (min)	125	125	125	NA
Average Sampling Rate (dscfm)	0.520	0.490	0.470	0.493
Metered Volume (dscf)	65.0	61.6	58.8	61.8
Metered Volume (dscm)	1.840	1.745	1.665	1.750
Average Stack Temperature (F)	240	236	244	240
O2 Concentration (%V)	14.2	12.5	14.3	13.7
CO2 Concentration (%V)	5.6	5.8	5.4	5.6
Stack Gas Moisture (%V)	27.5	28.4	28.1	28.0
Volumetric Flow Rate (dscfm)	34300	32600	31600	32800
Volumetric Flow Rate (dscmm)	971	925	894	930
Percent Isokinetic	107	106	105	106

NA = Not Applicable

2.3 PARTICULATE MATTER

2.3.1 Overview

Particulate matter emissions were measured using the front half particulate catch collected in the combined PM/metals train. Before metals speciation analysis, the filter and front half acetone rinse (e.g., rinsate from nozzle, probe, and filter holder) were analyzed gravimetrically as described in Section 5. The sampling and flue gas parameters have been presented previously in Table 2-5. Detailed sampling parameters are provided in Appendix C.1 and analytical results in Appendix E.1.

2.3.2 Particulate Matter Results

Table 2-10 summarizes the results of the gravimetric analyses. Exhaust grain loadings, corrected to 7 percent O_2, ranged from 0.0057 gr/dscf to 0.0121 gr/dscf with an average of 0.0089 gr/dscf, and emission rates ranged from 0.941 lb/hr to 1.69 lb/hr with an average of 1.24 lb/hr.

Table 2-2 summarizes emission factors for total PM which varied from 0.00386 lb/ton of product to 0.00690 lb/ton of product with an average of 0.00595 lb/ton of product. The variance may be related to transient emission conditions associated with process start-up, which coincided with sampling Run 1. The emission factors generated from sampling Runs 2 and 3 are in relatively close agreement, whereas the first run emission factor represents the high endpoint of the data set.

These emission factors are less than those currently published in AP-42 (0.02 lb/ton after baghouse control, from data from circa 1973-74). This difference may be attributable to a variety of factors, including product specifications for different testing programs or differences in baghouse design, operation, and maintenance between facilities tested.

2.4 PM_{10}/CPM RESULTS

2.4.1 Overview

Three test runs were conducted to determine the concentration and emission rate of PM_{10}. The testing procedures followed EPA Method 201A for the determination of PM_{10} emissions using the constant sampling rate (CSR) procedure coupled with EPA Method 202 for determining condensible emissions from the impinger's back half. The

Table 2-10
PARTICULATE MATTER CONCENTRATIONS EMISSIONS
MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Date	Location	Run #	Time (min)	FLUE GAS CONCENTRATION				FLUE GAS EMISSION RATE	
				(grains/dscf)	(grains/dscf @ 7% O2)	(grams/dscm)	(grams/dscm @ 7% O2)	(lb/hr)	(kg/hr)
09/19/91	Outlet	1	125	0.00589	0.0121	0.0135	0.0277	1.69	0.766
09/19/91	Outlet	2	125	0.00344	0.00571	0.00787	0.0131	0.941	0.428
09/20/91	Outlet	3	125	0.00415	0.00870	0.00951	0.0199	1.11	0.502
Average				0.00449	0.00885	0.0103	0.0202	1.24	0.565

CSR employs normal isokinetic sampling procedures except that the sample duration at each sampling point is proportional to the gas velocity at that point.

It should be noted that the smaller PM_{10} nozzles that would allow 100 percent isokinetic sampling and a cut size at 10 microns were several inches longer than a standard nozzle and would have required a 4-in. ID sampling port. The existing ports were only 3 in. ID, and therefore, a nozzle with a shorter, larger diameter had to be used. This resulted in a PM cut size of 7.9 microns rather than the expected 10 microns. The net effect on the results is that the fraction of PM present between 7.9 and 10 microns cannot be determined from the data collected. Therefore, these results are not comparable to PM_{10} data collected from other similar facilities, if they were collected at the targeted 10-micron cut size.

A full description of the method procedures can be found in Section 5 as well as in the EPA Reference Method located in Appendix I.2. The final results from this test procedure are presented in terms of the phase of PM caught as well as the cut size. The following PM weight fractions were determined:

- Noncondensible PM > 8 microns (cyclone fraction);
- Noncondensible PM < 8 microns (filter fraction);
- Inorganic CPM associated with the water fraction (< 8 microns); and
- Inorganic CPM associated with the methylene chloride fraction (<8 microns).

Stack gas velocities measured prior to PM_{10} sampling were up to 15 percent higher than those recorded for the other sampling trains. This difference may have resulted from reduced dampening of the exhaust between the fan and the sampling ports, but this is not certain. All other process parameters (e.g., production rates, fuel consumption rates, aggregate moisture) appear comparable to those monitored for the other tests.

2.4.2 PM_8 Emissions

The average emission rate and emission factor for PM less than 8 microns were equal to those for PM greater than 8 microns (1.65 lb/hr and 0.008 lb/ton of product,

each). Although the PM_8 emissions ranged from 45 to 55 percent of the total PM measured, the equal distribution of emissions above and below the cut size may be coincidental because only Runs 2 and 3 measured condensible emissions in both the water and methylene chloride fractions. No organic condensibles were found in the methylene chloride fraction of Run 1. This anomaly may be related to the process conditions or possible sampling or analytical errors, but a specific cause is not apparent.

Table 2-11 summarizes the PM_{10} emission factors and process operating data. Table 2-12 summarizes the analytical results, and Figure 2-1 illustrates the particle size contributions to mass emission rates. Figure 2-2 shows the particle size contributions to flue gas PM concentrations under actual conditions and conditions corrected to 7 percent O_2. Figure 2-3 illustrates the relative contribution of each sample fraction to total PM catch for each sample run.

2.5 ALDEHYDE RESULTS

2.5.1 Overview

A single sampling train was used to collect samples for analysis for 18 aldehydes (acetaldehyde, acetone, acetophenone/o-tolualdehyde, acrolein, benzaldehyde, butyraldehyde/isobutyraldehyde, crotonaldehyde, 2,5-dimethylbenzaldehyde, formaldehyde, hexanal, isophorone, isovaleraldehyde, MIBK/p-tolualdehyde, methyl ethyl ketone, propionaldehyde, quinone, m-tolualdehyde, and valeraldehyde). Three sampling runs were performed in order to ensure representative test results.

2.5.2 Process Operation

Table 2-13 presents the aldehyde emission factors with a summary of process operating data for the three test runs. During the emission tests, the plant was operating at slightly below normal load of 76 and 65 percent for Runs 1 and 3, but at a normal load of 95 percent for Run 2. Therefore, only Runs 1 and 3 will be used in averaging the operating data. The production rates were 229 tons/hr, 284 tons/hr, and 194 tons/hr for Runs 1, 2, and 3, respectively. Natural gas consumption varied from 5000 ft^3/hr (Run 3) to 6947 ft^3/ft/hr (Run 2).

Only the aldehydes detected are presented. The other aldehydes were analyzed, but they were not collected in detectable amounts. Of the 18 aldehydes analyzed,

Table 2-11

SUMMARY OF PM10/CPM EMISSION FACTORS AND PROCESS OPERATING DATA

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run Number		1	2	3	Average
Date		09/19/91	09/20/91	09/20/91	
Production Rate	(tons/hr)	224	213	215	217
Virgin Asphalt Rate	(tons/hr)	211	200.6	202.5	204.7
Recycled Asphalt Rate	(tons/hr)	0	0	0	0
Asphalt Cement Rate	(tons/hr)	13	12.4	12.5	12.6
Percent of Rated Capacity	(%)	75	71	72	73
Aggregate Moisture	(%)	4.3	4.4	4.7	4.5
Fuel Flow	(cubic foot/hr)	5180	4990	5230	5130
Heat Imput Rate	(1000 cubic foot/hr)	5.18	4.99	5.23	5.13
Burner Setting - Flame Meter	(%)	74	79	76	76
Ambient Temperature	(degree F)	51	53	58	54
Ambient Humidity	(%)	57	61	46	55
Kiln Exit Temperature	(degree F)	340	350	348	346
Stack Flow Rate	(dscfm)	33530	32660	32100	32840
Stack Flow Rate	(dscf/ton of product)	8981	9200	8960	8320
Stack Temperatrue	(degree F)	241	255	242	246
Stack Moisture	(% volume)	28.5	28.5	27.6	28.2
Stack CO2	(volume % dry)	5.1	5.4	5.6	5.4
Stack O2	(volume % dry)	14.1	14	13.6	13.9
Stack CO	(ppmV)	> 1000	1496	1567	1532
Control Device		Baghouse	Baghouse	Baghouse	
Particulate Emissions < Cut Size	(lbs/ton of product)	0.009	0.007	0.007	0.008
Particulate Emissions > Cut Size	(lbs/ton of product)	0.008	0.008	0.007	0.008
Particulate Emissions Total	(lbs/ton of product)	0.017	0.015	0.014	0.016

NOTE = Run averages were calculated from readings taken periodically throughout the duration of the emission test run.

See Table 3-1 and 3-2 for the individual readings.

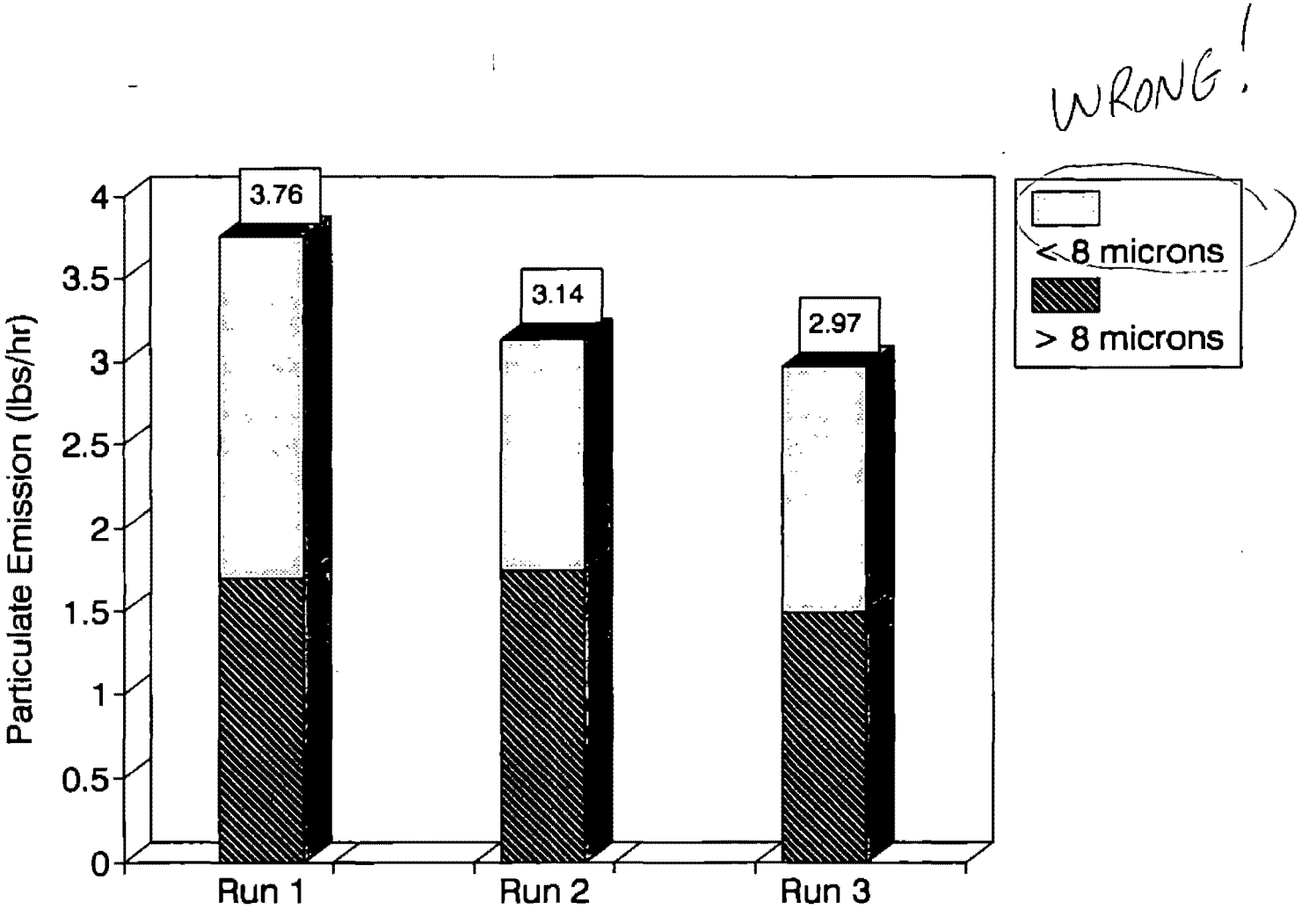
Table 2-12

PM10 EMISSIONS TEST RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

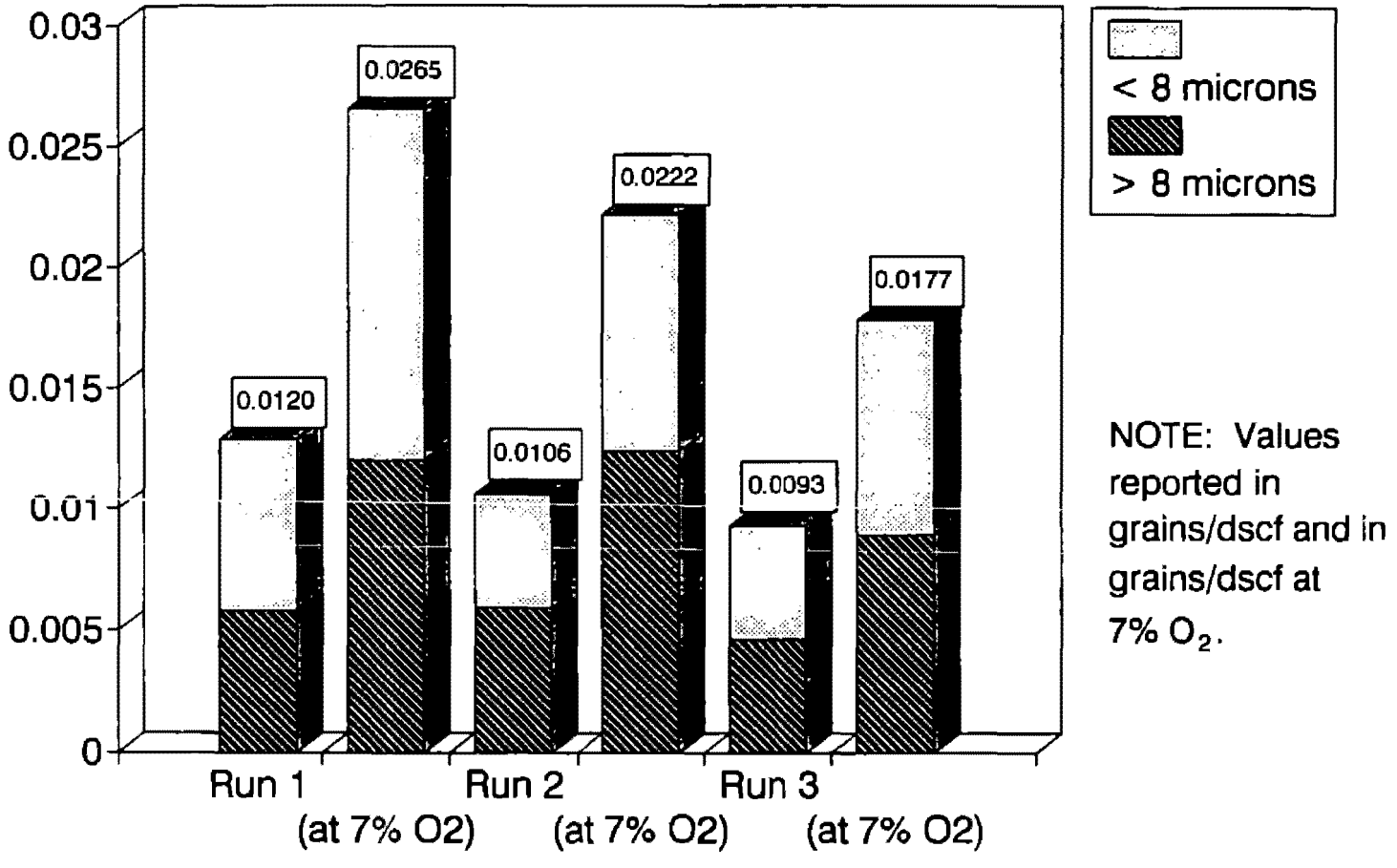
Stack Conditions	Run 1	Run 2	Run 3	Average
Corrected Barometric Pressure (in. Hg)	29.68	29.68	29.68	29.68
Stack Static Pressure (in. H2O)	0.80	0.80	0.80	0.80
Average Stack Temperature (degree F)	241	255	242	246
Carbon Dioxide Concentration (%V)	5.10	5.40	5.62	5.37
Oxygen Concentration (%V)	14.12	14.24	13.61	13.99
Nitrogen Concentration (%V)	80.78	80.36	80.77	80.64
Stack Moisture (%V)	28.5	28.5	27.6	28.2
Stack Gas Velocity,Vs (fps)	75.6	78.3	82.1	78.7
Volumetric Flow Rate (acfm)	63500	65700	69000	66100
Volumetric Flow Rate (dscfm)	34000	34500	37300	35300
Stack Viscosity (micropoise)	199	202	199	200
Sampling Parameters				
Total Sampling Time (min)	70.8	64.1	61.0	65.3
Average Meter Temperature (degree F)	65	70	73	69
Average Meter Pressure (in.H2O)	0.56	0.56	0.56	0.56
Average Sampling Rate (dscfm)	0.41	0.41	0.40	0.41
Average Sampling Rate (acfm - cyclone cond)	0.78	0.79	0.74	0.77
Standard Metered Volume,Vm(std) (dscf)	29	27	25	27
Percent Isokinetic	94.6	93.2	83.6	90.4
PM10 Results				
Cut Size (um)	7.80	7.82	8.04	7.89
Cyclone Particulate Catch (g)	0.0111	0.0102	0.0074	0.0096
PM10 Filter Particulate Catch (g)	0.0034	0.0029	0.0020	0.0028
H2O Back Half Particulate Catch (g)	0.0101	0.0037	0.0038	0.0059
MeCl Back Half Particulate Catch (g)	0.0000	0.0015	0.0016	0.0010
Total Particulate Catch (g)	0.0246	0.0183	0.0148	0.0192
Particulate Conc. < Cut Size (grains/dscf)	0.0071	0.0047	0.0047	0.0055
Particulate Conc. < Cut Size (grains/dscf @7%	0.0145	0.0098	0.0089	0.0111
Particulate Conc. > Cut Size (grains/dscf)	0.0058	0.0059	0.0047	0.0055
Particulate Conc. > Cut Size (grains/dscf @7%	0.0120	0.0124	0.0089	0.0111
Particulate Conc. Total (grains/dscf)	0.0129	0.0106	0.0093	0.0110
Particulate Conc. Total (grains/dscf @7% O2)	0.0265	0.0222	0.0177	0.0221
Particulate Emissions < Cut Size (lbs/hr)	2.07	1.39	1.49	1.65
Particulate Emissions > Cut Size (lbs/hr)	1.70	1.75	1.49	1.65
Particulate Emissions Total (lbs/hr)	3.77	3.14	2.98	3.30

flow rates don't match data sheets



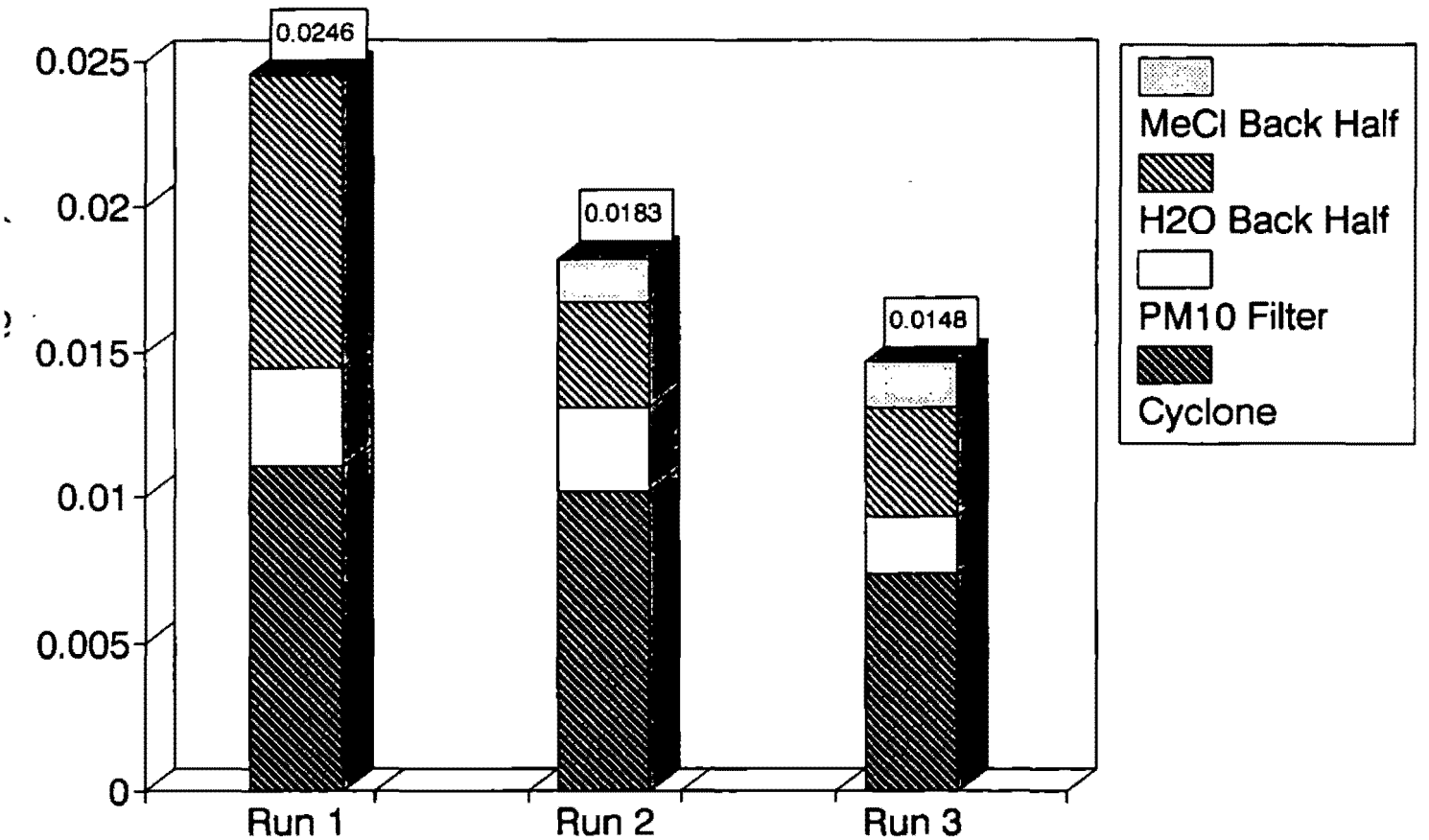
- Sample port and nozzle considerations reduced cut size to approximately 8 microns.

Figure 2-1. Emission Rates for PM_{10} (in lbs/hr)
Mathy Construction Company Facility No. 6



- Sample port and nozzle considerations reduced cut size to 8 microns.

Figure 2-2. Concentrations of PM_{10} (in grains/dscf) Mathy Construction Company Facility No. 6



* Sample port and nozzle considerations reduced cut size to approximately 8 micorns.

Figure 2-3. Filter Recoveries of PM_{10} (in grams)
Mathy Construction Company Facility No. 6

Table 2-13

SUMMARY OF ALDEHYDE EMISSION FACTORS AND PROCESS OPERATING DATA

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run number		1	2	3	Average
Date		09/19/91	09/20/91	09/20/91	
Production Rate	(ton/hr)	229	284	194	212
Virgin Asphalt Rate	(ton/hr)	215.7	267.5	182.7	199.2
Recycled Asphalt Rate	(ton/hr)	0	0	0	0
Asphalt Cement Rate	(ton/hr)	13.3	16.5	11.3	12.3
Percent of Rated Capacity	(%)	76	95	65	71
Aggregate Moisture	(%)	4.3	4.7	4.7	4.5
Fuel Flow	(cubic foot/hr)	5393	6947	5000	5197
Heat Imput Rate	(1000 cubic foot/hr)	5.39	6.95	5.00	5.20
Burner Setting - Flame Meter	(%)	70	84	75	73
Ambient Temperature	(degree F)	49	40	58	54
Ambient Humidity	(%)	58	80	46	52
Kiln Exit Temperature	(degree F)	341	347	348	345
Stack Flow Rate	(dscfm)	33187	32988	32475	32831
Stack Flow Rate	(dscf/ton of product)	8695	6969	10044	9370
Stack Temperature	(degree F)	240	238	234	237
Stack Moisture	(% volume)	28.7	29.5	27.1	27.9
Stack CO2	(volume % dry)	6.3	6.6	5.6	6.0
Stack O2	(volume % dry)	13.5	12.4	13.6	13.6
Stack CO	(ppmV)	> 1000	> 1000	1567	1567
Control Device		Baghouse	Baghouse	Baghouse	
Acetaldehyde	(lbs x 10(-4)/ton of product) *	5.07	5.35	8.78	6.92
	(lbs x 10(-4)/1000 cubic foot) *	215	219	341	278
Acetone	(lbs x 10(-4)/ton of product) *	105	23.5	ND	105
	(lbs x 10(-4)/1000 cubic foot) *	4460	962	ND	4460
Benzaldehyde	(lbs x 10(-4)/ton of product) *	1.23	1.02	1.58	1.41
	(lbs x 10(-4)/1000 cubic foot) *	52.2	41.8	61.4	56.8
Butyraldehyde/Isobutyraldehyde	(lbs x 10(-4)/ton of product) *	0.290	ND	0.566	0.428
	(lbs x 10(-4)/1000 cubic foot) *	12.3	ND	21.9	17.1
Crotonaldehyde	(lbs x 10(-4)/ton of product) *	0.229	0.152	0.488	0.358
	(lbs x 10(-4)/1000 cubic foot) *	9.72	6.22	18.9	14.3
Formaldehyde	(lbs x 10(-4)/ton of product) *	18.1	23.0	20.7	19.4
	(lbs x 10(-4)/1000 cubic foot) *	770	941	803	787
Hexanal	(lbs x 10(-4)/ton of product) *	0.251	0.323	0.150	0.201
	(lbs x 10(-4)/1000 cubic foot) *	10.7	13.2	5.82	8.24
Methyl Ethyl Ketone	(lbs x 10(-4)/ton of product) *	ND	ND	0.701	0.701
	(lbs x 10(-4)/1000 cubic foot) *	ND	ND	27.2	27.2
Propionaldehyde	(lbs x 10(-4)/ton of product) *	ND	ND	0.710	0.710
	(lbs x 10(-4)/1000 cubic foot) *	ND	ND	27.5	27.5
Quinone	(lbs x 10(-4)/ton of product) *	0.301	0.139	7.80	4.05
	(lbs x 10(-4)/1000 cubic foot) *	12.8	5.69	303	158
Valeraldehyde	(lbs x 10(-4)/ton of product) *	ND	ND	0.427	0.427
	(lbs x 10(-4)/1000 cubic foot) *	ND	ND	16.6	16.6

ND = Not Detected

* = 1 x 10 (-4) or 0.0001

NOTE : Run averages were calculated from readings taken periodically throughout the duration of the emission test run.

See Table 3-1 and 3-2 for the individual readings.

Aldehyde compounds analyzed, but not detected, are not included in this table.

Aldehyde compound concentrations have been blank corrected.

Run 2 was not calculated in the average. Runs 1 & 3 were at a minimum production load, whereas Run 2 was at a maximum production load.

All three tests were interrupted by plant shutdown. Trains were removed from stack and were not operated during down times.

11 were detected. These were acetaldehyde, acetone, benzaldehyde, formaldehyde, cutyraldehyde/isobutyraldehyde, crotonaldehyde, hexanal, methyl ethyl ketone, quinone propionaldehyde, and valeraldehyde. Aldehyde emissions from this plant are most likely a function of fuel consumption and any volatile fraction of the asphalt cement. The emission factors generated for aldehydes follow the same assumptions applied to the PAH emission factors.

2.5.3 Emissions

Table 2-14 presents the aldehyde emissions results for the three test runs. Also shown for each run are the date, metered volume (in dscm), O_2 concentration, and flow rate. Flue gas concentrations are given in terms of g/dscm, and g/dscm corrected to 7 percent O_2. Oxygen concentrations were collected from CEM data.

During the emission tests, acetone had the highest average mass rate with 1090 g/hr, followed by formaldehyde with 185 g/hr. After blank correction, acetophenone/o-tolualdehyde, 2,5-dimethylbenzaldehyde, isophorone, isovaleraldehyde, MIBK/p-tolualdehyde, and m-tolualdehyde were not collected in detectable amounts for any of the runs during these emission tests. Aldehyde values ranged from 1090 g/hr of acetone in Run 1, to 1.80 g/hr of quinone in Run 2.

The aldehyde values for the emission tests did not change significantly from Runs 1 to 3, except for acetone and quinone. Acetone fluctuated from 1090 g/hr in Run 1, 303 g/hr in Run 2, and not detected in Run 3. It should be noted that acetone is a compound that contaminates easily. This should be taken into consideration when accounting for it. Quinone varied from 3.13 g/hr in Run 1, 1.80 g/hr in Run 2, and 68.6 g/hr in Run 3. At this time, there are not enough data points on this compound to suggest a reason for the variation.

2.5.4 Flue Gas Aldehydes by Sample and Sample Parameters

Table 2-15 presents the aldehyde amounts in the flue gas sample for the emission tests in total μg for each run. Laboratory analytical results for each sample are presented in detail in Appendix E.3.

Sampling and flue gas parameters for the aldehyde runs are shown in Table 2-16. Total sampling times, sample volume and isokinetic results for each sampling run are

Table 2-14

ALDEHYDES CONCENTRATION AND EMISSION RATES

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run		Run 1	Run 2	Run 3	
Date		09/20/91	09/20/91	09/20/91	
Metered Volume (dscm)		0.860	0.843	0.836	
O2 Concentration (%V)		12.85	12.56	12.43	
Volumetric Flow Rate (dscm/s)		939.857	934.216	919.691	AVERAGE
Acetaldehyde	(ug/dscm)	934	1230	1400	1170
	(ug/dscm @ 7% O2)	1610	2000	2600	2100
	(g/hr)	52.7	68.9	77.2	65.0
Acetone	(ug/dscm)	19400	5410	ND	19400
	(ug/dscm @ 7% O2)	33500	8800	ND	33500
	(g/hr)	1090	303	ND	1090
Benzaldehyde	(ug/dscm)	226	235	252	239
	(ug/dscm @ 7% O2)	390	382	470	430
	(g/hr)	12.8	13.2	13.9	13.3
Butyraldehyde/Isobutyraldehyde	(ug/dscm)	53.4	ND	90.2	71.8
	(ug/dscm @ 7% O2)	92.2	ND	168	130
	(g/hr)	3.01	ND	4.98	3.99
Crotonaldehyde	(ug/dscm)	42.2	35.0	77.8	60.0
	(ug/dscm @ 7% O2)	72.8	57.0	145	109
	(g/hr)	2.38	1.96	4.29	3.33
Formaldehyde	(ug/dscm)	3340	5290	3300	3320
	(ug/dscm @ 7% O2)	5770	8610	6140	5960
	(g/hr)	188	297	182	185
Hexanal	(ug/dscm)	46.3	74.1	23.9	35.1
	(ug/dscm @ 7% O2)	79.9	121	44.5	62.2
	(g/hr)	2.61	4.16	1.32	1.96
Methyl Ethyl Ketone	(ug/dscm)	ND	ND	112	112
	(ug/dscm @ 7% O2)	ND	ND	208	208
	(g/hr)	ND	ND	6.17	6.17
Propionaldehyde	(ug/dscm)	ND	ND	113	113
	(ug/dscm @ 7% O2)	ND	ND	211	211
	(g/hr)	ND	ND	6.24	6.24
Quinone	(ug/dscm)	55.5	32.0	1244	650
	(ug/dscm @ 7% O2)	95.8	52.1	2315	1205
	(g/hr)	3.13	1.80	68.6	35.9
Valeraldehyde	(ug/dscm)	ND	ND	68.1	68.1
	(ug/dscm @ 7% O2)	ND	ND	127	127
	(g/hr)	ND	ND	3.76	3.76

NOTE: Concentrations given have been blank corrected.

Aldehyde compounds analyzed, but not detected, are not included in this table.

Run 2 was not calculated in the average. Runs 1 & 3 were at a minimum production load, whereas Run 2 was at a maximum production load.

All three tests were interrupted by plant shutdown. Trains were removed from stack and were not operated during down times.

TABLE 2-15

ALDEHYDES AMOUNTS IN FLUE SAMPLES - BLANK CORRECTED

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Field Blank (total ug)
Acetaldehyde	822	1040	1170	ND
Acetone	17060	4560	ND	6640
Acetophenone/o-Tolualdehyde	ND	ND	ND	ND
Acrolein	ND	ND	26.7	ND
Benzaldehyde	199	198	211	ND
Butyraldehyde/Isobutyraldehyde	47.0	ND	75.4	ND
Crotonaldehyde	37.1	29.5	65.0	ND
2,5-Dimethylbenzaldehyde	ND	ND	ND	ND
Formaldehyde	2940	4460	2760	10.7
Hexanal	40.7	62.5	20.0	ND
Isophorone	ND	ND	ND	ND
Isovaleraldehyde	ND	ND	ND	ND
MIBK/p-Tolualdehyde	ND	ND	ND	ND
Methyl Ethyl Ketone	ND	ND	93.5	ND
Propionaldehyde	ND	ND	94.6	ND
Quinone	48.8	27.0	1040	ND
m-Tolualdehyde	ND	ND	ND	ND
Valeraldehyde	ND	ND	56.9	ND

NOTE: Concentrations given have been blank corrected.

↑

ug is not a concentration!

Table 2-16

ALDEHYDES EMISSIONS SAMPLING AND FLUE GAS PARAMETERS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run No. Date	Run 1 09/20/91	Run 2 09/20/91	Run 3 09/20/91	Average
Total Sampling Time (min)	62.5	62.5	62.5	NA
Average Sampling Rate (dscfm)	0.500	0.480	0.470	0.483
Metered Volume (dscf)	31.1	29.8	29.5	30.1
Metered Volume (dscm)	0.880	0.843	0.836	0.853
Average Stack Temperature (F)	240	238	234	237
O2 Concentration (%V)	12.9	12.4	13.4	12.9
CO2 Concentration (%V)	6.2	6.6	5.6	6.1
Stack Gas Moisture (%V)	28.7	29.6	27.1	28.5
Volumetric Flow Rate (dscfm)	33200	33000	32500	32900
Volumetric Flow Rate (dscmm)	940	934	920	931
Percent Isokinetic	105	101	102	NA

NA = Not Applicable

presented. Appendix A.3 contains a complete listing of these parameters and additional sampling and flue gas parameters for each test run along with the field data sheets.

2.6 CONTINUOUS EMISSIONS MONITORING RESULTS

2.6.1 Overview

Continuous emissions monitoring was conducted at the outlet to the air pollution control device (APCD) during the two days of testing. Concentrations of O_2, CO_2, CO, NO_x, and SO_2 were determined on a dry basis by extracting the gas from the flue, transferring it to the CEM trailer through heated Teflon tubing (heat trace), passing it through gas conditioners to remove moisture and directing it to each respective analyzer. A full description of Radian's CEM system and methods is given in Section 5. Concentrations of THC were also monitored, with gas concentrations determined on a wet basis, by allowing a slipstream from the heated sample line to bypass the sample conditioners so that the wet flue gas was directed to the analyzer as it exited in the flue.

All CEM data were recorded as 30-second averages from multiple-readings-per-second input by Radian's CEM data acquisition system (DAS). The resulting CEM data files were averaged over the duration of each test run. The averages are presented in Section 2.7.2. The 30-second data are included in Appendix D along with the following additional CEM information:

LOCATION	CEM DATA TYPE
• Appendix D.1	CEM Tables
• Appendix D.2	Calibration Drifts
• Appendix D.3	Calibration and QC Gas Responses
• Appendix D.4	Response Time, NO_x Convertor Tests Linearity Calculation

2.6.2 CEM Results

The CEM averages are presented in Table 2-17. The average O_2 runs varied from 12.4 to 14.2 percent by volume. Carbon dioxide values were approximately 5 to 6 percent by volume. Nitrogen oxides concentrations were approximately 30 to 50 ppmv, dry at the stack. There were no SO_2 emissions. Concentrations of THC were also monitored, with the resulting concentrations consistently 50 to 70 ppmv/wet. The averages were taken over the time period that the CEM was on-line.

Table 2-17

CONTINUOUS EMISSIONS MONITORING DAILY TEST AVERAGES FOR ACTUAL CONCENTRATIONS - MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Test	Run Number	Date	Manual Run Time	CEM Run Time	O2 (%V)	THC (ppmv)	SO2 (ppmv)	CO (ppmv)	CO2 (%V)	NOx (ppmv)
PAH/Metals	1	09/19/91	0710-0923	0815-0923	14.1	52.1	0.0	a	5.7	48.0
	2	09/19/91	1328-1540	1341-1540	12.5	64.0	0.0	a	5.8	41.7
	3	09/20/91	0945-1203	1011-1203	14.2	60.3	0.0	1362.4 b	5.2	31.9
Aldehyde	1	09/19/91	0906-1117	0906-0923	13.5	51.2	0.0	a	6.3	52.0
	2	09/20/91	0745-1005	0745-0810	12.4	64.2	0.6	a	6.6	38.4
				0848-1005						
	3	09/20/91	1337-1524	1415-1524	13.6	72.8	0.2	1567.0 b	5.6	33.0
PM10	1	09/19/91	1036-1155	1125-1155	14.1	54.0	0.0	a	5.1	33.8
	2	09/20/91	1127-1252	1127-1252	14.0	70.6	0.0	1495.8 b	5.4	32.3
	3	09/20/91	1413-1524	1415-1524	13.6	72.8	0.2	1567.0 b	5.6	33.0

a = CO monitor out of range (> 1000ppmv)

b = CO monitor set to 25% of instrument range

2.6.3 Nonmethane Hydrocarbon Emission Test Results

EPA Method 18 analysis of the flue gas was performed using gas chromatography (GC) to separate the hydrocarbon species (C_1-C_6) present in the flue gas stream. Several samples were injected into the GC during each test day. Methane, benzene, toluene, ethylbenzene, and xylene were determined by this method. Total hydrocarbon emissions as methane were determined by EPA Method 25A.

Table 2-18 presents the hydrocarbon concentrations and emission rates in ppmv and lb/ton of asphalt produced. The nonmethane hydrocarbon emissions as methane were calculated by subtracting the methane concentration measured by the GC from the total hydrocarbons as methane measured by the total hydrocarbon analyzer (CEM) at the time of sample injection into the GC. The average nonmethane hydrocarbon emissions were 0.0103 to 0.0143 lb/ton for the two test days. The average emission factors of benzene, toluene, ethylbenzene, and xylene for the first test day were 0.00013, 0.00019, 0.000011, and 0.00017 lb/ton, respectively. The average emission factors for the second test day were 0.00023, 0.00019, 0.000011, and 0.00017 lb/ton, respectively. The data variability is believed to have resulted from process variability.

Table 2-18. Hydrocarbon Emission Rates and Concentrations

Date	Time	Stack Flow Rate (dscfm)	Asphalt Production Rate (tons/hr)	Methane By GC (ppmv)	Total Hydrocarbons By THC (ppmv)	Nonmethane Hydrocarbons		Benzene		Toluene		Ethylbenzene		Xylene	
						(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)	(ppmv)	(lbs/ton)
09/19/91	0836	33472	245	23.9	51.9	28.00	9.54E-03	0.338	5.61E-04	5.401	1.06E-02	0.267	6.04E-04	0.027	6.10E-05
	1059	33187	224	ND	a	NA	NA	ND	0.0	ND	0.0	ND	0	ND	0
	1341	31982	244	ND	54	54.00	1.76E-02	ND	0.0	ND	0.0	ND	0	0.037	8.02E-05
	1425	31982	244	62.25	71.9	9.65	3.15E-03	0.117	1.86E-04	7.350	1.38E-02	1.570	3.40E-03	0.495	1.07E-03
	1505	31982	244	39.86	71.7	31.84	1.04E-02	ND	0.00	ND	0.0	17.470	3.79E-02	38.51	8.35E-02
	1536	31982	244	ND	64.9	64.9	2.12E-02	0.027	4.30E-05	ND	0.0	14.530	3.15E-02	1.98	4.29E-03
AVERAGE		32431	241	21.0	52.4	31.40	1.03E-02	0.080	1.32E-04	2.13	4.06E-03	5.64	1.22E-02	6.84	1.48E-02
STANDARD DEVIATION				23.8	24.7	22.7		0.1		3.1		7.4		14.2	
09/20/91	0801	32988	284	13.68	55.5	41.82	1.21E-02	0.305	4.31E-04	0.001	1.66E-06	0.001	1.92E-06	0.596	1.15E-03
	1025	31086	222	5.77	54.6	48.83	1.70E-02	0.167	2.84E-04	0.009	1.81E-05	0.008	1.85E-05	0.003	6.95E-06
	1055	31086	222	ND	62.3	62.3	2.17E-02	0.022	3.74E-05	0.579	1.16E-03	ND	0	0.026	6.02E-05
	1151	31086	222	7.47	52.3	44.83	1.56E-02	0.332	5.65E-04	0.073	1.47E-04	0.001	2.32E-06	ND	0
	1246	32656	213	1.25	41.4	40.20	1.54E-02	ND	0.0	0.003	6.59E-06	0.019	4.82E-05	ND	0
	1451	32105	215	6.32	54.8	48.50	1.81E-02	ND	0.0	ND	0.0	0.003	7.41E-06	ND	0
	1520	32475	194	ND	0	0.00	0.0	0.156	3.17E-04	ND	0.0	ND	0	0.003	8.31E-06
AVERAGE		31926	225	4.93	45.84	40.93	1.43E-02	0.140	2.33E-04	0.095	1.91E-04	0.005	1.12E-05	0.090	1.74E-04
STANDARD DEVIATION				4.6	19.6	18.0		0.1		0.2		0.0		0.2	

[a]Instrument off-line.
ND = Not Detected
NA = Not Applicable

3. FACILITY DESCRIPTION

This section provides a description of the Mathy Construction Company's Facility No. 6 asphaltic concrete plant located in LaCrosse, Wisconsin. The process equipment and production materials used and the process parameters recorded during the emissions test are discussed.

3.1 PROCESS DESCRIPTION

The Mathy Construction Facility No. 6 is a typical batch asphaltic concrete plant with a rated production capacity of 300 tons/hour. The plant consists of the following components:

- Aggregate storage piles;
- Cold aggregate storage bins;
- A rotary kiln for aggregate drying;
- Hot screens and storage bins for dried aggregate, classified by aggregate size;
- A heated asphalt storage tank; and
- A pug mill for mixing hot aggregate and asphalt.

The aggregate must be dried to product specifications in the natural gas-fired rotary kiln before it is separated and classified by size by the hot screens and before it is mixed with asphalt in the pug mill. The asphalt is an amorphous solid that must be heated to a liquid state for injection into the pug mill where it is mixed with the dried aggregate. The final product leaving the pug mill may be loaded onto trucks or transferred to a hot mix storage bin.

Aggregate fines become entrained in the combustion exhaust leaving the rotary kiln and are transported to a cyclone, which returns heavier particles to the kiln. The exhaust gas then enters a fabric filter where smaller fines are collected. Process fugitive PM and VOCs from the hot screens, hot bins, and pug mill are also routed to the cyclone inlet. Emissions of VOCs from fuel combustion and asphalt mixing are not

controlled by the cyclone or fabric filter, nor are any VOC emission control devices used. The exhaust flow is an induced draft; that is, a clean-air-side fan is used.

The data collected only reflect emissions from vented process equipment as described above and do not include emissions from process and area fugitive sources such as:

- Aggregate storage piles and cold aggregate transport;
- Cold asphalt and hot mix storage tanks; or
- Plant vehicular traffic.

Point source PM, PM_{10}, and metal emissions are attributable primarily to aggregate-drying and hot transport mechanisms. Condensible PM, PAH, and aldehyde emissions are generally associated with fuel combustion products and the volatile fraction of the liquid asphalt, although relative contributions are not found in the literature. The aggregate/asphalt throughputs are functions of the desired product specifications, whereas fuel consumption is determined by the drying requirements, aggregate moisture, and throughput of the aggregate. For the emission factors developed, the final product composition is 5.8 percent asphalt cement by weight, and the kiln fuel is natural gas. The emission factors developed in this study reflect these constraints, and the effects of changes in these parameters on emission factors have not been evaluated.

3.2 PROCESS CONDITIONS DURING TESTING

Production monitoring data for all tests are presented in Tables 3-1 and 3-2.

3.2.1 Process Conditions During Metals/PAH Testing

Table 3-3 summarizes the production and operating conditions associated with the metals and PAH test data. Production rates for 3 runs varied from 222 tons/hour to 245 tons/hour (74 to 82 percent of capacity), while natural gas consumption ranged from 5170 ft^3/hr to 5250 ft^3/hr. Sampling runs for these trains experienced no interruptions of process upset conditions.

3.2.2 Process Conditions During Aldehyde Testing

Table 3-4 summarizes the production and operating data associated with aldehyde test data. Production rates varied from 194 tons/hr to 284 tons/hr (65 to 95 percent of capacity), while natural gas usage ranged from 5000 ft^3/hr to 6950 ft^3/hr. Sampling

Table 3-1

SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING EMISSION TESTING - SEPTEMBER 19, 1991

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Time (24hr)	Actual Fuel Use Natural Gas 100(cu.ft.)	Burner Setting		Kiln Exit Temp. (degree F)	Production Rate (tons/hr)	Injection Rate (tons/hr)	Mix Temp. (degree F)	Exhaust Gas Temp. (degree F)	Baghouse			Ambient Temp. (degree F)	Relative Humidity (%)	Exhaust Damper Position (%)
		Flame Meter (%)	Flame Eye (micro-amps)						Pressure Drop (w.g.)	Inlet Temp. (degree F)	Outlet Temp. (degree F)			
6:29	START OF WARM UP													
6:43*														
6:55	14	100	1.9	303	0		------	325	3.1	240	206	40.0	100	40.0
7:12	START OF TEST													
7:25	25	80	1.9	315	0		305	315	1.9	260	230	40.8	Not Taken	42.0
7:48	17	79	1.9	338	222	12.9	------	310	2.0	260	230	42.6	Not Taken	42.0
8:19	26	79	1.9	338	242	14.0	------	312	1.9	260	240	42.6	Not Taken	42.0
8:40	17	80	1.8	338	200	11.6	------	315	1.6	265	245	42.8	Not Taken	42.0
8:58	17	76	1.9	341	267	15.5	295	310	2.0	265	243	43.6	85	42.0
9:34	31	76	1.9	341	233	13.5	------	310	1.8	265	242	45.8	Not Taken	42.0
9:37	STOPPED PROCESS, TOOK AGGREATE FOR MOISTURE @ 9:45													
10:27**	7	63	1.9	340	6	0.3	------	285	3.0	260	195	52.0	Not Taken	43.0
11:00	31	63	1.9	340	236	13.7	------	310	1.2	260	240	52.0	58	43.0
11:20	18	81	1.9	338	210	12.2	301	315	1.0	265	244	50.6	Not Taken	43.0
11:46	22	79	1.9	342	242	14.1	------	310	1.8	264	245	50.0	57	43.0
12:01	12	79	2.0	338	280	16.2	------	312	1.6	268	244	51.2		42.0
	STOP													
13:24	START													
13:30 ***	5	92	1.9	317	100	5.8	------	312	2.5	250	165	51.0	58	42.0
14:00	27	75	1.8	343	210	12.2	------	315	1.8	262	242	51.0		42.0
14:30	30	79	1.9	340	270	15.7	------	313	1.9	265	243	52.3	50	41.0
15:00	26	80	1.9	337	280	16.2	305	312	1.7	262	241	51.0		41.0
15:30		74	1.9	344	200	11.6	------	310	1.7	264	242			41.0
15:44		79	1.9	341	193	11.2	------	310	1.8	264	242	51.4		41.0
15:46	35			Final	150	8.7	------						61	

* Start Production

** Started Plant

*** Start of Test

08:00 Crippled CEM on line

14:00 10 Burner pressure 0 sig ounces

11.5 Gas pressure psig

Table 3-2

SUMMARY OF PROCESS OPERATING DATA COLLECTED DURING EMISSION TESTING - SEPTEMBER 20, 1991

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Time (24hr)	Actual Fuel Use Natural Gas 100(cu.ft.)	Burner Setting		Kiln Exit Temp. (degree F	Production Rate (tons/hr)	Asphalt Injection Rate (tons/hr)	Mix Temp. (degree F	Exhaust Gas Temp. (degree F	Baghouse			Ambient Temp. (degree F	Relative Humidity (%)	Exhaust Damper Position (%)
		Flame Meter (%)	Flame Eye (micro-amps)						Presure Drop (w.g.)	Inlet Temp. (degree F	Outlet Temp. (degree F			
7:01	START OF WARM-UP											36.0	82	
7:36 *	4	93	1.9	358	0		---	310	0.9	250	155	36.0	Not Taken	42.0
7:45	START OF ALDEHYDES													
7:55	16	83	1.9	339	0		300	318	1.0	262	229	36.8	Not Taken	42.0
8:00	6	83	1.9	358	360	20.9	325		1.1	269	242	37.2	Not Taken	42.0
8:15	17	83	1.9	351	240	13.9	---	325	1.7	274	249	37.8	Not Taken	42.0
8:30	21	82	1.9	350	300	17.4	---	320	1.7	272	249	41.6	80	42.0
8:47	9	SHUT DOWN			141	8.2								
9:30	RESTART PLANT													
9:38	8	87	1.9	338	113	6.5	---	320	1.7	260	221	45.2	79	40.0
10:00	22	83	1.8	348	286	16.6	---	325	1.9	269	249	51.2	Not Taken	40.0
10:30	27	83	1.8	349	220	12.8	---	315	2.2	269	249	52.8	Not Taken	40.0
11:07	31	79	1.9	350	219	12.7	---	315	2.0	265	249		Not Taken	42.0
11:36	25	82	1.9	348	228	13.2	---	320	2.4	270	250	55.4	Not Taken	43.0
12:05	26	78	1.9	348	228	13.2	302	315	3.0	269	250	51.4	61	44.0
12:39	30	78	1.9	352	212	12.3	---	315	2.9	269	252		Not Taken	42.0
12:59	STOP				195	11.3								
	13													
13:35	4	100	1.9	314	17	1.0	---	305	2.8	260	220			40.0
14:12 ***	6	79	1.8	347	77	4.5	---	305	2.3	254	224	56.0	Not Taken	40.0
14:30	16	71	1.8	350	283	16.4	---	310	2.9	252	230	58.0	46	41
15:00	24	78	1.8	348	140	8.1	300	310	2.2	252	235	59.0	Not Taken	41
15:27	END OF PM TEST													
15:30	28	75	1.9	350	250	14.5	---	310	1.8	258	244	61.0	Not Taken	41

* Start Production

** Pegged

*** Restart

Table 3-3

SUMMARY OF METALS/PM AND PAH EMISSION FACTORS AND PROCESS OPERATING CONDITIONS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run Number		1	2	3	Average
Date		6.23	ND	5.92	
Production Rate	(tons/hr)	245	244	222	237
Virgin Asphalt Rate	(tons/hr)	230.8	229.8	209.1	223.2
Recycled Asphalt Rate	(tons/hr)	0	0	0	0
Asphalt Cement Rate	(tons/hr)	14.2	14.2	12.9	13.8
Percent of Rated Capacity	(%)	82	81	73	79
Aggregate Moisture	(%)	4.3	4.3	4.4	4.3
Fuel Flow	(cubic foot/hr)	5250	5280	5190	5240
Heat Imput Rate	(1000 cubic foot/hr)	5.25	5.28	5.19	5.24
Burner Setting - Flame Meter	(%)	78	80	81	80
Ambient Temperature	(degree F)	43	51	53	49
Ambient Humidity	(%)	85	54	70	70
Kiln Exit Temperature	(degree F)	339	341	349	343
Stack Flow Rate	(dscfm)	34280	32660	31570	32840
Stack Flow Rate	(dscf/ton of product)	8390	8030	8530	8320
Stack Temperatrue	(degree F)	240	236	244	240
Stack Moisture	(% volume)	27.5	28.4	28.1	28.0
Stack CO2	(volume % dry)	5.7	5.8	5.2	5.6
Stack O2	(volume % dry)	14.1	12.5	14.2	13.6
Stack CO	(ppmV)	> 1000	> 1000	1362	1362
Control Device		Baghouse	Baghouse	Baghouse	

NOTE = Run averages were calculated from readings taken periodically throughout the duration of the emission test run.
See Table 3-1 and 3-2 for the individual readings.

Table 3-4

SUMMARY OF ALDEHYDE EMISSION FACTORS AND PROCESS OPERATING CONDITIONS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run number		1	2	3	Average
Date		09/19/91	09/20/91	09/20/91	
Production Rate	(ton/hr)	229	284	194	212
Virgin Asphalt Rate	(ton/hr)	215.7	267.5	182.7	199.2
Recycled Asphalt Rate	(ton/hr)	0	0	0	0
Asphalt Cement Rate	(ton/hr)	13.3	16.5	11.3	12.3
Percent of Rated Capacity	(%)	76	95	65	71
Aggregate Moisture	(%)	4.3	4.7	4.7	4.5
Fuel Flow	(cubic foot/hr)	5393	6947	5000	5197
Heat Imput Rate	(1000 cubic foot/hr)	5.39	6.95	5.00	5.20
Burner Setting - Flame Meter	(%)	70	84	75	73
Ambient Temperature	(degree F)	49	40	58	54
Ambient Humidity	(%)	58	80	46	52
Kiln Exit Temperature	(degree F)	341	347	348	345
Stack Flow Rate	(dscfm)	33187	32988	32475	32831
Stack Flow Rate	(dscf/ton of product)	8695	6969	10044	9370
Stack Temperature	(degree F)	240	238	234	237
Stack Moisture	(% volume)	28.7	29.5	27.1	27.9
Stack CO2	(volume % dry)	6.3	6.6	5.6	6.0
Stack O2	(volume % dry)	13.5	12.4	13.6	13.6
Stack CO	(ppmV)	> 1000	> 1000	1567	1567
Control Device		Baghouse	Baghouse	Baghouse	

NOTE : Run averages were calculated from readings taken periodically throughout the duration of the emission test run.

See Table 3-1 and 3-2 for the individual readings.

Run 2 was not calculated in the average. Runs 1 & 3 were at a minimum production load, whereas Run 2 was at a maximum production load.

All three tests were interrupted by plant shutdown. Trains were removed from stack and were not operated during down times.

Run 1 was temporarily interrupted while aggregate moisture was measured, and sampling did not proceed during this stoppage. Sampling Run 2 was interrupted by a full plant shutdown. Sampling Run 3 was also interrupted by a full plant shutdown. In all cases, sampling was suspended and sampling equipment was removed from the stack and capped to prevent contamination during plant shutdowns.

3.2.3 Process Conditions During PM_{10}/CPM Testing

Table 3-5 summarizes the production and operating data that correspond to the PM_{10}/CPM test results. For three sampling runs, total asphaltic concrete production varied between 213 tons/hr and 224 tons/hr (71 to 75 percent of capacity), while natural gas consumption ranged from 5000 ft^3/hr to 5230 ft^3/hr. No PM_{10}/CPM sampling runs were interrupted by process upsets.

Table 3-5

SUMMARY OF PM10/CPM EMISSION FACTORS AND PROCESS OPERATING CONDITIONS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Run Number		1	2	3	Average
Date		09/19/91	09/20/91	09/20/91	
Production Rate	(tons/hr)	224	213	215	217
Virgin Asphalt Rate	(tons/hr)	211	200.6	202.5	204.7
Recycled Asphalt Rate	(tons/hr)	0	0	0	0
Asphalt Cement Rate	(tons/hr)	13	12.4	12.5	12.6
Percent of Rated Capacity	(%)	75	71	72	73
Aggregate Moisture	(%)	4.3	4.4	4.7	4.5
Fuel Flow	(cubic foot/hr)	5180	4990	5230	5130
Heat Input Rate	(1000 cubic foot/hr)	5.18	4.99	5.23	5.13
Burner Setting - Flame Meter	(%)	74	79	76	76
Ambient Temperature	(degree F)	51	53	58	54
Ambient Humidity	(%)	57	61	46	55
Kiln Exit Temperature	(degree F)	340	350	348	346
Stack Flow Rate	(dscfm)	33530	32660	32100	32840
Stack Flow Rate	(dscf/ton of product)	8981	9200	8960	8320
Stack Temperatrue	(degree F)	241	255	242	246
Stack Moisture	(% volume)	28.5	28.5	27.6	28.2
Stack CO2	(volume % dry)	5.1	5.4	5.6	5.4
Stack O2	(volume % dry)	14.1	14	13.6	13.9
Stack CO	(ppmV)	> 1000	1496	1567	1532
Control Device		Baghouse	Baghouse	Baghouse	

NOTE = Run averages were calculated from readings taken periodically throughout the duration of the emission test run. See Table 3-1 and 3-2 for the individual readings.

4. SAMPLING LOCATIONS

This section describes the locations where samples were taken during the emission testing program at Mathy Construction Company Facility No. 6. All samples collected by manual methods including PM_{10} samples were collected from six sampling ports at equal heights in the exhaust stack. Samples for the CEMS were collected from a single point near the manual sampling ports. The sampling location arrangement is shown in Figure 4-1.

The test ports were located according to EPA Method 1. The nearest upstream disturbance was 3.3 equivalent diameters away and the nearest downstream disturbance was 1.2 equivalent diameters away from the test ports.

The minimum number of traverse points required for manual sampling was 24. Five points at each of the six ports were used as shown in Figure 4-2.

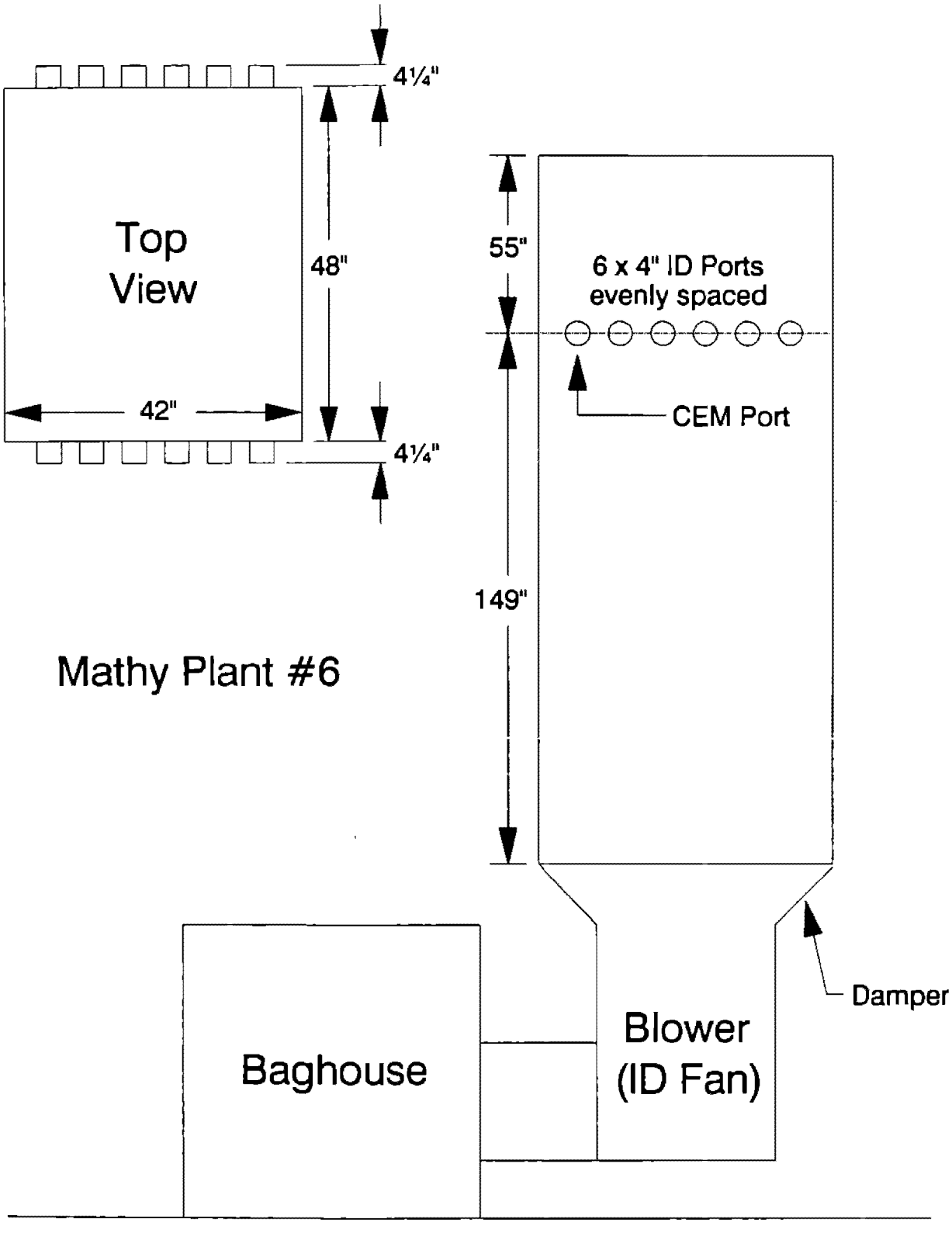
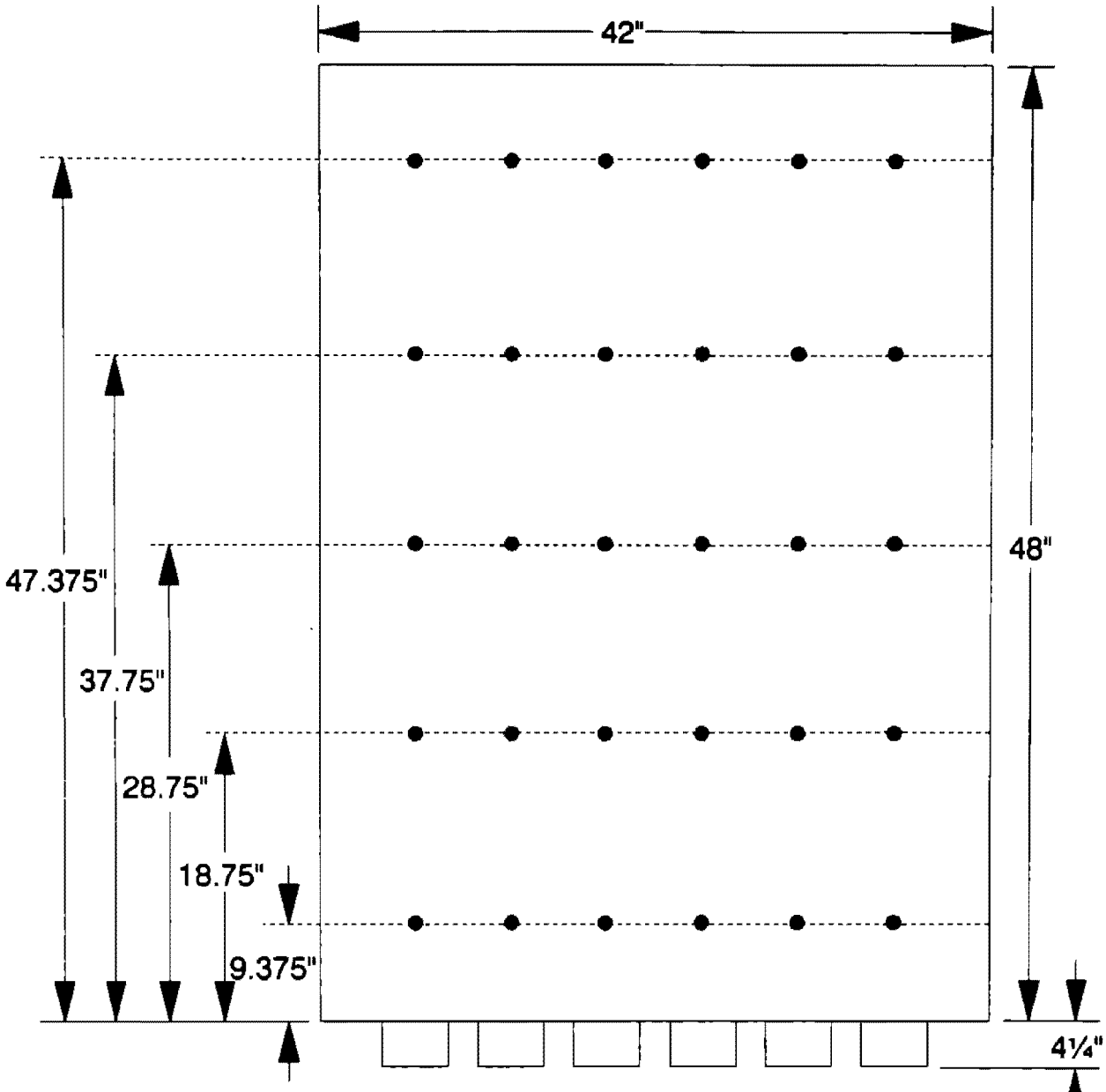


Figure 4-1. Sampling Location Arrangement.



Point	Probe Mark
1	9.375"
2	18.75"
3	28.75"
4	37.75"
5	47.375"

Figure 4-2. Traverse Point Layout at Stack.

5. SAMPLING AND ANALYTICAL PROCEDURES

The sampling and analytical procedures used for the asphalt plant test program were the most recent revisions of the published EPA methods or proposed EPA methods. In either case, state-of-the-art sampling and analytical methods were used. This section describes the sampling and analytical method used for each compound analyzed.

5.1 PARTICULATE MATTER AND METALS EMISSIONS TESTING METHOD

Sampling for particulate matter (PM) and metals was performed according to an EPA EMB draft protocol entitled "Methodology for the Determination of Metals Emissions in Exhaust Gases from Incineration Processes." The protocol is presented in Appendix J.1. This method is applicable for the determination of PM emissions and Pb, Ni, Zn, P, Cr, Cu, Mn, Se, Be, Tl, Ag, Sb, Ba, Cd, As, and Hg emissions from various types of processes. The test samples were not analyzed for Hg because Hg was not expected in the process stream. Particulate emissions were based on the weight gain of the filter and the front half acetone rinses of the probe, nozzle, and filter holder. After the gravimetric analyses were completed, the sample fractions were analyzed for the target metals as discussed in Section 5.2.5.

5.1.1 Sampling Equipment for Particulate Matter and Metals

This methodology used the sampling train shown in Figure 5-1. The sampling train consisted of a quartz nozzle/probe liner followed by a heated filter assembly with a Teflon® filter support, a series of five impingers, and the standard EPA Method 5 meterbox and vacuum pump. The sample was not exposed to any metal surfaces in this train. Two of the sequential impingers contained a 5 percent nitric acid (HNO_3)/10 percent hydrogen peroxide (H_2O_2) solution and one contained silica gel. The first and fourth impingers were empty knockout impingers not required by the method, but added because of the high moisture content of the flue gas. The second impinger containing HNO_3/H_2O_2 was of the Greenburg-Smith design; the other impingers had straight tubes. The impingers were connected together with clean glass

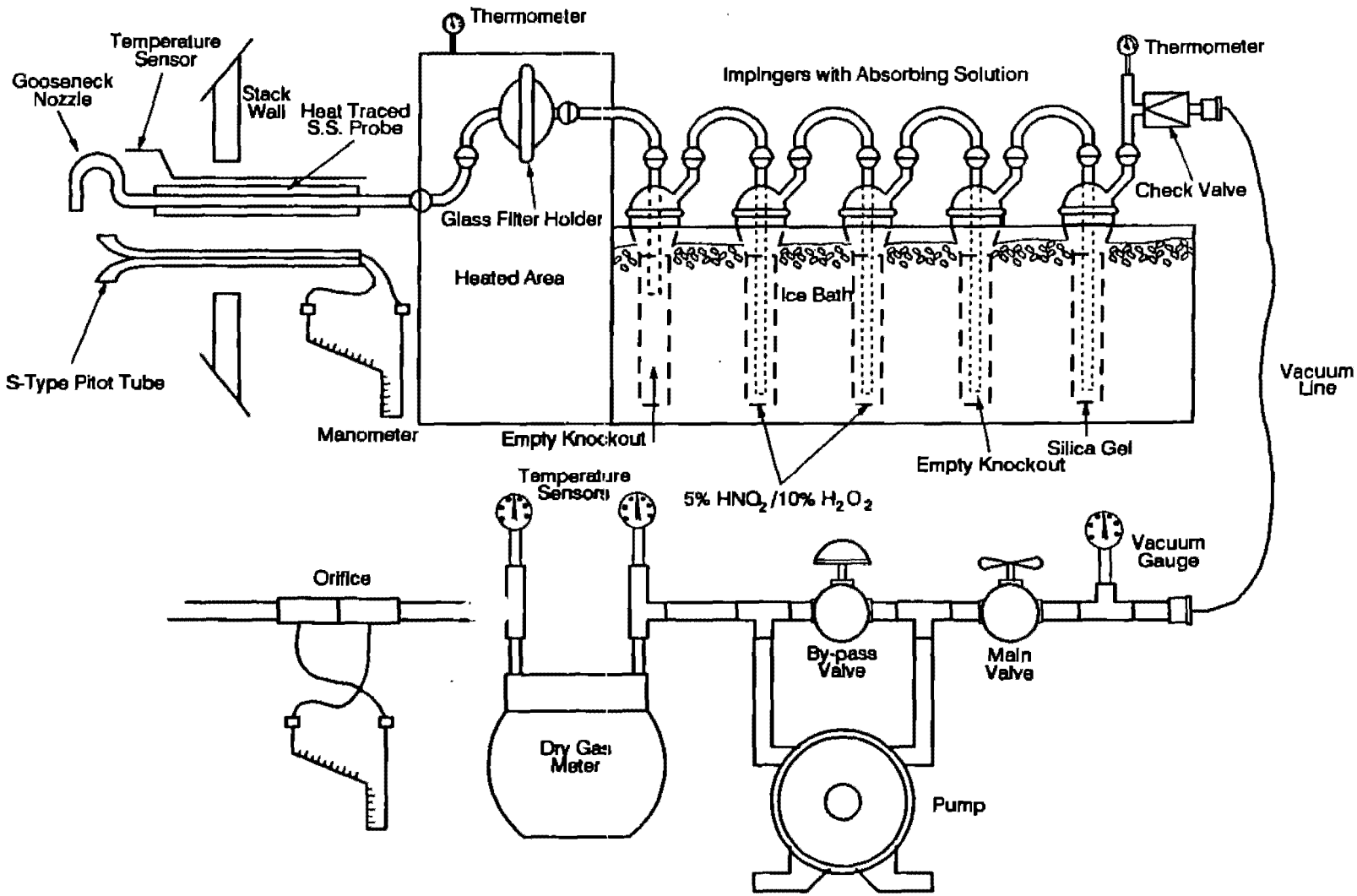


Figure 5-1. Schematic of Multiple Metals Sampling Train

5116047R

U-tube connectors and were arranged in an impinger bucket. Sampling train components were recovered and analyzed in separate front and back half fractions according to the described method.

5.1.2 Equipment Preparation for Particulate Matter and Metals Sampling

5.1.2.1 Glassware Preparation. Glassware was washed in hot, soapy water, rinsed three times with tap water and then rinsed three times with deionized distilled water. The glassware was then subjected to the following series of soaks and rinses:

- Soaked in a 10 percent HNO_3 solution for a minimum of 4 hours;
- Rinsed three times with deionized distilled water rinse; and
- Rinsed with acetone rinse.

The cleaned glassware was allowed to air dry in a contamination-free environment. The ends were then covered with parafilm. All glass components of the sampling train plus any other sample bottles, pipes, Erlenmeyer flasks, petri dishes, graduated cylinders, and other laboratory glassware used during sample preparation, recovery, and analysis were cleaned according to this procedure.

5.1.2.2 Reagent Preparation. The sample train filters were Pallflex Tissuequartz 2500QAS filters. The acids and H_2O_2 were Baker "Instra-analyzed" grade or equivalent. The H_2O_2 was purchased specifically for this test site.

The reagent water was Baker "Analyzed HPLC" grade or equivalent. The lot number, manufacturer, and grade of each reagent that was used were recorded in the laboratory notebook.

The HNO_3/H_2O_2 absorbing solution was prepared fresh daily according to Section 4.2.1 of the reference method. The analyst wore both safety glasses and protective gloves when the reagents were mixed and handled. Each reagent had its own designated transfer and dilution glassware. To avoid contamination, this glassware was marked for identification with a felt tip glass-marking pen and used only for the reagent for which it was designated.

5.1.2.3 Equipment Preparation. The remaining preparation included calibration and leak checking of all the train equipment, which included meterboxes, thermocouples, nozzles, pitot tubes, and umbilicals. Referenced calibration procedures were followed

when available, and the results were properly documented and retained. A discussion of the techniques used to calibrate this equipment is presented below.

Type-S Pitot Tube Calibration. The EPA has specified guidelines concerning the construction and geometry of an acceptable Type-S pitot tube. A pitot tube coefficient of 0.84 is used if the specified design and construction guidelines are met. Information pertaining to the design and construction of the Type-S pitot tube is presented in detail in Section 3.1.1 of EPA Document 600/4-77-027b. Only Type-S pitot tubes meeting the required EPA specifications were used. Pitot tubes were inspected and documented as meeting EPA specifications prior to field sampling.

Sampling Nozzle Calibration. Glass nozzles were used for isokinetic sampling. Calculation of the isokinetic sampling rate required that the cross sectional area of the sampling nozzle be accurately and precisely known. All nozzles were thoroughly cleaned, visually inspected, and calibrated according to the procedure outlined in Section 3.4.2 of EPA Document 600/4-77-027b.

Temperature Measuring Device Calibration. Accurate temperature measurements were required during source sampling. Thermocouple temperature sensors were calibrated using the procedure described in Section 3.4.2 of EPA document 600/4-77-027b. Each temperature sensor was calibrated at a minimum of two points over the anticipated range of use against an NBS-traceable mercury-in-glass thermometer. All sensors were calibrated prior to field sampling.

Dry Gas Meter Calibration. Dry gas meters (DGMs) were used in the sample trains to monitor the sampling rate and to measure the sample volume. All DGMs were calibrated to document the volume correction factor just before the equipment was shipped to the field. Post-test calibration checks were performed as soon as possible after the equipment has been returned to Research Triangle Park, North Carolina (RTP). Pre- and post-test calibrations agreed to within 5 percent. Prior to calibration, a positive pressure leak check of the system was performed using the procedure outlined in Section 3.3.2 of EPA document 600/4-77-237b. The system was placed under approximately 10 inches of water pressure and a gauge oil manometer was used to

determine if a pressure decrease was detected over a 1-minute period. If leaks were detected, they were eliminated before actual calibrations were performed.

After the sampling console was assembled and leak checked, the pump was allowed to run for 15 minutes. This allowed the pump and DGM to warm up. The valve was then adjusted to obtain the desired flow rate. For the pretest calibrations, data were collected at orifice manometer settings (ΔH) of 0.5, 1.0, 1.5, 2.0, 3.0 and 4.0 inches of H_2O. Gas volumes of 5 ft^3 were used for the two lower orifice settings, and volumes of 10 ft^3 were used for the higher settings. The individual gas meter correction factors were calculated for each orifice setting and averaged. The method required that each of the individual correction factors fall within ± 2 percent of the average correction factor or the meter was cleaned, adjusted, and recalibrated. In addition, Radian required that the average correction factor be within 1.00 ± 1 percent. For the post-test calibration, the meter was calibrated three times at the average orifice setting and vacuum used during the actual test.

Rockwell Model 175 DGMs in Research Appliance Company (RAC) enclosures were used for measuring gas sampling rates. The DGM calibrations were performed at Radian's RTP laboratory using an American wet test meter as an intermediate standard. The intermediate standard is calibrated every six months against the EPA spirometer at EPA's Emissions Measurement Laboratory in RTP.

5.1.3 Particulate Matter/Metals Sampling Operations

5.1.3.1 Preliminary Measurements. Before sampling began, preliminary measurements were required to ensure isokinetic sampling. These included determining the traverse point locations and performing a preliminary velocity traverse, cyclonic flow check, and moisture determination. These measurements were used to calculate a "K factor." The K factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements made during the pretest site survey were then checked for accuracy. Measurements were made of the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances. These measurements were used to verify sampling point locations by following EPA Reference

Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

5.1.3.2 Assembling the Train. Assembling the PM/metals sampling train components was initiated in the recovery trailer and final train assembly was completed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground-glass joint was carefully inspected for hairline cracks. The first impinger was a knockout impinger with a short tip. The purpose of this impinger was to collect condensate. The next two impingers were modified tip impingers, which each contained 100 ml of 5 percent HNO_2 and 10 percent H_2O_2. The fourth impinger was empty, and the fifth impinger contained 200 to 300 grams of blue-indicating silica gel. After the impingers were loaded, each impinger was weighed, and the initial weight and contents of each impinger were recorded on a recovery data sheet. The impingers were connected together by clean glass U-tube connectors and arranged in the impinger bucket. The height of all the impingers was approximately the same to obtain a leak free seal. The open ends of the train were sealed with parafilm or teflon tape.

The second step was to load the filter into the filter holder in the recovery trailer. The filter holder was then capped off and placed into the impinger bucket. A supply of parafilm and socket joints was also placed in the bucket in a clean plastic bag for use by the samplers. To avoid contamination of the sample, sealing greases were not used. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-1.

5.1.3.3 Sampling Procedures. After the train was assembled, the heaters for the probe liner and heated filter box were turned on. When the system reached the appropriate temperatures, the sampling train was ready for pretest leak checking. The filter skin temperature was maintained at 120 ±14°F (248 ±25°F). The probe temperature was maintained above 100°C (212°F).

The sampling trains were leak checked at the start and finish of sampling. (EPA Method 5 protocol required post-test leak checks and recommended pretest leak checks.) Radian protocol also incorporated leak checks before and after every port change. An

acceptable pretest leak rate was less than 0.02 acfm (ft^3/min) at approximately 15 inches of mercury (in. Hg). If, during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed, and after the train was reassembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

After a successful pretest leak check had been conducted, all train components were at their specified temperatures, and initial data were recorded (DGM reading), the test was initiated. Sampling train data were recorded periodically on standard data forms. A checklist for sampling is included in Table 5-1.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occurred during sampling were recorded on the task log such as pitot cleaning, thermocouple malfunctions, heater malfunctions, or any other unusual occurrences.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. (The post-test leak check procedure is identical to the pretest procedure; however, the vacuum should be at least 1 in. Hg higher than the highest vacuum attained during sampling.) An acceptable leak rate was less than 4 percent of the average sample rate, or 0.02 acfm (whichever was lower). If a final leak rate did not meet the acceptable criterion, the test run could still have been accepted upon approval of the test administrator.

5.1.4 Particulate Matter/Metals Sample Recovery

Recovery procedures began as soon as the probe was removed from the stack and the post-test leak check was completed.

Table 5-1

Sampling Checklist

Before Test Starts:

1. Check impinger set for correct order and number. Verify probe markings, and re-mark if necessary.

2. Verify that you have all the correct pieces of glassware.

3. Get data sheets and read barometric pressure.

4. Bag sampling equipment needs to be ready (with bags labeled and ready to go) if applicable.

5. Examine meter box; level it and confirm that the pump is operational.

6. Assemble train to the filter and leak check at 15 in Hg. Attach probe to train and do final leak check; record leak rate and pressure on sampling log.

7. Check out thermocouples; make sure they are reading correctly.

8. Turn on all heaters and check to see that they are increasing.

9. Leak check pitots.

10. Check that cooling water is flowing and on. Add ice to impinger buckets.

11. Check isokinetic K-factor; make sure it is correct. (Refer to previous results to confirm assumptions). (Two people should calculate this independently to double check it.)

12. Have a spare probe liner, probe sheath, meter box and filter ready to go at location.

Table 5-1

Continued

During Test:

1. Notify crew chief of any sampling problems immediately. Note problem on sampling log.

2. Perform simultaneous/concurrent testing with other locations (if applicable). Maintain filter temperature between 248°F ±25°F. Keep temperature as steady as possible. Maintain XAD trap and impinger temperatures below 68°F. Maintain probe temperature above 212°F.

3. Leak check between ports and record on sampling log.

4. Record sampling rate times and location for the fixed gas (CO, CO_2, O_2) sample (if applicable).

5. Blow back pitot tubes at inlet location every 15 minutes.

6. Change filter if pressure drop exceeds 15 in. Hg.

7. Check impinger solutions every 1/2 hr; if bubbling into impinger prior to silica gel, empty out first impinger into pre-weighed bottle and replace.

8. Check impinger silica gel every 1/2 hr; if indicator disappears request a pre-filled impinger from van lab and replace.

9. Check manometer fluid levels and zero every hour.

After Test Is Completed:

1. Record final meter reading.

2. Check completeness of data sheet.

3. Do final leak check of sampling train at 1 in Hg greater than maximum vacuum during test.

Table 5-1

Continued

4. Leak check each leg of pitot tubes.

5. Disassemble train. Cap sections. Take sections to recovery trailer.

6. Probe recovery (use 950 ml bottles)
   a) Bring probes into recovery trailer (or other enclosed area).
   b) For acetone rinses (all trains)
      - Attach flask to end of probe
      - Add about 50 mls of acetone
      - Put in brush down probe, and brush back and forth
      - Rinse back and forth in probe
      - Empty out acetone in sample jar
      - Do this 3 times
   c) For $MeCl_2$ rinses
      Rinse 3 times with flask attached (no brushing)

7. Reattach nozzle and cap for next day, store in dry safe place.

8. Make sure data sheets are completely filled out and give to location leader.

To facilitate its transfer from the sampling location to the recovery trailer, the sampling train was disassembled into three sections: the nozzle/probe liner, filter holder, and impingers in their bucket. Each of these sections was capped with Teflon® tape or parafilm before being transported to the recovery trailer.

Once in the trailers, the sampling train was recovered as separate front and back half fractions. Figure 5-2 is a diagram illustrating front half and back half sample recovery procedures. No equipment with exposed metal surfaces was used in the sample recovery procedures. The weight gain in each of the impingers was recorded to determine the moisture content in the flue gas. Following weighing of the impingers, the front half of the train was recovered, which included the filter and all sample-exposed surfaces forward of the filter. The probe liner was rinsed with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces were wetted. The acetone was quantitatively collected into the appropriate sample bottle. This rinse was followed by additional brush/rinse procedures using a nonmetallic brush; the probe was held in an inclined position and acetone was squirted into the upper end as the brush was pushed through with a twisting action. All of the acetone and particulate was caught in the sample container. This procedure was repeated until no visible particulate remained and was finished with a final acetone rinse of the probe and brush. The front half of the filter was also rinsed with acetone until all visible particulate was removed. After all front half acetone washes were collected, the cap was tightened, the liquid level marked and the bottle weighed to determine the acetone rinse volume. The method specifies that a total of 100 ml of acetone must be used for rinsing these components. However, a thorough rinse usually requires more reagent. For blank correction purposes, the exact weight or volume of acetone used was measured. An acetone reagent blank of approximately the same volume as the acetone rinses was analyzed with the samples.

The nozzle/probe liner, and front half of the filter holder was rinsed three times with 0.1N HNO_3 and the rinse was placed into a separate amber bottle. The container wascapped tightly, the weight of the combined rinse was recorded, and the liquid level

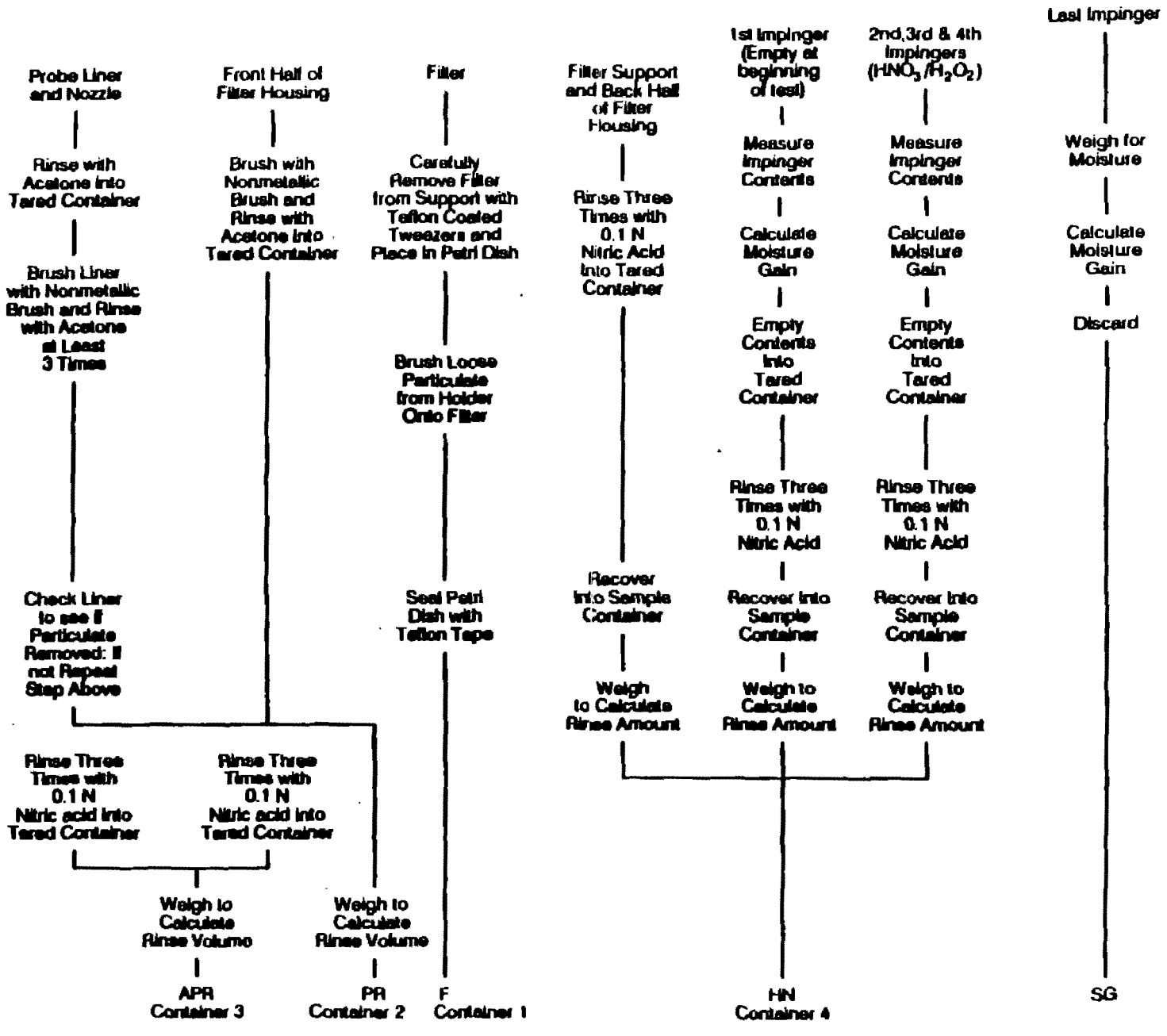


Figure 5-2. Metals Sample Recovery Scheme

was marked on the bottle. The filter was placed in a clean, well-marked glass petri dish and sealed with Teflon® tape.

Prior to recovering the back half impingers, the contents were weighed for moisture content. Any unusual appearance of the filter or impinger contents was noted in the logbook.

The contents in the knockout impinger was recovered into a preweighed, prelabeled bottle with the contents from the HNO_3/H_2O_2 impingers. These impingers and connecting glassware were rinsed thoroughly with 0.1N HNO_3, the rinse was captured in the impinger contents bottle, and a final weight was taken. Again, the method specifies a total of 100 ml of 0.1N HNO_3 be used to rinse these components. The weight of reagent used for rinsing was determined by weighing the impinger contents bottle before and after rinsing the glassware. A nitric acid reagent blank of approximately the same volume as the rinse volume was analyzed with the samples.

After final weighing, the silica gel from the train was saved for regeneration. The ground-glass fittings on the silica gel impinger were wiped off after sample recovery to ensure a leak tight fit for the next test.

A reagent blank was recovered in the field for each of the following reagents:

- Acetone blank--100-ml sample size;
- 0.1N HNO_3 blank--1000-ml sample size;
- 5 percent HNO_3/10 percent H_2O_2 blank--200-ml sample size;
- Dilution water--100-ml sample size; and
- Filter blank--one each.

Each reagent blank was from the same lot used during the sampling program. Each lot number and reagent grade was recorded on the field blank label and in the logbook.

The liquid level of each sample container was marked on the bottle in order to determine if any sample loss occurred during shipment. If sample loss had occurred, the

sample might have been voided or a method could have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

Approximate detection limits for the various metals of interest are summarized in Table 5-2.

5.1.5 Particulate Analysis

The general gravimetric procedure described in EPA Method 5, Section 4.3, was followed. Both filters and precleaned beakers were weighed to a constant weight before use in the field. The same balance used for taring was used for weighing the samples.

The acetone rinses were evaporated under a clean hood at 70°F to dryness in a tared beaker. The residue was desiccated for 24 hours in a desiccator containing fresh room temperature silica gel. The filter was also desiccated to a constant weight under the same conditions. Weight gain was reported to the nearest 0.1 mg. Each replicate weighing agreed to within 0.5 mg or 1 percent of total weight less tare weight, whichever was greater, between two consecutive weighings, and was at least 6 hours apart.

5.1.6 Metals Analytical Procedures

A diagram illustrating the sample preparation and analytical procedure for the target metals is shown in Figure 5-3.

The front half acetone and filter fractions were digested with concentrated HNO_3 and hydrofluoric acid (HF) in a microwave pressure vessel. The microwave digestion took place over a period of approximately 10 to 12 minutes in intervals of 1 to 2 minutes at 600 watts. The nitric probe rinse was digested by EPA SW 846 Method 3020. The digested filters and the digested probe rinses were combined to yield the front half sample fraction. The fraction was diluted to a specific volume with DI water and analyzed by applicable instrumentation.

The absorbing solutions from the HNO_3/H_2O_2 impingers were combined, acidified, and reduced to near dryness. The sample was then digested by conventional digestion, with 5 percent HNO_3. After the fraction has cooled, it was filtered and diluted to a specified volume with DI water.

Each sample fraction was analyzed by inductively coupled argon plasma spectroscopy (ICAP) using EPA Method 200.7. Interelement corrections were applied to

TABLE 5-2. APPROXIMATE DETECTION LIMITS

Metal	Method[a]	Analytical Detection Limits ($\mu g/ml$)	Instack Method Detection Limits[b]	
			Front Half (300 ml sample size) ($\mu g/m^3$)	Back Half (150 ml sample size) ($\mu g/m^3$)
Chromium	ICAP	0.006	1.4	0.7
Cadmium	ICAP	0.002	0.5	0.2
Arsenic[c]	GFAAS	0.004	1.0	0.5
Lead[c]	GFAAS	0.003	0.7	0.4
Nickel	ICAP	0.003	0.7	0.4
Barium	ICAP	0.001	0.2	0.1
Beryllium	ICAP	0.0001	0.2	0.1
Silver	ICAP	0.006	1.4	0.7
Antimony	ICAP	0.015	3.6	1.8
Thallium	ICAP	0.100	24	12
Zinc	ICAP	0.015	3.6	1.8
Copper	ICAP	0.004	1.0	0.5
Manganese	ICAP	0.003	0.7	0.4
Phosphorus	ICAP	0.300	72	36
Selenium	ICAP	0.005	1.2	0.6

[a]ICAP = Inductively Coupled Argon Plasma
GFAAS = Graphite Furnace Atomic Absorption Spectroscopy
CVAAS = Cold Vapor Atomic Absorption Spectroscopy

[b]These detection limits are based on a stack gas sample volume of 1.25 m^3. If 5 m^3 are collected, the instack detection method detection limits are 1/4 of the values indicated.

[c]If Fe and Al are present, samples will be diluted which may raise analytical detection limits.

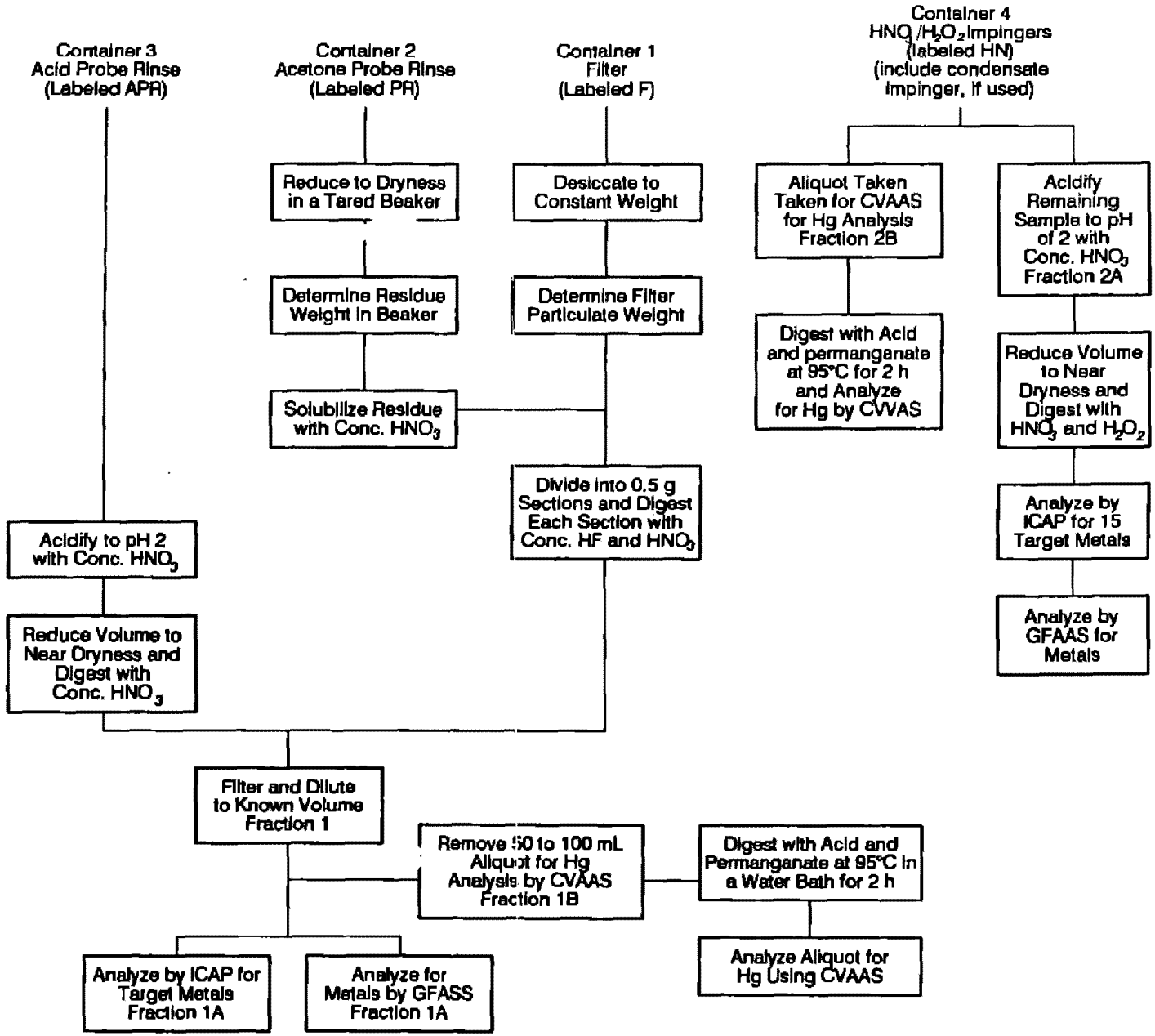


Figure 5-3. Metals Sample Preparation and Analysis Scheme

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the analytes to remove the effects of unwanted emissions. If arsenic or lead levels were less than 2 ppm, graphite furnace atomic absorption spectroscopy (GFAAS) was used to analyze for these elements by EPA Methods 7060 and 7421. Matrix modifiers such as specific buffering agents were added to these aliquots to make the matrix more volatile and/or stabilize the analyte element. The total volumes of the absorbing solutions and rinses for the various fractions were measured and recorded in the laboratory notebook.

5.1.7 Quality Control for Metals Analytical Procedures

All quality control (QC) procedures specified in the test method were followed. All field reagent blanks were processed, digested and analyzed as specified in the test method. To ensure optimum sensitivity in measurements, the concentrations of target metals in the solutions were at least 10 times the analytical detection limits.

5.1.7.1 Inductively Coupled Argon Plasma Spectroscopy Standards and Quality Control Samples. The QC procedures used for ICAP analysis include running two QC standards, and a calibration blank after every 10 samples. One interference check standard was analyzed at the beginning and the end of the analytical run. One duplicate analysis and one analytical spike were analyzed to check for precision and matrix effects.

Standards less than 1 μg/ml of a metal were prepared daily; those with concentrations greater than this were made monthly.

5.1.7.2 Graphite Furnace Standards and Quality Control Samples. Standards used for GFAAS analysis were matrix matched with the samples analyzed and the matrix modifiers added. Standards with less than 1 μg/ml of a metal were prepared daily; those with concentrations greater than this were made monthly. A minimum of five standards composed the standard curve. Quality control samples were prepared from a separate 10 μg/ml standard by diluting it into the range of the samples.

One analytical spike was analyzed for every 10 samples. If recoveries were below 80 percent of 100, the samples were analyzed by method of additions as explained in EPA SW 846 Method 7000. One QC sample was analyzed to verify the standard curve used to quanitate the samples.

5.2 EMISSIONS TESTING FOR PARTICULATE MATTER LESS THAN 10 MICRONS/CONDENSIBLE PARTICULATE MATTER

The sampling method for fine particulate matter/condensible particulate matter (PM_{10}/CPM) was a combination of the protocols outlined in EPA Method 201A [entitled "Determination of PM_{10} Emissions (Constant Sampling Rate Procedure)"] and EPA Method 202 (entitled "Determination of Condensible Emissions from Stationary Sources"). These methods are presented in Appendix J.2, and are summarized below. Method 201A is applicable to the measurement of PM emissions with aerodynamic diameters less than or equal to 10 microns (PM_{10}). Method 202 applies to the determination of CPM from various types of combustion devices. Condensible PM emissions are gaseous matter and aerosols that condense after passing through a filter that captures liquid and solid particulates. Analyses of the test samples were performed for total PM (including PM greater than 10 μm), PM_{10}, and CPM. Total PM emission rates were determined from the PM/metals train.

Particulate matter emissions larger than 10 microns were determined by measuring the weight of the catch of an in-stack PM_{10} cyclone. The PM_{10} emissions were determined from the weight gain of an in-stack backup filter that was downstream of the cyclone. The CPM emissions were determined from the evaporated residue of the impinger solution, as outlined in Section 5.2.5.2

5.2.1 Sampling Equipment for PM_{10}/CPM

Figure 5-4 shows the sampling train for the PM_{10}/CPM method, which combined the in-stack cyclone, filter assembly, and probe from EPA Method 201A with the impinger assembly from EPA Method 202. The sample train consisted of a tapered stainless steel inlet nozzle, an in-stack PM_{10} cyclone, a backup filter holder and filter behind the cyclone, a heated glass probe liner, a series of 4 impingers, and the standard EPA Method 17 meterbox and vacuum pump.

The instrument used in PM_{10} determination was a Sierra Instruments Series 280 Cyclade™ cyclone. This device collected particulates larger than 10 microns and allowed particulates smaller than 10 microns to pass through to a backup filter. The cyclone

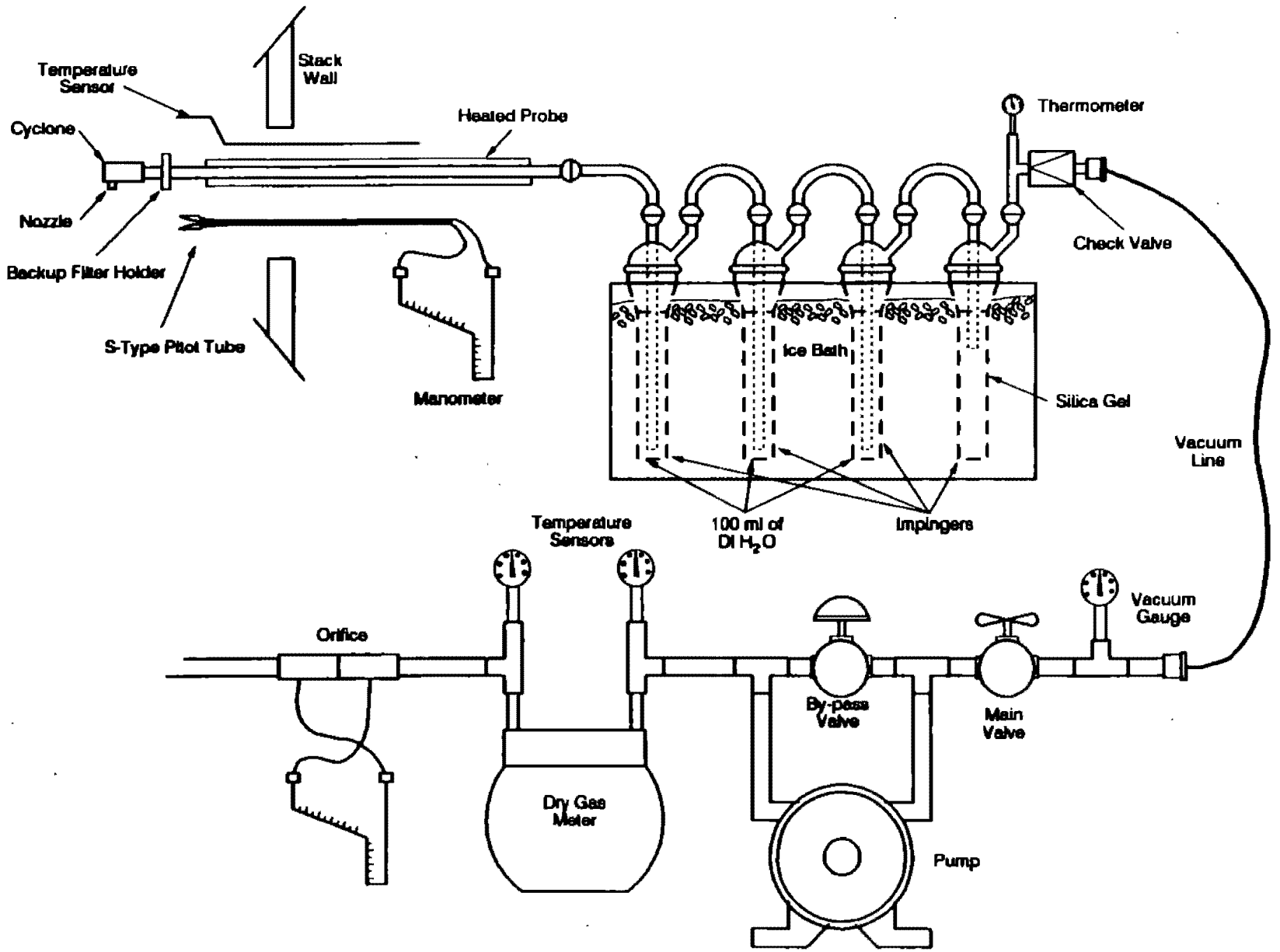

Figure 5-4. PM/CPM Sampling Train

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caused the gas stream to swirl in a vortex; larger particulates contacted the cyclone wall and fell into a collection cup.

The in-stack backup filter used after the cyclone had a demonstrated collection efficiency of greater than 99.95 percent on diocytylphepthalate (DOP) smoke particles, as required by ASTM Standard Method D 2986.

As outlined in EPA Method 202, the first two impingers each contained 100 ml of deionized distilled H_20, the third impinger was empty, and the fourth contained silica gel. The first two impingers were of the Greenburg-Smith design with standard tips; the other impingers had straight tubes. The impingers were connected together with clean glass U-tube connectors.

5.2.2 PM_{10}/CPM Sampling Equipment Preparation

5.2.2.1 Glassware Preparation. Glassware was washed as follows:

- Washed in hot soapy water;
- Rinsed with tap water;
- Rinsed with deionized distilled water;
- Rinsed with acetone; and
- Rinsed with methylene chloride ($MeCl_2$).

The cleaned glassware was allowed to air dry in a contamination-free environment. After drying, the ends were covered with parafilm. All glass components of the sampling train plus any sample bottles, pipets, Erlenmeyer flasks, petri dishes, graduated cylinders, and other laboratory glassware used during sample preparation, recovery, and analysis were cleaned according to this procedure.

The cyclone housing, nozzle, and interior surfaces were cleaned with hot, soapy water, rinsed with hot tap water, rinsed with distilled deionized water, and finally rinsed with acetone and dried.

5.2.2.2 Reagent Preparation. The deionized distilled reagent water used conformed to the American Society for Testing and Materials Specification D 1193-74, Type II.

5.2.2.3 Equipment Preparation. All measuring devices used during sampling were calibrated prior to use, as specified in EPA Method 17. This equipment included top loading scales, probe nozzles, pitot tubes, metering system, probe heater, temperature gauges, dry gas metering system, and barometer. A laboratory field notebook was maintained to record these calibration values.

Before they were used, all filters were desiccated and tared on a five-place balance. Replicate weighings at least 6 hours apart must agree to within 0.5 mg to yield an acceptable weight. Each filter was then stored in an individual petri dish with an identification number, and all data were recorded in the logbook.

5.2.3 Sampling Operations for PM_{10}/CPM

The sampling procedure for the PM_{10}/CPM method is similar to the procedure for EPA Method 5, except that the PM_{10}/CPM method includes a post-test nitrogen (N_2) purge to purge SO_2 from the sample, if considerable amounts of SO_2 are present in the flue gas. Also, a different method was used for nozzle size selection and sampling time, and no silicone grease was used in assembling the sample train in order to avoid contamination.

Prior to sampling for PM_{10}, a preliminary velocity traverse was performed. Moisture content, flue gas molecular weight, and temperature were determined using EPA Methods 1 through 4. These data were used to determine the appropriate sampling rate (as outlined in EPA Method 201A) through the cyclone and to select an appropriate sampling nozzle or nozzles. Since a constant sampling rate was required throughout a given run, more than one nozzle was required to maintain approximate isokinetic sampling conditions. In preparation for sampling, the tester calculated an appropriate nozzle size for each anticipated range of pitot readings (delta P), such that isokinetics could be maintained within ±20 percent of the constant sampling rate.

In addition to the above mentioned preliminary data, particulate loading was also known in order to calculate the required run duration to achieve a representative sample

in the cyclone. Because of the complexity of the PM_{10} method, only experienced samplers performed the sample tests.

The impinger train was prepared according to EPA Method 5. Teflon tape was used to provide leak-free connections between glassware. The impingers and impinger contents were weighed and the weights recorded. The sample train components were carefully assembled in the recovery trailer except for attachment of the cyclone, backup filter, and probe, which was performed at the stack sampling location.

The train was assembled at the stack location by connecting the cyclone, filter, and probe liner to the impinger train, which was connected to the meterbox. After the probe and filter heaters were turned on, the train was leak checked at 15 in. Hg. The leak rate must be below 0.02 cfm.

The samples were withdrawn at a constant flow rate from the stack at the traverse points determined by EPA Method 1. The sampling time at each point was based on the relative gas velocity at that point. A leak check was performed before and after each sample test. Parafilm or Teflon tape was used to seal the train components at the end of each test. As soon as possible after the post-test leak check, the probe was disconnected from the impinger train.

5.2.4 Sample Recovery for PM_{10}/CPM

Recovery procedures began as soon as the probe was removed from the stack at the end of the sampling period. The recovery scheme is shown in Figure 5-5. To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into four sections: the cyclone, the filter holder, the nozzle/probe liner, and the impingers in their bucket. Each of these sections was capped with parafilm or Teflon tape before being transported to the recovery trailer.

5.2.4.1 Cyclone Recovery. The cyclone was disassembled and the nozzle removed. The PM was quantitatively recovered from the interior surfaces of the nozzle, cyclone, and collection cup (excluding the exit tube) by brushing with a nylon bristle brush and rinsing with acetone until the rinse showed no visible particles. After this procedure, a final rinse of the cyclone surfaces and brush was performed. All particulate and acetone rinse was collected in a sample jar and sealed. The liquid level was marked,

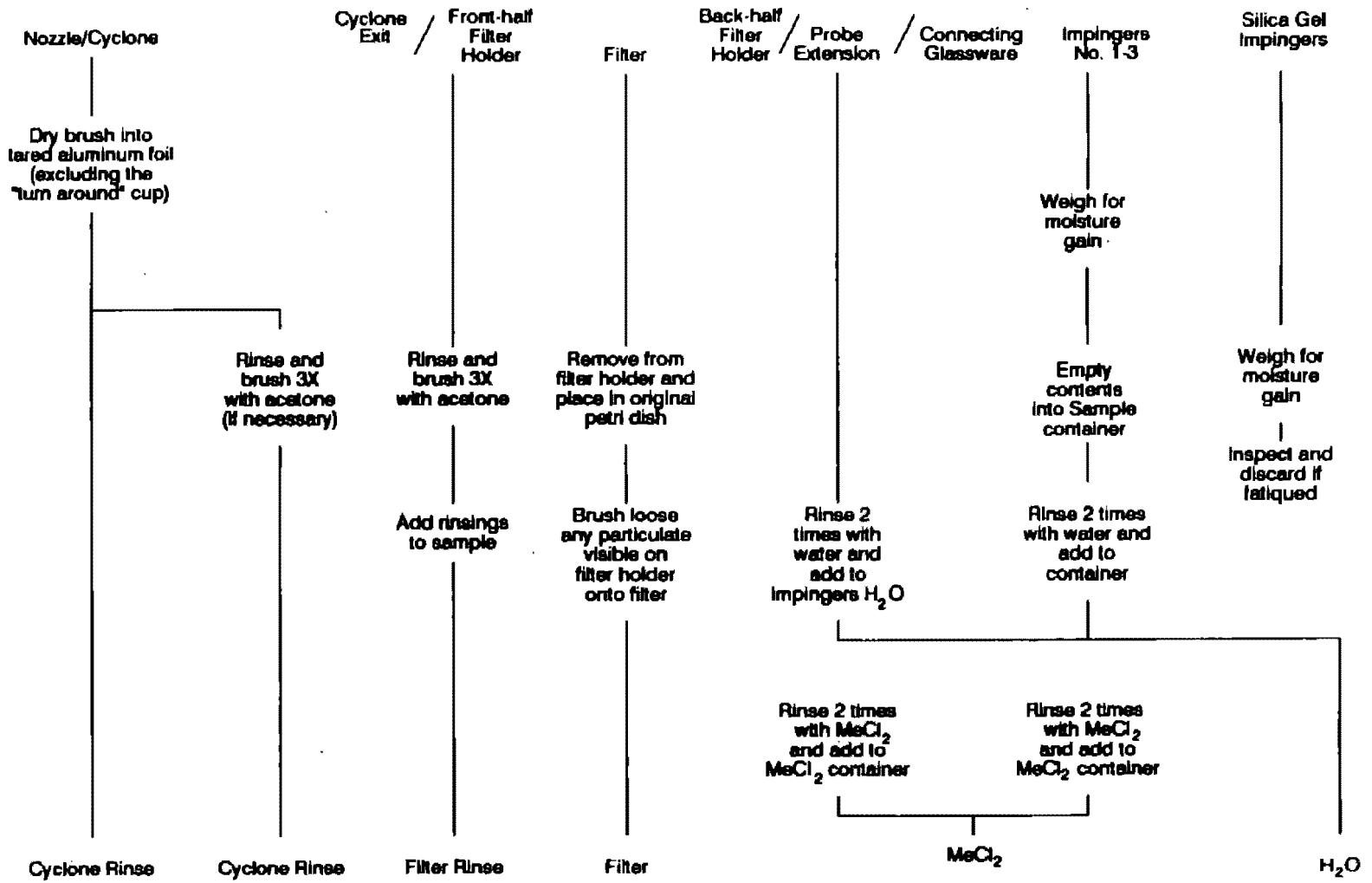

Figure 5-5. PM_{10}/CPM Sample Recovery Scheme

and the jar was identified. This information was logged into the field notebook.

The above procedure was repeated for all interior surfaces from the exit tube to the front half of the in-stack filter. The acetone rinse was collected in a separate sample jar, sealed, identified, the liquid level was marked, and the sample information was logged into the field notebook.

5.2.4.2 In-stack Filter Recovery. The in-stack filter holder was opened and the filter was removed with tweezers or rubber gloves. The filter was placed in a marked petri dish sealed with Teflon tape, and the filter number was logged into the field notebook.

5.2.4.3 Probe and Impingers Recovery. The weight or volume gain in each of the impingers was recorded and used to determine the moisture content in the flue gas. The liquid from the three impingers was transferred into a clean glass sample jar. The impinger bottles, back half of the filter holders, and probe liner were rinsed twice with water, the rinse water was added to the same sample bottle, and the liquid level was marked on the bottle.

Following the water rinses, the impingers, filter holder, and probe were rinsed twice with $MeCl_2$. The $MeCl_2$ rinse was saved in a clean glass sample jar and the liquid level was marked. The sample information was logged into the field notebook.

All sample jars were fully identified and sealed. Pertinent information was logged into the field notebook.

5.2.4.4 Field Blanks. Field blanks of water (500 ml), $MeCl_2$ (a volume approximately equal to the volume used for the $MeCl_2$ rinses), and acetone (200 ml) were taken. Each reagent blank was of the same lot as was used during the sampling program. Each lot number and reagent grade were recorded on the field blank label and recorded into the field notebook.

5.2.5 Analysis for PM_{10}/CPM

The PM_{10}/CPM gravimetric analyses were completed as shown in Figure 5-6. Sample jars were checked to ascertain if leakage during shipment had occurred. If sample loss occurred during shipment, the sample may have been voided or a method may have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

5.2.5.1 Cyclone Catch Analysis. The dry cyclone catch, stored in foil; the cyclone rinse; and the front half filter rinses were analyzed gravimetrically according to EPA Method 5. Each rinse was evaporated at 70°F in a tared beaker to dryness. The residue was then desiccated at room temperature for 24 hours to a constant weight in a desiccator containing anhydrous calcium sulfate. To be considered constant weight, each replicate weighing must agree to within 0.5 mg and must be at least 6 hours apart. Weight gain for each fraction was reported to the nearest 0.1 mg. This weight gain constituted the PM greater than 10 microns in size.

5.2.5.2 Filter Catch Analysis. The in-stack filter catch was analyzed gravimetrically according to EPA Method 5 requirements.

For each filter, the filter and loose particulates were transferred to a tared glass weighing dish and dried for 24 hours in a desiccator containing silica gel. The sample was weighed to a constant weight, with results reported to the nearest 0.1 mg. The resulting weight gain from the filter and exit tube acetone rinses constituted the noncondensible PM_{10} portion of the sample.

5.2.5.3 Impinger and Probe Sample Rinse Analysis. Data were recorded on the data sheet shown in Figure 5-7. The water sample was measured volumetrically.

The $MeCl_2$ sample was combined with the water sample in a 1000-ml separatory funnel. After mixing, the aqueous and organic phases were allowed to separate; most of the organic/$MeCl_2$ phase was drained off and collected in a tared 350-ml weighing tin (approximately 100 ml). Then 75 ml of $MeCl_2$ was added and mixed; again most of the organic $MeCl_2$ was drained into the weighing tin. This procedure was repeated with another 75 ml of $MeCl_2$. A total of approximately 250 ml of organic extract was drained into the weighing tin. No water was drained during this procedure.

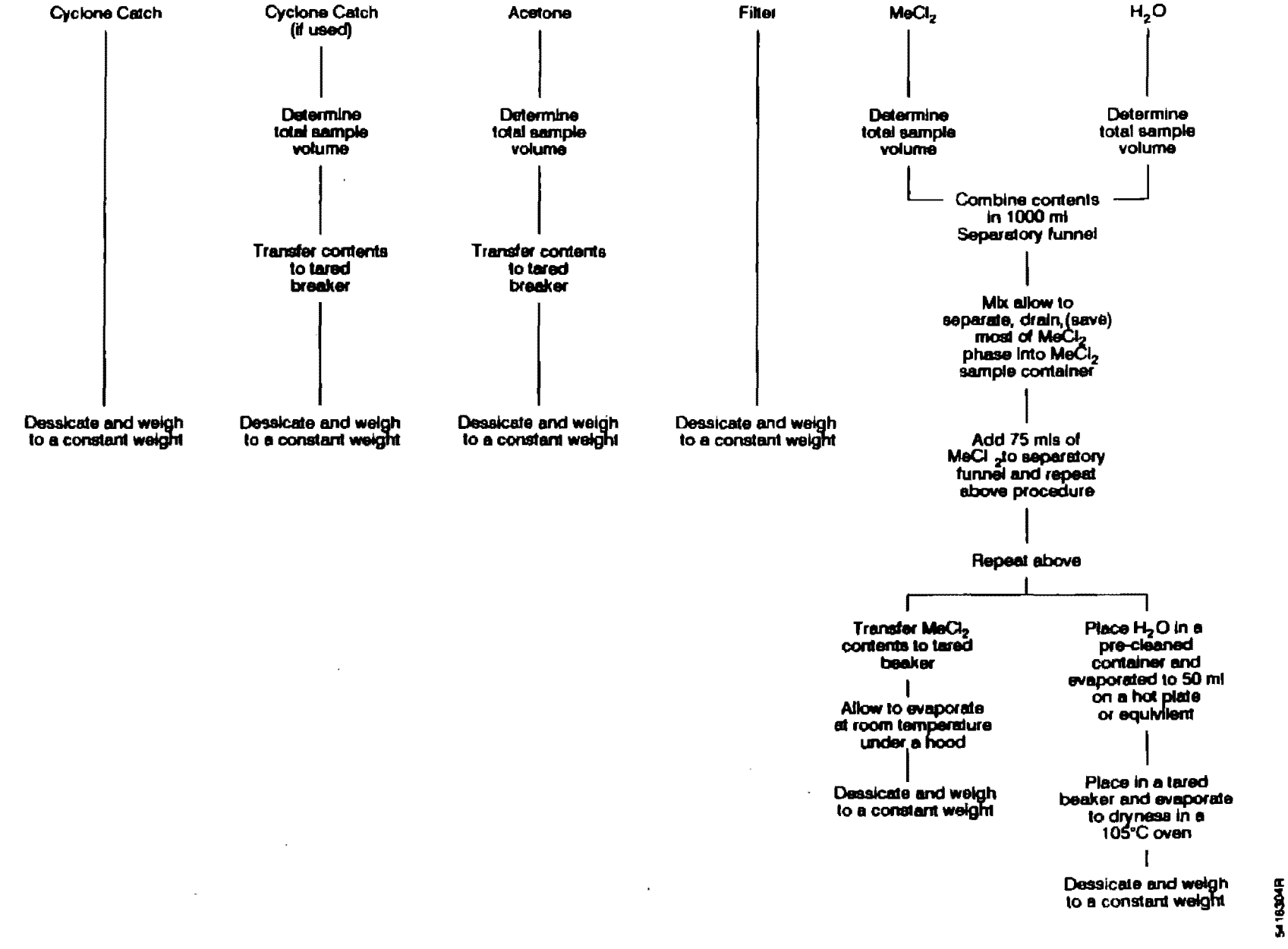

Figure 5-6. PM_{10}/CPM Analytical Scheme

Moisture Determination

Volume or weight of liquid in impingers _____ ml or g
Weight of moisture in silica gel _____ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport _____ ml
Final volume _____ ml
pH of sample prior to analysis _____
Addition of NH_4OH required? _____
Sample extracted 2X with 75 ml $MeCl_2$? _____

For Titration of Sulfate

Normality of NH_4OH _____ N
Volume of sample titrated _____ ml
Volume of titrant _____ ml

Sample Analysis

Container number	Weight of Condensible Particulate, mg		
	Final Weight	Tare Weight	Weight Gain
4 (Inorganic) 4 & 5 (Organic)			
		Total	
		Less Blank	
		Weight of Condensible Particulate	

Figure 5-7. Analytical Data Sheet

Organic Fraction Weight Determination

The organic extract was evaporated under a laboratory hood. Following evaporation, it was dried for 24 hours in a desiccator containing silica gel. The resulting sample was weighed to the nearest 0.1 mg.

Inorganic Fraction Weight Determination

The water sample was evaporated on a hot plate to approximately 50 ml, then evaporated to dryness in a 105°C oven. Because no N_2 recovery purge was used, the sample was then desiccated and weighed to a constant weight.

5.2.5.3 Field Blank Analysis. The acetone field blank was measured gravimetrically and transferred to a 250-ml beaker. The sample was evaporated to dryness, desiccated for 24 hours, and weighed to a constant weight.

The $MeCl_2$ and water field blanks were analyzed as described in Sections 1.2.5.2.1 and 1.2.5.2.2 of the test method, respectively. Blank correction was not required, because the sum of the values for the water blank and the $MeCl_2$ blank was less than 2 mg or 5 percent of the mass of the CPM, whichever is greater.

5.3 ALDEHYDES EMISSIONS TESTING

Sampling for aldehydes was performed according to EPA SW-846 Test Method 0011, "Sampling for Aldehyde and Ketone Emissions from Stationary Sources."

5.3.1 Sampling Equipment for Aldehydes

This methodology used the sampling train shown in Figure 5-8. The four-impinger train consisted of a quartz nozzle/probe liner followed by a series of impingers and the standard EPA Method 5 meterbox and vacuum pump. The contents of the sequential impingers were: the first two impingers with 2,4-dinitrophenylhydrazine (DNPH), the third impinger empty, and the fourth impinger with silica gel. The first, third, and fourth impingers were of the Greenburg-Smith design; the second impinger had a straight tube. The impingers were connected together with clean glass U-tube connectors. Sampling train components were recovered and analyzed in several fractions in accordance with the described method.

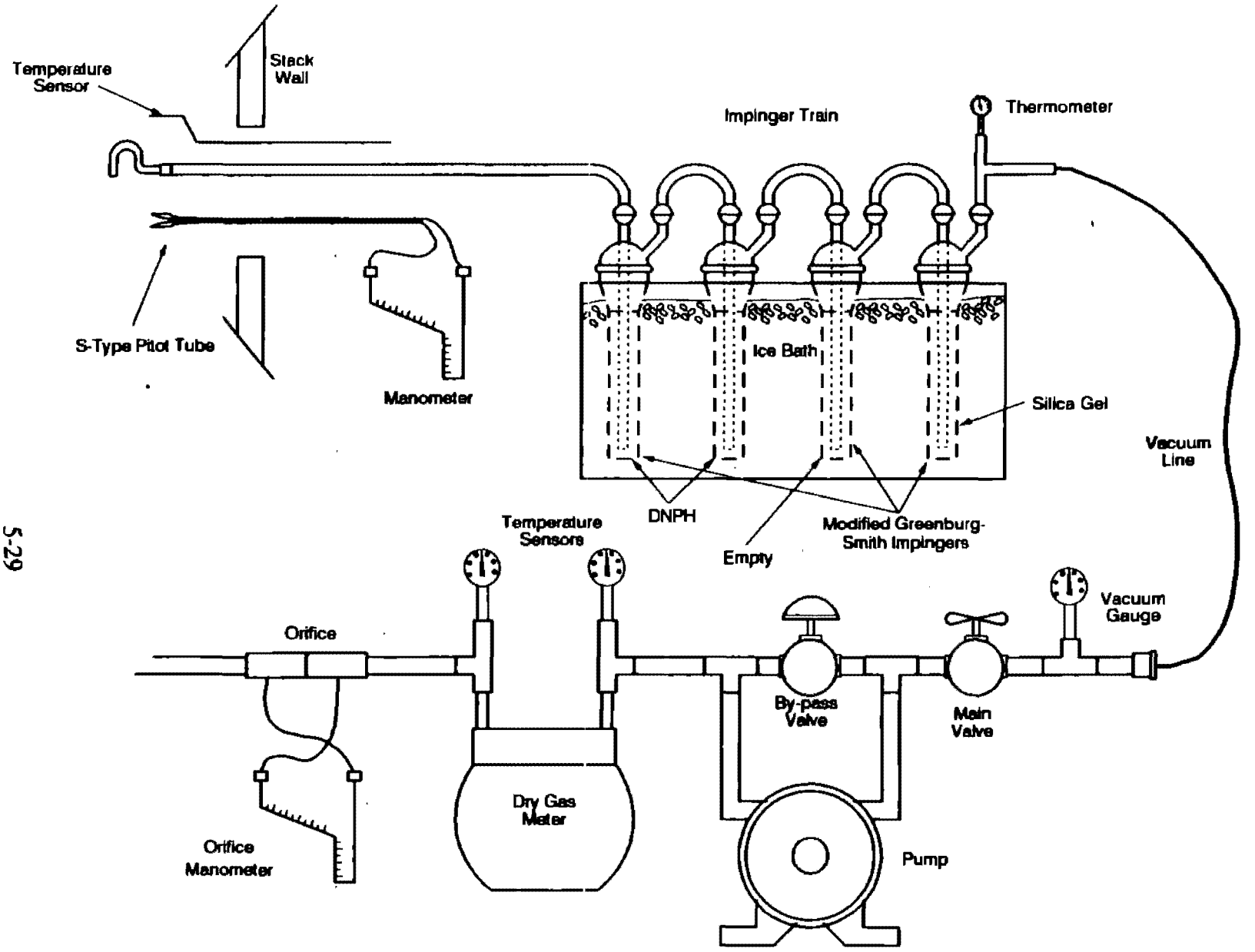

Figure 5-8. Aldehyde Sampling Train

5.3.2 Sampling Equipment Preparation for Aldehydes

5.3.2.1 Glassware Preparation. Glassware was washed in hot, soapy water, rinsed with tap water three times, and then rinsed with deionized distilled water three times. The glassware was then rinsed with methylene chloride, drained, dried, and heated in a laboratory oven at 130°C for several hours. Solvent rinses with methanol were substituted for the oven heating. After drying and cooling, glassware was stored in a clean environment to prevent any accumulation of dust or other contaminants.

5.3.2.2 Reagent Preparation. Reagent grade chemicals were used in all tests and conformed to the specifications of the Committee on Analytical Reagents of the American Chemical Society.

The reagent water was organic-free reagent water. The lot number, manufacturer, and grade of each reagent that was used were recorded in the laboratory notebook.

The DNPH absorbing solution was prepared according to Section 3.5.5.4.2 of the reference method. The analyst wore plastic gloves and safety glasses when handling DNPH crystals or solutions. Reagent bottles for storage of cleaned DNPH derivatizing solution were rinsed with acetonitrile and dried before use.

5.3.2.3 Equipment Preparation. The remaining preparation included calibration and leak checking of all train equipment as specified in EPA Method 5. This equipment included probe nozzles, pitot tubes, metering system, probe heater, temperature gauges, leak check metering system, and barometer. A field laboratory notebook was maintained to record these calibration values.

5.3.3 Aldehydes Sampling Operations

5.3.3.1 Preliminary Measurements. Prior to sampling, preliminary measurements were required to ensure isokinetic sampling. These included determining the traverse point locations, performing a preliminary velocity traverse, cyclonic flow check, and moisture determination. These measurements were used to calculate a K factor. The K-factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements were then made to verify the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances.

These measurements were then used to determine sampling point locations by following EPA Reference Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

5.3.3.2 Assembling the Train. Initial assembly of the aldehyde sampling train components was completed at the recovery trailer. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground glass joint was carefully inspected for hairline cracks. The first impinger was of the Greenburg-Smith design and contained DNPH. The second impinger was a straight tube and also contained DNPH. The third impinger, of the Greenburg-Smith design, was empty, and served as a knockout to collect condensate. The fourth impinger contained 200 to 300 grams of blue indicating silica gel.

After the impingers were loaded, each impinger was weighed, and the initial weight and contents of each impinger was recorded on a recovery data sheet. Final assembly of the sampling train components was completed at the stack location. The impingers were connected using clean, glass U-tube connectors. The height of all impingers was approximately the same to obtain a leak-free seal. The open ends of the train were sealed with ground-glass caps.

5.3.3.3 Sampling Procedures. After the train was assembled, the heaters for the probe liner were turned on. When the system reached the appropriate temperature, the sampling train was ready for pretest leak checking. The probe temperature was maintained above 100°C (212°F). The sampling trains were leak checked at the start and finish of sampling. An acceptable pretest leak rate was less than 0.02 acfm (ft^3/min) at approximately 15 in. Hg. If, during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed and after the train was reassembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled through the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the

train was systematically checked by first capping the train at the first impinger, the second impinger, etc., until the leak was located and corrected.

After a successful pretest leak check had been conducted, all train components were at their specified temperatures, and initial data were recorded (DGM reading), the test was initiated. Sampling train data were recorded periodically on standard data forms.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other occurences during sampling were recorded on the task log, such as pitot cleaning, thermocouple malfunctions, heater malfunctions, or any other unusual occurrence.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. The procedure was identical to the pretest procedure, but the vacuum should have been at least one in. Hg higher than the highest vacuum attained during sampling. An acceptable leak rate was less than 4 percent of the average sample rate or 0.02 acfm (whichever is lower). If a final leak rate did not meet the acceptable criterion, the test run may still have been accepted upon approval of the test administrator.

5.3.4 Aldehydes Sample Recovery

Recovery procedures began as soon as the probe was removed from the stack and the post-test leak check was completed.

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into two sections: the nozzle/probe liner, and the impingers in their bucket. Each of these sections was capped before being removed to the recovery trailer.

Once in the trailer, the entire sampling train was recovered into one sample container. The weight gain in each of the impingers was recorded to determine the moisture content in the flue gas. Following weighing of the impingers, the nozzle/probe was recovered. The probe liner was rinsed with methylene chloride by tilting and rotating the probe while squirting methylene chloride into its upper end so that all inside

surfaces were wetted. The methylene chloride was quantitatively collected into the sample container. This rinse was followed by additional brush/rinse procedures; the probe was held in an inclined position and methylene chloride was squirted into the upper end as the brush was pushed through with a twisting action. The procedure was performed three times. The brush was also rinsed with methylene chloride and the washing liquid was quantitatively collected in the sample container.

The first three impingers were then rinsed three times with methylene chloride and the washing was collected in the same sample container that was used for the probe. There were at least two liquid phases in the impingers. This two-phase mixture did not pour well and a significant amount of the impinger catch was left on the walls after the methylene chloride rinse. The use of water as a final rinse helped make the recovery quantitative.

After all methylene chloride and water washing and particulate matter had been collected in the sample container, the lid was tightened so solvent, water, and DNPH reagent did not leak out.

A sample blank was prepared by using an amber flint glass container and adding a volume of DNPH reagent and methylene chloride equal to the total volume in the first container.

The silica gel from the train was saved in a bag for regeneration after the job was completed. The ground-glass fittings on the silica gel impinger were wiped off after sample recovery to ensure a leak-tight fit for the next test.

The liquid level of each sample container was marked on the bottle in order to determine if any sample loss occurred during shipment. If sample loss had occurred, the sample would be voided or a method would have been used to incorporate a correction factor to scale the final results depending on the volume of the loss.

5.3.5 Aldehydes Analysis

The methylene chloride extract was solvent exchanged into acetonitrile prior to HPLC analysis. Liquid chromatographic conditions are described which permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

5.3.6 Quality Assurance for Aldehydes

The quality assurance (QA) program required for this method included the analysis of the field and method blanks, procedure validations, and analysis of field spikes. The assessment of combustion data and positive identification and quantitation of formaldehyde were dependent on the integrity of the samples received and the precision and accuracy of the analytical methodology. The QA procedures for this method were designed to monitor the performance of the analytical methodology and to provide the required information to take corrective action if problems were observed in laboratory operations or in field sampling activities.

Field blanks were submitted with the samples collected at each sampling site. The field blanks included the sample bottles containing aliquots of sample recovery solvents, methylene chloride and water, and unused DNPH reagent. At a minimum, one complete sampling train was assembled in the field staging area, taken to the sampling area, and leak checked at the beginning and end of testing. The probe of the blank train was heated during the sample test. The train was recovered as if it were an actual test sample. No gaseous sample was passed through the blank sampling train.

To evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory, a method blank was prepared for each set of analytical operations.

A field spike was performed by introducing 200 μl of the field spike standard into an impinger containing 200 ml of DNPH solution. Standard impinger recovery procedures were followed and the spike was used as a check on field handling and recovery procedures. An aliquot of the field spike standard was retained in the laboratory for derivatization and comparative analysis.

5.4 NONMETHANE HYDROCARBON ANALYSIS BY METHOD 25A AND C1-C6 BY METHOD 18

Benzene, toluene, and xylene concentrations were determined according to EPA Method 18. Total gaseous hydrocarbon (THC) concentrations were determined according to EPA Method 25A. Methane concentrations were determined by subtracting the results of EPA Method 18 from EPA Method 25A.

The instrument used to determine THC utilized a flame ionization detector (FID). For FIDs, the flue gas entered the detector and hydrocarbons were combusted in a hydrogen flame. The ions and electrons formed in the flame entered an electrode gap, decreased the gas resistance, and permitted a current flow in an external circuit. The resulting current was proportional to the instantaneous concentration of the total hydrocarbons.

The flue gas was analyzed by a Ratfisch Model 55 analyzer. The analyzer utilized a FID. The results are reported on a methane basis. Methane was used as the calibration gas.

EPA Method 18 analysis was performed using gas chromatography (GC) to separate the hydrocarbon (C_1 - C_6) species present in the gas stream. Prior to sampling of the source gas, the GC/FID system was calibrated with standard gas mixtures containing each hydrocarbon (CH_4, C_2H_6, C_3H_6, C_4H_{10}, C_5H_{12}, and C_6H_{14}) to establish calibration curves and retention times. The calibration curves and retention times were used to quantify the concentrations in the source samples.

A heat-traced slipstream from the stack was transferred to the Shimadzu Mini2 GC/FID to prevent any condensation of the sample gas. The gas sampling loop of the GC/FID was also heated and was purged each time a sample was analyzed. A schematic of the CEM and GC system is shown in Figure 5-9. Each analysis was approximately 10 to 15 minutes in duration. Thus, this analysis was semicontinuous with a result being generated approximately every 5 to 10 minutes during the sampling period.

The source sample was drawn into the GC sampling loop under conditions that prevented any condensation of the sample gas. The sample was injected into the GC and the hydrocarbon compounds were separated by absorbing them onto the column and

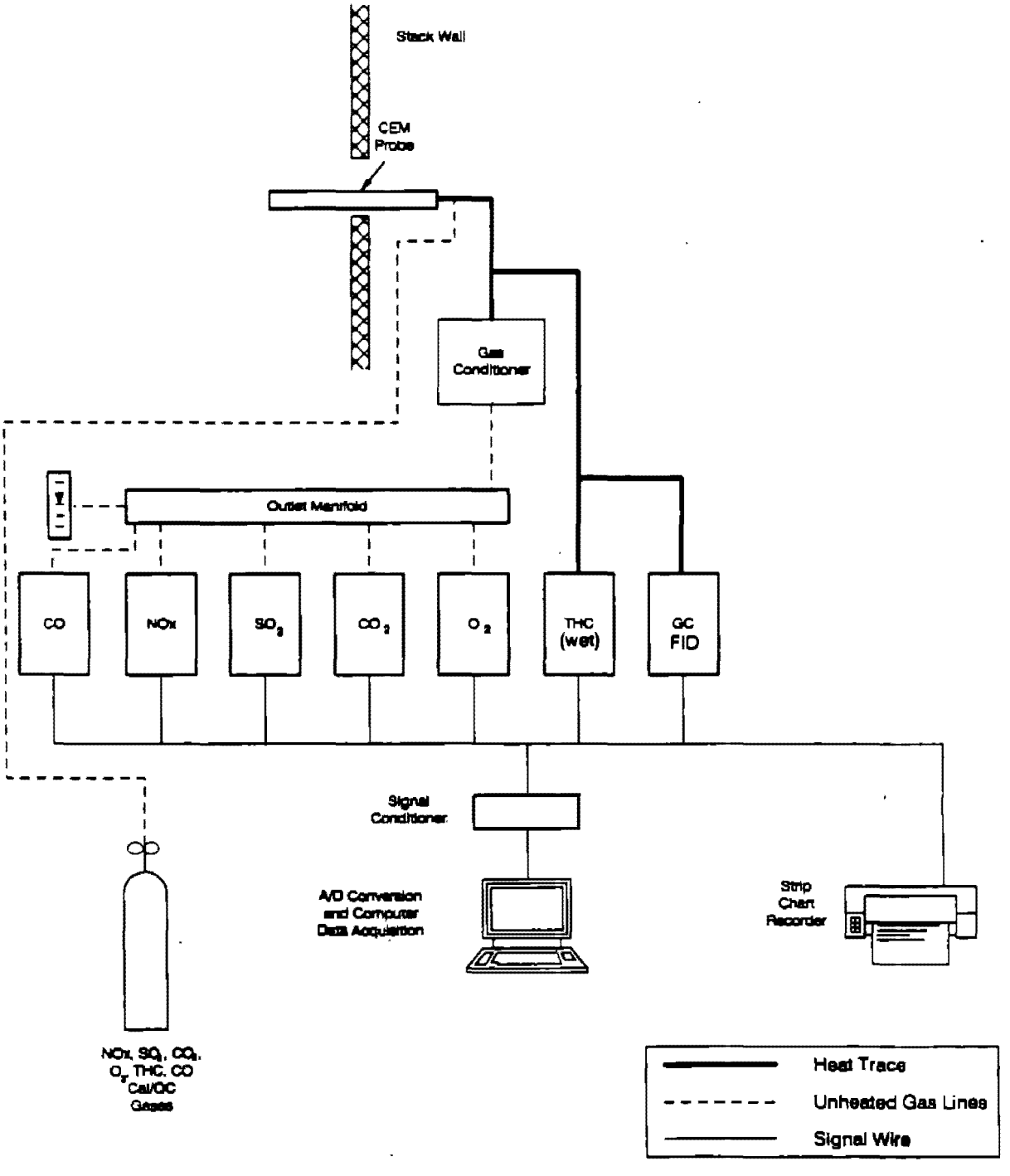

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Figure 5-9. Schematic of CEM System

desorbing them at different times. As each hydrocarbon compound was eluted, it was combusted in the hydrogen flame of the FID. The ions and electrons formed in the flame entered an electrode gas, decreased the gas resistance, and permitted a current flow in an external circuit. The resultant current was proportional to the instantaneous concentration of the hydrocarbon.

The response and retention times of the individual hydrocarbons were recorded on a strip chart recorder. A built-in integrator measured the peak areas and printed out the retention times and counts. The peaks were identified from the established retention times and the concentration of each hydrocarbon was determined by referring to the calibration curve.

The nonmethane hydrocarbon concentration was calculated by subtracting the average methane concentration as measured by GC/FID (EPA Method 18) from the average total hydrocarbon concentrations by EPA Method 25A.

5.5 EPA METHODS 1-4

5.5.1 Traverse Point Location By EPA Method 1

The number and location of sampling traverse points necessary for isokinetic and flow sampling was dictated by EPA Method 1 protocol. These parameters were based upon how much duct distance separated the sampling ports from the closest downstream and upstream flow disturbances. The minimum number of traverse points for a square duct of this size was 28. A set of perpendicular sampling ports was established in the stack.

5.5.2 Volumetric Flow Rate Determination by EPA Method 2

Volumetric flow rate was measured according to EPA Method 2. A Type K thermocouple and S-type pitot tube were used to measure flue gas temperature and velocity, respectively. All of the isokinetically sampled methods that were used incorporate EPA Method 2.

5.5.2.1 Sampling and Equipment Preparation. For EPA Method 2, the pitot tubes were calibrated before use following the directions in the method. Also, the pitots were leak checked before and after each run.

5.5.2.2 Sampling Operations. The parameters that were measured included the pressure drop across the pitots, stack temperature, stack static and ambient pressure. These parameters were measured at each traverse point, as applicable. A computer program was used to calculate the average velocity during the sampling period.

5.5.3 O_2 and CO_2 Concentrations by EPA Method 3A

The O_2 and CO_2 concentrations were determined by CEMs following EPA Method 3A. Flue gas was extracted from the duct and delivered to the CEM system through heated Teflon® tubing. The sample stream was then conditioned (particulate and moisture removed) and was directed to the analyzers. The O_2 and CO_2 concentrations were, therefore, determined on a dry basis. Average concentrations were calculated to coincide with each respective time period of interest. More information on the CEM system will be given in Section 5.6.

5.5.4 Average Moisture Determination by EPA Method 4

The average flue gas moisture content was determined according to EPA Method 4. Before sampling, the initial weight of the impingers was recorded. When sampling was completed, the final weights of the impingers were recorded, and the weight gain was calculated. The weight gain and the volume of gas sampled were used to calculate the average moisture content (percent) of the flue gas. The calculations were performed by computer. EPA Method 4 was incorporated in the techniques used for all of the manual sampling methods that were used during the test.

5.6 CONTINUOUS EMISSIONS MONITORING METHODS

EPA Methods 3A, 7E, 6C, and 10 were the continuous monitoring methods used for measuring CO_2/O_2, NO_x, SO_2, and CO concentrations, respectively. Total hydrocarbons were analyzed by EPA Method 25A. A diagram of the CEM system is shown in Figure 5-9.

One extractive system was used to obtain flue gas samples for the CEM systems. For the main CEM system, samples were withdrawn continuously at a single point from the outlet duct and transferred to the CEM trailer through heat-traced Teflon® line. The flue gas was conditioned (temperature lowered and moisture removed) before the flue gas stream was split through a manifold to the various analyzers. Total hydrocarbon

measurements were made on an unconditioned, hot basis. Therefore, this sample stream bypassed the gas conditioner.

5.6.1 CEM Sampling Equipment

5.6.1.1 Sample Probes. The main CEM probe consisted of a black iron pipe mounted to a Swagelok® reducing union which was attached directly to the heat trace tubing. The probe was placed approximately at a point of average velocity in the stack determined by a prior velocity traverse by EPA Method 2.

5.6.1.2 Heated Lines. Heated sample lines were used to transfer the flue gas samples to the instrument trailer for O_2, CO_2, NO_x, SO_2, CO, and THC analyses. These lines were heated in order to prevent condensation. Condensate could clog sample lines or provide a medium for the flue gas sample to react and change composition.

All heat trace lines contained three 3/8-inch Teflon® tubes. One tube carried the sample, one tube was used for calibration and QC gases, and the other was available as a backup. Calibration and QC gases were directed to the sampling probe through the transfer tube and then back through the entire sampling/conditioning system.

5.6.1.3 Gas Conditioning. Special gas conditioners were used to reduce the moisture content of the flue gas. A Radian designed gas conditioning system utilized a chiller system to cool a series of glass cyclones. An antifreeze liquid system was used to chill the glass cyclones. The hot flue gas was chilled by heat conduction through the glass wall causing the moisture to condense into droplets. The droplets and any PM were flung outward toward the glass walls by the centrifugal force. Particles impacted the glass walls and fell to the bottom of the cyclone where they were drained from the system. In this manner, both moisture and PM were effectively removed from the flue gas sample stream. This system operated under positive pressure eliminating the possibility of leakage which would dilute the gas samples. The gas conditioner was located in the CEM trailer.

5.6.2 CEM Principles of Operation

5.6.2.1 Sulfur Dioxide Analysis. The Western 721A SO_2 analyzer was essentially a continuous spectrophotometer in the ultraviolet range. Sodium dioxide selectively absorbed ultraviolet (UV) light at a wavelength of 202.5 nm. To take advantage of this

property of SO_2, the analyzer emitted UV light at 202.5 nm and measured the absorbance (A) of the radiation through the sample cell by the decrease in intensity. Beer's law, A = abc, was used to convert the absorbance into SO_2 concentration (A = absorbance, a = absorbitivity, b = path length, c = concentration). Sulfur dioxide measurements were performed using EPA Method 6C.

5.6.2.2 Nitrogen Oxide Analysis. The principle of operation of the TECO Model 10AR was a chemiluminescent reaction in which ozone (O_3) reacted with nitric oxide (NO) to form O_2 and nitrogen dioxide (NO_2). During this reaction, a photon was emitted which was detected by a photomultiplier tube. The instrument was capable of analyzing total oxides of nitrogen (NO + NO_2) by thermally converting NO_2 to NO in a separate reaction chamber prior to the photomultiplier tube. Nitrogen oxide measurements were performed using EPA Method 7E.

5.6.2.3 Oxygen Analysis. The Thermox WDG IV measured O_2 using an electrochemical cell. Porous platinum electrodes were attached to the inside and outside of the cell, which provided the instrument voltage response. Zirconium oxide contained in the cell conducted electrons when it was hot from the mobility of O_2 ions in its crystal structure. A difference in O_2 concentration between the sample side of the cell and the reference (outside) side of the cell produced a voltage. This response voltage was proportional to the logarithm of the O_2 concentration ratio. A linearizer circuit board was used to make the response linear. The reference gas was ambient air at 20.9 percent O_2 by volume.

5.6.2.4 Carbon Dioxide Analysis. Non-dispersive infrared (NDIR) CO_2 analyzers emitted a specific wavelength of infrared radiation which was selectively absorbed by CO_2 molecules through the sample cell. The intensity of radiation which reached the end of the sample cell was compared to the intensity of radiation through a CO_2-free reference cell. A reference cell was used to determine background absorbance which was subtracted from the sample absorbance. The detector used two chambers filled with CO_2 and connected by a deflective metallic diaphragm. One side received radiation from the sample cell and the other side received radiation from the reference cell. Since more radiation was absorbed in the sample cell than in the reference cell, less radiation

reached the sample side of the detector. This caused a deflection of the diaphragm due to increased heat from radiation absorption on the reference side. Deflection of the diaphragm created an electrical potential which was proportional to absorbance. Absorbance was directly proportional to CO_2 concentration in the gas. Carbon dioxide measurements were performed according to EPA Method 3A using a Beckman Model 880 NDIR anlayzer.

5.6.2.5 Carbon Monoxide Analysis. A TECO Model 48 analyzer was used to monitor CO emissions. The TECO analyzer measured CO using the same principle of operation as CO_2 analysis. A wave length of 5 nm is selective for CO. Carbon monoxide measurements were performed using EPA Method 10.

5.6.2.6 Total Hydrocarbon Analysis. A Ratfisch Model 55 was used to monitor THC emissions. By allowing the THC sample stream to bypass the gas conditioner, concentrations were determined on a wet basis. The analyzer employed an FID. As the flue gas entered the detector, the hydrocarbons were combusted in a hydrogen flame. The ions and electrons formed in the flame entered an electrode gap, decreased the gas resistance, and permitted a current flow in an external circuit. The resulting current was proportional to the instantaneous concentration of the total hydrocarbons. This method was not selective between species. EPA Method 25A applies to the continuous measurement of total gaseous organic concentrations of primarily alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The results were reported on a methane basis and methane was used as the calibration gas.

5.6.3 CEM Calibration

All the CEM instruments were calibrated once during the test program (and linearized, if necessary) using a minimum of three certified calibration gases (zero and two upscale points). Radian performed the multipoint calibrations with four general categories of certified gases: zero gas (generally N_2), a low scale gas concentration, a midrange concentration, and a high scale concentration (span gas). The criterion for acceptable linearity was a correlation coefficient (R^2) of greater than or equal to 0.998, where the independent variable was cylinder gas concentration and the dependent variable was instrument response. If an instrument did not meet these requirements, it

was linearized by adjusting potentiometers on the linearity card within the instrument or by other adjustments, if necessary.

The CEM analyzers were calibrated before and after each test run (test day) on a two point basis: zero gas (generally N_2), and a high-range span gas. These calibrations were used to calculate response factors used for sample gas concentration determinations. Instrument drift as a percent of span was also determined using these calibrations for each test run.

After each initial calibration, midrange gases for all instruments were analyzed, with no adjustment permitted, as a quality control (QC) check. If the QC midrange gas concentration observed was within ±2 percent of full scale, the calibration was accepted and the operator began sampling. If the QC check did not fulfill this requirement, another calibration was performed and linearization was performed if deemed necessary. Calibration procedures are further detailed in the daily operating procedure (Section 5.6.5).

Table 5-3 lists the concentration of all calibration and QC gases used on this test program.

5.6.4 Data Acquisition

The data acquisition system consisted of a Dianachart PC Acquisitor data logger, a signal conditioner, and a 386 desktop computer. All instrument outputs were connected in parallel to stripchart recorders and the data acquisition system. The stripchart recorders were a back-up system to the data logger. The PC Acquisitor scanned the instrument output and logged digitized voltages. A Radian computer program translated the digitized voltages into relevant concentrations in engineering units (ppmv, %V, etc.). The computer program had several modes of operation: calibration, data acquisition, data reduction, data view, data edit, and data import. The import function was used to combine other data files for comparison and correlation.

5.6.5 Daily Operating Procedure

The following is a detailed standard operating procedure for calibrating and operating the CEM system:

Table 5-3

CEM Operating Ranges And Calibration Gases

Analyte	Gas Concentration
CO_2	
Instrument	Beckman 880
Range	0 - 20%
Span Gas Value	18%
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	10%
Low Range QC Gas Value	5%
CO - dry	
Instrument	TECO 48H
Range	0 - 100 ppmvd
Span Gas Value	98 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	60 ppm
Low Range QC Gas Value	20 ppm
O_2	
Instrument	Thermox WDG III
Range	0 - 25%
Span Gas Value	20%
Zero Gas	0.2% O_2
Midrange QC Gas Value	10%
Low Range QC Gas Value	5%
SO_2	
Instrument	Western 721A
Range	0 - 200 ppmvd
Span Gas Value	180 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	100 ppm
Low Range QC Gas Value	30 ppm

Table 5-3

Continued

Analyte	Gas Concentration
NO_x	
Instrument	TECO 10AR
Range	0 - 250 ppmvd
Span Gas Value	200 ppm
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	100 ppm
Low Range QC Gas Value	50 ppm
THC (EPA Method 25A)	
Instrument	Ratfisch RS-55
Range	0 - 100 ppmvd
Span Gas Value	90% as methane
Zero Gas	100% N_2 (UHP)
Midrange QC Gas Value	45 ppm as methane
Low Range QC Gas Value	25 ppm as methane

1. Turn on computer and printer, put printer on-line, and load the CEM.EXE program. Be sure that the CEM instruments have been on for at least 20 hours.

2. Synchronize DAS clock with sample location leaders and the test leader.

3. Turn on strip chart recorders (SCR) and make appropriate notes on charts and in logbook (write down all procedures and observations in logbook and on SCRs as the day progresses).

4. Turn on the gas conditioners and blow back compressor. Blow back the system.

5. Open all calibration gas cylinders so that they may be introduced to the instruments via control panel valves.

6. Perform daily pretest leak check on CEMs by introducing ultra high purity nitrogen to the system. Zero all instruments except the Thermox O_2 analyzers. Make adjustments to the zero potentiometers as required to zero the instruments. Be sure to check and maintain all flows throughout calibration and operation.

7. Record the zero values in the computer calibration routine.

8. Introduce 2.0 percent O_2 to set the low scale response for the Thermox O_2 analyzers and repeat Step 7 for these instruments.

9. Introduce the mixed span gases for O_2, CO_2, and CO. Make adjustments as required to these instruments.

10. Enter these values in the computer calibration routine.

11. Introduce the NO_x span gas.

12. Make adjustments to the NO_x instruments as required and enter the value into the computer calibration routine.

13. Introduce the SO_2 span gas for the SO_2 analyzer, repeat Step 12 for the SO_2 analyzer. (Note that all calibration gases are passed through the entire sampling system.)

14. Switch the Western SO_2 analyzer range to 0-500 ppm introduce the span gas for this range and repeat Step 12 for this instrument.

15. Check the calibration table on the computer, and make a hardcopy. Put the computer in the standby mode.

16. Introduce QC gases to instruments in the same sequence as the calibration gases. Record three minutes of data for each, once the responses have stabilized. If the QC gas response is not within ±2 percent of the instrument range the operator should recalibrate the instrument, or perform other corrective actions.

17. Begin sampling routine, with the computer on stand by.

18. Start the data acquisition system when signaled by radio that system is in stack.

19. Carefully check all flows and pressures during the operation of the instruments and watch for apparent problems in any of the instruments, such as unusual readings or unreasonable fluctuations. Check the gas conditioning system periodically and drain the traps.

20. Stop the data acquisition system at the end of the test when signaled.

21. Perform final leak check of system.

22. Perform the final calibration (Repeat steps 6-16) except make no adjustments to the system.

23. Check for drift on each channel.

5.7 POLYNUCLEAR AROMATIC HYDROCARBON EMISSIONS TESTING

The polynuclear aromatic hydrocarbon (PAH) sampling and analytical method is a combination of EPA SW-846 Test Method 0010 and EPA SW-846 Test Method 8270.

5.7.1 Sampling Equipment

The PAH sampling method used the sampling train shown in Figure 5-10. Radian modified the protocol configuration to include a horizontal condenser rather than a vertical condenser. The horizontal condenser lowered the profile of the train and reduced breakage. The XAD trap following the condenser was maintained in a vertical position.

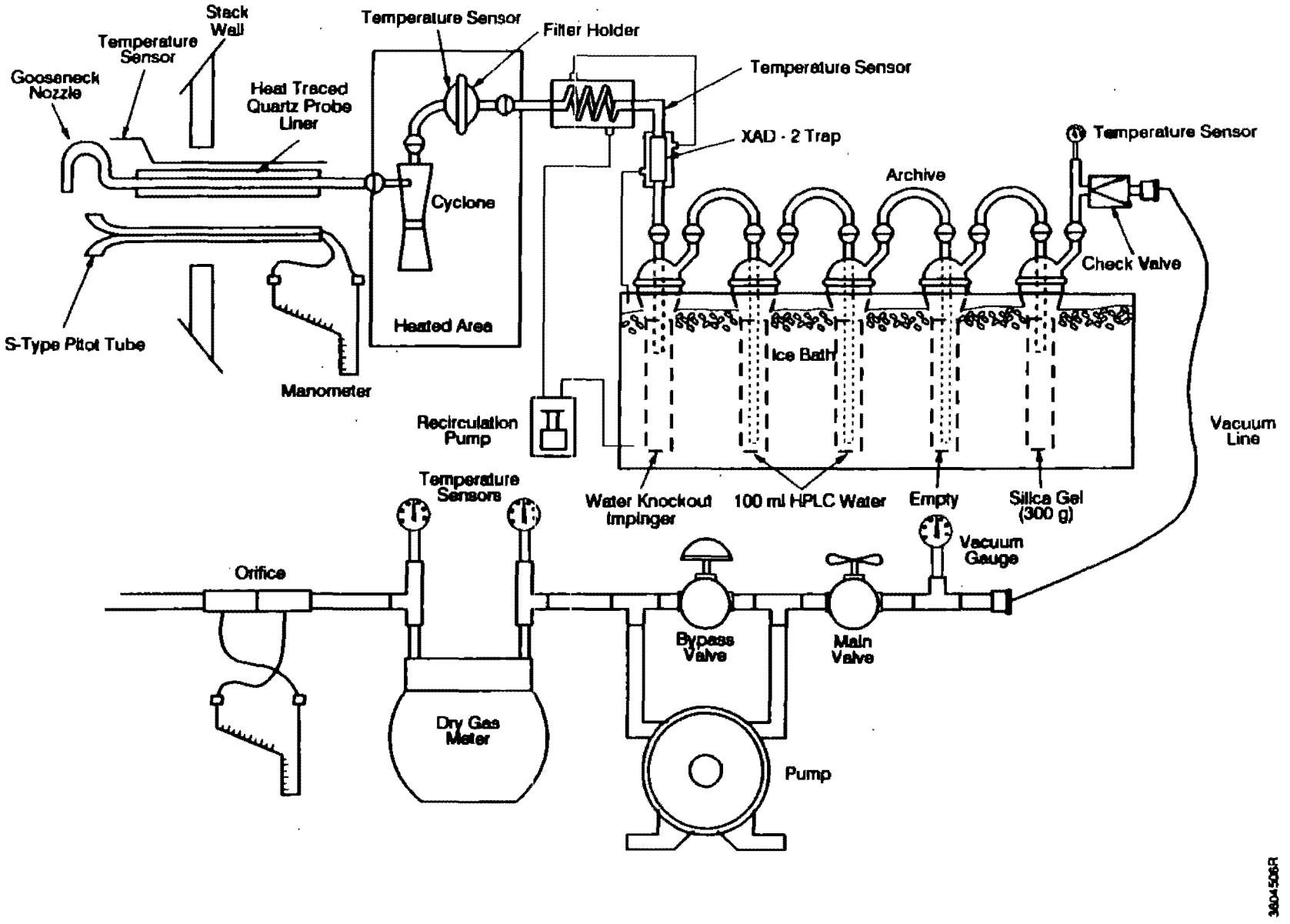

Figure 5-10. PAH Sampling Train Configuration

5.7.2 Sampling Equipment Preparation

In addition to the standard EPA Method 5 requirements, the PAH sampling method included several unique preparation steps which ensured that the sampling train components were not contaminated with organics that may interfere with analysis. The glassware, glass fiber filters, and XAD resins were cleaned and checked for residuals before being packed.

5.7.2.1 Glassware Preparation. Glassware was washed in soapy water, rinsed with distilled water, baked, and then rinsed with acetone followed by methylene chloride. This included all the glass components of the sampling train including the glass nozzles plus any sample bottles, Erlenmeyer flasks, petri dishes, graduated cylinders or stirring rods that were used during recovery. Nonglass components (such as the teflon-coated filter screens and seals, tweezers, teflon squeeze bottles, nylon probe brushes and nylon nozzle brushes) were cleaned following the same procedure except that no baking was performed. The specifics of the cleaning procedure are presented in Table 5-4.

5.7.2.2 XAD II and Filters Preparation. XAD resin and glass fiber filters were placed together in a soxhlet and extracted in HPLC-grade water, methyl alcohol, methylene chloride and hexane, sequentially. At the conclusion of the soxhlet extractions, one filter and 30 grams of XAD resin were analyzed for background contamination following the same procedure followed for the flue gas samples. The XAD and filter blank were analyzed for PAH compounds. The pressure drop for the XAD traps was checked before and after the resin was loaded to ensure that the pressure drop across the XAD traps was less than seven inches of mercury.

5.7.2.3 Method 5 Equipment Preparation. The EPA Method 5 equipment was prepared according to the protocol discussed in Section 5.1.2.3.

Table 5-4

Glassware Cleaning Procedure
(Train Components and Sample Containers)

NOTE: USE DISPOSABLE GLOVES AND ADEQUATE VENTILATION
1. Soak all glassware in hot soapy water (Alconox®).
2. Tap water rinse to remove soap.
3. Distilled/deionized H_2O rinse (X3).[a]
4. Bake at 450°F for 2 hours.[b]
5. Acetone rinse (X3), (pesticide grade).
6. Methylene Chloride (X3).
7. Cap glassware with clean glass plugs or methylene chloride rinsed aluminum foil.
8. Mark cleaned glassware with color-coded identification sticker.

[a](X3) = Three times.
[b]Step (4) is not used for probe liners and non-glass components of the train that cannot withstand 450°F (i.e., Teflon-coated filter screen and seals, tweezers, Teflon squeeze bottles, nylon probe and nozzle brushes). The probe liners are too large for the baking ovens.

5.7.3 Sampling Operations

5.7.3.1 Preliminary Measurements. Prior to sampling, preliminary measurements were made as described in Section 5.1.3.1.

5.7.3.2 Assembling the Train. Initial assembly of the PAH sampling train components was performed in the recovery trailer. Final assembly of the train with the probe, nozzle, and filter was performed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order. The first impinger was a knockout impinger which had a short tip. The purpose of this impinger was to collect condensate which formed in the coil and XAD trap. However, the gas was not bubbled through the condensate to prevent carry-over to the next impinger. The next two impingers were modified tip impingers that contained 100 ml of HPLC grade water each. The fourth impinger was empty, and the fifth impinger contained 200 to 300 grams of silica gel. When the impingers were loaded, they were wrapped with teflon tape to secure the two sections of the impinger. Then each impinger was weighed and the weight recorded along with information on the contents of the impingers. The impingers were connected together using cleaned glass U-tube connectors and arranged in the impinger bucket. The height of all the impingers should be approximately the same to obtain a leak-free seal. The open ends of the train were sealed with methylene chloride-rinsed aluminum foil.

The second step was to load the filter into the filter holder. The filter holder was then capped off and placed with the XAD trap and condenser coil (capped) into the impinger bucket. A supply of precleaned foil and socket joints were also placed in the bucket in a clean plastic bag. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-10. Sealing greases were not used to avoid contamination or adsorption of the sample.

5.7.3.3 Sampling Procedures. After the train was assembled, the heaters were turned on for the probe liner and heated filter box. When the system reached the appropriate temperatures, the sampling train was ready for leak checking.

The PAH train was leak checked at the start and finish of sampling as required in EPA Method 5 as well as before and after each port change. If a piece of glassware

needed to be emptied or replaced, a final leak check was performed before the glassware piece was removed. After the train was reassembled, an initial leak check was performed.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. The leak rate is required to be less than 0.02 acfm (ft^3/min). After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off.

If the leak-rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

In the event that a final leak rate was found to be above the minimum acceptable rate (0.02 acfm) upon removal from a port, acceptance is subject to the approved of the EPA administrator. Otherwise, the run was voided and repeated.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occurred during sampling were recorded on the task log such as pitot cleaning, thermocouple malfunctions, heater malfunctions and any unusual occurrences.

Sampling train data were recorded every five minutes on standard data forms. A checklist for sampling was given previously in Table 5-2. The purpose of the checklist is to remind samplers of the critical steps during sampling.

A sampling operation that was unique to PAH sampling was maintaining the gas temperature entering the XAD trap below 68°F. The gas was cooled by the condenser and the XAD trap, which both have a water jacket in which ice water was circulated.

5.7.4 Sample Recovery

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into four sections: the probe liner, the XAD trap and condenser, filter holder, and the impingers in their bucket. Each of these sections was capped with methylene chloride-rinsed aluminum foil before removal to the recovery trailer. Once in the trailer, field recovery personnel followed the scheme shown in Figure 5-11. The samples were placed in cleaned amber glass bottles to prevent light degradation.

The solvents used for train recovery were acetone (pesticide grade) followed by methylene chloride. The use of the highest grade acetone for train recovery was essential to prevent the introduction of chemical impurities which interfere with the quantitative analytical determinations.

Field recovery resulted in the sample components listed in Table 5-5. The samples were shipped to the analytical laboratory as expediently and carefully as possible.

5.7.5 Analytical Procedures

The analytical procedure used to determine PAH concentrations from the Modified Method 5 sample followed EPA SW-846 Test Method 8270 protocol. The detection limit for PAH was about 1 μg per train. The compounds/isomers of interest in the analysis are shown in Table 5-6.

5.7.5.1 Preparation of Samples for Extraction. Upon receiving the sample shipment, the samples were checked against the chain-of-custody forms and then assigned an analytical laboratory sample number. Each sample component was reweighed to determine if leakage occurred during travel. Color, appearance, and other particulars of the samples were noted. Samples were extracted within 21 days of collection.

Glassware used in the analytical procedures (including soxhlet apparatus and disposable bottles) was cleaned by washing twice with detergent, rinsing with distilled water, and then rinsing with acetone, methanol, and methylene chloride. The glassware was allowed to air dry.

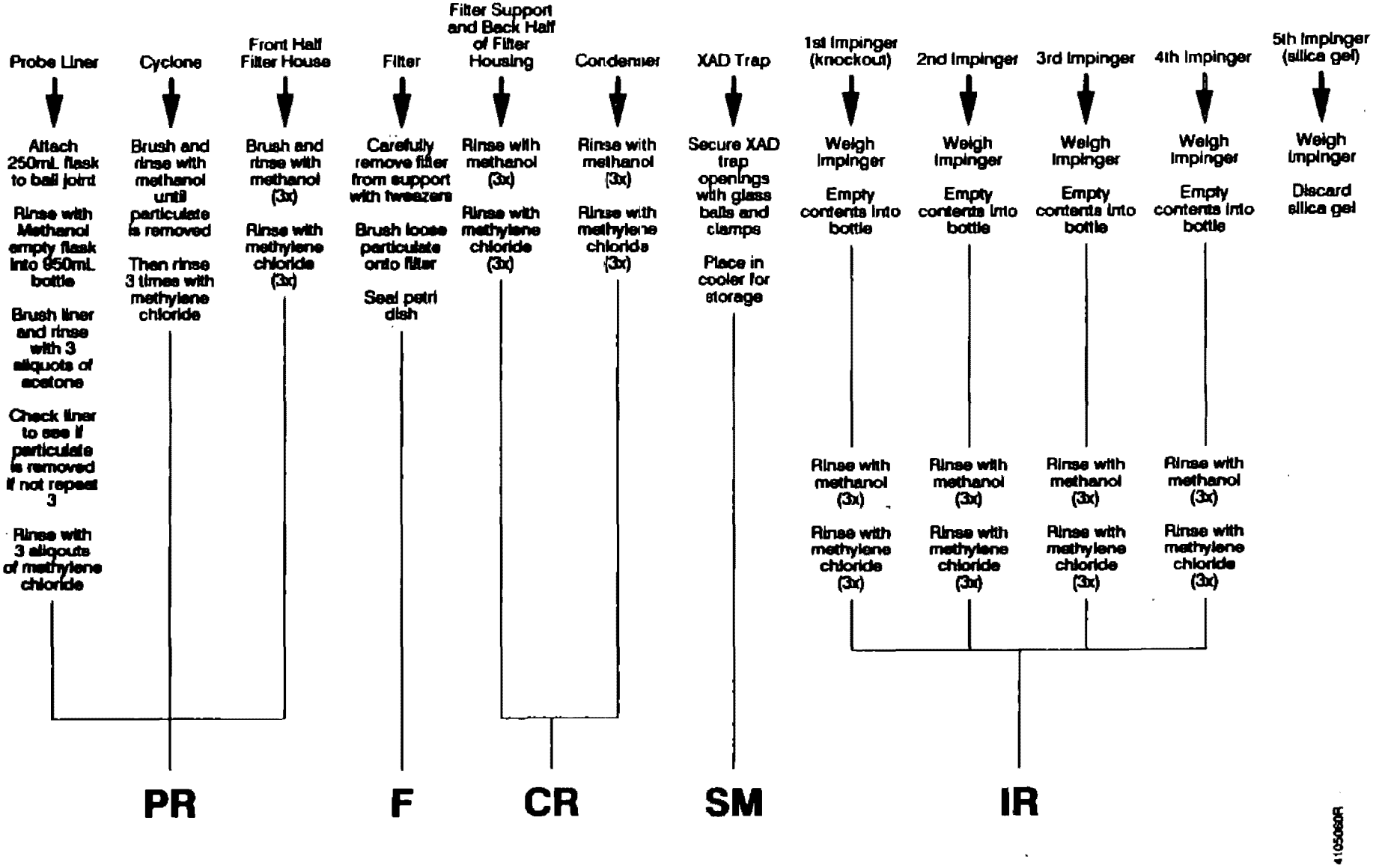

Figure 5-11. PAH Field Recovery Scheme

Table 5-5

Polynuclear Aromatic Hydrocarbon Sample Components
Shipped to Analytical Laboratory

Container/Component	Code	Glassware
1	F	Filter(s)
2	PR	Rinses[a] of nozzle, probe, and front half of filter holder
3	CR	Rinses[a] of back half of filter holder, filter support, and condenser
4	IR	First, second, third, and fourth impinger contents and rinses[a]
5	SM	XAD-2 resin

[a]Rinses include acetone and methylene chloride recovered into the same sample bottle.

Table 5-6

Polynuclear Aromatic Hydrocarbon Compounds To Be Analyzed

Naphthalene
Acenapthylene
Acenapthene
Fluorene
Phenathrene
Anthracene
Fluoranthene
Pyrene
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(a)pyrene
2-Methylnapthalene
2-Chloronapthalene
Benzo(k)fluoranthene
Benzo(e)pyrene
Perylene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Dibenzofuran
7,12-Dimethylbenz(a)anthracene
Benzo(g,h,i)perylene

5.7.5.2 Calibration of GC/MS System. An initial calibration of the GC/MS system was performed to demonstrate instrument linearity over the concentration range of interest. Analyses for PAH was performed using low resolution mass spectrometry. A typical calibration range consisted of points at 4:100, 40:100, and 400:100 for the ratio of analytes to isotopically labeled internal standards. Relative response factors were calculated for each compound of interest. The response factors were verified on a daily basis using a continuing calibration standard consisting of mid-level standard (typically the 40:100 standard).

5.7.5.3 Sample Extraction. For PAH analyses, isotopically labeled surrogate compounds were added to the samples before the extraction process was initiated. These surrogates were used to monitor the efficiency of the extraction/clean-up. The internal standards used in the quantitative analysis of these analytes were added to the samples immediately prior to analysis, and used to perform the quantitative calculations.

5.7.5.4 Analysis by GC/MS. The PAH analyses were performed by high-resolution GC followed by low resolution mass spectrometry.

Data from the MS were recorded and stored on a computer file as well as printed on paper. A duplicate analysis was performed on every tenth sample in the sample batch. A method blank which was carried through the complete extraction procedure was also analyzed. Results such as amount detected, detection limit, retention time, and internal standard and surrogate standard recoveries were calculated by computer. The chromatograms were retained by the analytical laboratory and also included in the analytical report.

5.7.6 Analytical QA/QC Procedures

This section discusses the general quality control procedures that were followed for the analytical methods. Method-specific analytical QA/QC procedures are also presented.

5.7.6.1 Quality Control. This section presents the PAH quality control requirements.

Blanks. Two different blanks were collected for the PAH analyses: a laboratory proof blank and a field blank. Proof blanks were obtained from a complete set of

Modified Method 5 sample train glassware that had been cleaned according to the procedure presented in Section 5.1.2. The precleaned glassware, which consisted of a probe liner, filter holder, condenser coil, and impinger set, was loaded and then recovered by rinsing with acetone and methylene chloride three times each. All sets of glassware were blanked.

A field blank was collected from a set of PAH glassware that was used to collect at least one sample and had been recovered. The train was reloaded and left at the sampling location during a test run. The train was then recovered. The field blank was used to measure the level of contamination that occurred from handling, loading, recovering, and transporting the sample train. The field blank was analyzed concurrently with the flue gas samples. If the field blank results were acceptable, the laboratory proof blank and reagent blanks were archived but not analyzed.

Analytical method (reagent) blanks were also analyzed as part of the QC program. The QC criteria for method blanks was concentrations less than or equal to the detection limit (in the noise range).

Standards Duplicates. Isotopically labelled internal standards and surrogate compounds were added to the sample before the extraction process began. Once added to the samples, the internal standards went through the entire extraction process and were measured on the GC/MS. The recoveries of the internal standards were determined and the results of the native species were adjusted according to the internal standard recoveries. The results contained in the analytical report were adjusted for internal standard recoveries. The surrogate compounds were added in a similar manner, but the surrogate recoveries were not used to adjust the results of the native species. Surrogate recoveries provided additional data on the efficiency of the extraction procedure and the performance of the instruments. The QC objective for internal standards and surrogate recoveries was 100 $\pm$50% recovery.

The purpose of duplicate analyses was to evaluate the reproductibility (precision) of the combined sample preparation and analytical methodology. The QC criterion for analysis of duplicates was agreement to within ±50% (for each PAH species). Analytical duplicates (two injections of the same sample) were also analyzed to assess the precision of the analytical methodology. For PAH flue gas samples, only analytical duplicates were performed. For every 10 samples, one duplicate analysis was performed.

5.8 ASTM METHODS

Standard ASTM methods were used to assess heat of combustion, ultimate analysis (ash, O_2, carbon, hydrogen, sulfur, and nitrogen), and chlorine content of the waste oil fuel. Aggregate moisture and ambient humidity were also analyzed. Descriptions of applicable ASTM methods follow.

5.8.1 Relative Humidity

Sampling for relative humidity was performed using ASTM Method E337-62, "Standard Test Method for Relative Humidity by Wet- and Dry-Bulb Psychrometer." This method covers the determination of the relative humidity of atmospheric air by means of wet- and dry-bulb temperature readings.

5.8.1.1 Sampling Equipment and Method. A sling psychrometer was used for measuring relative humidity. Two thermometers, one with a wet-bulb covering were mounted on the psychrometer. The wet-bulb covering was moistened and the psychrometer slung through the air for several minutes. The thermometers were read and the psychrometric chart was used to calculate the relative humidity.

5.8.2 Heat of Combustion Test Method

Heat of combustion of the fuel sample was determined according to ASTM Method D240-87, "Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter." This test method covered the determination of the heat of combustion of liquid hydrocarbon fuels ranging in volatility from that of light distillates to that of residual fuels.

5.8.2.1 Sampling Equipment and Method. Heat of combustion was determined using an O_2 bomb, calorimeter, stirred water jacket, and a thermometer. A weighed sample was burned in an O_2 bomb calorimeter under controlled conditions. The heat of

combustion was computed from temperature observations before, during, and after combustion with proper allowance for thermochemical and heat transfer corrections. Adiabatic calorimeter jackets were used.

5.8.3 Total Moisture Content Test Method

Total moisture content of the aggregate sample was determined according to ASTM Method C566-89, "Standard Test Method for Total Moisture Content of Aggregate by Drying." This test method covered the determination of the percentage of evaporative moisture in a sample. The plant routinely performed this test at least several times per day. This information was provided by the plant personnel and is included in the process data section of the test report.

5.8.3.1 Sampling Equipment and Method. Total moisture content was determined using a balance or scale accurately readable and sensitive to within 0.1 percent of the test load, a source of heat such as a ventilated oven capable of maintaining the temperature surrounding the sample at 110 ±5°C (230 ±9°F); and a sample container not affected by the heat and of sufficient volume to contain the sample without danger of spilling.

The sample was weighed to the nearest 0.1 percent and then dried in the sample container. The temperature was controlled when excessive heat may alter the character of the aggregate or where more precise measurement was needed. The dried sample was weighed to the nearest 0.1 percent after it had cooled sufficiently to prevent damage to the balance. Total moisture was calculated using the formulas presented in Section 7.1 of the reference method.

5.8.4 Sulfur Test Method

Sulfur concentrations in the sample were determined according to ASTM Method D1552-90, "Standard Test Method for Sulfur in Petroleum Products (High Temperature Method)." This test method covered the procedures for the determination of total sulfur in petroleum products including lubricating oils containing additives and in additive concentrates.

5.8.4.1 Sampling Equipment and Method. Sulfur content of a sample was determined using a furnace, an absorber, a buret, and other miscellaneous apparatus.

The IR detection system was used for the determination of sulfur. The sample was weighed into a special ceramic boat which was then placed into a combustion furnace in an O_2 atmosphere. Any sulfur was combusted to SO_2 which was then measured with an IR detector after moisture and dust were removed by traps. A microprocessor calculated the mass percent sulfur from the sample weight, the integrated detector signal and a predetermined calibration factor. Both the sample identification number and mass percent sulfur were then printed out. The calibration factor was determined using standards approximating the material to be analyzed.

5.8.5 Nitrogen Test Method

Nitrogen concentration in the sample was determined according to ASTM Method D3179-84, "Standard Test Methods for Nitrogen in the Analysis of Coal and Coke." This test method covered the determination of total nitrogen in samples of coal and coke.

5.8.5.1 Sampling Equipment and Method. Total nitrogen was determined using a digestion unit, digestion flasks, distillate unit, buret, Erlenmeyer flasks, rubber tubing, and pipets. Reagents included an alkali solution, ethyl alcohol, and sulfuric acid. Nitrogen in the sample was converted into ammonium salts by destructive digestion of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts were subsequently decomposed in a hot alkaline solution from which the ammonia was recovered by distillation and finally determined by alkalimetric or acidimetric titration.

5.8.6 Carbon and Hydrogen Test Method

Carbon and hydrogen concentrations in the sample were determined according to ASTM Method D3178-84, "Standard Test Method for Carbon and Hydrogen in the Analysis Sample of Coal and Coke."

5.8.6.1 Sampling Equipment and Method. Carbon and hydrogen content were determined using an O_2 purifying train that consisted of two water absorbers and a CO_2 absorber, a flow meter, a combustion unit, and reagents. A quantity of the sample was burned in a closed system. The products of combustion were fixed in an absorption train after complete oxidation and purification from interfering substances. This test method

gave the total percentages of carbon and hydrogen and included the carbon in carbonates and the hydrogen in the moisture and in the water of hydration of silicates.

6. QUALITY ASSURANCE AND QUALITY CONTROL

To ensure the production of useful and valid data, specific QA/QC procedures were strictly adhered to during this test program. A detailed presentation of QC procedures for all manual flue gas sampling, process sample collection, GC operations, and CEM operations can be found in the Mathy Test Plan. This section will report the test program QA parameters so that the degree of data quality can be shown.

Two days of testing were conducted at Mathy Construction Company, Plant 6. Three runs were completed successfully at near-design operating conditions while the plant was operating on natural gas. No sampling related problems were encountered during testing that would affect data quality.

In summary, a high degree of data quality was maintained throughout the project. Post-test leak checks for all sampling trains were within acceptable limits and all post-test calibration checks for the dry gas meters were within acceptable limits. All PM/metals, aldehydes, and PAH manual sampling trains met the isokinetic criterion of ±10 percent out of 100 percent, and the PM_{10}/CPM manual sampling trains met the isokinetic criterion of ±20 percent out of 100 percent, which is acceptable for this test method.

Method blank and field blank results for the manual sampling trains showed some contamination. Also, several method spike recovery values for the metals, aldehydes, and PAH analyses were not within the QA allowance. Method blank, field blank, and method spike results are further discussed in Section 6.2.

The CEM results showed acceptable calibration drift values and QC gas responses. All CEM QC procedures and objectives were followed as described in the Mathy Test Plan.

The GC used for EPA Method 18 analysis was calibrated each test day before and after flue gas sampling. Quality assurance/quality control results showed allowable response factor drift values for the calibration gases.

Section 6.1 presents the QA/QC definitions and data quality objectives. Section 6.2 presents manual flue gas sampling and recovery parameters and Section 6.3

presents method-specific analytical QA parameters. Section 6.4 discusses the CEM QA parameters and Section 6.5 presents the GC QA parameters.

6.1 QUALITY ASSURANCE/QUALITY CONTROL DEFINITIONS AND OBJECTIVES

The overall QA/QC objective is to ensure precision, accuracy, completeness, comparability, and representativeness for each major measurement parameter called for in this test program. For this test program, quality control and quality assurance can be defined as follows:

- Quality Control: The overall system of activities whose purpose is to provide a quality product or service. QC procedures are routinely followed to ensure high data quality.

- Quality Assurance: A system of activities whose purpose is to provide assurance that the overall quality control is being done effectively. Assessments can be made from QA parameters on what degree of data quality was achieved.

- Data Quality: The characteristics of a product (measurement data) that bear on its ability to satisfy a given purpose. These characteristics are defined as follows:

  - Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation and in this report will be expressed as the relative standard deviation or coefficient of variation.

  - Accuracy - The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, can be expressed as the difference between two values, X-T, the ratio X/T, or the difference as a percentage of the reference or true value, 100 (X-T)/T.

  - Completeness - A measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under prescribed test conditions.

  - Comparability - A measure of the confidence with which one data set can be compared with another.

- Representativeness - The degree to which data accurately and precisely represent a characteristic of a population, variations of a parameter at a sampling point, or an environmental condition.

A summary of the estimated precision, accuracy, and completeness objectives is presented in Table 6-1.

6.2 MANUAL FLUE GAS SAMPLING QUALITY ASSURANCE

The following section will report manual sampling QA parameters so that insight can be gained into the quality of the emissions test data produced from manual tests during the test program.

6.2.1 Particulate Matter/Metals Sampling Quality Assurance

Table 6-2 presents post-test leak check results for all of the manual sample trains. The acceptance criterion is that all post-test leak checks must be less than 0.02 cfm. All PM/metals post-test leak checks met the acceptance criterion and, therefore, no leak corrections were applied.

The isokinetic sampling rates for all of the manual sampling runs are presented in Table 6-3. The acceptance criterion for the PM/metals, aldehydes, and PAH sampling runs is that the average sampling rate must be within 10 percent of 100 percent isokinetic. Acceptance criterion for the PM_{10} runs allows sampling rates to be within 20 percent of 100 percent isokinetic. All PM/metals runs deviated by no more than 4 percent of 100 percent, thereby meeting the isokinetic criterion.

All dry gas meters used for manual sampling were fully calibrated within the last six months against an EPA approved intermediate standard. The full calibration factor or meter Y was used to correct actual metered volume to true sample volume. To verify the full calibration, a post-test calibration was performed. The full and post-test calibration coefficients must be within five percent to meet Radian's internal QA/QC acceptance criterion. The results of the full and post-test calibration check of the meter boxes used for manual sampling are presented in Table 6-4. The post-test calibration factor for the meter box used for PM/metals test runs was within the five percent criterion of the full calibration factor.

Table 6-1

Summary of Precision, Accuracy, and Completeness Objectives[a]

Parameter	Precision[a] (%)	Accuracy[a] (%)	Completeness[b] (%)
Flue Gas Formaldehyde	±15	±20	100
Flue Gas Metals	±15[d]	±30[c]	100
Polynuclear Aromatic Hydrocarbons	±15	±20	100
Flue Gas Total Particulate Matter	±11	±10	100
Continuous Monitoring System	±2[e]	±2[d]	95
Velocity/Volumetric Flow Rate	±6	±10	95
Fixed Gases/Molecular Weight	±0.3%V	±0.5%V	100
Moisture	±20	±10	95
Flue Gas Temperature	±2°F	±5°F	100

[a]Precision and accuracy estimated based on results of EPA collaborative tests. All values stated represent worst case values. All values are absolute percentages unless otherwise stated.

[b]Minimum valid data as a percentage of total tests conducted.

[c]Relative error (%) derived from audit analyses, where:

$$\text{Percent Error} = \frac{\text{Measured Value} - \text{Theoretical Value}}{\text{Theoretical Value}} \times 100$$

[d]Percent difference for duplicate anlayses, where:

$$\text{Percent Difference} = \frac{\text{First Value} - \text{Second Value}}{0.5(\text{First} + \text{Second Values})} \times 100$$

[e]Minimum requirements of EPA method 6C, based on percent of full scale.

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Table 6-2
LEAK CHECK RESULTS FOR MANUAL SAMPLE TRAINS
MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Sample Train	Date	Run Number	Maximum Vacuum	Avg. Sample Rate (acfm)	Measured Leak Rate	Vacuum (in. Hg)	Acceptable
PM/Metals	09/19/91	1	2	0.50	0.010	10	yes
PM/Metals	09/19/91	2	2	0.48	0.008	3.5	yes
PM/Metals	09/20/91	3	2	0.47	0.014	8	yes
PM10/CPM	09/19/91	1	NA	0.78	0.003	3	yes
PM10/CPM	09/20/91	2	NA	0.79	0.003	3	yes
PM10/CPM	09/20/91	3	NA	0.74	0.004	3	yes
Aldehydes	09/19/91	1	3	0.49	0.014	10	yes
Aldehydes	09/20/91	2	2.5	0.46	0.004	8	yes
Aldehydes	09/20/91	3	4	0.48	0.005	10	yes
PAH	09/19/91	1	7	0.51	0.012	10	yes
PAH	09/19/91	2	7	0.49	0.012	11	yes
PAH	09/20/91	3	8	0.47	0.006	11	yes

NA = Not Available

Table 6-3

ISOKINETIC SAMPLING RATES FOR MANUAL SAMPLING TEST RUN

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Sample Type	Date	Run Number	Isokinetic Sampling Rates	Acceptable *
PM/Metals	09/19/91	1	104	yes
PM/Metals	09/19/91	2	104	yes
PM/Metals	09/20/91	3	104	yes
PM10/CPM	09/19/91	1	87.0	yes
PM10/CPM	09/20/91	2	89.5	yes
PM10/CPM	09/20/91	3	80.4	yes
Aldehydes	09/19/91	1	105	yes
Aldehydes	09/20/91	2	101	yes
Aldehydes	09/20/91	3	102	yes
PAH	09/19/91	1	107	yes
PAH	09/19/91	2	106	yes
PAH	09/20/91	3	105	yes

* = PM/Metals, Aldehydes and PAH test metals specify isokinetic sampling rates must be within 10 percent of 100 percent isokinetic. The PM10 test method allows isokinetic sampling rates to be within 20 percent of 100 percent isokinetic

Table 6-4
DRY GAS METER POST-TEST CALIBRATION RESULTS
MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Meter Box I.D.	Sample Trains	Full Calibration Factor	Post-Test Calibration Factor	Post-Test Deviation (%)*	Acceptable
N-30	PAH	0.9998	1.0218	2.20	yes
N-32	Aldehydes	1.0006	1.0051	0.45	yes
N-33	PM/Metals - PM10/CPM	0.9875	0.9788	-0.88	yes

* = [(Post-Test)-(Full)]/(Full)*100

6.2.2 PM_{10}/CPM Sampling Quality Assurance

Post-test leak checks, isokinetic rates, and dry gas meter post-test calibrations for the PM_{10}/CPM test runs were with QA allowances and are presented in Tables 6-2 through 6-4, respectively. Note that the isokinetic acceptance criterion for PM_{10}/CPM runs is more lenient than other methods allowing the average sampling rate to be within 20 percent of 100 percent isokinetic.

6.2.3 Aldehydes Sampling Quality Assurance

The post-test leak checks for the aldehydes sample trains met the QA acceptance criterion. The isokinetic rates for the aldehydes test runs deviated by no more than 5 percent of 100 percent, meeting the QA acceptance criterion. The post-test calibration factor for the meter box used for aldehydes test runs was within the 5 percent criterion of the full calibration factor. Post-test leak check results, isokinetic values, and dry gas meter calibration results for the aldehydes runs are presented in Tables 6-2 through 6-4, respectively.

6.2.4 Polynuclear Aromatic Hydrocarbon Sampling Quality Assurance

The post-test leak check results for the PAH trains are presented in Table 6-2, and all trains met the QA acceptance criterion. Isokinetic rates, presented in Table 6-3, varied no more than 7 percent of 100 percent, meeting the acceptance criterion. The post-test calibration results of the dry gas meter used for PAH sampling are presented in Table 6-4 and show that the calibration factor is within the QA allowance.

6.3 ANALYTICAL QUALITY ASSURANCE

The following sections briefly report QA parameters for the metals, PM_{10}/CPM, aldehydes, and PAH analytical results. The analytical methods used for the flue gas samples are discussed fully in Section 5.

6.3.1 Metals Analytical Quality Assurance

Field blanks were collected for the PM/metals, PM_{10}/CPM, aldehydes, and PAH sampling trains. A train of each sample type was fully prepared, taken to the sample location, leak checked, and then recovered. Table 6-5 presents the results of the metals field blank analysis compared to the test run results. There was a noticeable contamination of certain metals in the field blank. The front half fraction was

Table 6-5

METALS FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

	Run 1			Run 2	Run 3			Field Blank		
Metals	Front Half (total ug)	Impinger 1,2,3 (total ug)	Total ug	Front Half (total ug)	Front Half (total ug)	Impinger 1,2,3 (total ug)	Total ug	Front Half (total ug)	Impinger 1,2,3 (total ug)	Total ug
Antimony	14.1	[1.57]	14.1	12.1	12.1	[1.58]	12.1	13.6	[1.67]	13.6
Arsenic	[1.00]	[0.418]	[1.42]	[1.00]	[1.00]	[0.421]	[1.42]	[1.00]	[0.444]	[1.44]
Barium	9.15	0.815	10.0	7.48	9.50	1.42	10.9	4.80	1.01	5.81
Beryllium	[0.250]	[0.104]	[0.354]	[0.250]	[0.250]	[0.105]	[0.355]	[0.250]	[0.111]	[0.361]
Cadmium	2.93	0.282	3.21	0.830	1.43	0.700	2.13	1.58	[0.222]	1.58
Chromium	7.75	1.87	9.62	3.20	3.38	1.28	4.66	3.78	0.811	4.59
Copper	[1.00]	6.23	6.23	[1.00]	[1.00]	5.92	5.92	[1.00]	[0.444]	[1.44]
Lead	1.92	0.886	2.81	0.750	6.35	1.66	8.01	1.74	0.373	2.11
Manganese	58.0	2.81	60.8	29.0	41.5	3.34	44.8	4.93	1.25	6.18
Nickel	45.0	2.17	47.2	2.43	3.50	1.72	5.22	3.30	0.955	4.26
Phosphorus	[75.0]	59.0	59.0	[75.0]	[75.0]	73.3	73.3	[75.0]	77.5	77.5
Selenium	[4.00]	[1.67]	[5.67]	[4.00]	[4.00]	[1.68]	[5.68]	[4.00]	[1.78]	[5.78]
Silver	7.00	[0.627]	7.00	5.35	[1.50]	[0.631]	[2.13]	4.05	0.700	4.75
Thallium	[25.0]	[10.4]	[25.4]	[25.0]	[25.0]	[10.5]	[35.5]	[25.0]	[11.1]	[36.1]
Zinc	23.8	16.1	39.9	15.4	35.0	13.4	48.4	21.6	2.24	23.8

NOTE: Run 2 impinger sample bottle broke during shipment. Therefore, only the front half is recorded. The average is based on Runs 1 and 3.

[ ] = Minimum Detection Limit

contaminated with Sb, Ba, Cd, Cr, Pb, Mn, Ni, Ag, and Zn. The back half fraction was contaminated with Ba, Cr, Pb, Mn, Ni, phosphorus (P), and Zn. The appropriate blank corrections were applied to the metals where contamination was found in the field blank. This resulted in 9 of the 15 metals exhibiting significant amounts detected in the flue gas samples. These metals are Ba, Cd, Cr, Cu, Pg, Mn, Ni, Ag, and Zn. Table 6-6 presents the metals method blank results for the flue gas samples. Lead, Mn and Ni were detected in the flue gas method blank at low levels. These amounts were negligible in comparison to the amounts found in the field blanks and, therefore, no blank corrections were applied based on these results.

Table 6-7 presents the method spike results for the metals analysis. All spiked recoveries for the front half fraction (except for Ag) were within the QA allowance of ±20 percent of 100 percent. Barium, Cu, and P were slightly below the 20 percent acceptance in the back half fraction, with 78.4 percent, 79.3 percent, and 78.2 percent, respectively. No spike corrections were applied.

6.3.2 PM_{10}/CPM Analytical Assurance

Table 6-8 presents the results of the PM_{10}/CPM field blank analysis compared to the test run results. Methylene chloride and acetone were detected in the field blank and were 0.8 percent and 3.1 percent, respectively, of the average run amount. The PM_{10}/CPM samples were gravimetrically analyzed according to EPA Method 5 requirements. Sample jars were checked to determine if leakage occurred during shipment. The residue for the cyclone catch, filter catch, organic fraction, and inorganic fraction were weighed to within 0.5 mg. The weight determinations were conducted at least six hours apart. Weight gain for each fraction was reported to the nearest 0.1 mg. Water and methylene chloride blanks were analyzed with the samples. Blank correction was not required because the sum of the values for the water blank and the methylene chloride blank was less than 2 mg.

6.3.3 Aldehydes Analytical Quality Assurance

Aldehydes field blank results are compared to the test run results in Table 6-9. Acetone was detected in the field blank at a noticeable level and formaldehyde was detected at a low level. The aldehydes method blank results for the flue gas samples are

Table 6-6

METALS AMOUNT IN FLUE GAS METHOD BLANK RESULT

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Metal	Flue Gas Method Blank	
	Front Half (total ug)	Impingers 1,2,3 (total ug)
Antimony	[1.50]	[1.59]
Arsenic	[0.400]	[0.424]
Barium	[0.100]	[0.106]
Beryllium	[0.100]	[0.106]
Cadmium	[0.200]	[0.212]
Chromium	[0.600]	[0.636]
Copper	[0.400]	[0.424]
Lead	[0.300]	(0.744)
Manganese	(0.330)	2.37
Nickel	[0.300]	(0.572)
Phosphorous	[30.0]	[31.8]
Selenium	[1.60]	[1.70]
Silver	[0.600]	[0.636]
Thallium	[10.0]	[10.6]
Zinc	(2.41)	[1.59]

[ ] = Minimum Detection Limit.

( ) = Estimated Value

Table 6-7

METALS METHOD SPIKE RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Metal	Method Spike (% rec)		Method Spike Duplicate (% rec)	
	Front Half	Impingers 1,2,3	Front Half	Impingers 1,2,3
Antimony	100%	86.8%	100%	96.8%
Arsenic	87.4%	86.0%	95.4%	97.8%
Barium	91.4%	88.2%	91.0%	78.4%
Beryllium	95.0%	83.4%	95.2%	93.4%
Cadmium	100%	98.5%	99.9%	88.5%
Chromium	99.6%	97.0%	99.4%	86.5%
Copper	93.5%	90.1%	93.9%	79.3%
Lead	97.8%	88.6%	102%	98.8%
Manganese	96.1%	94.0%	96.2%	84.9%
Nickel	99.9%	88.1%	100%	98.5%
Phosphorous	89.2%	78.2%	92.1%	86.5%
Selenium	99.0%	85.3%	99.6%	95.8%
Silver	74.5%	41.0%	24.7%	30.4%
Thallium	103%	94.7%	100%	97.5%
Zinc	103%	90.7%	104%	101%

Table 6-8

PM10/CPM FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Type	Test Run Average [a] (mg)	Field Blank (mg)
H2O	41.7	0.000 [b]
MeCl	5.03	0.040
Acetone	26.1	0.800

a = Runs 1,2,3

b = Showed a slightly negative weight gain, assumed to be zero

TABLE 6-9

ALDEHYDES FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Run 1 (total ug)	Run 2 (total ug)	Run 3 (total ug)	Field Blank (total ug)
Acetaldehyde	822	1040	1170	[5.80]
Acetone	23700	11200	2570	6640
Acetophenone/o-Tolualdehyde	[23.0]	[23.0]	[23.0]	[12.0]
Acrolein	[13.0]	[13.0]	26.7	[7.10]
Benzaldehyde	199	198	211	[11.0]
Butyraldehyde/Isobutyraldehyde	47.0	[18.0]	75.4	[9.40]
Crotonaldehyde	37.1	29.5	65.0	[9.40]
2,5-Dimethylbenzaldehyde	[24.0]	[24.0]	[24.0]	[13.0]
Formaldehyde	2950	4470	2770	10.7
Hexanal	40.7	62.5	20.0	[11.0]
Isophorone	[18.0]	[18.0]	[18.0]	[9.40]
Isovaleraldehyde	[18.0]	[18.0]	[18.0]	[9.80]
MIBK/p-Tolualdehyde	[23.0]	[23.0]	[23.0]	[12.0]
Methyl Ethyl Ketone	[18.0]	[18.0]	93.5	[9.40]
Propionaldehyde	[14.0]	[14.0]	94.6	[7.30]
Quinone	48.8	27.0	1040	[9.40]
m-Tolualdehyde	[23.0]	[23.0]	[23.0]	[12.0]
Valeraldehyde	[18.0]	[18.0]	56.9	[9.80]

[ ] = Minimum Detection Limit

presented in Table 6-10. Acetaldehyde, acetone, formaldehyde, and isovaleraldehyde were detected in the flue gas method blank at low levels. However, no corrections were made to the flue gas samples based on these results.

Table 6-11 presents the method spike results for the aldehyde analysis. All spike recoveries, except for acetophenone/o-tolualdehyde at 70 percent recovery, acrolein at 23 percent recovery, crotonaldehyde at 25 percent recovery, and quinone at 64 percent, are within the QA ±20 percent of the 100 percent. No spike corrections were applied.

6.3.4 Polynuclear Aromatic Hydrocarbon Results

Table 6-12 presents the PAH field blank results compared to the test run results. Naphthalene was detected in the field blank at a noticeable level, while methylnaphthalene and phenanthrene were detected at low levels. Blank corrections were applied based on these results. The PAH method blank results for the flue gas samples are presented in Table 6-13. Fluorene, 2-methylnaphthalene, naphthalene, and pyrene were detected in the flue gas method blank at low levels. No method blank corrections were applied.

Table 6-14 presents the method spike results for the PAH analysis. All spike recoveries, except for nitrophenol, pentachlorophenol, and di-n-butylphthalate, are within the QA ±20 percent of the 100 percent. No spike corrections were applied. Table 6-15 presents the PAH surrogate recovery results. 2-fluorobiphenyl was out of the lab control limits, but nitrobenzene-d5 and terphenyl-d14 were within control limits for all sample runs.

6.4 CONTINUOUS EMISSION MONITORING QUALITY ASSURANCES

Flue gas was analyzed continuously for O_2/CO_2, CO, SO_2, NO_x, and THC using EPA Reference Methods 3A, 10, 6C, 7E, and 25A, respectively. Daily QA/QC procedures were performed in accordance with QA/QC guidelines in the reference methods and Radian standard operating procedures. These procedures are fully detailed

Table 6-10

ALDEHYDES FLUE GAS METHOD BLANK RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Flue Gas Method Blank (total ug)	Flue Gas Field Blank (total ug)
Acetaldehyde	0.660	[5.80]
Acetone	1.74	6640
Acetophenone/o-Tolualdehyde	[1.20]	[12.0]
Acrolein	[0.710]	[7.10]
Benzaldehyde	[1.10]	[11.0]
Butyraldehyde/Isobutyraldehyde	[0.940]	[9.40]
Crotonaldehyde	[0.940]	[9.40]
2,5-Dimethylbenzaldehyde	[1.30]	[13.0]
Formaldehyde	1.06	10.7
Hexanal	[1.10]	[11.0]
Isophorone	[0.940]	[9.40]
Isovaleraldehyde	3.68	[9.80]
MIBK/p-Tolualdehyde	[1.20]	[12.0]
Methyl Ethyl Ketone	[0.940]	[9.40]
Propionaldehyde	[0.730]	[7.30]
Quinone	[0.940]	[9.40]
m-Tolualdehyde	[1.20]	[12.0]
Valeraldehyde	[0.980]	[9.80]

[ ] = Minimum Detection Limit.

Table 6-11

ALDEHYDES METHOD SPIKE RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Method Spike (% rec)
Acetaldehyde	90.0%
Acetone	83.0%
Acetophenone/o-Tolualdehyde	70.0%
Acrolein	23.0%
Benzaldehyde	119%
Butyraldehyde/Isobutyraldehyde	NA
Crotonaldehyde	25.0%
2,5-Dimethylbenzaldehyde	85.0%
Formaldehyde	87.0%
Hexanal	117%
Isophorone	91.0%
Isovaleraldehyde	NS
MIBK/p-Tolualdehyde	106%
Methyl Ethyl Ketone	NA
Propionaldehyde	107%
Quinone	64.0%
m-Tolualdehyde	93.0%
Valeraldehyde	82.0%

NA = Not Analyzed

NS = Not Spiked

TABLE 6-12

PAH FIELD BLANK RESULTS COMPARED TO TEST RUN RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Run 1 (Total ug)	Run 2 (Total ug)	Run 3 (Total ug)	Field Blank (Total ug)
Acenaphthylene	(41.8)	(37.7)	(48.1)	[50.0]
Acenoaphthene	(14.9)	[50.0]	[50.0]	[50.0]
Anthracene	(10.2)	(8.99)	(9.62)	[50.0]
Benzo(a)anthracene	[50.0]	[50.0]	[50.0]	[50.0]
Benzo(a)pyrene	[50.0]	[50.0]	[50.0]	[50.0]
Benzo(b)fluoranthene	[50.0]	[50.0]	[50.0]	[50.0]
Benzo(e)pyrene	[50.0]	[50.0]	[50.0]	[50.0]
Benzo(g,h,i)perylene	[50.0]	[50.0]	[50.0]	[50.0]
Benzo(k)fluoranthene	[50.0]	[50.0]	[50.0]	[50.0]
2-Chloronapthalene	[50.0]	[50.0]	[50.0]	[50.0]
Chrysene	[50.0]	[50.0]	[50.0]	[50.0]
Dibenz(a,h)anthracene	[50.0]	[50.0]	[50.0]	[50.0]
Dibenzofuran	[50.0]	[50.0]	[50.0]	[50.0]
7,12-Dimethylbenz(a)anthracene	[100]	[100]	[100]	[100]
Fluoranthene	[50.0]	[50.0]	[50.0]	[50.0]
Fluorene	[50.0]	[50.0]	[50.0]	[50.0]
Indeno(1,2,3-cd)pyrene	[50.0]	[50.0]	[50.0]	[50.0]
2-Methylnaphthalene	408	348	417	(2.36)
Naphthalene	416	776	1470	751
Perylene	[50.0]	[50.0]	[50.0]	[50.0]
Phenanthrene	(9.04)	(7.94)	(8.49)	(0.275)
Pyrene	(1.26)	(0.625)	(0.805)	[50.0]

[ ] = Minimum Detection Limit

( ) = Estimated Value

Table 6-13

PAH FLUE GAS METHOD BLANK RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Flue Gas Method Blank (total ug)
Acenaphthylene	[50.0]
Acenaphthene	[50.0]
Anthracene	[50.0]
Benzo(a)anthracene	[50.0]
Benzo(a)pyrene	[50.0]
Benzo(b)fluoranthene	[50.0]
Benzo(e)pyrene	[50.0]
Benzo(g,h,i)perylene	[50.0]
Benzo(k)fluoranthene	[50.0]
2-Chloronapthalene	[50.0]
Chrysene	[50.0]
Dibenz(a,h)anthracene	[50.0]
Dibenzofuran	[50.0]
7,12-Dimethylbenz(a)anthracene	[100]
Fluoranthene	[50.0]
Fuorene	(0.375)
Indeno(1,2,3-cd)pyrene	[50.0]
2-Methylnaphthalene	(0.680)
Naphthalene	260
Perylene	[50.0]
Phenanthrene	[50.0]
Pyrene	(0.805)

[ ] = Minimum Detection Limit.

( ) = Estimated Value

Table 6-14

PAH METHOD SPIKE RESULTS

MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Method Spike (% rec)
Phenol	84.8%
2-Chlorophenol	94.2%
1,4-Dichlorobenzene	95.6%
N-Nitroso-di-n-propylamine	104%
1,2,4-Trichlorobenzene	109%
4-Chloro-3-methylphenol	99.9%
4-Nitrophenol	53.0%
2,4-Dinitrotoluene	94.6%
Pentachlorophenol	26.6%
Di-n-butlyphthalate	2.15%

Table 6-15
PAH SURROGATE RECOVERY RESULTS
MATHY CONSTRUCTION COMPANY PLANT 6 (1991)

Compound	Run 1 (% Rec)	Run 2 (% Rec)	Run 3 (% Rec)	Field Blank (% Rec)	Method Bla (% Rec)	Method Spi (% Rec)	Control Limits
2-Fluorobiphenyl	146%	124%	153%	141%	130%	143%	30% to 115%
Nitrobenzene-d5	113%	93.9%	113%	96.4%	87.4%	105%	23% to 120%
Terphenyl-d14	112%	111%	121%	112%	111%	127%	18% to 137%

in the site-specific test plan prepared by Radian Corporation.[1] A summary of the QA/QC parameters and results is provided in this section. Deviations from the test plan and/or problems encountered during the test program are also discussed.

6.4.1 Calibration and Drift Assessments

Continuous monitoring instruments were calibrated at the beginning of the test period on a two-point basis using a zero gas (N_2) and a high-range span gas. A mid-range gas was analyzed with no adjustment permitted as a QC check at least once on site. The observed mid-range QC gas concentration was within ±2 percent of full scale for the linearity check to be considered acceptable. The results of this check are presented in Tables 6-16 through 6-21.

In addition to conducting the linearity check, the instrument drift was also determined for each analyzer on a daily basis. Typically, the mid-range gas was analyzed at mid-day and/or at the end of each test day to determine an "inter-run" drift; however, the span gas was used in some cases to conserve gases that were available in more limited quantities. A drift check was not determined between every manual run to allow completion of as many manual method tests as possible while the batch operated plant was actually on-line. The inter-run instrument drift value was calculated as a difference on a percent scale basis by comparing the current observed response to the previous response. The instrument drift over the entire test program was calculated similarly, except that the final mid-gas observed response was compared to the initial mid-gas observed response. These inter-run and overall drift values are also provided in Tables 6-16 through 6-21. The allowable drift of ±3 percent of full scale which was met in all cases except for one inter-run check of the NO_x analyzer; however, the overall drift for this analyzer over the entire test period was determined as 0.6 percent, which is within ±3 percent limit.

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[1]"Emission Testing for Asphalt Concrete Industry, Site-Specific Test Plan and Quality Assurance Project Plan, Mathy Construction Company Plant #6," Radian Corporation, September 1991.

Table 6-16

Method 3A Oxygen Analyzer and Drift Summary
Mathy Construction Company-Plant 6

Instrument: Thermox WDG III				Serial Number: 35983-1		
Span Gas Concentration: 25%V						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/19/91	08:35	Mid:	7.99	7.7	-0.29	-1.16
9/19/91	08:34	Span:	18.0	18.0	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Mid:	7.99	7.6	-0.1	-0.4
9/19/91	15:53	Mid:	7.99	7.6	0.0	0.0
9/20/91	6:51	Mid:	7.99	7.2	-0.4	-1.6
9/20/91	15:40	Mid:	7.99	7.4	0.2	0.8
Test Period Drift Summary[c]						
9/20/91	15:40	Mid:	7.99	7.4	-0.3	-1.2

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

Table 6-17

Method 3A Carbon Dioxide Analyzer and Drift Summary
Mathy Construction Company, Plant 6

Instrument: Beckman 865				Serial Number: 0103778		
Span Gas Concentration: 20%V						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/19/91	08:35	Mid:	9.91	10.3	0.4	2.0
9/19/91	08:34	Span:	17.0	17.0	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Mid:	9.91	10.3	0.0	0.0
9/19/91	15:53	Mid:	9.91	10.6	0.3	1.5
9/20/91	15:40	Mid:	9.91	10.2	-0.4	-2.0
Test Period Drift Summary[c]						
9/20/91	15:40	Mid:	9.91	10.2	-0.1	0.5

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

Table 6-18

Method 10 Carbon Monoxide Analyzer and Drift Summary
Mathy Construction Company, Plant 6

Instrument: TECO 48				Serial Number: 4829758-236		
Span Gas Concentration: 500 ppmv						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/19/91	08:35	Mid:	92.1	88.3	-3.8	-0.76
9/19/91	08:34	Span:	474	474	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Mid:	92.1	89.9	1.6	0.3
9/19/91	15:53	Mid:	92.1	93.7	3.8	0.8
9/20/91	06:51	Mid:	92.1	82.2	-11.5	-2.3
Test Period Drift Summary[c]						
9/20/91	06:51	Mid:	92.1	82.2	-6.1	-1.2

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

Table 6-19

Method 6C Sulfur Dioxide Analyzer and Drift Summary
Mathy Construction Company, Plant 6

Instrument: Western Research 721 AT2			Serial Number: 90-721AT2-7654-1			
Span Gas Concentration: 500 ppmv						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/20/91	06:51	Mid:	98.0	95.8	-2.2	-0.4
9/19/91	08:34	Span:	295	295	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Span:	295	299	4	0.8
9/19/91	15:53	Span:	295	300	1	0.2
9/20/91	12:38	Span:	295	287	-8	-1.6
Test Period Drift Summary[c]						
9/20/91	15:40	Low:	98.0	97.7	1.9	0.4

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

Table 6-20

Method 7E Nitrogen Oxides Analyzer and Drift Summary
Mathy Construction Company, Plant 6

Instrument: TECO 10AR				Serial Number: 2558-221		
Span Gas Concentration: 250 ppmv						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/20/91	06:51	Low:	44.0	44.0	0.0	0.0
9/19/91	08:34	Span:	201	201	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Span:	201	202	1	0.4
9/19/91	15:53	Span:	201	206	4	1.6
9/20/91	06:48	Span:	201	201	-5	-2.0
9/20/91	12:38	Span:	201	212	11	4.4
Test Period Drift Summary[c]						
9/20/91	15:40	Low:	44.0	45.5	1.5	0.6

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

Table 6-21

Method 25A Total Hydrocarbon Analyzer and Drift Summary
Mathy Construction Company, Plant 6

Instrument: Ratfisch 55				Serial Number: 212291		
Span Gas Concentration: 100 ppmv						
Date	Time		Certified Gas Conc (%V)	Observed Gas Conc (%V)	Difference (%V)	Difference Percent Scale (%)
Multipoint Linearity[a]						
9/19/91	08:34	Zero:	0.0	0.0	0.0	0.0
9/20/91	06:51	Low:	10.1	9.9	-0.2	-0.2
9/19/91	08:34	Span:	41.4	41.4	0.0	0.0
Inter-run Drift Summary[b]						
9/19/91	12:55	Span:	41.4	40.6	-0.8	-0.8
9/19/91	15:53	Span:	41.4	41.2	0.6	0.6
9/20/91	06:48	Span:	41.4	41.4	0.2	0.2
9/20/91	12:38	Span:	41.4	41.6	-0.2	-0.2
9/20/91	15:02	Span:	41.4	41.3	-0.3	-0.3
Test Period Drift Summary[c]						
9/20/91	15:02	Low:	10.1	9.4	-0.5	-0.5

[a]Difference Percent Scale = (observed conc. - certified conc.)/span value * 100%.

[b]Difference Percent Scale = (current observed conc. - previous observed conc.)/span value * 100%.

[c]Difference Percent Scale = (final observed conc. - initial observed conc.)/span value * 100%.

6.4.2 Line Bias Checks

Radian performed all multi-point and QC calibrations through the entire sampling system. A three-way valve was located between the reference method CEM probe and the heat-traced line. This valve was shut during calibration and QC drift checks, and the standard gases were directed from the gas cylinder through the heat-traced line to the analyzer probe and back before the gas was directed to the CEM analyzers. This procedure eliminated the need for performing line bias checks described in Methods 3A, 7E, and the test plan.

6.4.3 Leak Checks

Since Radian performed all calibrations through the entire sampling system, leak checks were incorporated in each calibration. The criterion used for this test was an O_2 response to a zero gas of less than 0.5 percent O_2. All leak checks performed at this test site met this criterion.

6.5 GAS CHROMATOGRAPHY QUALITY ASSURANCE

EPA Method 18 analysis of the flue gas was performed using a GC to separate the hydrocarbon (C_1-C_6) species in the gas stream. At the beginning of each test day prior to sampling the flue gas, the GC was calibrated with standard gas mixtures containing each hydrocarbon (methane, ethane, propane, butane, pentane, hexane) and an instrument response factor for each hydrocarbon was determined. Response factors for each hydrocarbon were determined again at the end of each test day after sampling was completed. In this way, a daily calibration drift was determined for these compounds. Additional calibrations were completed for benzene, toluene, and xylene (BTX). Only a single calibration was completed daily for BTX compounds. The calibration drift values are shown in Table 6-22. The drift values for C_1 through C_6 were determined to be less than 7 percent, which is within the QA allowance criterion of 10 percent. Based on these low drift values, the BTX drift values would not be expected to higher than the 10 percent criterion.

Table 6-22

GC Response Factor Drift Values
Mathy Construction Company-Plant 6
(1991)

Date	09/19/91			09/20/91		
Compound	Pre-Test Response Factor[a]	Post-Test Response Factor	Drift (%)[b]	Pre-Test Response Factor	Post-Test Response Factor	Drift (%)
Methane	2.823×10^{-5}	2.788×10^{-5}	-1.26	3.0855×10^{-5}	3.3123×10^{-5}	6.85
Ethane	1.371×10^{-5}	1.355×10^{-5}	-1.18	1.3928×10^{-5}	1.4493×10^{-5}	3.90
Propane	9.264×10^{-6}	9.123×10^{-6}	-1.55	9.4026×10^{-6}	9.7336×10^{-6}	3.40
Butane	6.710×10^{-6}	6.783×10^{-6}	1.08	6.4131×10^{-6}	6.7103×10^{-6}	4.43
Pentane	5.401×10^{-6}	5.414×10^{-6}	0.24	5.4065×10^{-6}	5.6770×10^{-6}	4.76
Hexane	4.522×10^{-6}	ND	--	4.6559×10^{-6}	4.8493×10^{-6}	3.99
Benzene	ND	4.413×10^{-6}	--	3.9334×10^{-6}	ND	--
Toluene	ND	3.632×10^{-6}	--	4.1761×10^{-6}	ND	--
Ethylbenzene	ND	2.838×10^{-6}	--	2.3932×10^{-6}	ND	--
Xylene	ND	2.500×10^{-6}	--	2.2779×10^{-6}	ND	--

ND = Not Determined

[a]Response Factor =

$$\frac{\text{Calibration Gas Concentration (ppm)}}{\text{Peak Area Count}}$$

[b]Drift Percent =

$$\frac{\text{(Post-Test Response Factor - Pre-Test Response Factor)}}{\text{Post-Test Response Factor}} \times 100$$

APPENDIX A

EMISSIONS TESTING FIELD DATA SHEETS

A.1 PM/Metals
A.2 PM_{10}/CPM
A.3 Aldehydes
A.4 PAH

APPENDIX A.1

PM/METALS

METALS

PLANT Murphy, La Cygne
DATE 4-19-91
SAMPLING LOCATION Cyclone
SAMPLE TYPE Metals
RUN NUMBER 1
OPERATOR JBF
AMBIENT TEMPERATURE 40
BAROMETRIC PRESSURE 29.1
STATIC PRESSURE (P_g)
FILTER NUMBER (s)
Initial Orifice at 15" Hg

PROBE LENGTH AND TYPE 6' glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 20 %
SAMPLE BOX NUMBER
METER BOX NUMBER N-33
METER $\Delta H_@$ 2.00
~~K~~ FACTOR .9875
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE ΔP K = 1.1

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 5 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0	0710	05.212									
A1	5	0715	11 8.7	.25		1.8	153	45	42	1	261	3[illegible]
2	10	720	11.6	1.8		1.2	257	49	42	1	251	46
3	15	725	14.6	1.2		.79	254	51	43	1	230	42
4	20	730	16.6	.85		.56	~~244~~ 231	53	45	1	244	41
5	25	735	16.725	.25		.16	226	54	46	1	245	42
STOP		736	"	PORT CHANGE								
B1	30	741	20.3	2.9		1.9	236	54	47	1	276	39
2	35	746	23.5	2.5		1.6	237	57	48	1	290	39
3	40	751	26.0	1.3		.85	234	~~56~~	49	1	270	40
4	45	756	27.9	.67		.60	235	59	50	1	274	40
5	50	801	29.378	.24		.16	226	57	50	1	277	41
STOP		802	"	PORT CHANGE								
C1	55	~~807~~ 807	33.0	2.8		1.9	239	58	51	1	280	40
2	60	812	36.4	2.4		1.6	234	59	51	1	280	40
3	65	817	38.8	1.0		69	234	62	52	1	282	40
4	70	822	41.0	.97		.68	240	60	53	1	285	41
5	75	827	42.632	.34		.25	254	63	57	1	275	45
STOP		831	"									
D1	80	836	46.5	.27		1.9	245	59	54	2	273	38
2	85	841	49.9	.26		1.8	246	63	55	2	267	41
3	90	846	52.4	~~1.1~~ 1.1		.70	246	64	55	1	266	40
4	95	851	53.9	.42		.31	244	63	56	1	272	41
5	100	856	54.951	.16		.11	230	60	57	1	278	42

COMMENTS:

TM 015

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m, ft³)	VELOCITY HEAD (Δp_s, in H_2O)	ORIFICE PRESSURE DIFFERENTIAL (ΔH, in H_2O)		STACK TEMPERATURE (T_s, °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE °F	IMPINGER TEMPERATURE °F
				DESIRED	ACTUAL		INLET ($T_{m\,in}$, °F	OUTLET ($T_{m\,out}$, °F			
	100 / 558	54.951									
E1	116/105 903	58.5	2.4		1.7	294	61	58	1	267	41
2	#5 110 908	61.9	2.1		1.5	243	64	58	1	275	40
3	#20 115 913	63.99	1.1		.71	243	66	59	1	249	43
4	+120 918	65.9	.60		.42	243	65	59	1	279	43
5	125 923	67.454	.22		.15	271	64	59	1	265	43
		FINAL	LEAK		0.010 at		10" Hg				

Metals Run 1

RADIAN CORPORATION

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)	
1	MT	—	MOD 6-S	Final	819.4
				Initial	478.4
				Wt. Gain	341.0
2	5% HNO_3/10% H_2O_2	200	MOD 6-S	Final	800.9
				Initial	649.4
				Wt. Gain	151.5
3	5% HNO_3/10% H_2O_2	200	MOD 6-S	Final	711.4
				Inital	661.3
				Wt. Gain	50.1
4	MT	—	MOD 6-S	Final	479.1
				Initial	478.6
				Wt. Gain	.5
5	Silica Gel	~200 (g)	MOD 6-S	Final	693.3
				Initial	682.8
				Wt. Gain	10.5
6				Final	
				Initial	
				Wt. Gain	
7				Final	
				Initial	
				Wt. Gain	

Total Impingers Weight Gain (Wc), grams 553.5 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

Filter no. TM-018

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = .0472(W_c) = .0472(_____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

PLANT Natly
DATE 9/19
SAMPLING LOCATION Outlet
SAMPLE TYPE Basics
RUN NUMBER 2
OPERATOR JBR
AMBIENT TEMPERATURE 56
BAROMETRIC PRESSURE 29.7
STATIC PRESSURE, (P_s)
FILTER NUMBER (s)

PROBE LENGTH AND TYPE 6' Glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 27.%
SAMPLE BOX NUMBER
METER BOX NUMBER N-33
METER $\Delta H_@$ 2.00
C FACTOR .9x75
PROBE HEATER SETTING 270
HEATER BOX SETTING 256
REFERENCE Δp .7

L
M7/M26
Dual

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 5 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0	1329	97.834									
A 1	5	1333	101.4	2.7		1.9	~~269~~ 225	58	59	1	~~275~~ 274	47
2	10	1338	104.7	2.2		1.5	211	58	60	1	265	43
3	15	1343	107.3	1.1		.77	222	58	61	1	271	46
4	20	1348	109.2	.63		.42	229	59	63	1	~~275~~	48
5	25	1353	110.376	.25		.18	228	59	63	1	275	49
STOP		1354	"									
B 1	30	1359	114.3	3.2		2.2	239	60	64	1	272	49
2	35	1404	~~120.~~ 117.0	2.3		1.6	242	60	66	1	263	49
3	40	1409	120.0	1.1		.77	242	61	70	1	273	50
4	45	1414	121.6	.36		.24	241	62	69	1	255	50
5	50	1419	122.496	.17		.12	232	63	68	1	266	50
STOP		1421	"									
C 1	55	1426	126.3	2.7		1.9	244	63	67	1	279	48
2	60	1431	129.2	1.8		1.2	244	64	70	1	274	45
3	65	1436	131.6	1.0		.75	244	64	71	1	267	44
4	70	1441	133.2	.33		.23	242	65	71	1	269	44
5	75	1446	133.917	.10		.07	230	66	70	1	267	45
		1447	"									
D 1	80	1452	137.0	2.7		1.9	244	65	70	1	275	43
2	85	1457	140.8	2.2		1.5	243	66	72	1	269	43
3	90	1502	143.3	1.1		.77	242	66	73	1	276	45
4	95	1507	144.9	.50		.35	240	66	72	1	268	44
5	100	1512	146.213	.17		.12	229	67	73		275	45

COMMENTS:

EPA (Dar) 235
4-72

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)		GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F			
	100	1515	146.213									
E1	105	1520	149.4	2.3		1.5	242	69	67	2	274	48
2	110	1525	152.5	2.1		1.3	243	71	67	2	257	44
3	115	1530	154.9	1.2		.80	243	73	68	2	263	46
4	120	1535	156.9	.67		.46	239	73	68	2	261	46
5	125	1540	157.911	.14		.10	221	72	68	1	263	47
			FINAL LEAK		.008 at 35" Hg							

RADIAN CORPORATION

Metals Run 2

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	MT	—	MOD G-S	Final 768.7 Initial 471.5 Wt. Gain 297.2
2	5% HNO_3/10% H_2O_2	200	MOD G-S	Final 784.1 Initial 642.8 Wt. Gain 141.3
3	5% HNO_3/10% H_2O_2	200	MOD G-S	Final 717.2 Inital 647.0 Wt. Gain 70.2
4	MT	—	MOD G-S	Final 475.3 Initial 474.3 Wt. Gain 1
5	Silica Gel	~200 (g)	MOD G-S	Final 652.7 Initial 642.4 Wt. Gain 10.3
6				Final Initial Wt. Gain
7				Final Initial Wt. Gain

Total Impingers Weight Gain (Wc), grams 520.0 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

Filter no. TM-005

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = $.0472(____ g)$ = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

PLANT Mathy CC
DATE 9-26-91
SAMPLING LOCATION Outlet
SAMPLE TYPE Metals
RUN NUMBER 3
OPERATOR JDC
AMBIENT TEMPERATURE 50
BAROMETRIC PRESSURE 29.7
STATIC PRESSURE, (P_g)
FILTER NUMBER (s)

PROBE LENGTH AND TYPE 6' glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 17.2
SAMPLE BOX NUMBER
METER BOX NUMBER 1-33
METER $\Delta H_@$ 2.00
C_p FACTOR .9875
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE Δp .65 70

Metals Dual

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 5 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	C'	C946	61.452									
E 1	5	0951	65.0	2.5		1.8	235	48	47	1	260	41
2	10	0956	67.9	2.0		1.4	240	50	47	1	260	40
3	15	1001	70.5	1.1		.68	244	54	48	1	263	42
4	20	1006	72.3	.67		.47	243	55	49	1	261	44
5	25	1011	73.300	.24		.17	240	57	53	1	260	44
		1013	"									
D 1	30	1018	76.8	2.7		1.9	247	56	52	1	262	45
2	35	1023	80.0	2.2		1.5	248	59	52	1	263	40
3	40	1028	82.2	1.0		.70	247	61	53	1	260	40
4	45	1033	83.9	.51		.36	245	63	57	1	259	39
5	50	1038	84.623	.10		.07	238	60	55	1	260	41
STOP		1042	"									
C 5	55	1047	87.9	2.8		2.1	245	62	57	1	260	42
4	60	1052	89.9	2.2		1.5	243	67	59	1	266	40
3	65	1057	94.0	1.0		.070	243	71	60	1	262	42
2	70	1102	95.8	.46		.32	243	73	62	1	254	42
1	75	1107	96.785	.16		.11	236	74	64		256	44
STOP		1111	"									
B 5	80	1116	99.5	1.5		1.0	249	74	65	1	271	45
4	85	1121	102.1	1.3		.91	250	79	68	1	266	42
3	90	1126	104.4	.98		.68	248	80	70	1	260	42
2	95	1131	106.2	.63		.45	249	81	71	1	265	44
1	100	1136	107.877	.40		~~.40~~ .36	235	82	73	1	262	44

COMMENTS:

EPA (Dm) 235
4/77

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)		GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F			
	100	1138	107.877									
A 1	105	1143	111.0	2.6		1.5	254	84	76	2	267	46
2	110	1148	114.3	2.0		1.4	253	86	75	2	266	44
3	115	1153	116.7	1.0		.7	256	88	75	1	264	45
4	120	1158	118.7	0.5		.35	253	89	78	1	263	46
5	125	1203	119.834	0.2		.14	243	86	78	1	264	48
			Final	0.014	at	5" Hg						

RADIAN CORPORATION

Metals Run 3

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)	
1	MT	—	MOD 6-S	Final	810.6
				Initial	481.1
				Wt. Gain	
2	5% HNO_3/10% H_2O_2	200	MOD 6-S	Final	809.4
				Initial	679.1
				Wt. Gain	
3	5% HNO_3/10% H_2O_2	200	MOD 6-S	Final	730.2
				Inital	692.8
				Wt. Gain	
4	MT	—	MOD 6-S	Final	480.8
				Initial	480.5
				Wt. Gain	
5	Silica Gel	~200 (g)	MOD 6-S	Final	702.4
				Initial	693.2
				Wt. Gain	
6				Final	
				Initial	
				Wt. Gain	
7				Final	
				Initial	
				Wt. Gain	

Total Impingers Weight Gain (Wc), grams 3026.7 (5007) Analyst VLL

Filter no. ~~TM-0006~~ Filter no. TM-001

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = $.0472(____ g)$ = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

APPENDIX A.2

PM_{10}/CPM

Run Code 1	Date 9/19/91	Stack Temperature 245 (°F)
Sampler ID ERP	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID 13 => .2130	End Time	Ambient Temperature 50 (°F)
Sampler Orientation vertical	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location Stack	DGM (initial) 67.844	Gas Velocity
Nozzle Diameter-ID .182 (in)	DGM (final)	System Leak Check .01 @ 10"
Operator(s) CH	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed? ✓		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes N-33 Y=.9875 ΔH@=2.00
Moisture Content		

Run Time	Port No / Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a]
1040:30	A1	1.8	.56		225	250	57/56	260
1043	A3	1.0	.56		230		60/58	150
1044:46	A5	.35	.56	71.4	231		61/57	106
1047:10	A4	.65	.56	72.4	232		62/58	144
1050:49	A2	1.5	.56	74.15	231		63/58	219
14:39								
1054				74.15				
1058:46	A1 B1	3.5	.56	76.04	246		66/59	230
11:02:46	A2 B2	2.3	.56	77.6	242		64/60	226
11:05:38	A3 B3	1.5	.56	78.82	245		63/6.	182
11:07:35	A4 B4	0.72	.56	79.7	247		68/62	122
11:08:54	A5 B5	.36	.56	80.514	247		69/61	92

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n} = 150.1\sqrt{P_n}$

Figure 6-1. CSR field data sheet.

Mutny 6

Run 1

Pg 2 of 2

624

Run Code 1	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Run Time	Port No / Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a]
11:10:36								
11:15:58	C1	3.2	.56		244		69/62	261
11:19:45	C2	2.3	.56	87.3	.245		70/67	227
11:22:17	C3	1.0	.56	85.4	.244		71/64	150
11:23:45	C4	.38	.56	~86.0	.244		71/64	93
11:24:52	C5	.20	.56	86.572	245		71/64	67
11:27:22				"				
11:28								
11:31:48	D1	3.2	.56	88.45	244		70/64	268
11:35:08	D2	2.3	.56	~90	244		71/65	225
11:37:59	D3	1.3	.56	91.0	245		71/65	171
11:39:34	D4	.75	.56	91.8	244		71/66	103
11:40:46	D5	.2	.56	92.288	244			62
13:46								

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_n}}$ = 150.11 $\sqrt{\Delta P_n}$

Run Code	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Run Time	Port No / Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a]
	E1	2.6		93.7	244		72/67	2.42
	E2	2.2		95.2	245		72/66	2.24
	E3	1.15		96.35	244		73/67	1.61
	E4	.60		97.1	245		73/64	1.16
	E5	.21		97.613	241		73/62	.71
		FINAL LEAK =			0.003 @ 3"			

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n}$

Figure 6-1. CSR field data sheet.

RADIAN CORPORATION

PM 10 Run 1

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	DI H_2O	100	MOD G-S	Final 750.4 Initial 548.7 Wt. Gain 201.7
2	DI H_2O	100	MOD G-S	Final 607.0 Initial 573.2 Wt. Gain 33.8
3	MT	—	MOD G-S	Final 478.4 Inital 472.10 Wt. Gain 6.3
4	Silica Gel	~200 (g)	MOD G-S	Final 662.1 Initial ~~671.5~~ 656.6 Wt. Gain 5.5
5				Final Initial Wt. Gain
6				Final Initial Wt. Gain
7				Final Initial Wt. Gain

Total Impingers Weight Gain (Wc), grams 275.5 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

Filter no. PM-1C

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3 .2130

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = .0472(____g) = ____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} =$$

Run Code PM_{10}-Run 2	Date 9/20/91	Stack Temperature ~245 (°F)
Sampler ID CRP	Start Time 11:27	Differential Stack Pressure +.8 (in. H_2O)
Filter ID	End Time	Ambient Temperature 55 (°F)
Sampler Orientation SITTING	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location Monty 6	DGM (initial) 27.576	Gas Velocity
Nozzle Diameter-ID .182 (in)	DGM (final)	System Leak Check
Operator(s) CRP	Sample Volume (ft^3)	Initial = 0-0.15 @ 10"
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content ~29 (Assumed)	✓ ✓	

1.3

+.8

Pt Duration	Clock 11287	Run Time	Port No / Trav. Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe oven	T DGM	Dwell Time[a] (sec)
3:34	11:30:34	~~2:34~~	E1	2.5	0.56	29.1	255	270	63/62	~~49~~ 214
3:20	11:33:54	6:54	E2	2.2	0.56	30.5	256	257	64/62	200
2:46	11:36:40	9:40	E3	1.5	0.56	31.6	253	250	66/64	166 ~~200~~
2:49	11:39:29	10:29	E4	.65	.56	32.3	250	250	67/66	107
2:03	11:37:32	11:32	E5	.22	.56	32.83	248	250	69/64	63
	1141	11:32				"				
4:02	11:45:02	15:34	D1	3.2	.56	34.5	252	241	64/64	242
3:34	11:48:36	19:08	D2	2.5	.56	35.95	240	260	68/64	214
2:37	11:51:13	21:45	D3	1.35	.56	37.1	234	261	70/65	157
2:40	11:52:53	23:25	D4	.55	.56	37.75	257	225	72/65	100
2:01	11:53:54	24:26	D5	.20	.56	38.364	254	214	71/66	61

25

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n} = 135.3 \sqrt{\Delta p}$

$\frac{213}{\sqrt{2.5}} = 135.2$

$t_n = \sqrt{\Delta p_n} \times 135.2$

V_m = 27.001

$T_{m_{avg}}$ = 70.10

ΔH_{avg} = 0.53

Figure 6-1. CSR field data sheet.

Run Code PM_{10}-2 (Pt 2 of 3)	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Dwell pt. Duration (min)	Clock 11:55	Run Time 24:26	Port No / Trav. Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a] (sec)
4:02	11:59:02	28:28	C1	3.2	.56	40	262	231	71/66	242
3:34	12:02:36	32:02	C2	2.5	.56	41.25	259	247	72/67	214
2:22	12:04:58	34:24	C3	1.1	.56	42.5	257	270	73/67	142
1:42	12:06:40	36:06	C4	0.57	.56	43.3	255	247	74/68	102
0:52	12:07:32	36:58	C5	.15	.56	43.703	250	271	73 68	52
	12:23:30	36:58								37
4:20	12:27:50	41:18	B1	3.7	.56	45.5	258	257	73/71	260
3:36	12:31:26	44:54	B2	2.55	.56	47.2	261	212	75/73	216
2:28	12:33:54	47:22	B3	1.2	.56	48	259	212	76/72	148
1:36	12:35:30	48:58	B4	0.50	.56	48.8	258	251	77 72	96
0:50	12:36:24	49:52	B5	0.16	.56	49.178	247	270	76/72	54
										50

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n} = \sqrt{\Delta p_n} \times 135.3$

Figure 6-1. CSR field data sheet.

Run Code PM_{10}-2 (3 of 3)	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Dwell (min)	Clock	Run Time	Port No / Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe Out	T DGM	Dwell Time[a]
	12:38:30	49:52								
1:04	12:42:34	53:56	A1	3.25	.56	50.9	261	2678	76/77	244
3:21	12:45:55	57:16	A2	2.2	.56	~52	259	276	78/74	201
1:34	12:48:29	59:50	A3	1.3	.56	53.35	256	272	78/74	154
:56	12:50:25	61:46	A4	0.76	.56	5415	254	254	79/74	116
:17	12:51:42	63:03	A5	.32	.56	54.737	252	255	78/74	77s
			Leak	.003	@ 3"					

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n}$

Figure 6-1. CSR field data sheet.

RADIAN CORPORATION

PM10 Run 2

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)	
1	DI H_2O	100	MOD 6-S	Final	717.4
				Initial	618.7
				Wt. Gain	[illegible]
2	DI H_2O	100	MOD 6-S	Final	767.6
				Initial	650.7
				Wt. Gain	[illegible]
3	MT	—	MOD 6-S	Final	499.6
				Inital	496.4
				Wt. Gain	[illegible]
4	Silicagel	~200(g)	MOD 6-S	Final	646.6
				Initial	640.7
				Wt. Gain	[illegible]
5				Final	
				Initial	
				Wt. Gain	
6				Final	
				Initial	
				Wt. Gain	
7				Final	
				Initial	
				Wt. Gain	

Total Impingers Weight Gain (Wc), grams 224.7 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3 Filter no. PM10-07

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

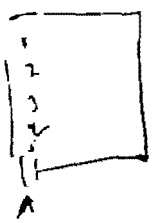
P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = $.0472(____ g)$ = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

Run Code: PM_{10} → Run 3	Date: 9/20/91	Stack Temperature (°F)
Sampler ID: CRP	Start Time: 14:13	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature: ~65 (°F)
Sampler Orientation: Horizontal	Sampling Duration: ~60 min (min)	Ambient Pressure (in. Hg)
Sampling Location: Mathy 6 stack	DGM (initial): 55.300	Gas Velocity
Nozzle Diameter-ID: .162 (in)	DGM (final)	System Leak Check: .012 @ 10" Initial
Operator(s): CRP	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed? ✓		
Magnehelics Zeroed?		
Gas Composition: %CO_2 %O_2 %CO	Pitot Leak Check: (Pos) (Neg)	Notes
Moisture Content		



Clock 1413

Run Time	Port No / Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a] (sec)	Dwell min
14:17:20	4:20	B1	3.7	.56	57.1	287	270	66/67	260	4:20
14:20:41	7:41	B2	2.2	.56	58.5	230	224	74/67	280?	3:21
14:23:06	10:06	B3	1.15	.56	59.44	235	270	70/67	142	2:25
14:24:46	11:46	B4	.55	.56	60.1	235	295	70/67	100	1:40
14:25:43	12:43	B5	.18	.56	60.506	233	295	71/67	57	0:57
14:26:30					"					
14:30:24	16:37	A1	3.0	.56	62	239	298	71/67	234	3:57
14:33:36	17:51	A2	2.05	.56	63.4	240	298	73/68	194	3:14
14:36:06	22:14	A3	1.2	.50	64.5	240	300	74/67	148	2:28
14:37:55	24:08	A4	.65	.56	65.2	236	300	75/67	109	1:49
14:39:04	25:17	A5	.26	.56	65.767	227	300	75/70	69	

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n}$

135.3 × $\sqrt{\Delta p}$ = Dwell Time

Figure 6-1. CSR field data sheet.

Run Code PM_{10} - 3 (2 or 3)	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Run Time	Port No Trav.Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a] (sec)	Dwell (min)
14.42 25:17									
14:45:44	29:01	E1	2.75	.56	67	243	300	71/70	224	3.44
14:48:55	32:12	E2	2.0	.56	68.6	248	275	76/70	191	3:11
14:51:35	34:52	E3	1.4	.56	69.73	247	300	76/71	160	2:40
14:53:13	36:30	E4	0.52	.56	70.4	243	300	76/71	98	1.38
14:53:56	37:13	E5	.1	.56	70.738	238	300	77/72	43	0:43
14:58:00	37:13								
15:01:54	41:07	D1	3.0	.56	72.3	249	292	77/72	234	3:57
15:05:12	44:25	D2	2.15	.56	738	250	295	78/72	198	3:18
15:07:31	46:44	D3	1.05	.56	741	248	297	77/73	139	2:19
15:09:51	48:04	D4	.38	.56	75.2	248	300	78/73	82	1:22
15:14:42	48:55	D5	.14	.56	75.578	248	288	78/74	51	0:51

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n}$

Figure 6-1. CSR field data sheet.

Run Code: PM10 - 3 (3 of 7)	Date	Stack Temperature (°F)
Sampler ID	Start Time	Differential Stack Pressure (in. H_2O)
Filter ID	End Time	Ambient Temperature (°F)
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)
Sampling Location	DGM (initial)	Gas Velocity
Nozzle Diameter-ID (in)	DGM (final)	System Leak Check
Operator(s)	Sample Volume (ft^3)	
Dual Manometer Leveled and Zeroed?		
Magnehelics Zeroed?		
Gas Composition %CO_2 %O_2 %CO	Pitot Leak Check (Pos) (Neg)	Notes
Moisture Content		

Run Time	Port No / Trav. Pt.	ΔP Pitot	ΔH Sample	DGM Volume	T Stack	T Probe	T DGM	Dwell Time[a] (sec)	Dwell (min)
15:11:30 48:23									
15:15:43	53:08	C1	3.5	.56	77.3	251	271	71/74	253	4:13
15:19:06	56:33	C2	2.3	.56	78.7	250	273	79/75	205	3:21
15:21:30	58:55	C3	1.1	.56	79.7	253	270	80/75	142	2:22
15:22:52		C4	.37	~~.37~~.56	80.25	250	273	80/78	82	1:22
15:23:35		C5	.10	.56	80.543	250	272	80/71	43	0:43
			Leak	.004	@ 3"				

[a]Dwell Time, $t_n = \frac{t_1}{\sqrt{\Delta P_1}} \sqrt{\Delta P_n}$

Figure 6-1. CSR field data sheet.

RADIAN CORPORATION

PM 10 Run 3

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	DI H_2O	100	Mod G-S	Final 748.3 Initial 616.0 Wt. Gain 132.3
2	DI H_2O	100	Mod G-S	Final 683.1 Initial 625.3 Wt. Gain 57.8
3	MT	—	Mod G-S	Final 475.6 Inital ~~475.9~~ 476.47 Wt. Gain 2.8
4	Silica Gel	~ 200(g)	Mod G-S	Final 667.4 Initial 661.5 Wt. Gain 5.9
5				Final Initial Wt. Gain
6				Final Initial Wt. Gain
7				Final Initial Wt. Gain

Total Impingers Weight Gain (Wc), grams 199.3 Analyst _____

Filter 08 => 0.2143 g

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

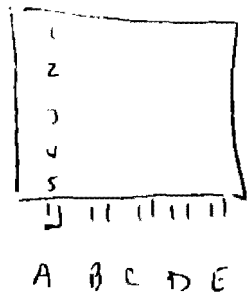
$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = $.0472(____ g)$ = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

APPENDIX A.3

ALDEHYDES

PLANT Mathy
DATE 9/19/91
SAMPLING LOCATION Stack
SAMPLE TYPE Aldehydes
RUN NUMBER 1
OPERATOR CFF
AMBIENT TEMPERATURE 50
BAROMETRIC PRESSURE
STATIC PRESSURE, (P_s)
FILTER NUMBER (s) NA



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 5' Glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 27
SAMPLE BOX NUMBER Aldehydes
METER BOX NUMBER N-32
METER $\Delta H_@$ 1.75
C_p FACTOR 0.61
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE Δp NA

K=.64 Y=1.0006

READ AND RECORD ALL DATA EVERY 2.5 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F (NO FILTER)	IMPINGER TEMPERATURE, °F
				DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0 / 906	62.714									
A1	0 / 908.5	64.4	2.2		1.53	245	59	56	2.5	270	46
A2	5 / 911	65.9	1.8		1.24	245	59	56		273	44
A3	7.5 / 913.5	67.2	1.1		0.76	245	62	56		268	43
A4	10 / 916	67.7	.66		.45	247	63	57		273	43
A5	12.5 / 918.5	68.595	.25		.17	246	64	58		270	44
		920	"								
B1	15 / 922.5	70.38	3.0		1.8	249	62	58		220	44
B2	17.5 / 925	72.03	2.4		1.46	247	66	59		220	43
B3	20 / 927.5	73.7	1.05		.63	248	66	59		230	44
B4	22.5 / 930	74.7	0.55		.33	246	67	60		230	45
B5	25 / 932.5	75.287	.25		.16	244	67	60		223	45
		1035	"								
C1	27.5 / 1037.5	77.2	2.8		1.8	225	58	58		240	42
2	30 / 1040	78.7	2.4		1.5	231	58	58		238	42
3	32.5 / 1042.5	80.1	1.3		.83	217	59	58		233	42
4	35 / 1045	80.8	.43		.27	211	60	59		234	43
5	37.5 / 1047.5	81.266	.16		.10	232	60	59		213	45
STOP		1049	"								
D1	40 / 1051.5	83.0	2.7		1.7	243	61	59		243	45
2	42.5 / 1054	84.6	2.4		1.5	245	63	59		229	44
3	45 / 1056.5	85.9	1.0		.64	246	66	60		235	48
4	47.5 / 1059	86.7	.53		.34	242	66	61		240	45
5	50 / 1101.5	87.238	.25		.16	233	66	61		242	45

COMMENTS

EPA (Draft 225

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)		GAS METER READING (V_m) ft³	VELOCITY HEAD (Δp_s) in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$) °F	OUTLET ($T_{m\,out}$), °F			
	50	1104	87.238									
E 1	52.5	1106.5	84.1	.24		1.7	245	61	67	3	232	46
2	55	1109	90.8	.22		1.4	247	62	68	3	218	45
3	57.5	1111.5	92.0	.13		.83	245	62	70	2	225	46
4	60	1114	93.0	.68		.44	244	63	70	1	230	46
5	62.5	1116.5	93.580	.28		.18	227	63	70	1	235	47
				~~.25~~								
			FINAL LEAK.		0.017		at 10" Hg					

RADIAN CORPORATION

Aldehydes Run 1

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)	
1	DNPH	200	MOD G-S	Final	801.2
				Initial	623.0
				Wt. Gain	178.2
2	DNPH	200	G-S	Final	706.5
				Initial	634.3
				Wt. Gain	72.2
3	MT	—	MOD G-S	Final	499.4
				Inital	493.0
				Wt. Gain	6.4
4	SilicaGel	~200 (g)	MODG-S	Final	673.6
				Initial	665.0
				Wt. Gain	8.6
5				Final	
				Initial	
				Wt. Gain	
6				Final	
				Initial	
				Wt. Gain	
7				Final	
				Initial	
				Wt. Gain	

Total Impingers Weight Gain (Wc), grams 265.4 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = .0472(____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

PLANT Mathy - LC
DATE 9-20-91
SAMPLING LOCATION Outlet
SAMPLE TYPE Aldehydes
RUN NUMBER 2
OPERATOR JB, J
AMBIENT TEMPERATURE 30
BAROMETRIC PRESSURE 29.7
STATIC PRESSURE, (P_g)
FILTER NUMBER (s)

Initial 0 on 2 or 3 kettles

PROBE LENGTH AND TYPE 6' glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 27.6
SAMPLE BOX NUMBER
METER BOX NUMBER N-32
METER $\Delta H_@$ 1.73
Y ~~Z~~ FACTOR 1.0006
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE Δp K = .62

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 1.5 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24-hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0	0745	98.052									
E 1	2.5	747.5	99.5	1.9		1.18	210	42	39	1	252	40
2	5	750	100.9	1.3		.91	218	49	40	1	268	39
3	7.5	752.5	102.1	.97		.68	220	45	40	1	252	38
4	10	755	103.0	.56		.40	221	46	41	1	283	40
5	12.5	757.5	103.507	.20		.14	228	47	41	1	280	40
		759	"									
D 1	15	801.5	104.9	2.8		1.7	236	47	42	2	242	40
2	17.5	804	106.8	2.8		1.7	239	51	43	2	241	41
3	20	806.5	107.9	1.3		.91	243	52	44	1	243	44
4	22.5	809	109.1	.74		.50	244	52	45	1	247	47
5	25	811.5	109.664	.30		.19	240	53	45	1	251	47
		813	"									
C 1	27.5	0815.5	111.6	3.0		1.9	250	53	46	1	275	46
2	30	818	113.0	2.2		1.3	252	54	51	1	268	47
3	32.5	820.5	114.3	1.2		.72	251	57	48	1	277	52
4	35	823	115.0	.44		.26	250	56	48	1	250	52
5	37.5	825.5	115.405	.16		.10	247	55	49	1	286	49
	39.5	938	115.772									
B 1	41	940.5	117.5	3.6		2.3	218	57	54	2.5	250	39
2	42.5	943	119	2.2		1.4	225	58	54	2	241	38
3	45	945.5	120.2	~~1.95~~		.72	231	59	54	2	234	40
4	47.5	948	121.15	.64		.39	237	61	55	2	245	40
	50	950.5	121.778	.28		.16	238	61	55	2	~~255~~ 257	42

Δp's .95 - 1.3

COMMENTS

235

EPA (Dec) 235

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM in Hg	SAMPLE BOX TEMPERATURE °F	IMPINGER TEMPERATURE °F
				DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0 / 0952	121,778 (start)									
A1	52.5 / 0954.5	123.3	2.5		1.45	246	61	56	2	243	37
A2	55 / 0957	124.8	2.25		1.3	249	63	56	2	244	40
A3	57.5 / 0959.5	125.95	1.15		.63	248	64	57	2	233	42
A4	60 / 1002	126.8	0.75		.43	249	65	58	2	246	43
A5	62.5 / 1004.5	127.400	0.25		.14	241	64	58	1.5	230	44
				CEM	- 0.4 =	.00					

RADIAN CORPORATION

Aldehydes Run 2

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	DNPH	200	mod G-S	Final 835.9 Initial ~~659.9~~ 666 Wt. Gain 173.3
2	DNPH	200	G-S	Final 719.9 Initial 652.1 Wt. Gain 67.8
3	MT	—	mod G-S	Final 499.9 Inital 487.4 Wt. Gain 12.5
4	Silicabel	200(g)	mod G-S	Final 650.6 Initial 639.7 Wt. Gain 10.9
5				Final ____ Initial ____ Wt. Gain ____
6				Final ____ Initial ____ Wt. Gain ____
7				Final ____ Initial ____ Wt. Gain ____

Total Impingers Weight Gain (Wc), grams ~~2,444.8~~ 264.5 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = .0472(W_c) = .0472(_____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

PLANT Mobay LC
DATE 9-20-91
SAMPLING LOCATION Control
SAMPLE TYPE Aldehydes
RUN NUMBER 3
OPERATOR JBP
AMBIENT TEMPERATURE 60
BAROMETRIC PRESSURE 29.7
STATIC PRESSURE (P_s)
FILTER NUMBER (s)
Initial 0004 at 11" Hg

PROBE LENGTH AND TYPE 6' glass
NOZZLE I.D. .191
ASSUMED MOISTURE, % 27%
SAMPLE BOX NUMBER
METER BOX NUMBER N-30
METER $\Delta H_@$ 1.84
γ FACTOR .9978
PROBE HEATER SETTING 250
HEATER BOX SETTING 250
REFERENCE Δp K=.65

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 25 MINUTES

TRAVERSE POINT NUMBER	SAMPLING TIME, min / CLOCK TIME (24 hr CLOCK)		GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	0	1337	99938									
E 1	2.5	1339.5	102.0	2.2		14	214	71	69	3	200	60
2	~~2~~	1341 ~~1342~~	102.653	2.0		13	219	71	69	3	214	58
~~3~~	~~7.5~~	STOP	PROCESS	SITUATION		PROBLEM						
~~4~~	~~10~~	~~1413~~										
~~5~~	~~12.5~~											
~~STOP~~	4	1413	102.653									
E 2	5	1414	103.1	1.4		.91	210	69	69	2	252	61
3	7.5	1416.5	104.5	1.2		.78	213	69	69	2	255	60
4	10	1419	105.5	.70		.46	216	69	69	1	250	59
5	12.5	1421.5	105.897	.15		.10	220	69	69	1	249	62
STOP		1422.5	"									
D 1	15	1425	107.7	2.7		18	229	70	69	3	241	61
2	17.5	1427.5	109.4	2.1		13	232	70	69	3	242	64
3	20	1430	110.6	1.0		.65	232	71	69	2	238	62
4	22.5	1432.5	111.5	.50		.33	232	72	70	1	239	61
5	25	1435	111.900	.17		.11	231	72	70	1	241	62
STOP		1443	"									
C 1	27.5	1445.5	113.7	2.7		18	236	72	70	3	246	69
2	30	1448	115.3	2.1		13	239	74	71	2	248	66
3	32.5	1450.5	116.5	.97		.62	240	74	71	1	247	61
4	35	1453	117.2	.40		.26	239	75	72	1	248	60
5	37.5	1455.5	117.638	.13		.08	234	75	72	1	258	63

COMMENTS:

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft³	VELOCITY HEAD (Δp_s), in H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in H_2O		STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM, in Hg	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
					DESIRED	ACTUAL		INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F			
	37.5	1457	117.638									
B 1	40	1459.5	119.5	3.0		2.0	240	75	72	4	264	61
2	42.5	1502	121.2	2.1		1.4	242	76	72	3	262	64
3	45	1504.5	122.4	1.2		.78	242	76	72	2	263	63
R 4	47.5	1507	123.4	.50		.33	241	77	73	1	262	66
1 5	50	1509.5	123.902	.25		.16	238	77	73	1	261	61
STOP		1511	"									
A 1	52.5	1513.5	125.8	2.5		1.7	243	76	73	3	259	60
2	55	1516	127.3	1.7		1.1	245	77	73	2	262	60
3	57.5	1518.5	128.5	1.0		.65	245	78	74	2	263	60
4	60	1521	129.4	.5		.33	244	78	74	1	263	61
5	62.5	1523.5	129.889	.22		.14	241	78	74	1	263	60
			FINAL LEAK 0.005 @ 0"									

RADIAN CORPORATION

Aldehydes Run 3

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	DNPH	200	mod 6-S	Final 872.2 Initial 684.9 Wt. Gain 187.3
2	DNPH	200	6-S	Final 691.8 Initial 655.7 Wt. Gain 36.1
3	MT	—	mod 6-S	Final 497.0 Inital 495.9 Wt. Gain 2.9
4	Silica Gel	~200 (g)	mod 6-S	Final 679.9 Initial 673.6 Wt. Gain 6.3
5				Final ____ Initial ____ Wt. Gain ____
6				Final ____ Initial ____ Wt. Gain ____
7				Final ____ Initial ____ Wt. Gain ____

Total Impingers Weight Gain (Wc), grams 232.6 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)$(DGMCF) = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = .0472(W_c) = .0472(_____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

APPENDIX A.4

PAH

RADIAN CORPORATION

MODIFIED METHOD 5
FIELD DATA

RUN 1
PAGE 2 OF 4

R
DIORIV

PLANT	Muddy LC	PROBE LENGTH AND TYPE		DUCT DIMENSIONS	
DATE	9-14-91	NOZZLE I.D. (in)		FILTER TYPE	
SAMPLING LOCATION	Outlet	METER BOX NUMBER		FILTER NUMBER	PAH 1
SAMPLE TYPE	~~Diorin~~ PAH	METER ΔH@		ASSUMED MOISTURE (%)	
RUN NUMBER	1	Yd		MOISTURE METHOD	
OPERATOR	JBP	K FACTOR		MOISTURE DATA	
AMBIENT TEMPERATURE	50	PROBE HEATER SETTING		O2/CO2 METHOD	
BAROMETRIC PRESSURE		HEATER BOX SETTING		O2	
STATIC PRESSURE (Ps)		XAD TRAP		CO2	
INITIAL LEAKCHECK		HEIGHT OF LOCATION (ft)		FINAL LEAKCHECK	

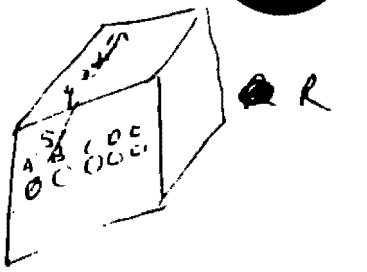
READ AND RECORD ALL DATA EVERY 5 MINUTES

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu.ft	Velocity Head (Δ Ps),	Flue Gas Temperature	Orifice Pressure Differential	Filter Temperature	Absorbent Trap	Temperature (°F) Dry Gas Meter Inlet	Temperature (°F) Dry Gas Meter Outlet	Temperature (°F) Impinger	Pump Vacuum
	75	831	45.768									
01	80	836	49.7	.27	245	1.9	273	43	58	56	46	7
2	85	841	53.0	.26	246	1.4	267	42	61	55	42	7
3	90	846	55.9	.16	246	.70	266	42	63	56	42	4
4	95	851	57.5	.42	244	1.47	272	43	62	58	43	3
5	100	856	58.584	.14	230	.11	278	43	64	59	43	2
stop		858										
E1	105	903	62.3	2.4	244	1.7	257	41	61	60	44	7
2	110	908	65.9	2.1	243	1.5	275	45	63	57	43	6
3	115	913	68.3	1.1	243	.71	264	47	65	59	46	4
4	120	918	70.3	.66	243	.42	279	47	66	61	46	3
5	125	923	71.120	.22	231	.15	265	47	65	61	46	3
			FINAL LEAK		.012 at 10"Hg							

MODIFIED METHOD 5
FIELD DATA

RUN 1
PAGE 1 OF
DUAL BOX
~~DIOXIN~~
PAH

PLANT	Nthg, LLC	PROBE LENGTH AND TYPE	6' Glass	DUCT DIMENSIONS	45" x
DATE	9-19-91	NOZZLE I.D. (in)	.191	FILTER TYPE	
SAMPLING LOCATION	Ol. Lk	METER BOX NUMBER	N-30	FILTER NUMBER	
SAMPLE TYPE	~~DIOXIN~~ PAH	METER Δ H@	1.84	ASSUMED MOISTURE (%)	20 %
RUN NUMBER	1	Yd	.9995	MOISTURE METHOD	
OPERATOR	TBP	K FACTOR	1.0	MOISTURE DATA	
AMBIENT TEMPERATURE	40	PROBE HEATER SETTING	250	O2/CO2 METHOD	
BAROMETRIC PRESSURE	29.7	HEATER BOX SETTING	250	O2	
STATIC PRESSURE (Pe)		XAD TRAP		CO2	
INITIAL LEAKCHECK	0.008 @ 17"	HEIGHT OF LOCATION (ft)	30'	FINAL LEAKCHECK	



READ AND RECORD ALL DATA EVERY 15 MINUTES

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu.ft	Velocity Head (Δ Pe),	Flue Gas Temperature	Orifice Pressure Differential	Filter Temperature	Absorbent Trap	Temperature (°F) Dry Gas Meter Inlet	Dry Gas Meter Outlet	Impinger	Pump Vacuum
	0	0710	07.532									
A1	5	715	8.7 11.1	.28	253	1.8	253	39	42	41	40	5
2	10	720	14.3	1.6	257	1.2	251	39	43	41	37	4
3	15	725	16	1.2	231	.79	244	40	44	42	38	3
4	20	730	18.6	.85	231	.56	244	41	47	43	39	2.5
5	25	735	19.814	.25	226	.16	245	41	48	44	40	2
STOP	27	736		PORT	CHANGE							
B1	36	741	23.0	2.9	236	1.9	276	41	50	45	38	4
2	35	746	26.3	2.5	233	1.6	260	42	54	46	39	6
3	46	751	28.9	1.3	234	.85	270	42	52	47	40	4
4	45	756	31.0	.67	235	.58	274	42	59	48	41	3.5
5	50	801	32.170	.24	226	.16	277	41	55	49	40	2
STOP		802										
C1	55	807	35.9	2.6	239	1.8	280	43	55	50	40	7
2	60	812	39.4	2.4	239	1.6	280	43	56	51	40	6
3	65	817	41.9	1.0	239	.69	282	41	58	52	40	4
4	70	822	44.1	.97	240	.68	285	41	58	53	40	4
5	75	827	45.768	.26	254	.25	278	41	59	54	42	2
STOP (Final)												

RADIAN CORPORATION

PAH Run 1

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	MT	—	MOD G-S	Final 854.4 Initial 478.0 Wt. Gain 376.4
2	HPLC H_2O	100	MOD G-S	Final 706.0 Initial 575.1 Wt. Gain 130.9
3	HPLC H_2O	100	MOD G-S	Final 591.8 Inital 591.7 Wt. Gain .1
4	MT	—	MOD G-S	Final 485.2 Initial 484.7 Wt. Gain .5
5	Silicagel	~200(g)	Mod G-S	Final 716.3 Initial 701.1 Wt. Gain 15.2
6				Final Initial Wt. Gain
7				Final Initial Wt. Gain

Total Impingers Weight Gain (Wc), grams _____ Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)$(DGMCF) = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = .0472(W_c) = .0472(_____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

KADIAN CORPORATION

MODIFIED METHOD 5
FIELD DATA

RUN 2
PAGE 1 OF

R
PAH
DUCT

PLANT	Marthy LC	PROBE LENGTH AND TYPE	6' glass	DUCT DIMENSIONS	48" x
DATE	7/14	NOZZLE I.D. (in)	.191	FILTER TYPE	
SAMPLING LOCATION	Outlet	METER BOX NUMBER	N-30	FILTER NUMBER	
SAMPLE TYPE	PAH	METER Δ H@	1.84	ASSUMED MOISTURE (%)	
RUN NUMBER	2	Yd	9998	MOISTURE METHOD	
OPERATOR	JBP	K FACTOR	.65	MOISTURE DATA	
AMBIENT TEMPERATURE	50	PROBE HEATER SETTING	250	O2/CO2 METHOD	
BAROMETRIC PRESSURE	29.7	HEATER BOX SETTING	250	O2	
STATIC PRESSURE (Pa)		XAD TRAP		CO2	
INITIAL LEAKCHECK	.008@10"	HEIGHT OF LOCATION (ft)	30'	FINAL LEAKCHECK	

READ AND RECORD ALL DATA EVERY 5 MINUTES

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu ft	Velocity Head (Δ Ps),	Flue Gas Temperature	Orifice Pressure Differential	Filter Temperature	Absorbent Trap	Temperature (°F) Dry Gas Meter Inlet	Temperature (°F) Dry Gas Meter Outlet	Temperature (°F) Impinger	Pump Vacuum
	0	1329	71.663		225							
A1	5	1333	74.9	2.7	264	1.9	264	45	56	54	41	5
2	10	1338	78.4	2.2	211	1.4	265	46	56	55	41	5
3	15	1343	80.9	1.1	222	.70	271	45	58	56	43	3
4	20	1348	82.9	.63	229	.45	275	45	60	57	43	2
5	25	1353	83.848	.25	228	.16	275	44	61	58	43	1
		1354	"									
B1	30	1359	87.5	3.2	239	2.1	272	42	62	58	46	6
2	35	1404	91.3	2.3	242	1.5	263	48	63	59	43	6
3	40	1409	93.9	1.1	242	.70	273	50	65	60	46	6
4	45	1414	95.5	.36	241	.25	255	48	66	61	48	3
5	50	1419	96.553	.17	232	.11		48	66	62	48	2
STOP		1421	"									
C1	55	1426	100.4	2.7	244	1.8	279	47	66	63	49	6
2	60	1431	103.7	1.8	244	1.2	274	50	67	63	47	5
3	65	1436	106.2	1.0	244	.65	267	47	69	64	47	3
4	70	1441	107.6	.33	242	.22	269	46	70	65	47	2
5	75	1446	108.317	.10	230	.07	267	47	69	66	47	1

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu ft	Velocity Head (Δ Pe), in. H2O	Flue Gas Temperature (°F)	Orifice Pressure Differential (Δ H, in H2O)	Filter Temperature (°F)	Absorbent Trap Inlet	Temperature (°F) Dry Gas Meter Inlet (Tm in)	Dry Gas Meter Outlet (Tm out)	Impinger Exit	Pump Vacuum (in Hg)
	75	1447	108.317									
D1	80	1452	111.9	2.1	244	1.8	275	46	66	69	42	7
2	85	1457	115.8	2.2	243	1.4	269	47	66	70	43	7
3	90	1502	118.3	1.1	242	.70	246	47	67	71	45	4
4	95	1508	120.0	.50	240	.33	268	46	67	71	46	3
5	100	1512	121.182	.17	229	.12	275	46	68	71	47	2
STOP		1515	"									
#1	105	1520	124.5	2.3	242	1.5	274	47	68	70	44	6
2	110	1525	127.7	2.1	243	1.3	257	44	68	71	46	5
3	115	1530	130.3	1.2	243	.80	263	46	68	72	45	5
4	120	1535	132.2	.67	239	.44	261	47	69	72	45	3
5	125	1540	133.271	.14	221	.09	263	46	69	72	46	1
			FINAL	LEAK		0	0.12 qt	11" Hg				

RADIAN CORPORATION

PAH's Run 2

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)
1	MT	—	MOD G-S	Final 877.2 Initial 478.6 Wt. Gain 398.6
2	HPLC H_2O	100	MOD G-S	Final 712.9 Initial 605.3 Wt. Gain 107.6
3	HPLC H_2O	100	MOD G-S	Final 611.8 Inital 612.1 Wt. Gain ~~-.3~~ -0.
4	MT	—	MOD G-S	Final 485.6 Initial 485.1 Wt. Gain .5
5	Silica Gel	~200(g)	MOD G-S	Final 728.5 Initial 716.1 Wt. Gain 12.4
6				Final Initial Wt. Gain 518.?
7				Final Initial Wt. Gain

Total Impingers Weight Gain (Wc), grams 518.8 Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = $.0472(W_c)$ = $.0472(____ g)$ = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

MODIFIED METHOD 5
FIELD DATA

RUN 3
PAGE 1 OF
FAH
DUAL

R

PLANT	MASON 6	PROBE LENGTH AND TYPE	6' glass	DUCT DIMENSIONS	48" x 42"
DATE	9/20/91	NOZZLE I D (in)	.191	FILTER TYPE	
SAMPLING LOCATION	STACK	METER BOX NUMBER	#30	FILTER NUMBER	
SAMPLE TYPE	PM4-5	METER Δ H@	1.54	ASSUMED MOISTURE (%)	
RUN NUMBER	3	Yd	.9998	MOISTURE METHOD	
OPERATOR	JBP	K FACTOR	.0.65	MOISTURE DATA	
AMBIENT TEMPERATURE	50	PROBE HEATER SETTING	250	O2/CO2 METHOD	
BAROMETRIC PRESSURE	29.7	HEATER BOX SETTING	250	O2	
STATIC PRESSURE (Pg)		XAD TRAP		CO2	
INITIAL LEAKCHECK	.016@10"	HEIGHT OF LOCATION (ft)		FINAL LEAKCHECK	

READ AND RECORD ALL DATA EVERY 5 MINUTES

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu.ft	Velocity Head (Δ Pg),	Flue Gas Temperature	Orifice Pressure Differential	Filter Temperature	Absorbent Trap	Temperature (°F) Dry Gas Meter Inlet	Dry Gas Meter Outlet	Impinger	Pump Vacuum
	0	0946	340.358									
E 1	5	0951	43.9	.25	235	1.9	260	39	49	50	46	6
2	10	0956	46.9	2.0	246	1.3	260	45	52	50	39	6
3	15	1001	49.5	1.1	244	.77	263	45	55	51	42	4
4	20	1006	51.4	.67	243	.42	261	44	56	52	43	4
5	25	1011	52.900	.24	240	.15	260	46	57	53	44	3
STOP		1013	"									
D 1	30	1018	55.9	2.7	247	1.9	262	47	58	54	42	8
2	35	1023	59.5	2.2	248	1.4	263	49	60	55	42	7
3	40	1028	61.7	1.0	247	.65	260	50	62	56	43	5
4	45	1033	63.7	.51	245	.33	259	45	63	57	43	4
5	50	1038	64.294	.10	238	.07	260	45	63	58	44	2
STOP		1042	"									
C 1	55	1047	67.3	.28	245	.20	260	46	62	59	42	8
2	66	1052	70.5	.22	243	.14	260	50	62	60	42	7
3	65	1057	74.0	1.0	243	.30	260	48	67	62	45	5
4	70	1102	75.6	.46	243	.30	254	49	68	63	47	4
5	75	1107	76.541	.16	236	.10	256	49	69	64	47	2

KAPPAN CORPORATION

METHOD 5
FIELD DATA

RUN 3
PAGE 1 OF 2

R

PLANT	Murphy LC
DATE	9.20.91
SAMPLING LOCATION	
SAMPLE TYPE	PAH
RUN NUMBER	3
OPERATOR	JBP

Traverse Point Number	Sampling Time (min)	Clock Time (24-hr)	Gas Meter Reading (Vm), cu.ft	Velocity Head (Δ Pe), in. H2O	Flue Gas Temperature (°F)	Orifice Pressure Differential (Δ H, in. H2O)	Filter Temperature (°F)	Temperature (°F)			Pump Vacuum (in. Hg)	XAD
								Dry Gas Meter				
								Inlet (Tm in)	Outlet (Tm out)	Impinger Exit		
	75	1111	76.541									
B5	80	1116	79.3	1.5	~~+0.24~~	1.0	271	75	67	45	~~4~~	48
4	85	1121	82.0	1.3	250	.85	266	76	66	44	6	49
3	90	1126	84.2	.98	246	.58	260	71	67	46	5	49
2	95	1131	86.1	.43	249	.40	265	72	68	47	4	49
1	100	1136	87.513	.40	235	.25	262	72	68	48	4	50
STOP		1138	"									
A1	105	1143	90.5	.26	254	.17	267	72	69	46	8	50
2	110	1148	94.0	.20	253	1.3	266	74	70	48	6	51
3	115	1153	96.4	1.0	256	.65	264	76	71	52	5	52
4	120	1158	98.2	.05	253	.32	263	76	71	52	4	52
5	125	1203	98.986	.02	243	.13	264	76	72	51	3	52
			Final	0.006 at 1.1"H								

COMMENTS:

Revision: 11/90

RADIAN CORPORATION

PAH Run 3

MOISTURE AND IMPINGER CATCH DATA SHEET

Impinger Number	Solution	Solution (mL)	Configuration	Weight (g)	
1	MT	—	Mod G-S	Final	876.7
				Initial	478.0
				Wt. Gain	398.7
2	HPLC H_2O	~200	Mod G-S	Final	712.2
				Initial	637.4
				Wt. Gain	74.8
3	HPLC H_2O	~200	Mod G-S	Final	624.6
				Inital	624.6
				Wt. Gain	0
4	MT	—	Mod G-S	Final	485.1
				Initial	484.8
				Wt. Gain	.3
5	Silica Gel	~200 (g)	Mod G-S	Final	740.6
				Initial	727.9
				Wt. Gain	12.7
6				Final	
				Initial	
				Wt. Gain	
7				Final	
				Initial	
				Wt. Gain	

Total Impingers Weight Gain (Wc), grams _____ Analyst _____

V_f = Final Meter Volume = _____ ft^3

V_i = Initial Meter Volume = _____ ft^3

DFMCF = Dry Gas Meter Correction Factor = _____

V_m = Metered Gas Volume = $(V_f - V_i)(DGMCF)$ = _____ ft^3

T_m = Average Meter Temperature = °F + 460 = _____ °R

P_m = Meter Pressure (Barometric Pressure) = _____ in.Hg.

$$V_{m(std)} = \frac{(17.64)(V_m)(P_m)}{T_m} = \frac{(17.64)(____ ft^3)(____ in.Hg.)}{(____ °R)} = ____ ft^3$$

$V_{w(std)}$ = Volume of Water Vapor = .0472(W_c) = .0472(_____g) = _____ ft^3

$$B_{ws} = \text{Moisture Fraction} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}} = \frac{____ ft^3}{____ ft^3 + ____ ft^3} = ____$$

APPENDIX B

PROCESS DATA SHEETS

Form REC#04 # Reading x 100 equals A.C. Volts NT - Not Taken

CSCO - Crippled CEM online

John Jorgensen - moisture analysis
#1 - 3.8
#2 - 4.3
#3 - 4.2

9/19/91 Mathy Plant # 6 Day 1 LR

1 Time (24 Hr) NOTE: check mail box on column when moisture sample is here	2 Fuel Use ✓Fuel Oil / Nat. Gas ✓ / Propane / Coal / other 100 (#2)	3 Burner Setting Flame (%)	Flame eye (microamps)	4 Kiln Exit ~~Bleed~~ Pressure / Temp (F) [340S]	5 Production Rate x 5 tph ✓Mix / Aggregate TPH (2155)	~~RAP~~ TPH	6 Asphalt Cement % 5.8%	7 Mix Temp °F (Kiln Exit Temp [340S])	8 ~~Bleed~~ Exhaust Gas Temp °F Stack	9 ✓Venturi Scrubber / Baghouse Pressure Drop in w.g.	~~Water~~ BH Pressure Inlet/out Temp. (F)	10 Ambient Temp °F	11 Relative Humidity %	12 [.275 to .375] Exhaust Damper Position %	Pressure (in-H2O)
:29	Start of warm up														
6:43	57670				0										
:55	~~57684~~ Start Production	69	1.9	303	0	0	5.8	—	325	3.1	240/20[illegible]	40.0	40/10	40	.35
7:12	Start of ~~rock~~														
7:25	57709	80	1.9	315	22	0	5.8	305	315	1.9	260/230	40.8	NT	42	.36
7:48	57726	79	1.9	338	39	0	5.8	—	310	2.0	260/230	~~42.6~~	NT	42	.33
8:19	57752	79	1.9	338	64	0	5.8	—	312	1.9	260/240	42.6	NT	42	.32
8:40	57769	80	1.8	338	78	0	5.8	—	315	1.8	265/245	42.8	NT	42	.34
8:58	57786	76	1.9	341	94	0	5.8	295	310	2.0	265/243	43.6	42/44/85	42	.34
9:34	57817	76	1.9	341	122	0	5.8	—	310	1.8	265/242	45.8		42	.36
09:37	~~Stopped~~ Started plant			Took	aggregate for	moisture	@	9:45							
10:27	57934	63	1.9	340	123	0	5.8	~~285~~	285	3.0	260/195	52.0	NT	43	.42
11:00	57855	63	1.9	340	149	0	5.8	—	310	1.2	260/240	~~52~~	~~45/50~~	43	.38
11:20	57873	81	1.9	338	163	0	5.8	301	315	1.0	265/244	50.6	NT	43	.40
11:46	57895	79	1.9	342	184	0	5.8	—	310	1.8	264/245	50.0	44/51/57	43	.43
12:01	57907	79	2.0	358	198	0	5.8	—	312	1.6	269/244	51.2	—	42	.41
	Stop														

Form REC#04

14:00 10 burner pressure 0 sig ounces
11.5 gas pressure psig

1/14/91 1 rev = 100 ft³

PAGE 2 OF 2

Day 1 2.72

1 Time (24 HR) NOTE: check small box in column when moisture sample is taken	2 Fuel Use: #Fuel Oil, Nat. Gas ✓, Propane, Coal, other	3 Burner Setting		4 Kiln Blower-Pressure Exit Temp.	5 Production Rate: Mix ✓, Aggregate; TPH	5 Production Rate: RAP; TPH	6 Asphalt Cement %	7 Mix Temp. °F	8 Exhaust Gas Temp. °F Stack	9 Venturi Scrubber ✓ Baghouse: Pressure Drop in w.g.	9 Waste-BH Pressure gage: Temp. In	9 Waste-BH Pressure gage: Temp. Out	10 Ambient Temp °F	11 Relative Humidity %	12 Exhaust Damper Position: in H₂O	12 Exhaust Damper Position: %
13:24	Start 57913															
13:30	Start of Test	92	1.9	317	200	0	5.8	~~312~~	312	2.5	250	165	51.0	44/52	42	.50
14:00	57939	95	1.8	343	221	0	5.8	—	315	1.8	262	242	51.0	—	42	.45
14:30	57969	79	1.9	340	248	0	5.8	—	313	1.9	265	243	52.3	45/54	41	.39
15:00	57995	80	1.9	337	276	0	5.8	305	312	1.7	262	241	51.0	—	41	.40
15:30	—	74	1.9	344	296	0	5.8	—	310	1.7	264	242			41	.45
15:44	—	79	1.9	341	305	0	5.8	—	310	1.8	264	242	51.4	—	41	.32
15:46	58030				306	Final								44/50		

REC#04

PAGE 1 OF 2

10/91 Mathy #6 (Day 2)

	2 Fuel Use (#Fuel Oil, Nat. Gas, Propane, Coal, other)	3 Burner Setting: Flame reg.	3 Burner Setting: Fuel Rate	4 Kiln Blower Pressure / Exit Temp.	5 Production Rate x 5 tph: Mix / Aggregate TPH	5 Production Rate x 5 tph: Pug / TPH	6 Asphalt Cement %	7 Mix Temp. °F	8 Exhaust Gas Temp. °F Stack	9 Vertical Scrubber / Baghouse: Pressure Drop in w.g.	9 Vertical Scrubber / Baghouse: Pressure BH Temp Inlet/outlet	10 Ambient Temp °F	11 Relative Humidity %	12 Exhaust Damper Position
1	Start of warm up										83/35	36.0	33/35 46	
	58030				0									
6	58037 Started production	83	1.9	358	0	0	5.8	—	310	0.9	250 155	36.0	—	42 5 plugged
5	Start of aldehydes													
55	58065	83	1.9	339	12	0	5.8	300	318	1.0	262 229	36.8	—	42 .33
00	58056	83	1.9	358	18	0	5.8	325		1.1	269 242	37.2	—	42 .3
15	58073	83	1.9	351	30	0	5.8	—	325	1.7	274 249	37.8	—	42 .33
30	58094	82	1.9	350	45	0	5.8	—	320	1.7	272 249	41.6	38/40 84	42 .33
47	58103 Shut down				53									
30	Restart plant													
38	58111	82	1.9	338	56	0	5.8	—	320	1.7	260 221	45.2	41/45	40 .28
00	58133	83	1.8	348	77	0	5.8	—	325	1.9	269 249	51.2	—	40 .35
30	58160	83	1.8	349	99	0	5.8	—	315	2.2	269 249	52.8	—	40 .38
07	58191	84	1.9	350	126	0	5.8	—	315	2.0	265 249		—	42 .35
36	58216	82	1.9	348	148	0	5.8	—	320	2.4	270 250	55.4	—	43 .32
05	58242	78	1.9	348	170	0	5.8	302	315	3.0	269 250	51.4	47/52	44 .38
39	58272	78	1.9	352	194	0	5.8	—	315	2.9	269 252		—	42 .38
59	Stop				207									

SEP 14 1991 NJDEP

C#04

1/22/91 Day 2

PAGE 2 OF 2

2 Fuel Use (#Fuel Oil / Nat. Gas ✓ / Propane / Coal / other)	3 Burner Setting		4 Kiln Blower Pressure Exit Temp	5 Production Rate ✓Mix / Aggregate TPH	5 Production Rate RAP TPH	6 Asphalt Content %	7 Mix Temp. °F	8 Exhaust Gas Temp. °F Stack	9 ✓Venturi Scrubber / Baghouse Pressure Drop in wg.	9 WEBB Pressure gal. In	9 Bit Temp Out	10 Ambient Temp °F	11 Relative Humidity %	12 Exhaust Damper Position ?	
Restart				● 209											
58294 Stop				210											
~~Restart~~ ~~58294~~				~~219~~											
58300	79	1.8	347	220	0	5.8½	—	305	2.3	254	224	56	—	40	0.35
58316	71	1.8	350	237	0	5.8½	—	310	2.9	252	230	58	50 61	41	0.48
58340	78	1.8	348	25●1	0	5.8	300	310	2.2	252	235	59	—	41	0.40
58368	75	1.9	350	276	0	5.8	—	310	1.8	258	274	61	—	41	0.42
End of Run	Test														

NAPA TEL NO. 3017314621 Sep 17,91 6:14 P.02

APPENDIX C

SAMPLE PARAMETER CALCULATION SHEETS

C.1 PM/Metals
C.2 PM_{10}/CPM
C.3 Aldehydes
C.4 PAH

APPENDIX C.1

PM/METALS

FACILITY : Mathy #6

DATE:	9/20/91
LOCATION:	Outlet
RUN NUMBER:	3.00

SAMPLING PARAMETER	TM
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	245.08
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	58.38
Average Meter Pressure (in.H2O)	0.86
Average Meter Temperature (deg. F)	65.66
Moisture Collected (g)	506.70
Carbon Dioxide Concentration (%V)	5.40
Oxygen Concentration (%V)	14.27
Nitrogen Concentration (%V)	80.33
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84
Particulate Catch (g)	0.01550
Average Sampling Rate (dscfm)	0.46
Standard Metered Volume,Vm(std) (dscf)	57.57
Standard Metered Volume,Vm(std) (dscm)	1.631
Standard Volume Water Vapor,Vw (scf)	23.89
Standard Volume Water Vapor,Vw (scm)	0.677
Stack Moisture (%V)	29.33
Mole Fraction Dry Stack Gas	0.707
Dry Molecular Weight	29.43
Wet Molecular Weight	26.08
Stack Gas Velocity,Vs (fpm)	4222.90
Stack Gas Velocity,Vs (mpm)	1287.14
Volumetric Flow Rate (acfm)	59120.55
Volumetric Flow Rate (acmm)	1674.294
Volumetric Flow Rate (dscfm)	31085.51
Volumetric Flow Rate (dscmm)	880.342
Percent Isokinetic	104.21
Percent Excess Air	205.23
Fuel Factor,Fo	1.228
Ultimate CO2	17.02
Concentration of Particulate (grains/acf)	0.00218
Concentration of Particulate (g/acm)	0.00500
Concentration of Particulate (grains/dscf)	0.00415
Concentration of Particulate (g/dscm)	0.00951
Concentration of Particulate (grains/dscf @12% CO2)	0.00923

FACILITY : Mathy #6

DATE: 9/19/91	
LOCATION: STACK	
RUN NUMBER: 1.00	
SAMPLING PARAMETER TM	
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	240.60
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	62.44
Average Meter Pressure (in.H2O)	0.96
Average Meter Temperature (deg. F)	63.28
Moisture Collected (g)	553.50
Carbon Dioxide Concentration (%V)	5.62
Oxygen Concentration (%V)	14.15
Nitrogen Concentration (%V)	80.23
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84
Particulate Catch (g)	0.02360
Average Sampling Rate (dscfm)	0.49
Standard Metered Volume,Vm(std) (dscf)	61.87
Standard Metered Volume,Vm(std) (dscm)	1.752
Standard Volume Water Vapor,Vw (scf)	26.10
Standard Volume Water Vapor,Vw (scm)	0.739
Stack Moisture (%V)	29.67
Mole Fraction Dry Stack Gas	0.703
Dry Molecular Weight	29.47
Wet Molecular Weight	26.06
Stack Gas Velocity,Vs (fpm)	4540.05
Stack Gas Velocity,Vs (mpm)	1383.81
Volumetric Flow Rate (acfm)	63560.69
Volumetric Flow Rate (acmm)	1800.039
Volumetric Flow Rate (dscfm)	33472.05
Volumetric Flow Rate (dscmm)	947.928
Percent Isokinetic	104.01
Percent Excess Air	200.80
Fuel Factor,Fo	1.201
Ultimate CO2	17.40
Concentration of Particulate (grains/acf)	0.00310
Concentration of Particulate (g/acm)	0.00709
Concentration of Particulate (grains/dscf)	0.00589
Concentration of Particulate (g/dscm)	0.01347
Concentration of Particulate (grains/dscf @12% CO2)	0.01257

FACILITY : Mathy #6

DATE: 9/19/91	
LOCATION: Outlet	
RUN NUMBER: 2.00	
SAMPLING PARAMETER TM	
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H20)	0.80
Average Stack Temperature (deg. F)	236.04
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	60.08
Average Meter Pressure (in.H20)	0.91
Average Meter Temperature (deg. F)	65.96
Moisture Collected (g)	920.00
Carbon dioxide Concentration (%V)	5.70
Oxygen Concentration (%V)	12.53
Nitrogen Concentration (%V)	81.69
Dry Gas Meter Factor	0.98750
Pitot Constant	0.84
Particulate Catch (g)	0.01320
Average Sampling Rate (dscfm)	0.47
Standard Metered Volume,Vm(std) (dscf)	59.22
Standard Metered Volume,Vm(std) (dscm)	1.677
Standard Volume Water Vapor,Vw (scf)	24.52
Standard Volume Water Vapor,Vw (scm)	0.694
Stack Moisture (%V)	29.28
Mole Fraction Dry Stack Gas	0.707
Dry Molecular Weight	29.43
Wet Molecular Weight	26.08
Stack Gas Velocity,Vs (fpm)	4286.10
Stack Gas Velocity,Vs (mpm)	1306.40
Volumetric Flow Rate (acfm)	60005.44
Volumetric Flow Rate (acmm)	1699.354
Volumetric Flow Rate (dscfm)	31981.67
Volumetric Flow Rate (dscmm)	905.721
Percent Isokinetic	104.18
Percent Excess Air	138.41
Fuel Factor,Fo	1.448
Ultimate CO2	14.43
Concentration of Particulate (grains/scf)	0.00183
Concentration of Particulate (g/scm)	0.00419
Concentration of Particulate (grains/dscf)	0.00344
Concentration of Particulate (g/dscm)	0.00787
Concentration of Particulate (grains/dscf @12% CO2)	0.00714

APPENDIX C.2

PM_{10}/CPM

SAMPLE PARAMETERS FOR PM10/CPM RUNS

MATHY CONSTRUCTION COMPANY - PLANT 6 (1991)

Parameter	Run 1	Run 2	Run 3	Average
Stack Conditions				
Corrected Barometric Pressure (in Hg)	29.68	29.68	29.68	29.68
Stack Static Pressure (in. H2O)	0.80	0.80	0.80	0.80
Average Stack Temperature (deg. F)	241.42	254.95	241.84	246.07
Carbon Dioxide Concentration (%V)	5.10	5.40	5.62	5.37
Oxygen Concentration (%V)	14.12	14.24	13.61	13.99
Nitrogen Concentration (%V)	80.78	80.36	80.77	80.64
Stack Moisture (%V)	28.53	28.52	27.63	28.22
Stack Gas Velocity,Vs (fps)	74.6*	72.9*	70.2*	72.59
Volumetric Flow Rate (acfm)	62705*	61261*	58967*	60,978
Volumetric Flow Rate (dscfm)	33530*	32656*	32105*	32,764
Stack viscosity (micropoise)	198.52	202.01	199.01	199.85
Sampling Parameters				
Total sampling time (min)	70.82	64.07	61.00	NA
Average Meter Temperature (deg. F)	65.18	69.68	72.60	69.15
Average Meter Pressure (in.H2O)	0.56	0.56	0.56	0.56
Average Sampling Rate (dscfm)	0.41	0.41	0.40	0.41
Average Sampling Rate (acfm - cyclone cond)	0.78	0.79	0.74	0.77
Standard Metered Volume,Vm(std) (dscf)	29.36	26.56	24.55	26.82
Percent Isokinetic	95.8	100	97.7	NA
Particulate Emissions < Cut Size (lbs/hr)	2.04	1.32	1.28	1.55
Particulate Emissions > Cut Size (lbs/hr)	1.68	1.66	1.28	1.54
Particulate Emissions Total (lbs/hr)	3.72	2.98	2.56	3.08

* Value taken from the average of all other trains as the PM10 flow rates (Site 6) were suspect (20 - 50 higher).

NA = Not Applicable

APPENDIX C.3

ALDEHYDES

FACILITY : Mathy #6

DATE: 9/20/91
LOCATION: STACK
RUN NUMBER:1

SAMPLING PARAMETER ALDEHYDES	
Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	239.80
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	30.87
Average Meter Pressure (in.H2O)	0.91
Average Meter Temperature (deg. F)	61.54
Moisture Collected (g)	265.40
Carbon Dioxide Concentration (%V)	6.17
Oxygen Concentration (%V)	12.85
Nitrogen Concentration (%V)	80.98
Dry Gas Meter Factor	1.00060
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.50
Standard Metered Volume,Vm(std) (dscf)	31.09
Standard Metered Volume,Vm(std) (dscm)	0.880
Standard Volume Water Vapor,Vw (scf)	12.51
Standard Volume Water Vapor,Vw (scm)	0.354
Stack Moisture (%V)	28.70
Mole Fraction Dry Stack Gas	0.713
Dry Molecular Weight	29.50
Wet Molecular Weight	26.20
Stack Gas Velocity,Vs (fpm)	4435.20
Stack Gas Velocity,Vs (mpm)	1351.85
Volumetric Flow Rate (acfm)	62092.73
Volumetric Flow Rate (acmm)	1758.466
Volumetric Flow Rate (dscfm)	33187.04
Volumetric Flow Rate (dscmm)	939.857
Percent Isokinetic	105.42
Percent Excess Air	150.38
Fuel Factor,Fo	1.305
Ultimate CO2	16.02

FACILITY : Mathy #6

DATE: 9-20-91
LOCATION: STACK
RUN NUMBER: 2.00

SAMPLING PARAMETER	ALDEHYDE
Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	237.52
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	28.98
Average Meter Pressure (in.H2O)	0.86
Average Meter Temperature (deg. F)	51.74
Moisture Collected (g)	264.50
Carbon Dioxide Concentration (%V)	6.63
Oxygen Concentration (%V)	12.36
Nitrogen Concentration (%V)	81.01
Dry Gas Meter Factor	1.00060
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.48
Standard Metered Volume,Vm(std) (dscf)	29.75
Standard Metered Volume,Vm(std) (dscm)	0.843
Standard Volume Water Vapor,Vw (scf)	12.47
Standard Volume Water Vapor,Vw (scm)	0.353
Stack Moisture (%V)	29.54
Mole Fraction Dry Stack Gas	0.705
Dry Molecular Weight	29.56
Wet Molecular Weight	26.14
Stack Gas Velocity,Vs (fpm)	4446.63
Stack Gas Velocity,Vs (mpm)	1355.33
Volumetric Flow Rate (acfm)	62252.76
Volumetric Flow Rate (acmm)	1762.998
Volumetric Flow Rate (dscfm)	32987.84
Volumetric Flow Rate (dscmm)	934.216
Percent Isokinetic	101.48
Percent Excess Air	136.68
Fuel Factor,Fo	1.288
Ultimate CO2	16.23

FACILITY : Mathy #6

DATE: 9-20-91
LOCATION: STACK
RUN NUMBER: 3.00

SAMPLING PARAMETER	ALDEHYDE
Total Sampling Time (min.)	62.50
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	233.74
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	29.95
Average Meter Pressure (in.H2O)	0.83
Average Meter Temperature (deg. F)	72.76
Moisture Collected (g)	232.60
Carbon Dioxide Concentration (%V)	5.63
Oxygen Concentration (%V)	13.43
Nitrogen Concentration (%V)	80.94
Dry Gas Meter Factor	0.99980
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.47
Standard Metered Volume,Vm(std) (dscf)	29.50
Standard Metered Volume,Vm(std) (dscm)	0.836
Standard Volume Water Vapor,Vw (scf)	10.97
Standard Volume Water Vapor,Vw (scm)	0.311
Stack Moisture (%V)	27.10
Mole Fraction Dry Stack Gas	0.729
Dry Molecular Weight	29.44
Wet Molecular Weight	26.34
Stack Gas Velocity,Vs (fpm)	4208.00
Stack Gas Velocity,Vs (mpm)	1282.60
Volumetric Flow Rate (acfm)	58912.05
Volumetric Flow Rate (acmm)	1668.389
Volumetric Flow Rate (dscfm)	32474.95
Volumetric Flow Rate (dscmm)	919.691
Percent Isokinetic	102.23
Percent Excess Air	168.84
Fuel Factor,Fo	1.327
Ultimate CO2	15.75

APPENDIX C.4

PAH

FACILITY : Mathy #6

DATE:	9-19-91
LOCATION:	STACK
RUN NUMBER:	1.00

SAMPLING PARAMETER	PAH
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	239.68
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	63.59
Average Meter Pressure (in.H2O)	0.96
Average Meter Temperature (deg. F)	53.86
Moisture Collected (g)	523.10
Carbon Dioxide Concentration (%V)	5.62
Oxygen Concentration (%V)	14.15
Nitrogen Concentration (%V)	80.23
Dry Gas Meter Factor	0.99980
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.52
Standard Metered Volume,Vm(std) (dscf)	64.96
Standard Metered Volume,Vm(std) (dscm)	1.840
Standard Volume Water Vapor,Vw (scf)	24.66
Standard Volume Water Vapor,Vw (scm)	0.698
Stack Moisture (%V)	27.52
Mole Fraction Dry Stack Gas	0.725
Dry Molecular Weight	29.47
Wet Molecular Weight	26.31
Stack Gas Velocity,Vs (fpm)	4505.27
Stack Gas Velocity,Vs (mpm)	1373.21
Volumetric Flow Rate (acfm)	63073.77
Volumetric Flow Rate (acmm)	1786.249
Volumetric Flow Rate (dscfm)	34274.71
Volumetric Flow Rate (dscmm)	970.660
Percent Isokinetic	106.64
Percent Excess Air	200.80
Fuel Factor,Fo	1.201
Ultimate CO2	17.40

FACILITY : Mathy #6

DATE: 9-19-91	
LOCATION: STACK	
RUN NUMBER: 2.00	
SAMPLING PARAMETER	PAH
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	236.04
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	61.61
Average Meter Pressure (in.H2O)	0.86
Average Meter Temperature (deg. F)	64.84
Moisture Collected (g)	518.80
Carbon Dioxide Concentration (%V)	5.78
Oxygen Concentration (%V)	12.53
Nitrogen Concentration (%V)	81.69
Dry Gas Meter Factor	0.99980
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.49
Standard Metered Volume,Vm(std) (dscf)	61.61
Standard Metered Volume,Vm(std) (dscm)	1.745
Standard Volume Water Vapor,Vw (scf)	24.46
Standard Volume Water Vapor,Vw (scm)	0.693
Stack Moisture (%V)	28.42
Mole Fraction Dry Stack Gas	0.716
Dry Molecular Weight	29.43
Wet Molecular Weight	26.18
Stack Gas Velocity,Vs (fpm)	4323.73
Stack Gas Velocity,Vs (mpm)	1317.87
Volumetric Flow Rate (acfm)	60532.27
Volumetric Flow Rate (acmm)	1714.274
Volumetric Flow Rate (dscfm)	32654.51
Volumetric Flow Rate (dscmm)	924.776
Percent Isokinetic	106.15
Percent Excess Air	138.41
Fuel Factor,Fo	1.448
Ultimate CO2	14.43

FACILITY : Mathy #6

DATE:	9-20-91
LOCATION:	STACK
RUN NUMBER:	3.00

SAMPLING PARAMETER	PAH
Total Sampling Time (min.)	125.00
Corrected Barometric Pressure (in. Hg)	29.68
Absolute Stack Pressure,Ps(in. Hg)	29.74
Stack Static Pressure (in. H2O)	0.80
Average Stack Temperature (deg. F)	244.28
Stack Area (sq.in.)	2016.00
Metered Volume,Vm (cu.ft.)	58.63
Average Meter Pressure (in.H2O)	0.82
Average Meter Temperature (deg. F)	63.40
Moisture Collected (g)	486.50
Carbon Dioxide Concentration (%V)	5.40
Oxygen Concentration (%V)	14.27
Nitrogen Concentration (%V)	80.33
Dry Gas Meter Factor	0.99980
Pitot Constant	0.84
Average Sampling Rate (dscfm)	0.47
Standard Metered Volume,Vm(std) (dscf)	58.79
Standard Metered Volume,Vm(std) (dscm)	1.665
Standard Volume Water Vapor,Vw (scf)	22.94
Standard Volume Water Vapor,Vw (scm)	0.650
Stack Moisture (%V)	28.07
Mole Fraction Dry Stack Gas	0.719
Dry Molecular Weight	29.43
Wet Molecular Weight	26.23
Stack Gas Velocity,Vs (fpm)	4208.90
Stack Gas Velocity,Vs (mpm)	1282.87
Volumetric Flow Rate (acfm)	58924.63
Volumetric Flow Rate (acmm)	1668.746
Volumetric Flow Rate (dscfm)	31570.07
Volumetric Flow Rate (dscmm)	894.065
Percent Isokinetic	104.77
Percent Excess Air	205.23
Fuel Factor,Fo	1.228
Ultimate CO2	17.02

APPENDIX D

CEM DATA

D.1 CEM DAS Printouts
D.2 Stripchart Tracings

APPENDIX D.1

CEM DAS PRINTOUTS

CALIBRATION SUMMARY 0733
09-19-1991 [illegible]
CALIBRATION FILE NAME =D: [illegible]P19CAL1.CAL

Chan.	Name	Units	Zero		Span		Slope	Int.
			Init.	Resp.	Conc.	Resp.		
1	O2	%	0.00	0.0008	18.00	[illegible]	[illegible]	[illegible]
2	THC	PPMV	0.00	-.1032	41.00	[illegible]	[illegible]	[illegible]
3	SO2	PPMV	0.00	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
4	CO	PPM	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
5	CO2	%	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
6	NOx	PPM	[illegible]	-.1.42	[illegible]	[illegible]	[illegible]	[illegible]
7			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
8			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
10			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
11			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
12			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
13			[illegible]	0.0006	0.0	[illegible]	[illegible]	[illegible]
14			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
15			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Press Shift-PrtSc to Print-Out Table
Press <C> to Continue

CEM INSTRUMENT DRIFT SUMMARY
09-19-1991 18:33:53

Chan.	Name	Units	Zero Conc.		Span		Drift % of Scale	
			Actual	Observed	Actual	Observed	Zero	Span
1	O2	%	0.000	0.177	18.00	17.62	0.71	-1.53
2	THC	PPMV	0.000	-.940	41.00	40.73	-0.74	-0.27
3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

[illegible]	[illegible]	[illegible]	0.00	0.00	0.00	0.00
[illegible]	[illegible]	[illegible]	0.00	0.00	0.00	0.00
[illegible]	0.000	0.000	0.00	0.00	0.00	0.00
[illegible]	0.000	0.000	0.00	0.00	0.00	0.00
11	0.000	0.000	0.00	0.00	0.00	0.00
12	0.000	0.000	0.00	0.00	0.00	0.00
13	0.000	0.000	0.00	0.00	0.00	0.00
14	0.000	0.000	0.00	0.00	0.00	0.00

Press Shift-PrtSc to Print Out Table
Press <C> to Continue

Avg.=	0.0	0.0	0.0	0.0	0.0	0.0

7168

RADIAN CORPORATION 0919RUN1

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
ONALASKA,WI
Performed for: MATHY 0734
Date Printed = 09-19-1991 Current Time = 08:34:56
File Name = D:\CEMDATA\091991.PRN Calibration File:D:\CEMDATA\0919CAL1.CAL

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
08:35:26	1.0	40.9	7.9	-7.8	-0.5	2.4
08:36:26	0.9	13.5	5.3	-5.3	-0.4	2.5
08:37:26	6.6	7.3	3.4	63.9	7.0	1.7
08:38:26	7.5	7.3	2.7	88.2	10.3	0.1
08:39:25	7.6	7.2	2.4	88.8	10.4	0.0
08:40:01	7.6	7.2	2.3	88.5	10.4	-0.0
08:40:22	7.7	7.1	2.3	88.9	10.4	-0.0
08:40:32	7.7	7.2	2.4	88.9	10.4	-0.0
08:40:42	7.9	7.1	2.1	88.1	10.4	-0.0
08:40:52	8.0	7.1	2.2	86.9	10.3	-0.1
08:41:01	7.8	7.1	2.3	86.7	10.2	-0.1
08:41:31	8.2	8.3	2.0	85.7	10.1	-0.1
08:42:01	7.6	8.7	25.3	81.0	9.8	43.7
08:42:31	3.9	8.6	173.2	40.3	6.4	134.6
08:43:01	1.2	8.5	256.4	-8.7	1.2	172.3
08:43:31	1.3	8.5	268.5	-9.1	-0.0	153.4
08:44:01	1.4	8.5	271.1	-10.4	-0.2	161.9
08:44:31	1.7	8.5	273.5	-11.5	-0.4	159.3
08:45:01	1.1	1.8	277.9	-12.0	-0.4	160.1
08:45:31	1.3	9.1	282.2	-12.0	-0.5	175.4
08:46:01	6.2	9.6	223.2	9.7	-0.4	86.8
08:46:31	17.6	9.7	69.7	14.4	-0.3	16.5
08:47:01	20.3	9.6	22.2	-3.9	-0.4	5.1
08:47:31	20.9	9.4	10.6	-7.7	-0.5	2.4
08:48:01	21.0	8.5	6.7	-8.3	-0.5	1.3
08:48:30	21.1	0.8	5.0	-8.8	-0.5	1.0
08:49:00	17.9	0.8	4.3	-8.3	-0.5	0.8
08:49:30	11.1	0.8	3.9	-9.1	-0.5	0.5
08:50:00	10.5	0.7	3.3	-9.0	-0.5	0.4
08:50:30	11.3	0.1	2.7	-8.8	-0.5	0.4
08:51:00	12.3	0.1	2.7	-9.3	-0.5	0.4
08:51:30	12.9	0.1	2.8	-9.3	-0.5	0.4
08:52:00	14.1	0.0	2.8	-9.3	-0.5	0.3
08:52:30	14.3	0.0	2.5	-9.3	-0.5	0.1
08:53:00	14.4	0.2	2.5	-9.2	-0.5	0.0
08:53:30	14.5	0.3	2.4	-9.2	-0.5	0.0
08:54:00	14.2	0.4	2.4	-9.2	-0.5	0.0
08:54:30	14.5	0.4	2.4	-9.2	-0.5	0.0
08:55:00	14.4	0.4	2.4	-9.2	-0.5	0.0
08:55:30	14.4	0.4	2.3	-9.2	-0.5	-0.0
08:55:59	14.5	0.3	2.1	-9.2	-0.5	-0.0
08:56:29	14.5	0.3	2.2	-9.2	-0.5	-0.0

Post Cal. QC
O2
CO
CO2

08:58:29	13.4	0.1	2.0	-9.3	-0.5	-0.2

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOx PPMV
~~08:58:59~~	16.0	0.1	1.9	-9.3	-0.5	-0.2
08:59:29 D750	17.0	0.1	1.8	-9.3	-0.5	-0.1
08:59:59	18.1	0.1	1.8	-9.3	-0.5	-0.1
09:00:29	17.1	0.1	1.7	-9.3	-0.5	-0.1
09:00:59	15.4	0.0	1.8	-9.3	-0.5	-0.1
09:01:29	13.7	-0.0	1.9	-9.2	-0.5	0.2
09:01:59	1.0	-0.0	3.1	-9.2	-0.5	0.4
09:02:29	-0.1	-0.0	2.1	-8.8	-0.5	-0.0
09:02:59	-0.1	-0.1	1.9	-9.3	-0.6	-0.1
09:03:28	-0.2	0.6	1.7	-9.3	-0.6	-0.2
09:03:58	-0.1	6.1	1.7	-9.0	-0.6	-0.2
09:04:28	2.6	6.6	1.9	5.7	0.4	-0.3
09:04:58	6.4	6.7	1.6	55.6	6.8	-0.3
09:05:28	7.3	6.8	1.0	82.7	9.7	-0.2
09:05:58	7.4	6.8	1.2	86.9	10.2	-0.2
09:06:28	7.5	6.8	1.1	87.9	10.3	-0.2
09:06:58	7.7	6.8	1.1	87.0	10.3	-0.3
09:07:28	7.8	6.8	1.1	86.4	10.2	-0.3
09:07:58	7.8	10.4	1.1	86.8	10.2	-0.3
09:08:28	8.1	38.0	1.0	86.1	10.3	-0.2
09:08:58	4.4	39.4	1.5	68.6	9.4	-0.0
09:09:28	1.5	37.5	1.0	14.6	3.4	-0.3
09:09:58	1.1	40.1	0.6	-4.4	0.5	-0.3
09:10:28	0.2	6.6	1.0	-5.0	-0.1	1.7
09:10:58	0.7	0.3	74.4	47.7	-0.0	74.1
09:11:27	-0.0	0.5	237.2	40.0	0.0	167.8
09:11:57	-0.0	0.4	279.6	-4.6	-0.4	138.5
09:12:27	0.6	7.5	263.4	-10.0	-0.5	173.4
09:12:57	3.9	56.0	244.9	-3.3	-0.3	140.7
09:13:27	7.1	60.4	134.5	65.8	-0.2	99.3
09:13:57	12.7	59.1	71.3	466.1	1.4	47.0
09:14:27	15.3	52.0	20.5	770.2	3.0	31.7
09:14:57	15.5	56.7	8.3	874.5	3.4	29.6
09:15:27	15.4	48.0	4.5	904.2	3.5	29.4
09:15:57	15.3	45.1	2.7	885.8	3.5	28.1
09:16:27	16.2	41.8	1.6	842.8	3.2	26.3
09:16:57	16.2	53.3	-1.1	830.5	3.2	26.1
09:17:27	16.1	54.[illegible]	0.8	871.7	3.3	26.6
09:17:57	16.4	53.[illegible]	0.6	844.6	3.2	26.3
09:18:26	16.4	49.2	0.3	829.9	3.2	26.0
09:18:56	16.6	49.8	0.0	809.1	3.0	26.4
09:19:26	15.3	47.0	-0.0	912.0	4.1	39.3
09:24:26	14.1	40.1	-0.1	1044.1	5.7	49.1
09:25:26	14.4	41.3	-0.3	1044.3	5.6	49.7
09:26:26	14.9	55.6	-0.2	1046.2	5.5	48.3
09:27:26	15.5	52.2	-0.2	1045.7	5.0	45.1
09:28:26	15.2	45.6	-0.4	1044.6	4.7	41.8
09:29:26	15.0	39.8	-0.7	1044.0	5.2	46.2
09:30:26	14.6	50.7	-0.7	1045.5	5.4	47.3
09:31:26	14.6	53.2	-0.7	1045.9	5.4	47.8
09:32:26	15.3	57.0	-0.9	1046.4	5.1	46.3
09:33:26	14.8	45.2	-1.1	1044.5	5.2	46.1
09:34:25	13.4	38.4	-1.1	1043.8	4.1	37.5

ON LINE

PAH/METALS ROLL #1 (PB)

Fyrite @ 0845
~14% O2
~4% CO2

09-17-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV	
~~09:38:25~~ 0838	14.7	40.5	-1.3	1044.2	5.3	47.0	
09:39:25	14.6	48.9	-1.0	1045.3	5.4	47.1	
09:40:25	14.4	55.5	-0.6	1046.2	5.4	47.2	
09:41:25	13.7	58.0	-0.7	1046.6	6.4	53.4	
09:42:25	14.1	56.5	-1.0	1046.4	5.8	49.2	
09:43:25	13.6	54.6	-0.8	1046.1	6.1	51.5	
09:44:25	13.9	59.1	-0.9	1046.8	6.2	52.2	
09:45:25	13.7	55.0	-1.0	1046.2	6.1	52.3	
09:46:25	13.7	45.7	-1.1	1044.9	6.2	52.7	
09:47:24	13.8	46.0	-1.2	1045.0	6.1	52.4	
09:48:24	14.0	60.5	-1.0	1047.1	5.9	50.3	
09:49:24	13.6	60.6	-0.9	1047.1	6.2	51.8	
09:50:24	13.7	52.5	-1.0	1045.9	6.3	53.1	
09:51:24	13.9	56.2	-1.0	1046.4	6.0	50.3	
09:52:24	13.6	61.5	-0.9	1047.2	6.3	52.2	
09:53:24	13.6	71.5	-0.6	1048.8	6.4	52.7	
09:54:24	13.8	65.7	-0.9	1047.9	6.2	51.7	
09:55:24	13.8	54.6	-1.2	1046.2	5.9	49.5	
09:56:24	13.3	51.2	-1.1	1045.7	6.4	52.3	
09:57:24	13.6	59.8	-1.0	1047.0	6.4	53.2	
09:58:24	13.2	65.6	-0.8	1047.9	6.5	53.2	
09:59:24	13.6	51.1	-1.1	1045.7	6.4	54.2	
10:00:23	13.8	48.4	-1.0	1045.3	6.0	49.7	
10:01:23	13.5	57.2	-0.9	1046.6	6.2	51.2	
10:02:23	13.4	62.2	-0.8	1047.3	6.4	53.1	
10:03:23	13.5	51.5	-1.1	1045.7	6.4	53.6	
10:04:23	13.6	54.2	-0.9	1046.1	6.1	50.8	
10:05:23	13.5	58.9	-0.8	1046.6	6.3	51.9	
~~10:06:23~~	13.5	55.2	-0.9	1046.3	6.2	51.6	START ALO #1
10:07:23	13.5	57.7	-0.8	1046.7	6.3	51.8	
10:08:23	13.7	53.6	-0.7	1046.1	6.1	51.0	
10:09:23	13.7	52.2	-1.0	1045.8	6.0	48.9	
10:10:23	13.5	52.3	-0.9	1046.0	6.1	50.1	
10:11:23	13.2	51.2	-1.0	1045.7	6.5	52.6	
10:12:23	13.5	52.2	-1.1	1045.9	6.4	53.5	
10:13:23	13.7	52.2	-0.8	1045.9	6.1	50.2	
10:14:23	13.6	50.9	-1.0	1045.7	6.1	50.9	
10:15:23	13.5	43.8	-1.0	1044.7	6.2	51.6	
10:16:23	13.4	40.2	-1.0	1044.3	6.3	52.8	
10:17:23	13.4	58.0	-0.7	1046.8	6.4	53.0	
10:18:23	13.3	58.4	-0.8	1046.8	6.5	53.6	
10:19:23	13.2	50.8	-0.8	1045.7	6.6	54.5	
10:20:23	13.3	45.5	-1.0	1045.0	6.4	52.9	
10:21:23	13.2	53.5	-0.7	1046.1	6.6	54.6	
10:22:23	13.4	45.2	-0.8	1045.0	6.2	51.0	END PARAMETERS #1
10:23:23	13.3	43.5	-0.9	1045.6	6.4	52.4	
10:24:22	13.3	47.6	-0.7	1045.3	6.4	52.9	
10:25:22	13.1	28.7	-0.6	1045.9	6.5	54.0	
10:26:22	12.4	0.1	-0.7	1045.7	6.8	55.7	
10:27:21	12.8	-0.3	-0.6	1045.7	6.1	51.5	
10:28:21	1.7	-0.4	-0.8	528.6	2.9	42.5	
10:29:21	0.1	-0.3	-0.0	56.0	-0.2	4.9	
10:30:21	-0.8	-0.4	-0.0	24.4	-0.4	1.8	

END 1st Test

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
~~10:34:21~~ 0934	12.4	10.9	-0.7	-0.8	-0.6	0.6
10:35:21	20.4	10.6	-0.9	-7.1	-0.6	-0.1
10:36:21	21.2	10.9	-1.0	-10.0	-0.6	-0.2
10:37:21	21.4	17.0	-1.1	-10.1	-0.6	-0.2
10:38:21	20.9	37.4	-0.9	61.4	-0.5	7.2
10:39:21	16.5	38.6	-0.8	756.4	2.3	25.8
10:40:20	16.3	39.5	-0.9	922.6	3.2	25.5
10:41:20	16.5	50.7	0.1	347.4	3.1	27.0
10:42:20	16.3	9.4	-0.8	895.2	3.4	18.6
10:43:20	20.7	10.0	-0.8	219.5	0.6	2.3
10:44:20	21.4	10.1	-0.9	16.9	-0.4	0.4
Avg.=	0.0	0.0	0.0	0.0	0.0	0.0

RADIAN CORPORATION 0919rum2

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
ONALASKA,WI
Performed for: MATHY 0946
Date Printed = 09-19-1991 Current Time = 10:46:05
File Name = D:\CEMDATA\091991.PRN Calibration File:D:\CEMDATA\0919CAL1.CAL

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
10:46:09	21.4	6.5	-0.6	-4.6	-0.5	-0.0
10:46:19	21.4	6.3	-0.5	-4.7	-0.5	-0.1
10:46:29	21.4	6.3	-0.5	-4.8	-0.5	-0.1
10:46:39	21.4	6.3	-0.4	-5.4	-0.5	-0.1
10:46:49	21.4	6.1	-0.5	-5.9	-0.5	-0.1
10:46:59	21.4	6.1	-0.5	-5.9	-0.5	-0.1
10:47:09	21.4	6.1	-0.6	-5.9	-0.5	-0.1
10:47:19	21.4	6.0	-0.6	-5.9	-0.5	-0.1
10:47:29	21.4	5.9	-0.9	-5.9	-0.5	-0.1
10:47:39	21.4	5.7	-0.6	-5.9	-0.5	-0.1
10:47:49	21.4	5.1	-0.5	-5.9	-0.5	-0.1
10:47:59	21.4	5.4	-0.5	-5.9	-0.5	-0.1
10:48:09	21.4	5.5	-0.5	-5.9	-0.5	-0.1
10:48:19	21.4	5.4	-0.3	-5.9	-0.5	-0.1
10:48:29	21.5	5.3	-0.6	-5.9	-0.5	-0.1
10:49:29	18.1	2.9	-0.6	-6.1	-0.5	-0.1
10:50:29	6.6	1.8	-0.6	-7.6	-0.5	-0.2
10:51:29	7.2	2.3	-0.7	-8.1	-0.5	-0.3
10:52:29	7.7	2.2	-0.9	-8.1	-0.5	-0.3
10:53:29	8.0	2.2	-0.7	-8.1	-0.5	-0.3
10:54:29	6.7	2.0	-0.8	-8.1	-0.5	-0.2
10:55:29	5.0	1.9	-0.6	-8.1	-0.5	-0.2
10:56:29	4.8	1.9	-0.6	-8.1	-0.5	-0.2
10:57:29	4.8	1.8	-0.7	-8.1	-0.5	-0.2
10:58:29	4.7	1.8	-0.6	-8.1	-0.5	-0.2
10:59:29	4.4	1.7	-0.6	-8.1	-0.5	-0.2
11:00:29	4.6	1.6	-0.5	-8.1	-0.5	-0.2
11:01:29	4.5	1.6	-0.5	-8.1	-0.5	-0.2
11:02:29	5.1	1.6	-0.5	-8.1	-0.5	-0.3
11:03:27	7.1	1.6	-0.6	-8.1	-0.5	-0.3
11:04:27	9.6	3.0	-0.5	-8.6	-0.5	-0.3
11:05:27	17.5	7.6	-0.4	-8.9	-0.5	-0.3
11:06:27	21.0	9.5	-0.3	-9.2	-0.6	-0.4
11:07:27	21.0	9.5	-0.4	-9.2	-0.6	-0.4
11:08:27	21.5	9.5	-0.3	-9.2	-0.6	-0.4
11:09:27	21.5	9.4	-0.3	-9.2	-0.6	-0.4
11:10:27	21.5	9.4	-0.5	-9.2	-0.6	-0.4
11:11:27	21.5	9.4	-0.3	-9.3	-0.6	-0.4
11:12:27	21.5	9.4	-0.2	-9.2	-0.6	-0.4
11:13:27	21.5	9.5	-0.3	-9.2	-0.6	-0.4
11:14:27	21.5	9.4	-0.4	-9.2	-0.6	-0.4
11:15:27	21.5	9.7	-0.6	-9.5	-0.6	-0.4

11:19:26	21.5	9.3	-0.6	-9.2	-0.6	-0.4

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
11:20:26	21.6	9.3	-0.5	-9.2	-0.6	-0.4
11:21:26	21.5	9.2	-0.6	-9.2	-0.6	-0.4
11:22:26	21.5	9.2	-0.5	-9.2	-0.6	-0.4
11:23:26	21.5	9.2	-0.5	-9.2	-0.6	-0.4
11:24:26	21.6	9.2	-0.4	-9.2	-0.6	-0.4
11:25:26	21.5	9.2	-0.4	-9.2	-0.6	-0.4
11:26:26	21.0	6.6	-0.5	-8.8	-0.6	-0.3
11:27:26	21.3	5.6	-0.7	-8.2	-0.5	-0.3
11:28:26	21.4	7.6	-0.7	-7.6	-0.5	-0.3
11:29:25	20.2	7.9	-0.5	-2.7	-0.5	-0.2
11:30:25	2.9	1.7	-0.7	-8.0	-0.6	-0.3
11:31:25	1.0	1.3	-0.6	-8.2	-0.6	-0.3
11:32:25	0.5	1.2	-0.7	-8.6	-0.6	-0.3
11:33:25	0.2	1.0	-0.7	-9.1	-0.6	-0.3
11:34:25	0.4	0.9	-0.8	-9.3	-0.6	-0.3
11:35:25	0.3	0.9	-0.9	-9.1	-0.6	-0.3
11:36:25	4.4	1.2	-0.8	-9.2	-0.6	-0.3
11:37:25	10.2	2.9	-0.6	-1.2	0.0	-0.3
11:38:25	13.2	5.3	-0.7	40.6	5.0	-0.3
11:39:25	3.6	5.5	-0.8	59.2	7.2	-0.3
Avg.=	15.2	5.5	-0.6	-6.0	-0.3	-0.3

RADIAN CORPORATION 0919RUN5

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MARTY #6
ONALASKA,WI
Performed for: MARTY 1100
Date Printed = 09-19-1991 Current Time = 12:00:24
File Name = D:\CEMDATA\091991.PRN Calibration File:D:\CEMDATA\0919CAL1.CAL

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
12:00:26	17.4	45.7	-0.6	740.7	2.8	34.6
12:00:36	17.5	46.6	-0.7	737.4	2.8	35.6
12:00:46	17.4	51.0	-0.6	723.8	2.8	35.0
12:00:56	17.6	55.0	-0.7	728.7	2.9	34.6
12:01:17	17.6	57.8	-0.9	727.3	3.2	34.2
12:01:27	17.6	54.4	-0.9	726.3	3.2	33.7
12:02:27	17.1	59.2	-1.0	762.6	3.3	33.9
12:03:27	17.2	59.4	-0.8	772.3	3.4	35.3
12:04:27	17.6	51.9	-1.0	689.6	3.0	32.8
12:05:27	17.8	51.9	-0.8	640.0	2.8	-4.2
12:06:27	17.5	56.3	-1.0	649.9	2.9	30.2
12:07:27	17.6	56.3	-1.0	651.7	2.5	31.7
12:08:27	17.8	53.0	-0.9	647.3	2.4	27.3
12:09:27	18.7	42.4	-1.1	500.1	1.9	14.8
12:10:27	18.8	40.9	-1.2	464.2	1.8	13.4
12:11:27	18.8	50.2	-1.3	469.8	1.8	15.1
12:12:27	18.8	41.2	-1.2	476.7	1.8	14.9
12:13:26	19.0	52.9	-1.0	465.4	1.5	14.3
12:14:26	19.0	56.8	-1.0	446.2	1.6	14.2
12:15:26	19.1	58.3	-1.0	420.5	1.6	13.2
12:16:26	19.3	46.4	-1.4	389.4	1.3	11.5
12:17:26	19.4	44.8	-1.3	383.7	1.2	10.7
12:18:26	19.4	55.5	-1.0	380.2	1.2	10.5
12:19:26	19.5	56.7	-0.9	358.1	1.2	10.0
12:21:26	19.7	54.9	-1.1	338.2	1.0	9.3
12:22:26	19.9	42.8	-1.2	291.6	0.8	7.3
12:23:26	19.9	41.2	-1.3	273.4	0.7	6.1
12:23:26	20.3	42.8	-1.4	218.0	0.6	4.8
12:24:26	14.7	47.6	-1.2	458.1	2.5	39.9
12:25:26	13.8	57.0	-1.0	1044.7	5.6	35.7
12:26:26	14.1	59.4	-0.8	1044.7	5.0	34.1
12:27:26	14.2	56.9	-1.0	1044.6	4.9	33.8
12:28:26	14.2	57.3	-1.1	1044.7	4.9	34.3
12:29:26	14.0	62.0	-0.8	1044.8	5.0	34.2
12:30:26	14.2	63.0	-1.0	1044.9	5.0	34.2
12:31:26	14.3	55.7	-1.2	1044.6	5.2	32.5
12:32:37	14.3	45.9	-1.2	1043.8	4.7	32.9
12:33:36	14.3	44.1	-1.0	1043.7	4.8	34.0
12:34:36	14.1	46.0	-1.2	1044.1	4.9	34.0
12:35:36	14.2	51.7	-1.0	1044.6	4.9	33.7
12:36:36	14.2	57.4	-1.1	1044.7	5.0	34.2
12:37:36	14.2	59.9	-1.2	1044.7	4.9	33.4

START PM10 RUN #1 (2B)

ANALYZERS READY (2B)

12:41:36 14.1 59.7 -1.3 1044.7 5.0 34.5

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
12:42:36 M42	14.0	54.5	-1.4	1044.5	5.0	34.7
12:43:36	13.9	47.8	-1.4	1044.1	5.1	34.8
12:44:36	14.1	52.2	-1.5	1044.4	5.0	33.5
12:45:36	14.1	42.7	-1.3	1043.5	4.9	32.6
12:46:35	14.1	47.1	-1.5	1043.9	4.9	33.8
12:47:35	14.0	50.0	-1.5	1044.3	5.0	34.3
12:48:35	14.1	44.7	-1.2	1043.7	4.9	33.3
12:49:35	14.2	52.5	-1.3	1044.5	4.9	33.1
12:50:35	14.2	58.6	-1.1	1044.7	5.0	33.5
12:51:35	14.2	62.0	-0.9	1045.3	5.3	33.7
12:52:35	14.0	57.4	-1.3	1045.5	5.9	34.3
12:53:35	14.0	48.3	-1.4	1043.9	6.0	34.7
12:54:35	14.0	56.7	-1.4	1045.3	6.0	33.9
12:55:35	14.1	62.8	-1.1	1044.8	5.9	33.7
12:56:35	14.2	69.7	-1.0	1045.9	5.9	33.1
12:57:35	14.3	54.9	-1.4	1044.8	5.7	32.2
12:58:35	14.1	58.4	-1.0	1045.3	5.8	33.3
12:59:34	14.2	67.3	-0.9	1045.6	5.9	33.2
13:00:34	14.4	66.7	-0.9	1045.0	5.7	31.8
13:01:34	14.2	64.2	-1.0	1045.1	5.7	33.7
13:02:34	14.2	69.1	-1.0	1045.9	5.5	33.9
13:03:34	14.3	62.7	-1.2	1046.0	4.9	32.5
13:04:34	14.4	69.2	-1.0	1047.3	4.8	32.1
13:05:34	14.4	71.4	-0.9	1047.6	4.9	32.2
13:06:34	14.3	65.7	-1.1	1046.8	4.9	33.1
13:07:34	14.1	54.7	-1.0	1045.1	5.0	34.0
13:08:34	14.3	45.8	-0.1	1047.2	5.2	31.3
13:09:34	21.3	11.3	-1.6	462.5	1.9	5.8
13:10:34	21.4	9.3	-0.8	45.9	-0.2	1.7
13:11:34	21.4	9.2	-0.9	12.1	-0.4	1.0
13:12:34	21.5	9.2	-1.0	2.7	-0.4	0.8
13:13:33	21.5	8.9	-0.8	-1.7	-0.4	0.7
13:14:33	21.6	8.1	-0.6	-4.0	-0.5	0.6
13:15:33	21.6	7.2	-0.5	-5.1	-0.5	0.6
13:16:33	21.6	6.8	-0.5	-6.1	-0.5	0.6
13:17:33	21.6	6.6	-0.6	-6.1	-0.5	0.6
13:18:33	21.5	6.1	-0.5	-6.1	-0.5	0.5
13:19:33	21.6	5.8	-0.3	-6.7	-0.5	0.5
13:20:33	21.6	5.6	-0.2	-7.1	-0.5	0.5
13:21:33	21.6	5.3	-0.3	-7.2	-0.5	0.5
13:22:33	21.6	5.4	-0.2	-7.2	-0.5	0.5
13:23:33	21.6	5.4	-0.2	-7.2	-0.5	0.5
Avg.=	0.0	0.0	0.0	0.0	0.0	0.0

END PM10 RUN #1 (2B)

END 2nd Test

RADIAN CORPORATION 0919[illegible]6

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
GNALASKA,WI
Performed for: MATHY 1240
Date Printed = 09-19-1991 Current Time = ~~13:39:56~~
File Name = D:\CEMDATA\091991.PRN Calibration File:D:\CEMDATA\0919CAL1.CAL

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
~~13:39:57~~ 1238	1.2	2.0	0.2	-8.2	-0.6	0.1
13:40:19	1.0	1.9	0.3	-8.3	-0.6	0.1
13:40:39	1.2	1.8	0.1	-8.3	-0.6	0.1
13:40:59	1.5	1.8	0.0	-8.2	-0.6	0.0
13:41:19	2.1	1.7	-0.1	-8.2	-0.6	0.0
13:41:39	2.4	1.7	0.0	-8.2	-0.6	0.1
13:41:58	3.4	1.7	0.1	-8.2	-0.6	0.1
13:42:18	4.4	1.7	-0.1	-8.2	-0.5	0.1
13:42:38	10.2	2.0	-0.0	-9.2	-0.5	0.0
13:42:58	15.5	2.2	0.2	-9.2	-0.5	-0.0
13:43:18	16.1	2.2	0.1	-9.2	-0.5	0.0
13:43:38	16.3	2.2	0.2	-9.2	-0.5	0.0
13:43:58	16.8	2.1	0.1	-9.2	-0.5	0.0
13:44:18	16.9	2.1	0.0	-9.2	-0.5	0.0
13:44:38	16.8	2.1	-0.1	-9.2	-0.5	0.0
13:44:58	16.4	2.1	-0.1	-9.2	-0.5	-0.0
13:45:18	16.8	2.1	-0.2	-9.2	-0.5	-0.0
13:45:38	17.0	2.1	-0.1	-9.2	-0.5	0.0
13:45:58	17.2	2.2	-0.2	-9.2	-0.5	-0.0
13:46:18	17.7	2.2	-0.2	-8.2	-0.5	-0.0
13:46:38	18.7	2.3	0.1	-8.2	-0.5	-0.0
13:46:58	19.2	2.3	-0.1	-8.2	-0.5	-0.0
13:47:17	19.5	2.3	-0.1	-8.2	-0.5	-0.0
13:47:37	19.9	2.4	-0.3	-8.2	-0.5	-0.0
13:47:57	20.1	2.4	-0.2	-8.2	-0.5	-0.0
13:48:17	20.2	2.4	-0.3	-8.2	-0.5	0.0
13:48:37	18.3	3.4	-0.1	-5.8	-0.5	0.1
13:48:57	20.1	3.4	-0.2	-3.1	-0.5	0.0
13:49:17	20.4	2.5	-0.4	-4.8	-0.5	-0.0
13:49:37	20.0	2.2	-0.5	-7.5	-0.5	-0.0
13:49:57	16.8	2.2	-0.4	-8.1	-0.5	0.0
13:50:17	5.3	2.6	-0.4	-7.4	-0.5	0.1
13:50:37	7.3	5.6	-0.5	4.5	-0.3	0.1
13:50:57	7.8	7.5	-0.4	43.3	4.3	0.1
13:51:17	7.6	8.0	-0.3	75.7	9.6	0.1
13:51:38	7.6	8.2	-0.5	85.9	11.2	0.1
13:51:53	7.6	8.3	-0.6	88.8	11.5	0.1
13:52:07	7.6	8.2	-0.5	89.4	11.6	0.1
13:52:22	7.6	8.2	-0.8	89.0	11.6	0.1
13:52:37	7.6	8.2	-0.7	88.9	11.6	0.1
13:52:52	7.6	8.2	-0.8	89.1	11.6	0.1
13:53:07	7.6	8.2	-0.8	89.9	11.6	0.1

13:54:07	7.5	8.2	-0.8	89.9	10.3	0.1

09-19-1991 Time	O2 %	THC PPMV	NOX PPMV	CO PPMV	CO2 %	NOX PPMV
13:54:22	7.5	8.2	-0.8	89.2	10.3	0.1
13:54:37	7.5	8.1	-0.9	89.4	10.3	0.1
13:54:52	7.6	8.1	-0.9	89.1	10.3	0.1
13:55:07	7.6	8.1	-0.7	89.9	10.3	0.1
13:55:22	7.6	8.2	-0.6	89.9	10.3	0.1
13:55:37	7.6	8.2	-0.6	89.9	10.3	0.1
13:55:52	7.6	8.2	-0.6	89.9	10.3	0.1
13:56:07	7.6	8.1	-0.6	89.9	10.4	0.1
13:56:21	7.6	8.2	-0.8	89.5	10.3	0.1
13:57:21	4.2	4.7	101.4	65.9	9.2	113.7
13:58:23	-0.1	1.8	287.9	-6.7	0.5	199.8
13:58:43	-0.2	1.6	299.6	-11.0	-0.4	202.0
13:59:01	-0.2	1.5	300.6	-12.2	-0.5	202.2
13:59:10	-0.1	1.4	301.0	-11.5	-0.4	202.7
13:59:20	-0.2	1.3	300.6	-12.1	-0.5	202.6
13:59:30	-0.2	1.3	301.5	-12.1	-0.5	203.5
13:59:40	-0.2	1.2	302.3	-12.2	-0.5	202.3
14:00:40	0.0	21.9	158.3	-11.1	-0.5	56.7
14:01:40	0.1	33.2	11.3	-10.3	-0.6	3.6
14:02:40	0.2	38.2	3.8	-9.9	-0.6	1.3
14:03:40	0.1	40.1	1.9	-9.7	-0.6	0.7
14:04:40	0.5	44.4	1.4	-9.1	-0.6	0.7
14:05:40	-0.1	48.1	-0.2	-7.5	-0.6	-0.0
14:06:22	-0.2	40.6	-1.0	-10.6	-0.7	-0.1
14:06:32	-0.1	40.6	-0.8	-10.6	-0.7	-0.1
14:06:42	-0.1	40.5	-1.2	-10.7	-0.7	-0.1
14:06:52	-0.1	40.5	-1.3	-10.7	-0.7	-0.1
14:07:02	-0.1	40.5	-1.2	-10.7	-0.7	-0.1
14:07:12	-0.1	40.5	-1.3	-10.7	-0.7	-0.1
14:07:22	-0.2	38.6	-1.1	-10.8	-0.7	-0.1
14:07:32	0.6	13.5	-1.3	-11.9	-0.7	-0.1
14:07:41	10.5	24.7	-0.4	-11.9	-0.7	1.2
14:07:51	5.0	26.2	1.2	-11.7	-0.7	0.8
14:08:01	5.7	23.6	1.4	-11.7	-0.6	0.8
14:08:11	7.6	15.1	1.3	-11.1	-0.6	0.9
14:08:21	12.1	7.1	1.4	-9.2	-0.6	0.8
14:08:31	14.7	7.7	1.5	-9.3	-0.6	0.4
14:08:41	17.8	5.3	1.1	-8.1	-0.6	0.3
14:08:51	19.3	4.5	0.6	-8.1	-0.6	0.2
14:09:11	20.2	4.2	0.4	-8.1	-0.6	0.1
14:10:01	21.1	3.4	-0.1	-8.1	-0.6	0.0
14:11:01	21.4	2.3	-0.3	-8.1	-0.6	-0.0
14:12:01	21.4	2.2	-0.4	-8.1	-0.6	-0.0
14:13:01	21.4	1.9	-0.8	-8.1	-0.6	-0.0
14:14:01	21.4	1.9	-0.8	-8.1	-0.6	-0.1
14:15:01	21.4	2.0	-0.6	-8.1	-0.6	-0.1
14:16:00	21.4	1.8	-0.7	-8.1	-0.6	-0.1
14:17:00	21.4	0.8	-0.7	-8.2	-0.6	-0.1
14:18:00	21.4	1.2	-0.8	-8.2	-0.6	-0.1
14:19:00	21.5	1.6	-1.0	-8.1	-0.6	-0.1
14:20:00	21.5	1.6	-0.8	-8.1	-0.6	-0.1
14:21:00	21.4	1.6	-1.0	-8.1	-0.6	-0.1

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
14:26:00	21.4	1.9	-1.3	-8.1	-0.6	-0.1
14:27:00	21.4	1.7	-1.2	-8.1	-0.6	-0.1
14:28:00	21.4	1.7	-1.3	-8.1	-0.6	-0.1
14:28:59	21.4	1.7	-1.4	-8.1	-0.6	-0.1
14:29:59	21.4	2.4	-1.5	-8.1	-0.6	-0.1
14:30:59	21.4	3.1	-1.5	-8.1	-0.6	-0.1
14:31:59	21.3	25.3	-1.4	-9.4	-0.6	-0.1
14:32:59	21.1	30.4	-1.3	10.1	-0.5	1.5
14:33:59	18.5	81.3	-0.7	203.3	0.9	14.5
14:34:59	16.4	58.8	-1.6	967.5	2.6	21.2
14:35:59	17.2	64.9	-1.4	954.8	2.5	18.0
14:36:59	18.8	90.7	-0.3	523.7	1.7	13.8
14:37:59	19.0	49.4	-1.9	620.1	1.5	17.1
14:38:59	17.8	48.8	-1.8	895.9	1.9	15.5
14:39:59	18.8	54.2	-1.7	542.4	1.5	10.7
14:40:59	14.7	46.0	-1.7	738.1	2.7	37.5
14:41:58	12.8	54.0	-1.5	1045.8	5.5	41.9
14:42:58	12.7	49.9	-1.5	1045.1	5.5	42.3
14:43:58	12.7	56.9	-1.5	1046.1	5.5	42.8
14:44:58	12.7	55.1	-1.5	1045.9	5.6	43.2
14:45:58	12.7	52.1	-1.5	1045.4	5.6	43.4
14:46:58	12.9	59.4	-1.5	1046.4	5.6	39.4
14:47:58	13.8	62.6	-1.4	1045.5	5.1	35.0
14:48:58	14.4	60.7	-1.4	1009.3	4.6	33.0
14:49:58	14.8	46.5	-1.8	939.9	4.2	30.9
14:50:58	14.8	48.1	-1.7	888.9	3.9	31.7
14:51:58	15.2	58.9	-1.7	874.1	4.0	26.0
14:52:58	15.6	61.7	-1.7	722.4	3.0	30.3
14:53:58	14.0	56.6	-1.7	1018.5	4.7	34.6
14:54:57	14.7	45.4	-2.0	890.9	4.2	31.2
14:55:57	15.1	40.2	-1.9	880.3	3.9	29.1
14:56:57	15.4	48.3	-1.8	830.3	3.6	26.7
14:57:57	15.9	54.6	-1.8	813.4	3.3	25.6
14:58:57	15.9	52.3	-1.7	847.4	3.3	26.8
14:59:57	15.6	51.6	-1.8	848.5	3.3	27.0
15:00:57	13.4	49.5	-1.8	991.9	4.4	40.3
15:01:57	12.7	51.8	-1.5	1044.4	5.6	41.5
15:02:57	12.7	59.4	-1.4	1044.7	5.7	42.4
15:03:57	12.5	60.3	-1.4	1044.7	5.9	44.3
15:04:57	12.3	56.5	-1.2	1044.7	6.1	44.7
15:05:57	12.5	59.6	-1.4	1044.7	6.0	43.4
15:06:57	12.6	66.0	-1.3	1045.6	5.9	42.7
15:07:56	12.7	69.2	-1.0	1046.6	5.8	41.9
15:08:56	12.7	58.8	-1.3	1044.9	5.8	42.3
15:09:56	12.3	54.8	-1.2	1045.7	6.0	44.1
15:11:00	12.1	65.9	-1.2	1047.1	6.2	44.5
15:12:00	12.2	66.3	-1.1	1047.2	6.1	43.1
15:13:00	12.1	62.9	-1.0	1046.5	6.1	44.0
15:14:00	11.9	68.5	-0.6	1047.5	6.3	46.1
15:15:00	11.7	76.8	-0.7	1049.0	6.4	47.1
15:16:00	11.7	77.4	-0.6	1049.0	6.5	46.9
15:17:00	12.0	78.0	-0.6	1049.2	6.4	45.4
15:18:00	12.1	77.9	-0.4	1049.2	6.3	44.9

START PAH/METAL #2 (EB)

1340 plant on-line

ANALYZERS REBOOT (PB)

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
[illegible]	12.0	64.6	-0.9	1045.2	6.3	44.8
15:22:59	12.1	57.1	-1.1	1044.7	6.1	44.4
15:23:59	12.1	68.1	-0.9	1045.8	6.1	44.9
15:24:59	12.0	73.5	-0.8	1047.7	6.3	45.8
15:25:59	12.0	71.9	-0.8	1047.1	6.3	45.2
15:26:59	12.0	71.5	-0.9	1047.0	6.3	44.9
15:27:59	12.2	59.0	-0.9	1044.7	6.1	42.8
15:28:59	12.0	50.4	-1.0	1044.4	6.1	44.6
15:29:59	11.6	68.7	-0.7	1046.0	6.5	46.1
15:30:59	12.0	71.0	-0.6	1046.8	6.4	44.8
15:31:59	12.1	74.4	-0.5	1047.9	6.3	43.4
15:32:59	12.1	67.8	-0.6	1045.9	6.1	44.1
15:33:59	11.9	55.7	-0.8	1044.6	6.3	44.9
15:34:58	11.9	46.8	-0.9	1044.4	6.3	44.9
15:35:58	11.8	49.2	-0.9	1044.3	6.3	44.9
15:36:58	11.9	66.7	-0.7	1045.5	6.4	44.9
15:37:58	12.1	74.9	-0.4	1048.2	6.3	43.4
15:38:58	12.2	79.4	-0.0	1049.1	6.1	42.9
15:39:58	12.0	85.9	0.6	1050.3	6.3	45.3
15:40:58	12.0	87.3	0.6	1050.6	6.5	44.8
15:41:58	12.2	76.7	0.3	1048.6	6.2	42.6
15:42:58	12.4	74.5	-0.1	1047.9	5.9	41.6
15:43:58	12.3	79.4	0.0	1049.2	6.0	43.7
15:44:58	12.2	82.1	0.1	1049.6	6.2	43.9
15:45:58	12.1	73.8	-0.3	1047.5	6.2	44.2
15:46:58	12.0	65.9	-0.4	1044.7	6.2	44.5
15:47:57	12.1	63.3	-0.2	1045.1	6.2	44.2
15:48:57	12.0	65.1	-0.2	1044.6	6.2	44.3
15:49:57	11.8	70.7	0.2	1046.7	6.4	45.3
15:50:57	11.8	75.3	0.1	1048.2	6.4	44.5
15:51:57	11.9	74.2	0.1	1048.0	6.4	44.2
15:52:57	12.1	74.3	0.1	1048.0	6.3	42.4
15:53:57	12.5	62.5	-0.2	1045.2	5.9	40.1
15:54:57	12.4	66.7	-0.1	1045.5	5.9	42.1
15:55:57	12.4	72.6	0.2	1047.4	6.0	41.4
15:56:57	12.6	74.0	0.3	1047.6	5.8	40.0
15:57:57	13.9	59.3	-0.0	1044.7	4.9	34.9
15:58:57	12.1	48.4	-0.0	1044.3	5.8	43.0
15:59:57	12.1	61.3	0.1	1045.1	6.1	42.2
16:00:56	12.2	56.1	-0.0	1044.6	6.0	41.9
16:01:56	12.4	54.1	0.2	1044.6	5.9	40.4
16:02:56	12.6	61.3	0.2	1044.7	5.7	40.3
16:03:56	12.2	50.3	-0.0	1044.5	5.9	42.4
16:04:56	12.1	60.0	-0.0	1045.5	6.1	42.8
16:05:56	12.3	71.7	0.2	1047.1	6.1	41.7
16:06:56	12.1	71.4	0.3	1047.0	6.1	44.0
16:07:56	12.3	77.0	0.8	1048.5	6.2	41.8
16:08:56	12.1	70.1	0.5	1046.5	6.1	43.7
16:09:56	12.2	74.3	0.6	1047.8	6.2	42.9
16:10:56	12.4	74.1	0.7	1047.8	6.0	40.9
16:11:56	12.4	64.3	0.2	1045.2	5.9	41.2
16:12:56	12.1	59.9	0.3	1044.8	6.0	44.0
16:13:55	12.0	59.3	0.5	1049.1	6.3	43.5

09-19-1991 Time	O2 %	THC PPMV	[illegible] PPMV	CO PPMV	CO2 %	NOX PPMV
~~16:17:55~~ 1517	12.2	72.8	0.7	1047.4	6.1	42.3
16:18:55	12.1	73.3	0.8	1047.5	6.2	43.5
16:19:55	12.1	72.9	0.8	1047.5	6.2	43.2
16:20:55	12.3	56.0	0.5	1044.6	6.0	41.4
16:21:55	12.2	58.6	0.6	1044.5	6.0	42.2
16:22:55	12.1	63.6	0.5	1044.9	6.1	43.5
16:23:55	12.1	69.5	0.7	1046.3	6.2	44.0
16:24:55	12.2	78.8	1.0	1049.0	6.2	42.7
16:25:55	12.1	59.4	0.6	1045.0	6.0	43.1
16:26:54	12.1	74.8	1.2	1048.0	6.3	43.2
16:27:54	12.3	73.6	0.9	1047.5	6.0	41.6
16:28:54	12.0	61.4	0.6	1044.8	6.1	44.0
16:29:54	12.0	65.4	0.6	1045.3	6.3	43.8
16:30:54	12.1	66.3	0.6	1045.6	6.2	42.5
16:31:54	12.2	67.8	1.0	1046.3	6.1	42.4
16:32:54	12.5	69.5	0.9	1046.5	5.9	40.6
16:33:54	12.2	63.3	0.9	1044.9	6.0	42.1
16:34:54	12.1	65.4	0.6	1045.6	6.2	43.5
16:35:54	12.3	70.6	0.7	1046.7	6.2	41.3
16:36:54	12.1	64.9	0.7	1045.4	6.1	43.5
16:37:54	11.9	64.1	0.8	1045.2	6.3	43.9
16:38:54	12.3	76.0	1.0	1048.4	6.3	41.2
16:39:53	12.6	65.1	0.9	1046.0	5.8	39.6
16:40:53 1540	12.4	49.3	0.8	1044.4	5.8	41.4
16:41:53	12.3	44.6	0.7	1043.9	6.0	41.8
16:42:53	12.3	54.3	0.8	1044.4	6.0	41.7
16:43:53	12.4	53.6	0.8	1045.3	6.0	40.8
16:44:53	12.5	70.6	0.9	1046.7	5.9	40.6
16:45:53	12.0	61.1	0.9	1044.8	6.1	43.7
16:46:53	11.9	69.1	1.2	1046.1	6.4	44.3
16:47:53	12.0	71.9	1.4	1047.1	6.3	43.3
16:48:53	11.6	72.6	1.2	1047.4	6.5	45.5
16:49:53	11.6	68.4	1.1	1046.1	6.7	45.4
16:50:53	11.7	52.5	0.9	1044.1	6.5	44.5
16:51:53	7.6	11.3	0.4	755.8	6.6	7.8
Avg.=	12.1	59.5	9.2	567.8	3.9	28.9

END PAH/METALS Run #2 (RB)

RADIAN CORPORATION 09[redacted]

Field Testing and Process E[redacted]ering Dept.
Continuous Emissions Monitor[redacted] Data
MAHTY #6
ONALASKA,WI
Performed for: MATHY 1553
Date Printed = 09-19-1991 Current Time = 16:53:10
File Name = D:\CEMDATA\091991.PRN Calibration File:D:\CEMDATA\0919CAL1.CAL

09-19-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
~~16:52:42~~ 1553	7.6	8.2	0.6	127.6	10.4	0.8
16:53:27	7.6	7.2	0.5	95.9	10.6	0.5
16:53:42	7.6	7.1	0.2	95.1	10.6	0.5
16:53:57	7.6	6.9	0.4	95.1	10.6	0.4
16:54:12	7.6	4.6	0.4	95.0	10.6	0.4
16:54:27	7.6	6.5	0.2	94.5	10.6	0.4
16:54:42	7.6	6.4	0.4	93.9	10.6	0.4
16:54:57	7.6	6.4	0.5	93.6	10.6	0.4
16:55:12	7.6	6.4	0.3	93.9	10.6	0.3
16:55:27	7.6	6.1	0.3	93.1	10.6	0.3
16:55:42	7.5	2.1	0.7	93.0	10.6	38.0
16:55:57	3.3	0.9	45.1	76.5	10.4	171.6
16:56:12	0.3	0.7	187.0	35.2	7.5	200.9
16:56:27	-0.1	0.6	260.1	2.8	2.4	204.9
16:56:42	-0.2	0.5	284.4	-8.9	-0.0	206.4
16:56:57	-0.3	0.4	294.3	-11.2	-0.4	207.0
16:57:11	-0.3	0.4	299.1	-11.4	-0.5	207.4
16:57:26	-0.3	0.4	302.0	-12.2	-0.6	207.7
16:57:41	-0.3	0.3	304.1	-12.2	-0.6	207.9
16:57:56	1.4	0.4	261.1	-9.8	-0.6	91.8
16:58:11	14.2	0.4	77.8	-8.1	-0.5	17.2
16:58:26	17.1	1.5	24.6	-8.7	-0.5	11.9
16:58:41	18.1	2.0	17.6	-9.3	-0.6	10.8
16:58:56	19.0	2.1	14.5	-9.3	-0.6	9.4
16:59:11	18.7	2.3	13.0	-9.3	-0.6	14.7
16:59:26	19.3	2.[redacted]	26.9	-9.4	-0.6	6.3
16:59:41	21.1	2.[redacted]	15.8	-8.3	-0.6	1.6
17:00:41	6.8	0.[redacted]	161.5	-11.7	-0.7	169.8
17:01:08	-0.3	0.[redacted]	301.6	-13.2	-0.7	207.6
17:01:18	-0.3	0.0	307.2	-12.5	-0.7	207.7
17:01:28	-0.3	-0.1	308.0	-13.1	-0.7	207.7
17:01:38	-0.3	-0.1	308.8	-13.2	-0.7	207.7
17:01:48	-0.2	14.6	307.7	-12.2	-0.7	149.7
17:01:58	-0.1	32.8	243.6	-10.1	-0.6	21.6
17:02:08	-0.1	35.8	120.6	-10.0	-0.6	3.5
17:02:18	-0.2	36.2	56.9	-9.0	-0.6	1.4
17:02:27	-0.2	36.4	32.2	-9.9	-0.6	0.9
17:02:37	-0.2	36.5	20.9	-10.0	-0.6	0.8
17:02:47	-0.2	36.7	15.1	-10.0	-0.6	0.7
17:02:57	-0.2	36.8	11.7	-10.0	-0.6	0.6
17:03:07	-0.2	37.0	9.5	-10.0	-0.7	0.6

O2
CO
CO2
SO2
NOx
THC

17:03:57	-0.1	41.2	4.9	-9.6	-0.6	0.4

09-19-1991	O2	[illegible]	CO	CO2	NOI
Time	%	[illegible]	PPMV	%	PPMV
17:04:07	-0.1	41.2	4.5	-9.6	-0.6	0.4
Avg.=	4.6	13.5	99.3	18.0	2.5	63.9

CALIBRATION SUMMARY 0648
09-20-1991 07:48:45
CALIBRATION FILE NAME =D:\CEMDATA\0920CAL1.CAL

Chan.	Name	Units	Zero Conc.	Zero Resp.	Span Conc.	Span Resp.	Slope	Int.
1	O2	%	0.00	0.0006	18.00	0.076	240.373	-0.16
2	THC	PPMV	0.00	0.0025	41.00	4.084	10.045	-0.03
3	SO2	PPMV	0.00	0.0008	295.00	0.058	5177.991	-4.27
4	CO	PPMV	0.00	0.0003	474.00	0.047	10243.321	-2.62
5	CO2	%	0.00	0.0012	17.00	0.083	207.578	-0.24
6	NOX	PPMV	0.00	0.0078	201.00	2.080	96.990	-0.76
7			0.00	0.0000	0.00	0.000	1.000	0.00
8			0.00	0.0000	0.00	0.000	1.000	0.00
9			0.00	0.0000	0.00	0.000	1.000	0.00
10			0.00	0.0000	0.00	0.000	1.000	0.00
11			0.00	0.0000	0.00	0.000	1.000	0.00
12			0.00	0.0000	0.00	0.000	1.000	0.00
13			0.00	0.0000	0.00	0.000	1.000	0.00
14			0.00	0.0000	0.00	0.000	1.000	0.00

Press Shift-PrtSc to Print Out Table
Press (C) to Continue

CEM INSTRUMENT DRIFT SUMMARY
09-20-1991 07:49:03

Chan.	Name	Units	Zero Conc. Actual	Zero Conc. Observed	Span Actual	Span Observed	Drift % of Scale Zero	Drift % of Scale Span
1	O2	%	0.000	-.027	18.00	18.66	-0.11	2.65
2	THC	PPMV	0.000	1.035	41.00	41.01	1.03	0.01
3	SO2	PPMV	0.000	3.127	295.00	305.59	0.06	0.21

7	0.000	0.000	0.00	0.00	0.00	0.00
8	0.000	0.000	0.00	0.00	0.00	0.00
9	0.000	0.000	0.00	0.00	0.00	0.00
10	0.000	0.000	0.00	0.00	0.00	0.00
11	0.000	0.000	0.00	0.00	0.00	0.00
12	0.000	0.000	0.00	0.00	0.00	0.00
13	0.000	0.000	0.00	0.00	0.00	0.00
14	0.000	[redacted]	0.00	0.00	0.00	0.00

Press Shift-PrtSc to Print Out Table
Press <C> to Continue

RADIAN CORPORATION 0920CCK

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
ONALASKA,WI
Performed for: MATHY
Date Printed = 09-20-1991 Current Time = ~~07:50:33~~ 0658
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CAL1.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
~~07:51:17~~ 0651	1.0	41.1	1.6	0.0	-0.1	-1.5
07:52:17	1.8	28.6	1.9	4.5	0.1	-0.8
07:53:11	7.2	4.5	1.1	82.8	8.6	-1.7
07:53:31	7.2	4.1	0.9	96.9	10.8	-1.8
07:53:51	7.2	4.1	0.6	96.9	10.8	-1.8
07:54:11	7.3	4.0	0.6	97.0	10.7	-1.8
07:54:31	7.4	4.0	0.6	97.0	10.6	-1.9
07:55:31	7.1	2.9	4.9	95.9	10.6	14.8
07:56:01	0.1	-2.4	84.6	31.4	5.7	95.6
07:56:31	-0.3	-2.8	95.9	-1.2	0.3	97.3
07:57:01	-0.3	-2.7	94.9	-2.1	-0.1	97.5
07:57:31	-0.3	-2.9	95.3	-2.1	-0.1	97.7
07:58:01	-0.3	-3.0	95.5	-2.1	-0.1	97.8
07:58:31	-0.3	-3.0	95.8	-2.1	-0.1	98.0
07:59:01	-0.3	-3.0	95.6	-2.1	-0.1	98.1
07:59:31	-0.3	-3.0	95.7	-2.1	-0.1	98.2
08:00:01	-0.3	-3.0	95.8	-2.1	-0.1	98.3
08:00:31	-0.3	-3.0	95.9	-2.1	-0.1	98.3
08:01:01	-0.3	-3.0	95.9	-2.1	-0.1	98.5
08:01:31	-0.3	-3.2	96.0	-2.1	-0.1	98.6
08:02:00	-0.3	-3.2	96.1	-2.1	-0.1	98.7
08:02:00	-0.3	-3.4	96.0	-2.1	-0.1	98.9
08:12:00	10.0	1.1	49.2	-0.7	-0.1	46.1
08:12:57	21.1	7.0	-0.5	-0.8	-0.1	-2.5
08:13:07	21.1	9.9	-0.7	-2.6	-0.1	-2.6
08:13:17	21.1	9.9	-1.2	-2.9	-0.1	-2.6
08:13:27	21.1	9.9	-1.2	-2.9	-0.1	-2.6
08:13:37	21.1	9.9	-1.1	-2.9	-0.1	-2.6
08:13:47	21.1	9.9	-1.0	-2.9	-0.1	-2.6
08:13:57	21.1	9.8	-1.1	-2.9	-0.1	-2.6
08:14:07	21.1	9.8	-1.2	-2.9	-0.1	-2.6
08:14:17	21.1	9.8	-1.3	-2.9	-0.1	-2.6
08:14:26	21.1	9.8	-1.4	-2.9	-0.1	-2.6
08:14:56	18.8	7.1	-0.6	-1.2	-0.1	16.2
08:15:26	1.1	2.4	-0.6	0.7	-0.0	43.4
08:15:56	-0.1	1.9	-1.0	1.1	-0.0	44.0
08:16:26	-0.1	1.7	-0.8	1.2	-0.0	44.2
08:16:56	-0.1	1.6	-0.9	1.0	-0.0	44.3
08:17:06	-0.1	1.5	-0.9	0.8	-0.1	44.4
08:17:16	-0.1	1.4	-1.0	1.2	-0.0	44.4
08:17:26	-0.1	1.4	-1.0	1.2	-0.0	44.4
08:17:36	-0.2	1.4	-1.2	1.2	-0.0	44.4
08:17:46	-0.1	1.3	-1.0	0.6	-0.1	44.4
08:17:56	-0.2	1.4	-1.0	0.8	-0.0	44.4
08:18:06	-0.1	1.2	-1.0	1.2	-0.1	44.4
08:18:16	-0.1	0.1	-1.0	1.2	-0.1	44.4

O2
CO
CO2
SO2
THC
NOx

RADIAN CORPORATION 0920RUN1

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #5
ONALASKA,WI
Performed for: MATHY 0718
Date Printed = 09-20-1991 Current Time = ~~08:18:39~~
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CAL1.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
~~08:18:40~~ 0718	-0.2	0.1	-1.3	1.2	-0.1	44.5
08:19:40	-0.1	-0.0	-1.1	1.1	-0.1	44.5
08:20:40	-0.1	-0.1	-1.0	1.1	-0.1	44.5
08:21:40	-0.1	-0.2	-0.9	1.1	-0.1	44.5
08:22:40	-0.2	-0.2	-0.9	1.1	-0.0	44.5
Avg.=	-0.2	-0.1	-1.0	1.1	-0.1	44.5

NOx QC(?) (PB) 44.0 ?? 49.3

RADIAN CORPORATION 0920RUN1

TIME CHANGED

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
ONALASKA,WI
Performed for: MATHY
Date Printed = 09-20-1991 Current Time = 07:19:00
File Name = D:\CEMDATA\092091.PSN Calibration File:D:\CEMDATA\0920CAL1.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
07:19:06	-0.3	-0.3	-1.0	0.3	-0.0	-2.7
07:19:16	-0.2	-0.3	-0.9	0.2	-0.0	-2.7
07:19:26	-0.2	-0.3	-0.8	0.2	-0.0	-2.7
07:19:35	-0.2	-0.3	-1.2	0.2	-0.0	-2.7
07:19:45	-0.2	-0.3	-1.1	0.2	-0.0	-2.7
07:19:55	-0.2	-0.4	-1.0	0.2	-0.0	-2.7
07:20:05	-0.2	-0.3	-0.9	0.2	-0.0	-2.7
07:20:15	-0.2	-0.4	-0.8	0.2	-0.0	-2.7
07:20:25	-0.2	-0.3	-0.9	0.2	-0.0	-2.7
07:20:35	-0.2	-0.3	-0.8	0.2	-0.0	-2.7
07:20:45	-0.2	-0.3	-0.9	0.2	-0.0	-2.7
07:20:55	-0.2	-0.4	-0.7	0.2	-0.0	-2.7
07:21:05	-0.2	-0.3	-0.8	0.2	-0.0	-2.7
07:21:15	-0.2	-0.3	-1.0	0.2	-0.0	-2.7
07:21:25	-0.2	-0.3	-0.8	0.2	-0.0	-2.7
07:21:35	-0.2	-0.4	-0.9	0.2	-0.0	-2.7
07:21:45	-0.2	-0.4	-0.7	0.2	-0.0	-2.8
07:21:55	-0.2	-0.5	-0.5	0.2	-0.1	-2.7
07:22:05	-0.2	-0.4	-0.7	0.2	-0.0	-2.8
07:22:15	0.1	-0.2	-0.3	0.2	-0.1	-2.7
07:22:24	10.3	0.9	-0.3	0.4	-0.0	-2.6
07:22:34	18.3	1.6	0.4	0.3	-0.0	-2.6
07:22:44	20.1	1.9	0.7	0.3	-0.0	-2.6
07:22:54	20.7	2.6	0.7	0.3	0.0	-2.6
07:23:04	20.9	3.2	1.0	0.4	0.0	-2.6
07:23:14	15.9	2.0	0.5	0.3	0.0	-2.7
07:23:24	3.6	1.3	0.2	0.3	0.0	-2.7
07:23:34	1.1	1.0	-0.2	0.2	-0.0	-2.7
07:23:44	0.5	0.9	-0.2	0.3	-0.0	-2.8
07:23:54	0.3	0.7	-0.4	0.3	-0.0	-2.7
07:24:04	0.2	0.6	-0.5	0.3	-0.0	-2.7
07:24:14	0.2	0.6	-0.6	0.3	-0.0	-2.8
07:24:24	0.1	0.5	-0.4	0.3	-0.0	-2.8
07:24:34	0.1	0.5	-0.5	0.3	-0.0	-2.8
07:24:44	0.1	0.5	-0.6	0.3	-0.0	-2.8
07:24:54	0.0	0.4	-0.6	0.3	-0.0	-2.8
07:25:04	0.0	0.5	-0.5	0.3	-0.0	-2.8
07:25:13	0.0	0.4	-0.5	0.3	-0.0	-2.8
07:25:23	-0.1	1.6	-0.4	0.3	-0.0	-2.8
07:25:33	-0.0	0.2	-0.5	0.3	-0.0	-2.8
07:25:43	-0.1	0.1	-0.4	0.3	-0.0	-2.8
07:25:53	-0.0	-0.1	-0.6	0.3	-0.0	-2.8
07:26:03	-0.0	-0.1	-0.4	0.2	-0.1	-2.8
07:26:13	-0.1	-0.2	-0.1	0.2	-0.0	-2.8

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV
07:26:43	-0.2	-0.3	-0.2	0.2	-0.0	-2.8
07:26:53	-0.2	-0.3	-0.3	0.2	-0.0	-2.8
07:27:03	-0.1	-0.4	-0.1	0.2	-0.0	-2.8
07:29:03	-0.1	-0.2	-0.2	0.4	-0.0	-2.8
07:29:03	10.3	5.1	1.9	1.3	-0.0	-2.6
07:30:03	20.6	3.9	1.1	2.3	0.0	-2.6
07:31:03	20.9	3.2	0.3	1.4	0.0	-2.7
07:32:02	21.0	2.8	0.2	1.4	0.0	-2.7
07:33:02	21.0	2.5	-0.0	1.4	0.0	-2.8
07:34:02	21.0	3.0	0.0	1.4	0.0	-2.8
07:35:02	21.3	22.5	-0.3	-1.0	-0.0	-2.7
07:36:02	12.2	1.4	-0.2	16.4	0.5	0.3
07:37:02	0.4	4.7	-0.4	1.1	0.0	-2.6
07:38:02	8.8	24.6	-0.6	450.3	2.0	25.7
07:39:02	16.4	19.0	-0.5	406.8	3.7	17.4
07:40:02	14.4	39.5	-0.7	566.3	3.7	29.7
07:41:02	12.4	49.7	-0.4	1022.8	6.0	38.1
07:42:02	13.0	38.9	-0.5	1018.5	5.1	33.9
07:43:02	14.6	47.3	-0.6	954.3	4.7	27.0
07:44:02	12.8	64.4	-0.4	1001.9	5.4	[illegible]
07:45:01	12.0	58.1	-0.4	1023.9	6.6	40.1
07:46:01	13.5	46.4	-0.7	1015.9	5.9	32.5
07:47:01	13.3	75.7	-0.2	1010.6	5.4	34.1
07:48:01	12.2	72.5	-0.4	1026.1	6.6	[illegible]
07:49:01	12.6	62.2	-0.6	1024.5	6.7	[illegible]
07:50:01	14.6	79.8	-0.3	1030.3	5.1	[illegible]
07:51:01	15.0	96.5	2.6	1035.1	5.0	[illegible]
07:52:01	12.6	100.4	4.9	1047.1	6.3	[illegible]
07:53:01	[illegible]					
07:54:01	[illegible]					
07:55:01	[illegible]					
07:56:01	[illegible]					
07:57:01	[illegible]					
07:58:00	[illegible]					
07:59:01	[illegible]					
08:00:01	[illegible]					
[illegible]	[illegible]					
[illegible]	[illegible]					
[illegible]

FAIRBAN DISPERSION 0920RUN2

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MATHY #5
CHALASKA,WI
Performed for: MATHY
Date Printed = 09-20-1991 Current Time = 08:33:19
File Name = Drv:\EMDATA.PRO\91.PRN Calibration File:D: \EMDATA\091220A1.CAL

09-20-1991 Time	O2 %	CO ppm	SO2 ppm	CO ppm	CO2 %	NOx ppm								
08:33:14	12.3	42.6	-0.4	1022.4	6.3	37.3	-3.9	-4.1	-4.7	-4.7	23.6	23.6	23.5	24.3	3
08:34:14	12.5	39.9	-0.4	1022.8	6.5	38.3	-4.0	-4.1	-4.7	-4.7	23.6	23.5	23.5	24.3	3
08:35:14	12.6	37.0	0.2	1026.0	6.5	39.0	-4.0	-4.1	-4.7	-4.6	23.5	23.5	23.5	24.3	3
08:36:16	12.4	71.4	0.6	1027.0	6.7	39.5	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	3
08:37:16	13.1	77.0	1.0	1028.6	6.6	35.3	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:38:16	12.5	62.9	0.2	1025.2	6.1	44.2	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	3
08:39:16	12.3	64.9	0.7	1025.9	6.8	43.8	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	3
08:40:15	12.4	78.6	1.5	1029.4	6.8	43.9	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
08:41:15	12.1	76.5	1.4	1028.7	7.0	44.7	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:42:15	12.3	72.2	1.0	1027.3	6.9	43.6	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:43:15	12.3	74.0	1.2	1027.9	6.8	44.2	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:44:15	12.1	55.2	0.1	1023.9	6.8	43.8	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:45:15	12.3	62.2	0.4	1025.2	6.8	43.3	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	3
08:46:15	12.5	71.5	0.8	1027.0	6.7	42.9	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:47:15	12.4	71.6	0.9	1027.2	6.7	42.7	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
08:48:15	12.3	56.4	0.3	1024.1	6.7	43.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:49:15	14.0	90.5	4.9	1042.1	7.1	35.1	-3.9	-4.0	-4.8	-4.5	23.4	23.5	23.5	24.3	3
08:50:15	16.9	52.8	0.3	1023.9	3.2	7.1	-4.3	-4.3	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:51:15	20.6	27.5	-0.3	1021.1	0.7	-0.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:52:15	20.8	18.6	-0.5	1020.7	0.3	-1.8	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:53:15	20.9	13.2	-0.5	1020.8	0.1	-2.5	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:54:15	21.0	10.6	-0.6	1021.0	0.1	-2.7	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:55:15	21.0	10.4	-0.9	1020.7	0.0	-2.8	-4.1	-4.1	-4.7	-4.7	23.6	23.5	23.5	24.3	3
08:56:14	20.9	8.5	-0.7	1022.7	0.1	-2.8	-4.1	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:57:14	20.9	7.2	-0.7	1023.1	0.1	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:58:14	20.9	5.7	-0.7	1023.1	0.1	-2.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
08:59:14	20.9	5.5	-0.9	1023.2	0.0	-2.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:00:14	20.9	4.7	-1.0	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:01:14	20.9	4.5	-0.9	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:02:14	20.9	4.5	-1.2	1023.2	0.0	-2.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:03:14	20.9	4.5	-1.1	1023.2	0.0	-2.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:04:14	20.9	4.5	-1.3	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:05:14	20.9	4.6	-1.3	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:06:14	20.9	4.4	-1.3	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:07:14	20.9	4.0	-1.5	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:08:14	20.9	4.1	-1.6	1023.2	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:09:14	20.9	3.9	-1.7	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:10:14	20.9	3.5	-1.9	977.0	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:11:14	20.9	3.4	-2.0	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:12:13	20.9	3.2	-2.0	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:13:13	20.9	3.2	-2.0	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:14:13	20.9	3.2	-2.0	1023.1	0.0	-3.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:15:13	20.9	2.9	-2.1	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:16:13	20.9	2.7	-2.1	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
09:17:13	20.9	2.8	-2.0	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV								
09:19:13	20.9	2.[illegible]	-2.0	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:20:13	20.9	2.[illegible]	-1.9	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:21:13	20.9	2.[illegible]	-2.0	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:22:13	20.9	2.5	-2.0	1023.1	0.0	-3.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:23:13	14.6	2.5	-2.0	1023.1	2.4	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:24:13	17.3	2.6	-2.2	1023.2	15.8	-3.2	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:25:13	17.9	2.5	-2.0	1023.2	17.1	-3.2	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:26:13	17.9	2.5	-2.0	1023.2	17.2	-3.2	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3
09:47:25	17.9	2.4	-1.9	1023.2	17.2	-3.2	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3

CEM INSTRUMENT DRIFT SUMMARY
09-20-1991 10:07:04

Chan.	Name	Units	Zero Conc. Actual	Zero Conc. Observed	Span Actual	Span Observed	Drift % of Scale Zero	Drift % of Scale Span
1	O2	%	0.000	0.000	18.00	0.00	0.00	-72.00
2	THC	PPMV	0.000	0.000	41.00	0.00	0.00	-41.00
3	SO2	PPMV	0.000	0.000	295.00	0.00	0.00	-5.90
4	CO	PPMV	0.000	0.000	474.00	470.04	0.00	-0.20
5	CO2	%	0.000	0.000	17.00	0.00	0.00	-85.00
6	NOX	PPMV	0.000	0.000	201.00	0.00	0.00	-20.10
7			0.000	0.000	0.00	0.00	0.00	0.00
8			0.000	0.000	0.00	0.00	0.00	0.00
9			0.000	0.000	0.00	0.00	0.00	0.00
10			0.000	0.000	0.00	0.00	0.00	0.00
11			0.000	0.000	0.00	0.00	0.00	0.00
12			0.000	0.000	0.00	0.00	0.00	0.00
13			0.000	0.000	0.00	0.00	0.00	0.00
14			0.000	0.000	0.00	0.00	0.00	0.00

Press Shift-PrtSc to Print Out Table
Press (C) to Continue

CALIBRATION SUMMARY
09-20-1991 10:07:27
CALIBRATION FILE NAME =D:\CEMDATA\0920CALA.CAL

Chan.	Name	Units	Zero Conc.	Zero Resp.	Span Conc.	Span Resp.	Slope	Int.
1	O2	%	0.00	0.0006	18.00	0.076	240.373	-0.16
2	THC	PPMV	0.00	0.0025	41.00	4.084	10.045	-0.03
3	SO2	PPMV	0.00	0.0008	295.00	0.058	5177.990	-4.27
4	CO	PPMV	0.00	0.0003	474.00	0.010	50405.125	-12.91
5	CO2	%	0.00	0.0012	17.00	0.083	207.578	-0.24

10	0.00	0.0000	0.00	0.000	1.000	0.00
11	0.00	0.0000	0.00	0.000	1.000	0.00
12	0.00	0.0000	0.00	0.000	1.000	0.00
13	0.00	0.0000	0.00	0.000	1.000	0.00
14	0.00	0.0000	0.00	0.000	1.000	0.00

Press Shift-PrtSc to Print Out Table
Press <C> to Continue

Avg.=		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0		

RADIAN CORPORATION 0920RUN2

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MARTY #6
ONALASKA,WI
Performed for: MARTY
Date Printed = 09-20-1991 Current Time = 10:08:59
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CALA.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV								
10:09:01	18.2	0.2	-0.6	475.8	16.8	-3.2	-4.0	-4.0	-4.7	-4.8	23.6	23.6	23.5	24.3
10:10:00	18.7	27.7	-0.4	380.8	12.6	6.6	-4.0	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3
10:11:00	14.0	42.3	-0.6	1034.5	4.5	31.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:12:00	14.0	37.4	-0.5	1235.0	5.3	31.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:13:00	14.2	45.9	-0.3	1117.6	5.0	30.5	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:14:00	14.1	46.3	-0.4	1179.4	5.1	31.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:15:00	13.9	53.0	-0.5	1281.8	5.4	33.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:16:00	13.9	52.6	-0.2	1289.9	5.4	33.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:17:00	13.9	60.6	-0.3	1296.4	5.4	33.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:18:00	14.1	63.1	-0.1	1139.3	5.3	32.6	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:19:00	13.8	60.9	-0.3	1277.9	5.4	33.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:20:00	14.2	50.0	-0.5	1220.8	5.2	29.8	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:21:00	14.4	62.9	-0.3	1074.6	5.0	31.9	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:22:00	14.1	62.0	-0.4	1082.6	5.2	32.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:23:00	13.9	61.8	-0.5	1146.2	5.4	33.9	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:24:00	14.0	47.9	-0.7	1153.2	5.3	31.5	-4.2	-4.2	-4.7	-4.7	23.5	23.6	23.5	24.3
10:24:59	14.2	53.6	-0.7	1047.8	5.1	31.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:25:59	14.1	54.6	-0.8	999.1	5.2	32.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:26:59	14.0	52.3	-0.9	1058.3	5.3	32.7	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:27:59	14.3	66.7	-0.4	1076.2	5.2	31.1	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:28:59	14.5	66.9	-0.6	1144.9	5.0	30.1	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:29:59	14.4	55.2	-0.8	1150.4	5.0	30.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:30:59	14.2	57.2	-0.7	983.5	5.2	32.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:31:59	14.3	54.4	-0.6	1018.7	5.1	30.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:32:59	14.4	51.7	-0.8	1050.1	5.1	30.8	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:33:59	14.1	55.2	-0.7	1108.4	5.2	32.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:34:59	14.0	57.3	-0.7	1421.8	5.4	32.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:35:59	14.3	57.9	-0.6	1213.3	5.2	31.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:36:59	14.1	47.5	-0.8	1226.2	5.2	32.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:37:59	14.0	39.8	-0.9	1385.0	5.3	31.5	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:38:58	14.3	34.6	-1.0	1213.7	5.0	30.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:39:58	14.4	46.8	-0.9	1032.5	5.0	29.8	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:40:58	14.6	54.0	-0.9	1209.8	4.9	28.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:41:58	14.7	57.6	-1.0	1246.9	4.8	28.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:42:58	14.5	51.3	-0.9	1116.7	4.9	30.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:43:58	14.6	67.7	-0.8	1065.8	5.0	29.6	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:44:58	14.6	69.9	-0.7	1156.1	5.0	29.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:45:58	14.6	66.7	-0.6	1124.2	4.9	29.3	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:46:58	14.6	50.6	-1.2	1108.3	4.9	29.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:47:58	14.6	44.7	-1.1	1153.1	4.8	29.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:48:58	14.5	63.3	-1.0	1058.4	5.0	30.7	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:49:58	14.6	68.6	-0.9	1094.8	5.0	30.4	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:50:58	14.6	69.3	-0.9	1126.1	5.0	30.6	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3
10:51:58	14.5	59.1	-1.3	1164.0	5.0	30.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3
10:52:57	14.5	57.3	-1.3	1141.4	4.8	31.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
10:54:57	14.5	58.[illegible]	-1.3	1126.1	5.0	31.2	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
10:55:57	14.4	62.[illegible]	-1.1	1165.1	5.1	31.1	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
10:56:57	14.4	68.[illegible]	-1.0	1247.4	5.2	31.7	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
10:57:57	14.1	72.[illegible]	-0.7	1591.6	5.4	32.7	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
10:58:57	14.2	69.0	-0.8	1623.1	5.4	32.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
10:59:57	14.3	56.4	-1.3	1310.2	5.2	31.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:00:57	14.1	56.7	-1.3	1443.2	5.2	32.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:01:57	14.3	67.1	-0.9	1408.1	5.2	31.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:02:57	14.2	73.1	-0.5	1376.5	5.3	32.6	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:03:57	14.0	66.0	-1.0	1774.8	5.4	32.9	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:04:57	14.3	64.0	-1.2	1385.6	5.3	31.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:05:57	14.5	54.5	-1.6	1248.9	5.0	30.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:06:57	14.5	46.0	-1.8	1210.9	4.9	31.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:07:56	14.3	60.9	-1.1	1229.9	5.1	32.5	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:08:56	14.3	53.0	-1.4	1278.5	5.2	31.5	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:09:56	14.5	46.9	-1.2	1163.8	5.1	30.8	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:10:56	14.5	47.1	-1.4	1160.9	5.0	31.2	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:11:56	14.4	63.4	-1.4	1104.4	5.1	31.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:12:56	14.4	47.1	-2.0	1113.9	5.1	31.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:13:56	14.3	41.3	-1.9	1148.8	5.1	31.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:14:56	14.3	40.0	-2.0	1144.7	5.1	31.5	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:15:56	14.4	56.8	-1.8	1163.8	5.1	31.8	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:16:56	14.3	70.3	-1.4	1246.7	5.2	32.6	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:17:56	14.2	75.7	-1.0	1470.9	5.4	32.9	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:18:56	14.1	64.6	-1.6	1449.1	5.4	32.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:19:56	14.4	56.0	-1.8	1265.1	5.2	31.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:20:56	14.5	66.2	-1.6	1256.9	5.0	31.1	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:21:55	14.4	59.4	-2.0	1167.8	5.1	32.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:22:55	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:23:55	14.3	73.5	-0.8	1247.9	5.2	31.8	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:24:55	14.5	77.1	-1.0	1234.5	5.2	32.7	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:25:55	14.3	65.9	-1.6	1366.1	5.1	33.1	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:26:55	14.2	77.1	-0.9	1549.9	5.3	33.3	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:27:55	14.3	75.4	-1.1	1551.2	5.3	33.3	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:28:55	14.3	72.6	-1.5	1448.6	5.3	32.4	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:29:55	14.3	62.2	-2.0	1371.5	5.1	32.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:30:55	14.2	68.6	-1.6	1495.8	5.2	33.0	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:31:55	14.1	55.3	-1.8	1514.8	5.2	32.8	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:32:55	14.3	63.1	-1.9	1465.6	5.2	32.4	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:33:55	14.6	73.[illegible]	-1.3	1278.8	5.1	30.7	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:34:55	14.7	67.[illegible]	-1.7	1282.3	4.9	30.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:35:55	14.3	65.[illegible]	-1.7	1279.2	5.1	32.4	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:36:54	14.2	64.4	-2.0	1448.2	5.2	31.8	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:37:54	14.8	66.5	-1.7	1417.5	4.7	29.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:38:54	13.4	66.0	-1.7	2158.9	5.7	34.1	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:39:54	13.6	60.6	-1.8	2053.0	5.7	34.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:40:54	13.9	60.1	-1.7	1593.4	5.6	32.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:41:54	14.0	50.3	-1.9	1415.6	5.3	32.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:42:54	13.7	49.0	-2.0	1779.7	5.5	33.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:43:54	13.6	60.5	-1.9	2103.5	5.7	33.9	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:44:54	13.8	69.2	-1.6	2052.8	5.7	33.7	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:45:54	13.6	57.7	-1.9	2011.9	5.6	33.5	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:46:54	13.8	83.0	-0.1	2138.3	5.8	33.8	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:47:54	13.4	79.3	-0.5	2808.7	5.9	34.3	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
11:48:54	13.7	69.0	-1.5	2233.7	5.7	33.7	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
11:50:53	13.7	66.4	-1.7	1828.5	5.6	33.7	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:51:53	13.4	52.[illegible]	-2.1	2251.3	5.7	33.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:52:53	13.7	62.[illegible]	-1.9	1986.2	5.7	33.5	-4.1	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:53:53	13.8	68.[illegible]	-1.6	1697.6	5.6	33.6	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
11:54:53	13.8	64.0	-1.9	1580.3	5.6	33.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:55:53	13.8	63.9	-1.8	1628.1	5.6	32.8	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:56:53	13.8	66.3	-1.7	1640.7	5.6	33.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
11:57:53	13.7	55.1	-2.0	1571.9	5.6	33.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:58:53	13.9	50.7	-1.9	1503.7	5.5	33.1	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
11:59:53	14.1	49.0	-2.0	1331.3	5.3	30.5	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:00:53	14.5	66.7	-1.6	1357.8	5.1	29.6	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:01:53	14.5	71.6	-1.5	1345.3	5.1	30.7	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:02:53	14.2	70.6	-1.5	1190.6	5.3	32.2	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:03:53	14.3	74.8	-1.1	1217.6	5.4	31.0	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:04:53	14.4	54.5	-2.0	1441.4	5.1	29.6	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:05:52	14.1	50.0	-2.0	1271.6	5.1	32.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:06:52	14.1	66.8	-1.7	1213.0	5.4	32.9	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:07:52	14.0	68.4	-1.4	1285.4	5.5	33.2	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:08:52	14.0	63.9	-1.5	1284.8	5.5	33.0	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:09:52	14.1	48.4	-1.9	1188.5	5.3	31.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:10:52	14.1	42.5	-1.8	1218.9	5.2	31.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:11:52	14.1	55.1	-1.7	1266.1	5.3	31.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:12:52	14.1	48.8	-1.8	1251.8	5.3	31.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:13:52	14.3	69.6	-1.2	1277.0	5.3	30.7	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:14:52	14.4	84.7	0.3	1262.8	5.4	31.9	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:15:52	14.3	95.2	1.8	1454.5	5.7	33.1	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:16:52	14.5	91.8	1.2	1474.0	5.5	31.9	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:17:52	14.6	91.5	1.1	1420.6	5.5	31.8	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:18:52	14.5	79.3	-0.5	1376.3	5.2	32.3	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:19:52	14.7	91.2	1.2	1290.5	5.4	32.9	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:20:52	14.7	98.9	2.1	1292.5	5.6	33.9	-4.1	-4.1	-4.8	-4.5	23.4	23.5	23.5	24.3	32
12:21:52	14.6	94.5	1.5	1314.8	5.6	33.4	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:22:52	14.7	88.5	0.6	1285.4	5.3	31.4	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:23:52	14.5	70.0	-1.3	1303.4	5.1	30.9	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:24:52	14.6	77.8	-0.6	1304.7	5.2	31.1	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:25:52	14.8	91.6	1.1	1355.0	5.3	31.7	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.4	32
12:26:52	15.1	100.4	3.7	1330.9	5.8	34.5	-4.0	-4.0	-4.8	-4.5	23.4	23.5	23.5	24.3	32
12:27:52	15.4	100.4	5.4	1483.2	6.2	37.0	-3.9	-4.0	-4.8	-4.4	23.4	23.5	23.5	24.3	32
12:28:52	14.9	100.4	4.6	1723.5	6.2	37.8	-4.0	-4.0	-4.8	-4.5	23.4	23.5	23.5	24.3	32
12:29:52	14.7	100.4	3.0	1529.8	5.8	36.2	-4.1	-4.1	-4.8	-4.5	23.4	23.5	23.5	24.3	32
12:30:52	14.6	92.4	1.9	1376.2	5.5	34.8	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:31:52	14.4	73.[illegible]	-0.6	1336.4	5.3	33.6	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:32:52	14.5	76.0	-0.3	1363.1	5.2	32.2	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:33:52	14.8	92.7	1.5	1461.5	5.3	31.1	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:34:52	14.8	91.3	1.3	1432.5	5.2	32.2	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:35:52	14.7	97.6	2.2	1318.2	5.5	34.6	-4.1	-4.1	-4.8	-4.5	23.4	23.5	23.5	24.3	32
12:36:52	14.3	78.1	-0.2	1390.0	5.4	34.8	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:37:50	14.3	66.3	-0.8	1364.5	5.3	34.3	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
Avg.=	14.3	64.0	-0.8	1346.4	5.4	31.7	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32

RADIAN CORPORATION 0920QCCK

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MAHTY #6
GNALASKA,MI
Performed for: MATHY
Date Printed = 09-20-1991 Current Time = 12:38:28
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CALA.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
12:38:50	14.1	22.0	-1.4	1235.3	5.9	22.6	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:39:50	17.5	5.9	-1.6	578.4	15.2	-1.8	-4.1	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:40:23	17.7	3.8	-1.5	466.8	16.8	-2.5	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:40:33	17.8	3.3	-1.5	463.6	17.0	-2.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:40:43	17.8	3.2	-1.4	463.2	17.0	-2.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:40:53	17.8	3.0	-1.3	463.3	17.0	-2.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:03	17.7	2.8	-1.4	463.2	17.0	-2.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:13	17.7	2.7	-1.3	463.2	17.0	-2.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:23	17.7	2.6	-1.2	463.2	17.0	-2.7	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:33	17.8	2.4	-1.4	463.1	17.0	-2.7	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:43	17.7	2.4	-1.4	463.3	17.0	-2.7	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:41:53	17.8	2.2	-1.3	463.2	17.0	-2.7	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:42:53	10.8	2.5	46.2	397.9	15.5	112.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:43:53	0.3	2.2	258.4	39.6	2.1	212.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:44:03	0.0	2.0	281.0	0.5	0.3	211.0	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.4	24.3	3
12:44:13	0.0	1.9	283.1	-4.1	0.3	211.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:44:23	-0.0	1.8	285.3	-4.1	0.2	211.7	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:44:33	-0.0	1.8	287.6	-4.1	0.2	211.7	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:44:43	-0.0	1.8	289.3	-4.0	0.1	211.9	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:44:53	-0.0	1.7	290.4	-4.0	0.1	211.8	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:45:03	-0.1	1.7	291.5	-4.0	0.1	212.1	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:46:03	0.3	39.1	237.5	-6.0	0.1	118.6	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	3
12:46:13	1.0	41.4	67.3	-5.3	0.2	2.5	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:46:23	1.0	41.5	36.5	-4.8	0.1	-0.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:46:33	1.0	41.6	26.1	-5.3	0.1	-0.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:46:43	1.0	41.6	19.6	-5.2	0.0	-1.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:46:53	1.0	41.7	14.9	-5.3	0.0	-1.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
12:47:03	1.0	41.8	12.2	-8.9	0.0	-1.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
Avg.=	3.1	12.4	96.7	243.7	7.5	68.5	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	3

O2
CO
CO2
SO2
NOX
THC

RADIAN CORPORATION 0920RUN3

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MARTY #6
ONALASKA,WI
Performed for: MARTY
Date Printed = 09-20-1991 Current Time = 12:47:38
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CALA.CAL

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV								
12:48:08	1.0	41.9	6.5	-6.5	0.0	-1.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:49:08	4.0	70.1	6.8	103.2	0.3	9.3	-4.1	-4.1	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:50:08	13.9	76.2	8.1	1115.2	4.4	29.1	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:51:08	14.6	62.3	2.3	1329.5	4.9	29.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
12:52:08	14.8	70.1	1.3	1238.7	4.9	29.5	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
12:53:08	14.7	65.6	0.1	1129.5	5.0	30.5	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
12:54:08	14.9	73.2	1.3	1096.0	5.2	30.4	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
12:55:08	17.3	15.7	-1.1	877.5	4.0	8.8	-4.2	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:00:08	21.3	34.7	-1.6	8.5	0.1	-2.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:05:08	21.1	16.0	-2.6	-14.4	-0.0	-2.7	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:10:07	21.0	9.3	-2.7	-9.8	0.0	-2.7	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:15:07	5.1	4.3	-2.6	-5.9	-0.0	-2.7	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:20:07	-0.2	1.6	-2.8	-7.0	-0.0	-2.9	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:25:07	19.3	6.3	-2.5	-3.2	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:30:07	21.3	8.2	-2.6	-6.1	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:32:59	16.4	53.0	-1.2	1239.0	3.1	19.6	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:33:59	17.6	80.6	0.2	601.7	3.6	15.0	-4.1	-4.1	-4.7	-4.6	23.5	23.5	23.5	24.3	32
13:34:59	15.0	100.4	5.2	363.5	4.9	28.9	-3.8	-3.9	-4.8	-4.4	23.4	23.5	23.5	24.3	32
13:35:59	12.7	95.7	5.5	2139.2	7.0	33.4	-4.0	-4.1	-4.8	-4.5	23.4	23.5	23.5	24.3	32
13:36:59	13.3	66.6	-1.6	3672.4	6.1	32.1	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
13:37:59	13.9	67.5	-1.5	1586.7	5.5	31.0	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
13:38:59	13.9	76.2	-0.9	1409.9	5.6	32.7	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
13:39:59	14.0	97.6	0.5	1412.2	5.8	32.1	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
13:40:59	14.1	87.2	0.2	1571.1	5.6	31.2	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
13:41:59	13.8	64.5	-1.4	1521.5	5.4	32.4	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
13:42:59	14.0	82.1	0.7	1410.9	5.8	32.8	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
13:43:59	18.6	19.3	-1.8	811.4	3.6	4.9	-4.3	-4.2	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:44:59	20.8	11.1	-1.7	23.5	0.2	-2.1	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:45:59	20.9	11.8	-1.8	0.5	0.1	-2.5	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:46:59	20.9	12.3	-1.9	-1.6	0.0	-2.5	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:47:59	20.9	10.6	-1.9	-6.5	0.0	-2.7	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:48:59	20.9	8.8	-1.7	-3.6	0.0	-2.8	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:49:59	20.9	7.8	-1.7	-0.9	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:50:59	20.9	6.9	-1.5	-0.8	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:51:59	20.9	6.7	-1.4	-1.6	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:52:59	20.9	9.4	-1.5	-5.4	0.0	-2.9	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:53:59	21.0	11.0	-1.6	-11.5	-0.0	-2.9	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:54:59	21.0	11.3	-1.5	-11.5	-0.0	-2.9	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:55:59	20.9	10.0	-1.3	-12.6	-0.0	-2.9	-4.1	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32
13:56:57	21.2	8.9	-1.4	-5.6	0.0	-2.9	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
13:57:57	22.5	7.9	-1.5	-3.0	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
13:58:57	21.0	7.2	-1.2	-3.6	0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32

This was pending all cal. checks, see previous page.

Plant Down

PLANT COMING ON LINE 1330

PLANT DOWN

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
14:03:57	0.7	2.[illegible]	-0.7	-4.1	-0.0	-2.9	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
14:04:57	18.4	5.[illegible]	-0.7	-5.3	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
14:05:57	20.7	5.[illegible]	-0.7	-5.3	0.0	-2.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
14:06:57	20.9	6.[illegible]	-0.6	-6.1	0.0	-2.9	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
14:07:57	20.9	18.4	-0.8	-8.7	-0.0	-1.6	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
14:08:57	19.7	39.9	-0.3	10.7	1.0	3.4	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
14:09:57	20.9	14.7	-0.8	-8.7	0.2	-2.6	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
14:10:56	20.0	16.7	-0.8	-1.7	0.2	3.5	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:11:56	15.7	99.8	6.0	314.7	4.0	27.7	-3.7	-3.9	-4.8	-4.5	23.4	23.5	23.5	24.3	32
14:12:56	12.6	83.4	2.9	3101.0	6.7	32.6	-4.1	-4.1	-4.8	-4.5	23.4	23.5	23.5	24.3	32
14:13:56	13.8	48.3	-0.3	2708.6	5.8	27.4	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:14:56	14.1	46.1	-0.7	1209.2	4.9	29.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:15:56	13.5	65.0	-0.3	1389.1	5.6	32.7	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
14:16:56	13.4	60.3	-0.4	1543.1	5.7	32.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:17:56	13.9	74.7	0.2	1614.3	5.6	30.0	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:18:56	14.0	66.4	0.0	1559.6	5.4	30.6	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
14:19:56	13.9	74.1	0.5	1508.6	5.5	32.5	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:20:56	13.8	76.2	0.5	1515.3	5.7	32.5	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:21:56	14.1	86.1	1.7	1572.9	5.6	30.1	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:22:56	14.2	90.3	2.2	1707.6	5.6	29.9	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:23:55	14.1	81.7	1.3	1735.6	5.5	30.4	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:24:55	14.1	83.7	1.4	1639.9	5.5	31.3	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:25:55	14.1	86.1	1.7	1610.5	5.6	30.9	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:26:55	14.2	85.1	1.6	1703.5	5.5	30.1	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:27:55	14.2	77.7	0.8	1688.8	5.3	30.5	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:28:55	13.8	62.3	-0.1	1578.2	5.4	32.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:29:55	14.1	75.3	0.6	1615.5	5.5	29.8	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32
14:30:55	14.4	84.8	1.6	1576.2	5.3	29.0	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:31:55	14.4	78.8	0.7	1499.7	5.2	29.1	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:32:55	14.2	77.7	0.9	1363.3	5.3	30.6	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:33:55	14.4	64.2	-0.1	1362.7	5.0	28.3	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
14:34:55	14.5	64.6	-0.2	1370.5	5.0	27.3	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
14:35:55	14.6	80.0	1.0	1363.4	5.0	27.2	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32
14:36:55	14.4	63.3	-0.2	1339.8	4.9	28.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32
14:37:55	14.2	66.7	-0.1	1381.3	5.2	28.8	-4.2	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	32
14:38:55	14.4	80.3	0.9	1498.3	5.2	28.8	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:39:54	14.1	81.6	1.1	1466.4	5.4	31.5	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:40:54	14.2	85.2	1.8	1404.7	5.6	31.2	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:41:54	14.4	80.1	1.0	1609.6	5.3	29.3	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:42:54	14.1	61.[illegible]	-0.4	1616.1	5.2	30.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:43:54	14.0	57.[illegible]	-0.7	1461.8	5.3	31.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:44:54	13.7	60.[illegible]	-0.4	1462.1	5.4	32.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:45:54	14.0	80.1	0.7	1410.1	5.6	33.2	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:46:54	14.0	86.3	1.5	1482.1	5.7	32.5	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:47:54	14.1	85.0	1.2	1616.6	5.5	31.5	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:48:54	13.8	76.0	0.4	1544.3	5.6	33.2	-4.2	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:49:54	13.9	62.2	-0.7	1594.3	5.5	31.0	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:50:54	13.9	57.8	-0.8	1650.3	5.3	30.2	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:51:54	13.9	54.8	-0.6	1733.5	5.3	30.1	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:52:54	14.0	54.1	-0.7	1732.9	5.3	29.9	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:53:54	14.1	70.4	0.1	1739.1	5.3	29.8	-4.1	-4.2	-4.7	-4.6	23.5	23.5	23.5	24.3	3
14:54:53	14.0	60.2	-0.7	1757.3	5.2	30.3	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	3
14:55:53	13.9	73.0	-0.1	1581.9	5.5	32.5	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	3
14:56:53	13.9	83.3	1.0	1525.2	5.8	32.2	-4.1	-4.1	-4.8	-4.6	23.4	23.5	23.5	24.3	3
14:57:53	14.2	83.4	0.8	1655.4	5.5	30.7	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	3

post test THC QC/SPAN/zero check

09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
14:59:53	13.9	72.8	-0.1	1584.6	5.6	32.8	-4.1	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32.
15:00:53	14.0	81.0	0.7	1568.5	5.7	33.0	-4.1	-4.2	-4.8	-4.6	23.4	23.5	23.5	24.3	32.
15:01:53	13.4	71.3	0.3	1572.6	5.7	34.4	-4.2	-4.2	-4.7	-4.6	23.4	23.5	23.5	24.3	32.
15:02:53	2.1	47.9	-0.7	681.1	2.9	-0.5	-4.3	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:03:56	1.1	43.5	-0.7	9.7	0.0	-2.4	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:06	1.1	41.1	-0.4	0.0	0.0	-2.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:16	1.0	41.2	-0.6	-0.5	-0.0	-2.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:26	1.0	41.2	-0.7	-0.5	-0.0	-2.6	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:36	1.0	41.1	-0.8	-0.5	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:46	1.0	41.2	-0.7	-2.8	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:04:55	1.0	41.3	-0.7	-5.3	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:05:05	1.0	41.3	-0.6	-5.4	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:05:15	1.0	41.3	-0.9	-5.5	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:05:25	1.0	41.2	-0.8	-5.3	-0.0	-2.7	-4.2	-4.2	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:06:25	10.3	11.0	-0.8	-3.2	-0.0	-2.6	-4.2	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:07:18	20.5	7.7	-0.6	-4.2	-0.0	-2.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:07:28	20.7	9.3	-0.7	-5.0	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:07:38	20.7	9.4	-0.5	-13.7	-0.1	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:07:48	20.7	9.4	-0.8	-16.0	-0.1	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:07:58	20.7	9.4	-0.7	-15.0	-0.1	-2.8	-4.0	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32.
15:08:08	20.7	9.4	-1.0	-16.2	-0.1	-2.8	-4.0	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32.
15:08:18	20.7	9.3	-0.6	-16.1	-0.1	-2.8	-4.0	-4.1	-4.7	-4.8	23.6	23.5	23.5	24.3	32.
15:08:29	20.7	4.5	-0.8	-9.5	-0.0	-2.8	-4.1	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:08:38	20.5	-1.1	-0.6	-6.2	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:08:48	14.2	-1.1	-0.7	-6.4	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:08:58	3.4	-1.2	-0.7	-6.1	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:09:08	0.3	-0.9	-0.7	-5.7	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:09:18	0.2	-1.0	-0.6	-6.1	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:09:28	0.1	-1.0	-0.7	-6.0	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:09:38	-0.0	-1.0	-0.7	-6.1	-0.0	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:09:48	-0.0	-0.7	-0.6	-6.2	-0.0	-2.8	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:10:48	1.0	-0.9	-0.7	536.0	1.8	31.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:11:47	11.9	-0.9	-0.7	1815.1	6.3	40.3	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:12:47	12.0	-0.9	-0.5	2071.5	6.3	40.5	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:13:47	11.8	-0.9	-0.4	1810.5	6.5	42.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:14:47	11.6	-0.7	-0.5	1763.7	6.7	43.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:15:47	12.0	-1.2	-0.4	1603.3	6.5	41.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:16:47	12.1	-2.9	-0.2	1526.3	6.2	39.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:17:47	12.1	-3.0	-0.4	1549.5	6.2	40.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:18:47	12.1	-2.9	-0.5	1412.5	6.2	40.7	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:19:47	12.2	-3.0	-0.3	1458.7	6.2	39.3	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:20:47	12.3	-3.0	-0.4	1542.9	6.1	38.8	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:21:47	12.1	-3.0	-0.3	1555.7	6.2	40.6	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:22:47	12.1	-3.0	-0.2	1545.7	6.4	40.4	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:23:47	12.1	-2.9	-0.3	1635.3	6.3	40.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:24:47	12.1	-3.0	-0.1	1614.2	6.3	39.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:25:47	12.3	-2.9	-0.2	1642.3	6.0	37.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:26:47	12.2	-2.9	-0.1	1645.3	6.0	39.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:27:46	12.2	-2.9	0.1	1471.4	6.3	39.0	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:28:46	12.4	-3.0	0.1	1608.7	6.0	38.5	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:29:46	12.3	-2.9	-0.0	1528.3	6.1	38.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:30:46	12.3	-2.9	-0.0	1467.0	6.1	39.1	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
15:31:46	11.9	-2.9	-0.0	1637.8	6.3	41.3	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:32:46	12.1	-3.0	-0.1	2324.6	6.4	38.3	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:33:46	11.8	-2.9	-0.2	2290.4	6.3	41.1	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:34:46	11.6	-2.9	-0.2	2284.8	6.7	41.4	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.

THC SP

THC QC

THC ZERO

CAL GAS

ALLEN GAS #3

09-20-1991	O2	THC	SO2	CO	CO2	NOX									
Time	%	PPMV	PPMV	PPMV	%	PPMV									
5:35:46	11.8	[illegible]	[illegible]	2433.2	6.5	40.0	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
5:36:46	11.8	[illegible]	[illegible]	2423.5	6.5	40.3	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
5:37:46	11.7	[illegible]	[illegible]	2428.5	6.5	41.1	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
5:38:46	11.6	[illegible]	[illegible]	2425.0	6.9	42.9	-4.0	-4.0	-4.7	-4.7	23.5	23.5	23.5	24.3	32.
5:39:46	9.2	-2.9	0.1	1796.1	7.2	12.5	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
Avg.=	13.6	36.2	-0.1	971.4	3.4	18.9	-4.1	-4.1	-4.7	-4.7	23.5	23.5	23.5	24.3	32.

RADIAN CORPORATION 09209CCK

Field Testing and Process Engineering Dept.
Continuous Emissions Monitoring Data
MARTY #6
ONALASKA,WI
Performed for: MARTY
Date Printed = 09-20-1991 Current Time = 15:40:05
File Name = D:\CEMDATA\092091.PRN Calibration File:D:\CEMDATA\0920CALA.CAL

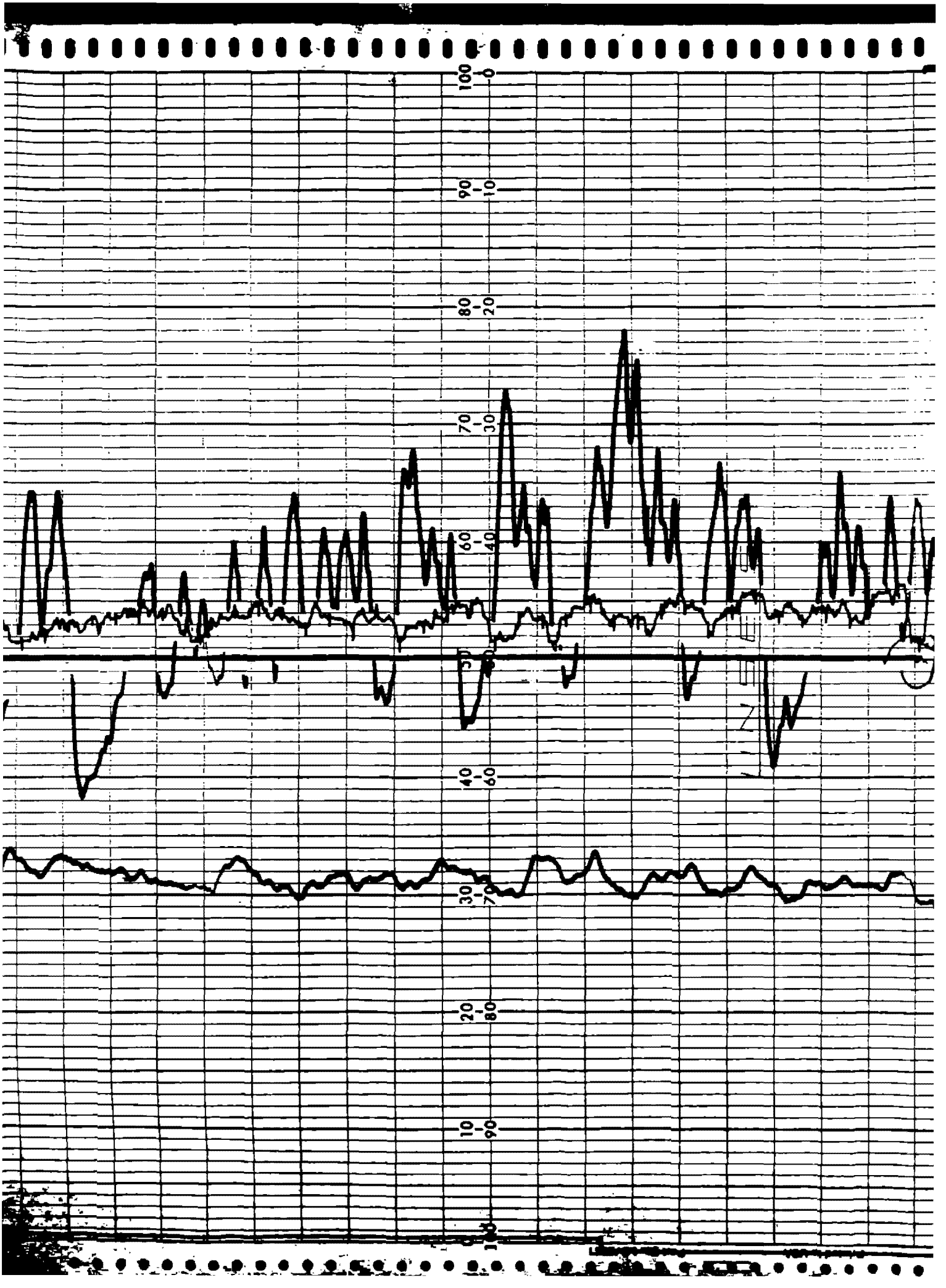
09-20-1991 Time	O2 %	THC PPMV	SO2 PPMV	CO PPMV	CO2 %	NOX PPMV									
15:40:46	7.4	-2.9	0.2	131.1	10.8	-2.4	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:41:45	7.4	-2.9	0.3	81.0	10.9	-2.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:42:19	7.4	-2.9	0.3	78.0	10.9	-2.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:42:39	7.4	-3.0	0.4	77.7	10.9	-2.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:42:59	7.4	-3.0	0.2	77.3	10.9	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:43:19	7.4	-2.9	0.1	77.3	10.9	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:43:39	7.4	-2.9	0.3	77.3	11.0	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:43:59	7.4	-2.9	0.0	78.8	11.0	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:44:19	7.4	-2.9	-0.2	82.3	10.9	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:44:39	7.4	-2.9	-0.2	82.2	10.7	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:44:59	7.4	-2.9	-0.1	82.2	10.5	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:45:19	7.4	-2.9	-0.2	82.2	10.4	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:45:39	7.4	-2.9	-0.3	82.2	10.3	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:45:59	7.4	-2.9	-0.5	82.3	10.1	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:46:19	7.4	-2.9	-0.5	82.3	10.1	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:46:38	5.9	-2.9	0.3	79.9	10.1	29.9	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:46:58	1.6	-2.9	14.3	58.6	9.1	93.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:47:18	0.4	-2.9	34.0	18.5	5.5	100.4	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:47:38	-0.1	-2.9	42.6	-1.7	0.5	101.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:47:58	-0.1	-2.9	46.3	-4.3	0.1	101.7	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:48:18	-0.1	-2.9	49.0	-7.4	0.1	101.9	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:48:38	-0.1	-2.9	50.2	-7.4	0.0	102.0	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:48:58	-0.1	-2.9	51.7	-4.8	0.0	102.1	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:49:18	-0.1	-2.9	53.0	-7.4	0.0	102.2	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:49:38	-0.2	-2.9	54.0	-7.4	0.0	102.3	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:49:58	-0.2	-2.9	54.9	-7.3	0.0	98.7	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:50:18	0.5	-2.9	55.8	-7.3	-0.0	100.8	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:50:38	-0.1	-2.9	84.6	-7.4	-0.0	102.2	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:50:58	-0.2	-2.9	94.8	-7.4	-0.0	102.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:51:18	-0.2	-2.9	96.7	-7.3	-0.0	102.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:51:34	-0.2	-2.9	97.5	-7.2	-0.1	102.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:51:44	-0.2	-2.9	97.5	-6.9	-0.1	102.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:51:54	-0.2	-2.9	97.8	-7.3	-0.1	102.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:52:04	-0.2	-2.9	98.3	-7.2	-0.1	102.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:52:14	-0.2	-2.9	98.1	-7.2	-0.1	102.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:52:24	-0.2	-2.9	98.0	-7.2	-0.1	102.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:52:33	-0.2	-2.9	98.4	-7.3	-0.1	102.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:53:03	-0.1	-2.9	81.8	-4.3	-0.1	71.3	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:53:33	-0.2	-2.9	20.0	-2.1	-0.0	46.8	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:54:03	-0.2	-2.9	6.5	-5.8	-0.1	45.9	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:54:13	-0.1	-2.9	3.7	-5.2	-0.1	45.7	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.4	24.2	32
15:54:23	-0.2	-2.9	3.2	-6.7	-0.1	45.6	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:54:33	-0.2	-2.9	2.7	-6.7	-0.1	45.6	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32
15:54:43	-0.2	-2.9	2.4	-6.7	-0.1	45.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32

09-20-1991	O2	THC	SO2	CO	CO2	NOX									
Time	%	PPMV	[illegible]	PPMV	%	PPMV									
15:55:13	-0.2	-2.[illegible]	[illegible]	-6.6	-0.1	45.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:55:23	-0.2	-2.[illegible]	[illegible]	-6.8	-0.1	45.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:55:33	-0.2	-2.[illegible]	1.3	-6.9	-0.1	45.5	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:55:43	-0.2	-2.9	1.3	-5.7	-0.1	37.3	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:55:53	3.1	-2.9	1.1	7.5	-0.1	10.4	-4.0	-4.1	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:56:53	16.3	-2.9	1.0	322.5	11.1	-1.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:57:23	17.6	-2.9	0.7	477.1	15.5	-2.6	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:57:43	17.6	-2.9	1.1	478.6	15.6	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:58:03	17.6	-3.0	0.8	478.4	15.6	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:58:23	17.6	-3.0	0.6	478.4	15.6	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:58:43	17.6	-2.9	0.4	478.5	16.5	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:59:03	17.7	-2.9	0.3	478.4	17.2	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:59:22	17.7	-2.9	0.3	478.5	17.2	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
15:59:42	17.7	-2.9	0.3	478.5	17.2	-2.7	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
16:00:02	17.7	-2.9	0.4	478.4	17.2	-2.8	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
16:01:02	16.9	-3.0	0.8	515.8	17.2	9.4	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.
Avg.=	5.0	-2.9	25.9	102.5	5.8	44.0	-4.0	-4.0	-4.7	-4.8	23.5	23.5	23.5	24.3	32.

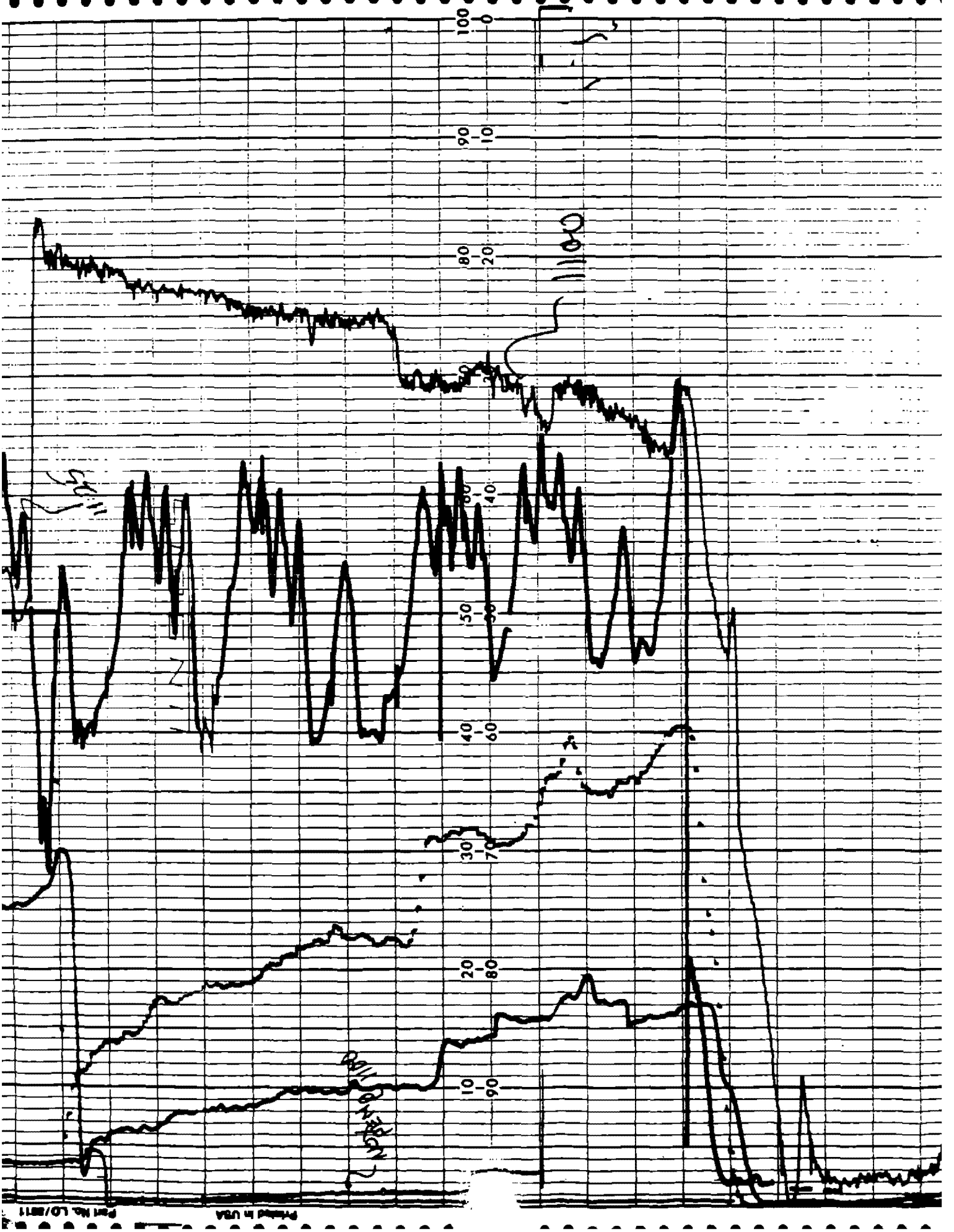
APPENDIX D.2

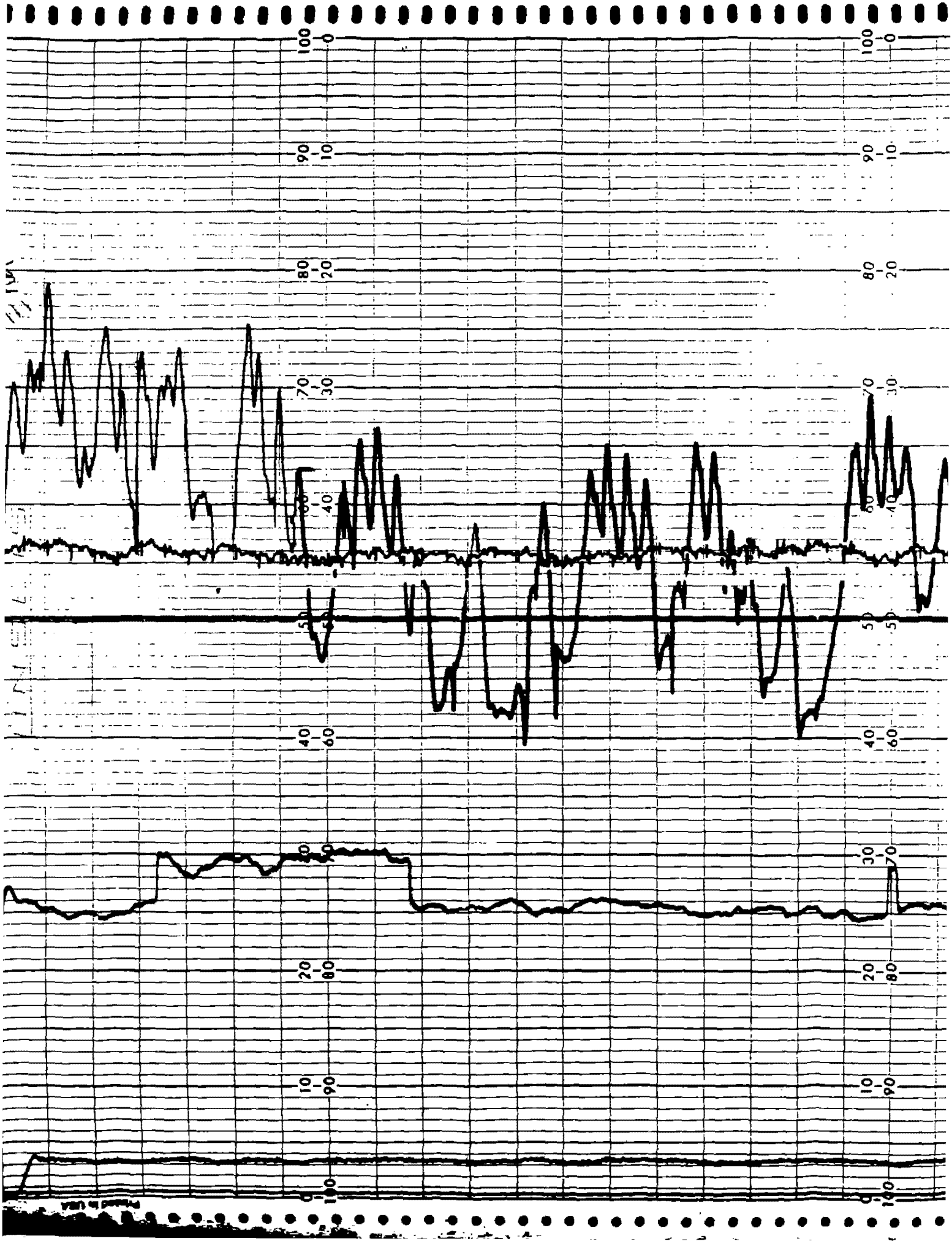
STRIPCHART TRACINGS

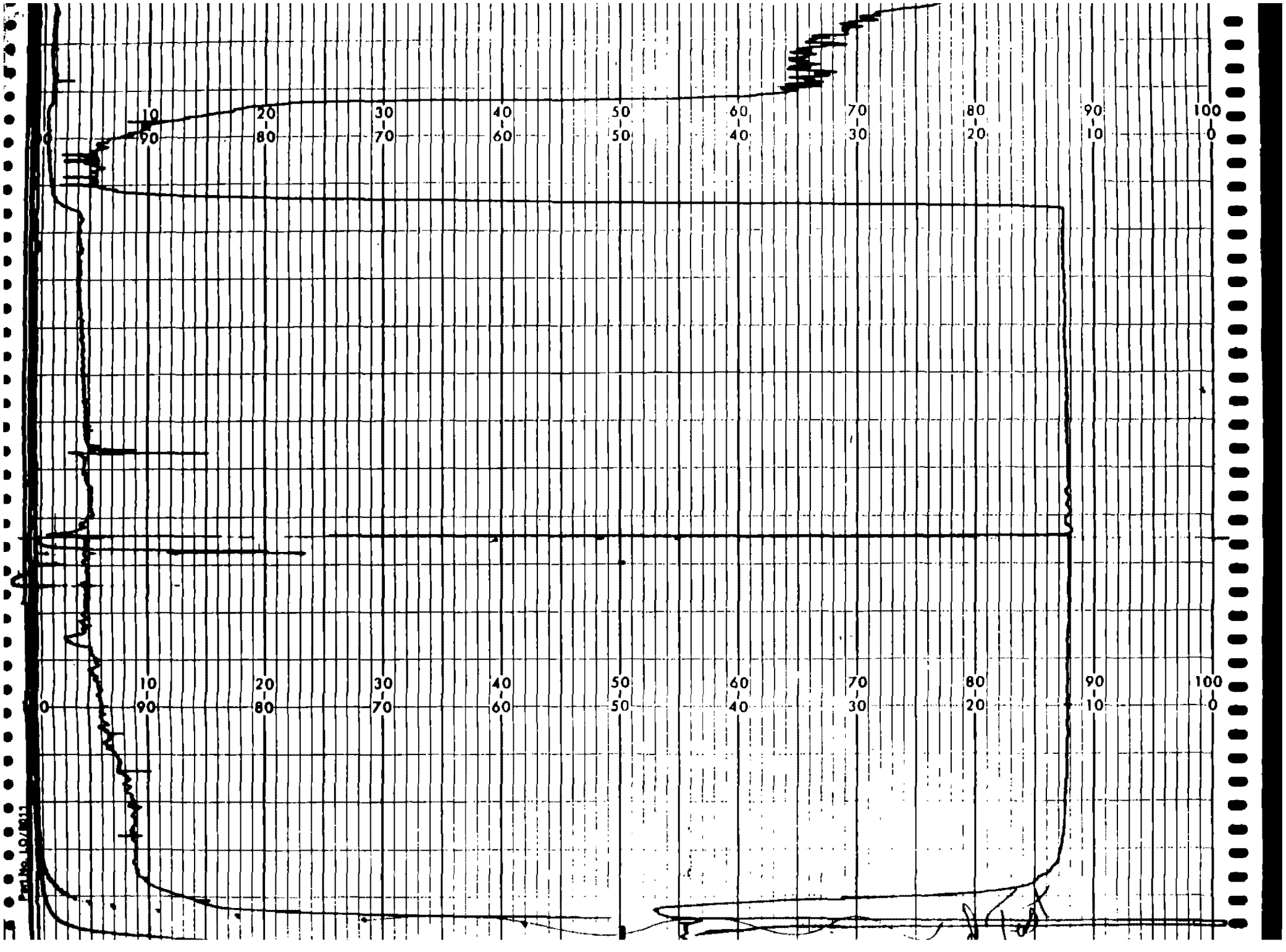
100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
20dB
Voltage Gain
Output
Input
0-100mV


100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90

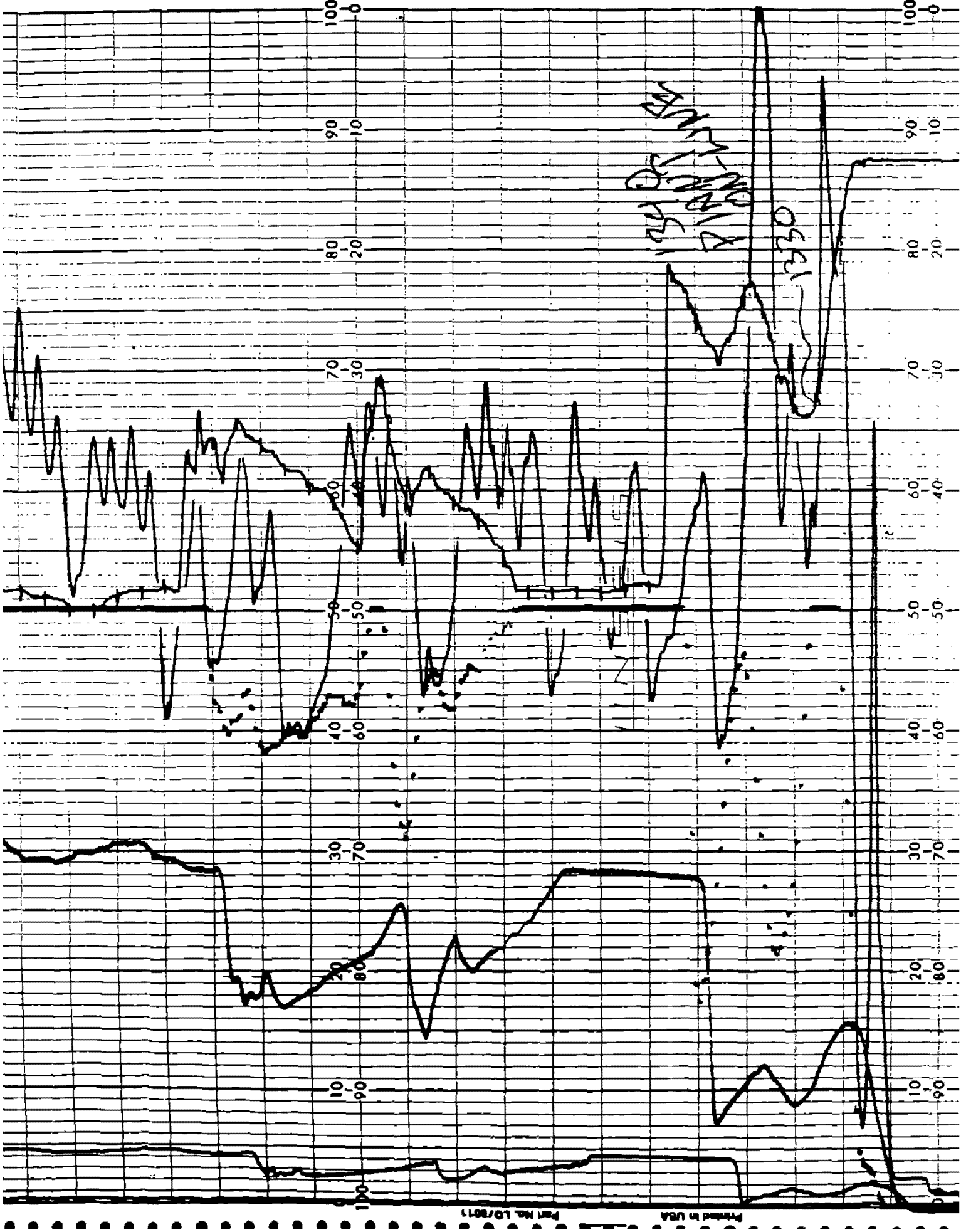
light
dark
dark
light

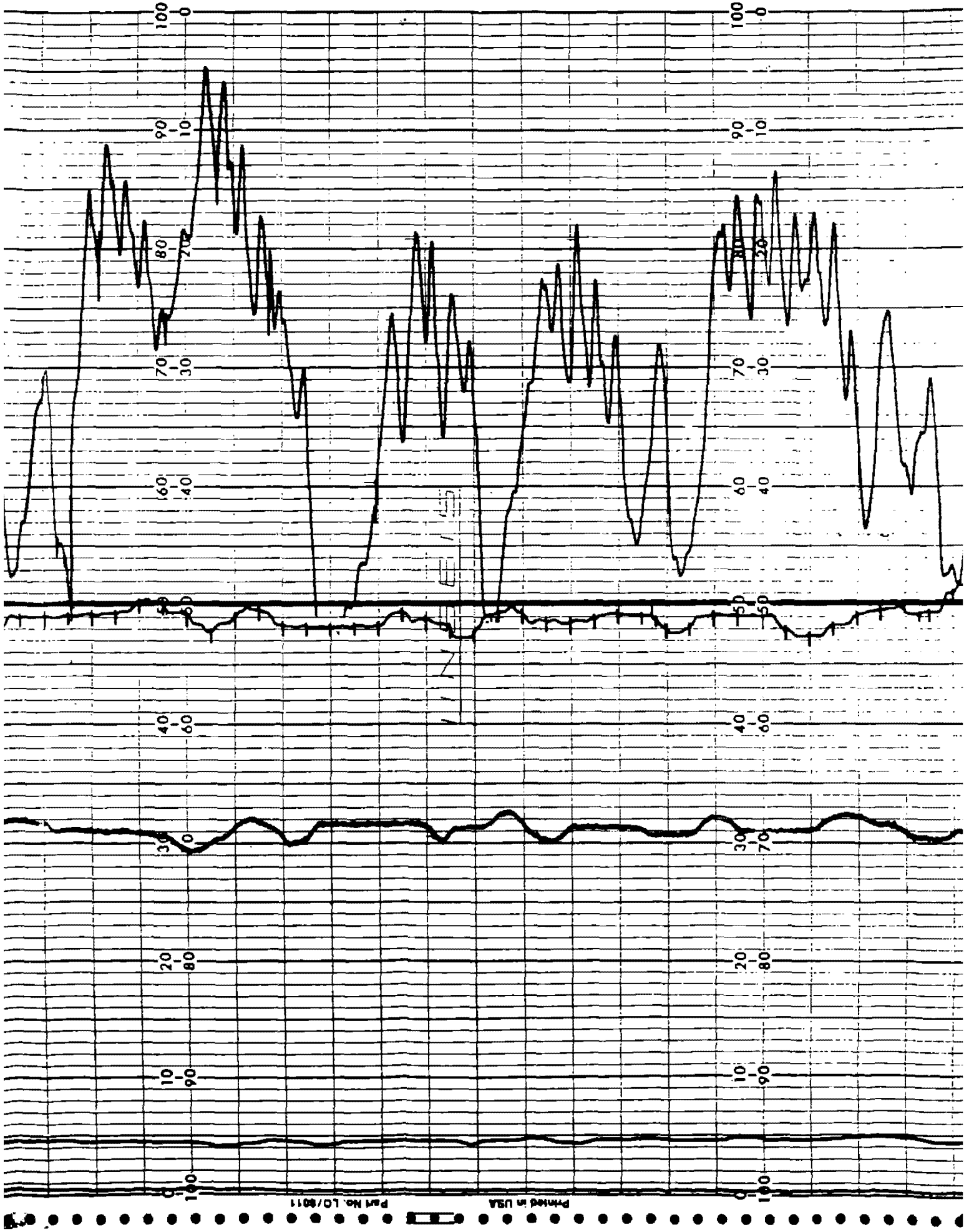

100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90

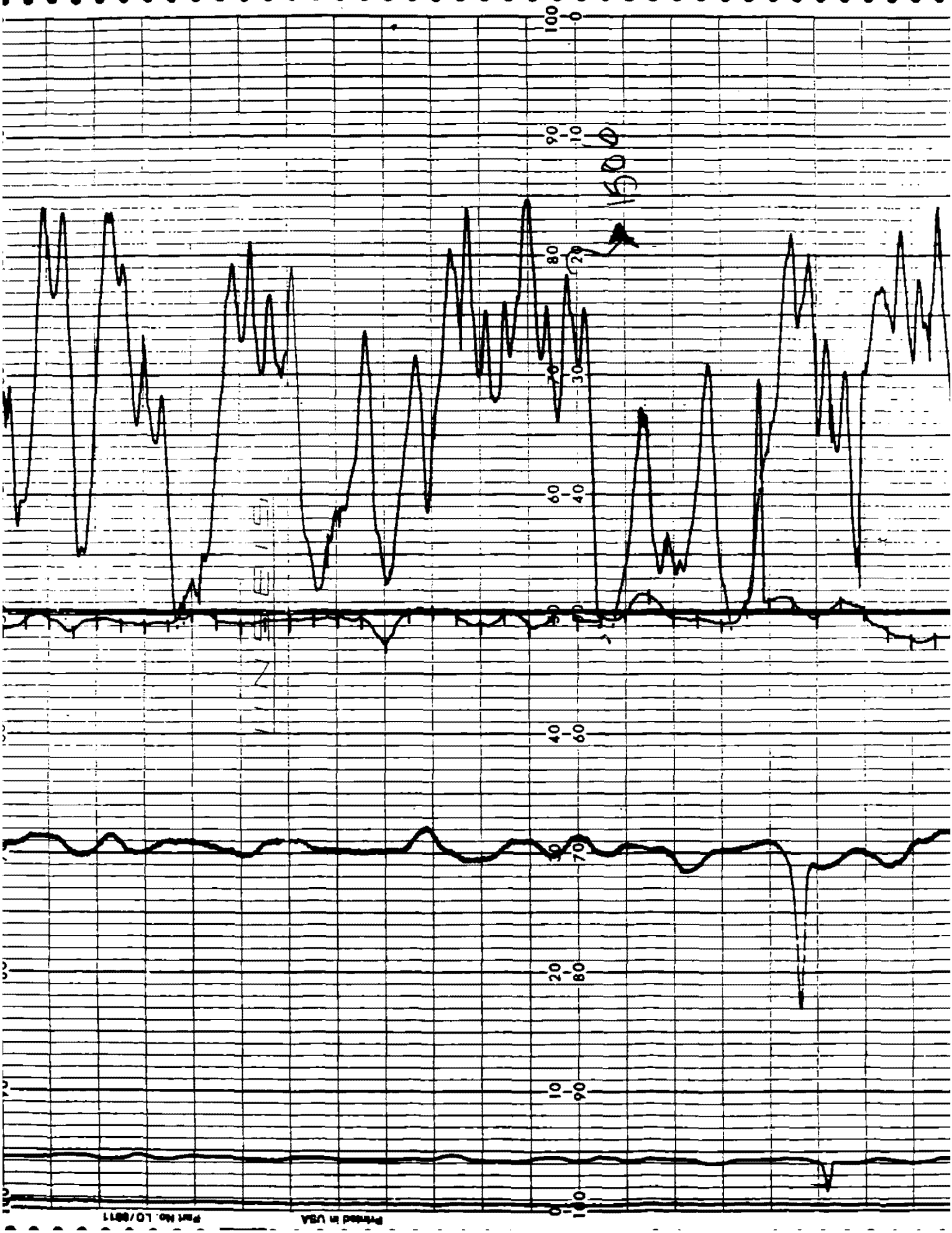



10 20 30 40 50 60 70 80 90 100
90 80 70 60 50 40 30 20 10 0
10 20 30 40 50 60 70 80 90 100
90 80 70 60 50 40 30 20 10 0

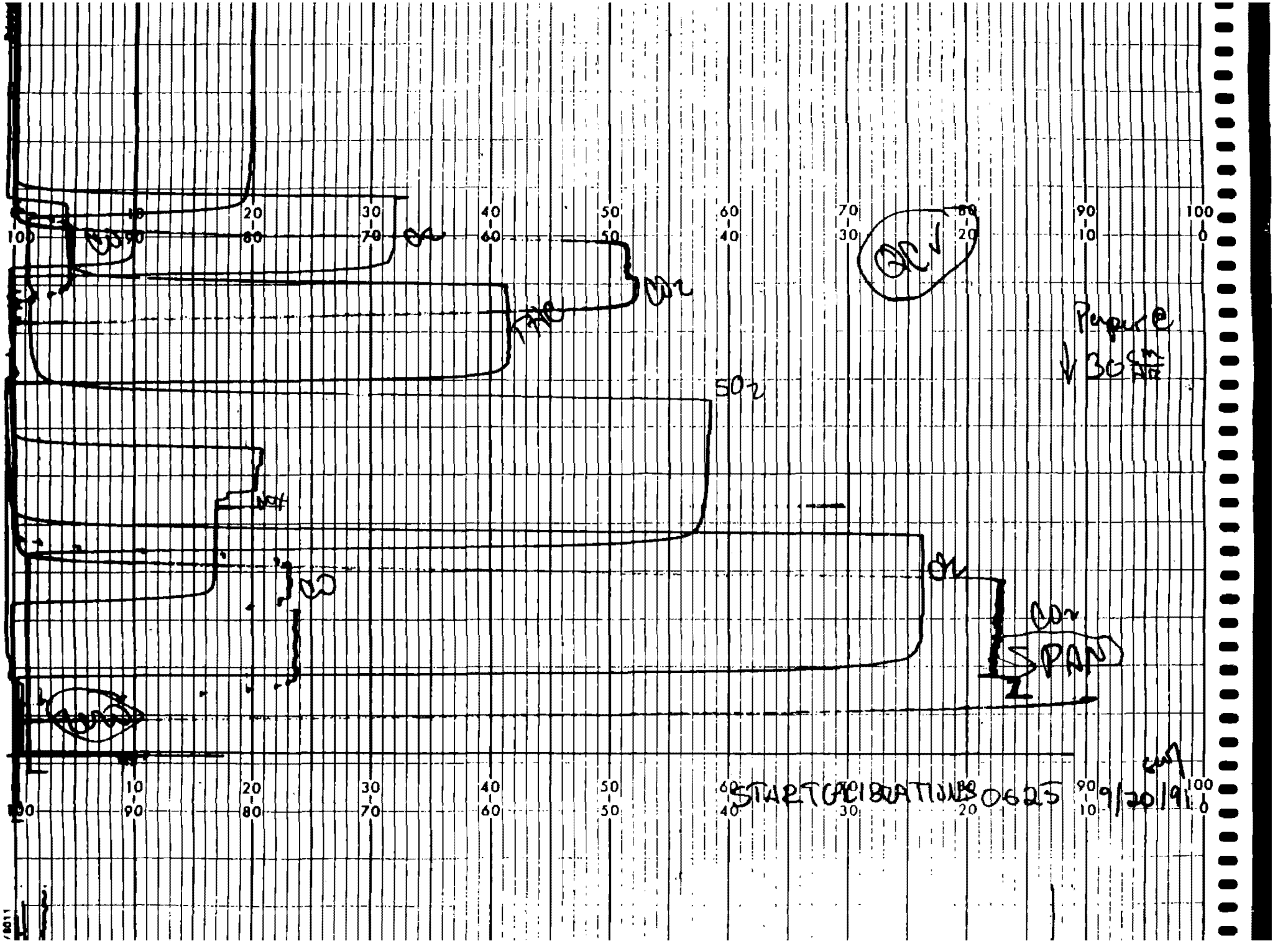
100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
100
OIL LINE
PLANT Cmpd UP
TEMP
Pen No. 10/8011

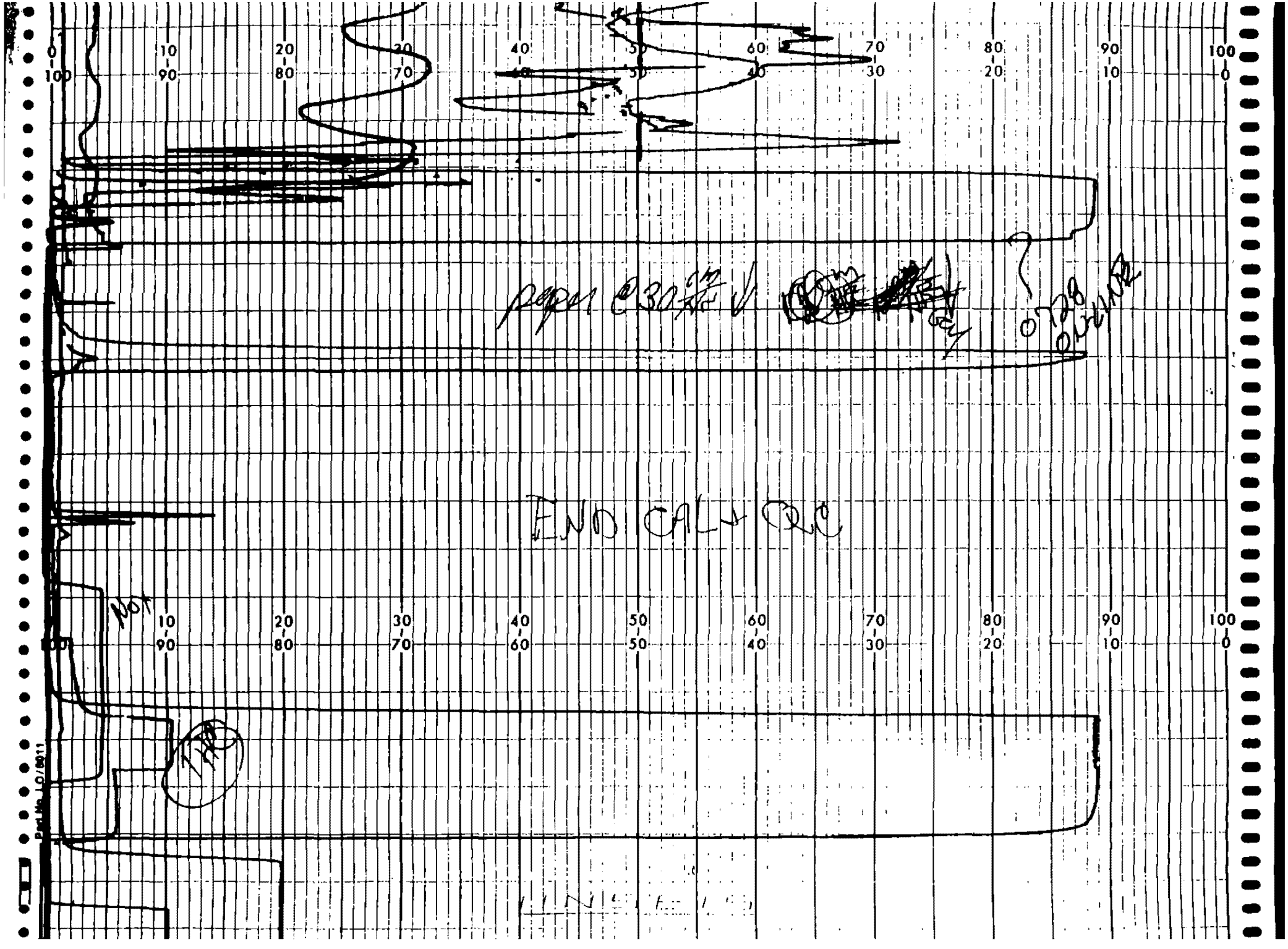

1350
Part No. LD/8011
Printed in USA

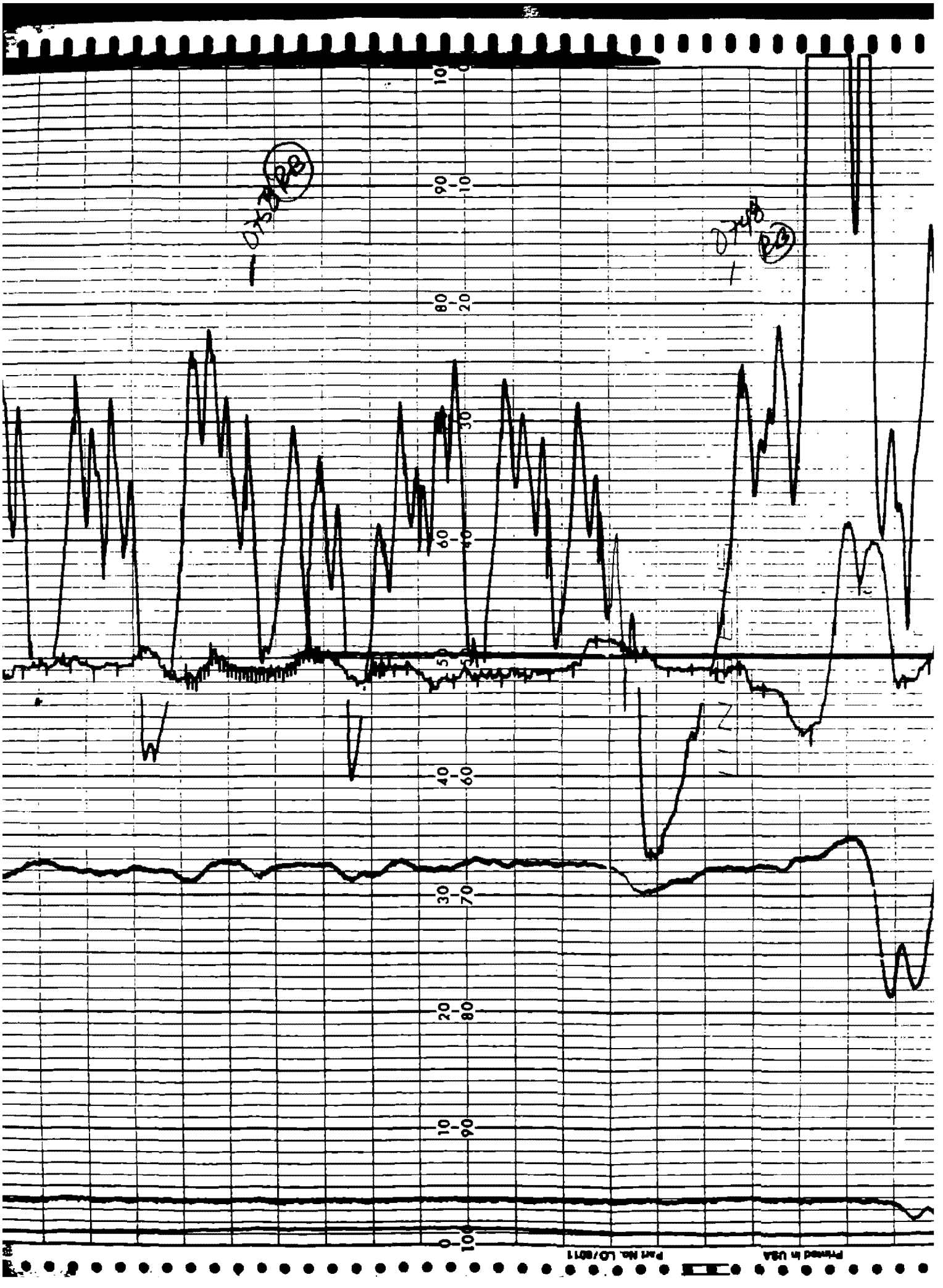

100 0
90 10
80 20
70 30
60 40
50 50
40 60
30 70
20 80
10 90
0 100
Printed in USA
Part No. LO/8011

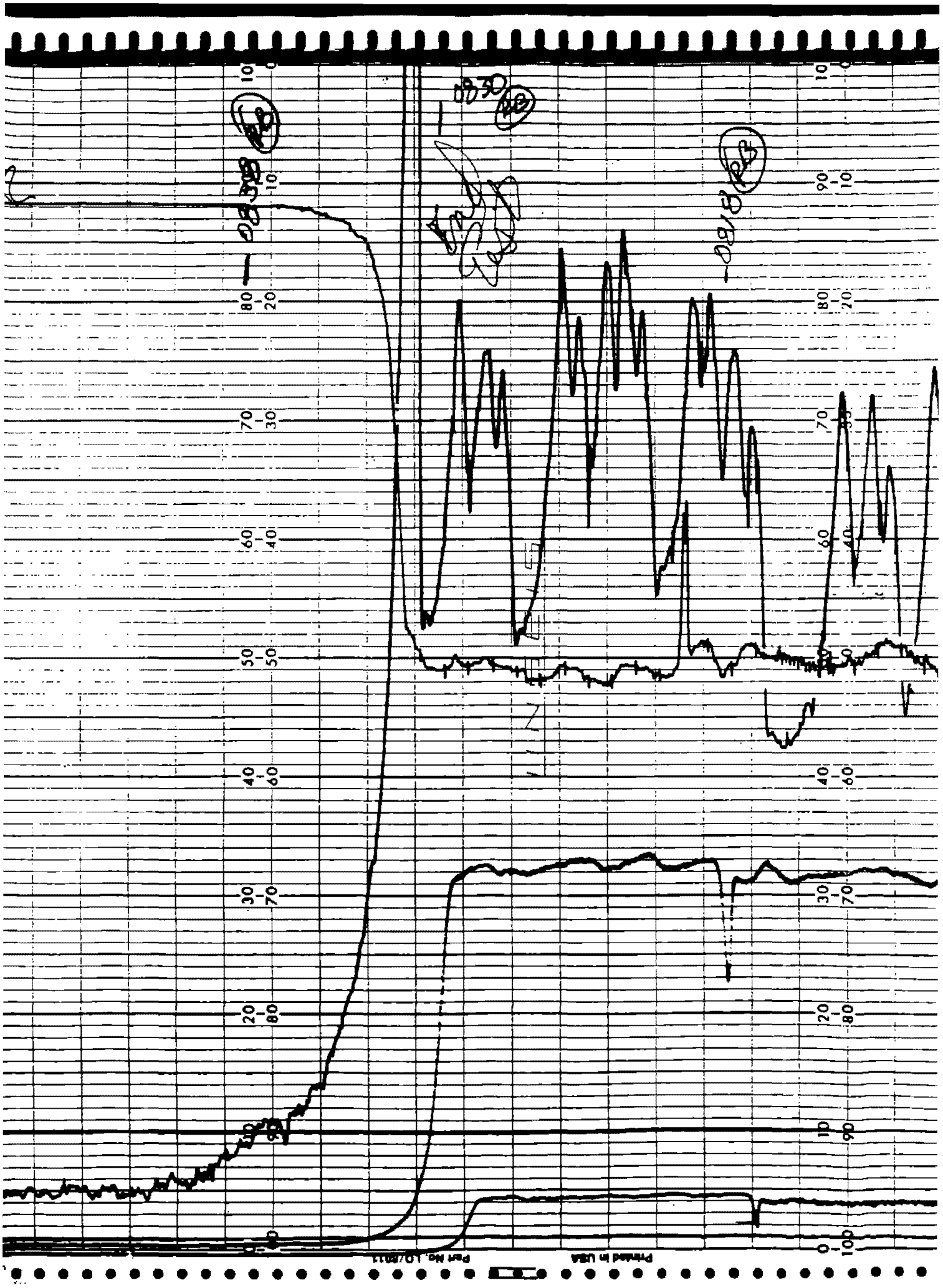

100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
100
Printed in USA
Part No. LO/8011

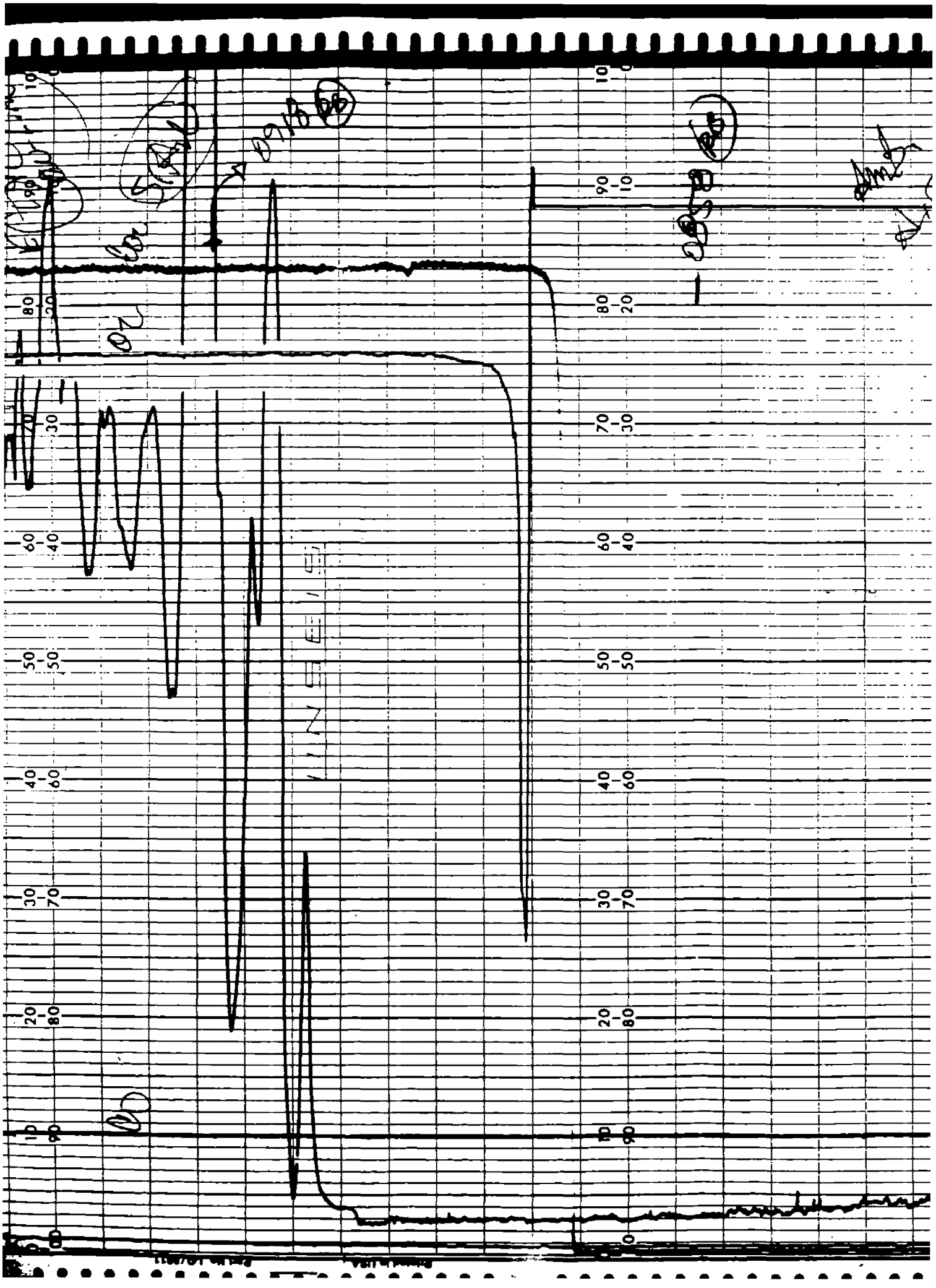
100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
100

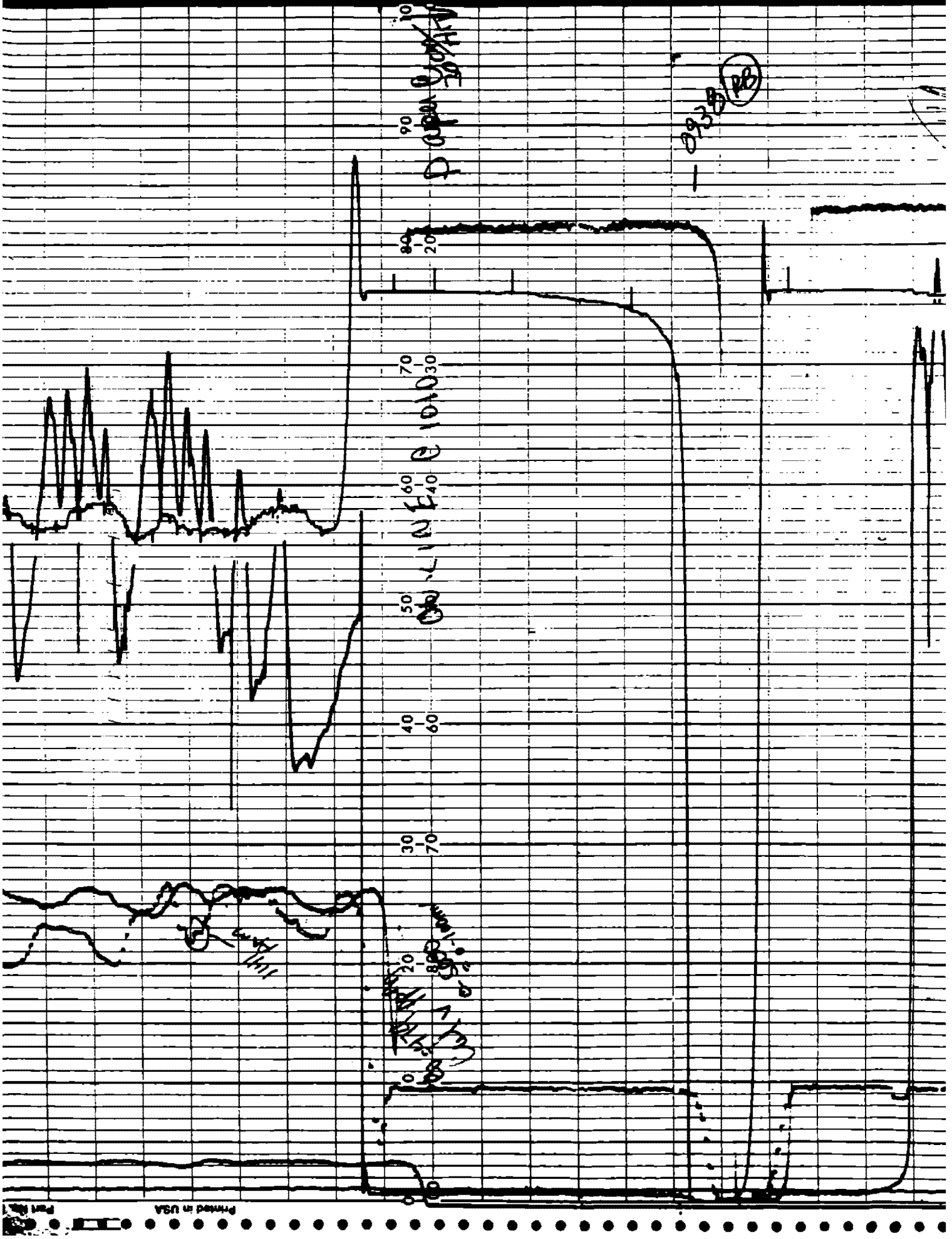

NO
SO2
CO
CO2
SPAN
START CALIBRATIONS 0625
9/20/91


END CAL

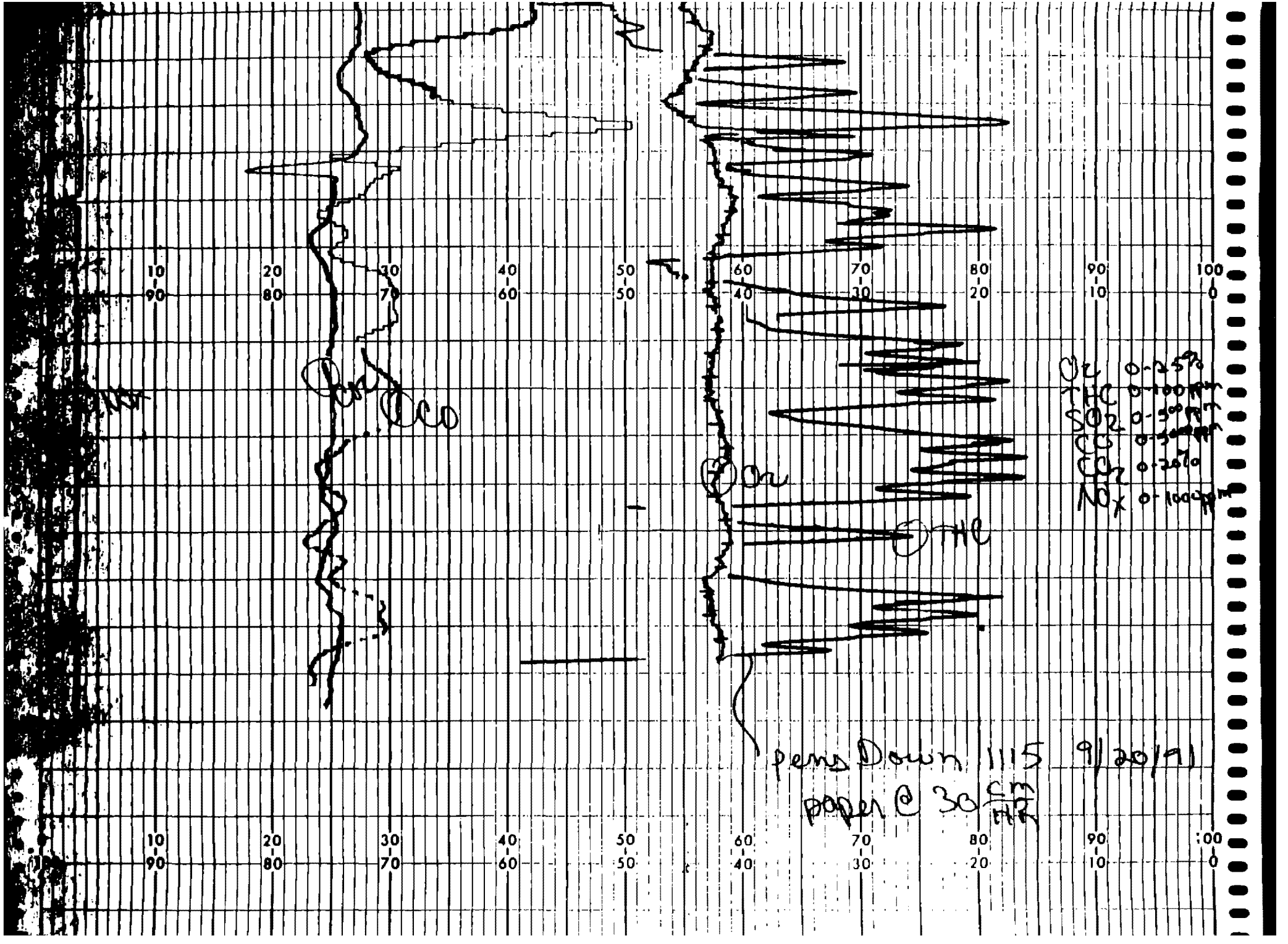

10
90
10
80
20
30
60
40
5
5
40
60
30
70
20
80
10
90
0
100
V
V
Z
Printed in USA
Part No. LD/8011

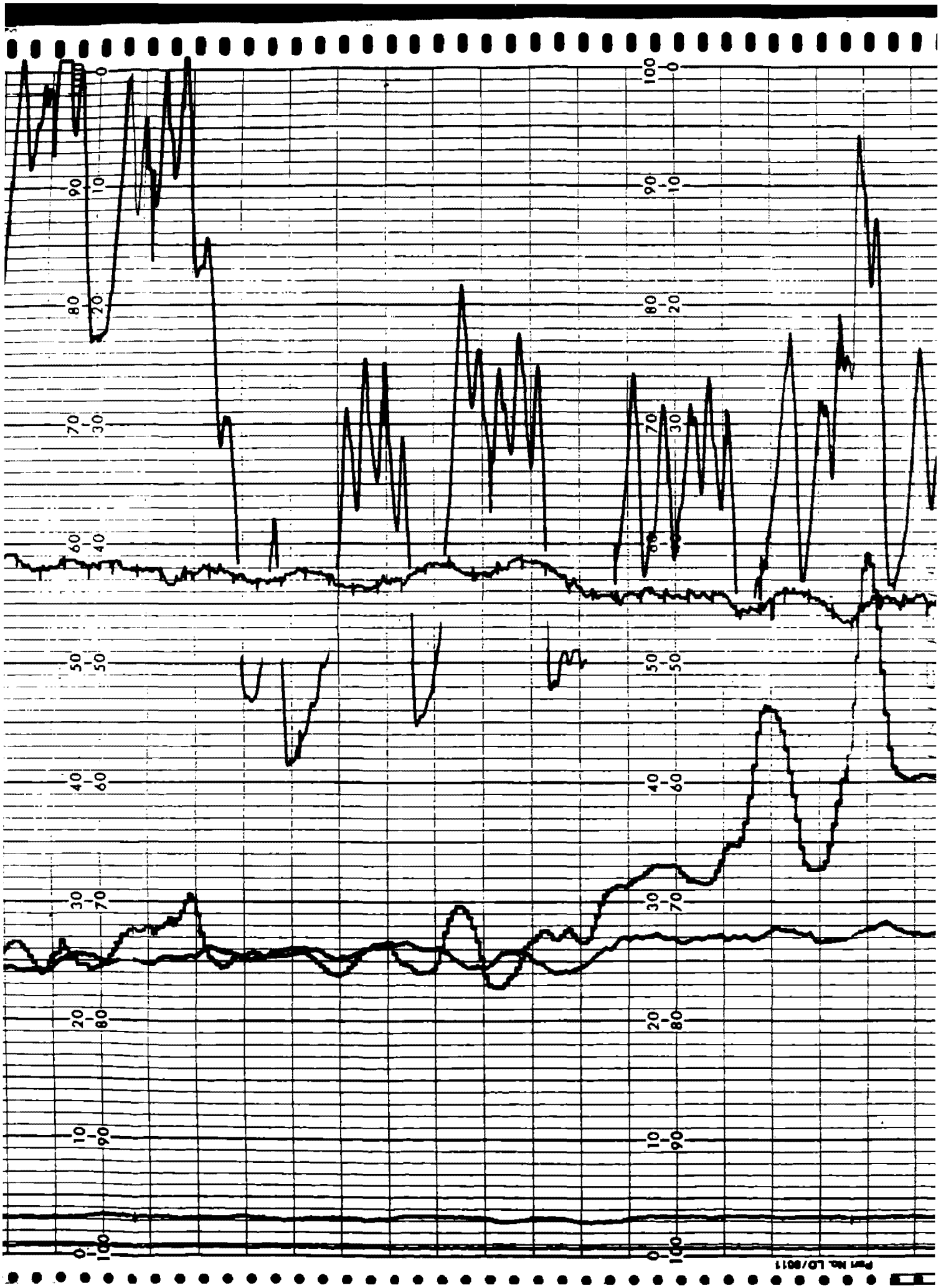


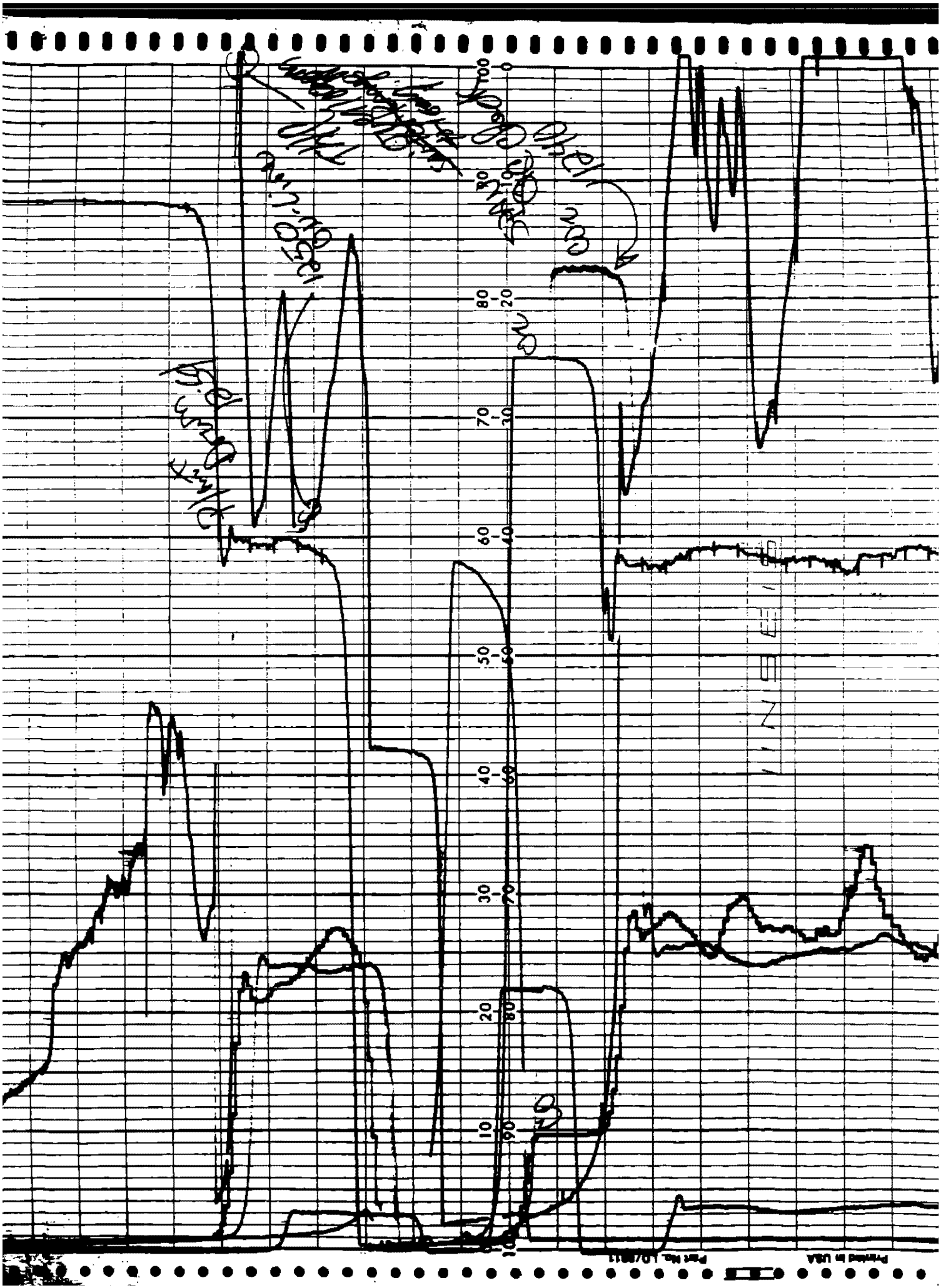


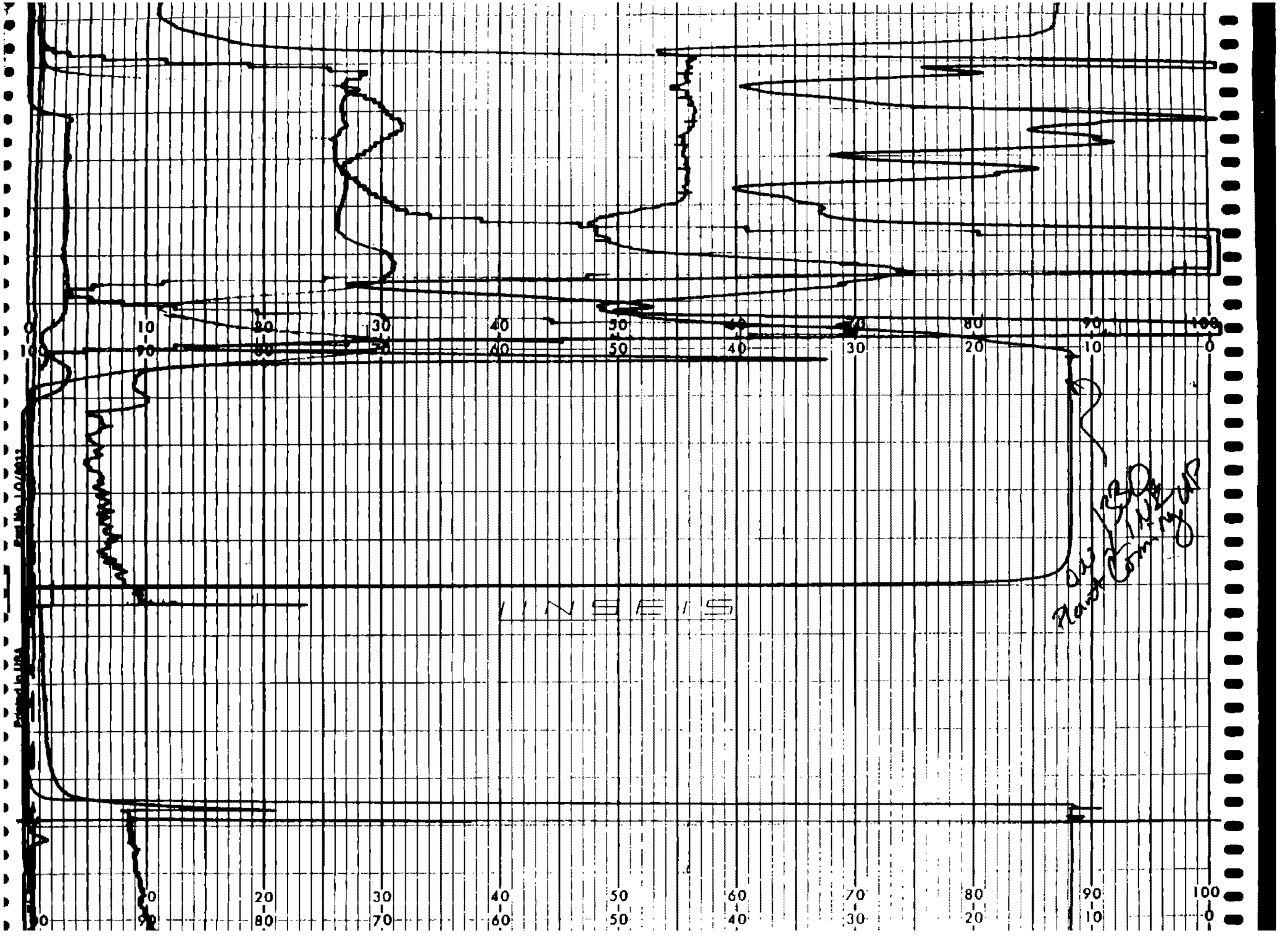

Printed in USA

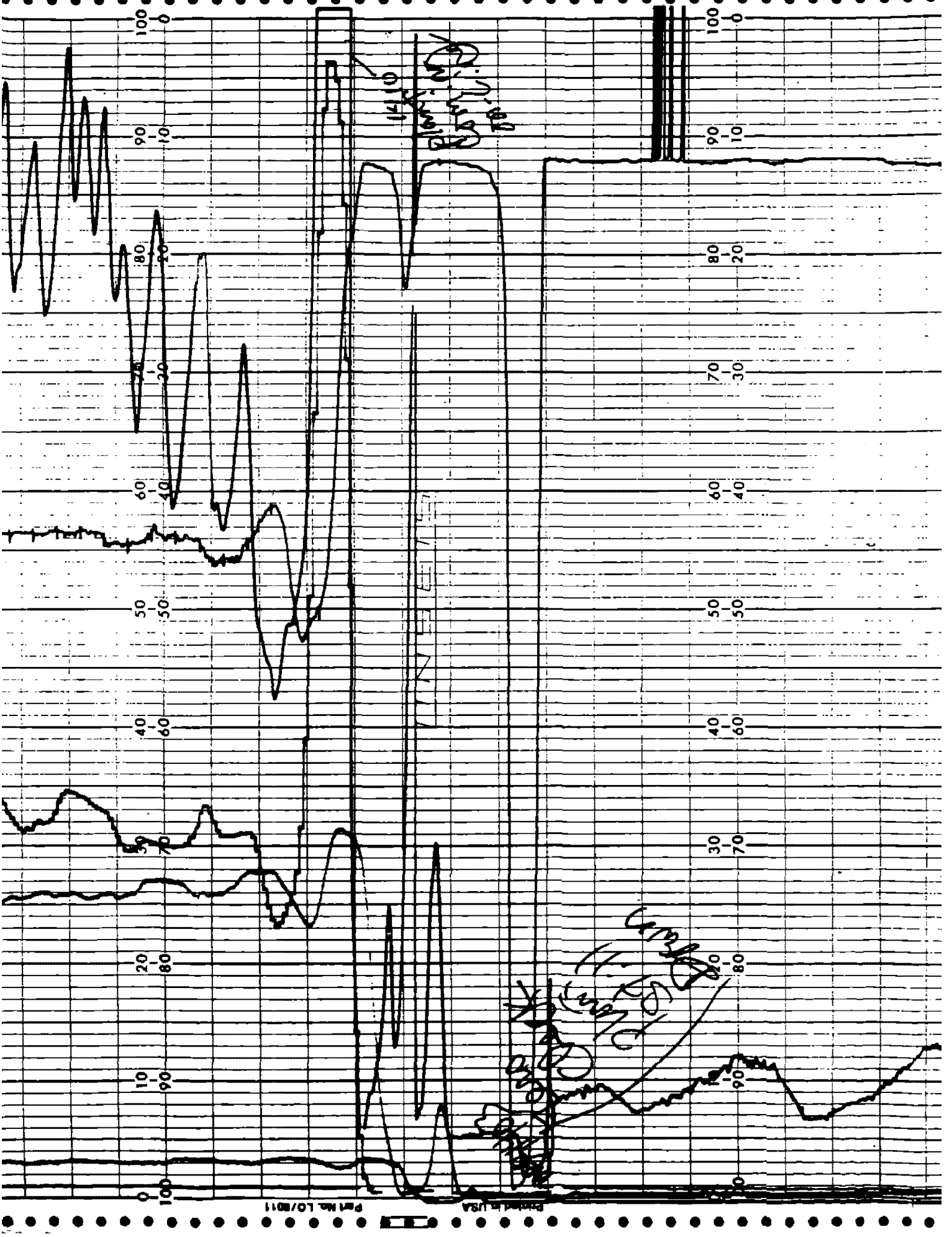
Endpoint @ 9/22/91
Inlet
Outlet
Pressure

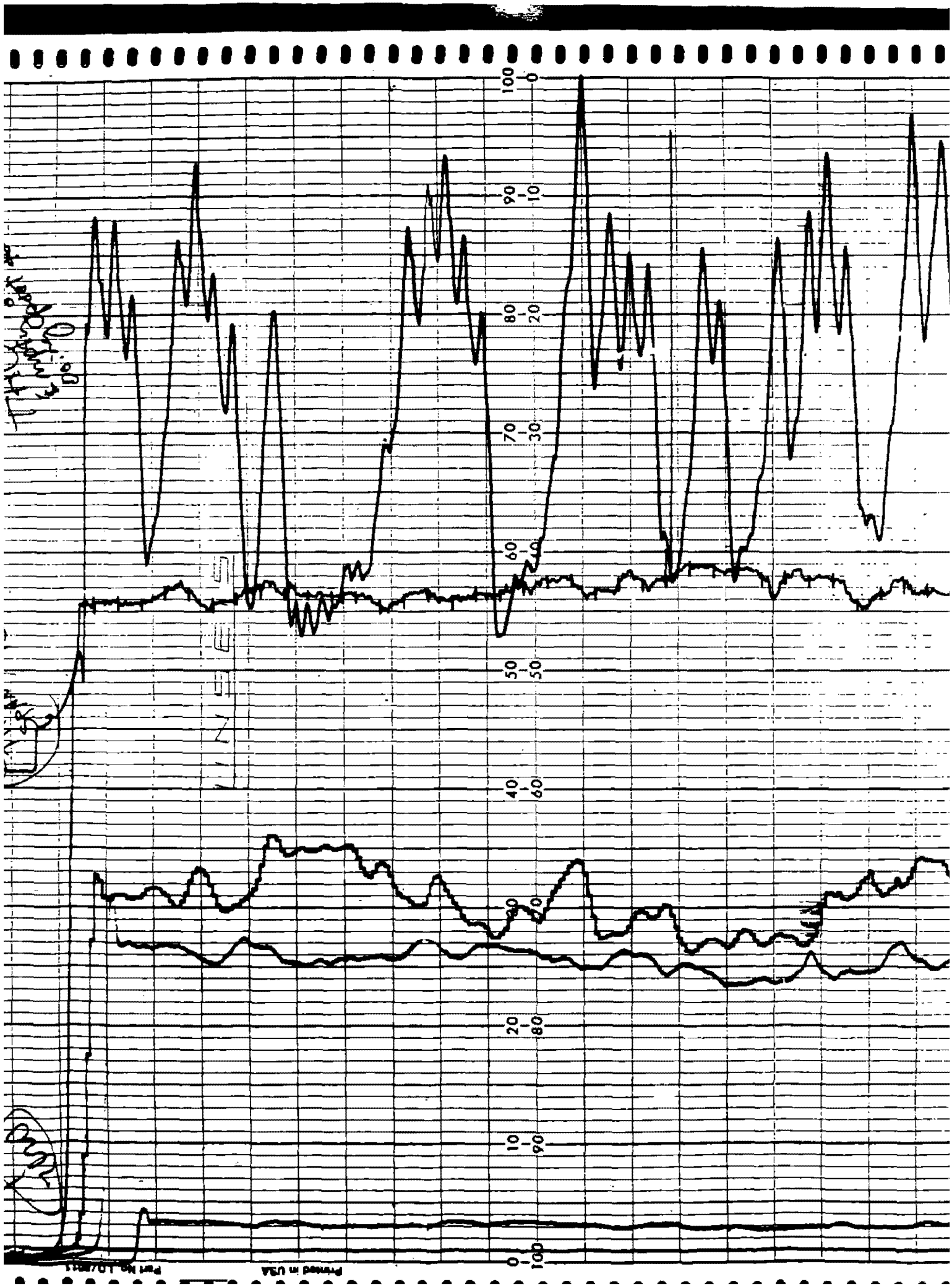

10 90
20 80
30 70
40 60
50 50
60 40
70 30
80 20
90 10
100 0
CO
O2
HC
O2 0-25%
HC 0-100 PPM
SO2 0-50 PPM
CO 0-20%
NOx 0-100 PPM
Pens Down 1115 9/20/91
paper @ 30 cm/hr


Part No. LD/8011




VINSETS



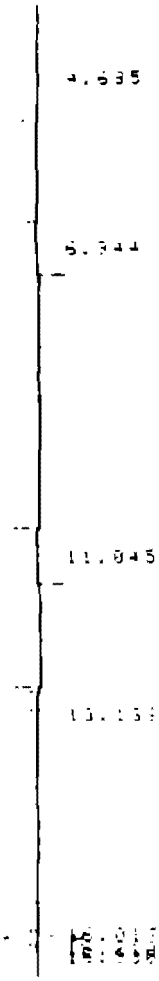

100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
100

POST TEST
QC check
1540
(3) diastolic
(2) systolic
(1) MAP

100
0
90
10
80
20
70
30
60
40
50
50
40
60
30
70
20
80
10
90
0
100

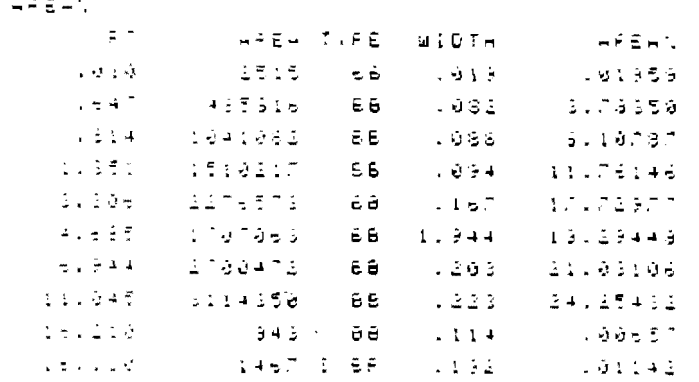
APPENDIX E

GC DATA



STOP

RUN# 35 SEP 20, 1991 07:03:00



FT	mFEm	TYPE	WIDTH	mFEm'
.010	1515	SB	.019	.001855
.047	485516	BB	.082	3.783350
.114	1041041	BB	.088	5.107787
1.391	1510217	SB	.094	11.761446
2.104	227e571	BB	.167	17.713977
4.685	1707063	BB	1.944	13.239449
5.944	1703473	BB	.203	21.031106
11.045	3114150	BB	.223	24.194311
13.210	943	BB	.114	.000507
15....	1467	BF	.132	.001141

TOTAL mFE=1.13940E-07
ALL FACTOR=1.00000E-00

9/20/91 1 of 63

TIME TABLE STOP

RUN# 34 SEP 20, 1991 06:36:36

AREA

RT	AREA	TYPE	WIDTH	AREA
.625	39589	PB	.049	.10457
.636	53560	BB	.049	.14153
1.019	33294	BB	.220	.08794
3.320	226	PB	.049	.00060
6.217	124006	BB	1.204	.32755
6.575	1356	BB	.131	.00359
7.624	15659	I VB	.191	.04136
8.790	11322	BB	.220	.02991
8.858	2057	BB	.038	.00543
9.477	55150	BB	.796	.14568
9.933	5059229	BB	.212	13.36362
10.633	5714	BB	.167	.01509
11.037	7573	BB	.131	.02000
11.703	133	BB	.053	.00032
11.971	24356	BB	.136	.06433
12.364	785	BB	.100	.00207
12.577	3071	BB	.301	.00811
14.695	4980730	BB	.194	13.15637
15.497	54531	BB	.223	.14404
15.883	498	BB	.100	.00132
16.057	2534	BB	.266	.00669
16.419	76	BB	.039	.00020
16.739	27680	BB	.321	.07311
17.566	8730088	BB	.364	23.06790
18.436	204	PB	.064	.00054
20.030	91043	BB	.541	.24313
21.173	194	PB	.055	.00078
21.735	3939	BB	.137	.01054
22.319	15525456	BB	.458	48.93373

TOTAL AREA=3.7858E+07
MULTIPLICATION FACTOR=1.0000E+00

TIME7:00:35
SEP 20, 1991 07:00:35

RUN # 35 SEP 20, 1991 07:03:00
START

0.010
0.514
1.351

BTEX

* RUN # 34 SEP 20, 1991 05:36:36
START

IP

0.686

1.019

3.330

4.717

6.317

6.875

7.624

8.396

8.477

9.331

10.683

11.037

11.705

11.971

12.264

12.577

14.699

15.447

16.419

16.739

17.666

19.436

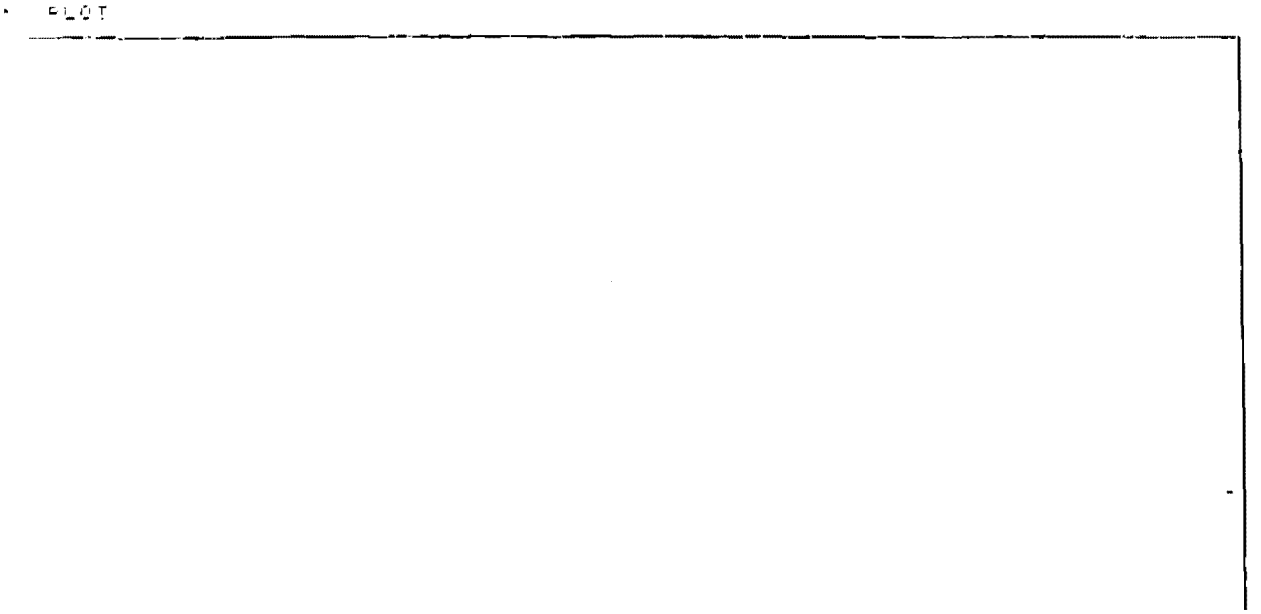
20.030

21.173

21.735

23.515

```
* PLOT
```



```
STOP

* PLOT
STOP

* LIST TIME @

   0.000 INTG # =  5
  30.000 STOP


* TIME 25 STOP
* DELETE TIME 30 @

* TIME 0 INTG # 2 @
* LIST TIME @

   0.000 INTG # =  5
   0.000 INTG # =  2
  25.000 STOP
```

```
* PREP CALIB @

E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E/I/N]: N

REF % RTW [   5.000]:
NON-REF % RTW [   5.000]: -   ?
NON-REF % RTW [   5.000]: -5

RF BASED ON AREA OR HEIGHT [A/H]:
CAL#      RT          AMT          NAME
  1   :1.647       :15.0         :METHANE
  2   :1.819       :14.5         :ETHANE
  3   :1.35  NO MATCH
  3   :1.351@@@@@1.351
                   :4.2@@@14.2   :PROPANE
  4   :3.306       :14.6         :PENTANE
  5   :6.944       :14.6         :PENTANE
  6   :11.045      :14.5         :HEANE@@@AANE
  7   :9.933@@@@@-9.933  NO MATCH
  7   :
REF PK CAL#:  1
REF PK CAL#:  3.08.TE-05   ?
REF PK CAL#:

GROUP PEAKS [Y N]:

CALIBRATION OPTIONS
RF or uncalibrated peaks [0.0000E+00 ]: 3.08.TE-05
Replace calibration rts [Y N]:
Disable post-run RT update [Y/N]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E-00 ]:

* EDIT CALIB @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  4
RT:
AMT:
AMT AREA:
NAME:  BUTANE

CAL#:

SECTION TO BE EDITED:

* LIST: CALIB @

NORM
```

```
LE EL: 1                SELECTIONS: 1

CHL#      RT      EV      AMT        AMT-APEA
   1P     0.647     1  1.5000E+01   3.0576E-05
   2      0.814     1  1.4500E+01   1.3918E-05
   3      1.351     1  1.4200E+01   9.4036E-06
   4      3.306     1  1.4600E+01   6.4133E-06
   5      6.944     1  1.4600E+01   5.4065E-06
   6     11.045     1  1.4500E+01   4.6559E-06

CHL#      NAME
   1   METHANE
   2   ETHANE
   3   PROPANE
   4   BUTANE
   5   PENTANE
   6   HEXANE

CALIBRATION OPTIONS
RF or uncalibrated peak .... 3.0370E-05
Calibration fit ............ P
Disable post-run RT update .. NO
MUL FACTOR ................. 1.0000E+00

*  OF # 3

CALIBRATION OPTIONS
RF or uncalibrated peaks [3.0370E-05 ]:
Replace calibration fit [Y N*]:
Disable post-run RT update [Y N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

*  EDIT  CALIB    0

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CHL#:  7
RT:   9.931
AMT:   19.9
AMT-APEA:   3.935E-06
NAME:   BENZENE

CHL#:

SECTION TO BE EDITED: 4&3

CHL#:   8
RT:   114.995&&&&&14.895
AMT:   20.84
AMT-APEA:   4.154E-06
NAME:   TOLUENE

CHL#:   9
RT:   17.666
AMT:   20.86
AMT-APEA:   2.388E-06
```

```
[-L*:   .0
RT:   22,511E+5
AMT:   21.05
AMT/AREA:   2.275E-06
NAME:   XYLENE

CAL*:

SECTION TO BE EDITED:

*   OP # 4

REPORT OPTIONS

Suppress local report [Y/N*]:
HEIGHT% report [Y/N*]:
Replace report title [Y/N*]:
Replace amount label [Y/N*]:
Report uncalibrated peaks [Y/N*]: Y
Extended report [Y/N]:

*   LIST: LIST
PEAK CAPACITY:   1221

ZERO    =   0, -5,682
ATT 2   =  -1
CHT SF  =   1.0
AP REJ  =   0
THRSH   =  -1
PK WD   =   0.06


*   THRSH 0   0
*   Run #    36      SEP 20, 1991   07:47:05
START
   IF
     STOP


Run#     36           SEP 20, 1991   07:47:05

NO RUN PEAKS STORED


*   Run #    37      SEP 20, 1991   07:47:50
START: not ready
   IF
     STOP


Run#     37           SEP 20, 1991   07:47:50

NO RUN PEAKS STORED


*
BREAK

*   PLOT
   IF
STOP
```

```
START GAS READ
   IF
   STOP


RUN#     38            SEP 20, 1991   07:49:58

NO RUN PEAKS STORED



*   THRSH    0
*   LIST: LIST
PEAK CAPACITY:   1221

ZERO    =   0, -6.297
ATT 2   =  -1
CHT SP  =   1.0
AR REJ  =   0
THRSH   =   2
FW WD   =   0.08


*   THRSH 1   0
*
```

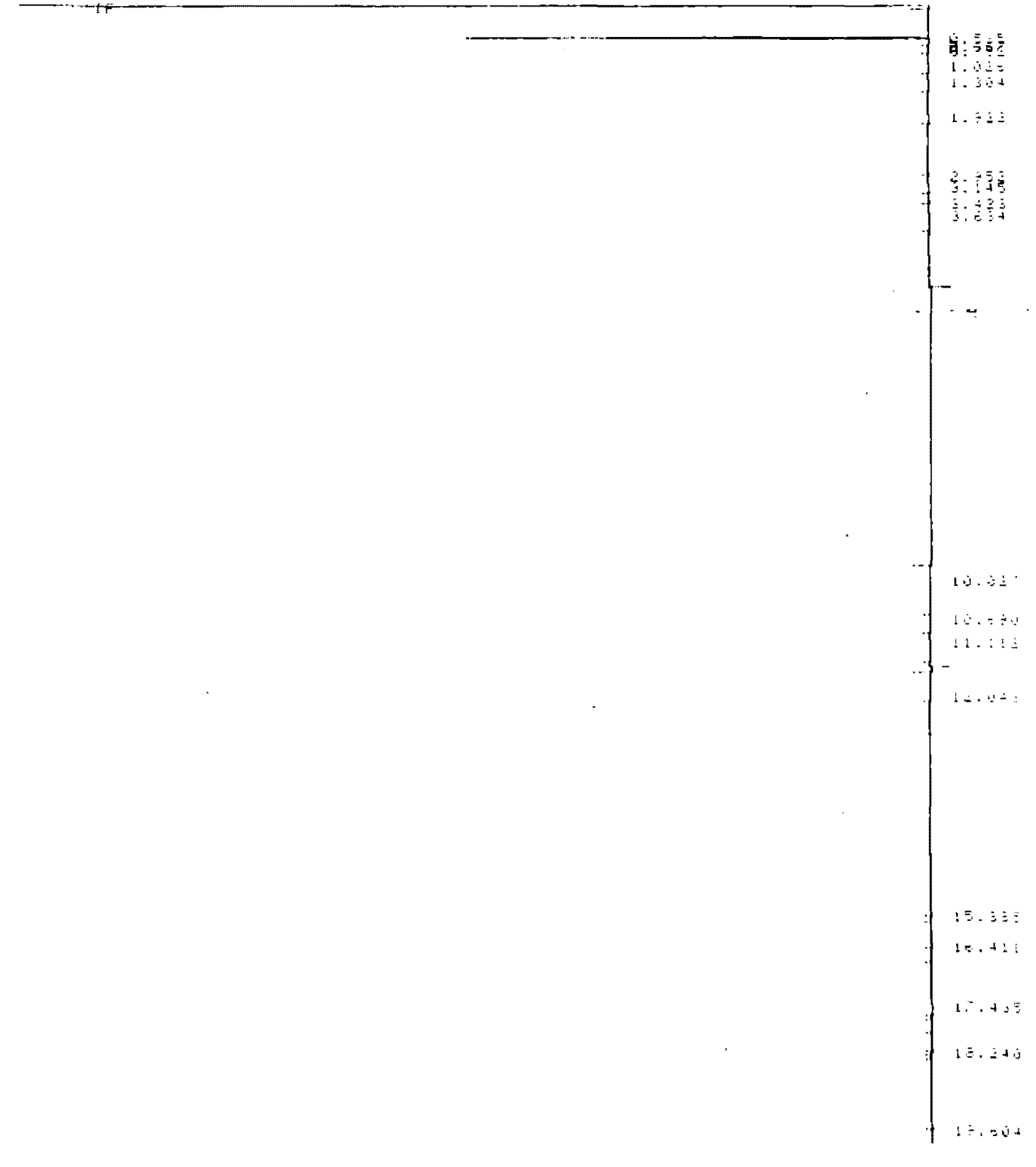
```
*   LIST: CALIB    0

NORM
REF % FTW:    5.000   NON-REF % FTW:   -5.000

LEVEL: 1              RECALIBRATIONS: 1

CAL#      RT      CV      AMT        AMT AREA
   1F     0.647    1  1.5000E+01   3.0876E-05
   2      0.914    1  1.4500E+01   1.3926E-05
   3      1.191    1  1.4200E+01   9.4026E-06
   4      3.304    1  1.4600E+01   6.4132E-06
   5      4.944    1  1.4600E+01   5.4065E-06
   6     11.045    1  1.4500E+01   4.6559E-06
   7      9.933    1  1.9900E+01   1.9350E-06
   8     14.395    1  2.0340E+01   4.1340E-06
   9     17.966    1  2.0860E+01   3.3860E-06
  10     33.615    1  2.1080E+01   1.3750E-06

CAL#      NAME
   1  METHANE
   2  ETHANE
   3  PROPANE
   4  BUTANE
   5  PENTANE
   6  HEXANE
   7  BENZENE
   8  TOLUENE
   9  ETH-BENZENE
  10  XYLENE
```

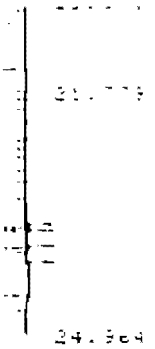
[illegible] ..... F
[illegible] ..... NO
MUL FACTOR ..... 1.0000E+00

* LIST: LIST
PEAK CAPACITY: 1331

DEPO = 0, -10.877
ATT 2' = -1
CNT SP = 1.0
HR REJ = 0
THRSH = 1
FR WD = 0.03

* RUN # 39 SEP 20, 1991 03:01:16
ETHRT





TIMETABLE STOP

RUN# 39 SEP 20, 1991 08:01:18

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.535	BB	189175	.246	12829		15.311	
.667	BB	443262	.090	83176	1R	36.043	METHANE
.752	BB	205870	.069	50349	2	7.699	ETHANE
1.036	BB	245233	.160	25617		19.933	
1.304	BB	171669	.108	26429	3	4.353	PROPANE
1.432	BB	47643	.094	8476		3.874	
3.145	BB	87602	.141	10345		7.139	
3.433	BB	4113	.113	612	4	.069	BUTANE
3.634	BB	47744	.173	4607	5	.680	PENTANE
10.017	BB	77625	.256	5060	7	.805	BENZENE
10.690	BB	3545	.037	1151		.207	
11.113	BB	6171	.131	939	6	.076	HEXANE
13.043	PB	5416	.233	338		.440	
16.411	PB	245	.035	116	8	.003	TOLUENE
17.435	PB	9613	.304	785		.782	
18.340	PB	373	.046	136	9	.002	ETH-BENZENE
19.604	PB	7630	1.124	113		.620	
20.176	BB	4308	.455	107		.342	
20.577	BB	449	.230	47		.053	
21.779	BB	247	.055	75		.030	
24.964	BP	2e1824	.245	10779	10	1.569	XYLENE

TOTAL AREA=1313041
MUL FACTOR=1.0000E+00

* ZERO ? ? ? BREAK

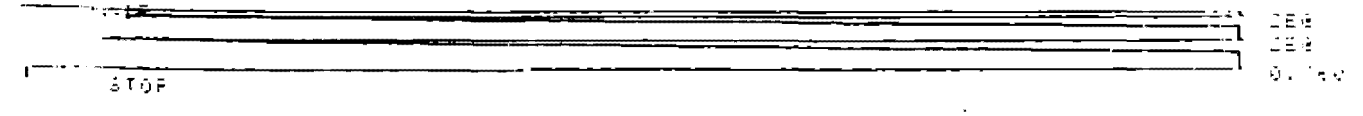
* LIST ZERO = 0. -10.877

* LIST ZERO = 0. -10.875

*

* RUN # 40 SEP 20, 1991 08:30:29

ETHET



RUN# 40 SEP 20, 1991 08:30:29

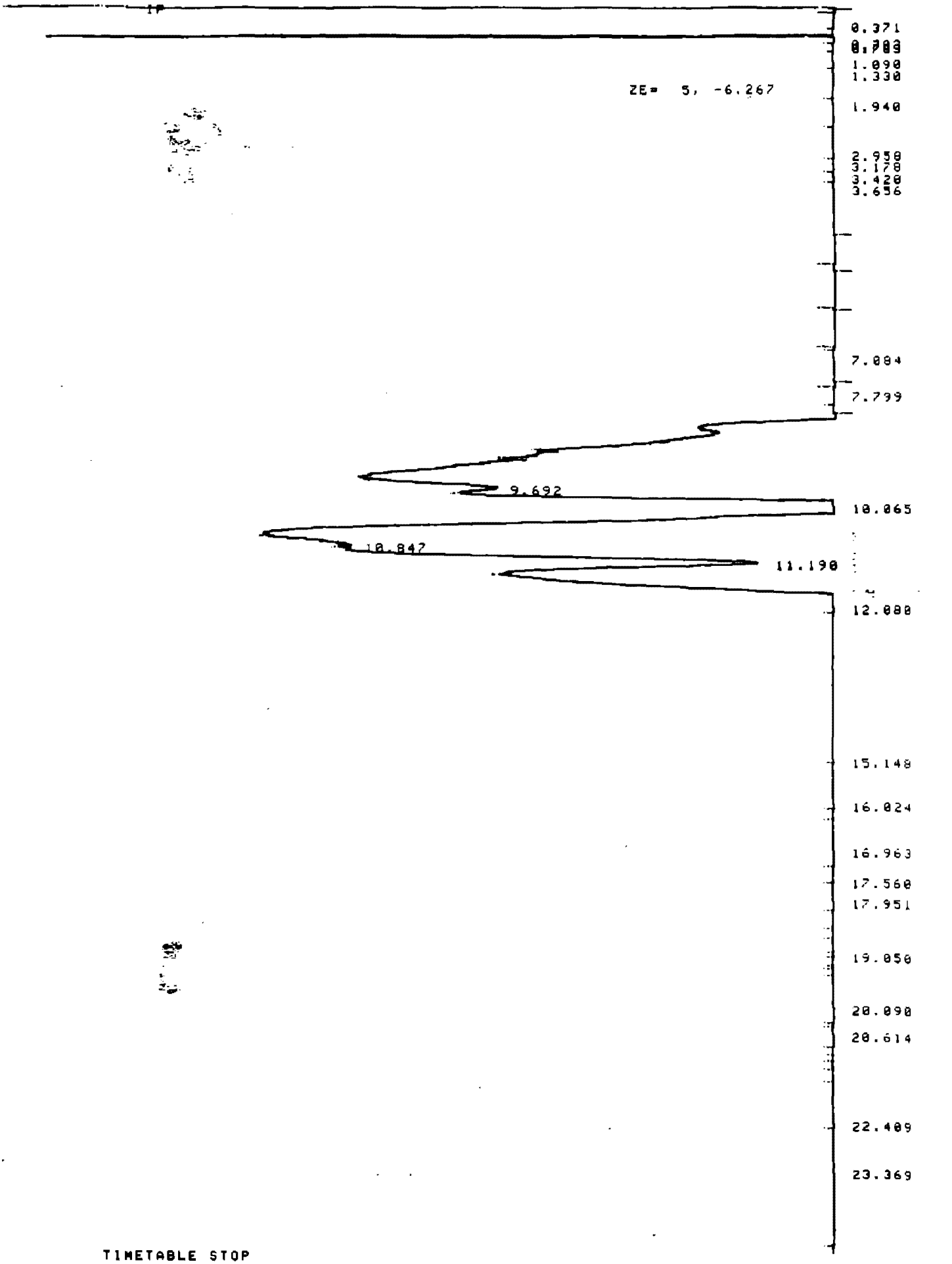
```
      RT TYPE        AREA   WIDTH      HEIGHT CAL# CONC.        NAME
    .768 I BP      700963    .365       32027   2      100.000  ETHANE

TOTAL AREA= 700963
MUL FACTOR=1.0000E+00


* RUN #    41      SEP 20, 1991   08:31:48
START
   STOP1F


RUN#     41           SEP 20, 1991   08:31:48

NO RUN PEAKS STORED


*  ZERO   BREAK

*  LIST: ZERO =  0, -3.642

*  PLOT

     STOP2E0

*  LIST: ZERO =  0, -1.463

*ATT 2^   BREAK

*  LIST: ZERO =  0, -1.279

*  LIST: ZERO =  0, -1.067

*  ZERO 10  0
*  PLOT

STOP

*  LIST: ZERO =  10, -0.706

*  PLOT


 ZE=  10, -2.545

                                                ZE=  10, -0.035
 ZE=  10, -0.971  10, -0.787
 ZE=0 0, -1.651
 ZE=0 5, -2.238

 ZE=  5, -3.484
 ZE=  5, -7.530
 ZE=0 0, -7.987
                  STOP

*  LIST: ZERO =  0, -0.304
```

```
*  PLOT
 ZE=  5; -8.928ZE5E
                  STOP

*p  BREAK

*P

INVALID SYSTEM COMMAND

*
```

```
*
*  OP # 5

POST-RUN LIST OPTIONS
Store post-run report [Y/N*]:
External post-run report [Y/N*]:
List run parameters [Y/N*]: Y
List timetable [Y/N*]: N
List calibration table [Y/N*]:
List remote method [Y/N*]:
Form-feed before report [Y/N*]:
Form-feed after report [Y/N*]: Y
Skip perforations in report [Y/N*]:

*
```

* RUN # 42 SEP 26, 1991 08:48:49

START



TIMETABLE STOP

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.371	BB	1676	.195	143		.322	
.703	PB	141244	.150	15740		27.097	
.783	BB	263013	.069	63267	2	22.766	ETHANE
1.090	BB	170303	.170	15931	3	9.952	PROPANE
2.958	BB	4426	.036	2023		.849	
3.178	BB	36497	.130	4695		7.002	
3.420	BB	8504	.102	1392	4	.339	BUTANE
3.656	BB	34667	.127	4533		6.651	
7.084	VB	34192	.334	1704	5	1.149	PENTANE
7.799	BB	3876	.163	397		.744	
9.692	PB	3226	.144	373		.619	
10.065	BB	100418	.298	5610	7	2.456	BENZENE
10.847	BB	578	.091	106		.111	
11.190	BB	21934	.207	1763	6	.635	HEXANE
12.080	BB	2613	.864	679		.501	
16.024	BB	50061	.830	1005	8	1.302	TOLUENE
16.963	PB	27054	.304	1482		5.190	
17.560	BB	1480	.224	110		.284	
17.951	BB	11710	.298	655		2.247	
19.050	PB	3863	.153	420	9	.057	ETH-BENZENE
20.096	PB	18084	.443	680		3.469	
20.614	VB	5430	.268	338		1.042	
22.409	PB	11911	.804	247		2.285	
23.369	BB	207496	.986	3506	10	2.934	XYLENE

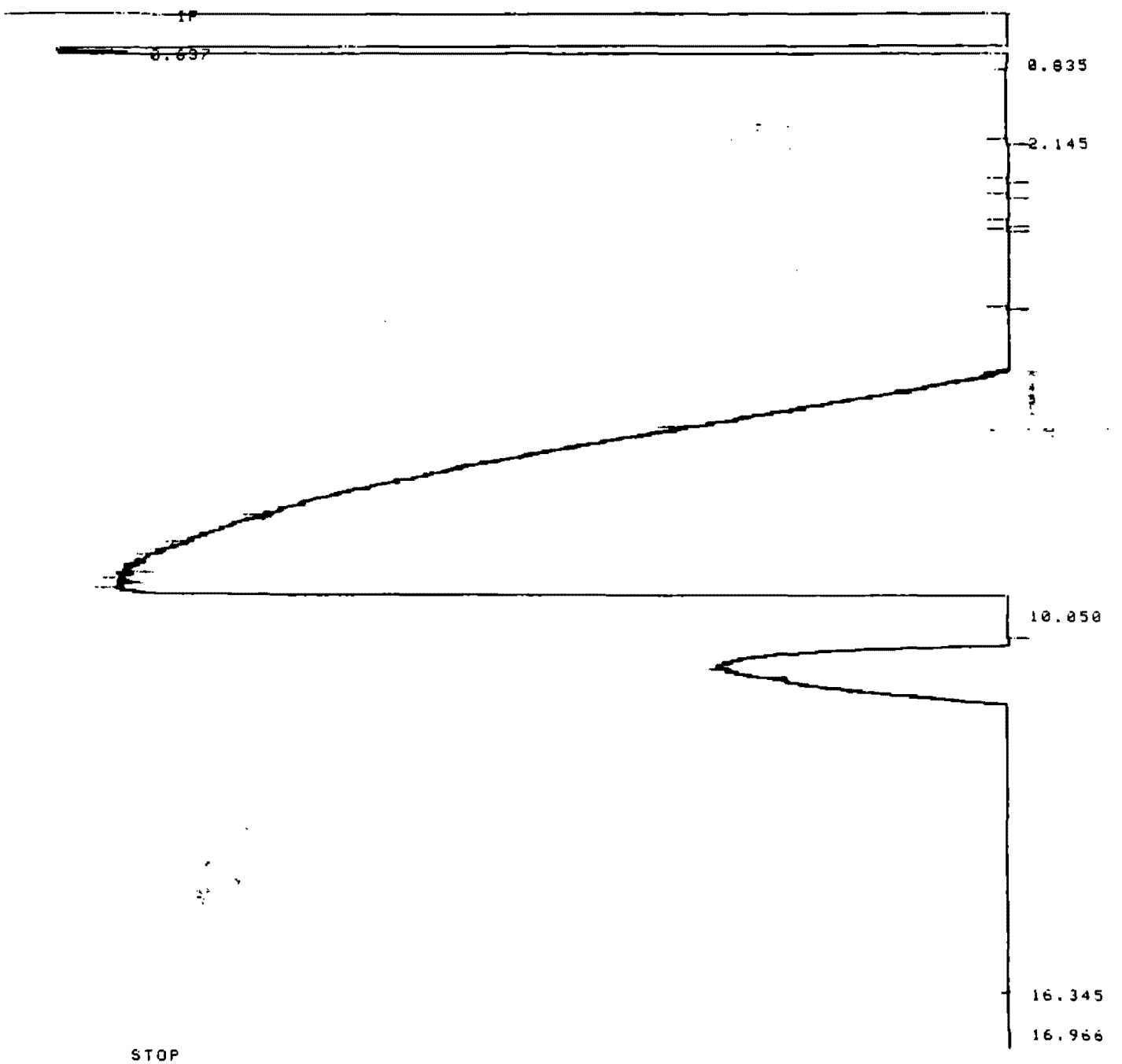
TOTAL AREA=1164256
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 5
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

Q6 102 ppm BENZENE

* RUN # 43 SEP 20, 1991 09:30:06

START



STOP

RUN# 43 SEP 20, 1991 09:30:06

NORM-AREA

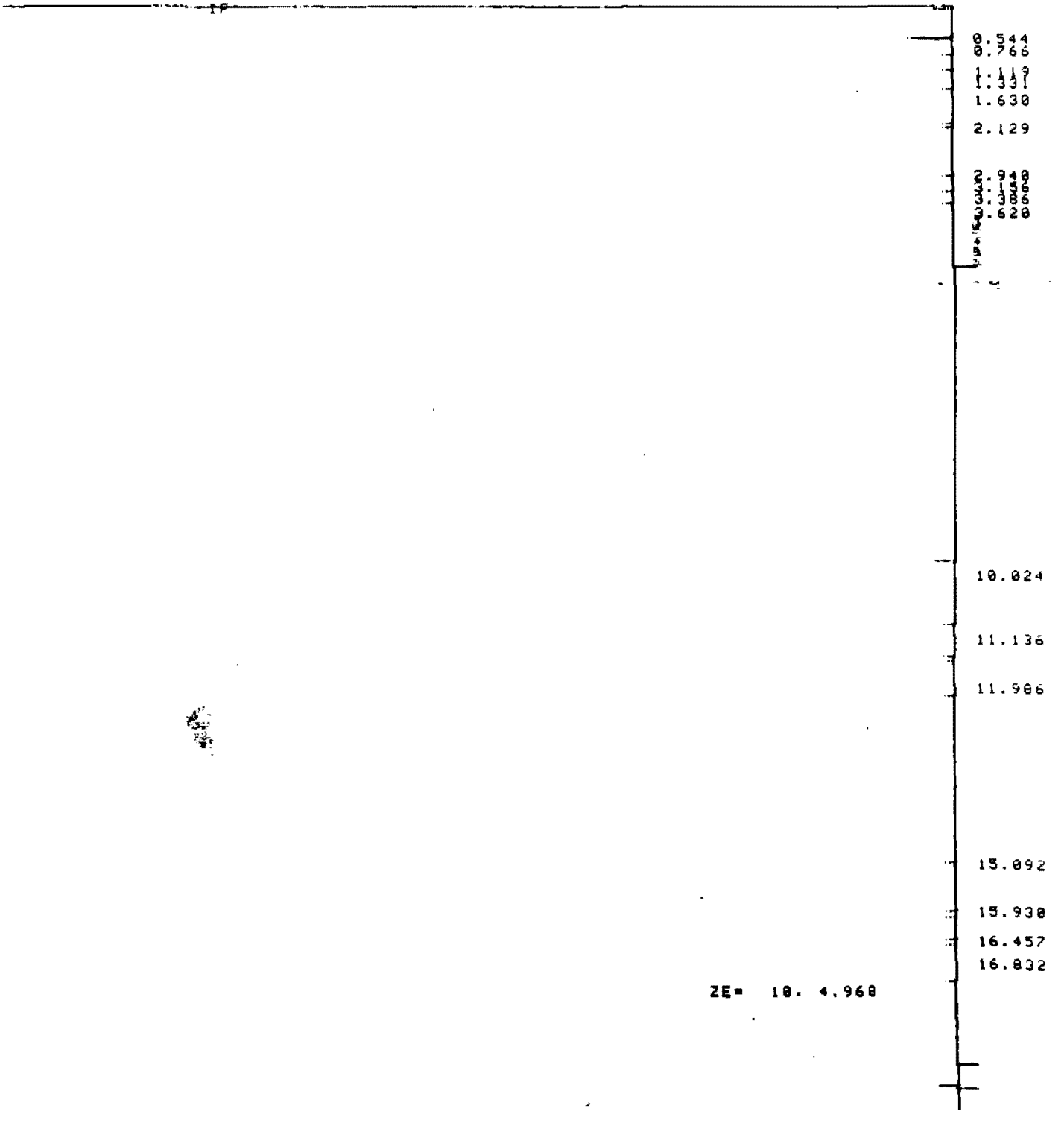
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.637	BB	20539	.057	6038	1R	.688	METHANE
.835	BB	131776	.287	7644	2	1.969	ETHANE
2.145	BB	1532	.026	965	3	.015	PROPANE

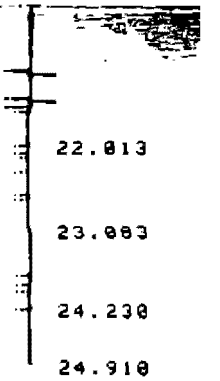
TOTAL AREA=2.3238E+07
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 5
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

SAMPLE #3

```
*  LIST: ZERO =   5, -10.875

*ZERO    10  @
*  LIST: ZERO =   10, -10.876

*  RUN #    44      SEP 20, 1991   09:52:20
START
```





TIMETABLE STOP

RUN# 44 SEP 20, 1991 09:52:20

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.544	BB	398413	.265	25069		23.770	
.766	BB	937423	.098	159617	2	25.234	ETHANE
1.119	BB	29945	.232	2154		1.787	
1.331	BB	225877	.131	28777		13.476	
1.630	BB	40894	.085	7894	3	.729	PROPANE
2.129	BB	842	.028	497		.050	
3.156	BB	48154	.135	5946		2.873	
3.386	BB	14482	.110	2197	4	.179	BUTANE
3.620	BB	106376	.363	4882	5	1.112	PENTANE
10.024	BB	92217	.213	7218	7	.781	BENZENE
11.136	BB	27950	.220	2113	6	.252	HEXANE
11.996	PB	17912	.231	1291		1.069	
15.930	BB	59229	1.723	573	8	.479	TOLUENE
16.832	VB	17464	.318	916	9	.081	ETH-BENZENE
22.813	PB	1219	.207	98		.073	
23.083	VB	52476	.685	1445	10	.231	XYLENE
24.230	PB	680	.155	73		.041	
24.910	I BP	467046	.416	18716		27.865	

TOTAL AREA=2537798
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.00

SAMPLE #4

* RUN # 45 SEP 28, 1991 10:25:02
START

IF

0.509
0.568
1.006
1.294

1.898

3.118
3.590

9.590

11.125

11.975

16.140
16.628
16.890
17.315

19.984

20.590

22.425

22.989

TIMETABLE STOP

RUN# 45 SEP 20, 1991 10:25:02

NORM-AREA

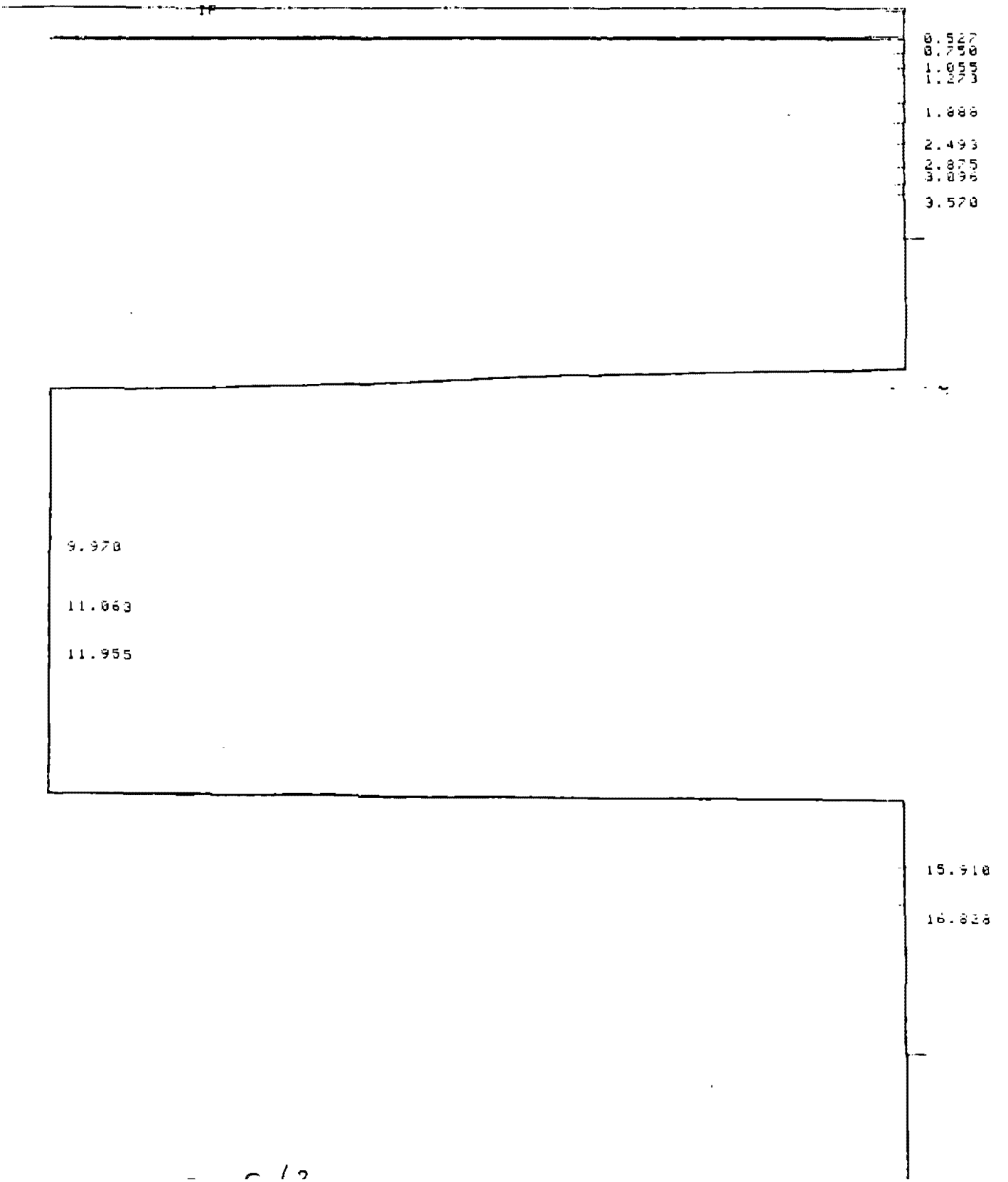
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.509	BB	412519	.173	39760		36.967	
.672	BB	186893	.079	39419	1R	16.751	METHANE
.763	BB	197526	.066	50116	2	7.986	ETHANE
1.006	BB	301008	.159	31624		26.974	
1.294	BB	4426	.004	19638		.397	
1.898	BB	1006	.003	6357	3	.028	PROPANE
3.110	BB	65006	.134	9102		5.825	
3.590	PB	11089	.061	3008	4	.206	BUTANE
9.990	BB	42569	.161	4415	7	.486	BENZENE
11.125	BB	15902	.186	1425		1.425	
11.975	BB	12457	.225	922	6	.168	HEXANE
16.628	BB	2157	.455	79	8	.026	TOLUENE
16.890	BB	4851	.218	371		.435	
17.315	BB	1422	.121	196		.127	
19.984	PB	3340	.261	213	9	.023	ETH-BENZENE
20.590	PB	12321	.412	498		1.104	
22.425	PB	11866	2.747	72		1.063	
22.989	PB	1137	.188	101	10	.008	XYLENE

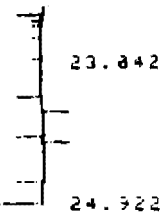
TOTAL AREA=1387496
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

Sample #5

* RUN # 46 SEP 20, 1991 10:55:34
START





TIMETABLE STOP

RUN# 46 SEP 20, 1991 10:55:34

NORM-AREA

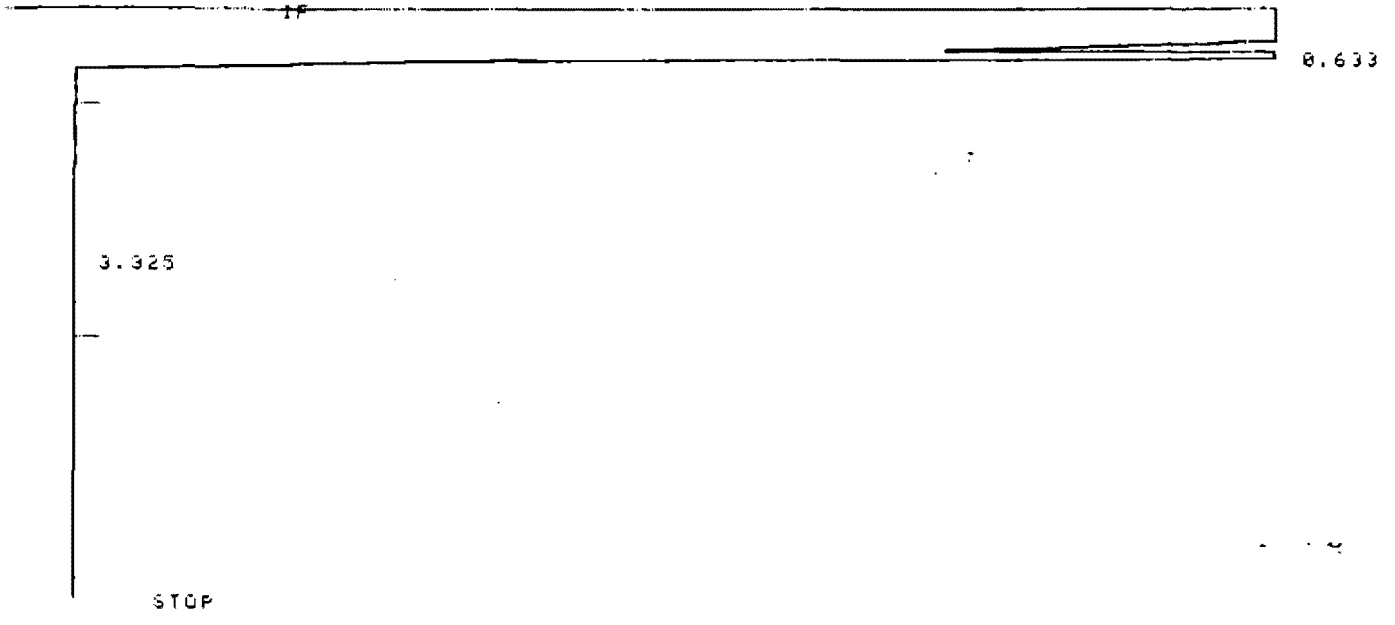
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.527	BB	697183	.262	44357		46.959	
.750	BB	897987	.095	157869	2	27.289	ETHANE
1.055	BB	49510	.123	6687		3.335	
1.273	BB	55296	.037	24957		3.724	
1.888	BB	20424	.124	2751	3	.419	PROPANE
2.493	BB	1404	.152	154		.095	
2.875	BB	20576	.177	1941		1.386	
3.096	BB	35958	.131	4590		2.422	
3.570	VB	55444	.210	4405	4	.776	BUTANE
9.970	BB	5694	.032	2951	7	.049	BENZENE
11.063	BB	5491	.115	799	6	.056	HEXANE
11.955	BB	5803	.194	498		.391	
16.828	VB	147259	1.138	2156	8	1.344	TOLUENE
23.042	VB	11596	.219	861	10	.058	XYLENE
24.922	I BP	173680	.198	14593		11.698	

TOTAL AREA=2183306
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

METHANE
CAL .198 ppm

* RUN # 47 SEP 20, 1991 11:29:36
START



RUN# 47 SEP 20, 1991 11:29:36

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.633	BB	63842	.125	8505	1R	73.968	METHANE
3.325	BB	108173	.127	14247	4	26.032	BUTANE

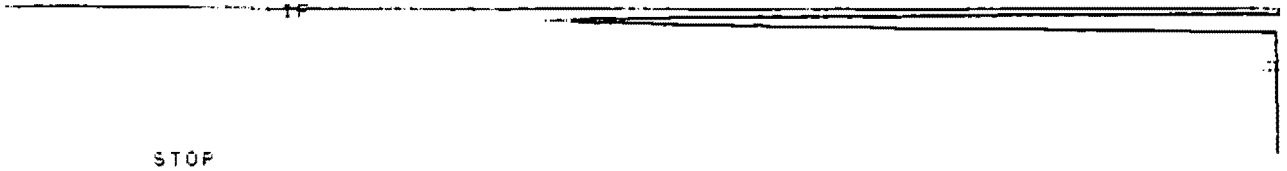
TOTAL AREA= 172015
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

```
>  RUN #    48      SEP 20, 1991   11:43:45
START
```

```
        STOP


RUN#     48           SEP 20, 1991   11:43:45

NORM-AREA
      RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
    .658   PB       22054   .049        7555   1R     100.000  METHANE


TOTAL AREA=  22054
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  10
ATT Z   = -1
CHT SP  =  1.0
AR REJ  =  0
THRSH   =  1
PK WD   =  0.08
```

```
*  RUN #    49      SEP 20, 1991  11:47:14
START
```

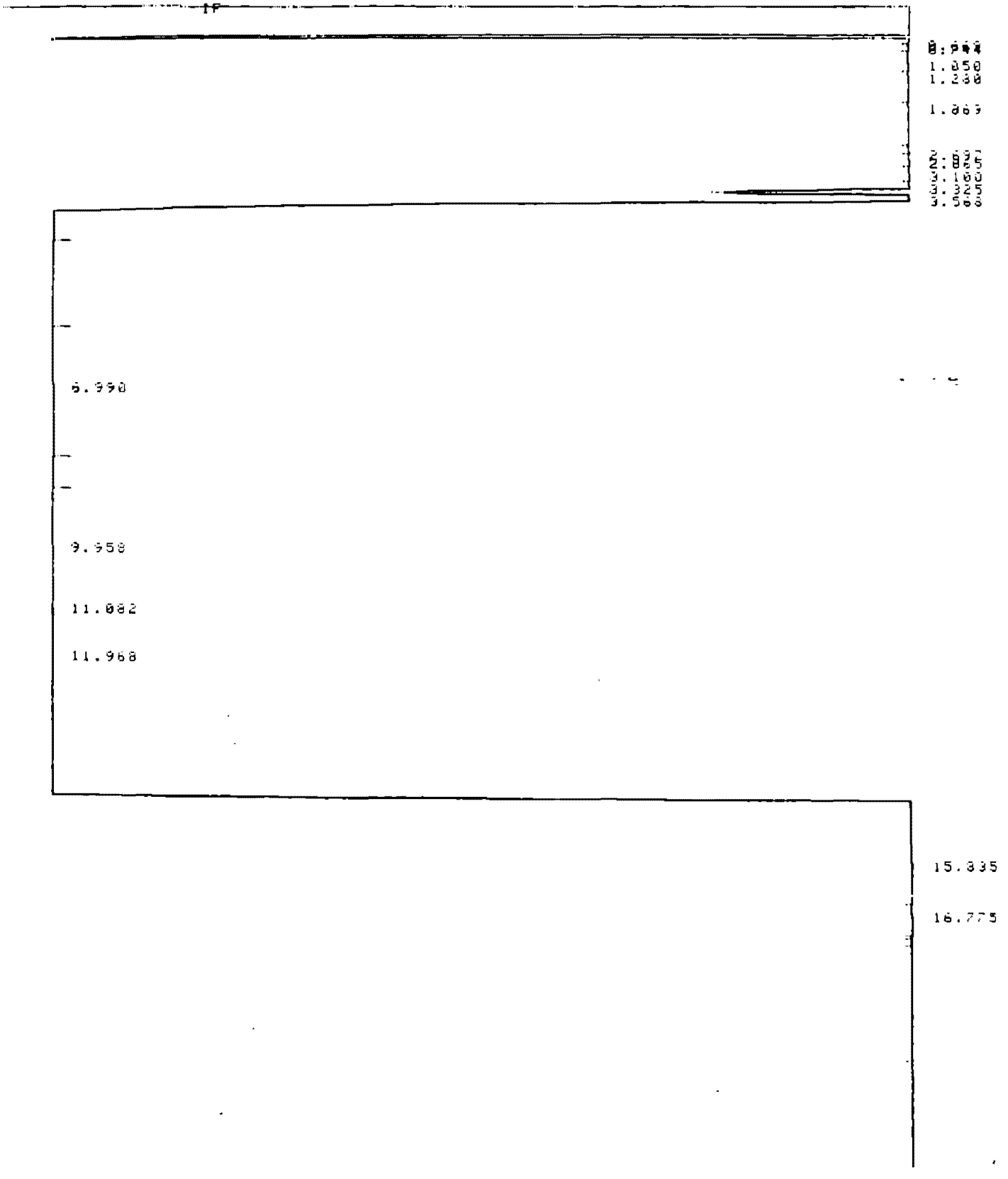


```
        STOP


RUN#     49           SEP 20, 1991  11:47:14

NORM-AREA
       RT TYPE        AREA  WIDTH      HEIGHT CAL# CONC.        NAME
     .649   PB       49927   .093        8515   1R      63.630  METHANE
     .792   BB         949   .081         196   2         .546  ETHANE
    1.260  I BP       92102   .730        2102   3       35.774  PROPANE


TOTAL AREA= 142978
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  10
ATT 2^  = -1
CHT SP  =  1.0
AR REJ  =  0
THRSH   =  1
PK WD   =  0.08
```

AMRE #6

* RUN # 50 SEP 20, 1991 11:51:18

START



TIMETABLE STOP

RUN# 50 SEP 20, 1991 11:51:18

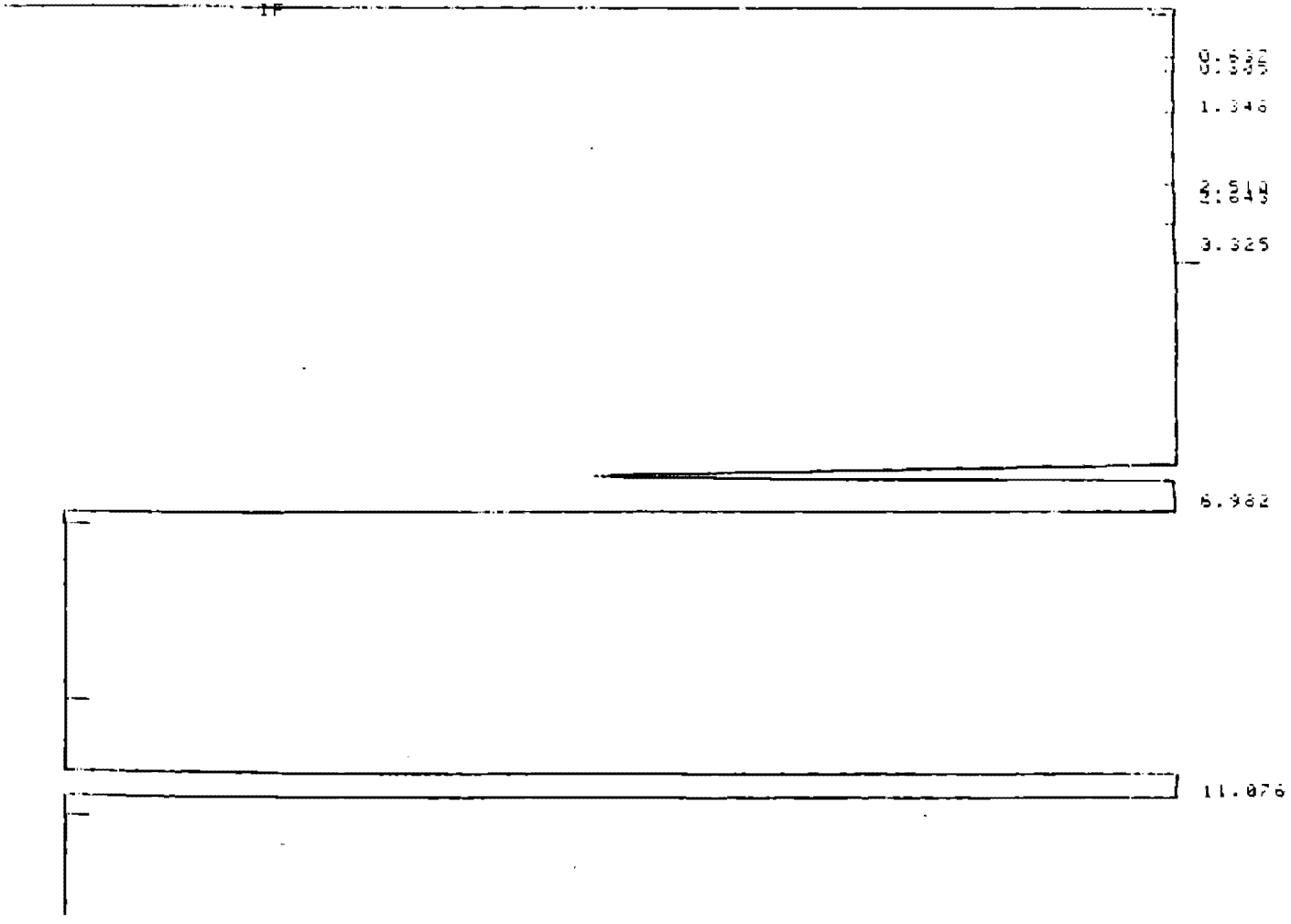
NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.669	PB	241818	.103	39050	1R	26.023	METHANE
.744	BB	208152	.066	57731	2	10.104	ETHANE
1.050	BB	219307	.199	19210	3	7.187	PROPANE
2.697	BB	1296	.142	152		.139	
2.865	BB	11731	.113	1723		1.262	
3.100	BB	30319	.127	3970		3.262	
3.325	BB	7211	.108	1110		.776	
3.568	BB	20936	.094	3696	4	.468	BUTANE
6.990	PB	18705	.229	1360	5	.352	PENTANE
9.958	VB	84495	.296	4762	7	1.159	BENZENE
11.682	PB	17448	.202	1439		1.877	
11.963	PB	8493	.199	712	6	.138	HEXANE
15.835	BB	436761	.572	12736		46.992	
16.775	BB	17537	.296	986	8	.256	TOLUENE
21.799	PB	589	.094	104	9	.005	ETH-BENZENE

TOTAL AREA=1324798
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.00

```
* EDIT CALIB @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 1


E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E/I/N*]: E


SECTION TO BE EDITED:

* RUN # 51 SEP 20, 1991 12:21:56
START
```



16.145

STOP

RUN# 51 SEP 20, 1991 12:21:56

ESTD-AREA

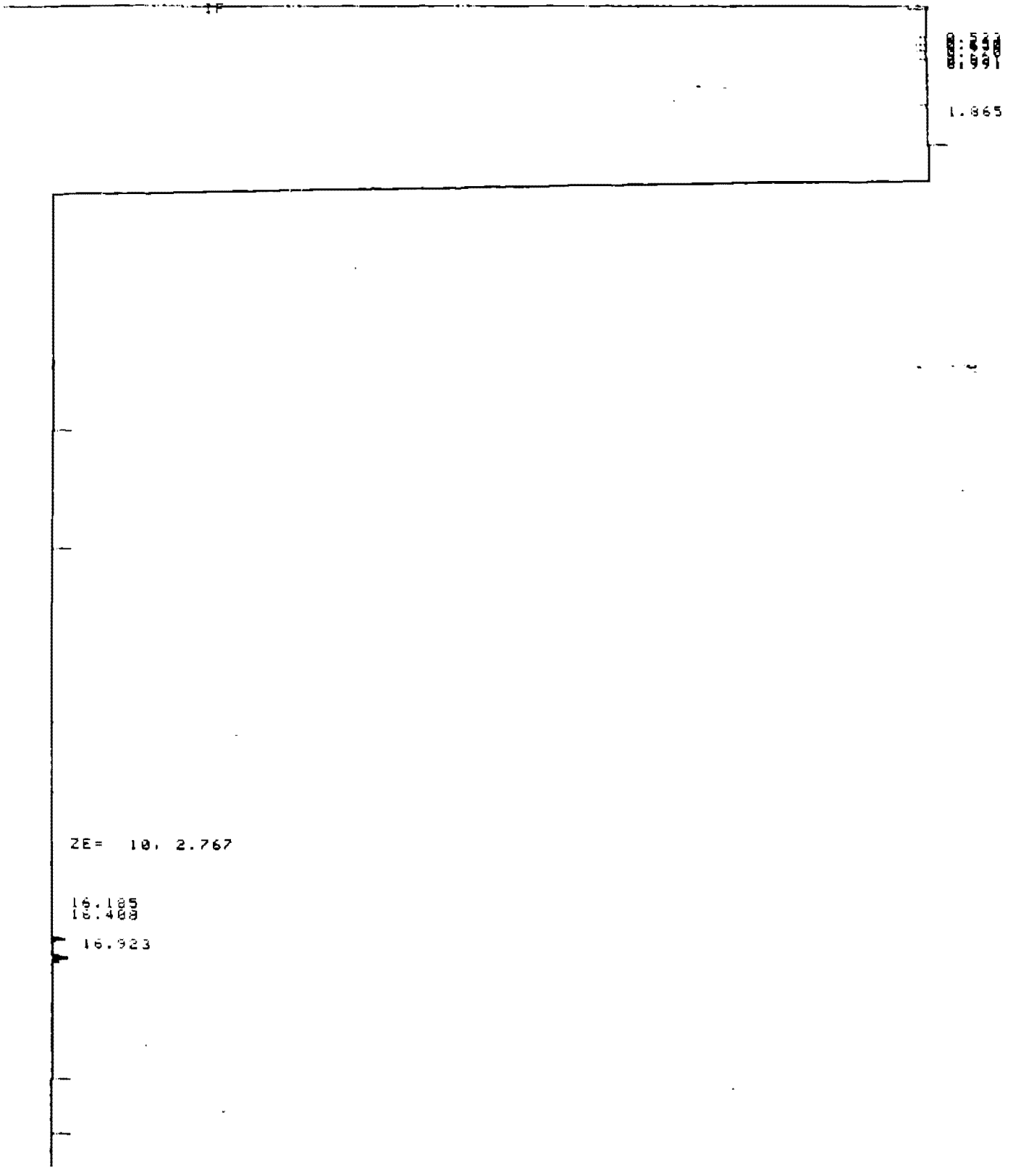
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.637	PB	492096	.083	98622	1R	15.194	METHANE
.805	BB	1004442	.085	196906	2	13.990	ETHANE
1.348	BB	1461869	.092	263489	3	13.745	PROPANE
2.510	BB	56078	3.769	248		1.731	
2.643	BB	14688	.398	615		.453	
3.325	BB	2077438	.158	219717	4	13.323	BUTANE
6.982	BB	2579718	.201	213673	5	13.347	PENTANE
11.076	BB	2990469	.222	224790	6	13.923	HEXANE

TOTAL AREA=1.0677E+07
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

EXAMPLE #7

*
* RUN # 52 SEP 20, 1991 12:46:00
START



TIMETABLE STOP

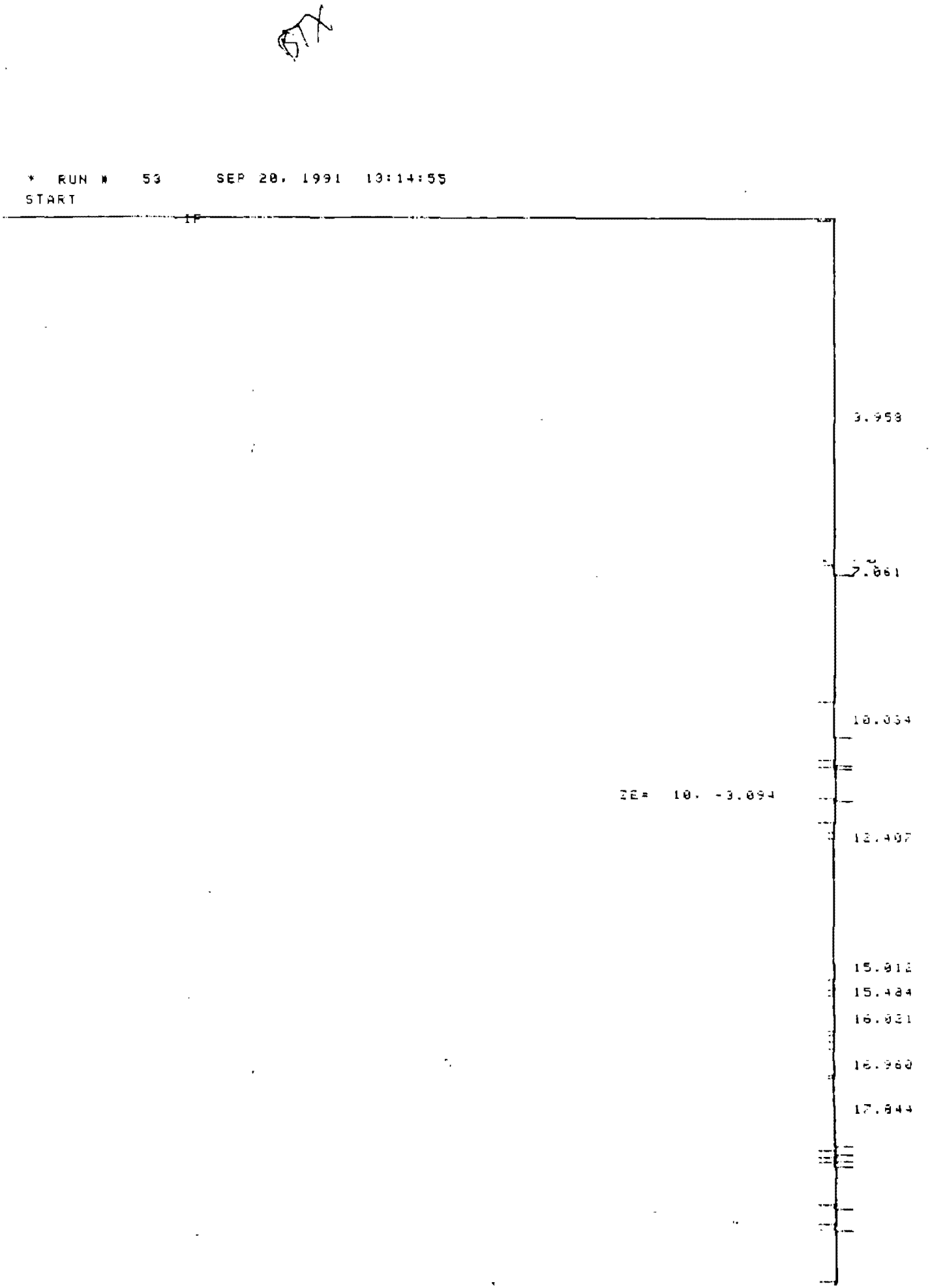
RUN# 52 SEP 20, 1991 12:46:00

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.523	PB	34630	.291	1982		1.069	
.650	BB	41578	.080	8632	1R	1.284	METHANE
.770	BB	42942	.025	28563	2	.598	ETHANE
.921	BB	17408	.096	3008	3	.164	PROPANE
16.408	BB	617	.085	121	8	.003	TOLUENE
16.923	BB	8055	.265	506	9	.019	ETH-BENZENE

TOTAL AREA= 145230
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 3
THRSH = 1
PK WD = 0.09


* RUN # 53 SEP 20, 1991 13:14:55
START
IF
3.958
7.061
10.034
ZE= 10. -3.094
12.407
15.012
15.484
16.031
16.960
17.844

TIMETABLE STOP

RUN# 53 SEP 20, 1991 13:14:55

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
3.958	PB	22245264	4.617	80300	4	142.662	BUTANE
7.061	BB	626136	.091	114878	5	3.385	PENTANE
10.034	BB	2102330	.211	166412	7	8.273	BENZENE
12.407	PB	1023	.078	218	6	.005	HEXANE
15.812	BB	1112613	.103	179642		34.346	
15.484	BB	2468	.146	281		.076	
16.021	PB	12706	.324	654		.392	
16.960	PB	9308	.265	585	8	.039	TOLUENE
17.844	VB	3819142	.371	171495	9	9.120	ETH-BENZENE
21.823	PB	2316	.142	272		.071	
23.082	VP	5499792	.889	103070	10	12.512	XYLENE

TOTAL AREA=3.5433E+07
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.09

```
*  LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:   -5.000

LEVEL: 1              RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1R     0.649     1  1.5000E+01   3.0876E-05
  2      0.782     1  1.4500E+01   1.3928E-05
  3      1.278     1  1.4200E+01   9.4026E-06
  4      3.424     1  1.4600E+01   6.4132E-06
  5      6.961     1  1.4600E+01   5.4065E-06
  6     11.466     1  1.4500E+01   4.6553E-06
  7     10.051     1  1.9900E+01   3.9350E-06
  8     16.532     1  2.0340E+01   4.1840E-06
  9     19.982     1  2.0860E+01   2.3880E-06
 10     23.361     1  2.1000E+01   2.2750E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   TOLUENE
  9   ETH-BENZENE
 10   XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.0870E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

*  DELETE  CALIB  @
DELETE ALL  (Y/N*): N

*  EDIT  CALIB   @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  8
RT:  5@15.012
```

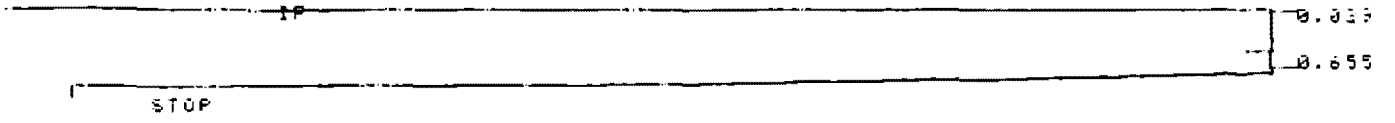
NAME: TOLUENE

CAL#:

SECTION TO BE EDITED:

METHANE ~10

* RUN # 54 SEP 20, 1991 13:48:54
START



RUN# 54 SEP 20, 1991 13:48:54

ESTD-AREA

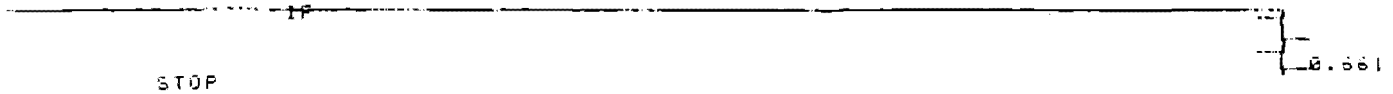
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.029	BB	2727	.023	1976		.084	
.655	BB	349734	.088	66219	1R	10.798	METHANE

TOTAL AREA= 352461
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2* = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
FR WD = 0.03

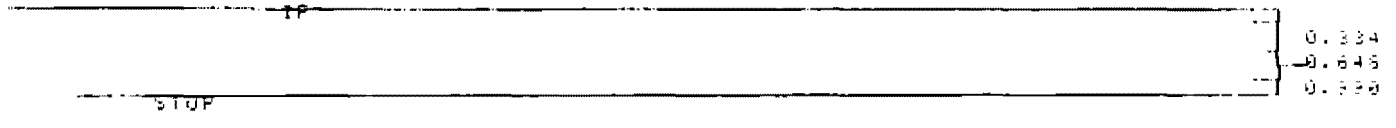
METH - 12

```
*  RUN #    55      SEP 20, 1991   13:52:30
START
```




```
        STOP


RUN#     55            SEP 20, 1991   13:52:30

ESTD-AREA
       RT TYPE        AREA  WIDTH      HEIGHT CHL# CONC.        NAME
     .661   BB      369861   .093       66175   IR      11.420  METHANE


TOTAL AREA= 369861
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  10
ATT 2   = -1
CHT SP  =  1.0
AR REJ  =  0
THRSH   =  1
PK WD   =  0.03
```

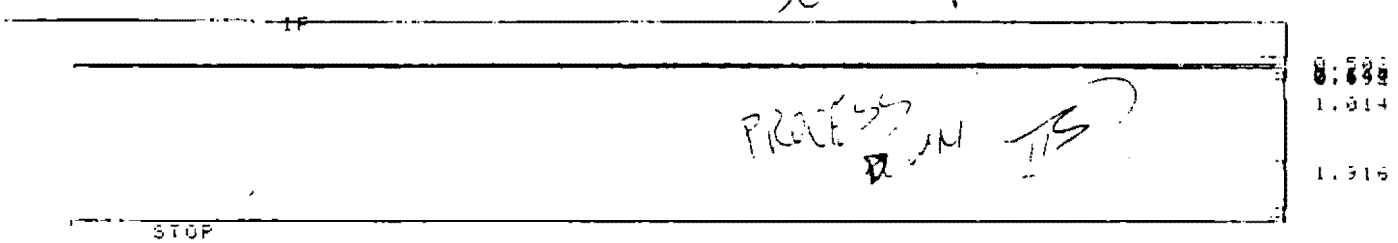
```
* EDIT CALIB @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 1


E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E*/I/N]: N


SECTION TO BE EDITED:

* RUN # 56 SEP 20, 1991 13:55:45
START
```




```
RUN# 56 SEP 20, 1991 13:55:45

NORM-AREA
      RT TYPE      AREA  WIDTH     HEIGHT CAL# CONC.       NAME
    .334   BB     24600   .198       2071          5.157
    .648   BB    352075   .088      66563   1R    73.926  METHANE
    .990 1 BP    222198   .380       9754   2     21.017  ETHANE

TOTAL AREA= 598873
MUL FACTOR=1.0000E+00




RUN PARAMETERS
ZERO   =  10
ATT 2  = -1
CHT SP =  1.0
AR REJ =  0
THRSH  =  1
PK WD  =  0.08
```

```
* EDIT CALIB @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 1


E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E/I/N*]: E


SECTION TO BE EDITED:

* RUN # 57 SEP 20, 1991 14:00:26
START
```

SCRIPED RUN

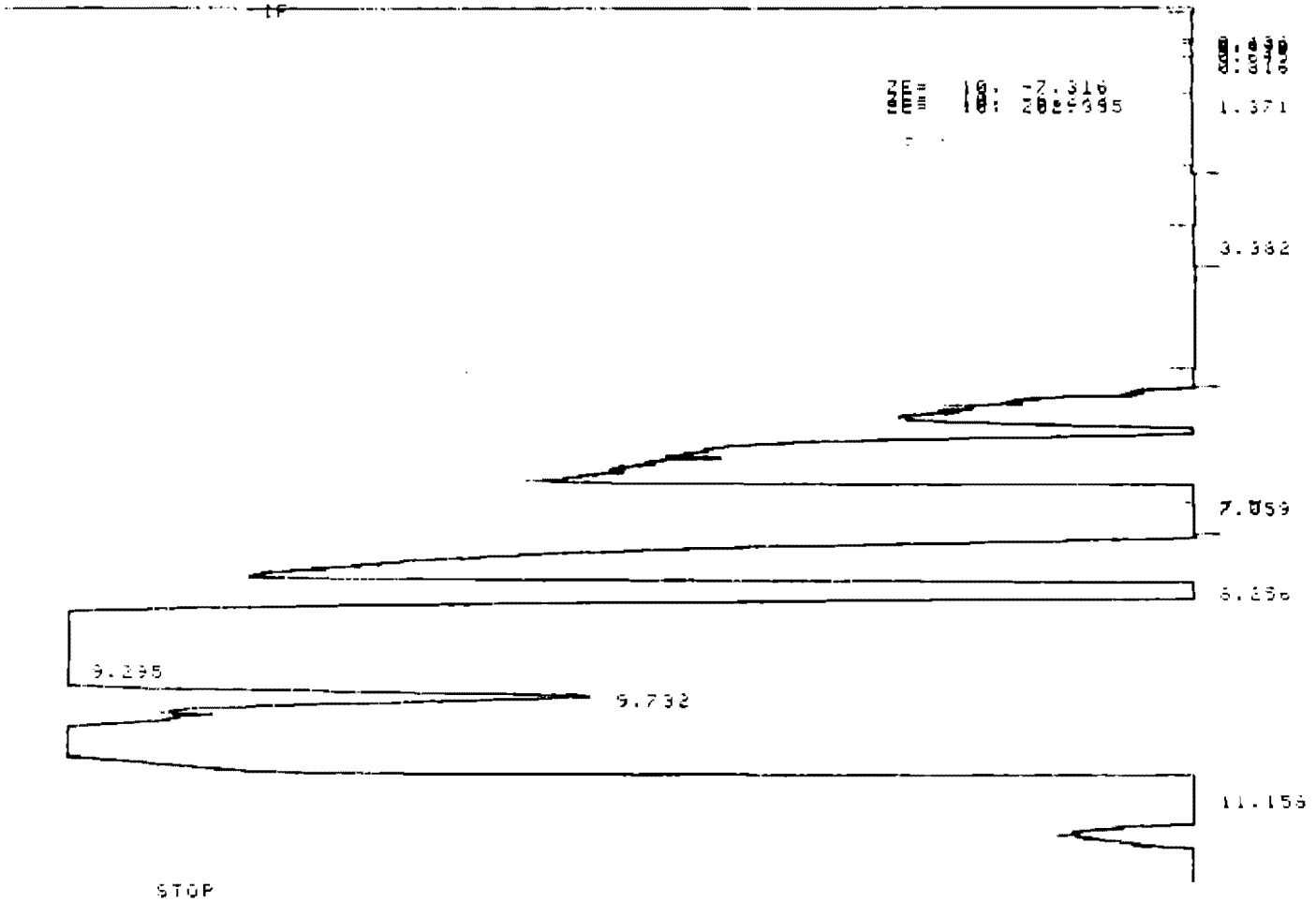



```
RUN# 57 SEP 20, 1991 14:00:26

ESTD-AREA
      RT TYPE      AREA  WIDTH     HEIGHT CAL# CONC.      NAME
    .502   BB     42357   .154       4580        1.308
    .644   BB     53358   .052      17021   1R   1.647  METHANE
    .692   BB     13707   .025       9169   2     .191  ETHANE
   1.014   BB   1165926   .434      44782   3   10.963  PROPANE
   1.916   BB      3753   .039       1587   4     .024  BUTANE

TOTAL AREA=1279101
MUL FACTOR=1.0000E+00


RUN PARAMETERS
ZERO   =  10
ATT 2^ = -1
```

THRSH = 1
FR WD = 0.03

C1-C6 ~100 ppmv

* RUN # 58 SEP 20, 1991 14:05:31
START



RUN# 58 SEP 20, 1991 14:05:31

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.436	BB	2509	.236	177		.077	
.496	BB	289	.033	146		.009	
.645	BB	3347253	.088	637333	1R	103.349	METHANE
.816	SPB	6795779	.088	1287059	2	94.650	ETHANE
1.371	SPB	10429224	.098	1779933	3	99.062	PROPANE
3.382	BB	13798136	.158	1452983	4	88.490	BUTANE
7.059	PB	17395760	.202	1435809	5	94.050	PENTANE
8.256	BB	120419	.208	9632		3.717	
9.295	PB	894	.213	70		.028	
9.732	PB	18367	.171	1795	7	.072	BENZENE
11.158	PB	21060208	.222	1582767	6	98.054	HEXANE

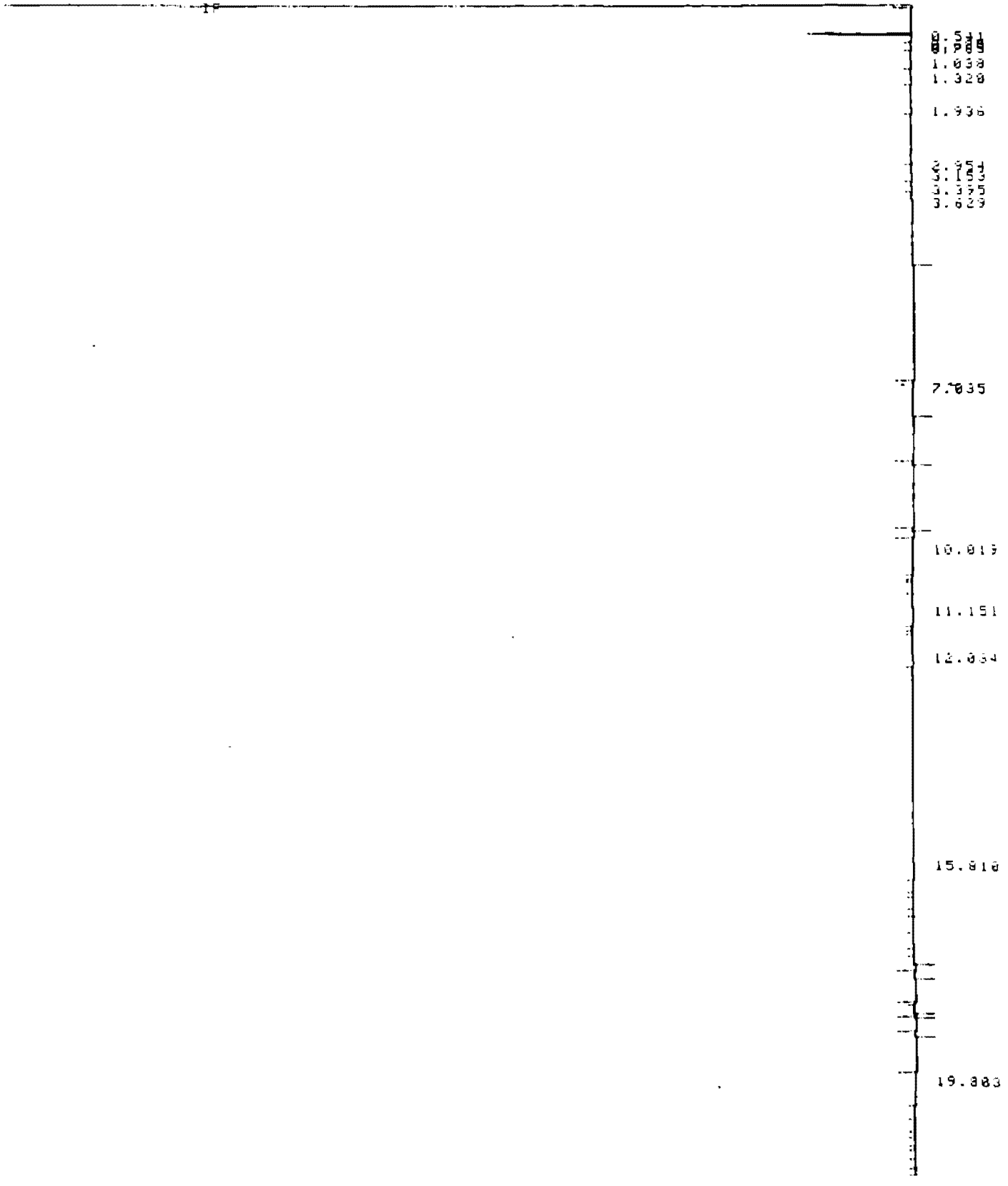
TOTAL AREA=7.2969E+07

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
FR WD = 0.00

SAMPLE #3

▸ RUN # 59 SEP 28, 1991 14:21:19

START



TIMETABLE STOP

RUN# 59 SEP 28, 1991 14:21:19

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.541	BB	204301	.260	13091		6.307	
.685	BB	181739	.117	25988		5.610	
.765	BB	241649	.071	56510	2	3.366	ETHANE
1.038	BB	204911	.159	21448		6.326	
1.320	BB	198453	.123	26995	3	1.866	PROPANE
1.936	BB	5655	.024	3881		.125	
3.153	BB	56250	.134	6971	4	.361	BUTANE
3.395	BB	10934	.106	1715		.338	
3.629	BB	144153	.439	5473		4.450	
7.035	BB	13226	.123	1115	5	.866	PENTANE
10.019	BB	66294	.257	4301	7	.261	BENZENE
11.151	VB	40445	.232	2907	6	.188	HEXANE
12.034	BB	18496	.249	1184	8	.143	TOLUENE
19.883	BB	11522	.392	583	9	.028	ETH-BENZENE

TOTAL AREA=1395328

MULT FACTOR=1.000000E+00

PEAK PARAMETERS

TDBL = 10

WTT 2? = -1

AMT SF = 1.0

WB BL1 = 0

THRSH = 1

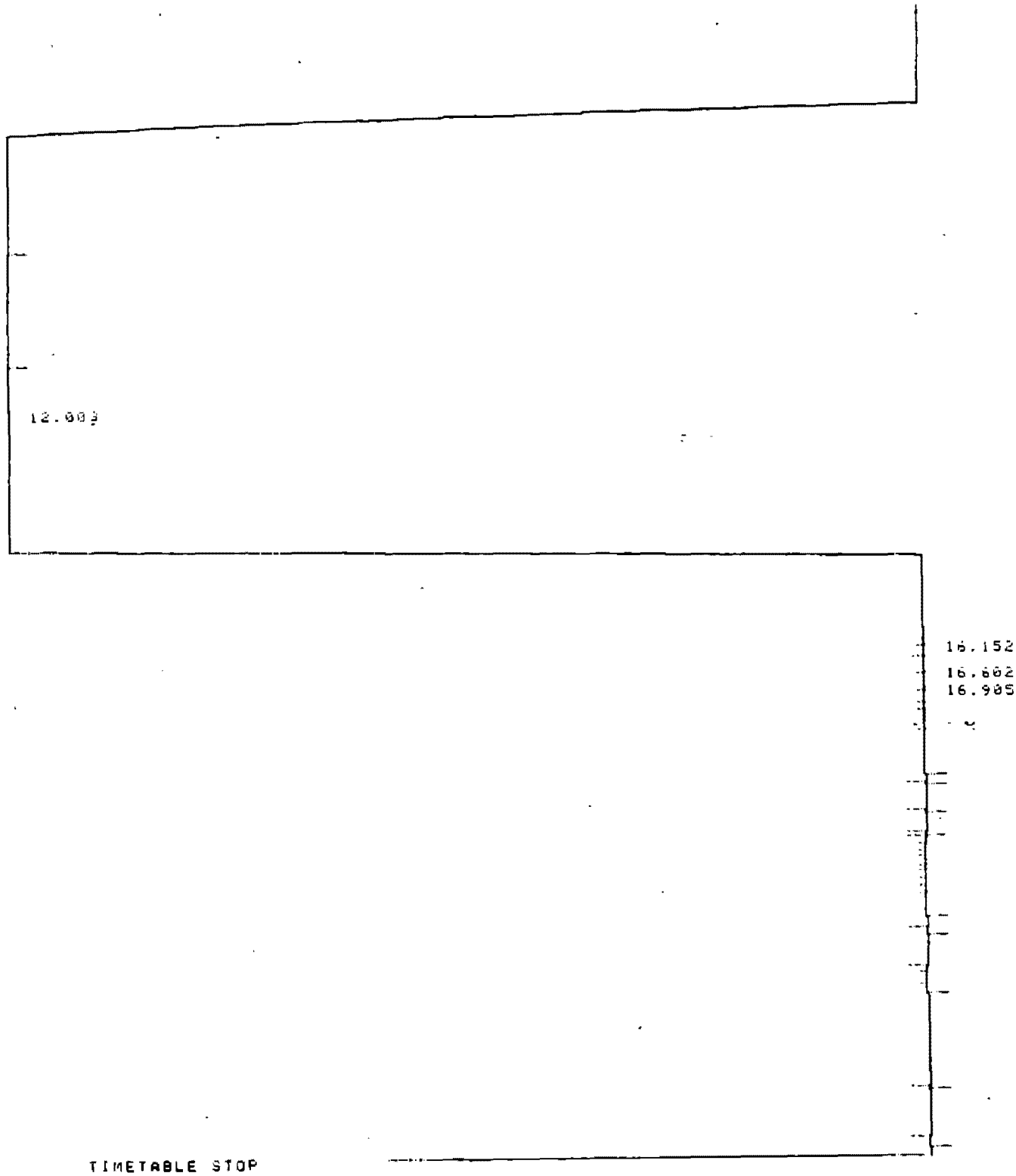
PK WD = 0.03

```
* LIST:
BREAK

* LIST: CALIB   @

ESTD
REF % RTW:    5.000  NON-REF % RTW:   -5.000

LEVEL: 1              RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1R     0.648     1  1.5000E+01   3.0876E-05
  2      0.803     1  1.4500E+01   1.3928E-05
  3      1.251     1  1.4200E+01   9.4026E-06
  4      3.130     1  1.4600E+01   6.4132E-06
  5      7.302     1  1.4600E+01   5.4065E-06
  6     11.389     1  1.4500E+01   4.6559E-06
  7      9.972     1  1.9900E+01   3.3350E-06
  8     15.012     1  9.6600E+00   3.6820E-06
  9     19.982     1  2.0860E+01   2.3880E-06
 10     23.361     1  2.1080E+01   2.2750E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   TOLUENE
  9   ETH-BENZENE
 10   XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks .... 3.0870E-05
Calibration fit ............. P
Disable post-run RT update .. NO
SAMPLE AMT .................. 0.0000E+00
MUL FACTOR .................. 1.0000E+00

* RUN #   60     SEP 20, 1991  14:51:44
START
```

SAMPLE #9

IF

0.525
0.678
0.993
1.300
1.931
2.401
2.685
3.123
3.596



RUN# 60 SEP 20, 1991 14:51:44

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.535	BB	547162	.260	35113		16.891	
.672	BB	204524	.115	29646	1R	6.315	METHANE
.767	BB	287048	.069	69639	2	3.998	ETHANE
.998	BB	170228	.161	17591		9.255	
1.300	BB	106140	.098	17981	3	.998	PROPANE
1.921	PB	21608	.124	2918		.667	
2.401	PB	6748	.230	489		.208	
2.885	VB	13806	.132	1741		.426	
3.123	BB	34088	.138	4103	4	.219	BUTANE

16.905	88	1187	.250	79	7	.003	ETH-BENZENE

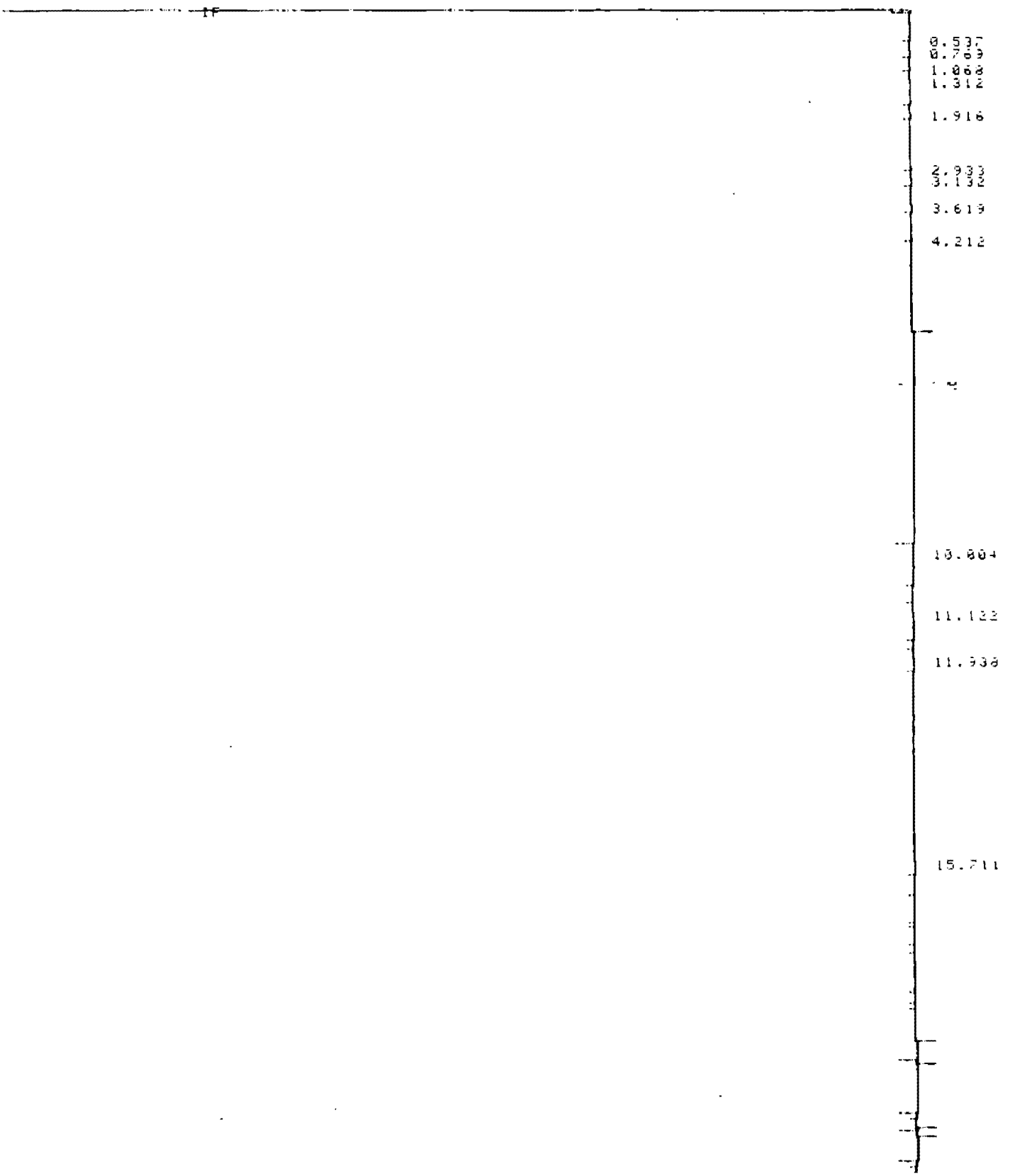
TOTAL AREA=1418497
MUL FACTOR=1.0000E+00

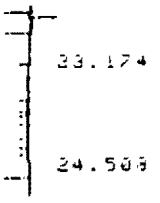
RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

SAMPLE #10

* RUN # 61 SEP 20, 1991 15:20:05

START





TIMETABLE STOP

RUN# 61 SEP 20, 1991 15:20:05

ESTD-AREA

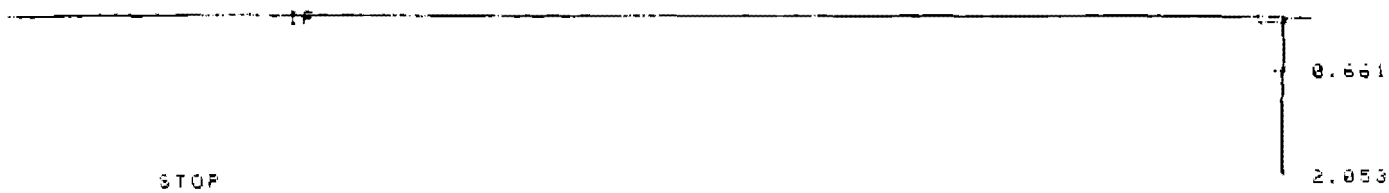
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.537	BB	301424	.256	19645		9.305	
.769	BB	1070322	.112	158859	2	14.907	ETHANE
1.068	BB	40156	.102	6593		1.240	
1.312	BB	360005	.178	33774	3	3.385	PROPANE
1.916	BB	22814	.113	3355		.704	
3.132	BB	48115	.132	6058	4	.309	BUTANE
3.619	BB	42179	.163	4305		1.302	
4.213	BB	8897	.494	300	5	.043	PENTANE
10.004	BB	39591	.200	3293	7	.156	BENZENE
11.122	PB	9693	.156	1036	6	.045	HEXANE
11.938	VB	7236	.210	574	8	.063	TOLUENE
23.174	BB	3477	.325	258	10	.008	XYLENE
24.508	I PB	1255	.082	255		.039	

TOTAL AREA=1955163
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2 = -1
CHT SP = 1.0
AF REJ = 0
THRSH = 1
PK WD = 0.08

Methane Cal .996 ppm

* RUN # 62 SEP 20, 1991 15:50:27
START



STOP

RUN# 62 SEP 20, 1991 15:50:27

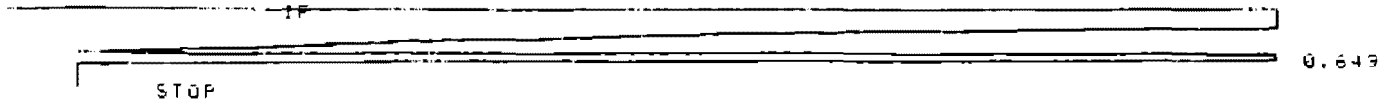
ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.661	PB	50277	.107	7847	1R	1.552	METHANE
2.053	I BP	1580205	.717	36740	3	14.858	PROPANE

TOTAL AREA=1630482
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2** = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

```
*  RUN #    63      SEP 20, 1991   15:53:53
START
```

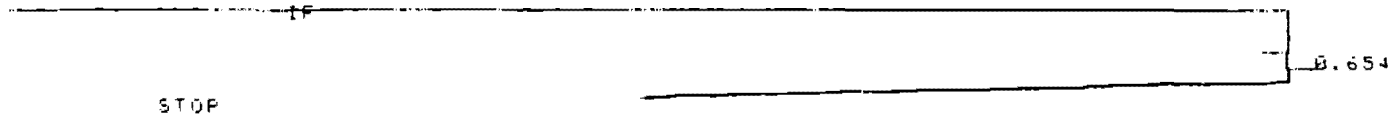



```
RUN#     63           SEP 20, 1991   15:53:53

ESTD-AREA
       RT TYPE        AREA   WIDTH      HEIGHT CAL# CONC.        NAME
     .649 I BP      529838    .622       14186    1R      16.359  METHANE


TOTAL AREA= 529838
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  10
ATT 2   = -1
CHT SP  =  1.0
AR REJ  =  0
THRSH   =  1
PK WD   =  0.08
```

METHANE CAL 9.96 ppmv

* RUN # 64 SEP 20, 1991 15:56:07
START

IF



STOP

RUN# 64 SEP 20, 1991 15:56:07

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.654	BB	349779	.089	65691	1R	10.000	METHANE

TOTAL AREA= 349779
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
HF REJ = 0
THRSH = 1
PK WD = 0.08

METHANE
CAL
9.96 ppm

* RUN # 65 SEP 20, 1991 16:00:17
START

IF

STOP

RUN# 65 SEP 20, 1991 16:00:17

ESTD-AREA

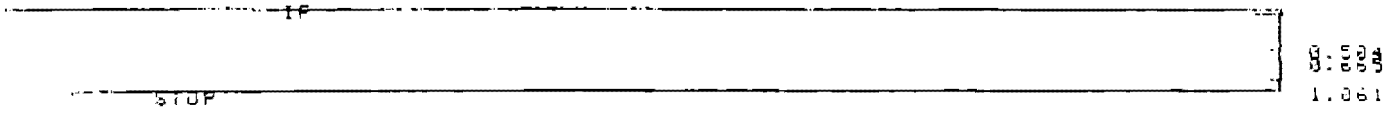
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.496	PB	731	.033	393		.024	
.655	BB	371079	.093	66193	1R	11.457	METHANE

TOTAL AREA= 371960
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2 = -1
CHT SF = 1.0
AR REJ = 0
THRSH = 1
FL WD = 0.08

METHANE
CAL
.998

* RUN # 66 SEP 20, 1991 16:03:10
START



0:50
0:55
1.061

RUN# 66 SEP 20, 1991 16:03:10

ESTD-AREA

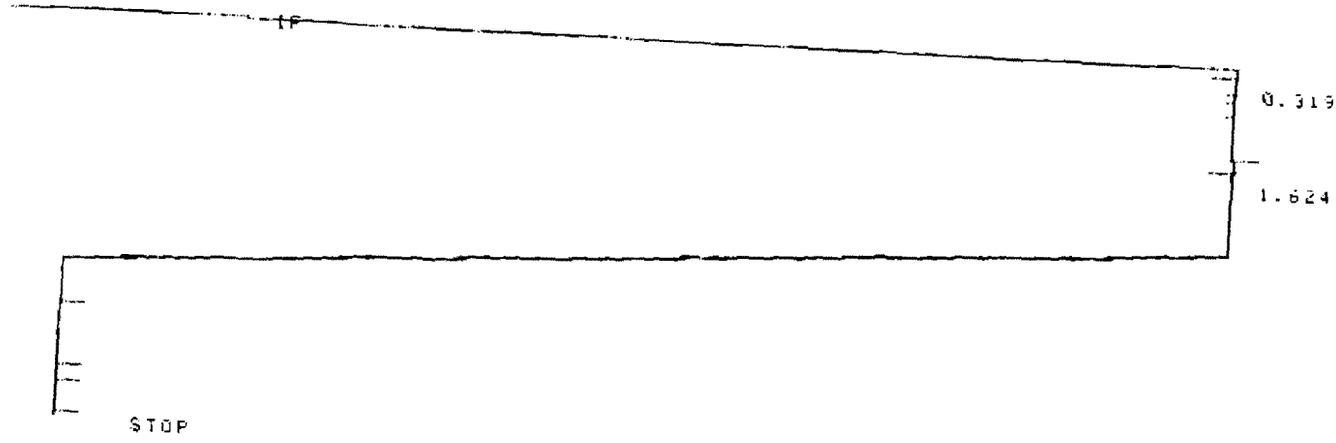
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.665	BB	74219	.126	9801	1R	2.292	METHANE
1.061	I BP	140669	.082	28752	2	1.959	ETHANE

TOTAL AREA= 214888
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2 = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

BLANK

* RUN # 67 SEP 20, 1991 16:05:09
START



RUN# 67 SEP 20, 1991 16:05:09

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.319	BB	649	.155	70	3	.009	ETHANE
1.624	BB	113001	.163	11527	3	1.063	PROPANE

TOTAL AREA= 113650
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
HIT 2^ = -1
CNT SP = 1.0
AR REJ = 0
THRSH = 1
FR WD = 0.08

METHANE CAL .998

* RUN # 68 SEP 20, 1991 16:12:06
START

0:000
1.094

RUN# 68 SEP 20, 1991 16:12:06

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.656	PB	32078	.071	7541	1R	.990	METHANE
.800	BB	862	.065	221	2	.012	ETHANE
1.094	I BP	141873	.140	16947	3	1.334	PROPANE

TOTAL AREA= 174812
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2 = 1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.09

METHANE
998

* RUN # 69 SEP 20, 1991 16:14:47
START

STOP

RUN# 69 SEP 20, 1991 16:14:47

ESTD-AREA

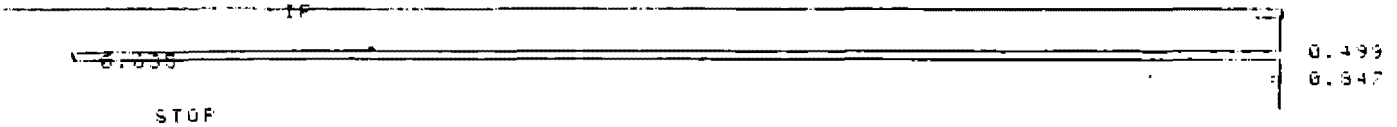
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.667	PB	27250	.066	6885	1R	.841	METHANE
.915	I BH	174602	.228	12771	2	2.432	ETHANE

TOTAL AREA= 201852
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2 = -1
CHT SF = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.08

PLANT (AIR)

RUN # 70 SEP 20, 1991 16:16:33
START



STOP

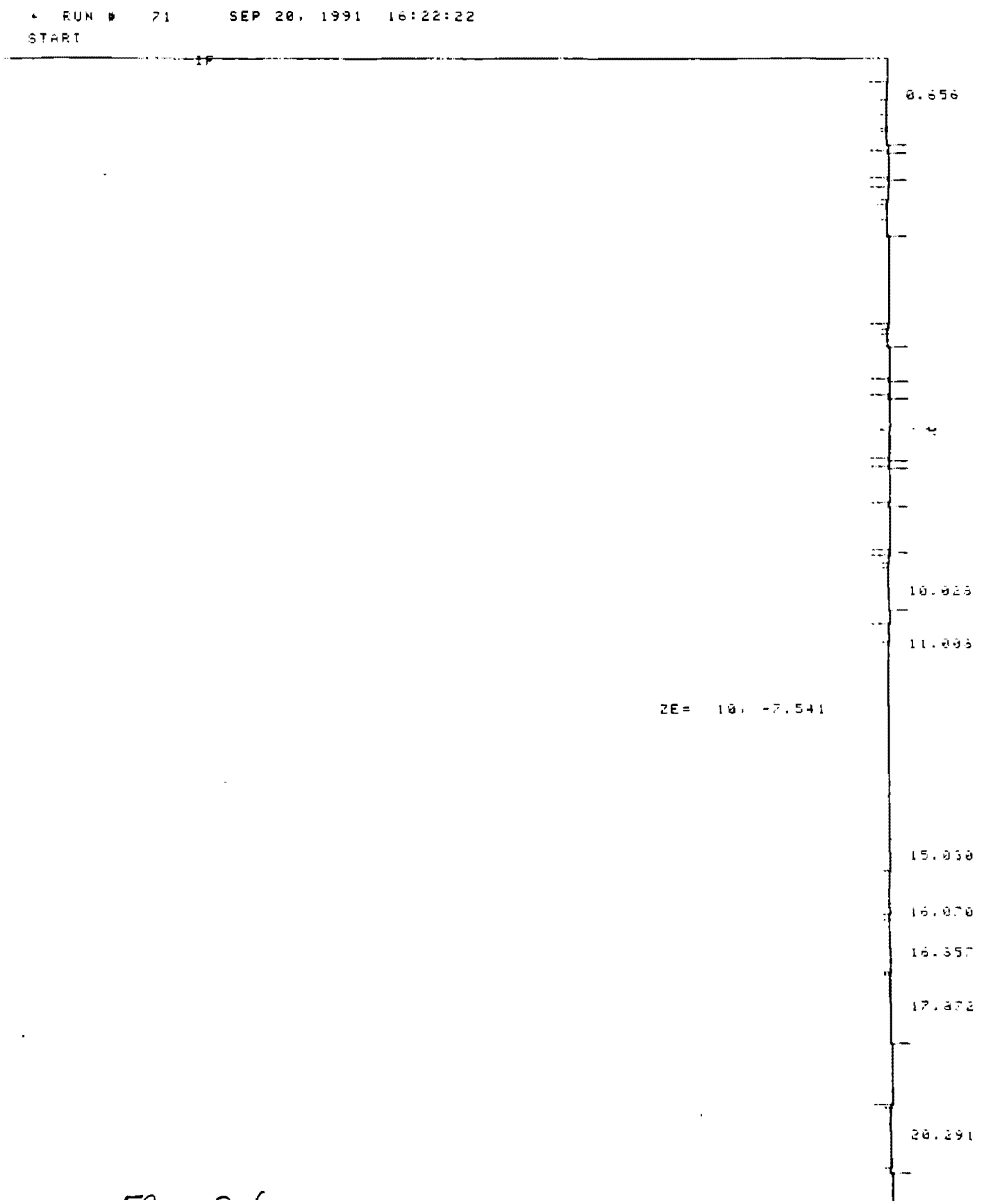
RUN# 70 SEP 20, 1991 16:16:33

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.499	BB	330197	.258	21338		10.193	
.635	BB	31145	.055	9359	1R	.962	METHANE
.647	BB	124252	.647	3201	2	1.731	ETHANE

TOTAL AREA= 485594
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
HTT 3 = -1
CHT SP = 1.0
AP REJ = 0
THRSH = 1
PK WD = 0.08


RUN # 71 SEP 20, 1991 16:22:22
START
0.656
10.026
11.006
ZE= 10, -7.541
15.030
16.070
16.357
17.872
20.291

TIMETABLE STOP

RUN# 71 SEP 20, 1991 16:22:22

ESTD-AREA

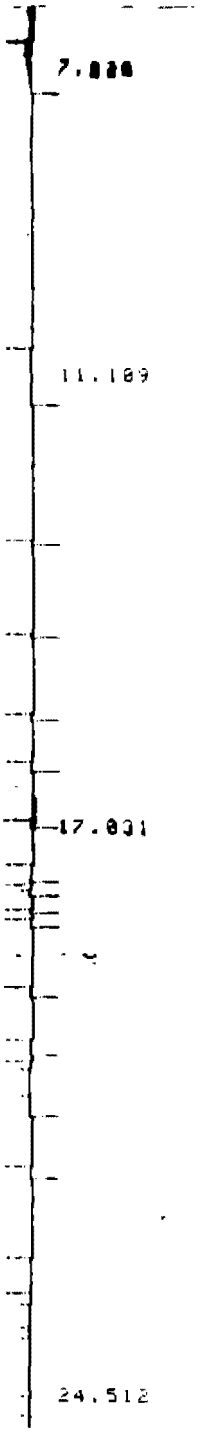
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.656	BB	5497	.098	939	1R	.170	METHANE
10.023	PB	2401066	.216	185496	7	9.448	BENZENE
15.030	BB	76871	.006	227755		2.373	
16.070	BB	30230	.684	737	8	.262	TOLUENE
16.857	PB	49355	.455	1807		1.524	
17.872	VB	5399392	.371	242448		166.679	
20.291	VB	42838	.519	1377	9	.102	ETH-BENZENE
22.155	PB	2977	.382	130		.092	
23.131	BB	5416730	.653	138226	10	12.323	XYLENE

TOTAL AREA=1.3425E+07
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 10
ATT 2^ = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.06

```
* LIST: CALIB   @

ESTD
PEF % RTW:    5.000   NON-PEF % RTW:   -5.000

LEVEL: 1              RECALIBRATIONS: 1

CHL#      RT      LV      AMT       AMT AREA
  1P     0.653     1  1.5000E+01   3.0876E-05
  2      0.856     1  1.4500E+01   1.3925E-05
  3      1.460     1  1.4200E+01   9.4036E-06
  4      3.133     1  1.4600E+01   6.4132E-06
  5      6.375     1  1.4600E+01   5.4045E-06
  6     11.333     1  1.4500E+01   4.6559E-06
  7      7.584     1  1.5000E+01   7.9150E-06
  8     16.546     1  9.6600E+00   3.6520E-06
  9     20.059     1  2.0660E+01   2.1580E-06
 10     33.228     1  2.1060E+01   2.2750E-06

CHL#      NAME
  1  METHANE
  2  ETHANE
  3  PROPANE
  4  BUTANE
  5  PENTANE
  6  HEXANE
  7  BENZENE
  8  TOLUENE
  9  ETHYLBENZENE
 10     XYLENE

CALIBRATION OPTIONS
PEF in uncalibrated areas .... 3.0870E-05
Calibration fit ............. P
Disable Midstream RT update .. NO
SAMPLE AMT ................... 0.0000E+00
ALL PRINT .................... 1.0000E+00

*  RUN #    72      SEP 20, 1991  16:30:46
STOP
```

$C_1 - C_6$ ~14.5



TIMETABLE STOP

RUN# 72 SEP 28, 1991 16:50:46

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.645	PB	452561	.078	96344	1R	13.973	METHANE
.815	BB	1000457	.086	193353	2	13.934	ETHANE
1.365	BB	1458863	.094	258986	3	13.717	PROPANE
3.360	BB	2175754	.166	218532	4	13.953	BUTANE
4.471	BB	56062	1.895	493		1.731	
4.765	BB	186121	1.103	2813		5.746	
7.020	BB	2571776	.201	212937	5	13.904	PENTANE
11.109	BB	2990144	.221	225404	6	13.922	HEXANE
17.031	BB	465	.028	272	8	.004	TOLUENE

TOTAL AREA=1.0093E+07

```
*  LIST: LIST
PEAK CAPACITY:  1221

ZERO    =  10, -10.000
ATT 2^  =  -1
CHT SP  =  1.0
AR REJ  =  0
THRSH   =  1
PK WD   =  0.02


*  LIST: THRSH =  1

*  LIST: CALIB   0

ESTD
REF % RTW:   5.000  NON-REF % RTW:  -5.000

LEVEL: 1              RECALIBRATIONS: 1

CAL#      RT      LV     AMT        AMT/AREA
  1P     0.651     1  1.5000E+01   3.0876E-05
  2      0.947     1  1.4500E+01   1.3928E-05
  3      1.436     1  1.4200E+01   9.4026E-06
  4      3.921     1  1.4600E+01   6.4132E-06
  5      6.536     1  1.4600E+01   5.4065E-06
  6     11.319     1  1.4500E+01   4.6559E-06
  7      8.966     1  1.9900E+01   3.9350E-06
  8     16.667     1  9.6600E+00   8.6020E-06
  9     20.059     1  2.0860E+01   2.3080E-06
 10     23.228     1  2.1000E+01   2.2750E-06

CAL#      NAME
  1   METHANE
  2   ETHANE
  3   PROPANE
  4   BUTANE
  5   PENTANE
  6   HEXANE
  7   BENZENE
  8   TOLUENE
  9   ETH-BENZENE
```

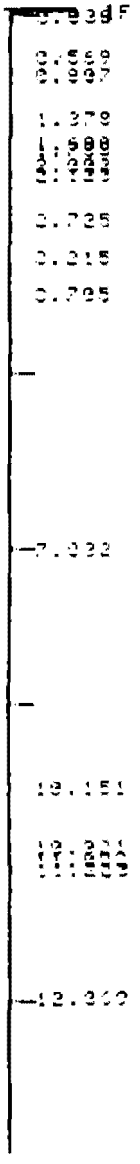
9/20/91

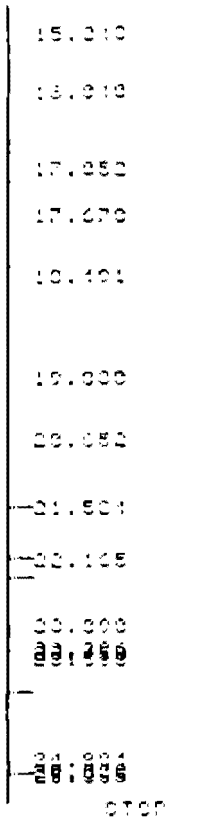
RUN PARAMETERS
ZERO = 10
ATT Z[illegible] = -1
CHT SP = 1.0
AR REJ = 0
THRSH = 1
PK WD = 0.00

SAMPLE #1

* RUN # 12 SEP 19, 1991 09:36:21

START





RUN# 10 SEP 19, 1991 09:36:21

AREA%

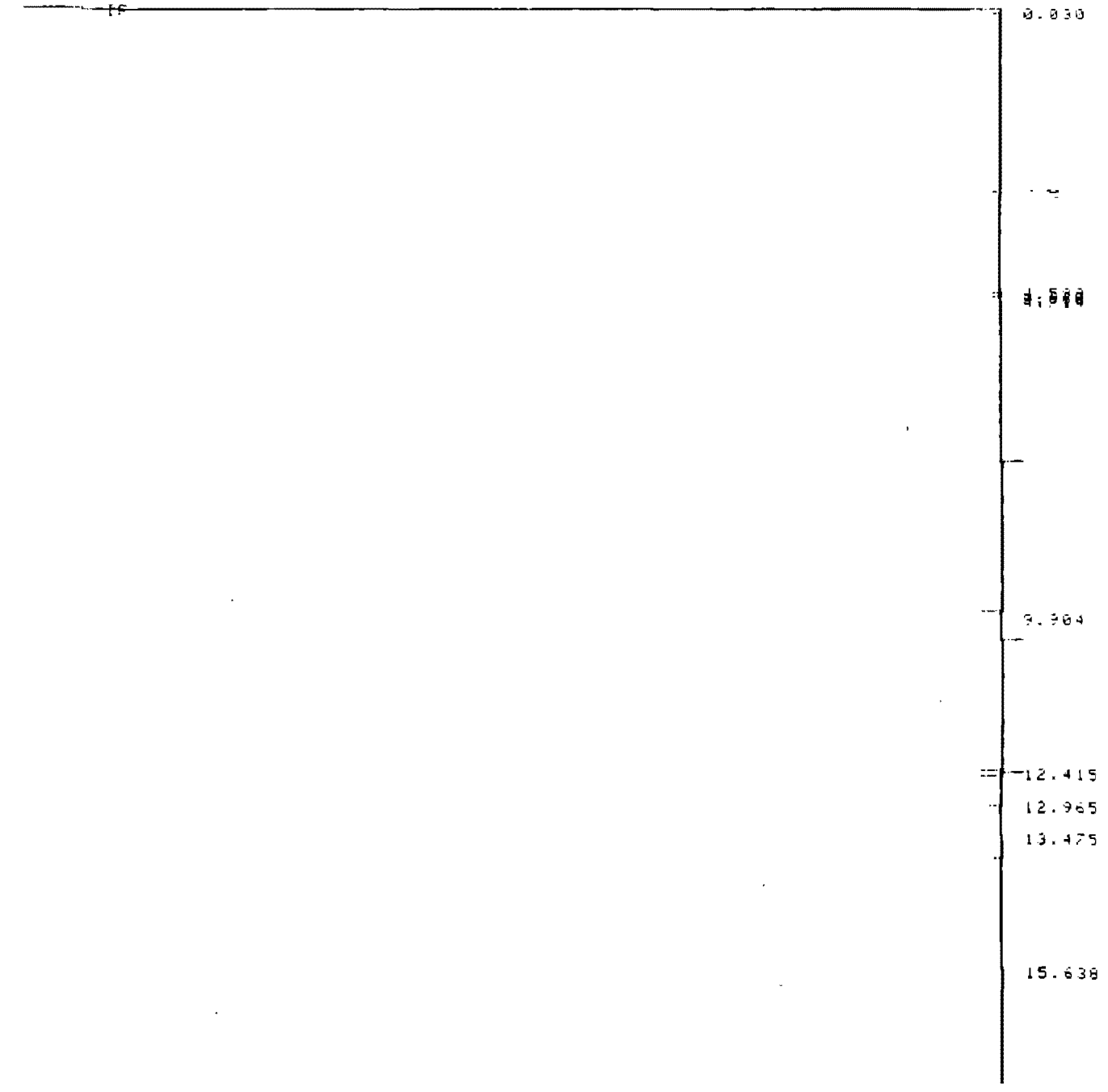
RT	AREA	TYPE	WIDTH	AREA%
.030	3133	BB	.021	.01131
.569	946769	BP	.279	3.05812
.807	1641852	PV	.172	5.92958
1.378	3953992	VV	.526	14.27993
1.690	1415453	VV	.230	5.11194
1.798	660444	VV	.109	2.38521
2.002	1191951	VV	.204	4.30476
2.123	402274	VV	.071	1.45282
2.725	5206099	VV	.930	18.80195
3.215	1522631	VV	.396	5.49901
3.705	2007976	VB	.663	7.25149
7.032	545	BB	.011	.00197
10.151	76506	BV	.312	.27630
10.921	8845	VV	.120	.03194
11.093	6709	VV	.074	.02423
11.223	95043	VP	.769	.34325
12.869	160	PB	.028	.00058
15.248	1486841	PV	.854	5.36976
16.040	2607234	VV	1.425	9.41608
17.052	1198524	VV	.772	4.32850
17.670	93938	VV	.073	.33926
18.491	2168547	VV	1.394	7.83176
19.830	25599	VV	.051	.09245
20.652	810106	VP	.918	2.92572
21.524	249	BB	.014	.00090
22.165	483	PB	.057	.00174
23.090	537	BV	.050	.00194
23.386	10782	VV	.175	.03894
23.450	3843	VV	.058	.01388
23.510	27338	VB	.362	.09870
24.804	931	PV	.109	.00336

TOTAL AREA=2.7599E-07
MUL FACTOR=1.0000E-00

*

BLANK

* RUN # 14 SEP 19. 1991 09:04:11
START



STOP

RUN# 14 SEP 19, 1991 09:04:11

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.020	1617	BP	.019	.00440
4.598	17656432	PV	2.918	48.07229
4.660	375498	VV	.062	1.02235
4.714	8464352	VB	1.429	23.04546
9.904	27601	BB	.306	.07515
12.415	3722	BB	.029	.01013
12.965	35370	PV	.272	.09630
13.475	605525	VV	.462	1.64863
15.638	7606139	VV	3.074	20.70882
18.038	1523402	VV	1.148	4.14791
19.703	42222	VV	.092	.11496
19.911	298225	VV	.641	.81196
20.550	23722	VV	.103	.06459
20.697	27040	VV	.122	.07362
20.940	9788	VV	.051	.02665
20.993	28239	VB	.029	.07688

TOTAL AREA=3.6729E+07
MUL FACTOR=1.0000E+00

BLANK

* RUN # 15 SEP 19, 1991 09:38:35
START

STOP

RUN# 15 SEP 19, 1991 09:38:35

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.030	3767	BB	.029	15.65019
.570	649	BB	.039	2.69630
.660	2165	BP	.050	8.99460
.751	17489	PB	.226	72.65891

TOTAL AREA= 24070
MUL FACTOR=1.0000E+00

BLANK

* RUN # 16 SEP 19, 1991 09:49:42
START
0.036F
0.667
2.039
2.240
2.335

STOP

RUN# 16 SEP 19, 1991 09:49:42

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.036	2149	BP	.018	.06426
.667	206712	PV	.304	6.18103
2.039	1885016	VV	1.247	56.36506
2.240	353036	VV	.253	10.55635
2.335	897386	VB	.678	26.83331

TOTAL AREA=3344299
MUL FACTOR=1.0000E+00

* RUN # 17 SEP 19, 1991 09:59:26
START
0.031F

BLANK

3.385
3.684
3.809

STOP

RUN# 17 SEP 19, 1991 09:59:26

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.031	253	BB	.003	.00168
3.385	6505661	BV	1.798	43.20227
3.684	474868	VV	.133	3.15347
3.809	8077827	VV	2.287	53.64261

TOTAL AREA=1.5059E-07
MUL FACTOR=1.0000E+00
*

$C_1 - C_6$ CAL ~14.5 - 15

* LIST: CALIB @

NO CALIB TBL

* RUN # 19 SEP 19, 1991 10:28:01
START

IF

0.699
0.863
1.440
2.111
2.399
2.896
3.488
3.835
4.286
4.620
4.900

9.120

10.148

11.274

STOP

RUN# 19 SEP 19, 1991 10:28:01

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.689	531434	BV	.098	3.66104
.868	1057679	VB	.098	7.28633
1.440	1532790	BB	.104	10.55937
2.414	1005	BB	.050	.00692
2.565	451	BP	.041	.00311
2.635	51	I PB	.004	.00035
2.896	599	PB	.055	.00413
3.488	2175387	BV	.161	14.98621
3.835	34229	VV	.343	.23580
4.196	7949	VV	.091	.05476
4.292	31104	VV	.308	.21428
4.620	12116	VV	.133	.08347
4.900	54999	VB	.380	.37889
7.175	2702862	BB	.201	18.61999
8.351	14764	VV	.195	.10171
9.120	3083272	VV	.387	21.24062
10.148	68704	VP	.343	.47330
11.274	3206534	PP	.220	22.08976

TOTAL AREA=1.4516E+07
MUL FACTOR=1.0000E+00

* PREP CALIB @

E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E*/I/N]:

REF % RTW [5.000]:
NON-REF % RTW [5.000]:

RF BASED ON AREA OR HEIGHT [A*/H]:

CAL#	RT	AMT	NAME
1	:.689■■■■-.699		
		:15.0	:METHANE
2	:-.868	:14.5	:ETHANE
3	:-1.44	:14.2	:PROPANE
4	:-3.48 NO MATCH		
4	:-3.488	:14.6	:BUTANE
5	:-7.175	:14.6	:PENTANE
6	:11.274■■■■■■■-11.274		
		:14.5	:HE■EXANE
7	:		

GROUP PEAKS [Y/N*]:

CALIBRATION OPTIONS
RF of uncalibrated peaks [0.0000E-00]: 2.73E-05
Replace calibration fit [Y/N=]:
Disable post-run RT update [Y/N=]:
SAMPLE AMT [0.0000E+00]: 15##
MUL FACTOR [1.0000E+00]:

* RUN # 20 SEP 19, 1991 10:59:06
START

APPL. #2

0.025F
0.530
0.766
1.175
1.328
2.932
3.154
3.397
3.620
6.240
7.791
8.057
8.975
9.403
9.828
10.002 ← BENZENE
10.###
11.135
11.967
NO ~~ETHYL BNZ~~ TOLUENE
15.771
16.434
16.850
17.211
17.405
17.823 ← ETHYL-BNZ
18.746
19.637
19.###
20.###
20.###
21.419
23.826 ← M-P-XYLENE

[redacted]

25.031

STOP

RUN# 20 SEP 19, 1991 10:59:06

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
3.620	VB	655984	.549	19098	4R	4.403	BUTANE
11.135	VV	28215	.223	2112	6R	.128	HEXANE

TOTAL AREA=2.6833E+07
MUL FACTOR=1.0000E+00

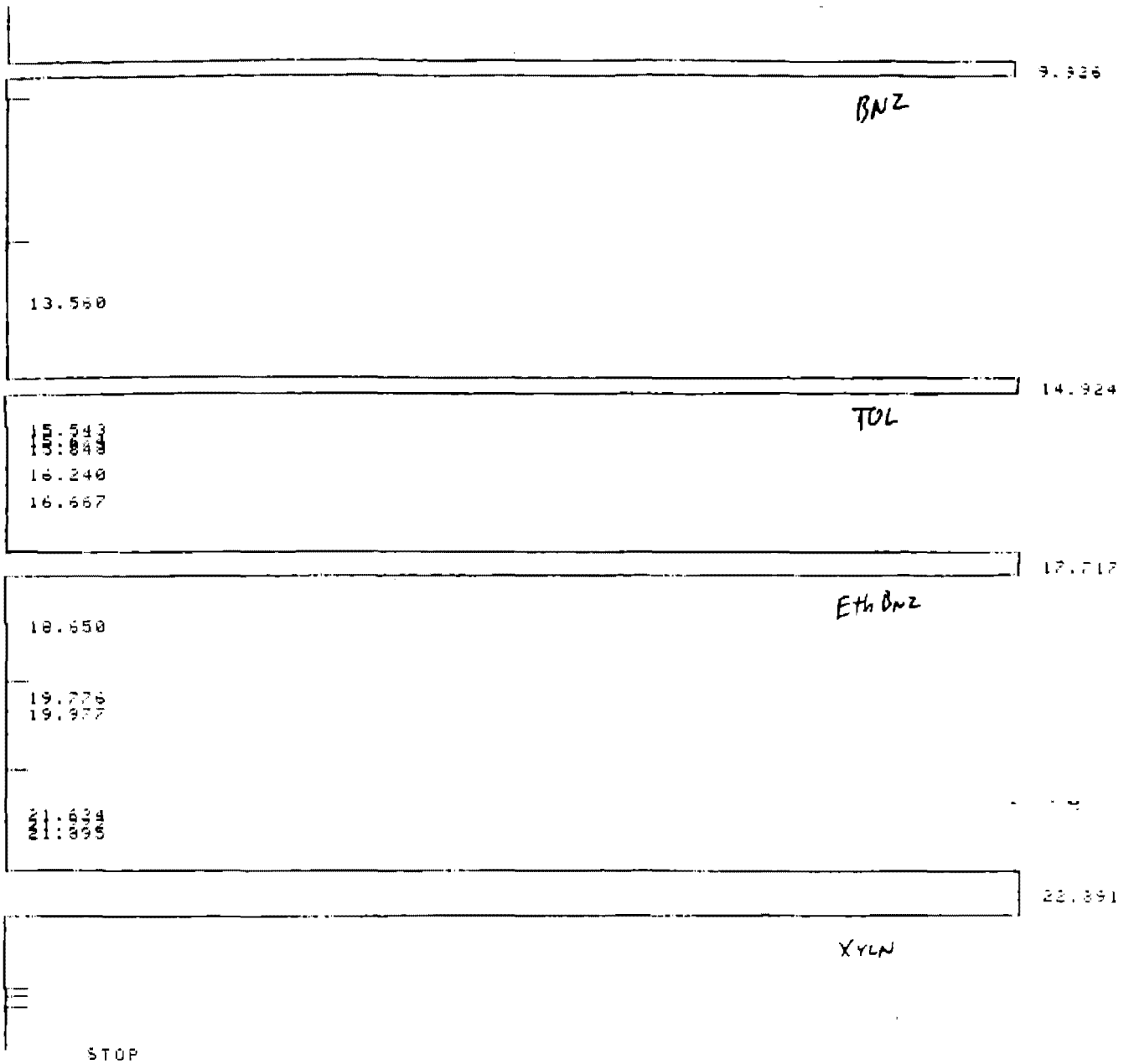
* LIST: TIME @

0.000 INTG # = 8
30.000 STOP

* RUN # 21 SEP 19, 1991 11:28:10
START

0.033F
0.597
0.896

4.470




```
      STOP

RUN#     21          SEP 19, 1991  11:28:16

ESTD-AREA
      RT TYPE       AREA  WIDTH     HEIGHT CAL# CONC.        NAME
    .996   VV     1884989   .224     139983   2R     25.842  ETHANE


TOTAL AREA=1.2496E+08
MUL FACTOR=1.0000E+00

*
```

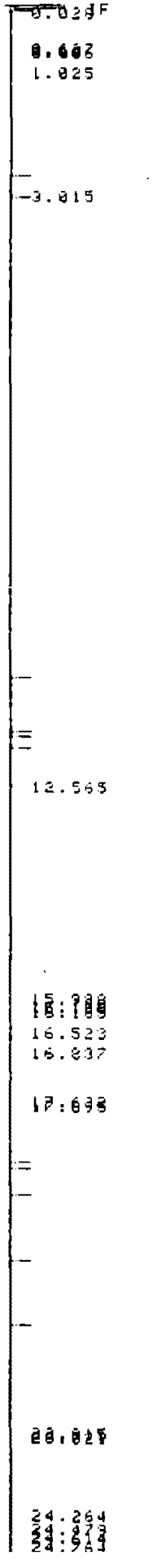
```
1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 3

CAL#:  7
RT:  9.926
AMT:  19.9
AMT/AREA:

SECTION TO BE EDITED: 1


E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION

CALIB PROCEDURE [E*/I/N]:


SECTION TO BE EDITED: 2

REF % RTW [   5.000]:
NON-REF % RTW [   5.000]:

SECTION TO BE EDITED: 5

CALIBRATION OPTIONS
RF of uncalibrated peaks [2.7300E-05 ]:
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:

SECTION TO BE EDITED: 3

CAL#:  7
RT:  -9.926
AMT:  19.9
AMT/AREA:  3.86E-06
NAME:  BENENE■■■ZENE

CAL#:  8
RT:  -14.924
AMT:  20.8
AMT/AREA:  2.85E-06
NAME:  TOLUENE

CAL#:  9
RT:  -17.717
AMT:  20.9
AMT/AREA:  2.09E-06
NAME:  ETH-BENZENE

CAL#:  10
RT:  22■■-22.898■1
AMT:  21.1
AMT/AREA:  1.18E-06
NAME:  XYLENE
```

SECTION TO BE EDITED:

* RUN # 22 SEP 19, 1991 12:09:53

SAMPLE #3

START



STOP

RUN# 22 SEP 19, 1991 12:09:53

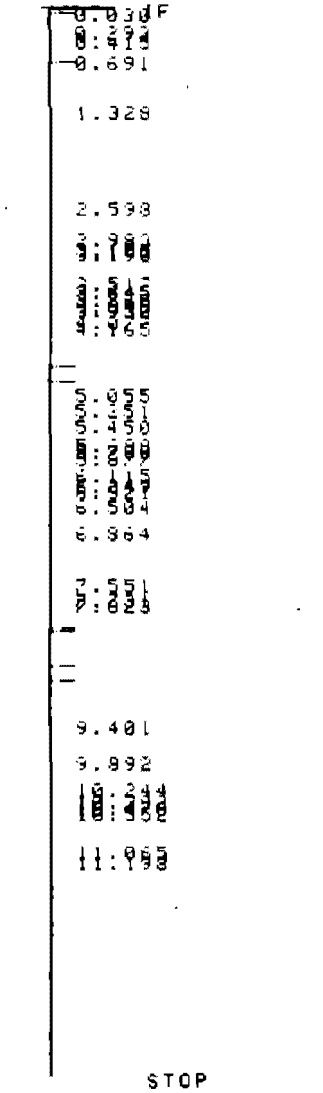
ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.686	VV	313606	.059	88110	1R	8.852	METHANE
16.837	VV	963752	.827	19417	9R	2.014	ETH-BENZENE
23.021	VP	30419	.231	2191	10R	.036	XYLENE

TOTAL AREA=1.1515E+07
MUL FACTOR=1.0000E+00

BLANK

* RUN # 23 SEP 19, 1991 13:00:24
START



STOP

RUN# 23 SEP 19, 1991 13:00:24

ESTD AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.691	PB	27046	.053	8435	1R	.753	METHANE
3.517	PV	475	.072	110	4R	.003	BUTANE
6.864	VV	4482	.070	1060	5R	.024	PENTANE
9.892	BV	23834	.252	1576	7R	.092	BENZENE
11.065	VV	49778	.364	2280	8R	.225	HEXANE

TOTAL AREA= 595702
MUL FACTOR=1.0000E+00

* LIST: CALIB @

ESTD
REF % RTW: 5.000 NON-REF % RTW: 5.000

LEVEL: 1 RECALIBRATIONS: 1

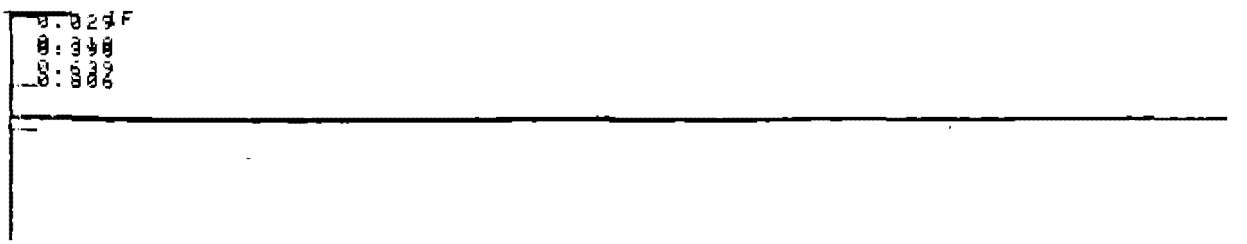
CAL#	RT	LV	AMT	AMT/AREA
1R	0.699	1	1.5000E-01	2.8236E-05
2R	0.868	1	1.4500E+01	1.3709E-05
3R	1.440	1	1.4200E+01	9.3642E-06
4R	3.488	1	1.4600E+01	6.7114E-06
5R	7.175	1	1.4600E+01	5.4017E-06
6R	11.274	1	1.4500E+01	4.5220E-06
7R	9.926	1	1.9900E+01	3.8600E-06
8R	14.924	1	2.0800E+01	2.8500E-06
9R	17.717	1	2.0900E+01	2.0900E-06
10R	22.391	1	2.1100E+01	1.1900E-06

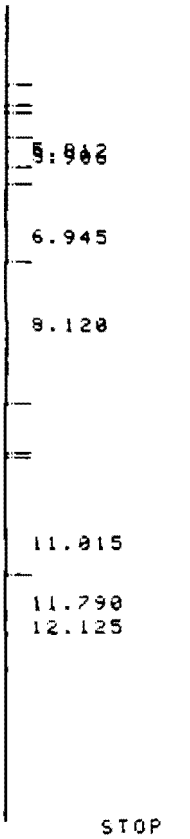
CAL#	NAME
1	METHANE
2	ETHANE
3	PROPANE
4	BUTANE
5	PENTANE
6	HEXANE
7	BENZENE
8	TOLUENE
9	ETH-BENZENE
10	XYLENE

CALIBRATION OPTIONS
RF of uncalibrated peaks 2.7300E-05
Calibration fit ..... P
Disable post-run RT update .. NO
SAMPLE AMT ..... 0.0000E+00
MUL FACTOR ..... 1.0000E+00

E-66

* RUN # 24 SEP 19, 1991 13:22:25
START





STOP

RUN# 24 SEP 19, 1991 13:22:25

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
6.945	BB	2596552	.201	215249	5R	14.026	PENTANE
11.015	VB	3073397	.223	229777	6R	13.898	HEXANE

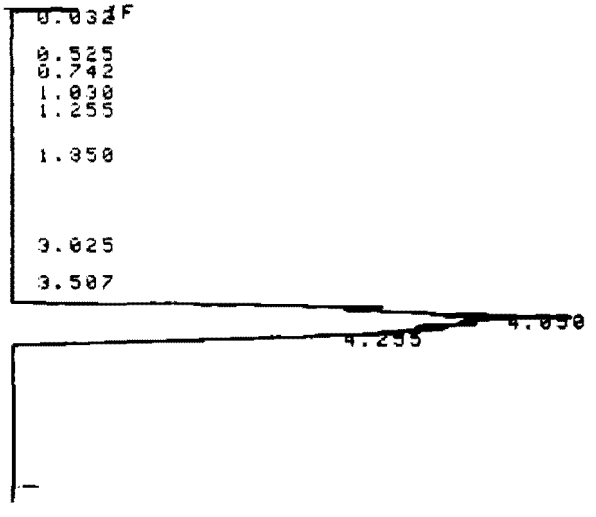
TOTAL AREA=1.0815E+07
MUL FACTOR=1.0000E+00

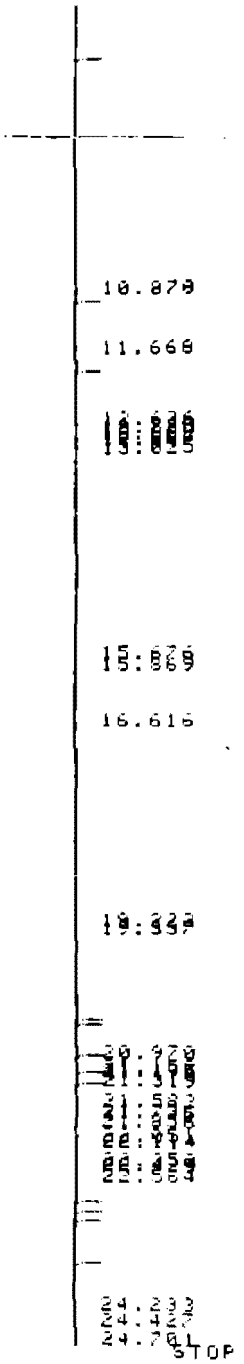
SAMPLE #5

* CALIB BREAK

* RUN # 25 SEP 19, 1991 13:41:06

START





RUN# 25 SEP 19, 1991 13:41:06

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
3.507	VV	3369430	.453	123380	4R	32.614	BUTANE
10.878	BB	13904	.290	800	6R	.063	HEXANE
23.564	VB	23267	.261	1496	10R	.027	XYLENE

TOTAL AREA=4.1939E+07
MUL FACTOR=1.0000E+00

* EDIT CALIB @

3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED: 5

CALIBRATION OPTIONS
RF of uncalibrated peaks [2.7300E-05]:
Replace calibration fit [Y/N*]:
Disable post-run RT update [Y/N*]:
SAMPLE AMT [0.0000E+00]:
MUL FACTOR [1.0000E+00]:

SECTION TO BE EDITED: 1

E = EXTERNAL STANDARD
I = INTERNAL STANDARD
N = NORMALIZATION
CALIB PROCEDURE [E*/I/N]: N

SECTION TO BE EDITED:

* RUN # 26 SEP 19, 1991 14:11:56
START
IF
0.267
0.497

1.093
STOP

RUN# 26 SEP 19, 1991 14:11:56

NO CALIB PEAKS FOUND
AREA%

RT	AREA	TYPE	WIDTH	AREA%
.267	422922	BV	.164	31.22186
.497	435796	VV	.161	32.17227
1.093	495852	VV	.294	36.60587

TOTAL AREA=1354570
MUL FACTOR=1.0000E+00

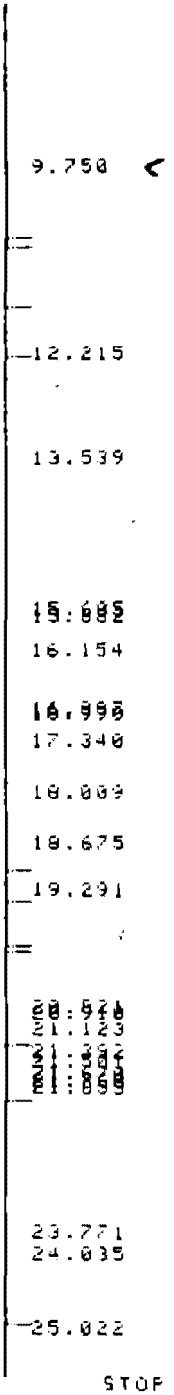
SAMPLE #6

* RUN # 27 SEP 19, 1991 14:25:40
START
0.034F

0.510
0.723
1.010
1.232

1.919

2.799
2.975
3.222
3.444



RUN# 27 SEP 19, 1991 14:35:40

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.723	PV	2204909	.158	232710	1R	70.706	METHANE
3.444	VB	2759470	.672	68444	4R	21.041	BUTANE
9.750	BB	26508	.105	4206	7R	.116	BENZENE
15.685	VV	2024582	1.048	32198	8R	6.555	TOLUENE
17.340	VV	553965	.511	18056	9R	1.315	ETH-BENZENE
24.035	VP	198128	.835	3957	10R	.266	XYLENE

TOTAL AREA=2.7723E+07
MUL FACTOR=1.0000E+00

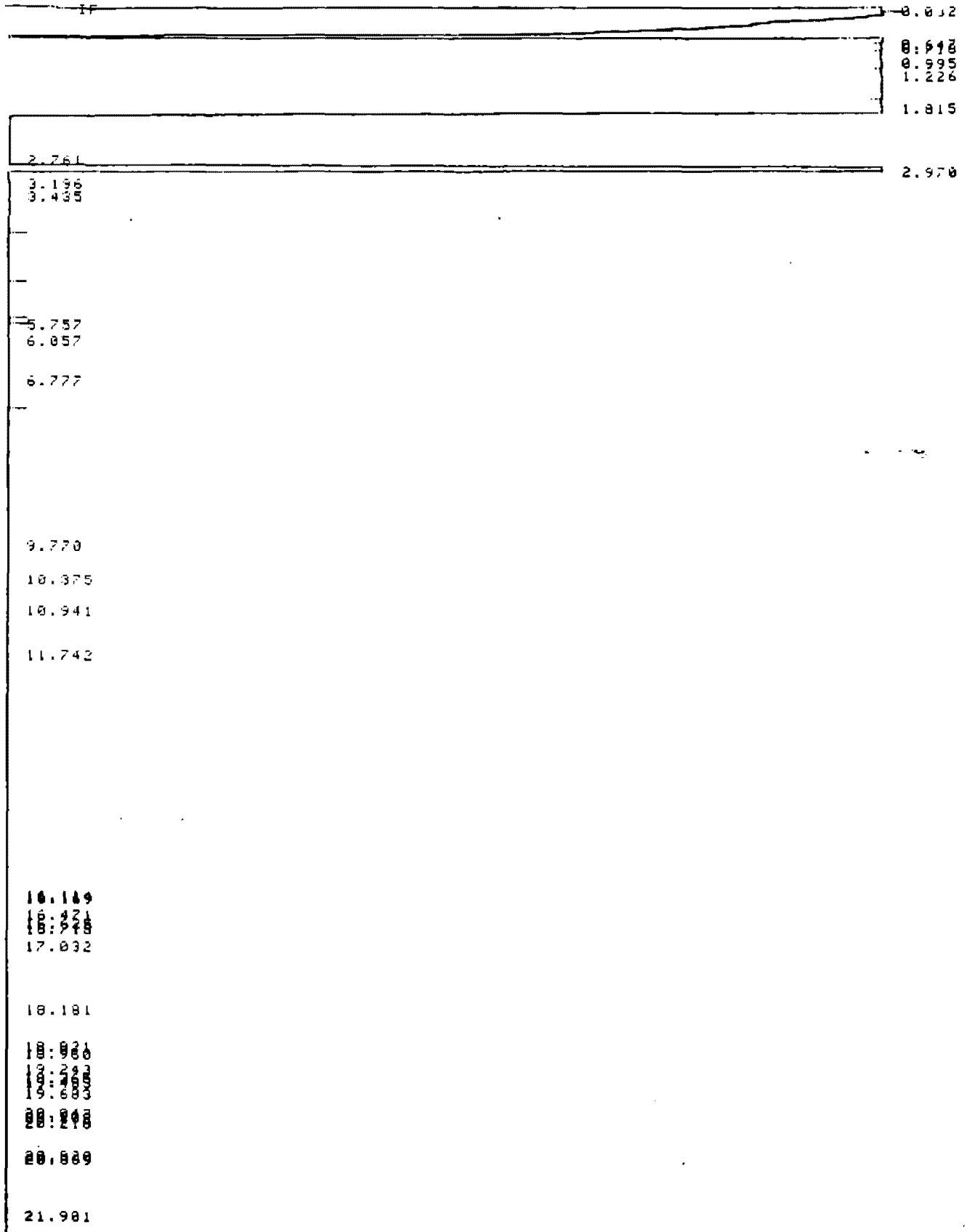
STOP

• PLOT

STOP

• RUN # 20 SEP 19, 1991 15:05:22
START

SAMPLE #7



24.310

STOP

RUN# 28 SEP 19, 1991 15:05:22

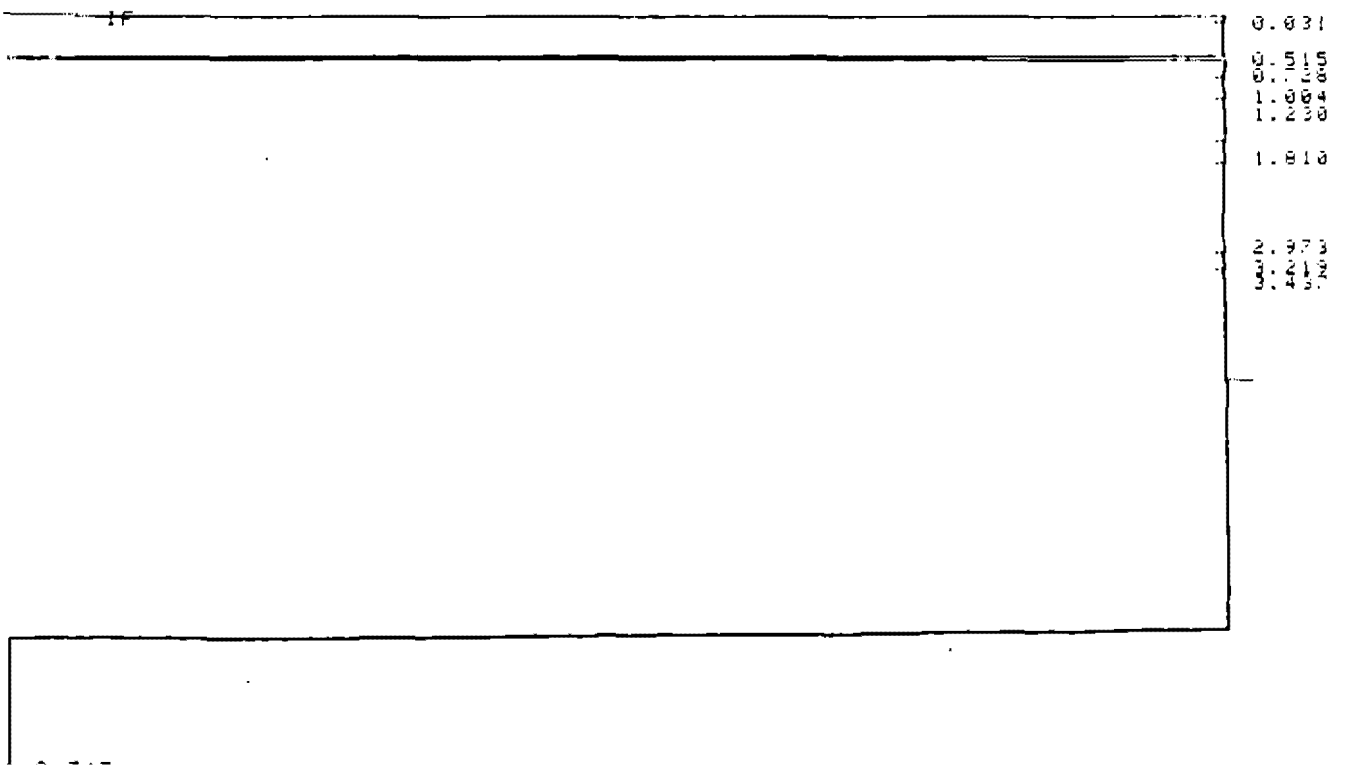
NORM-AREA

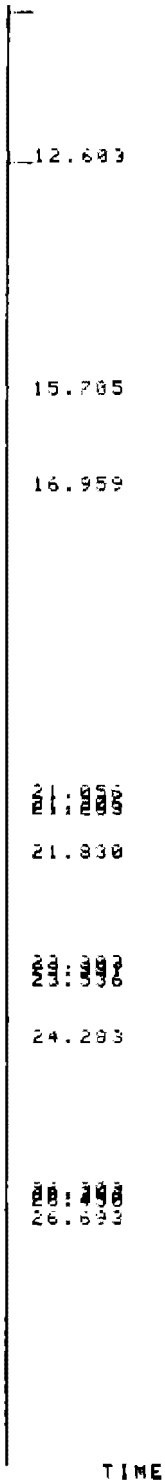
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
.718	VV	1412130	.100	235121	1R	52.537	METHANE
3.435	VB	528495	.408	21585	4R	4.675	BUTANE
9.770	BP	99501	.287	5782	7R	.506	BENZENE
11.742	VV	228851	.491	7769	8R	1.364	HEXANE
17.032	VV	6156963	1.048	97939	9R	16.961	ETH-BENZENE
21.901	VV	15401976	2.130	120530	10R	23.956	XYLENE

TOTAL AREA=9.1966E+07
MUL FACTOR=1.0000E+00

Sample #8

* RUN # 29 SEP 19, 1991 15:36:01
START





TIMETABLE STOP

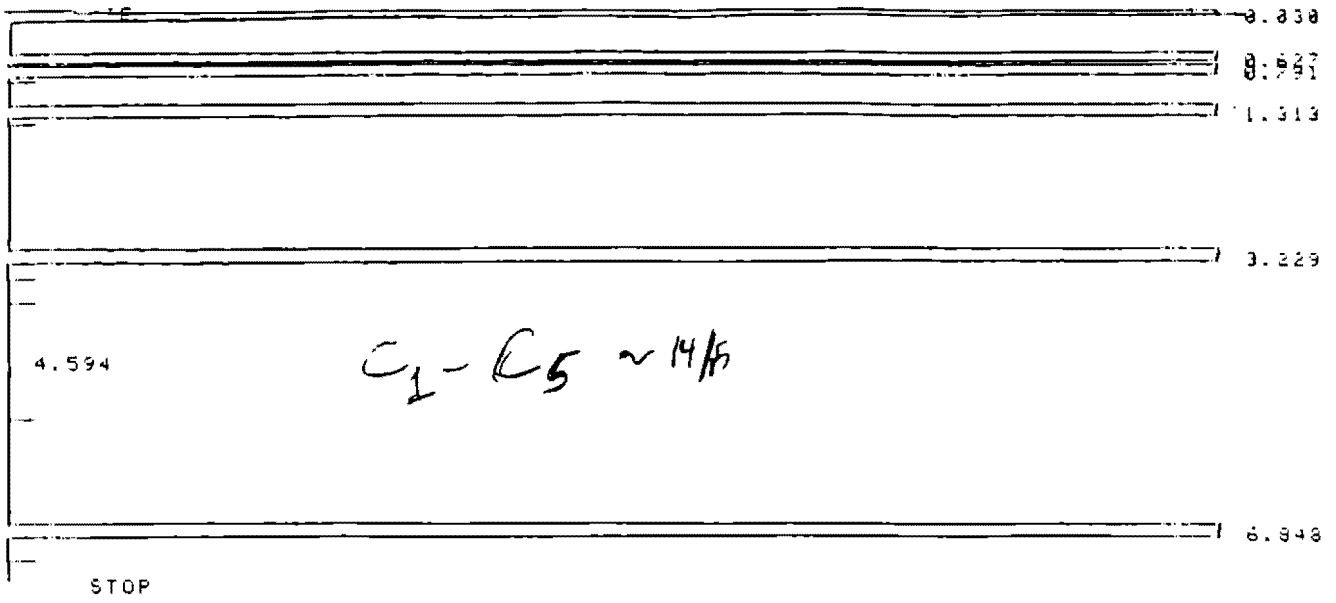
RUN# 29 SEP 19, 1991 15:36:01

NORM-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	CONC.	NAME
3.437	VB	3966397	.808	81781	4R	69.545	BUTANE
9.747	BB	6182	.024	4381	7R	.062	BENZENE
16.959	VV	5118682	2.529	33732	9R	27.949	ETH-BENZENE
21.830	VV	792772	1.301	10159	10R	2.444	XYLENE

TOTAL AREA=3.6686E+07
MUL FACTOR=1.0000E+00

START



$C_1 - C_5 \sim 14/15$

RUN# 31 SEP 19, 1991 16:37:37

AREA%

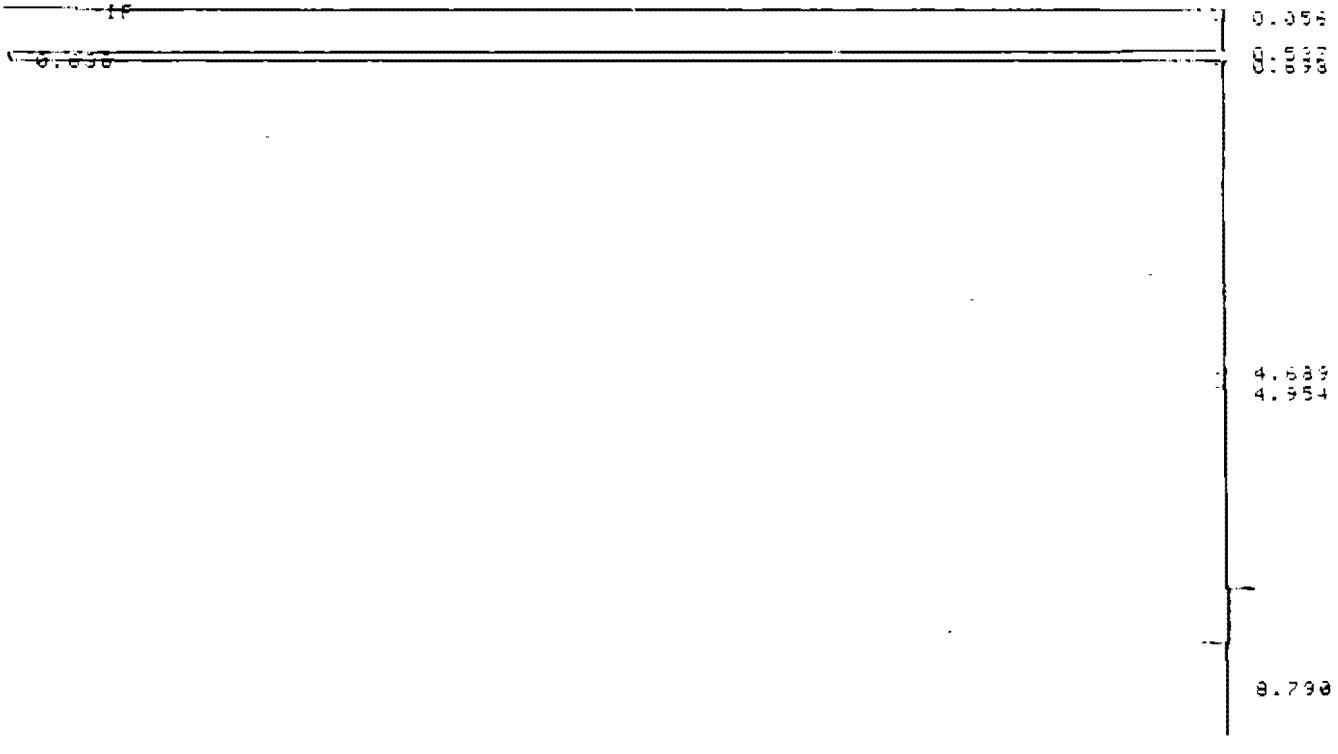
RT	AREA	TYPE	WIDTH	AREA%
.030	7668	BB	.022	.09535
.627	537969	BV	.084	6.68975
.791	1069719	VB	.084	13.30210
1.313	1556456	BB	.092	19.35485
3.329	2152123	BB	.156	26.76210
4.594	21486	BB	.357	.26718
6.848	2696262	BB	.202	33.52858

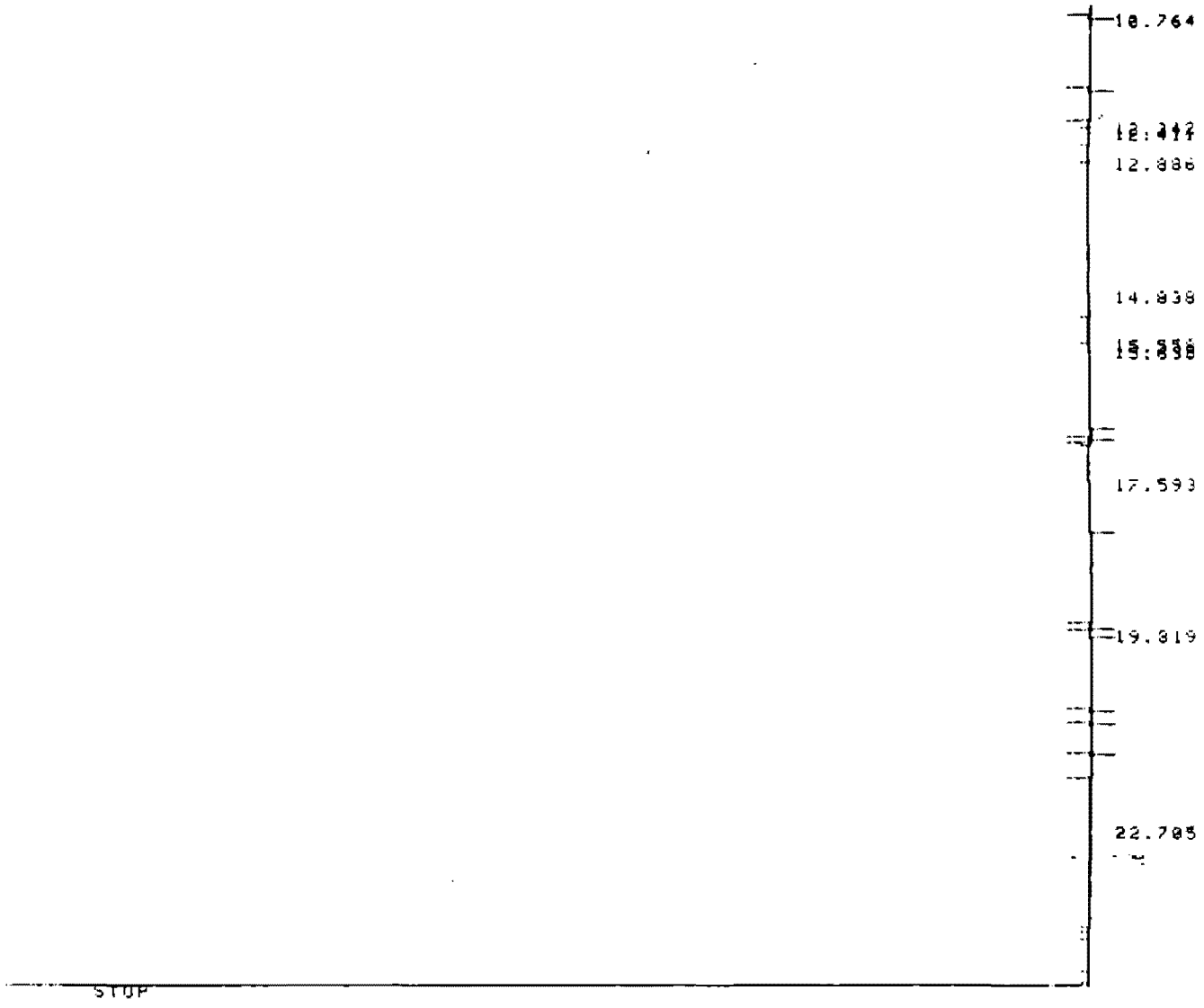
TOTAL AREA=8041683
MUL FACTOR=1.0000E+00

RUN # 32 SEP 19, 1991 16:47:14

BTX ~ 2°

START





RUN# 32 SEP 19, 1991 16:47:14

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.056	23746	BV	.072	.03939
.537	676261	VP	.253	1.12186
.638	68092	PV	.048	.11296
.698	154703	VV	.052	.25664
4.689	14751848	VV	3.446	24.47210
4.954	5892682	VB	1.465	9.77548
8.790	3101685	BP	.364	5.14544
9.833	4508643	PB	.215	7.47947
10.764	1441	BB	.012	.00239
12.342	670	BV	.061	.00111
12.411	3161	VP	.154	.00524
14.838	5726813	PV	.246	9.50031
15.558	435849	VV	.411	.72304
15.638	696543	VB	.683	1.15551
17.593	7363475	VB	.359	12.21540
19.819	794	BB	.101	.00132
22.705	16873872	BV	.694	27.99235

TOTAL AREA=6.0288E+07
MUL FACTOR=1.0000E+00

APPENDIX F

ANALYTICAL DATA

F.1 PM/Metals
F.2 PM_{10}/CPM
F.3 Aldehydes
F.4 PAH
F.5 Sample Log

APPENDIX F.1

PM/METALS

Run # M6

Client _____ Date 10/7/91

Plant MATHY Technician TWOOD

Sample type Acetone MET PR Sheet 1 of 1

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
MET 1 PR	M6 26 Beaker LL17	150mL		104.8933	104.8987		Run 1
				104.8935	104.8990		
						0.0054	
				104.8934	104.8988	~~0.0054~~	
MET 2 PR	M6 43 Beaker RX88	175mL		113.5755	113.5777		Run 2
				113.5756	113.5778		
				113.5756	113.5778	0.0022	
MET #3 PR	M6 62 Beaker M10	280mL		113.6989	113.7030		Run 3
				113.6989	113.7031		
				113.6989	113.7030	0.0041	
MET FB PR	M6 3 Beaker LL20	225mL		103.1333	103.1342		FB
				103.1333	103.1340		
				103.1333	103.1341	0.0008	
Met FB IC	M6 1 Beaker M6 4			-		503	Met FB IC
				-		227	Met FB APR
Met Run 1	M6 24 Beaker M6 27			-		1161.1	Run 1 IC
				-		203.1	Run 1 APR
Met Run 2	M6 42 Beaker M6 44			-		966.5	Run 2 IC
				-		143.0	Run 2 APR
Met Run 3	M6 59 Beaker M6 61			-		1019.1	Run 3 IC
				-		312.1	Run 3 APR

1181672R

Method 5 Analysis Data Sheet

Run # M6

Client EPA Date 9/30/91

Plant MATHY Technician T WOOD

Sample type PM10 filters, metal filters Sheet 1 of 1

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Samples Weight (g)	Comments
PM10 FB	M6 PM10 16 Filter # 03			0.2118	0.2120		
				0.2116	0.2122		
				0.2117	0.2121	0.0004	
PM10 01	M6 PM10 30 Filter # 10			0.2137	0.2163		
				0.2138	0.2160		
				0.2138	0.2162	0.0024	
PM10 02	PM10 M6 58 07			0.2130	0.2160		
				0.2129	0.2158		
				0.2130	0.2159	0.0029	
PM10 03	M6 PM10 68 09			0.2143	0.2163		
				0.2143	0.2163		
				0.2143	0.2163	0.0020	
MET FB	M6 MET M6 # 2 Filter # TM008			0.6405	0.6410		FB met
				0.6407	0.6412		
				0.6406	0.6411	0.0005	
MET 01	M6 MET 25 Filter # TM018			0.6410	0.6547		Run 1
				0.6410	0.6585		
				0.6410	0.6546	0.0136	
MET 02	M6 MET 45 TM005			0.6466	0.6535		Run 2
				0.6461	0.6575		
				0.6460	0.6535	0.0115	
MET 03	MET M6 60 TM001			0.6450	0.6570		Run 3
				0.6450	0.6571		
				0.6450	0.6571	0.0121	

1010726

Method 5 Analysis Data Sheet

ble 2.
(TNT - Asphalt: Back 1/2
sults in total ug.

SAR Number	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF	Final Volume	Initial Volume	Digested Volume
1-10-010-16a	M6 - 1	* 0.700	1.01	< 0.111	< 0.222	* 0.811	< 0.444	* 1.25	* 0.955	* 77.5	* 0.373	< 1.67	< 1.78	< 11.1	* 2.24	< 0.444	0.100	0.5030	0.4530
1-10-010-17a	M6 - 24	< 0.627	0.815	< 0.104	* 0.282	* 1.87	6.23	2.81	2.17	* 59.0	* 0.886	< 1.57	< 1.67	10.4	16.1	< 0.418	0.100	1.1615	1.1115
1-10-010-18a	M6 - 42	< 0.633	10.5	< 0.105	10.1	11.0	198	40.5	30.6	191	29.5	< 1.58	< 1.69	< 10.5	245	* 1.10	0.100	0.9605	0.9105
1-10-010-19a	M6 - 59	< 0.631	1.42	< 0.105	* 0.700	1.28	5.92	3.34	1.72	* 73.3	1.66	< 1.58	< 1.68	< 10.5	13.4	< 0.421	0.100	1.0191	0.9691
1-10-010-12a	M26 - 14	< 0.671	< 0.112	< 0.112	< 0.224	< 0.671	< 0.447	* 0.447	* 0.369	* 50.2	< 0.335	< 1.68	< 1.79	< 11.2	< 1.68	< 0.447	0.100	0.4755	0.4255
1-10-010-13a	M26 - 30	< 0.634	1.32	< 0.106	* 0.254	* 1.26	* 1.07	1.84	2.59	* 64.9	* 0.355	< 1.59	< 1.69	< 10.6	14.4	< 0.423	0.100	0.9246	0.8746
1-10-010-14a	M26 - 37	< 0.631	1.66	< 0.105	* 0.716	* 3.07	2.89	24.8	4.69	* 73.1	3.50	< 1.58	< 1.68	< 10.5	24.3	< 0.421	0.100	1.0069	0.9569
1-10-010-15a	M26 - 66	< 0.631	1.33	< 0.105	< 0.210	* 1.30	< 0.421	2.01	2.04	* 67.5	* 0.969	< 1.58	< 1.68	< 10.5	* 5.84	< 0.421	0.100	1.0163	0.9663
1-10-010-20a-M	Method Spike	41.0%	88.2%	83.4%	98.5%	97.0%	90.1%	94.0%	88.1%	78.2%	88.6%	86.8%	85.3%	94.7%	90.7%	86.0%	0.100	0.100	0.100
1-10-010-21a-M	Method Spike-dup	30.4%	78.4%	93.4%	88.5%	86.5%	79.3%	84.9%	98.5%	86.5%	98.8%	96.8%	95.8%	97.5%	101%	97.8%	0.100	0.100	0.100
1-10-010-22a-B	Method Blank	< 0.636	* 0.106	< 0.106	< 0.212	< 0.636	< 0.424	2.37	* 0.572	< 31.8	* 0.744	< 1.59	< 1.70	< 10.6	< 1.59	< 0.424	0.100	0.8834	0.8334
1-10-010-	LCS	107%	91.6%	97.4%	104%	100%	96.4%	97.1%	102%	99.0%	96.0%	109%	106%	98.5%	105%	107%			

less than 5 times the detection limit.

LCULATIONS:

Sample Result (ug/L) X Final Volume (L) X Initial Volume / Digested Volume = Total ug

e 2.
T - Asphalt: Front 1/2
lts in total ug.

SAN umber	Sample Description	Ag ICAP	Ba ICAP	Be ICAP	Cd ICAP	Cr ICAP	Cu ICAP	Mn ICAP	Ni ICAP	P ICAP	Pb AAS GF	Sb ICAP	Se ICAP	Tl ICAP	Zn ICAP	As AAS GF	Final Volume
10-010-01a	M26 - 15,16,17	< 1.50	45.0	= 0.400	= 1.18	= 24.5	= 2.60	55.3	15.3	= 112	8.13	= 9.38	< 4.00	< 25.0	69.5	16.1	0.250
10-010-02a	M26 - 31,32,33	= 5.18	64.3	= 0.550	= 2.25	= 101	23.0	82.0	138	= 315	31.0	= 15.1	< 4.00	< 25.0	249	23.5	0.250
10-010-03a	M26 - 34,35,36	= 7.45	60.0	= 0.625	3.10	105	19.4	94.0	99.8	= 306	28.3	= 11.8	< 4.00	< 25.0	268	20.2	0.250
10-010-04a	M26 - 67,68,69	= 5.05	79.8	= 0.275	4.70	27.8	37.0	105	13.3	= 360	36.8	= 9.58	< 4.00	< 25.0	316	27.0	0.250
10-010-05a	M6 - 2,3,4	= 4.05	4.80	< 0.250	= 1.58	= 3.78	< 1.00	4.93	= 3.30	< 75.0	= 1.74	= 13.6	< 4.00	< 25.0	21.6	< 1.00	0.250
10-010-06a	M6 - 25,26,27	= 7.00	9.15	< 0.250	2.93	= 7.75	< 1.00	58.0	45.0	< 75.0	= 1.92	= 14.1	< 4.00	< 25.0	23.8	< 1.00	0.250
10-010-07a	M6 - 43,44,45	= 5.35	7.48	< 0.250	= 0.83	= 5.20	< 1.00	29.0	= 2.43	< 75.0	< 0.750	= 12.1	< 4.00	< 25.0	= 15.4	< 1.00	0.250
10-010-08a	M6 - 60,61,62	< 1.50	9.50	< 0.250	= 1.43	= 3.38	< 1.00	61.5	= 3.50	< 75.0	6.35	= 12.1	< 4.00	< 25.0	35.0	< 1.00	0.250
10-010-09a-M	Method Spike	76.5%	91.4%	95.0%	100%	99.6%	93.5%	96.1%	99.9%	89.2%	97.8%	100%	99.0%	103%	103%	87.4%	0.100
10-010-10a-M	Method Spike-dup	26.7%	91.0%	95.2%	99.9%	99.4%	93.9%	96.2%	100%	92.1%	102%	100%	99.6%	100%	104%	95.4%	0.100
10-010-11a-B	Method Blank	< 0.600	= 0.100	< 0.100	< 0.200	< 0.600	< 0.400	= 0.330	< 0.300	< 30.0	< 0.300	< 1.50	< 1.60	< 10.0	= 2.41	< 0.40	0.100
10-003-	LCS	107%	91.6%	97.4%	104%	100%	96.4%	97.1%	102%	99.0%	101%	109%	106%	98.5%	105%	91.4%	54.0

as than 5 times the detection limit.

ULATIONS:
Sample Result (ug/L) X Final Volume (L) = Total ug

APPENDIX F.2

PM_{10}/CPM

Run # m6

Client _____ Date 10/10

Plant Mathy Technician JWOD

Sample type $MECl_2$ + H_2O pmD Sheet 1 of 1

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
FB	m6 pm10 MEC 14 Beaker #	280mL		101.7803	101.7807		
				101.7800	101.7806		FB
$MECl_2$	LL13			101.7800	101.7806	0.0006	MECL
FB	m6 pm10 IC 15 Beaker #	680mL		168.3412	168.3406		
				168.3412	168.3404		H_2O FB
H_2O	LL08			168.3412	168.3405	0.0007	
ME	m6 pm10 ME 33 Beaker #	430mL		104.0258	104.0264		
1				104.0259	104.0266		
$MECl_2$	LL19			104.0258	104.0265	0.0007	
IC	m6 pm10 IC 34 Beaker #	710mL		168.9670	168.9781		
				168.9673	168.9779		
1							
H_2O	KZ100			168.9672	168.9780	0.0108	
ME	m6 pm10 ME 57 Beaker #	440mL		102.6840	102.6867		
2				102.6842	102.6864		
$MECl_2$	LL21			102.6841	102.6865	0.0024	
IC	m6 pm10 IC 56 Beaker #	820mL		169.2318	169.2325		
2				169.2319	169.2324		
H_2O	LL05			169.2318	169.2324	0.0046	
ME	m6 pm10 ME 67 Beaker #	390mL		108.8665	108.8688		
3				108.8660	108.8683		
$MECl_2$	M7			108.8662	108.8686	0.0024	
IC	m6 pm10 IC ~~AA~~ 66 Beaker #	820mL		175.8725	175.8775		
				175.8730	175.8775		
3							
H_2O	LL11			175.8728	175.8775	0.0047	

11816728

Method 5 Analysis Data Sheet

Client EPA

Plant Mathy

Sample type PM10 filters, metal filters

Run # M6

Date 9/30/91

Technician T WOOD

Sheet 1 of 1

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
Pm10 FB	M6 Pm10 16 Filter # 03			0.2118 0.2116 0.2117	0.2120 0.2122 0.2121	 0.0004	FB
Pm10 01	M6 Pm10 30 Filter # 10			0.2127 0.2128 0.2128	0.2163 0.2160 0.2162	 0.0034	
Pm10 02	Pm10 M6 58 07			0.2130 0.2129 0.2130	0.2160 0.2158 0.2159	 0.0029	
Pm10 03	M6 Pm10 68 08			0.2143 0.2143 0.2143	0.2163 0.2163 0.2163	 0.0020	
MET FB	M6 MET 16 Filter # TM008			0.6405 0.6407 0.6406	0.6410 0.6412 0.6411	 0.0005	
MET 01	M6 MET 25 Filter # TM018			0.6410 0.6410 0.6410	0.6547 0.6595 0.6596	 0.0186	
MET 02	M6 MET 45 TM005			0.6460 0.6461 0.6460	0.6575 0.6575 0.6575	 0.0115	
MET 03	MET M6 60 TM001			0.6450 0.6450 0.6450	0.6570 0.6572 0.6571	 0.0121	

1181672R

Method 5 Analysis Data Sheet

Client _____ Run # M6

Plant Mathy Date 10/7/91

Sample type Acetone PM10 CYC Technician T WOOD

Sheet 1 of 1

Run #	Sample ID#	Sample Vol. (ml)	Blank Corr. (ml)	Tare Weight (g)	Final Weight (g)	Sample Weight (g)	Comments
FB PM10 CYC 1	M6 PM10 31 CYC Beaker LL13	125mL		104.6220	104.6229		
				104.6220	104.6230		
				104.6220	104.6230	0.0010	
1 PM10 CYC	M6 PM10 32 CYC Beaker RX81	100mL		112.2134	112.2245		
				112.2132	112.2247		
				112.2133	112.2246	0.0113	
PM10 2 CYC	M6 55 Beaker LL14	125mL		102.2612	102.2718		
				102.2614	102.2716		
				102.2613	102.2717	0.0104	
PM10 3 CYC	M6 45 Beaker RX96	75mL		100.8620	100.8695		
				100.8622	100.8698		
				100.8621	100.8696	0.0075	
	Beaker						

1181872R

Method 5 Analysis Data Sheet

APPENDIX F.3

ALDEHYDES

Radian Work Order P1-09-010

Analytical Report
12/16/91

EMB

EMB
Radian Corporation
RTP, NC

Larry Romesburg

Customer Work Identification Asphalt Test Site 6
Purchase Order Number 275-026-48-27

Contents:

1 Analytical Data Summary
2 Sample History
3 Comments Summary
4 Notes and Definitions

Radian Analytical Services
900 Perimeter Park
Morrisville, NC 27560

919-481-0212

Client Services Coordinator: LROMESBURG

Certified by: _____

EMB
Radian Work Order: P1-09-010

Method:Aldehydes, Mod TO11, HPLC (1)
List:Compendium Method TO-11

Sample ID:	M6-0918-ALD-F-B (12, 13)	M6-0919-ALD-1 (28, 29)	M6-0920-ALD-2 (46, 47)	M6-0920-ALD-3 (63, 64)
Factor:	5.4	10	10	10
Results in:	Total ug	Total ug	Total ug	Total ug
	09A	10A	11A	12A
Matrix:	Stack	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acetaldehyde	ND	5.8	822	120	1040	120	1170	120
Acetone	6640 Y	7.3	23700 Y	150	11200	150	2570	150
Acetophenone/o-Tolualdehyde	ND	12	ND	23	ND	23	ND	23
Acrolein	ND	7.1	ND	13	ND	13	26.7 *	13
Benzaldehyde	ND	11	199	21	198	21	211	21
Butyraldehyde/Isobutyraldehyde	ND	9.4	47.0 *	18	ND	18	75.4 *	18
Crotonaldehyde	ND	9.4	37.1 *	18	29.5 *	18	65.0 *	18
2,5-Dimethylbenzaldehyde	ND	13	ND	24	ND	24	ND	24
Formaldehyde	10.7 *	4.8	2950	96	4470	96	2770	96
Hexanal	ND	11	40.7 *	20	62.5 *	20	ND	20
Isophorone	ND	9.4	ND	18	ND	18	ND	18
Isovaleraldehyde	ND	9.8	ND	18	ND	18	ND	18
MIBK/p-Tolualdehyde	ND	12	ND	23	ND	23	ND	23
Methyl Ethyl Ketone	ND	9.4	ND	18	ND	18	93.5	18
Propionaldehyde	ND	7.3	ND	14	ND	14	94.6	14
Quinone	ND	9.4	48.8 *	18	27.0 *	18	1040	18
m-Tolualdehyde	ND	12	ND	23	ND	23	ND	23
Valeraldehyde	ND	9.8	ND	18	ND	18	56.9 *	18

ND Not detected at specified detection limit

* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-09-010

Method:Aldehydes, Mod TO11, HPLC (1)
List:Compendium Method TO-11

Sample ID:	METHOD BLANK	METHOD SPIKE	Calibration Check QC
Factor:	.54	1	1
Results in:	Total ug	%	%
	17A	18A	19A
Matrix:	DNPH	DNPH	ACN

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acetaldehyde	0.66 *	0.58	90		116 Q			
Acetone	1.74 *	0.73	83		NS			
Acetophenone/o-Tolualdehyde	ND	1.2	70 Q		NS			
Acrolein	ND	0.71	23 Q		NS			
Benzaldehyde	ND	1.1	119		NS			
Butyraldehyde/Isobutyraldehyde	ND	0.94	NA		104			
Crotonaldehyde	ND	0.94	25 Q		NS			
2,5-Dimethylbenzaldehyde	ND	1.3	85		NS			
Formaldehyde	1.06 *	0.48	87		112			
Hexanal	ND	1.1	117		NS			
Isophorone	ND	0.94	91		NS			
Isovaleraldehyde	3.68 *	0.98	NS		NS			
MIBK/p-Tolualdehyde	ND	1.2	106		NS			
Methyl Ethyl Ketone	ND	0.94	NA		NS			
Propionaldehyde	ND	0.73	107		165 Q			
Quinone	ND	0.94	64 Q		NS			
m-Tolualdehyde	ND	1.2	93		NS			
Valeraldehyde	ND	0.98	82		NS			

* Est. result less than 5 times detection limit
Q Outside control limits
NS Not spiked
ND Not detected at specified detection limit
NA Not analyzed

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-09-010

Sample Identifications and Dates

Sample ID	M6-0920-ALD-2 (46, 47)	M6-0920-ALD-3 (63, 64)	METHOD BLANK	METHOD SPIKE	METHOD BLANK	METHOD SPIKE
Date Sampled	09/20/91	09/20/91				
Date Received	09/23/91	09/23/91	09/23/91	09/23/91	09/23/91	09/23/91
Matrix	Stack	Stack	XAD-2/FI	XAD-2/FI	DNPH	DNPH
	11	12	15	16	17	18
SW8270-Semi-Volatiles						
Prepared			09/30/91	10/11/91		
Analyzed			11/04/91	11/05/91		
Analyst			RK	RK		
File ID			4586532.TI	4586534.TI		
Blank ID			4586532.TI	4586533.TI		
Instrument			GC/MS B	GC/MS B		
Report as			received	received		
Aldehydes, Mod TO11, HPLC						
Prepared	09/30/91	09/30/91			09/30/91	09/30/91
Analyzed	10/18/91	10/18/91			10/04/91	10/04/91
Analyst	LKK	LKK			LKK	LKK
File ID	OWEN127/174	OWEN128/173			LLWA75	LLWA76
Blank ID	LLWA75	LLWA75			LLWA75	LLWA75
Instrument	V5000	V5000			V5000	V5000
Report as	received	received			received	received

EMB
Radian Work Order: P1-09-010

Sample Identifications and Dates

Sample ID	Calibration Check QC
Date Sampled	
Date Received	09/23/91
Matrix	ACN
	19

Aldehydes, Mod TO11, HPLC						
Prepared						
Analyzed	10/18/91					
Analyst	LKK					
File ID	OWEN124					
Blank ID						
Instrument	V5000					
Report as	received					

Appendix A

Comments, Notes and Definitions

EMB
Radian Work Order: P1-09-010

General Comments

Aldehyde Data: Y - Outside of calibration curve.

Q - Spike recovery QC limits range from 80 to 120 percent.
Calibration check QC limits range from 85 to 115 percent.

NA - The butyraldehydes and MEK coeluted.

EMB
Radian Work Order: P1-09-010

B This flag indicates that the analyte was detected in the reagent blank but the sample results are not corrected for the amount in the blank.

J Indicates an estimated value for GC/MS data. This flag is used either when estimating a concentration for tentatively identified compounds where a response factor of 1 is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit.

NA This analyte was not analyzed.

ND This flag (or <) is used to denote analytes which are not detected at or above the specified detection limit. The value to the right of the < symbol is the method specified detection limit for the sample.

NS This analyte or surrogate was not added (spiked) to the sample for this analysis.

Q This quality control standard is outside method or laboratory specified control limits. This flag is applied to matrix spike, analytical QC spike, and surrogate recoveries; and to RPD(relative percent difference) values for duplicate analyses and matrix spike/matrix spike duplicate result.

* The asterisk(*) is used to flag results which are less than five times the method specified detection limit. Studies have shown that the uncertainty of the analysis will increase exponentially as the method detection limit is approached. These results should be considered approximate.

EMB
Radian Work Order: P1-09-010

TERMS USED IN THIS REPORT:
Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/ digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units -	ug/L	micrograms per liter (parts per billion);liquids/water
	ug/kg	micrograms per kilogram (parts per billion); soils/solids
	ug/M3	micrograms per cubic meter; air samples
	mg/L	milligrams per liter (parts per million);liquids/water
	mg/kg	milligrams per kilogram (parts per million);soils/solids
	%	percent; usually used for percent recovery of QC standards
	uS/cm	conductance unit; microSiemens/centimeter
	mL/hr	milliliters per hour; rate of settlement of matter in water
	NTU	turbidity unit; nephelometric turbidity unit
	CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX F.4

PAH

Radian Work Order P1-09-010

Analytical Report
12/16/91

EMB

EMB
Radian Corporation
RTP, NC

Larry Romesburg

Customer Work Identification Asphalt Test Site 6
Purchase Order Number 275-026-48-27

Contents:

1 Analytical Data Summary
2 Sample History
3 Comments Summary
4 Notes and Definitions

Radian Analytical Services
900 Perimeter Park
Morrisville, NC 27560

919-481-0212

Client Services Coordinator: LROMESBURG

Certified by: _____

EMB
Radian Work Order: P1-09-010

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	M6-0918-PAH-F-B (6-11)	M6-0919-PAH-1 17,18,20-23	M6-0919-PAH-2 (35, 37-41)	M6-0920-PAH-3 (48, 50-54)
Factor:	5	5	5	5
Results in:	Total ug 02A	Total ug 04A	Total ug 06A	Total ug 08A
Matrix:	XAD-2/FI	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Acenaphthene	ND	50	14.9 J	50	ND	50	ND	50
Acenaphthylene	ND	50	41.8 J	50	37.7 J	50	48.1 J	50
Anthracene	ND	50	10.2 J	50	8.99 J	50	9.62 J	50
Benzo(a)anthracene	ND	50	ND	50	ND	50	ND	50
Benzo(a)pyrene	ND	50	ND	50	ND	50	ND	50
Benzo(b)fluoranthene	ND	50	ND	50	ND	50	ND	50
Benzo(g,h,i)perylene	ND	50	ND	50	ND	50	ND	50
Benzo(k)fluoranthene	ND	50	ND	50	ND	50	ND	50
Chrysene	ND	50	ND	50	ND	50	ND	50
Dibenz(a,h)anthracene	ND	50	ND	50	ND	50	ND	50
Dibenzofuran	ND	50	ND	50	ND	50	ND	50
7,12-Dimethylbenz(a)anthracene	ND	100	ND	100	ND	100	ND	100
Fluoranthene	ND	50	ND	50	ND	50	ND	50
Fluorene	ND	50	ND	50	ND	50	ND	50
Indeno(1,2,3-cd)pyrene	ND	50	ND	50	ND	50	ND	50
2-Methylnaphthalene	2.36 J	50	408	50	348	50	417	50
Naphthalene	751 B	50	416 B	50	776 B	50	1470 B	50
Phenanthrene	0.275 J*	50	9.04 J	50	7.94 J	50	8.49 J	50
Pyrene	ND	50	1.26 J	50	0.625 JB*	50	0.805 J*	50

(See next page for tentatively identified compounds.)

ND Not detected at specified detection limit
B Detected in blank,result not corrected
J Detected at less than detection limit
* Est. result less than 5 times detection limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-09-010

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	M6-0918-PAH-F-B (6-11)	M6-0919-PAH-1 17,18,20-23	M6-0919-PAH-2 (35, 37-41)	M6-0920-PAH-3 (48, 50-54)
Factor:	5	5	5	5
Results in:	Total ug	Total ug	Total ug	Total ug
	02A	04A	06A	08A
Matrix:	XAD-2/Fi	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit
Surrogate Recovery(%)								
2-Fluorobiphenyl	141 Q		146 Q		124 Q		153 Q	
Control Limits: 30 to 115								
Nitrobenzene-d5	96.4		113		93.9		113	
Control Limits: 23 to 120								
Terphenyl-d14	112		111		112		121	
Control Limits: 18 to 137								

(See next page for tentatively identified compounds.)

Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-09-010

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	METHOD BLANK	METHOD SPIKE
Factor:	5	5
Results in:	Total ug	%
	15A	16A
Matrix:	XAD-2/FI	XAD-2/FI

	Result	Det. Limit	Result	Det. Limit
Acenaphthene	ND	50	109	
Acenaphthylene	ND	50	NS	
Anthracene	ND	50	NS	
Benzo(a)anthracene	ND	50	NS	
Benzo(a)pyrene	ND	50	NS	
Benzo(b)fluoranthene	ND	50	NS	
Benzo(g,h,i)perylene	ND	50	NS	
Benzo(k)fluoranthene	ND	50	NS	
Chrysene	ND	50	NS	
Dibenz(a,h)anthracene	ND	50	NS	
Dibenzofuran	ND	50	NS	
7,12-Dimethylbenz(a)anthracene	ND	100	NS	
Fluoranthene	ND	50	NS	
Fluorene	0.375 J*	50	NS	
Indeno(1,2,3-cd)pyrene	ND	50	NS	
2-Methylnaphthalene	0.68 J*	50	NS	
Naphthalene	260	50	NS	
Phenanthrene	ND	50	NS	
Pyrene	0.805 J*	50	135	

ND Not detected at specified detection limit
J Detected at less than detection limit
* Est. result less than 5 times detection limit
NS Not spiked

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-09-010

Method:SW8270-Semi-Volatiles (1)
List:PAHs by SW846 8270

Sample ID:	METHOD BLANK	METHOD SPIKE
Factor:	5	5
Results in:	Total ug	%
	15A	16A
Matrix:	XAD-2/FI	XAD-2/FI

	Result	Det. Limit	Result	Det. Limit		
Surrogate Recovery(%)						
2-Fluorobiphenyl	130 Q		143 Q			
Control Limits: 30 to 115						
Nitrobenzene-d5	87.4		105			
Control Limits: 23 to 120						
Terphenyl-d14	111		127			
Control Limits: 18 to 137						

Q Outside control limits

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.
(2) 4-Methylphenol co-elutes with 3-methylphenol. The value reported is the combined total of the 2 compounds.

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
M6-0918-PAH-F8 (6-11)				
	1-Methylnaphthalene	1.94 B*J	Total ug	
	Diethylphthalate	7.27 B*J	Total ug	
	~~N-nitrosodiphenylamine~~	~~0.72 *J~~	~~Total ug~~	
	Di-n-butylphthalate	9.44 B*J	Total ug	
	Diphenylamine	0.72 *J	Total ug	
	Bis-(2-ethylhexyl)-phthalate	11.4 B*J	Total ug	
M6-0919-PAH-1 17,18,20-23				
	Phenol	34.6 *J	Total ug	
	Acetophenone	93.5	Total ug	
	3-Methylphenol	5.70 *J	Total ug	
	Indene	421	Total ug	
	1-Methylnaphthalene	249 B	Total ug	
	Butylbenzylphthalate	0.67 *J	Total ug	
	Bis-(2-ethylhexyl)-phthalate	47.4 B*J	Total ug	
	Di-n-octylphthalate	0.66 *J	Total ug	
	2,6-Dimethylheptadecane	NQ	Total ug	
	2,6-Dimethylnonane	NQ	Total ug	
	Tetramethylbenzene	NQ	Total ug	
	1-Ethyl-2,3-dimethylbenzene	NQ	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	5,6-Dimethylundecane	NQ	Total ug	
	2,3-Dihydro-4-methyl-1H-Indene	NQ	Total ug	
	(1,1-Dimethylpropyl)-benzene	NQ	Total ug	
	6-Methyldodecane	NQ	Total ug	
	2,4-Dimethyl-1-	NQ	Total ug	
	(1-methylpropyl)-benzene		Total ug	
	7-Methyltridecane	NQ	Total ug	
	4,7-Dimethylundecane	NQ	Total ug	
	3-Methyltridecane	NQ	Total ug	
	1,1'-Biphenyl	NQ	Total ug	
	2-Methylundecane	NQ	Total ug	
	2-Ethylnaphthalene	NQ	Total ug	
	Dimethylnaphthalenes	NQ	Total ug	
M6-0919-PAH-2 (35, 37-41)				
	Acetophenone	83.4	Total ug	
	3-Methylphenol	3.46 *J	Total ug	
	Indene	387	Total ug	
	Diethylphthalate	12.2 B*J	Total ug	
	Bis-(2-ethylhexyl)-phthalate	390 B	Total ug	
	1-Methylnaphthalene	213 B	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	Decane	NQ	Total ug	
	1,3,5-Trimethylbenzene	NQ	Total ug	
	1-Heptyl-2-methylcyclopropane	NQ	Total ug	
	Ethylmethylbenzenes	NQ	Total ug	
	Uknown oxygenated hydrocarbons	NQ	Total ug	
	Unkown branch hydrocarbons	NQ	Total ug	
	2,3-Dimethyloctane	NQ	Total ug	
	(1-Methylethyl)-benzene	NQ	Total ug	
	Dimethylbenzenes	NQ	Total ug	
	1-Heptyl-2-methylcyclopropane	NQ	Total ug	
	2,3,4-Trimethylhexane	NQ	Total ug	
	Unknown alcoxy alcohol	NQ	Total ug	
	1-Octene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	Octane	NQ	Total ug	
	2-Propenylcyclohexane	NQ	Total ug	
	(1-Methylpropyl)-cyclooctane	NQ	Total ug	
	Toluene	NQ	Total ug	
M6-0920-PAH-3 (48, 50-54)				
	Phenol	24.0 *J	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	Acetophenone	114	Total ug	
	3-Methylphenol	2.74 *J	Total ug	
	Indene	426	Total ug	
	Cyclohexane	996	Total ug	
	1-Methylnaphthalene	262	Total ug	
	Diethylphthalate	17.9 *J	Total ug	
	Bis-(2-ethylhexyl)-phthalate	36.9 *J	Total ug	
	Di-n-octylphthalate	1.17 *J	Total ug	
	Cyclohexene	NQ	Total ug	
	Unknown branched hydrocarbons	NQ	Total ug	
	Toluene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	Xylene isomers	NQ	Total ug	
	1-Ethyl-2-methylbenzene	NQ	Total ug	
	Decane	NQ	Total ug	
	1,3-Diethylbenzene	NQ	Total ug	
	1,4-Diethylbenzene	NQ	Total ug	
	1-Methyl-2-(2-propenyl)-benzen	NQ	Total ug	
	Undecane	NQ	Total ug	
	(E)-(1-Methyl-1-propenyl)-benz	NQ	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	2,3-Dihydro-4-methyl-1H-indene	NQ	Total ug	
	4,7-Dimethylundecane	NQ	Total ug	
	2-Fluoro-1,1'-biphenyl	NQ	Total ug	
METHOD BLANK				
	1-Methylnaphthalene	0.365 *J	Total ug	
	Diethylphthalate	22.3 *J	Total ug	
	Di-n-butylphthalate	11.7 *J	Total ug	
	Bis-(2-ethylhexyl)-phthalate	19.5 *J	Total ug	
	Cyclohexene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	2,2'-Oxybis-ethanol diacetate	NQ	Total ug	
	Diethylbenzene isomers	NQ	Total ug	
	Benzoic acid, methyl ester	NQ	Total ug	
	Unsaturated branched benzyl compound	NQ	Total ug	
	Oxygenated hydrocarbons	NQ	Total ug	
METHOD SPIKE				
	Phenol	84.8	%	
	2-Chlorophenol	94.2	%	
	1,4-Dichlorobenzene	95.6	%	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: SW8270-Semi-Volatiles (1)
List: PAHs by SW846 8270

Sample ID	Analyte	Result	Units	Scan
	N-Nitroso-di-n-propylamine	104	%	
	1,2,4-Trichlorobenzene	109	%	
	4-Chloro-3-methylphenol	99.9	%	
	4-Nitrophenol	53	%	
	2,4-Dinitrotoluene	94.6	%	
	Pentachlorophenol	26.6	%	
	Di-n-butylphthalate	2.15	%	

EMB
Radian Work Order: P1-09-010

Method:Twenty TICS to be reported (1)
List:

Sample ID:	M6-0918-PAH-F-B (6-11)	M6-0919-PAH-1 17,18,20-23	M6-0919-PAH-2 (35, 37-41)	M6-0920-PAH-3 (48, 50-54)
Factor:	5	5	5	5
Results in:	Total ug	Total ug	Total ug	Total ug
	02A	04A	06A	08A
Matrix:	XAD-2/Fi	Stack	Stack	Stack

	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit	Result	Det. Limit

(1) For a detailed description of flags and technical terms in this report refer to Appendix A in this report.

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
M6-0918-PAH-FB (6-11)				
	Toluene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	Cyclohexene	NQ	Total ug	
	1,1'-Bicyclopropyl	NQ	Total ug	
	2,3-Dimethylheptane	NQ	Total ug	
	2-Methyloctane	NQ	Total ug	
	1,4-Diethylbenzene	NQ	Total ug	
	1,3-Diethylbenzene	NQ	Total ug	
	1-Methyl-2-(2-propenyl)-benzen	NQ	Total ug	
	1-Ethenyl-4-ethylbenzene	NQ	Total ug	
	(E)-(1-methyl-1-propenyl)-	NQ	Total ug	
	benzene		Total ug	
	(1,1-Dimethylpropyl)-benzene	NQ	Total ug	
	Decahydro-2,3-dimethyl-	NQ	Total ug	
	naphthalene		Total ug	
	1,3,5-Triethylbenzene	NQ	Total ug	
	Unknown oxygenated branched	NQ	Total ug	
	hydrocarbons		Total ug	
	Unkown unsaturated branched	NQ	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
	hydrocarbon		Total ug	
M6-0919-PAH-1 17,18,20-23				
	Cyclohexene	NQ	Total ug	
	Toluene	NQ	Total ug	
	2-Methyl-1-Heptene	NQ	Total ug	
	1-Octene	NQ	Total ug	
	4-Methyl-3-penten-2-one	NQ	Total ug	
	Octane	NQ	Total ug	
	2-Methyloctane	NQ	Total ug	
	Trimethylnaphthalenes	NQ	Total ug	
	Dimethylbenzenes	NQ	Total ug	
	1-Heptyl-2-methylcyclopropane	NQ	Total ug	
	Heptadecane	NQ	Total ug	
	Hexadecane	NQ	Total ug	
	Propylcyclohexane	NQ	Total ug	
	3-Methylnonane	NQ	Total ug	
	2,3-Dimethyloctane	NQ	Total ug	
	Ethylmethylbenzenes	NQ	Total ug	
	2,6-Dimethylheptadecane	NQ	Total ug	
	1-Heptyl-2-methylcyclopropane	NQ	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
	Trimethylbenzene	NQ	Total ug	
	Decane	NQ	Total ug	
M6-0919-PAH-2 (35, 37-41)				
	2,6-Dimethylnonane	NQ	Total ug	
	Trimethylbenzene isomer	NQ	Total ug	
	1-Ethenyl-2-methylbenzene	NQ	Total ug	
	1,3-Diethylbenzene	NQ	Total ug	
	1,4-Diethylbenzene	NQ	Total ug	
	1,2-Diethylbenzene	NQ	Total ug	
	2-Ethyl-1,4-Dimethylbenzene	NQ	Total ug	
	1-Methyl-2-(2-propenyl)-benzen	NQ	Total ug	
	4,7-Dimethylundecane	NQ	Total ug	
	1,3-Diethyl-5-methylbenzene	NQ	Total ug	
	1-Ethyl-2,3-dimethylbenzene	NQ	Total ug	
	(E)-(1-Methyl-1-propenyl)-benz	NQ	Total ug	
	5,6-Dimethylundecane	NQ	Total ug	
	naphthalene substituted with a	NQ	Total ug	
	branch alkyl chain		Total ug	
	1,1-Dimethylpropylbenzene	NQ	Total ug	
	1,6-Dimethylnaphthalene	NQ	Total ug	

EMB
Radian Work Order: P1-09-010

Tentatively Identified Compounds
Method: Twenty TICS to be reported (1)
List:

Sample ID	Analyte	Result	Units	Scan
	2,6-Dimethylheptadecane	NQ	Total ug	
	Hexadecane	NQ	Total ug	
M6-0920-PAH-3 (48, 50-54)				
	4,7-Dimethylundecane	NQ	Total ug	
	1,6-Dimethylnaphthalene	NQ	Total ug	
	1,8-Dimethylnaphthalene	NQ	Total ug	
	1,5-Dimethylnaphthalene	NQ	Total ug	
	2,6-Dimethylheptadecane	NQ	Total ug	
	Hexadecane	NQ	Total ug	
	Heptadecane	NQ	Total ug	

Appendix A

Comments, Notes and Definitions

EMB
Radian Work Order: P1-09-010

Sample Identifications and Dates

Sample ID	M6-0918-PAH-F-B (6-11)	M6-0919-PAH-1 17,18,20-23	M6-0919-PAH-2 (35, 37-41)	M6-0920-PAH-3 (48, 50-54)	M6-0918-ALD-F-B (12, 13)	M6-0919-ALD-1 (28, 29)
Date Sampled	09/18/91	09/19/91	09/19/91	09/20/91	09/18/91	09/19/91
Date Received	09/23/91	09/23/91	09/23/91	09/23/91	09/23/91	09/23/91
Matrix	XAD-2/FI	Stack	Stack	Stack	Stack	Stack
	02	04	06	08	09	10
SW8270-Semi-Volatiles						
Prepared	09/30/91	09/30/91	09/30/91	09/30/91		
Analyzed	11/05/91	11/05/91	11/05/91	11/05/91		
Analyst	RK	RK	RK	RK		
File ID	4586538.TI	4586535.TI	4586536.TI	4586537.TI		
Blank ID	4586532.TI	4586532.TI	4586532.TI	4586532.TI		
Instrument	GC/MS B	GC/MS B	GC/MS B	GC/MS B		
Report as	received	received	received	received		
Aldehydes, Mod TO11, HPLC						
Prepared					09/30/91	09/30/91
Analyzed					10/18/91	10/18/91
Analyst					LKK	LKK
File ID					OWEN125	OWEN126/175
Blank ID					LLWA75	LLWA75
Instrument					V5000	V5000
Report as					received	received
Twenty TICS to be reported						
Prepared	09/30/91	09/30/91	09/30/91	09/30/91		
Analyzed	11/05/91	11/05/91	11/05/91	11/05/91		
Analyst	RK	RK	RK	RK		
File ID	4586538.TI	4586535.TI	4586536.TI	4586537.TI		
Blank ID	4586532.TI	4586532.TI	4586532.TI	4586532.TI		
Instrument	GC/MS B	GC/MS B	GC/MS B	GC/MS B		
Report as	received	received	received	received		

EMB
Radian Work Order: P1-09-010

General Comments
Aldehyde Data: Y - Outside of calibration curve.
Q - Spike recovery QC limits range from 80 to 120 percent.
Calibration check QC limits range from 85 to 115 percent.
NA - The butyraldehydes and MEK coeluted.

EMB
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B This flag indicates that the analyte was detected in the reagent blank but the sample results are not corrected for the amount in the blank.

J Indicates an estimated value for GC/MS data. This flag is used either when estimating a concentration for tentatively identified compounds where a response factor of 1 is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit.

NA This analyte was not analyzed.

ND This flag (or <) is used to denote analytes which are not detected at or above the specified detection limit. The value to the right of the < symbol is the method specified detection limit for the sample.

NS This analyte or surrogate was not added (spiked) to the sample for this analysis.

Q This quality control standard is outside method or laboratory specified control limits. This flag is applied to matrix spike, analytical QC spike, and surrogate recoveries; and to RPD(relative percent difference) values for duplicate analyses and matrix spike/matrix spike duplicate result.

* The asterisk(*) is used to flag results which are less than five times the method specified detection limit. Studies have shown that the uncertainty of the analysis will increase exponentially as the method detection limit is approached. These results should be considered approximate.

EMB
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TERMS USED IN THIS REPORT:

Analyte - A chemical for which a sample is to be analyzed. The analysis will meet EPA method and QC specifications.

Compound - See Analyte.

Detection Limit - The method specified detection limit, which is the lower limit of quantitation specified by EPA for a method. Radian staff regularly assess their laboratories' method detection limits to verify that they meet or are lower than those specified by EPA. Detection limits which are higher than method limits are based on experimental values at the 99% confidence level. The detection limits for EPA CLP (Contract Laboratory Program) methods are CRQLs (contract required quantitation limits) for organics and CRDLs (contract required detection limits) for inorganics. Note, the detection limit may vary from that specified by EPA based on sample size, dilution or cleanup. (Refer to Factor, below)

EPA Method - The EPA specified method used to perform an analysis. EPA has specified standard methods for analysis of environmental samples. Radian will perform its analyses and accompanying QC tests in conformance with EPA methods unless otherwise specified.

Factor - Default method detection limits are based on analysis of clean water samples. A factor is required to calculate sample specific detection limits based on alternate matrices (soil or water), reporting units, use of cleanup procedures, or dilution of extracts/digestates. For example, extraction or digestion of 10 grams of soil in contrast to 1 liter of water will result in a factor of 100.

Matrix - The sample material. Generally, it will be soil, water, air, oil, or solid waste.

Radian Work Order - The unique Radian identification code assigned to the samples reported in the analytical summary.

Units -	ug/L	micrograms per liter (parts per billion);liquids/water
	ug/kg	micrograms per kilogram (parts per billion); soils/solids
	ug/M3	micrograms per cubic meter; air samples
	mg/L	milligrams per liter (parts per million);liquids/water
	mg/kg	milligrams per kilogram (parts per million);soils/solids
	%	percent; usually used for percent recovery of QC standards
	uS/cm	conductance unit; microSiemens/centimeter
	mL/hr	milliliters per hour; rate of settlement of matter in water
	NTU	turbidity unit; nephelometric turbidity unit
	CU	color unit; equal to 1 mg/L of chloroplatinate salt

APPENDIX F.5

SAMPLE ID LOG

Project No.

LE Book No. 25284

om Page No.

Sequential Number

Sample ID		Date	Sample soln./type	Sample wt.	Analysis	
6-0918-MET-IC-FB	1	9/18	HNO_3/H_2O_2 ✓	503.0(g)	Metals ~~PAH~~ vol	Field
6-0918-MET-F-FB	2	9/18	Filter ✓	—	Part./Metals	Field
6-0918-MET-PR-FB	3		Acetone probe rinse ✓	160.2	Part	Field
6-0918-MET-APR-FB	4		Nitric probe rinse ✓	227.9	Metals	
6-0918-PAH-IC-FB	5		HPLC H_2O	912.6	PAH's	
6-0918-PAH-APR-FB	6		Acetone probe rinse	128.2	PAH's	
6-0918-PAH-MPR-FB	7		$MeCl_2$ probe rinse	277.37	PAH's	
6-0918-PAH-ACR-FB	8		Acetone IMP. coil rinse	112.7	PAH's	
6-0918-PAH-MCR-FB	9		$MeCl_2$ IMP. coil rinse	105.0	PAH's	
6-0918-PAH-F-FB	10		Filter	—	PAH's	
6-0918-PAH-SM-FB	11		XAD trap	—	PAH's	
6-0918-ALD-PR-FB	12 ✓		$MeCl_2$ probe rinse	310.4	Aldehydes	
6-0918-ALD-IC-FB	13 ✓		DNPH	392.1	⇓	
6-0918-PM10-ME-FB	14		$MeCl_2$ imp. rinse	169.5	PM10	
6-0918-PM10-IC-FB	15 ✓		H_2O imp. soln.	675.3	⇓	
6-0918-PM10-F-FB	16		Filter	—	⇓	
6-0919-PAH-F-1	17	9/19	Filter	—	PAH's	R
6-0919-PAH-SM-1	18 ✓		XAD trap	—		
6-0919-PAH-IC-1	19 ✓		HPLC H_2O	702.8		
6-0919-PAH-ACR-1	20		Acetone imp./coil rinse	246.6		
6-0919-PAH-MCR-1	21		$MeCl_2$ imp./coil rinse	357.6		
6-0919-PAH-APR-1	22		Acetone probe/nozzle rinse	151.5		
6-0919-PAH-MPR-1	23 ✓		$MeCl_2$ probe/nozzle rinse	185.0		
6-0919-MET-IC-1	24 ✓		HNO_3/H_2O_2 ✓	299.6 + Metals = 1161.1	Metals	
6-0919-MET-F-1	25 ✓	✓	Filter ✓	—	Part./Metals	
6-0919-MET-PR-1	26 ✓	✓	Acetone probe/nozzle rinse	98.1 ✓	Particulate	
6-0919-MET-APR-1	27 ✓		Aceto probe/nozzle rinse	203.1 ✓	Metals	
6-0919-ALD-PR-1	[redacted]	✓	$MeCl_2$ probe/nozzle rinse	127.9	Aldehydes	
6-0919-ALD-IC-1	[redacted]	✓	DNPH	1090.1	⇓	
6-0919-PM10-F-1	29 ✓	✓	Filter	— ✓	Part PM10	✓
6-0918-PM10-CYC-FB	31 ✓	9/18	cyclone contents + Acetone probe/cyc rinse	78.6 ✓		Ru
6-0919-PM10-CYC-1	32 ✓	9/19	cyclone contents + Acetone probe/cyc rinse	66.1 ✓		
6-0919-PM10-ME-1	33		$MeCl_2$ probe & imp. rinse	378.2 ✓		
6-0919-PM10-IC-1	34	✓	DI H_2O	701.5 ✓	⇓	To Page

Witnessed & Understood by me,	Date	Invented by	Date

RADIAN CORPORATION Project No. _____ Book No. 25284 TITLE _____

om Page No. __

Sample ID		Date	Sample sdn./type	Sample wt.		Analysis	
16-0919-PAH-SM-2	35	9/19	XAD trap	—		PAH's	Run.
16-0919-PAH-IC-2	36		HPLC H_2O	754.8			
16-0919-PAH-F-2	37		Filter	—			
16-0919-PAH-ACR-2	38		Acetone imp./coil rinse	248.5			
16-0919-PAH-MCR-2	39		$MeCl_2$ imp./coil rinse	304.1			
16-0919-PAH-APR-2	40		Acetone probe/nozzle rinse	87.9			
16-0919-PAH-MPR-2	41		$MeCl_2$ probe/nozzle rinse	126.5			
16-0919-MET-IC-2	42		HNO_3/H_2O_2	960.5	✓	Metals	
16-0919-MET-PR-2	43	✓	Acetone probe/nozzle rinse	111.6	✓	particulate	
16-0919-MET-APR-2	44		Acid probe/nozzle rinse	143.0		Metals	
16-0919-MET-F-2	45	✓	Filter	—	✓	Particulate/Metals	
16-0920-ALD-IC-2	46	9/20	DNPH	1076.4		Aldehydes	Ru.
16-0920-ALD-PR-2	47		$MeCl_2$ rinse	441.9			
16-0920-PAH-SM-3	48	✓	XAD trap	—		PAH's	Run
16-0920-PAH-IC-3	49	✓	HPLC H_2O	767.2			
16-0920-PAH-ACR-3	50	✓	Acetone imp./coil rinse	309.2			
16-0920-PAH-MCR-3	51	✓	$MeCl_2$ imp./coil rinse	472.4			
16-0920-PAH-F-3	52	✓	Filter	—			
16-0920-PAH-APR-3	53	✓	Acetone probe/nozzle rinse	208.1			
16-0920-PAH-MPR-3	54	✓	$MeCl_2$ probe/nozzle rinse	334.9			
16-0920-PM10-CYC-2	55	✓	cyclone contents + Acetone rinses	73.4	✓	PM10	Run
16-0920-PM10-IC-2	56	✓	DI H_2O	816	✓		
16-0920-PM10-ME-2	57	✓	$MeCl_2$ probe/imp. rinses	388.7	✓		
16-0920-PM10-F-2	58	✓	Filter	—	✓		
16-0920-MET-IC-3	59	✓	HNO_3/H_2O_2	1019.1	✓	Metals	Run
16-0920-MET-F-3	60	✓	Filter	—	✓	Part./Metals	
16-0920-MET-APR-3	61	✓	Acid probe/nozzle rinse	312.1	✓	Metals	
16-0920-MET-PR-3	62	✓	Acetone probe/nozzle rinse	218.5	✓	particulate	
16-0920-ALD-IC-3	63	✓	DNPH	1061.7		Aldehydes	Run
16-0920-ALD-PR-3	64	✓	$MeCl_2$ probe/nozzle rinse	425.4			
16-0920-PM10-CYC-3	65	✓	Cyclone contents + Acetone rinses	47.5	✓	PM-10	Run
16-0920-PM10-IC-3	66	✓	DI H_2O	811.4	✓	PM10	
16-0920-PM10-ME-3	67	✓	$MeCl_2$ probe/imp. rinse	311.5	✓	PM-10	
16-0920-PM10-F-3	68	✓	Filter	—	✓	PM-10	

To Page !

Witnessed & Understood by me. | Date | Invented by | Date

APPENDIX G

CALIBRATION DATA SHEETS

Meter Box Calibration

Post Test - Mathey

Date	10/3/91	Cal Meter #	6830284	Pbar	29.9
Box #	N-30	Cal Meter Yd	0.9967	Vaccum	10 " Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Cal. Temp Out	Meter Temp In	Meter Temp Out	Time	Yd	Delta H@
1.1	-0.07	Final	43.149	52.743	68	67	70	68	6		
		Init.	39.743	49.400	68	67	71	69		1.0164	1.92
		Total	3.406	3.343	Avg.	67.5	Avg.	69.5			
1.1	-0.07	Final	49.823	59.250	66	67	71	68	11		
		Init.	43.515	53.100	66	67	70	69		1.0252	1.87
		Total	6.308	6.150	Avg.	66.5	Avg.	69.5			
1.1	-0.07	Final	57.947	67.182	67	67	70	69	14.25		
		Init.	49.823	59.250	68	68	73	70		1.0237	1.90
		Total	8.124	7.932	Avg.	67.5	Avg.	70.5			
									Average	1.0218	1.90

Meter Box Calibration

Post Test - Mathey

Date 10/4/91 Cal Meter # 6830284 Pbar 29.88

Box # N-32 Cal Meter Yd 0.9967 Vaccum 12 " Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Cal. Temp Out	Meter Temp In	Meter Temp Out	Time	Yd	Delta H@
1.0	-0.06	Final	32.507	47.087	65	65	71	69	18		
		Init.	22.882	37.450	66	66	73	70		1.0028	1.95
		Total	9.625	9.637	Avg.	65.5	Avg.	70.8			
1.0	-0.06	Final	37.323	51.885	66	66	73	70	9		
		Init.	32.507	47.087	67	67	74	71		1.0083	1.95
		Total	4.816	4.798	Avg.	66.5	Avg.	72.0			
1.0	-0.06	Final	44.398	58.962	67	67	73	71	13.25		
		Init.	37.323	51.885	67	67	75	71		1.0042	1.96
		Total	7.075	7.077	Avg.	67.0	Avg.	72.5			
									Average	1.0051	1.95

Meter Box Calibration

Post Test - Mathey

Date	10/15/91	Cal Meter #	6830284	Pbar	29.88
Box #	N-33	Cal Meter Yd	0.9967	Vaccum	11 " Hg

Orifice Setting	Cal. Meter Pressure	Gas Volume Cal. Meter		Gas Volume Meter Box	Cal. Temp In	Cal. Temp Out	Meter Temp In	Meter Temp Out	Time	Yd	Delta H@
1.1	-0.07	Final	67.375	76.428	70	70	73	70	11		
		Init.	61.057	70.000	72	72	78	72		0.9810	1.89
		Total	6.318	6.428	Avg.	71.0	Avg.	73.3			
1.1	-0.07	Final	73.116	82.298	73	73	80	75	10		
		Init.	67.375	76.428	72	72	77	73		0.9788	1.89
		Total	5.741	5.870	Avg.	72.5	Avg.	76.3			
1.1	-0.007	Final	79.410	88.777	73	73	82	78	11		
		Init.	73.116	82.298	72	72	79	75		0.9765	1.90
		Total	6.294	6.479	Avg.	72.5	Avg.	78.5			
									Average	0.9788	1.89

APPENDIX H

SAMPLE EQUATIONS

SAMPLE CALCULATIONS

COMPANY : BORGESS MEDICAL CENTER
PLANT SITE : KALAMAZOO, MI
SAMPLING LOCATION : BAGHOUSE OUTLET
DATE : 09/07/91
EXAMPLE #1: PM/Metals , RUN 02
STANDARD CONDITIONS: 68 F, 29.92 in Hg

INPUT PARAMETERS:

As	= 551.55 sq.in.	Ps	= 29.48 in. Hg	PM COLLECT.	= 0.0102 gran		
Cp	= 0.84	%CO2	= 4.69	AVG SQRT DEL.P	= 0.2546 IN H		
Dn	= 0.313 in.	%N2	= 80.26	MOISTURE	= 211.60 gran		
Kp	= 84.59	%O2	= 15.05	COLLECTED			
P(std)	= 29.92 in. Hg	T(std)	= 68.00 F	SAMPL TIME	= 240 min.		
Pb	= 29.52 in. Hg	Tm(avg)	= 110.09 F	Ts(avg)	= 294.42 F		
Pmg(avg)	= 0.45 in. H2O	Vm	= 91.40 ft^3	Y	= 1.0108		

1) Volume of dry gas sampled at standard conditions:

$$Pm = Pmg/13.6 + Pb \qquad Pm = 29.5530$$

$$Vm(std) = \frac{Y * Vm * (T(std) + 460) * Pm}{P(std) * (Tm(avg) + 460)}$$

$$Vm(std) = (1.011 * 91.40 * (68 + 460) * 29.55) / (29.92 * (110.09 + 460))$$

$$Vm(std) = 84.52 \text{ dscf}$$

2) Volume of water vapor at standard conditions:

$$Vw(gas) = 0.04707 \text{ ft3/g} * (\text{moisture collected})$$

$$Vw(gas) = (0.04707 * 211.60)$$

$$Vw(gas) = 9.95 \text{ scf}$$

3) Percent moisture in stack:

$$\%V = \frac{100 * Vw(gas)}{Vm(std) + Vw(gas)}$$

$$\%V = (100 *9.95) / (84.52 + 9.95)$$

$$\%V = 10.53$$

4) Mole fraction of dry stack gas:

$$MFd = \frac{100 - \%V}{100}$$

$$MFd = (100 - 10.53) / 100$$

$$MFd = 0.895$$

DEFINITION OF TERMS

SYMBOL	DEFINITIONS	UNITS
As	AREA OF STACK	in.^2
Cp	PITOT COEFFICIENT	--
Ca	CONCENTRATION OF PARTICULATE	grains/ft^3
Dn	DIAMETER OF SAMPLING NOZZLE	in.
ER	EMISSION RATE OF PARTICULATE	lb/hr
Kp	PITOT TUBE COEFFICIENT	--
MFd	MOLE FRACTION OF DRY STACK GAS	--
MWd	MOLECULAR WEIGHT OF DRY STACK GAS	lb/lb-mole
MWw	MOLECULAR WEIGHT OF WET STACK GAS	lb/lb-mole
	MOISTURE COLLECTED IN IMPINGERS	grams
P(std)	STANDARD PRESSURE (29.92 in. Hg)	in. Hg
Pb	BAROMETRIC PRESSURE	in. Hg
Pmg(avg)	AVERAGE GAUGE METER PRESSURE	in. Hg
Ps	ABSOLUTE STACK PRESSURE	in. Hg
	PARTICULATE CATCH	grams
Qsd	AVERAGE STACK DRY VOLUMETRIC FLOW RATE	dry ft^3/min.
%CO2	PERCENT CO2 IN STACK GAS	--
%N2	PERCENT N2 IN STACK GAS	--
%O2	PERCENT O2 IN STACK GAS	--
%V	PERCENT MOISTURE IN STACK	--
%XS	PERCENT EXCESS AIR	--
	TOTAL SAMPLING TIME	min.
T(std)	STANDARD TEMPERATURE (68 F)	F
Tm(avg)	AVERAGE TEMPERATURE OF THE METER	F
Ts(avg)	AVERAGE TEMPERATURE OF THE STACK	F
Vm	TOTAL METERED VOLUME	ft^3
Vm(std)	STANDARD METERED VOLUME	dry standard ft^3
Vw(gas)	VOLUME OF WATER IN STACK GAS	standard ft^3
Vs	VELOCITY OF STACK GAS	ft/min
Y	TEST METER CALIBRATION COEFFICIENT	--

10) Excess air (%):

$$\%XS = \frac{100 * \%O2}{(0.264 * \%N2) - \%O2}$$

%XS = (100 * 15.05) / ((0.264 * 80.26) - 15.05)

%XS = 244.53

11) Concentration of particulate:

Ca = (particulate catch) / Vm(std) / 453.59 * 7000

Ca = 0.0102 / 84.52 / 453.59 * 7000

Ca = 0.00186 grains/dscf

12) Particulate Emissions Rate:

ER = (concentration) * (Qsd) * 60 / 7000

ER = 0.0019 * 2444.95 * 60 / 7000

ER = 0.039 lb/hr

5) Average molecular weight of dry stack gas:

MWd = (0.44 * %CO2) + (0.32 * %O2) + (0.28 * %N2)

MWd = (0.44 * 4.69) + (0.32 * 15.05) + (0.28 * 80.26)

MWd = 29.35 lb/lb-mole

6) Average molecular weight of wet stack gas:

MWw = MWd * MFd + 18.0 * (1.0 - MFd)

MWw = 29.35 * 0.895 + 18.0 * (1.0 - 0.895)

MWw = 28.16 lb/lb-mole

7) Stack velocity (feet/min) at stack conditions:

Vs = Kp*Cp*[SQRT(dP)]avg*(SQRT[(Ts)avg])*[SQRT(1/Ps*MWw)]*60

Vs = 84.59 * 0.84 * 0.25 * SQRT[(294.42 +460) / (29.48 * 28.16)]

Vs = 17.24665 fps
1034.799 fpm

8) Average stack dry volumetric flow rate:

$$Qsd = \frac{Vs * As * MFd * (T(std)+460) * Ps}{144\ sq.in./cu.ft. * (Ts(avg) + 460) * P(std)}$$

Qsd = 17.25 * 551.55 * 0.895 * (68.0 + 460) * 29.48 / (144 * (294.42 + 460) * 29.92)

Qsd = 2444.95 dscfm
69.24238 dscmm

9) Isokinetic sampling rate (%):

$$\%I = \frac{1039.5746 * Vm(std) * (Ts(avg) + 460)}{Vs * samp.\ time * Ps * MFd * (Dn)^2}$$

%I = (1039.5746 * 84.52 * (294.42 + 460)) / (1034.80 * 240 * 29.48 * 0.895 * (0.313)^2)

%I = 103.24

APPENDIX I

PROJECT PARTICIPANTS

PROJECT PARTICIPANTS

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APPENDIX J

SAMPLING AND ANALYTICAL PROTOCOLS

J.1 PM/Metals
J.2 PM_{10}/CPM
J.3 Aldehydes
J.4 PAH
J.5 CEM and GC

APPENDIX J.1

PM/METALS

METHODOLOGY FOR THE DETERMINATION OF METALS EMISSIONS IN EXHAUST GASES FROM INCINERATION PROCESSES

1. Applicability and Principle

1.1 Applicability. This method is applicable for the determination of arsenic (As), beryllium (Be), cadmium (Cd), total chromium (Cr), lead (Pb), mercury (Hg), nickel (Ni), and zinc (Zn) emissions from municipal waste incinerators and similar combustion processes. These elements are referred to hereafter as the primary metals. This method may also be used for the determination of antimony (Sb), barium (Ba), copper (Cu), manganese (Mn), phosphorus (P), selenium (Se), silver (Ag), and thallium (Tl) emissions from these sources. These elements are referred to hereafter as the secondary metals.

In addition, the method may be used to determine particulate emissions by following the additional procedures described. Modifications to the sample recovery and analysis procedures described in this protocol for the purpose of determining particulate emissions may potentially impact the front half mercury determination.*

1.2 Principle. The stack sample is withdrawn isokinetically from the source, with particulate emissions collected in the probe and on a heated filter and gaseous emissions collected in a series of chilled impingers containing a solution of dilute nitric acid in hydrogen peroxide in two impingers, and acidic potassium permanganate solution in two (or one) impingers. Sampling train components are recovered and digested in separate front and back half fractions. Materials collected in the sampling train are digested with acid solutions to dissolve inorganics and to remove organic constituents that may create analytical interferences. Acid digestion is performed using conventional Parr® Bomb or microwave digestion techniques. The

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*Field tests to date have shown that of the total amount of mercury measured by the method, only 0 to <2% was measured in the front half. Therefore, it is tentatively concluded, based on the above data, that particulate emissions may be measured by this train, without significantly altering the mercury results.

nitric acid and hydrogen peroxide impinger solution, the acidic potassium permanganate impinger solution, and the probe rinse and digested filter solutions are analyzed for mercury by cold vapor atomic absorption spectroscopy (CVAAS). Except for the permanganate solution, the remainder of the sampling train fractions are analyzed for As, Be, Cd, Cr, Pb, Ni, and Zn (and Sb, Ba, Cu, Mn, P, Se, Ag, and Tl, if desired) by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of As, Cd, and Pb (and Sb, Se, and Tl, when measured) if these elements require greater analytical sensitivity than can be obtained by ICAP. Additionally, if desired, the tester may use AAS for analyses of all target metals if the resulting in-stack method detection limits (combined sampling and analytical detection limits) meet the data quality objectives of the testing program. For convenience, aliquots of each digested sample fraction can be combined proportionally for a single analytical determination. The efficiency of the analytical procedure is quantified by the analysis of spiked quality control samples containing each of the target metals including actual sample matrix effects checks.

2. Range, Sensitivity, Precision, and Interferences

2.1 Range. For the analyses described in this methodology and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per milliliter (ng/ml) to micrograms per milliliter (ug/ml) range in the analytical finish solution can be analyzed using this technique. Samples containing greater than approximately 50 ug/ml of arsenic, chromium, or lead should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20 ug/ml of cadmium should be diluted to that level before analysis.

2.2 Analytical Sensitivity. ICAP analytical detection limits for the primary [and secondary] metals in the sample solutions (based on SW-846, Method 6010) are approximately as follows: As (53 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Pb (42 ng/ml), Ni (15 ng/ml), Zn (2 ng/ml) [Sb (32 ng/ml), Ba (2 ng/ml), Cu (6 ng/ml), P (75 ng/ml), Mn (2 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Ti (40 ng/ml)]. The actual method detection limits are sample dependent and may vary as the sample matrix may affect the limits. The

analytical detection limits for the primary [and secondary] metals in sample solutions analyzed by direct aspiration AAS (based on SW-846, Method 7000) are approximately as follows: As (2 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Pb (100 ng/ml), Ni (40 ng/ml), Zn (5 ng/ml) [Sb (200 ng/ml), Ba (100 ng/ml), Cu (20 ng/ml), Mn (10 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml)]. The detection limit for mercury by CVAAS is approximately 0.2 ng/ml. The use of GFAAS can give added sensitivity compared to the use of direct aspiration AAS for the following primary and secondary metals: As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Pb (1 ng/ml), Sb (3 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml).

Using (1) the procedures described in this method, (2) the analytical detection limits described in the previous paragraph, (3) a volume of 300 ml for the front half and 150 ml for the back half samples, and (4) a stack gas sample volume of 1.25 m^3, the corresponding in-stack method detection limits are presented in Table A-1 and calculated as shown:

$$\frac{A \times B}{C} = D$$

where: A = analytical detection limit, ug/ml.
B = volume of sample prior to aliquot for analysis, ml.
C = stack sample volume, dscm (dsm^3).
D = in-stack detection limit, ug/m^3.

Values in Table A-1 are calculated for the front and back half and/or the total train.

To ensure optimum sensitivity in obtaining the measurements, the concentrations of target metals in the solutions are suggested to be at least ten times the analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, this concentration can be as low as approximately three times the analytical detection limit. In all cases, repetitive analyses, method of standard additions (MSA), serial dilution, or matrix spike addition should be used to establish the quality of the data.

Actual in-stack method detection limits will be determined based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be made more sensitive than those shown in Table A-1 for a specific test by using one or more of the following options:

TABLE A-1. IN-STACK METHOD DETECTION LIMITS (ug/m^3)
FOR TRAIN FRACTIONS USING ICAP AND AAS

Metal	Front Half Fraction 1 Probe and Filter	Back Half$_1$ Fraction 2 Impingers 1-3	Back Half$_2$ Fraction 3 Impingers 4-5	Total Train
		Primary Metals		
Arsenic	12.7 (0.3)*	6.4 (0.1)*		19.1 (0.4)*
Beryllium	0.07 (0.05)*	0.04 (0.03)*		0.11 (0.08)*
Cadmium	1.0 (0.02)*	0.5 (0.01)*		1.5 (0.03)*
Chromium	1.7 (0.2)*	0.8 (0.1)*		2.5 (0.3)*
Lead	10.1 (0.2)*	5.0 (0.1)*		15.1 (0.3)*
Mercury	0.05**	0.03**	0.03**	0.11**
Nickel	3.6	1.8		5.4
Zinc	0.5	0.3		0.8
		Secondary Metals		
Antimony	7.7 (0.7)*	3.8 (0.4)*		11.5 (1.1)*
Barium	0.5	0.3		0.8
Copper	1.4	0.7		2.1
Manganese	0.5 (0.2)*	0.2 (0.1)*		0.7 (0.3)*
Phosphorus	18	9		27
Selenium	18 (0.5)*	9 (0.3)*		27 (0.8)*
Silver	1.7	0.9		2.6
Thallium	9.6 (0.2)*	4.8 (0.1)*		14.4 (0.3)*

( )* Detection limit when analyzed by GFAAS.
** Detection limit when analyzed by CVAAS.
Actual method in-stack detection limits will be determined based on actual source sampling parameters and analytical results as described earlier in this section.

- A normal 1-hour sampling run collects a stack gas sampling volume of about 1.25 m^3. If the sampling time is increased and 5 m^3 are collected, the in-stack method detection limits would be one fourth of the values shown in Table A-1 (this means that with this change, the method is four times more sensitive than normal).
- The in-stack detection limits assume that all of the sample is digested (with exception of the aliquot for mercury) and the final liquid volumes for analysis are 300 ml for the front half and 150 ml for the back half sample. If the front half volume is reduced from 300 ml to 30 ml, the front half in-stack detection limits would be one tenth of the values shown above (ten times more sensitive). If the back half volume is reduced from 150 ml to 25 ml, the in-stack detection limits

would be one sixth of the above values. Matrix effects checks are necessary on analyses of samples and typically are of greater significance for samples that have been concentrated to less than the normal sample volume. A volume less than 25 ml may not allow resolubilization of the residue and may increase interference by other compounds.

- o When both of the above two improvements are used on one sample at the same time, the resultant improvements are multiplicative. For example, where stack gas volume is increased by a factor of five and the total liquid sample digested volume of both the front and back halves is reduced by factor of six, the in-stack method detection limit is reduced by a factor of thirty (the method is thirty times more sensitive).
- o Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase limits. The front half and back half samples (Fractions 1 and 2) can be combined prior to analysis. The resultant liquid volume (excluding Fraction 3, which must be analyzed separately) is recorded. Combining the sample as described does not allow determination (whether front or back half) of where in the train the sample was captured. The in-stack method detection limit then becomes a single value for all target metals except mercury, for which the contribution of Fraction 3 must be considered.
- o The above discussion assumes no blank correction. Blank corrections are discussed later in this method.

2.3 Precision. The precisions (relative standard deviations) for each of the primary and secondary metals detected in a method development test at a sewage sludge incinerator, are as follows: As (13.5%), Cd (11.5%), Cr (11.2%), Pb (11.6%), Zn (11.8%), Sb (12.7%), Ba (20.6%), Cu (11.5%), P (14.6%), Se (15.3%), and Tl (12.3%). The precision for nickel was 7.7% for another test conducted at a source simulator. Beryllium, manganese and silver were not detected in the tests; however, based on the analytical sensitivity of the ICAP for these metals, it is assumed that their precisions should be similar to those for the other metals, when detected at similar levels.

2.4 Interferences. Iron can be a spectral interference during the analysis of arsenic, chromium, and cadmium by ICAP. Aluminum can be a spectral interference during the analysis of arsenic and lead by ICAP. Generally, these interferences can be reduced by diluting the sample, but this increases the

in-stack method detection limit. Refer to EPA Method 6010 (SW-846) for details on potential interferences for this method. For all GFAAS analyses, matrix modifiers should be used to limit interferences, and standards should be matrix matched.

3. Apparatus

3.1 Sampling Train. A schematic of the sampling train is shown in Figure A-1. It is similar to the Method 5 train. The sampling train consists of the following components.

3.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as Method 5, Sections 2.1.1 and 2.1.2. Glass nozzles are requiredunless an alternate probe tip prevents the possibility of contamination or interference of the sample with its materials of construction. If a probe tip other than glass is used, no correction of the stack sample test results can be made because of the effect on the results by the probe tip.

3.1.2 Pitot Tube and Differential Pressure Gauge. Same as Method 2, Sections 2.1 and 2.2, respectively.

3.1.3 Filter Holder. Glass, same as Method 5, Section 2.1.5, except that a Teflon filter support must be used to replace the glass frit.

3.1.4 Filter Heating System. Same as Method 5, Section 2.1.6.

3.1.5 Condenser. The following system shall be used for the condensation and collection of gaseous metals and for determining the moisture content of the stack gas. The condensing system should consist of four to six impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is optional and is recommended as a water knockout trap for use during test conditions which require such a trap. The impingers to be used in the metals train are now described. When the first impinger is used as a water knockout, it shall be appropriately-sized for an expected large moisture catch and constructed generally as described for the first impinger in Method 5, Paragraph 2.1.7. The second impinger (or the first HNO_3/H_2O_2 impinger) shall also be as described for the first impinger in Method 5. The third impinger (or the impinger used as the second HNO_3/H_2O_2 impinger) shall be the same as the Greenburg Smith impinger with the standard tip described as the second impinger in Method 5, Paragraph 2.1.7. All other impingers used in the metals train are the same as the second impinger (the first HNO_3/H_2O_2 impinger) previously described in this paragraph. In summary,

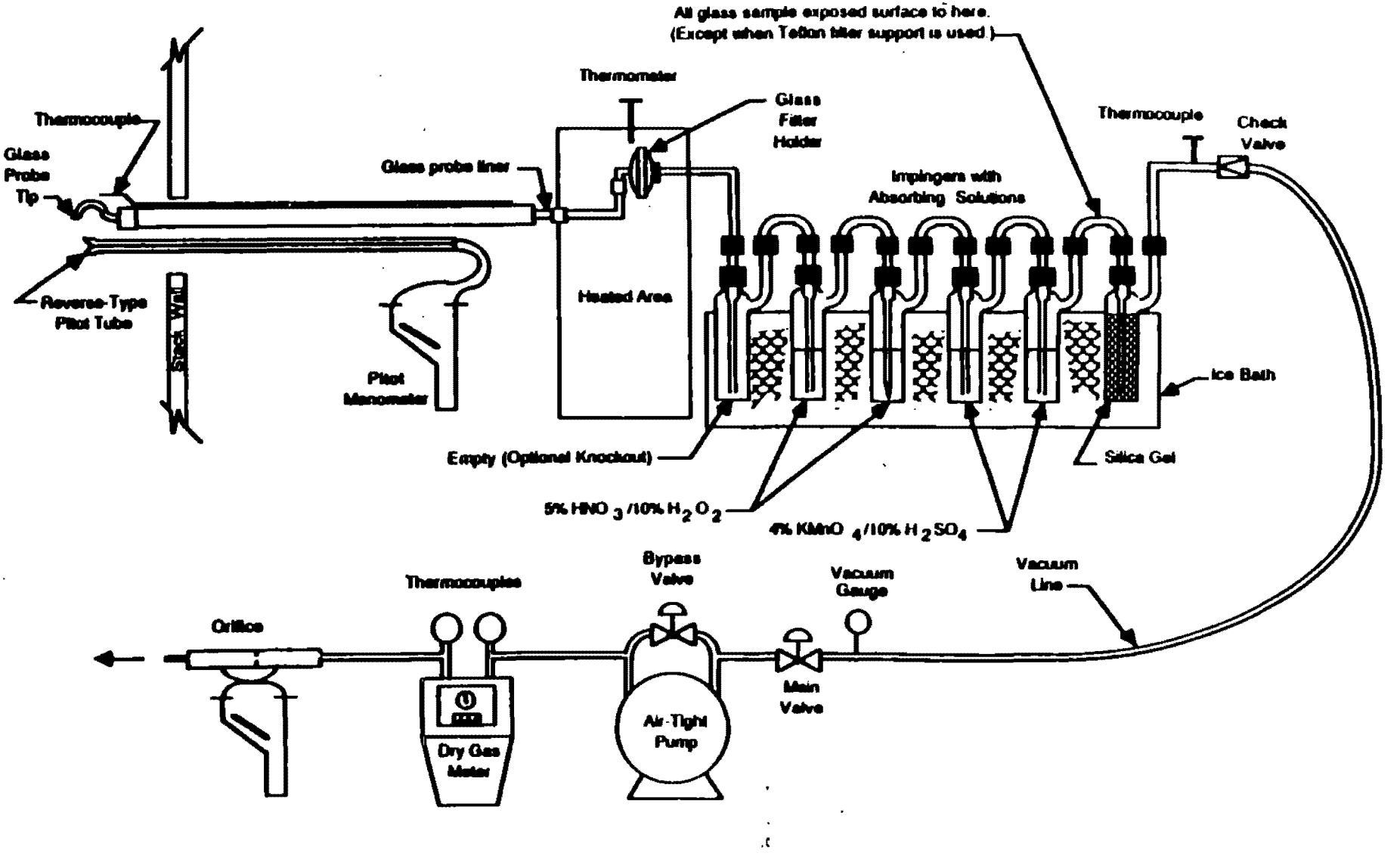


Figure A-1. Schematic of multiple metals sampling train.

the first impinger should be empty, the second and third shall contain known quantities of a nitric acid/hydrogen peroxide solution (Section 4.2.1), the fourth (and fifth, if required) shall contain a known quantity of acidic potassium permanganate solution (Section 4.2.2), and the last impinger shall contain a known quantity of silica gel or equivalent desiccant. A thermometer capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger. When the water knockout impinger is not needed, it is removed from the train and the other impingers remain the same. If mercury analysis is not needed, the potassium permanganate impingers are removed.

3.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1 through 2.1.10, respectively.

3.1.7 Teflon Tape. For capping openings and sealing connections on the sampling train.

3.2 Sample Recovery. Same as Method 5, Sections 2.2.1 through 2.2.8 (Nonmetallic Probe-Liner and Probe-Nozzle Brushes, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

3.2.1 Nonmetallic Probe-Liner and Probe-Nozzle Brushes. For quantitative recovery of materials collected in the front half of the sampling train. Description of acceptable all-Teflon component brushes to be included in EPA's Emission Measurement Technical Information Center (EMTIC) files.

3.2.2 Sample Storage Containers. Glass bottles with Teflon-lined caps, 1000- and 500-ml, shall be used for $KMnO_4$-containing samples and blanks. Polyethylene bottles may be used for other sample types.

3.2.3 Graduated Cylinder. Glass or equivalent.

3.2.4 Funnel. Glass or equivalent.

3.2.5 Labels. For identification of samples.

3.2.6 Polypropylene Tweezers and/or Plastic Gloves. For recovery of the filter from the sampling train filter holder.

3.3 Sample Preparation and Analysis. For the analysis, the following equipment is needed:

3.3.1 Volumetric Flasks, 100 ml, 250 ml, and 1000 ml. For preparation of standards and sample dilution.

3.3.2 Graduated Cylinders. For preparation of reagents.

3.3.3 Parr® Bombs or Microwave Pressure Relief Vessels with Capping

Station (CEM Corporation model or equivalent).

3.3.4 Beakers and Watchglasses. 250-ml beakers for sample digestion with watchglasses to cover the tops.

3.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

3.3.6 Filter Funnels. For holding filter paper.

3.3.7 Whatman 541 Filter Paper (or equivalent). For filtration of digested samples.

3.3.8 Disposable Pasteur Pipets and Bulbs.

3.3.9 Volumetric Pipets.

3.3.10 Analytical Balance. Accurate to within 0.1 mg.

3.3.11 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures.

3.3.12 Hot Plates.

3.3.13 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

3.3.13.1 Graphite Furnace Attachment. With As, Cd, and Pb (and Sb, Se, and Tl, if measured) hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). Same as EPA Methods 7060 (As), 7131 (Cd), 7421 (Pb), 7041 (Sb), 7740 (Se), and 7841 (Tl).

3.3.13.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL. The equipment needed for the cold vapor mercury attachment includes an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp should be capable of raising the ambient temperature at the quartz cell by 10°C such that no condensation forms on the wall of the quartz cell. Same as EPA Method 7470.

3.3.14 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. Same as EPA Method 6010.

4. Reagents

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

4.1 Sampling. The reagents used in sampling are as follows:

4.1.1 Filters. The filters shall contain less than 1.3 ug/in.^{2} of each of

the metals to be measured. Analytical results provided by filter manufacturers are acceptable. However, if no such results are available, filter blanks must be analyzed for each target metal prior to emission testing. Quartz fiber or glass fiber filters without organic binders shall be used. The filters should exhibit at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986-71 (incorporated by reference). For particulate determination in sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3, as described in EPA Method 5. Quartz fiber filters meeting these requirements are recommended.

4.1.2 Water. To conform to ASTM Specification D1193.77, Type II (incorporated by reference). Analyze the water for all target metals prior to field use. All target metals should be less than 1 ng/ml.

4.1.3 Nitric Acid. Concentrated. Baker Instra-analyzed or equivalent.

4.1.4 Hydrochloric Acid. Concentrated. Baker Instra-analyzed or equivalent.

4.1.5 Hydrogen Peroxide, 30 Percent (V/V).

4.1.6 Potassium Permanganate.

4.1.7 Sulfuric Acid. Concentrated.

4.1.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.4, respectively.

4.2 Pretest Preparation for Sampling Reagents.

4.2.1 Nitric Acid (HNO_3)/Hydrogen Peroxide (H_2O_2) Absorbing Solution. 5 Percent HNO_3/10 Percent H_2O_2. Add 50 ml of concentrated HNO_3 and 333 ml of 30 percent H_2O_2 to a 1000-ml volumetric flask or graduated cylinder containing approximately 500 ml of water. Dilute to volume with water. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.2 Acidic Potassium Permanganate ($KMnO_4$) Absorbing Solution, 4 Percent $KMnO_4$ (W/V). Prepare fresh daily. Dissolve 40 g of $KMnO_4$ in sufficient 10 percent H_2SO_4 to make 1 liter. Prepare and store in glass bottles to prevent degradation. The reagent shall contain less than 2 ng/ml of Hg.

Precaution: To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper. Also, due to reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottle; these bottles should not be fully filled

and should be vented both to relieve excess pressure and prevent explosion due to pressure buildup. Venting is highly recommended, but should not allow contamination of the sample; a No. 70-72 hole drilled in the container cap and Teflon liner has been used.

4.2.3 Nitric Acid, 0.1 N. Add 6.3 ml of concentrated HNO_3 (70 percent) to a graduated cylinder containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.4 Hydrochloric Acid (HCl), 8 N. Add 690 ml of concentrated HCl to a graduated cylinder containing 250 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of Hg.

4.3 Glassware Cleaning Reagents.

4.3.1 Nitric Acid, Concentrated. Fisher ACS grade or equivalent.

4.3.2 Water. To conform to ASTM Specifications D1193-77, Type II.

4.3.3 Nitric Acid, 10 Percent (V/V). Add 500 ml of concentrated HNO_3 to a graduated cylinder containing approximately 4000 ml of water. Dilute to 5000 ml with water.

4.4 Sample Digestion and Analysis Reagents.

4.4.1 Hydrochloric Acid, Concentrated.

4.4.2 Hydrofluoric Acid, Concentrated.

4.4.3 Nitric Acid, Concentrated. Baker Instra-analyzed or equivalent.

4.4.4 Nitric Acid, 10 Percent (V/V). Add 100 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

4.4.5 Nitric Acid, 5 Percent (V/V). Add 50 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Reagent shall contain less than 2 ng/ml of each target metal.

4.4.6 Water. To conform to ASTM Specifications D1193-77, Type II.

4.4.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See EPA Method 7470 for preparation.

4.4.8 Stannous Chloride.

4.4.9 Potassium Permanganate, 5 Percent (W/V).

4.4.10 Sulfuric Acid, Concentrated.

4.4.11 Nitric Acid, 50 Percent (V/V).

4.4.12 Potassium Persulfate, 5 Percent (W/V).

4.4.13 Nickel Nitrate, $Ni(NO_3)_2 \cdot 6H_2O$.

4.4.14 Lanthanum Oxide, La_2O_3.

4.4.15 AAS Grade As Standard, 1000 ug/ml.

4.4.16 AAS Grade Be Standard, 1000 ug/ml.

4.4.17 AAS Grade Cd Standard, 1000 ug/ml.

4.4.18 AAS Grade Cr Standard, 1000 ug/ml.

4.4.19 AAS Grade Pb Standard, 1000 ug/ml.

4.4.20 AAS Grade Hg Standard, 1000 ug/ml.

4.4.21 AAS Grade Ni Standard, 1000 ug/ml.

4.4.22 AAS Grade Zn Standard, 1000 ug/ml.

4.4.23 AAS Grade Al Standard, 1000 ug/ml.

4.4.24 AAS Grade Fe Standard, 1000 ug/ml.

4.4.25 AAS Grade Sb Standard, 1000 ug/ml. Optional.

4.4.26 AAS Grade Ba Standard, 1000 ug/ml. Optional.

4.4.27 AAS Grade Cu Standard, 1000 ug/ml. Optional.

4.4.28 AAS Grade Mn Standard, 1000 ug/ml. Optional.

4.4.29 AAS Grade P Standard, 1000 ug/ml. Optional.

4.4.30 AAS Grade Se Standard, 1000 ug/ml. Optional.

4.4.31 AAS Grade Ag Standard, 1000 ug/ml. Optional.

4.4.32 AAS Grade Tl Standard, 1000 ug/ml. Optional.

4.4.33 The metals standards may also be made from solid chemicals as described in EPA Method 200.7. EPA Method 7470 or Standard Methods for the Analysis of Water and Wastewater. 15th Edition, Method 303F should be referred to for additional information on mercury standards.

4.4.34 Mercury Standards and Quality Control Samples. Prepare fresh weekly a 10 ug/ml intermediate mercury standard by adding 5 ml of 1000 ug/ml mercury stock solution to a 500-ml volumetric flask; dilute to 500 ml by first adding 20 ml of 15 percent HNO_3 and then adding water. Prepare a working mercury standard solution fresh daily: add 5 ml of the 10 ug/ml intermediate standard to a 250 ml volumetric flask and dilute to 250 ml with 5 ml of 4 percent $KMnO_4$, 5 ml of 15 percent HNO_3, and then water. At least six separate aliquots of the working mercury standard solution should be used to prepare the standard curve. These aliquots should contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution. Quality control samples should be prepared by making a separate 10 ug/ml standard and diluting until in the range of the calibration.

4.4.35 ICAP Standards and Quality Control Samples. Calibration standards

for ICAP analysis can be combined into four different mixed standard solutions as shown below.

MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

Solution	Elements (secondary metals in parantheses)
I	As, Be, Cd, Pb, Zn (Mn, Se)
II	Fe (Ba, Cu)
III	Al, Cr, Ni
IV	(Sb, P, Ag, Tl)

Prepare these standards by combining and diluting the appropriate volumes of the 1000 ug/ml solutions with 5 percent nitric acid. A minimum of one standard and a blank can be used to form each calibration curve. However, a separate quality control sample spiked with known amounts of the target metals in quantities in the midrange of the calibration curve should be prepared. Suggested standard levels are 50 ug/ml for Al, 25 ug/ml for Cr and Pb, 15 ug/ml for Fe, and 10 ug/ml for the remaining elements. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.36 Graphite Furnace AAS Standards for Arsenic, Cadmium, and Lead (and Antimony, Selenium, and Thallium). Prepare a 10 ug/ml standard by adding 1 ml of 1000 ug/ml standard to a 100-ml volumetric flask. Dilute to 100 ml with 10 percent nitric acid. For graphite furnace AAS, the standards must be matrix matched; e.g., if the samples contain 6 percent nitric acid and 4 percent hydrofluoric acid, the standards should also be made up with 6 percent nitric acid and 4 percent hydrofluoric acid. Prepare a 100 ng/ml standard by adding 1 ml of the 10 ug/ml standard to a 100-ml volumetric flask and dilute to 100 ml with the appropriate matrix solution. Other standards should be prepared by dilution of the 100 ng/ml standards. At least five standards should be used to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Quality control samples should be prepared by making a separate 10 ug/ml standard and diluting until it is in the range of the samples. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.37 Matrix Modifiers.

4.4.37.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of $Ni(NO_3)_2 \cdot 6H_2O$ in approximately 50 ml of water in a 100-ml volumetric flask.

Dilute to 100 ml with water.

4.4.37.2 Nickel Nitrate, One-tenth Percent (V/V). Dilute 10 ml of 1 percent nickel nitrate solution to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for As.

4.4.37.3 Lanthanum. Dissolve 0.5864 g of La_2O_3 in 10 ml of concentrated HNO_3 and dilute to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for Pb.

5. Procedure

5.1 Sampling. The complexity of this method is such that, to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. All sampling train glassware should first be rinsed with hot tap water and then washed in hot soapy water. Next, glassware should be rinsed three times with tap water, followed by three additional rinses with water. All glassware should then be soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, rinsed a final time with acetone, and allowed to air dry. All glassware openings where contamination can occur should be covered until the sampling train is assembled, prior to sampling.

5.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2.

5.1.3 Preparation of Sampling Train. Follow the same general procedures given in Method 5, Section 4.1.3, except place 100 ml of the nitric acid/hydrogen peroxide solution (Section 4.2.1) in the two HNO_3/H_2O_2 impingers (normally the second and third impingers), place 100 ml of the acidic potassium permanganate solution (Section 4.2.2) in the fourth and fifth impinger, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to train assembly.

Several options are available to the tester based on the sampling conditions. The use of an empty first impinger can be eliminated if the moisture to be collected in the impingers is calculated or determined to be less than 150 ml. The tester shall include two impingers containing the acidic potassium permanganate solution for the first test run, unless past testing experience at the same or similar sources has shown that only one is

necessary. The last permanganate impinger may be discarded if both permanganate impingers have retained their original deep purple permanganate color. A maximum of 200 ml in each permanganate impinger (and a maximum of three permanganate impingers) may be used, if necessary, to maintain the desired color in the last permanganate impinger.

Retain for reagent blanks, 100 ml of the nitric acid/hydrogen peroxide solution and 100 ml of the acidic potassium permanganate solution. These solutions should be labeled and treated as described in Section 7. Set up the sampling train as shown in Figure A-1. If necessary to ensure leak-free sampling train connections, Teflon tape should be used instead of silicone grease to prevent contamination.

Precaution: Extreme care should be taken to prevent contamination within the train. Prevent the mercury collection reagent (acidic potassium permanganate) from contacting any glassware of the train which is washed and analyzed for Mn. Prevent hydrogen peroxide from mixing with the acidic potassium permanganate.

5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

5.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

5.2 Sample Recovery. Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period.

The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling. This normally causes a vacuum to form in the filter holder, thus causing the undesired result of drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the

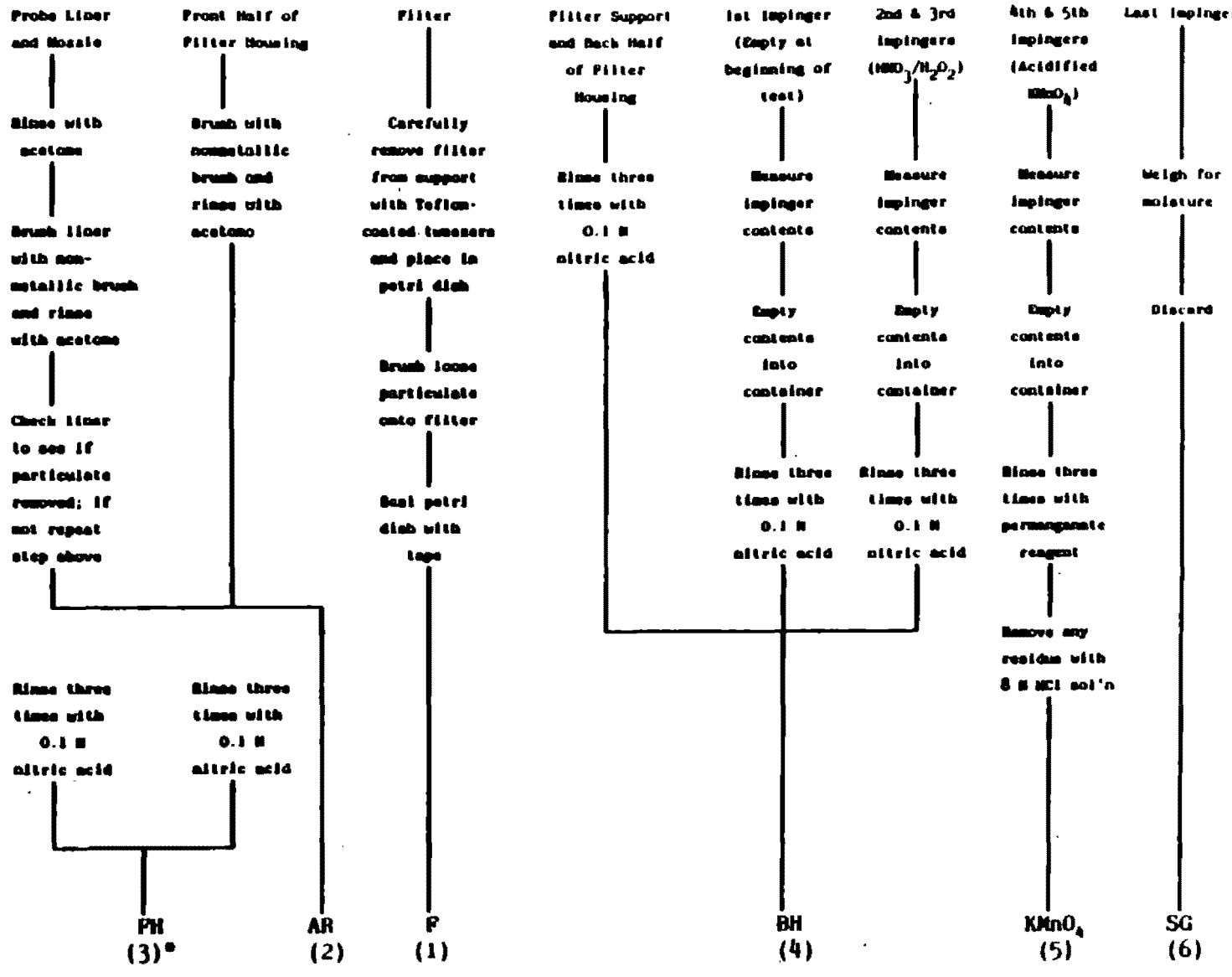
impinger. Cap off the filter holder outlet and impinger inlet. Use non-contaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon tape to close these openings.

Alternatively, the train can be disassembled before the probe and filter holder/oven are completely cooled, if this procedure is followed: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. The sample is recovered and treated as follows (see schematic in Figure A-2). Assure that all items necessary for recovery of the sample do not contaminate it.

5.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. Acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water should be used to handle the filters. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metal-containing materials when recovering this train. Seal the labeled petri dish.

5.2.2 Container No. 2 (Acetone Rinse). Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 100 ml of acetone and placing the wash in a glass container. Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nonmetallic brush. Brush until



* Number in parantheses indicates container number.

Figure A-2. Sample recovery scheme.

the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a nonmetallic probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a samplecontainer underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the front half of the filter holder by rubbing surfaces with a nonmetallic nylon bristle brush and rinsing with aceton Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container clearly to identify its contents.

5.2.3 Container No. 3 (Probe Rinse). Rinse the probe liner, probe nozzle, and front half of the filter holder thoroughly with 100 ml of 0.1 N nitric acid and place the wash into a sample storage container. Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Perform the rinses as described in Method 12, Section 5.2.2. Record the volume of the combined rinse. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during

transport. Seal the container and clearly label the contents. Finally, rinse the nozzle, probe liner, and front half of the filter holder with water followed by acetone and discard these rinses.

5.2.4 Container No. 4 (Impingers 1 through 3, Contents and Rinses). Due to the large quantity of liquid involved, the tester may place the impinger solutions in more than one container. Measure the liquid in the first three impingers volumetrically to within 0.5 ml using a graduated cylinder. Record the volume of liquid present. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N nitric acid as described in Method 12, Section 5.2.4. Note: The use of exactly 100 ml of 0.1 N nitric acid rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the volume. Calculate the 0.1 N nitric acid rinse volume by difference. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. Seal the container and clearly label the contents.

5.2.5 Container No. 5 (Acidified Potassium Permanganate Solution and Rinses, Impingers No. 4 & 5). Pour all the liquid from the permanganate impingers (fourth and fifth, if two permanganate impingers are used) into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Using 100 ml total of the acidified potassium permanganate solution, rinse the permanganate impinger(s) and connecting glass pieces a minimum of three times. Combine the rinses with the permanganate impinger solution. Finally, rinse the permanganate impinger(s) and connecting glassware with 50 ml of 8 N HCl to remove any residue. Note: The use of exactly 100 ml and 50 ml for the two rinses is necessary for the subsequent blank correction procedures. Place the combined rinses and impinger contents in a labeled glass storage bottle. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport. See the following note and the Precaution in Paragraph 4.2.2 and properly seal the bottle and clearly label the contents.

Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles should not be filled full and should be vented to relieve excess

pressure. Venting is highly recommended. A No. 70-72 hole drilled in the container cap and Teflon liner has been found to allow adequate venting without loss of sample.

5.2.6 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

5.2.7 Container No. 7 (Acetone Blank). Once during each field test, place 100 ml of the acetone used in the sample recovery process into a labeled container for use in the front half field reagent blank. Seal the container.

5.2.8 Container No. 8 (0.1 N Nitric Acid Blank). Once during each field test, place 200 ml of the 0.1 N nitric acid solution used in the sample recovery process into a labeled container for use in the front half and back half field reagent blanks. Seal the container.

5.2.9 Container No. 9 (5% Nitric Acid/10% Hydrogen Peroxide Blank). Once during each field test, place 200 ml of the 5% nitric acid/10% hydrogen peroxide solution used as the nitric acid impinger reagent into a labeled container for use in the back half field reagent blank. Seal the container.

5.2.10 Container No. 10 (Acidified Potassium Permanganate Blank). Once during each field test, place 300 ml of the acidified potassium permanganate solution used as the impinger solution and in the sample recovery process into a labeled container for use in the back half field reagent blank for mercury analysis. Seal the container.

Note: This container should be vented, as described in Section 5.2.4, to relieve excess pressure.

5.2.11 Container No. 11 (8 N HCl Blank). Once during each field test, place 50 ml of the 8 N hydrochloric acid used to rinse the acidified potassium permanganate impingers into a labeled container for use in the back half reagent blank for mercury.

5.2.12 Container No. 12 (Filter Blank). Once during each field test,

place an unused filter from the same lot as the sampling filters in a labeled petri dish. Seal the petri dish. This will be used in the front half field reagent blank.

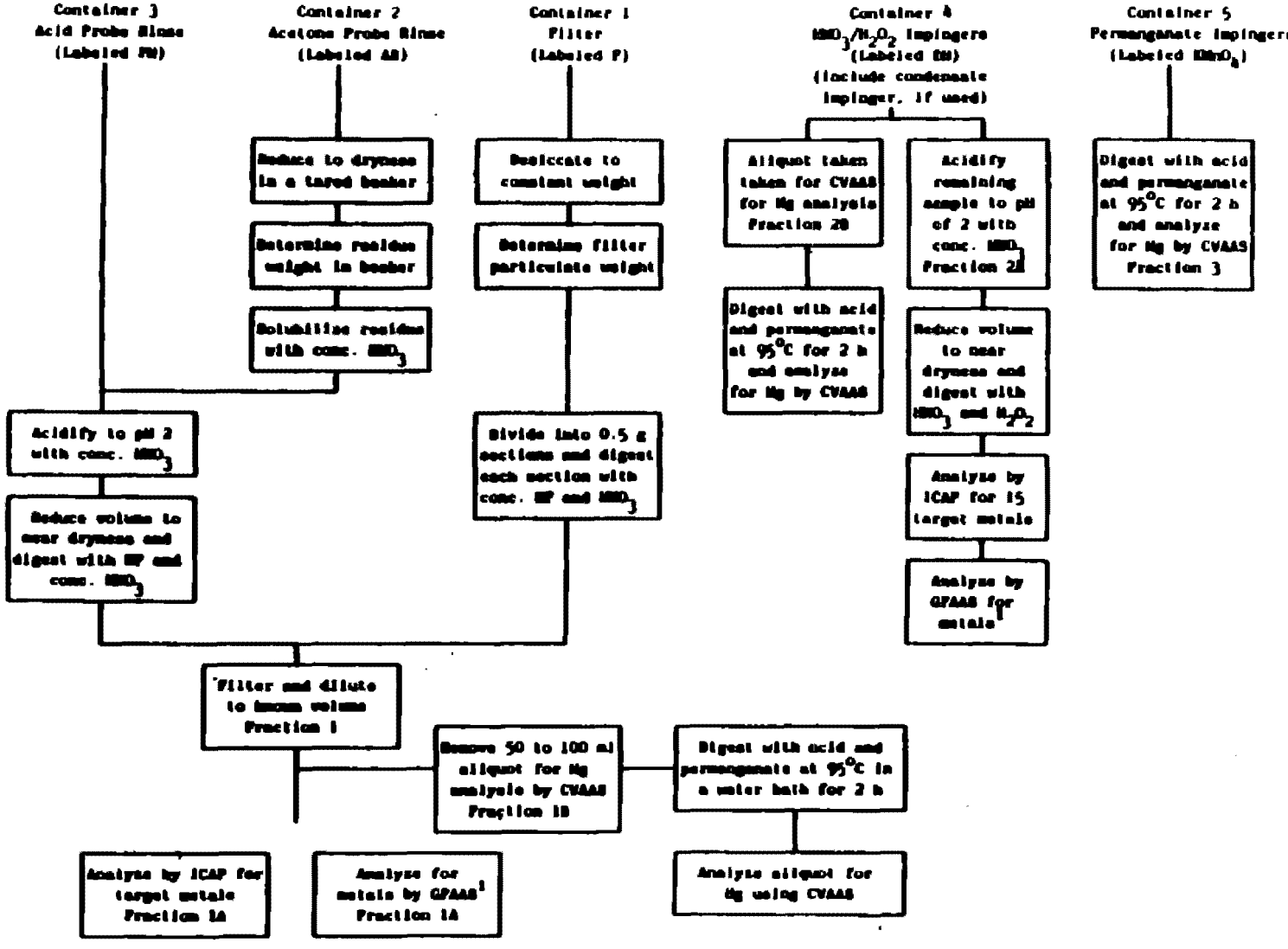
5.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure A-3.

5.3.1 Container No. 1 (Filter). If particulate emissions are being determined, then desiccate the filter and filter catch without heat and weigh to a constant weight as described in Section 4.3 of Method 5. For analysis of metals, divide the filter with its filter catch into portions containing approximately 0.5 g each and place into the analyst's choice of either individual microwave pressure relief vessels or Parr® Bombs. Add 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid to each vessel. For microwave heating, microwave the sample vessels for approximately 12-15 minutes in intervals of 1 to 2 minutes at 600 Watts. For conventional heating, heat the Parr Bombs at 140°C (285°F) for 6 hours. Then cool the samples to room temperature and combine with the acid digested probe rinse as required in Section 5.3.3, below.

Notes: 1. Suggested microwave heating times are approximate and are dependent upon the number of samples being digested. Twelve to 15 minute heating times have been found to be acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by sorbent reflux within the vessel.

2. If the sampling train uses an optional cyclone, the cyclone catch should be prepared and digested using the same procedures described for the filters and combined with the digested filter samples.

5.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to an acid-cleaned tared 250-ml beaker and evaporate to dryness at ambient Figure



[1]Analysis by AAS for metals found at less than 2 ug/ml in digestate solution, if desired. Or analyse for each metal by AAS, if desired.

Figure A-3. Sample preparation and analysis scheme.

temperature and pressure. (If particulate emissions are being determined, desiccate for 24 hours without heat, weigh to a constant weight according to the procedures described in Section 4.3 of Method 5, and report the results to the nearest 0.1 mg.) Resolubilize the residue with concentrated nitric acid and combine the resultant sample including all liquid and any particulate matter with Container No. 3 prior to beginning the following Section 5.3.3.

5.3.3 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. If the pH is higher, the sample should be acidified with concentrated nitric acid to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 50 ml by heating on a hot plate at a temperature just below boiling. Inspect the sample for visible particulate matter, and depending on the results of the inspection, perform one of the following. If no particulate matter is observed, combine the sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. If particulate matter is observed, digest the sample in microwave vessels or Parr® Bombs following the procedures described in Section 5.3.1; then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. The resultant combined sample is referred to as Fraction 1. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. Measure and record the combined volume of the Fraction 1 solution to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as Fraction 1B. Label the remaining 250-ml portion as Fraction 1A. Fraction 1A is used for ICAP or AAS analysis. Fraction 1B is used for the determination of front half mercury.

5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample (Fraction 2) to within 0.5 ml. Remove a 50-ml aliquot for mercury analysis and label as Fraction 2B. Label the remaining portion of Container No. 4 as Fraction 2A. The Fraction 2B aliquot should be prepared and analyzed as described in Section 5.4.3. Fraction 2A shall be pH 2 or lower. If necessary, use concentrated nitric acid to lower Fraction 2A to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Then follow either of the digestion procedures described in Sections

5.3.4.1 and 5.3.4.2, below.

5.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent nitric acid and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent hydrogen peroxide and heat for 10 more minutes. Add 50 ml of hot water and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water.

5.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent nitric acid and heat for 6 minutes in intervals of 1 to 2 minutes at 600 Watts. Allow the sample to cool. Add 10 ml of 3 percent hydrogen peroxide and heat for 2 more minutes. Add 50 ml of hot water and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water.

Note: All microwave heating times given are approximate and are dependant upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.

5.3.5 Container No. 5 (Impingers 4 & 5). Measure and record the total volume of this sample to within 0.5 ml. This sample is referred to as Fraction 3. Follow the analysis procedures described in Section 5.4.3.

5.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

5.4 Sample Analysis. For each sampling train, five individual samples are generated for analysis. A schematic identifying each sample and the prescribed sample preparation and analysis scheme is shown in Figure A-3. The first two samples, labeled Fractions 1A and 1B, consist of the digested samples from the front half of the train. Fraction 1A is for ICAP or AAS analysis as described in Sections 5.4.1 and/or 5.4.2. Fraction 1B is for determination of front half mercury as described in Section 5.4.3.

The back half of the train was used to prepare the third through fifth samples. The third and fourth samples, labeled Fractions 2A and 2B, contain the digested samples from the H_2O and HNO_3/H_2O_2 Impingers 1 through 3. Fraction 2A is for ICAP or AAS analysis. Fraction 2B will be analyzed for mercury.

The fifth sample, labeled Fraction 3, consists of the impinger contents and

rinses from the permanganate Impingers 4 and 5. This sample is analyzed for mercury as described in Section 5.4.3. The total back half mercury catch is determined from the sum of Fraction 2B and Fraction 3.

5.4.1 ICAP Analysis. Fraction 1A and Fraction 2A are analyzed by ICAP using EPA Method 200.7 (40 CFR 136, Appendix C). Calibrate the ICAP, and set up an analysis program as described in Method 200.7. The quality control procedures described in Section 7.3.1 of this method shall be followed. Recommended wavelengths for use in the analysis of the primary, secondary, and interferring metals are listed below.

Element	Wavelength (nm)
Arsenic	193.696
Beryllium	313.042
Cadmium	226.502
Chromium	267.716
Lead	220.353
Nickel	231.604
Zinc	213.856
Antimony	206.833
Barium	455.403
Copper	324.754
Manganese	257.610
Selenium	196.026
Silver	328.068
Thallium	190.864
Aluminum	308.215
Iron	259.940

The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

Initially, analyze all samples for the target metals plus iron and aluminum. If iron and aluminum are present in the sample, the sample may have to be diluted so that each of these elements is at a concentration of less than 50 ppm to reduce their spectral interferences on arsenic and lead.

Note: When analyzing samples in a hydrofluoric acid matrix, an alumina torch should be used; since all front half samples will contain hydrofluoric acid, use an alumina torch.

5.4.2 AAS by Direct Aspiration and/or Graphite Furnace. If analysis of metals in Fraction 1A and Fraction 2A using graphite furnace or direct aspiration AAS is desired, Table A-2 should be used to determine which techniques and methods should be applied for each target metal. Table A-2 should also be consulted to determine possible interferences and techniques to be followed for their minimization. Calibrate the instrument according to Section 6.3 and follow the quality control procedures specified in Section 7.3.2.

5.4.3 Cold Vapor AAS Mercury Analysis. Fraction 1B, Fraction 2B, and Fraction 3 should be analyzed for mercury using cold vapor atomic absorption spectroscopy following the method outlined in EPA Method 7470 or in Standard Methods for Water and Wastewater Analysis, 15th Edition, Method 303F. Set up the calibration curve as described in Section 7.3 of Method 303F. Add approximately 5 ml of each sample to BOD bottles. Record the amount of sample added. The amount used is dependent upon the expected levels of mercury. Dilute to approximately 120 ml with mercury-free water. Add approximately 15 ml of 5 percent potassium permanganate solution to the Fraction 2B and Fraction 3 samples. Add 5 percent potassium permanganate solution to the Fraction 1B sample as needed to produce a purple solution lasting at least 15 minutes. A minimum of 25 ml is suggested. Add 5 ml of 50 percent nitric acid, 5 ml of concentrated sulfuric acid, and 9 ml of 5 percent potassium persulfate to each sample and each standard. Digest the solution in the capped BOD bottle at 95°C (205°F) in a convection oven or water bath for 2 hours. Cool. Add 5 ml of hydroxylamine hydrochloride solution and mix the sample. Then add 7 ml of stannous chloride to each sample and analyze immediately.

6. Calibration

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in Section 4.4. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures using the

TABLE A-2. APPLICABLE TECHNIQUES, METHODS, AND MINIMIZATION OF INTEFERENCE FOR AAS ANALYSIS

Metal	Technique	Method No.	Wavelength (nm)	Interference	
				Cause	Minimization
Sb	Aspiration	7040	217.6	1000 mg/ml Pb Ni, Cu, or acid	Use secondary wavelenght of 231.1 nm. Match sample & standards acid concentration or use nitrous oxide/acetylene flame
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction
As	Furnace	7060	193.7	Arsenic volati - zation Aluminium	Spiked samples & add nickel nitrate solution to digestates prior to analyses Use Zeeman background correction
Ba	Aspiration	7080	553.6	Calcium Barium ionization	High hollow cathode current & narrow band set 2 mL of KCl per 100 mL of sample
Be	Aspiration	7090	234.9	500 ppm Al High Mg & Si	Add 0.1% fluoride Use method of standard additions
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects
Cd	Aspiration	7130	228.8	Absorption & light scattering	Background correction is required
Cd	Furnace	7131	228.8	As above Excess chloride Pipet tips	As above Ammonium phosphate used as a matrix modifier Use cadmium-free tips
Cr	Aspiration	7190	357.9	Alkali metal Absorption & scatt	KCl ionization suppressant in sample & stand Consult manufacturer's literature
Cr	Furnace	7191	357.9	200 mg/L calcium & phosphate	All calcium nitrate for a know constant effect and to eliminate effect of phosphate

(continued)

TABLE A-2 (CONTINUED)

Metal	Technique	Method No.	Wavelength (nm)	Interference	
				Cause	Minimization
Cu	Aspiration	7210	324.7	Absorpt & scatter	Consult manufacturer's manual
Fe	Aspiration	7380	248.3	Contamination	Great care taken to aviod contamination
Pb	Aspiration	7420	283.3	217.0 nm alternat	Background correction required
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 uL of phosphorus acid to 1-mL of prepared sample in sampler cup
Mn	Aspiration	7460	279.5	403.1 nm alternat	Background correction required
Ni	Aspiration	7520	232.0	352.4 nm alternat	Background correction required
				Fe, Co, & Cr	Matrix matching or a nitrous-oxide/acety flame
				Nonlinear respons	Sample dilution or use 352.4 nm line
Se	Furnace	7740	196.0	Volitality	Spike samples & reference materials & add nickel nitrate to minimize volatilization
				Adsorpt & scatter	Background correction is required & Zeeman background correction can be useful
Ag	Aspiration	7760	328.1	Absorpt & scatter	Background correction is required
				AgCl insoluble	Avoid hydrochloric acid unless silver is in solution as a chloride complex
				Viscosity	Sample & standards monitored for apiration rate
Tl	Aspiration	7840	276.8		Background correction is required Hydrochloric acid should not be used
Tl	Furnace	7841	276.8		Background correction is required
				Hydrochloric acid or chloride	Verify that losses are not occuring for volitization by spiked samples or standad addt Palladium is a suitable matrix modifier
Zn	Aspiration	7950	213.9	High Si, Cu & P	Strontium removes Cu and phosphate
				Contamination	Care should be taken to avid contamination

above standards. The instrument calibration should be checked once per hour. If the instrument does not reproduce the concentrations of the standard within 10 percent, the complete calibration procedures should be performed.

6.3 Atomic Absorption Spectrometer - Direct Aspiration, Graphite Furnace and Cold Vapor Mercury Analyses. Prepare the standards as outlined in Section 4.4. Calibrate the spectrometer using these prepared standards. Calibration procedures are also outlined in the EPA methods referred to in Table A-2 and in Standard Methods for Water and Wastewater, 15th Edition, Method 303F (for mercury). Each standard curve should be run in duplicate and the mean values used to calculate the calibration line. The instrument should be recalibrated approximately once every 10 to 12 samples.

7. Quality Control

7.1 Sampling. Field Reagent Blanks. The blank samples in Container Numbers 7 through 12 produced previously in Sections 5.2.7 through 5.2.11, respectively, shall be processed, digested, and analyzed as follows. Digest and process Container No. 12 contents per Section 5.3.1, Container No. 7 per Section 5.3.2, and half of Container No. 8 per Section 5.3.3. This produces Fraction Blank 1A and Fraction Blank 1B from Fraction Blank 1. Combine the remaining half of Container No. 8 with the contents of Container No. 9 and digest and process the resultant volume per Section 5.3.4. This produces Fraction Blank 2A and Fraction Blank 2B from Fraction Blank 2. Container No. 1(and Container No. 11 contents are Fraction Blank 3. Analyze Fraction Blank 1A and Fraction Blank 2A per Section 5.4.1 and/or 5.4.2. Analyze Fraction Blank 1B, Fraction Blank 2B, and Fraction Blank 3 per Section 5.4.3. The analysis of Fraction Blank 1A produces the front half reagent blank correction values for the metals except mercury; the analysis of Fraction Blank 1B produces the front half reagent blank correct value for mercury. The analysis of Fraction Blank 2A produces the back half reagent blank correction values for the metals except mercury, while separate analysis of Fraction Blanks 2B and 3 produce the back half reagent blank correction value for mercury.

7.2 An attempt may be made to determine if the laboratory reagents used in Section 5.3 caused contamination. They should be analyzed by the procedures in Section 5.4. The Administrator will determine whether or not the laboratory blank reagent values can be used in the calculation of the stationary source test results.

7.3 Quality Control Samples. The following quality control samples should be analyzed.

7.3.1 ICAP Analysis. Follow the quality control shown in Section 8 of Method 6010. For the purposes of a three run test series, these requirements have been modified to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (must be within 25% or analyze by standard addition), one quality control sample to check the accuracy of the calibration standards (must be within 25% of calibration), and one duplicate analysis (must be within 5% of average or repeat all analysis).

7.3.2 Direct Aspiration and/or Graphite Furnace AAS Analysis for Arsenic, Beryllium, Cadmium, Chromium, Lead, Mercury, Nickel, and Zinc (and Antimony, Barium, Copper, Manganese, Phosphorus, Selenium, Silver, and Thallium, if measured). All samples should be analyzed in duplicate. Perform a matrix spike on one front half sample and one back half sample or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the method of additions. A quality control sample should be analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration repeated.

7.3.3 Cold Vapor AAS Analysis for Mercury. All samples should be analyzed in duplicate. A quality control sample should be analyzed to check the accuracy of the calibration standards (within 10% or repeat calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). Additional information on quality control can be obtained from EPA Method 7470 or in Standard Methods for Water and Wastewater, 15th Edition, Method 303F.

8. Calculations

8.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

8.2 Volume of Water Vapor and Moisture Content. Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

8.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

8.4 Metals (Except Mercury) in Source Sample.

8.4.1 Fraction 1A, Front Half, Metals (except Hg). Calculate the amount of each metal collected in Fraction 1 of the sampling train using the following equation:

$$M_{fh} = C_a F_d V_{soln.1} \qquad \text{Eq. 1*}$$

where:

M_{fh} = total mass of each metal (except Hg) collected in the front half of the sampling train (Fraction 1), ug.
C_a = concentration of metal in sample Fraction 1A as read from the standard curve (ug/ml).
F_d = dilution factor (F_d = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument t produce the reading C_a. For example, when the dilution of Fraction 1 is from 2 to 10 ml, F_d = 5).
$V_{soln.1}$ = total volume of digested sample solution (Fraction 1), ml.

8.4.2 Fraction 2A, Back Half, Metals (except Hg). Calculate the amount of each metal collected in Fraction 2 of the sampling train using the following equation.

$$M_{bh} = C_a F_a V_a \qquad \text{Eq. 2*}$$

where:

M_{bh} = total mass of each metal (except Hg) collected in the back half of the sampling train (Fraction 2), ug.
C_a = concentration of metal in sample Fraction 2A, as read from the standard curve (ug/ml).
F_a = aliquot factor, volume of Fraction 2 divided by volume of aliquot Fraction 2A.
V_a = volume of digested sample analyzed (concentrated Fraction 2A), ml.

8.4.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{fh} - M_{fhb}) + (M_{bh} - M_{bhb}) \qquad \text{Eq. 3*}$$

*If Fractions 1A and 2A are combined, proportional aliquots must be used. Appropriate changes must be made in Equations 1-3 to reflect this approach.

where:

M_t = total mass of each metal (separately stated for each metal) collected in the sampling train, ug.
M_{fhb} = blank correction value for mass of metal detected in front half field reagent blank, ug.
M_{bhb} = blank correction value for mass of metal detected in back half field reagent blank, ug.

Note: If the measured blank value for the front half (m_{fhb}) is in the range 0.0 to A ug [where A ug equals the value determined by multiplying 1.4 ug per square inch (1.4 ug/in.^{2}) times the actual area in square inches (in.^{2}) of the filter used in the emission sample], m_{fhb} may be used to correct the emission sample value (m_{fh}); if m_{fhb} exceeds A ug, the greater of the two following values (either I. or II.) may be used:

I. A ug, or

II. the lesser of (a) m_{fhb}, or (b) 5 percent of m_{fh}.

If the measured blank value for the back half (m_{bhb}) is in the range 0.0 to 1 ug, m_{bhb} may be used to correct the emission sample value (m_{bh}); if m_{bhb} exceeds 1 ug, the greater of the two following values may be used: 1 ug or 5 percent of m_{bh}.

8.5 Mercury in Source Sample.

8.5.1 Fraction 1B, Front Half, Hg. Calculate the amount of mercury collected in the front half, Fraction 1, of the sampling train using the following equation:

$$Hg_{fh} = \frac{Q_{fh}}{V_{f1s}} \times V_{soln,t} \qquad \text{Eq. 4}$$

where:

Hg_{fh} = total mass of mercury collected in the front half of the sampling train (Fraction 1), ug.
Q_{fh} = quantity of mercury in analyzed sample, ug.
$V_{soln,t}$ = total volume of digested sample solution (Fraction 1), ml.
V_{f1s} = volume of Fraction 1B analyzed, ml. See the following Note.

Note: V_{f1s} is the actual amount of Fraction 1B analyzed. For example, if 1 ml of Fraction 1B were diluted to 100 ml to bring it into the proper analytical range, and 1 ml of the 100-ml dilution was analyzed, V_{f1s} would be 0.01.

8.5.2 Fraction 2B and Fraction 3, Back Half, Hg. Calculate the amount of mercury collected in Fractions 2 and 3 using Equations 5 and 6, respectively.

Calculate the total amount of mercury collected in the back half of the sampling train using Equation 7.

$$Hg_{bh2} = \frac{Q_{bh2}}{V_{f2s}} \times V_{soln,2} \qquad \text{Eq. 5}$$

where:

Hg_{bh2} = total mass of mercury collected in Fraction 2, ug.
Q_{bh2} = quantity of mercury in analyzed sample, ug.
V_{f2s} = volume of Fraction 2B analyzed, ml (see Note in Section 8.5.1).
$V_{soln,2}$ = total volume of Fraction 2, ml.

$$Hg_{bh3} = \frac{Q_{bh3}}{V_{f3s}} \times V_{soln,3} \qquad \text{Eq. 6}$$

where:

Hg_{bh3} = total mass of mercury collected in Fraction 3, ug.
Q_{bh3} = quantity of mercury in analyzed sample, ug.
V_{f3} = volume of Fraction 3 analyzed, ml (see Note in Section 8.5.1).
$V_{soln,3}$ = total volume of Fraction 3, ml.

$$Hg_{bh} = Hg_{bh2} + Hg_{bh3} \qquad \text{Eq. 7}$$

where:

Hg_{bh} = total mass of mercury collected in the back half of the sampling train, ug.

8.5.3 Total Train Mercury Catch. Calculate the total amount of mercury collected in the sampling train using Equation 8.

$$M_t = (Hg_{fh} - Hg_{fhb}) + (Hg_{bh} - Hg_{bhb}) \qquad \text{Eq. 8}$$

where:

M_t = total mass of mercury collected in the sampling train, ug.
Hg_{fhb} = blank correction value for mass of mercury detected in front half field reagent blank, ug.

Hg_{bhb} = blank correction value for mass of mercury detected in back half field reagent blank, ug.

Note: If the total of the measured blank values (Hg_{fhb} + Hg_{bhb}) is in the rang of 0 to 3 ug, then the total may be used to correct the emission sample value (Hg_{fh} + Hg_{bh}); if it exceeds 3 ug, the greater of the following two values may be used: 3 ug or 5 percent of the emission sample value (Hg_{fh} + Hg_{bh}).

8.6 Metal Concentration of Stack Gas. Calculate the concentrations of arsenic, beryllium, cadmium, total chromium, lead, mercury, nickel, and zinc (and antimony, barium, copper, manganese, phosphorus, selenium, silver, and thallium, if measured) in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_s = K_4 \, (M_t / V_{m(std)}) \qquad \text{Eq.9}$$

where:

C_s = concentration of each metal in the stack gas, ug/dscm.
K_4 = 10^{-3} ug/ug.
M_t = total mass of each metal collected in the sampling train, ug.
$V_{m(std)}$ = volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

8.7 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

9. Bibliography

9.1 Method 303F in Standard Methods for the Examination of Water Wastewater, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street N.W., Washington, D.C. 20036.

9.2 EPA Methods 6010, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response. U. S. Environmental Protection Agency, Washington, D.C. 20460.

9.3 EPA Method 200.7, Code of Federal Regulations, Title 40, Part 136, Appendix C. July 1, 1987.

9.4 EPA Methods 1 through 5, Code of Federal Regulations, Title 40, Part 60, Appendix A, July 1, 1987.

APPENDIX J.2

PM_{10}/CPM

them to be erroneously measured as PM_{10}.

Drying and shrinking is not thought to be a problem. Should it be considered a problem, the tester could choose Method 201A in which there is no recycle gas.

Another commenter said that the use of recycle gas increases velocity in the cyclone which could cause friable particles to break up, becoming PM_{10}.

Prior to size classification by a PM_{10} cyclone, there is no known or suspected mechanism by which friable particles, should they exist, may break up. When particles greater than 10 μm aerodynamic size reach the cyclone wall due to their inertia, they are collected.

One commenter said that no consideration is made in either method of the gas density, gas viscosity, or of the density of the particulate matter being measured.

Gas density and viscosity are compensated for in the calculations for both PM_{10} methods. Because the aerodynamic diameter of PM_{10} emissions is used in both PM_{10} methods, determination of particle densities, volumes, or shapes is not necessary and would be redundant.

Another commenter said if the PM_{10} measurement is made downstream of an electrostatic precipitater (ESP), then the particles will carry an electric charge and the measurement of PM_{10} by these methods will be affected.

The effect of an ESP on particle sizing when using Method 201 or 201A is considered to be negligible.

There was concern by the commenters that the particulate matter may settle out inside the sample train.

The trains have been calibrated with test aerosols and the relative accuracies to each other have been established. These tests results and the operation principles of both methods have shown that "settling" is not a problem. The particles travel only 1.5 to 3 in. (nozzle length) prior to size classification by the trains.

IV. Administrative

The docket is an organized and complete file of all the information considered by EPA in the development of this rulemaking. The docket is a dynamic file since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to identify readily and locate documents so that they can effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated test method revisions and EPA responses to significant comments, the contents of the docket, except for interagency review materials, will serve as the record in case of judicial review (section 307(d)(7)(A)).

Under Executive Order 12291, EPA is required to judge whether a regulation is a "major rule" and, therefore, subject to the requirements of a regulatory impact analysis. The Agency has determined that this regulation would result in none of the adverse economic effects set forth in section 1 of the Order as grounds for finding a regulation to be a "major rule." The Agency has, therefore, concluded that this regulation is not a "major rule" under Executive Order 12291.

The Regulatory Flexibility Act (RFA) of 1980 requires the identification of potentially adverse impacts of Federal regulations upon small business entities. The Act specifically requires the completion of a RFA analysis in those instances where small business impacts are possible. Because this rulemaking imposes no adverse economic impacts, an analysis has not been conducted.

List of Subjects in 40 CFR Part 51

Administrative practice and procedure, Air pollution control, Carbon monoxide, Intergovernmental relations, Lead, Nitrogen dioxide, Ozone, Particulate matter, Reporting and recordkeeping requirements, Sulfur oxides, Volatile organic compounds.

Dated: March 22, 1990.

William K. Reilly,

Administrator.

The EPA amends title 40, chapter I, part 51 of the Code of Federal Regulations as follows:

PART 51—[AMENDED]

1. The authority citation for part 51 is revised to read as follows:

Authority: 42 U.S.C. 7401(b)(1), 7410, 7470–7479, 7501–7508, and 7601(a), unless otherwise noted.

2. Subpart K, § 51.212 is amended by adding paragraph (c) to read as follows:

§ 51.212 Testing, inspection, enforcement, and complaints.

* * * * *

(c) Enforceable test methods for each emission limit specified in the plan. As an enforceable method, States may use:

(1) Any of the appropriate methods in appendix M to this part, Recommended Test Methods for State Implementation Plans; or

(2) An alternative method following review and approval of that method by the Administrator; or

(3) Any appropriate method in appendix A to 40 CFR part 60.

3. Appendix M is added to part 51 to read as follows:

Appendix M—Recommended Test Methods for State Implementation Plans

Method 201—Determination of PM_{10} Emissions (Exhaust Gas Recycle Procedure).

Method 201A—Determination of PM_{10} Emissions (Constant Sampling Rate Procedure).

Presented herein are recommended test methods for measuring air pollutants emanating from an emission source. They are provided for States to use in their plans to meet the requirements of Subpart K—Source Surveillance.

The State may also choose to adopt other methods to meet the requirements of Subpart K of this part, subject to the normal plan review process.

The State may also meet the requirements of Subpart K of this part by adopting, again subject to the normal plan review process, any of the relevant methods in appendix A to 40 CFR part 60.

Method 201—Determination of PM_{10} Emissions

(Exhaust Gas Recycle Procedure)

1. Applicability and Principle

1.1 Applicability. This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 μm (PM_{10}) from stationary sources. The EPA recognizes that condensible emissions not collected by an in-stack method are also PM_{10}, and that emissions that contribute to ambient PM_{10} levels are the sum of condensible emissions and emissions measured by an in-stack PM_{10} method such as this method or Method 201A. Therefore, for establishing source contributions to ambient levels of PM_{10}, such as for emission inventory purposes, EPA suggests that source PM_{10} measurement include both in-stack PM_{10} and condensible emissions. Condensible missions may be measured by an impinger analysis in combination with this method.

1.2 Principle. A gas sample is isokinetically extracted from the source. An in-stack cyclone is used to separate PM greater than PM_{10}, and an in-stack glass fiber filter is used to collect the PM_{10}. To maintain isokinetic flow rate conditions at the tip of the probe and a constant flow rate through the cyclone, a clean, dried portion of the sample gas at stack temperature is recycled into the nozzle. The particulate mass is determined gravimetrically after removal of uncombined water.

2. Apparatus

Note: Method 5 as cited in this method refers to the method in 40 CFR part 60, appendix A.

2.1 Sampling Train. A schematic of the exhaust of the exhaust gas recycle (ECR) train is shown in Figure 1 of this method.

2.1.1 Nozzle with Recycle Attachment. Stainless steel (316 or equivalent) with a sharp tapered leading edge, and recycle attachment welded directly on the side of the nozzle (see schematic in Figure 2 of this method). The angle of the taper shall be on the outside. Use only straight sampling nozzles. "Gooseneck" or other nozzle

extensions designed to turn the sample gas flow 90°, as in Method 5 are not acceptable. Locate a thermocouple in the recycle attachment to measure the temperature of the recycle gas as shown in Figure 3 of this method. The recycle attachment shall be made of stainless steel and shall be connected to the probe and nozzle with stainless steel fittings. Two nozzle sizes, e.g., 0.125 and 0.160 in., should be available to allow isokinetic sampling to be conducted over a range of flow rates. Calibrate each nozzle as described in Method 5, Section 5.1.

2.1.2 PM_{10} Sizer. Cyclone, meeting the specifications in Section 5.7 of this method.

2.1.3 Filter Holder. 63mm, stainless steel. An Andersen filter, part number SE274, has been found to be acceptable for the in-stack filter.

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

2.1.4 Pitot Tube. Same as in Method 5, Section 2.1.3. Attach the pitot to the pitot lines with stainless steel fittings and to the cyclone in a configuration similar to that shown in Figure 3 of this method. The pitot lines shall be made of heat resistant material and attached to the probe with stainless steel fittings.

2.1.5 EGR Probe. Stainless steel, 15.9-mm (⅝-in.) ID tubing with a probe liner, stainless steel 9.53-mm (⅜-in.) ID stainless steel recycle tubing, two 6.35-mm (¼-in.) ID stainless steel tubing for the pitot tube extensions, three thermocouple leads, and one power lead, all contained by stainless steel tubing with a diameter of approximately 51 mm (2.0 in.). Design considerations should include minimum weight construction materials sufficient for probe structural strength. Wrap the sample and recycle tubes with a heating tape to heat the sample and recycle gases to stack temperature.

2.1.6 Condenser. Same as in Method 5, Section 2.1.7.

2.1.7 Umbilical Connector. Flexible tubing with thermocouple and power leads of sufficient length to connect probe to meter and flow control console.

2.1.8 Vacuum Pump. Leak-tight, oil-less, noncontaminating, with an absolute filter, "HEPA" type, at the pump exit. A Gast Model 0522-V103 C16DX pump has been found to be satisfactory.

2.1.9 Meter and Flow Control Console. System consisting of a dry gas meter and calibrated orifice for measuring sample flow rate and capable of measuring volume to ±2 percent, calibrated laminar flow elements (LFE's) or equivalent for measuring total and sample flow rates, probe heater control, and manometers and magnehelic gauges (as shown in Figures 4 and 5 of this method), or equivalent. Temperatures needed for calculations include stack, recycle, probe, dry gas meter, filter, and total flow. Flow measurements include velocity head (Δp), orifice differential pressure (ΔH), total flow, recycle flow, and total back-pressure through the system.

2.1.10 Barometer. Same as in Method 5, Section 2.1.9.

2.1.11 Rubber Tubing, 6.35-mm (¼-in.) ID flexible rubber tubing.

2.2 Sample Recovery.

2.2.1 Nozzle, Cyclone, and Filter Holder Brushes. Nylon bristle brushes properly sized and shaped for cleaning the nozzle, cyclone, filter holder, and probe or probe liner, with stainless steel wire shafts and handles.

2.2.2 Wash Bottles, Glass Sample Storage Containers, Petri Dishes, Graduated Cylinder and Balance, Plastic Storage Containers, and Funnels. Same as Method 5, Sections 2.2.2 through 2.2.8 and 2.2.8, respectively.

2.3 Analysis. Same as in Method 5, Section 2.3.

3. Reagents

The reagents used in sampling, sample recovery, and analysis are the same as that specified in Method 5, Sections 3.1, 3.2, and 3.3, respectively.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. Same as in Method 5, Section 4.1.1.

4.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2, except use the directions on nozzle size selection in this section. Use of the EGR method may require a minimum sampling port diameter of 0.2 m (8 in.). Also, the required maximum number of sample traverse points at any location shall be 12.

4.1.2.1 The cyclone and filter holder must be in-stack or at stack temperature during sampling. The blockage effects of the EGR sampling assembly will be minimal if the cross-sectional area of the sampling assembly is 3 percent or less of the cross-sectional area of the duct and a pitot coefficient of 0.84 may be assigned to the pitot. If the cross-sectional area of the assembly is greater than 3 percent of the cross-sectional area of the duct, then either determine the pitot coefficient at sampling conditions or use a standard pitot with a known coefficient in a configuration with the EGR sampling assembly such that flow disturbances are minimized.

4.1.2.2 Construct a setup of pressure drops for various Δp's and temperatures. A computer is useful for these calculations. An example of the output of the EGR setup program is shown in Figure 6 of this method, and directions on its use are in section 4.1.5.2 of this method. Computer programs, written in IBM BASIC computer language, to do these types of setup and reduction calculations for the EGR procedure, are available through the National Technical Information Services (NTIS), Accession number PB90-500000, 5285 Port Royal Road, Springfield, Virginia 22161.

4.1.2.3 The EGR setup program allows the tester to select the nozzle size based on anticipated average stack conditions and prints a setup sheet for field use. The amount of recycle through the nozzle should be between 10 and 80 percent. Inputs for the EGR setup program are stack temperature (minimum, maximum, and average), stack velocity (minimum, maximum, and average), atmospheric pressure, stack static pressure, meter box temperature, stack moisture, percent O_2, and percent CO_2 in the stack gas, pitot coefficient (C_p), orifice $\Delta H_@$, flow rate measurement calibration values (slope (m) and y-intercept (b) of the calibration curve), and the number of nozzles available and their diameters.

4.1.2.4 A less rigorous calculation for the setup sheet can be done manually using the equations on the example worksheets in Figures 7, 8, and 9 of this method, or by a Hewlett-Packard HP41 calculator using the program provided in appendix D of the EGR operators manual, entitled *Applications Guide for Source PM_{10} Exhaust Gas Recycle Sampling System*. This calculation uses an approximation of the total flow rate and agrees within 1 percent of the exact solution for pressure drops at stack temperatures from 38 to 260 °C (100 to 500 °F) and stack moisture up to 50 percent. Also, the example worksheets use a constant stack temperature in the calculation, ingoring the complicated temperature dependence from all three pressure drop equations. Errors for this at stack temperatures ±28 °C (±50 °F) of the temperature used in the setup calculations are within 5 percent for flow rate and within 5 percent for cyclone cut size.

4.1.2.5 The pressure upstream of the LFE's is assumed to be constant at 0.8 in. Hg in the EGR setup calculations.

4.1.2.6 The setup sheet constructed using this procedure shall be similar to Figure 8 of this method. Inputs needed for the calculation are the same as for the setup computer except that stack velocities are not needed.

4.1.3 Preparation of Collection Train. Same as in Method 5, Section 4.1.3, except use the following directions to set up the train.

4.1.3.1 Assemble the EGR sampling device, and attach it to probe as shown in Figure 3 of this method. If stack temperatures exceed 260 °C (500 °F), then assemble the EGR cyclone without the O-ring and reduce the vacuum requirement to 130 mm Hg (5.0 in. Hg) in the leak-check procedure in Section 4.1.4.3.2 of this method.

4.1.3.2 Connect the probe directly to the filter holder and condenser as in Method 5. Connect the condenser and probe to the meter and flow control console with the umbilical connector. Plug in the pump and attach pump lines to the meter and flow control console.

4.1.4 Leak-Check Procedure. The leak-check for the EGR Method consists of two parts: the sample-side and the recycle-side. The sample-side leak-check is required at the beginning of the run with the cyclone attached, and after the run with the cyclone removed. The cyclone is removed before the post-test leak-check to prevent any disturbance of the collected sample prior to analysis. The recycle-side leak-check tests the leak tight integrity of the recycle components and is required prior to the first test run and after each shipment.

4.1.4.1 Pretest Leak-Check. A pretest leak-check of the entire sample-side, including the cyclone and nozzle, is required. Use the leak-check procedure in Section 4.1.4.3 of this method to conduct a pretest leak-check.

4.1.4.2 Leak-Checks During Sample Run. Same as in Method 5, Section 4.1.4.1.

reading shall not exceed 1 mm (0.05 in.). If the differences exceed the limit specified, adjust or replace the pressure gauge. After each field use, check the calibration of the pressure gauges.

5.3.3 Total LFE. Same as the metering system in Method 5, Section 5.3.

5.3.4 Recycle LFE. Same as the metering system in Method 5, Section 5.3, except completely close both the coarse and fine recycle valves.

5.4 Probe Heater. Connect the probe to the meter and flow control console with the umbilical connector. Insert a thermocouple into the probe sample line approximately half the length of the probe sample line. Calibrate the probe heater at 88 °C (150 °F), 121 °C (250 °F), and 177 °C (350 °F). Turn on the power, and set the probe heater to the specified temperature. Allow the heater to equilibrate, and record the thermocouple temperature and the meter and flow control console temperature to the nearest 0.5 °C (1 °F). The two temperatures should agree within 5.5 °C (10 °F). If this agreement is not met, adjust or replace the probe heater controller.

5.5 Temperature Gauges. Connect all thermocouples, and let the meter and flow contol console equilibrate to ambient temperature. All thermocouples shall agree to within 1.1 °C (2.0 °F) with a standard mercury-in-glass thermometer. Replace defective thermocouples.

5.6 Barometer. Calibrate against a standard mercury-in-glass barometer.

5.7 Probe Cyclone and Nozzle Combinations. The probe cyclone and nozzle combinations need not be calibrated if the cyclone meets the design specifications in Figure 12 of this method and the nozzle meets the design specifications in appendix B of the *Application Guide for the Source PM and Exhaust Gas Recycle Sampling System*, EPA/600/3–88–058. This document may be obtained from Roy Huntley at (919) 541–1060. If the nozzles do not meet the design specifications, then test the cyclone and nozzle combination for conformity with the performance specifications (PS's) in Table 1 of this method. The purpose of the PS tests is to determine if the cyclone's sharpness of cut meets minimum performance criteria. If the cyclone does not meet design specifications, then, in addition to the cyclone and nozzle combination conforming to the PS's, calibrate the cyclone and determine the relationship between flow rate, gas viscosity, and gas density. Use the procedures in Section 5.7.5 of this method to conduct PS tests and the procedures in Section 5.8 of this method to calibrate the cyclone. Conduct the PS tests in a wind tunnel described in Section 5.7.1 of this method and using a particle generation system described in Section 5.7.2 of this method. Use five particle sizes and three wind velocities as listed in Table 2 of this method. Perform a minimum of three replicate measurements of collection efficiency for each of the 15 conditions listed, for a minimum of 45 measurements.

5.7.1 Wind Tunnel. Perform calibration and PS tests in a wind tunnel (or equivalent test apparatus) capable of establishing and maintaining the required gas stream velocities within 10 percent.

5.7.2 Particle Generation System. The particle generation system shall be capable of producing solid monodispersed dye particles with the mass median aerodynamic diameters specified in Table 2 of this method. The particle size distribution verification should be performed on an integrated sample obtained during the sampling period of each test. An acceptable alternative is to verify the size distribution of samples obtained before and after each test, with both samples required to meet the diameter and monodispersity requirements for an acceptable test run.

5.7.2.1 Establish the size of the solid dye particles delivered to the test section of the wind tunnel using the operating parameters of the particle generation system, and verify the size during the tests by microscopic examination of samples of the particles collected on a membrane filter. The particle size, as established by the operating parameters of the generation system, shall be within the tolerance specified in Table 2 of this method. The precision of the particle size verification technique shall be at least ±0.5 μm, and the particle size determined by the verification technique shall not differ by more than 10 percent from that established by the operating parameters of the particle generation system.

5.7.2.2 Certify the monodispersity of the particles for each test either by microscopic inspection of collected particles on filters or by other suitable monitoring techniques such as an optical particle counter followed by a multichannel pulse height analyzer. If the proportion of multiplets and satellites in aerosol exceeds 10 percent by mass, the particle generation system is unacceptable for purposes of this test. Multiplets are particles that are agglomerated, and satellites are particles that are smaller than the specified size range.

5.7.3 Schematic Drawings. Schematic drawings of the wind tunnel and blower system and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques shall be furnished with calibration data to the reviewing agency.

5.7.4 Flow Rate Measurement. Determine the cyclone flow rates with a dry gas meter and a stopwatch, or a calibrated orifice system capable of measuring flow rates to within 2 percent.

5.7.5 Performance Specification Procedure. Establish the test particle generator operation and verify the particle size microscopically. If monodispersity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements may be made at this time.

5.7.5.1 The cyclone cut size (D_{50}) is defined as the aerodynamic diameter of a particle having a 50 percent probability of penetration. Determine the required cyclone flow rate at which D_{50} is 10 μm. A suggested procedure is to vary the cyclone flow rate while keeping a constant particle size of 10 μm. Measure the PM collected in the cyclone (m_c), exit tube (m_t), and filter (m_f). Compute the cyclone efficiency (E_c) as follows:

$$E_c = \frac{m_c}{(m_c + m_t + m_f)} \times 100$$

5.7.5.2 Perform three replicates and calculate the average cyclone efficiency as follow:

$$E_{avg} = \frac{(E_1 + E_2 + E_3)}{3}$$

where E_1, E_2, and E_3 are replicate measurements of E_c.

5.7.5.3 Calculate the standard deviation (σ) for the replicate measurements of E_c as follows:

$$\sigma = \left[\frac{(E_1^2 + E_2^2 + E_3^2) - \frac{(E_1 + E_2 + E_3)}{3}}{2} \right]^{1/2}$$

if σ exceeds 0.10, repeat the replicate runs.

5.7.5.4 Using the cyclone flow rate that produces D_{50} for 10 μm, measure the overall efficiency of the cyclone and nozzle, E_o, at the particle sizes and nominal gas velocities in Table 2 of this method using this following procedure.

5.7.5.5 Set the air velocity in the wind tunnel to one of the nominal gas velocities from Table 2 of this method. Establish isokinetic sampling conditions and the correct flow rate through the sampler (cyclone and nozzle) using recycle capacity so that the D_{50} is 10 μm. Sample long enough to obtain ±5 percent precision on the total collected mass as determined by the precision and the sensitivity of the measuring technique. Determine separately the nozzle catch (m_n), cyclone catch (m_c), cyclone exit tube catch (m_t), and collection filter catch (m_f).

5.7.5.6 Calculate the overall efficiency (E_o) as follows:

4.1.4.3 Post-Test Leak-Check. A leak-check is required at the conclusion of each sampling run. Remove the cyclone before the leak-check to prevent the vacuum created by the cooling of the probe from disturbing the collected sample and use the following procedure to conduct a post-test leak-check.

4.1.4.3.1 The sample-side leak-check is performed as follows: After removing the cyclone, seal the probe with a leak-tight stopper. Before starting pump, close the coarse total valve and both recycle valves, and open completely the sample back pressure valve and the fine total valve. After turning the pump on, partially open the coarse total valve slowly to prevent a surge in the manometer. Adjust the vacuum to at least 381 mm Hg (15.0 in. Hg) with the fine total valve. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

Caution: Do not decrease the vacuum with any of the valves. This may cause a rupture of the filter.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

4.1.4.3.2 Leak rates in excess of 0.00057 m^3/min (0.020 ft^3/min) are unacceptable. If the leak rate is too high, void the sampling run.

4.1.4.3.3 To complete the leak-check, slowly remove the stopper from the nozzle until the vacuum is near zero, then immediately turn off the pump. This procedure sequence prevents a pressure surge in the manometer fluid and rupture of the filter.

4.1.4.3.4 The recycle-side leak-check is performed as follows: Close the coarse and fine total valves and sample back pressure valve. Plug the sample inlet at the meter box. Turn on the power and the pump, close the recycle valves, and open the total flow valves. Adjust the total flow fine adjust valve until a vacuum of 25 inches of mercury is achieved. If the desired vacuum is exceeded, either leak-check at this higher vacuum, or end the leak-check and start over. Minimum acceptable leak rates are the same as for the sample-side. If the leak rate is too high, void the sampling run.

4.1.5 EGR Train Operation. Same as in Method 5, Section 4.1.5, except omit references to nomographs and recommendations about changing the filter assembly during a run.

4.1.5.1 Record the data required on a data sheet such as the one shown in Figure 10 of this method. Make periodic checks of the manometer level and zero to ensure correct ΔH and Δp values. An acceptable procedure for checking the zero is to equalize the pressure at both ends of the manometer by pulling off the tubing, allowing the fluid to equilibrate and, if necessary, to re-zero. Maintain the probe temperature to within 11 °C (20 °F) of stack temperature.

4.1.5.2 The procedure for using the example EGR setup sheet is as follows: Obtain a stack velocity reading from the pitot manometer (Δp), and find this value on the ordinate axis of the setup sheet. Find the stack temperature on the abscissa. Where these two values intersect are the differential pressures necessary to achieve isokineticity and 10 μm cut size (interpolation may be necessary).

4.1.5.3 The top three numbers are differential pressures (in. H_2O), and the bottom number is the percent recycle at these flow settings. Adjust the total flow rate valves, coarse and fine, to the sample value (ΔH) on the setup sheet, and the recycle flow rate valves, coarse and fine, to the recycle flow on the setup sheet.

4.1.5.4 For startup of the EGR sample train, the following procedure is recommended. Preheat the cyclone in the stack for 30 minutes. Close both the sample and recycle coarse valves. Open the fine total, fine recycle, and sample back pressure valves halfway. Ensure that the nozzle is properly aligned with the sample stream. After noting the Δp and stack temperature, select the appropriate ΔH and recycle from the EGR setup sheet. Start the pump and timing device simultaneously. Immediately open both the coarse total and the coarse recycle valves slowly to obtain the approximate desired values. Adjust both the fine total and the fine recycle valves to achieve more precisely the desired values. In the EGR flow system, adjustment of either valve will result in a change in both total and recycle flow rates, and a slight iteration between the total and recycle valves may be necessary. Because the sample back pressure valve controls the total flow rate through the system, it may be necessary to adjust this valve in order to obtain the correct flow rate.

Note: Isokinetic sampling and proper operation of the cyclone are not achieved unless the correct ΔH and recycle flow rates are maintained.

4.1.5.5 During the test run, monitor the probe and filter temperatures periodically, and make adjustments as necessary to maintain the desired temperatures. If the sample loading is high, the filter may begin to blind or the cyclone may clog. The filter or the cyclone may be replaced during the sample run. Before changing the filter or cyclone, conduct a leak-check (Section 4.1.4.2 of this method). The total particulate mass shall be the sum of all cyclone and the filter catch during the run. Monitor stack temperature and Δp periodically, and make the necessary adjustments in sampling and recycle flow rates to maintain isokinetic sampling and the proper flow rate through the cyclone. At the end of the run, turn off the pump, close the coarse total valve, and record the final dry gas meter reading. Remove the probe from the stack, and conduct a post-test leak-check as outlined in Section 4.1.4.3 of this method.

4.1.6 Calculation of Percent Isokinetic Rate and Aerodynamic Cut Size. Calculate percent isokinetic rate and the aerodynamic cut size (D_{50}) (see Calculations, Section 6 of this method) to determine whether the test was valid or another test run should be made. If there was difficult in maintaining isokinetic rates or a D_{50} of 10 μm because of source conditions, the Administrator may be consulted for possible variance.

4.2 Sample Recovery. Allow the probe to cool. When the probe can be safely handled, wipe off all external PM adhering to the outside of the nozzle, cyclone, and nozzle attachment, and place a cap over the nozzle to prevent losing or gaining PM. Do not cap the nozzle tip tightly while the sampling train is cooling, as this action would create a vacuum in the filter holder. Disconnect the probe from the umbilical connector, and take the probe to the cleanup site. Sample recovery should be conducted in a dry indoor area or, if outside, in an area protected from wind and free of dust. Cap the ends of the impingers and carry them to the cleanup site. Inspect the components of the train prior to and during disassembly to note any abnormal conditions. Disconnect the pitot from the cyclone. Remove the cyclone from the probe. Recover the sample as follows:

4.2.1 *Container Number 1* (Filter). The recovery shall be the same as that for Container Number 1 in Method 5, Section 4.2.

4.2.2 *Container Number 2* (Cyclone or Large PM Catch). The cyclone must be disassembled and the nozzle removed in order to recover the large PM catch. Quantitatively recover the PM from the interior surfaces of the nozzle and the cyclone, excluding the "turn around" cup and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.3 *Container Number 3* (PM_{10}). Quantitatively recover the PM from all of the surfaces from cyclone exit to the front half of the in-stack filter holder, including the "turn around" cup and the interior of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.4 *Container Number 4* (Silica Gel): Same as that for Container Number 3 in Method 5, Section 4.2.

4.2.5 *Impinger Water*. Same as in Method 5, Section 4.2, under "Impinger Water."

4.3 Analysis. Same as in Method 5, Section 4.3, except handle EGR Container Numbers 1 and 2 like Container Number 1 in Method 5, EGR Container Numbers 3, 4, and 5 like Container Number 3 in Method 5, and EGR Container Number 6 like Container Number 3 in Method 5. Use Figure 11 of this method to record the weights of PM collected.

4.4 Quality Control Procedures. Same as in Method 5, Section 4.4.

5. Calibration

Maintain an accurate laboratory log of all calibrations.

5.1 Probe Nozzle. Same as in Method 5, Section 5.1.

5.2 Pitot Tube. Same as in Method 5, Section 5.2.

5.3 Meter and Flow Control Console.

5.3.1 Dry Gas Meter. Same as in Method 5, Section 5.3.

5.3.2 LFE Gauges. Calibrate the recycle, total, and inlet total LFE gauges with a manometer. Read and record flow rates at 10, 50, and 90 percent of full scale on the total and recycle pressure gauges. Read and record flow rates at 10, 20, and 30 percent of full scale on the inlet total LFE pressure gauge. Record the total and recycle readings to the nearest 0.3 mm (0.01 in.). Record the inlet total LFE readings to the nearest 3 mm (0.1 in.). Make three separate measurements at each setting and calculate the average. The maximum difference between the average pressure reading and the average manometer

The flow rate, at actual cyclone conditions, is calculated as follows:

$$Q_c = \frac{T_c}{K_1 P_c}\left[Q_{actual} + \frac{V_{wfuel}}{\Theta} \right]$$

6.6.1 Determine the water fraction of the mixed gas through the cyclone by using the equation below.

$$B_c = \frac{V_{wfuel}}{Q_{actual}\,\Theta + V_{wfuel}}$$

6.6.2 Calculate the cyclone gas viscosity as follows:

$$\mu_{cyc} = C_1 + C_2 T_c + C_3 T_c^2 + C_4 I_{21} - C_5 B_c$$

6.6.3 Calculate the molecular weight on a wet basis of the cyclone gas as follows:

$M_c = M_d(1 - B_c) + 18.0(B_c)$

6.6.4 If the cyclone meets the design specification in Figure 12 of this method, calculate the actual D_{50} of the cyclone for the run as follows:

$$D_{50} = \beta_1 \left[\frac{T_c}{M_c P_c} \right]^{0.2501} \left[\frac{\mu_{cyc}}{Q_c} \right]^{0.7501}$$

where $\beta_1 = 0.1582$.

6.6.5 If the cyclone does not meet the design specifications in Figure 12 of this method, then use the following equation to calculate D_{50}.

$$D_{50} = (3)(10)^b (7.378 \times 10^{-4})^m \left[\frac{M_c P_c}{T_c} \right] \left[\frac{4 Q_c}{\pi \mu_{cyc}} \right] d^{(1.5-m)}$$

6.8 Aerodynamic Cut Size. Use the following procedure to determine the aerodynamic cut size (D_{50}).

where:

m = Slope of the calibration curve obtained in Section 5.8.2.

b = y-intercept of the calibration curve obtained in Section 5.8.2.

6.7 Acceptable Results. Acceptability of anisokinetic variation is the same as Method 5, Section 6.12.

6.7.1 If 9.0 μm < D_{50} < 11 μm and 90 < I < 110, the results are acceptable. If D_{50} is greater than 11 μm, the Administrator may accept the results. If D_{50} is less than 9.0 μm, reject the results and repeat the test.

7. Bibliography

1. Same as Bibliography in Method 5.

2. McCain, J.D., J.W. Ragland, and A.D. Williamson. Recommended Methodology for the Determination of Particles Size Distributions in Ducted Sources. Final Report. Prepared for the California Air Resources Board by Southern Research Institute. May 1986.

3. Farthing, W.E., S.S. Dawes, A.D. Williamson, J.D. McCain, R.S. Martin, and J.W. Ragland. Development of Sampling Methods for Source PM-10 Emissions. Southern Research Institute for the Environmental Protection Agency. April 1989.

4. *Application Guide for the Source PM_{10} Exhaust Gas Recycle Sampling System.* EPA/600/3-88-058.

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$$E_o = \frac{(m_a + m_c)}{(m_a + m_c + m_i + m_r)} \times 100$$

5.7.5.7 Do three replicates for each combination of gas velocities and particle sizes in Table 2 of this method. Calculate E_o for each particle size following the procedures described in this section for determining efficiency. Calculate the standard deviation (σ) for the replicate measurements. If σ exceeds 0.10, repeat the replicate runs.

5.7.6 Criteria for Acceptance. For each of the three gas stream velocities, plot the average E_o as a function of particle size on Figure 13 of this method. Draw a smooth curve for each velocity through all particle sizes. The curve shall be within the bonded region for all sizes, and the average E_o for a D_{50} for 10 μm shall be 50 ± 0.5 percent.

5.8 Cyclone Calibration Procedure. The purpose of this section is to develop the relationship between flow rate, gas viscosity, gas density, and D_{50}. This procedure only needs to be done on those cyclones that do not meet the design specifications in Figure 12 of this method.

5.8.1 Calculate cyclone flow rate. Determine the flow rates and D_{50}'s for three different particle sizes between 5 μm and 15 μm, one of which shall be 10 μm. All sizes must be within 0.5 μm. For each size, use a different temperature within 80°C (108°F) of the temperature at which the cyclone is to be used and conduct triplicate runs. A suggested procedure is to keep the particle size constant and vary the flow rate. Some of the values obtained in the PS tests in Section 5.7.5 may be used.

5.8.1.1 On log-log graph paper, plot the Reynolds number (Re) on the abscissa, and the square root of the Stokes 50 number $[(STK_{50})^{1/2}]$ on the ordinate for each temperature. Use the following equations:

$$Re = \frac{4\rho K_{cyc}}{d_{cyc}\pi\mu_{cyc}}$$

$$(Stk_{50})^{1/2} = \left[\frac{4\,Q_{cyc}\,(D_{50})^3}{9\pi\,\mu_{cyc}\,(d_{cyc})^3}\right]^{1/2}$$

where:

Q_{cyc} = Cyclone flow rate cm^3/sec.
ρ = Gas density, g/cm^3.
d_{cyc} = Diameter of cyclone inlet, cm.
μ_{cyc} = Viscosity of gas through the cyclone, poise.
D_{50} = Cyclone cut size, cm.

5.8.1.2 Use a linear regression analysis to determine the slope (m), and the y-intercept (b). Use the following formula to determine Q, the cyclone flow rate required for a cut size of 10 μm.

$$Q = \frac{\pi\,\mu_{cyc}}{4}\left[(3000)(K_1)^b\right]^{-(0.5-m)}\left[\frac{T_s}{M_s P_s}\right]^{m/(m-0.5)} d^{(m-0.5)b/(m-0.5)}$$

where:

Q = Cyclone flow rate for a cut size of 10 μm, cm^3/sec.
T_s = Stack gas temperature, °K.
d = Diameter of nozzle, cm.
$K_1 = 4.077 \times 10^{-3}$.

5.8.2 Directions for Using Q. Refer to Section 5 of the EGR operators manual for directions in using this expression for Q in the setup calculations.

6. Calculations

6.1 The EGR data reduction calculations are performed by the EGR reduction computer program, which is written in IBM BASIC computer language and is available through NTIS. Accession number PB90-500000, 5285 Port Royal Road, Springfield, Virginia 22161. Examples of program inputs and outputs are shown in Figure 14 of this method.

6.1.1 Calculations can also be done manually, as specified in Method 5, Sections 6.3 through 6.7, and 6.9 through 6.12, with the addition of the following:

6.1.2 Nomenclature.

B_c = Moisture fraction of mixed cyclone gas, by volume, dimensionless.
C_1 = Viscosity constant, 51.12 micropoise for °K (51.05 micropoise for °R).
C_2 = Viscosity constant, 0.372 micropoise/°K (0.207 micropoise/°R).
C_3 = Viscosity constant, 1.05×10^{-6} micropoise/°K^2 (3.24×10^{-6} micropoise/°R^2).
C_4 = Viscosity constant, 53.147 micropoise/fraction O_2.
C_5 = Viscosity constant, 74.143 micropoise/fraction H_2O.
D_{50} = Diameter of particles having a 50 percent probability of penetration, μm.
f_{o2} = Stack gas fraction O_2, by volume, dry basis.
K_1 = 0.3858 °K/mm Hg (17.64 °R/in. Hg).
M_c = Wet molecular weight of mixed gas through the PM_{10} cyclone, g/g-mole (lb/lb-mole).
M_d = Dry molecular weight of stack gas, g/g-mole (lb/lb-mole).
P_{bar} = Barometer pressure at sampling site, mm Hg (in. Hg).
P_{tot} = Gauge pressure at inlet to total LFE, mm H_2O (in. H_2O).
P_s = Absolute stack pressure, mm Hg (in. Hg).
Q_s = Total cyclone flow rate at wet cyclone conditions, m^3/min (ft^3/min).
Q_{std} = Total cyclone flow rate at standard conditons, dscm/min (dscf/min).
T_m = Average temperature of dry gas meter, °K (°R).
T_s = Average stack gas temperature, °K (°R).
$V_{w(std)}$ = Volume of water vapor in gas sample (standard conditions), scm (scf).
X_T = Total LFE linear calibration constant, m^3/[(min)(mm H_2O)] (ft^3/[(min)(in. H_2O)]).
Y_T = Total LFE linear calibration constant, dscm/min (dscf/min).
ΔP_T = Pressure differential across total LFE, mm H_2O, (in. H_2O).
θ = Total sampling time, min.
μ_{cyc} = Viscosity of mixed cyclone gas, micropoise.
μ_{LFE} = Viscosity of gas laminar flow elements, micropoise.
μ_{std} = Viscosity of standard air, 180.1 micropoise.

6.2 PM_{10} Particulate Weight. Determine the weight of PM_{10} by summing the weights obtained from Container Numbers 1 and 3, less the acetone blank.

6.3 Total Particulate Weight. Determine the particulate catch for PM greater than PM_{10} from the weight obtained from Container Number 2 less the acetone blank, and add it to the PM_{10} particulate weight.

6.4 PM_{10} Fraction. Determine the PM_{10} fraction of the total particulate weight by dividing the PM_{10} particulate weight by the total particulate weight.

6.5 Total Cyclone Flow Rate. The average flow rate at standard conditions is determined from the average pressure drop across the total LFE and is calculated as follows:

$$Q_{s(std)} = K_1\left[X_T\,\Delta P\,\frac{\mu_{std}}{\mu_{LFE}} + Y_T\right]\frac{P_{bar} + P_{tot}/13.6}{T_m}$$

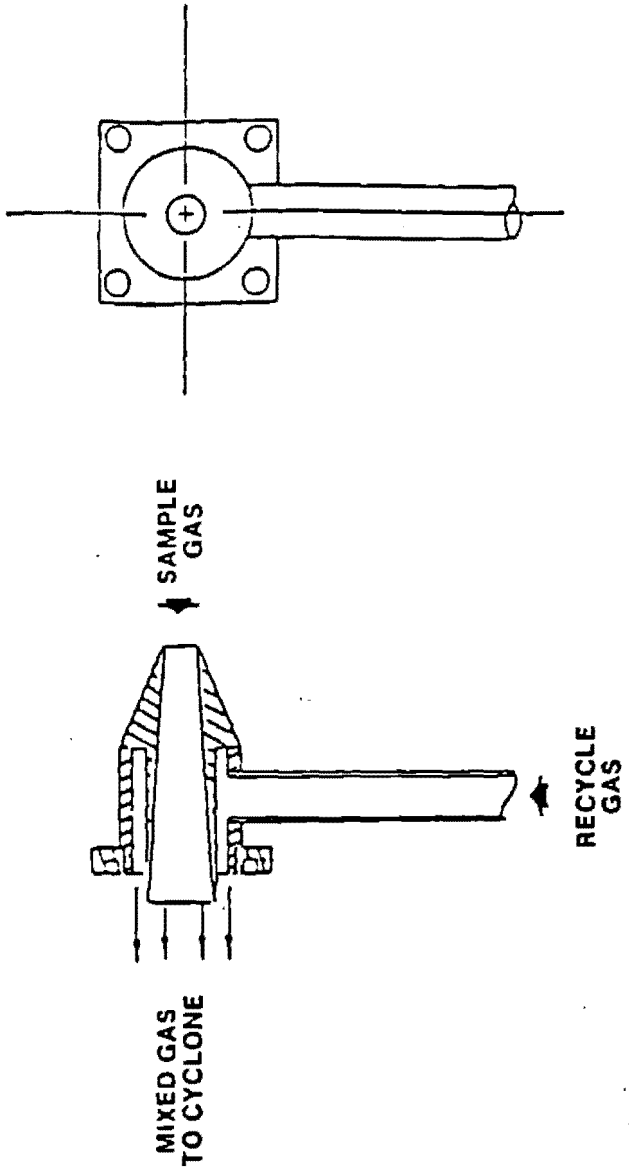

Figure 2. Schematic of EGR nozzle assembly.

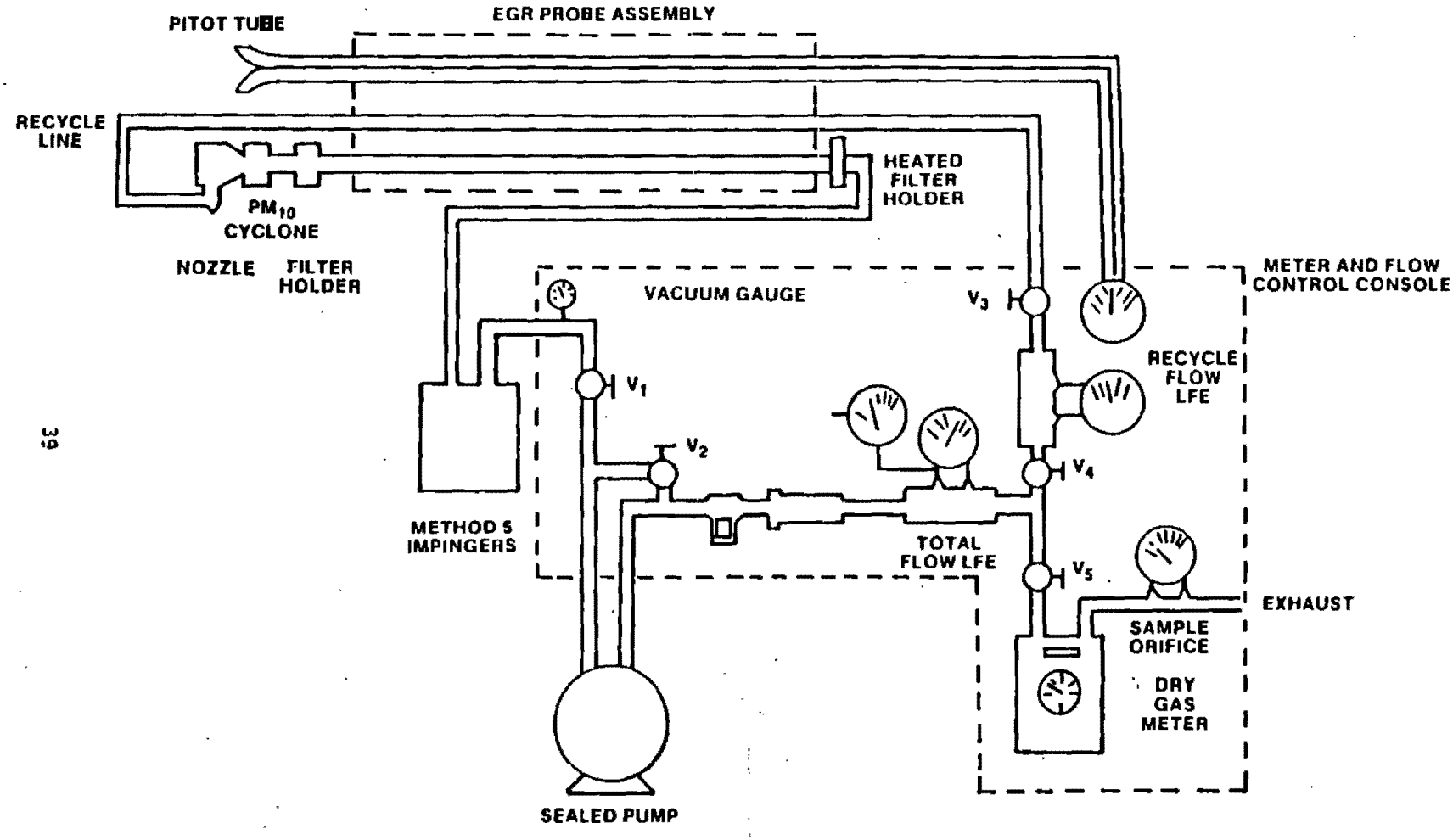

Figure 1. Schematic of the exhaust gas recycle train.

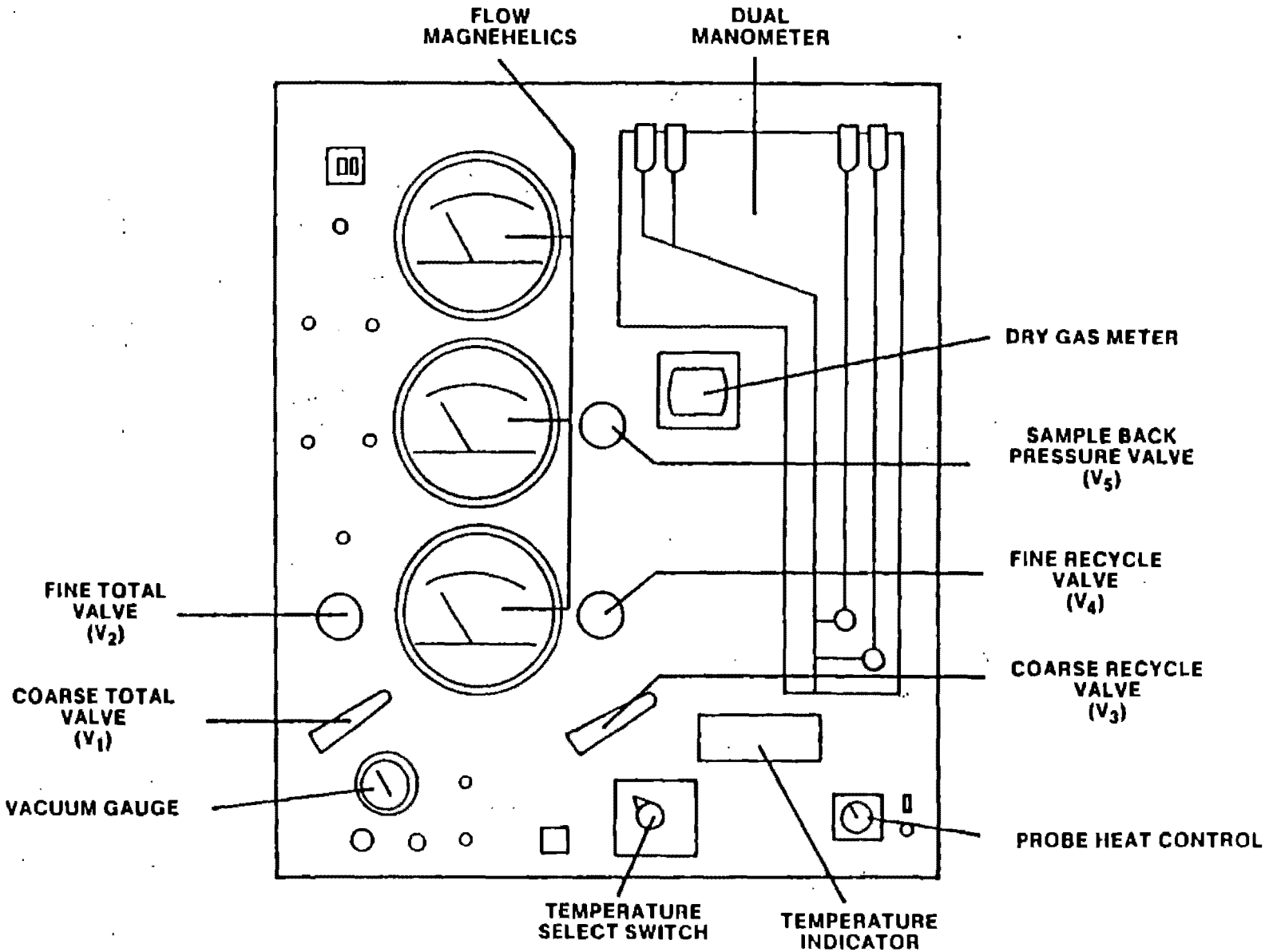

Figure 4. Example EGR control module (front view) showing principle components.

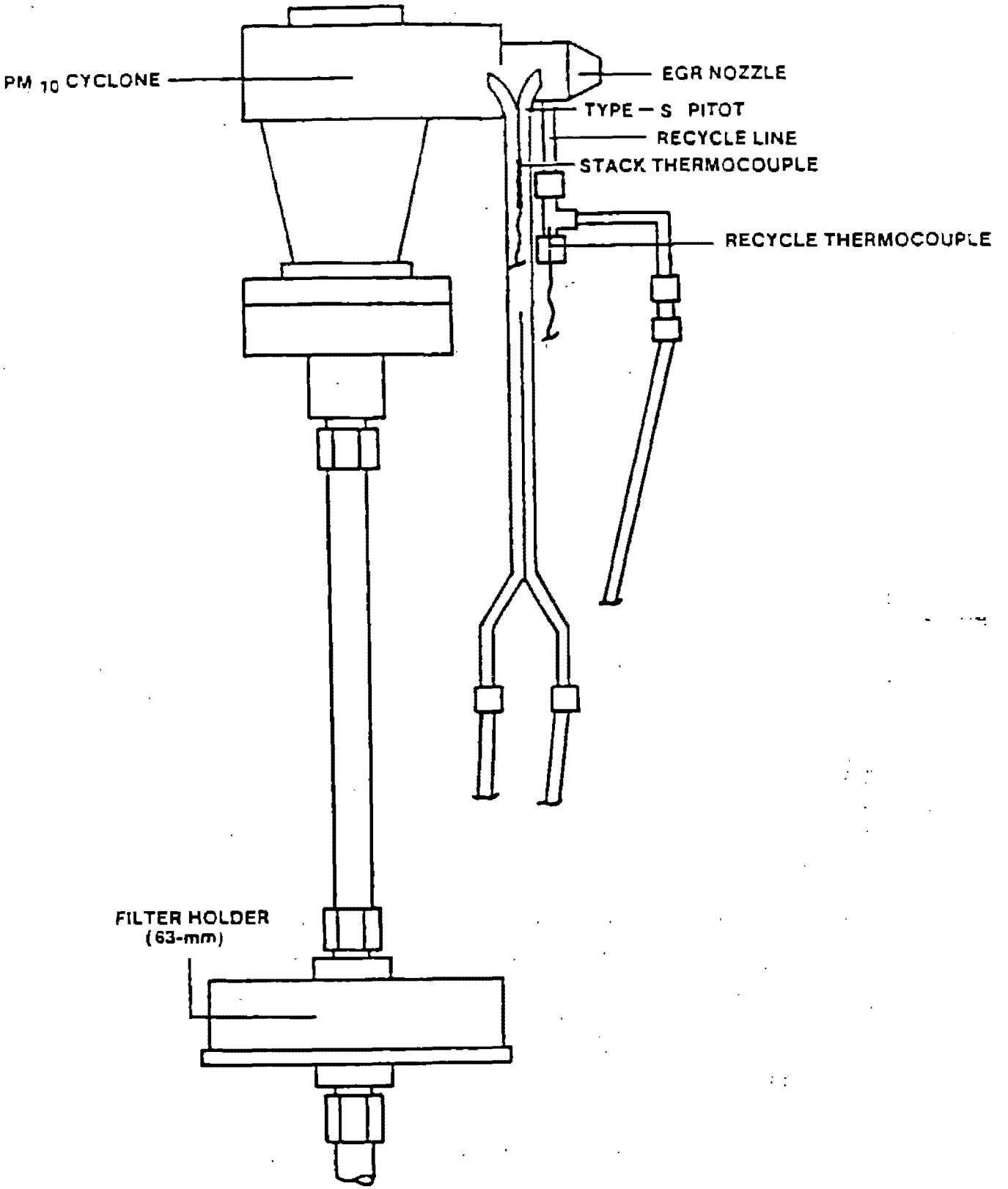

Figure 3. EGR PM_{10} cyclone sampling device.

EXAMPLE EMISSION GAS RECYCLE SETUP SHEET

VERSION 1.1 MAY 1986

TEST I.D.: SAMPLE SETUP
RUN DATE: 11/24/88
LOCATION: SOURCE SIM
OPERATOR(S): RH JB
NOZZLE DIAMETER (IN): .25

STACK CONDITIONS:

AVERAGE TEMPERATURE (F): 200.0
AVERAGE VELOCITY (FT/SEC): 15.0
AMBIENT PRESSURE (IN HG): 29.92
STACK PRESSURE (IN H20): .10

GAS COMPOSITION:
H20 = 10.0% MD = 28.84
02 = 20.9% MW = 27.75
CO2 = .0% (LB/LB MOLE)

TARGET PRESSURE DROPS

TEMPERATURE (F)

DP(PTO)		150	161	172	183	194	206	217	228
0.026	SAMPLE	.49	.49	.48	.47	.46	.45	.45	.45
	TOTAL	1.90	1.90	1.91	1.92	1.92	1.92	1.92	1.93
	RECYCLE	2.80	2.92	2.94	2.97	3.00	3.02	3.05	3.05
	%RCL	61%	61%	62%	62%	60%	60%	60%	60%
.031		.58	.56	.55	.55	.54	.53	.53	.52
		1.88	1.89	1.89	1.90	1.91	1.91	1.91	1.92
		2.71	2.74	2.77	2.80	2.82	2.85	2.88	2.90
		57%	57%	58%	58%	58%	59%	60%	60%
.035		.67	.65	.64	.63	.62	.61	.60	.59
		1.88	1.88	1.89	1.89	1.90	1.90	1.91	1.91
		2.57	2.60	2.63	2.66	2.69	2.72	2.74	2.78
		54%	55%	55%	56%	56%	57%	57%	57%
.039		.75	.74	.72	.71	.70	.69	.67	.66
		1.87	1.88	1.88	1.89	1.89	1.90	1.90	1.91
		2.44	2.47	2.50	2.53	2.56	2.59	2.62	2.67
		51%	52%	52%	53%	53%	54%	54%	55%

Figure 6. Example EGR setup sheet.

Barometric pressure, P_{bar}, in. Hg. = _____
Stack static pressure, P_g, in. H_2O. = _____
Average stack temperature, t_s, °F. = _____
Meter temperature, t_m, °F. = _____
Gas analysis:
%CO_2 = _____
%O_2 = _____
%N_2 + %CO = _____
Fraction moisture content, B_{ws} = _____

Calibration data:
Nozzle diameter, D_n in. = _____
Pitot coefficient, C_p = _____
$\Delta H@$, in. H_2O = _____
Molecular weight of stack gas, dry basis:
$M_d = 0.44$ (%CO_2) + 0.32 (%O_2) + 0.28 (%N_2 + %CO). = _____ lb/lb mole

Molecular weight of stack gas, wet basis:
$M_s = M_d (1 - B_{ws}) + 18B_{ws}$. = _____ lb/lb mole
Absolute stack pressure:
$P_s = P_{bar} + (P_g/13.6)$. = _____ in. Hg

$$K = 846.72\ D_n^4\ \Delta H@\ C_p^2\ (1 - B_{ws})^2 \frac{M_d\ (t_m + 460)\ P_s}{M_s\ (t_s + 460)\ P_{bar}} = _____$$

Desired meter orifice pressure (ΔH) for velocity head of stack gas (Δp):
$\Delta H = K\ \Delta p$ = _____ in. H_2O

Figure 7. Example worksheet 1, meter orifice pressure head calculation.

Barometric pressure, P_{bar}, in. Hg. = _____

Absolute stack pressure, P_s, in. Hg. = _____
Average stack temperature, T_s, °R. = _____
Meter temperature, T_m, °R. = _____

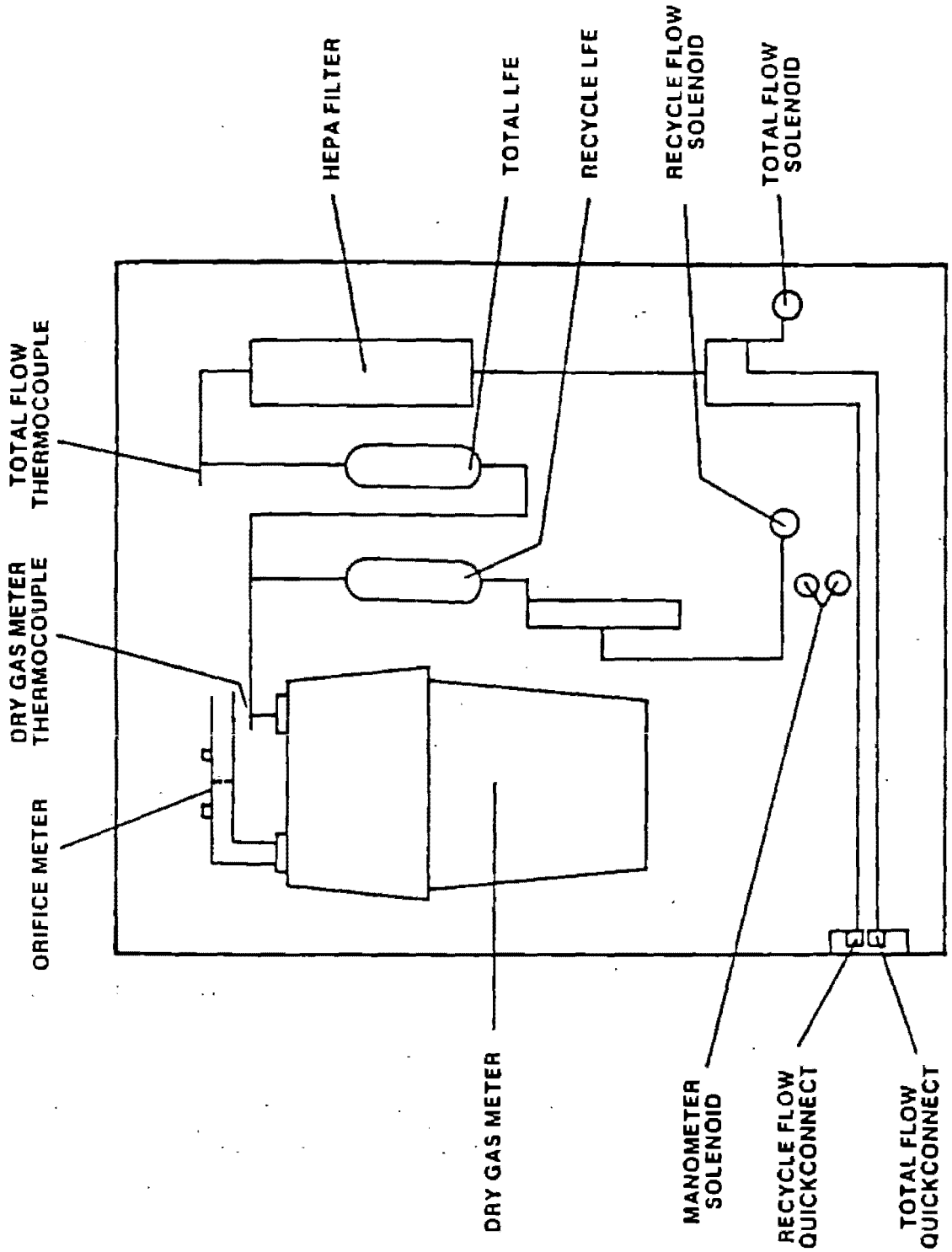

Figure 5. Example EGR control module (rear view) showing principle components.

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Figure 9. Example worksheet 3, recycle LFE pressure head.

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Molecular weight of stack gas, wet basis, M_s lb/lb mole. = _____

Pressure upstream of LFE, in. Hg. = 0.6

Gas analysis:

$\%O_2$ ..... = _____

Fraction moisture content, B_{ws}. = _____

Calibration data:

Nozzle diameter, D_n, in. = _____

Pitot coefficient, C_p ..... = _____

Total LFE calibration constant, X_t. = _____

Total LFE calibration constant, T_t. = _____

Absolute pressure upstream of LFE:

$P_{LFE} = P_{bar} + 0.8$ ..... = _____ in. Hg

Viscosity of gas in total LFE:

$\mu_{LFE} = 152.418 + 0.2552\ T_m + 3.2355 \times 10^{-5}\ T_m^2 + 0.53147\ (\%O_2)$. = _____

Viscosity of dry stack gas:

$\mu_d = 152.418 + 0.2552\ T_s + 3.2355 \times 10^{-5}\ T_s^2 + 0.53147\ (\%O_2)$. = _____

Constants:

$$K_1 = 1.5752 \times 10^{-5} \frac{\mu_{LFE}\ T_m\ P_s^{0.7031}\ \mu_d}{P_{LFE}\ M_d^{0.2949}\ T_s^{0.7031}} = _____$$

$$K_2 = 0.1539 \frac{\mu_{LFE}\ T_m\ D_n^4\ C_p}{P_{LFE}} \left[\frac{P_s}{T_s}\right]^{1/2}$$

$$K_3 = \frac{B_{ws}\ \mu_d\ [1 - 0.2949\ (1 - 18/M_d)] + 74.143\ B_{ws}\ (1 - B_{ws})}{\mu_d - 74.143\ B_{ws}} = _____$$

Figure 8. Example worksheet 2, total LFE pressure head.

$$A_1 \frac{K_1}{X_t} = \frac{\mu_{LFE}\ Y_t}{180.1\ X_t} = _____$$

$$B_1 = \frac{K_2\ K_3}{(M_d)^{1/2}\ X_t} = _____$$

Total LFE pressure head:

$\Delta p_t = A_1 - B_1\ (\Delta p)^{1/2}$ = _____ in. H_2O

Barometric pressure, P_{bar}, in. Hg = _____

Absolute stack pressure, P_s, in. Hg = _____

Average stack temperature, T_s, °R = _____

Meter temperature, T_m, °R = _____

Molecular weight of stack gas, dry basis, M_d, lb/lb mole = _____

Viscosity of LFE gas, μ_{LFE}, poise = _____

Viscosity of dry stack gas, μ_d, poise = _____

$$K_1 = 1.5752 \times 10^{-5} \frac{\mu_{LFE}\ T_m\ P_s^{0.7031}\ \mu_d}{P_{LFE}\ M_d^{0.2949}\ T_s^{0.7031}} = _____$$

$$K_2 = 0.1539 \frac{M_{LFE}\ T_m\ D_n^4\ C_p}{P_{LFE}} = \left[\frac{P_s}{T_s} =\right]^{1/2}$$

$$K_3 = \frac{\mu_d}{M_w^{0.7031}\ M_d^{0.2949}\ (\mu_d - 74.143\ B_{ws})} = _____$$

Absolute pressure upstream of LFE, P_{LFE}, in. Hg = _____

Calibration data:

Nozzle diameter, D_n, in. = _____

Pitot coefficient, C_p = _____

Recycle LFE calibration constant, X_r = _____

Recycle LFE calibration constant, Y_r = _____

$$A_2 = \frac{K}{X_r} - \frac{\mu_{LFE}}{180.1\ X_r} = _____$$

$$B_2 = \frac{K_2}{X_r} = _____$$

Pressure head for recycle LFE:

$\Delta P_r = A_2 - B_2\ (\Delta p)^{1/2}$ = _____ in. H_2O

Plant _____
Date _____
Run no. _____
Filter no. _____
Amount liquid lost during transport_____
Acetone blank volume, ml_____
Acetone wash volume, ml
(2)_____(3)_____
Acetone blank conc., mg/mg (Equation 5–4, Method 5)_____
Acetone wash blank, mg (Equation 5–5, Method 5)_____

Container number	Weight of particulate matter, mg		
	Final weight	Tare weight	Weight gain
1			
3			
Total			
Less acetone blank			
Weight of PM			
2			
Less acetone blank			

Container number	Weight of particulate matter, mg		
	Final weight	Tare weight	Weight gain
Total particulate weight			

Figure 11. EGR method analysis sheet.

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Run Code	Date	Stack Temperature (°F)	Gas Composition % CO_2 % O_2 % CO
Sampler ID	Start Time	Stack Static Pressure (in. H_2O)	Moisture Content
Filter ID	End Time	Ambient Temperature (°F)	
Sampler Orientation	Sampling Duration (min)	Ambient Pressure (in. Hg)	
Sampling Location	DGM (initial)	Gas Velocity	Pitot Leak Check (Pos) (Neg)
Nozzle Diameter-ID (in.)	DGM (final)	System Leak Check ($\geq$ 15 in. Hg)	Notes
Operator (s)	Sample Volume (ft^3)		
Dual Manometer Leveled and Zeroed?			
Magnehelics Zeroed?			

Run Time	Port No / Trav. Pt.	ΔP Pilot	$\Delta H_@$ Sample	DGM Volume	ΔP Total	P Inlet	ΔP Recycle	T_1 Stack	T_2 Recycle	T_3 Probe	T_4 LFE	T_5 DGM

Figure 10. Example EGR Procedure data sheet.

TABLE 1. PERFORMANCE SPECIFICATIONS FOR SOURCE PM_{10} Cyclones and Nozzle Combinations

Parameter	Units	Specification
1. Collection efficiency	Percent	Such that collection efficiency falls within envelope specified by Section 5.7.6 and Figure 13.
2. Cyclone cut size (D50).	μm	10 ± 1 μm aerodynamic diameter.

TABLE 2. PARTICLE SIZES AND NOMINAL GAS VELOCITIES FOR EFFICIENCY

Particle size (μm)[a]	Target gas velocities (m/sec)		
	7 ± 1.0	15 ± 1.5	25 ± 2.5
5 ± 0.5			
7 ± 0.5			
10 ± 0.5			
14 ± 1.0			
20 ± 1.0			

(a) Mass median aerodynamic diameter.

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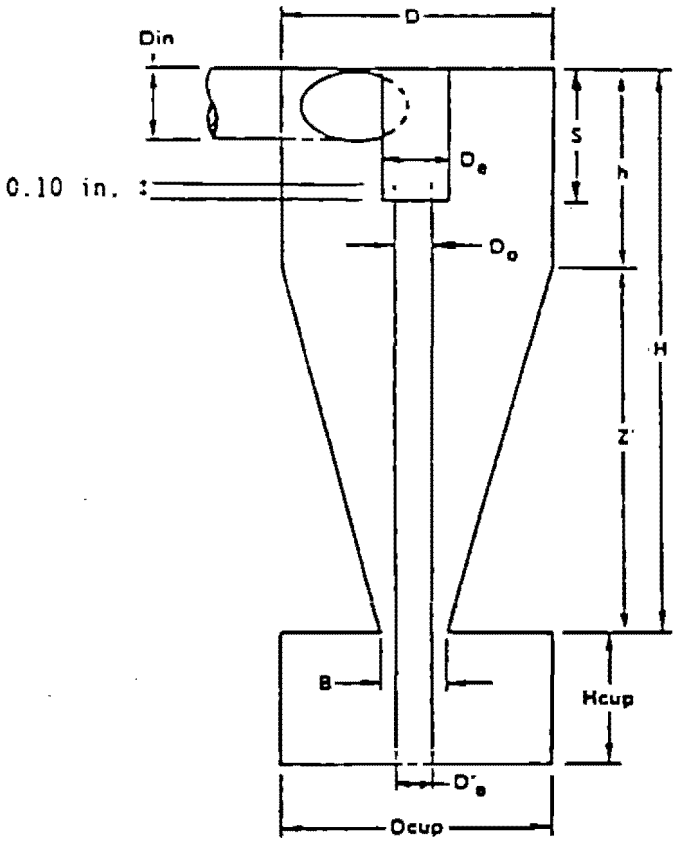
Cyclone Interior Dimensions



	Dimensions (±0.02 cm, ±0.01 in.)											
	Din	D	De	B	H	h	Z	S	Hcup	Dcup	D'_e	D_o
cm	1.27	4.47	1.50	1.88	6.95	2.24	4.71	1.57	2.25	4.45	1.02	1.24
inches	0.50	1.76	0.59	0.74	2.74	0.88	1.85	0.62	0.89	1.75	0.40	0.49

Figure 12. Cyclone design specifications.

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Emission Gas Recycle, Data Reduction,
Version 3.4 MAY 1986

Test ID. Code: Chapel Hill 2.
Test Location: Baghouse Outlet.
Test Site: Chapel Hill.
Test Date: 10/20/86.
Operators(s): JD RH MH.

Entered Run Data:

Temperatures:
T(STK)..... 251.0 F
T(RGL)..... 250.0 F
T(LFL)..... 81.0 F
T(DGM)..... 78.0 F
System Pressures:
DH(ORI)..... 1.10 INWG
DP(TOT)..... 1.91 INWG
P(INL)..... 12.15 INWG
DP(RCL)..... 2.21 INWG
DP(PTO)..... 0.00 INWG
Miscellanea:
P(BAR)..... 29.98 INWG

Entered Run Data—Continued

DP(STD)..... 0.10 INWG
V(DGM)..... 13.744 FTJ
TIME..... 60.00 MIN
%CO2..... 8.00
..... 20.00
%)..... 0.2500
ontent:
Estimate..... 0.0%
or
Condenser..... 7.0 ML
Column..... 0.0 GM
Rinse Masses:
Cyclone 1..... 21.7 MG
Filter..... 11.7 MG
Impinger Residue..... 0.0 MG
Blank Values:
CYC Rinse..... 0.0 MG
Filter Holder Rinse..... 0.0 MG
Filter Blank..... 0.0 MG
Impinger Rinse..... 0.0 MG

Calibration Values
CP(PITOT)..... 0.840
DH@(ORI)..... 10.000
M(TOT LFE)..... 0.2500
B(TOT LFE)..... −.0050
M(RCL LFE)..... 0.0948
B(RCL LFE)..... −.0007
DGM GAMMA..... 0.9940

Reduced Data

Stack Velocity (FT/SEC)..... 15.85
Stack Gas Moisture (%)..... 2.4
Sample Flow Rate (ACFM)..... 0.5108
Total Flow Rate (ACFM)..... 0.5815
Recycle Flow Rate (ACFM)..... 0.2700
Percent Recycle..... 46.7
Isokinetic Ratio (%)..... 95.1

	(Particulate)		(MG/DNCM)	(GR/ACF)	(GR/DCF)	(LB/DSCF) (X 1E6)
	(UM)	(% <)				
Cyclone 1	10.15	35.8	56.6	0.01754	0.02470	3.53701
Backup Filter			30.5	0.00966	0.01332	1.907
Particulate Total			87.2	0.02762	0.03802	5.444

Note: Figure 14. Example inputs and outputs of the EGR reduction program.

Method 201A—Determination of PM_{10} Emissions (Constant Sampling Rate Procedure)

1. Applicability and Principle

1.1 Applicability. This method applies to the in-stack measurement of particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10 (PM_{10}) from stationary sources. The EPA recognizes that condensible emissions not collected by an in-stack method are also PM_{10} and that emissions that contribute to ambient PM_{10} levels are the sum of condensible emissions and emissions measured by an in-stack PM_{10} method, such as this method or Method 201. Therefore, for establishing source contributions to ambient levels of PM_{10}, such as for emission inventory purposes, EPA suggests that source PM_{10} measurement include both in-stack PM_{10} and condensible emissions. Condensible emissions may be measured by an impinger analysis in combination with this method.

1.2 Principle. A gas sample is extracted at a constant flow rate through an in-stack sizing device, which separates PM greater than PM_{10}. Variations from isokinetic sampling conditions are maintained within well-defined limits. The particulate mass is determined gravimetrically after removal of uncombined water.

2. Apparatus

Note: Methods cited in this method are part of 40 CFR part 60, appendix A.

2.1 Sampling Train. A schematic of the Method 201A sampling train is shown in Figure 1 of this method. With the exception of the PM_{10} sizing device and in-stack filter, this train is the same as an EPA Method 17 train.

2.1.1 Nozzle. Stainless steel (316 or equivalent) with a sharp tapered leading edge. Eleven nozzles that meet the design specification in Figure 2 of this method are recommended. A larger number of nozzles with small nozzle increments increase the likelihood that a single nozzle can be used for the entire traverse. If the nozzles do not meet the design specifications in Figure 2 of this method, then the nozzles must meet the criteria in Section 5.2 of this method.

2.1.2 PM_{10} Sizer. Stainless steel (316 or equivalent), capable of determining the PM_{10} fraction. The sizing device shall be either a cyclone that meets the specifications in Section 5.2 of this method or a cascade impactor that has been calibrated using the procedure in Section 5.4 of this method.

2.1.3 Filter Holder, 63-mm, stainless steel. An Andersen filter, part number SE274, has been found to be acceptable for the in-stack filter. Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.4 Pitot Tube. Same as in Method 5, Section 2.1.3. The pitot lines shall be made of heat resistant tubing and attached to the probe with stainless steel fittings.

2.1.5 Probe Liner. Optional, same as in Method 5, Section 2.1.2.

2.1.6 Differential Pressure Gauge, Condenser, Metering System, Barometer, and Gas Density Determination Equipment. Same as in Method 5, Sections 2.1.4, and 2.1.7 through 2.1.10, respectively.

2.2 Sample Recovery.

2.2.1 Nozzle, Sizing Device, Probe, and Filter Holder Brushes. Nylon bristle brushes with stainless steel wire shafts and handles, properly sized and shaped for cleaning the nozzle, sizing dedvice, probe or probe liner, and filter holders.

2.2.2 Wash Bottles, Glass Sample Storage Containers, Petri Dishes, Graduated Cylinder and Balance, Plastic Storage Containers, Funnel and Rubber Policeman, and Funnel. Same as in Method 5, Sections 2.2.2 through 2.2.8, respectively.

2.3 Analysis. Same as in Method 5, Section 2.3.

3. Reagents

The reagents for sampling, sample recovery, and analysis are the same as that specified in Method 5, Sections 3.1, 3.2, and 3.3, respectively.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. Same as in Method 5, Section 4.1.1.

4.1.2 Preliminary Determinations. Same as in Method 5, Section 4.1.2, except use the directions on nozzle size selection and sampling time in this method. Use of any nozzle greater than 0.16 in. in diameter require a sampling port diameter of 6 inches. Also, the required maximum number of traverse points at any [illegible] be 12.

4.1.2.1 The sizing [illegible] be in-stack or maintained at stack temperature during sampling. The blockage effect of the CSR sampling assembly will be minimal if the cross-sectional area of the sampling assembly is 3 percent or less of the cross-sectional area of the duct. If the cross-sectional area of the assembly is greater than 3 percent of the cross-sectional area of the duct, then either determine the pitot coefficient at sampling conditions or use a standard pitot with a known coefficient in a configuration with the CSR sampling

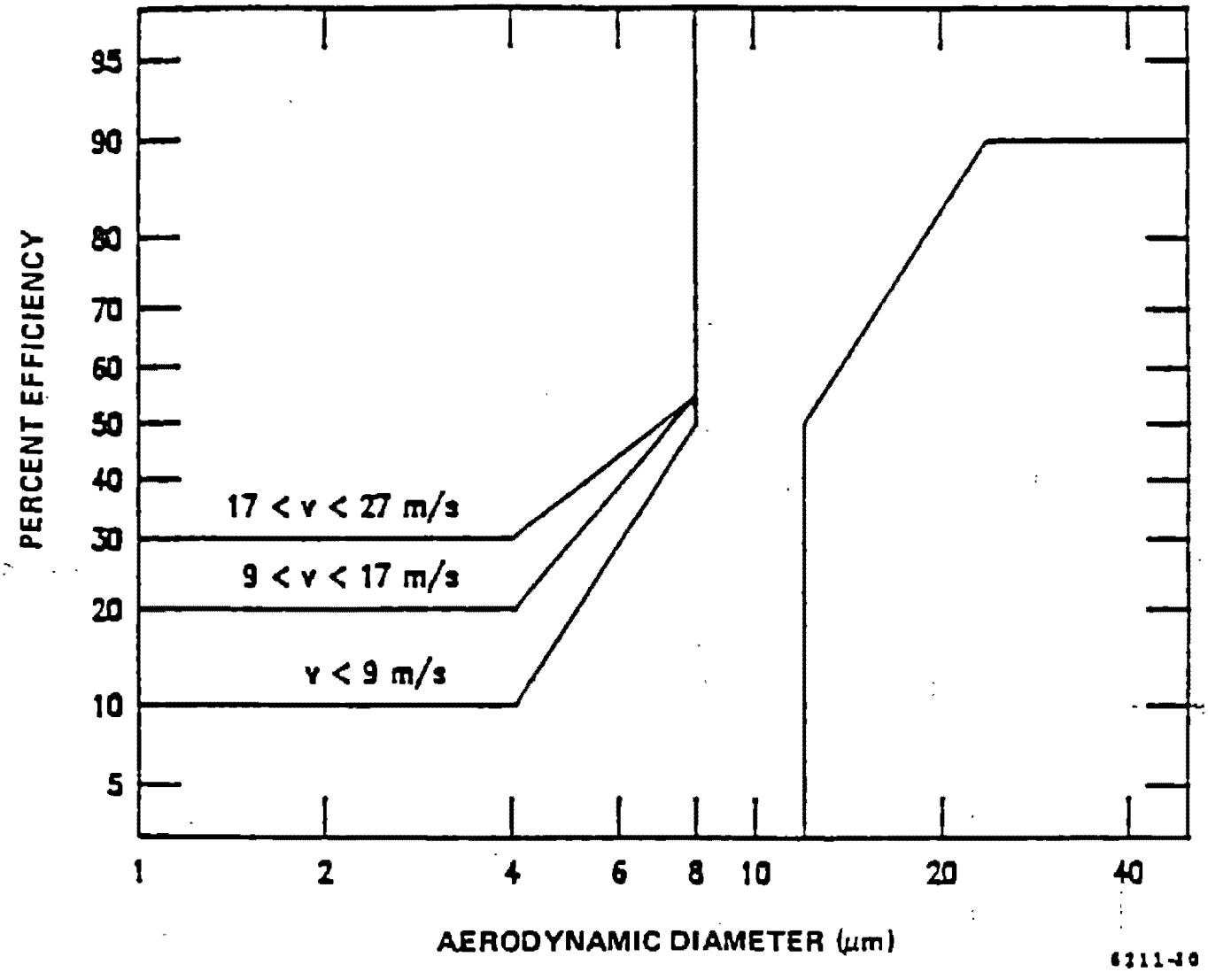

Figure 13. Efficiency envelope for the PM_{10} cyclone.

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operating parameters of the particle generation system.

5.2.2.2 Certify the monodispersity of the particles for each test either by microscopic inspection of collected particles on filters or by other suitable monitoring techniques such as an optical particle counter followed by a multichannel pulse height analyzer. If the proportion of multiplets and satellites in an aerosol exceeds 10 percent by mass, the particle generation system is unacceptable for the purpose of this test. Multiplets are particles that are agglomerated, and satellites are particles that are smaller than the specified size range.

5.2.3 Schematic Drawings. Schematic drawings of the wind tunnel and blower system and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques shall be furnished with calibration data to the reviewing agency.

5.2.4 Flow Measurements. Measure the cyclone air flow rates with a dry gas meter and a stopwatch, or a calibrated orifice system capable of measuring flow rates to within 2 percent.

5.2.5 Performance Specification Procedure. Establish test particle generator operation and verify particle size microscopically. If monodisperity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements may be made at this time.

5.2.5.1 The cyclone cut size, or D_{50}, of a cyclone is defined here as the particle size having a 50 percent probability of penetration. Determine the cyclone flow rate at which D_{50} is 10 μm. A suggested procedure is to vary the cyclone flow rate while keeping a constant particle size of 10 μm. Measure the PM collected in the cyclone (m_c), the exit tube (m_t), and the filter (m_f). Calculate cyclone efficiency (E_c) for each flow rate as follows:

$$E_c = \frac{m_c}{(m_c + m_t + m_f)} \times 100$$

5.2.5.2 Do three replicates and calculate the average cyclone efficiency [$E_{c(avg)}$] as follows:

$$E_{c(avg)} = (E_1 + E_2 + E_3)/3$$

Where E_1, E_2, and E_3 are replicate measurements of E_c.

5.2.5.3 Calculate the standard deviation (σ) for the replicate measurements of E_c as follows:

$$\left[\sigma = \left[\frac{(E_1^2 + E_2^2 + E_3^2) - \frac{(E_1 + E_2 + E_3)^2}{3}}{2}\right]^{1/2}\right.$$

If σ exceeds 0.10, repeat the replicated runs.

5.2.5.4 Measure the overall efficiency of the cyclone and nozzle, E_o, at the particle sizes and nominal gas velocities in Table 2 of this method using the following procedure.

5.2.5.5 Set the air velocity and particle size from one of the conditions in Table 2 of this method. Establish isokinetic sampling conditions and the correct flow rate in the cyclone (obtained by procedures in this section) such that the D_{50} is 10 μm. Sample long enough to obtain ±5 percent precision on total collected mass as determined by the precision and the sensitivity of measuring technique. Determine separately the nozzle catch (m_n), cyclone catch (m_c), cyclone exit tube (M_t), and collection filter catch (m_f) for each particle size and nominal gas velocity in Table 2 of this method. Calculate overall efficiency (E_o) as follows:

$$E_o = \frac{(m_n + m_c)}{(m_n + m_c + m_t + m_f)} \times 100$$

5.2.5.6 Do three replicates for each combination of gas velocity and particle size in Table 2 of this method. Use the equation below to calculate the average overall efficiency [$E_{o(avg)}$] for each combination following the procedures described in this section for determining efficiency.

$$E_{o(avg)} = (E_1 + E_2 + E_3)/3$$

Where E_1, E_2, and E_3 are replicate measurements of E_o.

5.2.5.7 Use the formula in Section 5.2.5.3 to calculate σ for the replicate measurements. If σ exceeds 0.10 or if the particle sizes and nominal gas velocities are not within the limits specified in Table 2 of this method, repeat the replicate runs.

5.2.6 Criteria for Acceptance. For each of the three gas stream velocities, plot the $E_{o(avg)}$ as a function of particle size on Figure 8 of this method. Draw smooth curves through all particle sizes. $E_{o(avg)}$ shall be within the banded region for all sizes, and the $E_{c(avg)}$ shall be 50±0.5 percent at 10 μm.

5.3 Cyclone Calibration Procedure. The purpose of this procedure is to develop the relationship between flow rate, gas viscosity, gas density, and D_{50}.

5.3.1 Calculate Cyclone Flow Rate. Determine flow rates and D_{50}'s for three different particle sizes between 5 μm and 15 μm, one of which shall be 10 μm. All sizes must be determined within 0.5 μm. For each size, use a different temperature within 60 °C (108 °F) of the temperature at which the cyclone is to be used and conduct triplicate runs. A suggested procedure is to keep the particle size constant and vary the flow rate.

5.3.1.1 On log-log graph paper, plot the Reynolds number (Re) on the abscissa, and the square root of the Stokes 50 number [$(Stk_{50})^{1/2}$] on the ordinate for each temperature. Use the following equations to compute both values:

$$Re = \frac{4 \rho Q_{cyc}}{d_{cyc} \pi \mu_{cyc}}$$

$$(Stk_{50})^{1/2} = \left[\frac{4 Q_{cyc} (D_{50})^2}{9 \pi \mu_{cyc})^3}\right]^{1/2}$$

where:

Q_{cyc} = Cyclone flow rate, cm^3/sec.
ρ = Gas density, g/cm^3.
d_{cyc} = Diameter of cyclone inlet, cm.
μ_{cyc} = Viscosity of gas through the cyclone, micropoise.
D_{50} = Aerodynamic diameter of a particle having a 50 percent probability of penetration, cm.

5.3.1.2 Use a linear regression analysis to determine the slope (m) and the Y-intercept (b). Use the following formula to determine Q_c, the cyclone flow rate required for a cut size of 10 μm.

$$Q_c = \frac{\pi \mu_{cyc}}{4}\left[(3000)(K_1) - b\right]^{-(6+m)}\left[\frac{T_s}{M_s P_s}\right]^{m/(m-0.5)} d^{(m+1.5)/(m-0.5)}$$

where:

m = Slope of the calibration line.
b = y-intercept of the calibration line.
Q_c = Cyclone flow rate for a cut size of 10 μm, cm^3/sec.
d = Diameter of nozzle, cm.
T_s = Stack gas temperature, °R.
P_s = Absolute stack pressure, in. Hg.
M_s = Molecular weight of the stack gas, lb/lb-mole.
$K_1 = 4.077 \times 10^{-5}$.

5.3.1.3 Refer to the Method 201A operators manual, entitled *Application Guide for Source PM_{10} Measurement with Constant*

assembly such that flow disturbances are minimized.

4.1.2.2 The setup calculations can be performed by using the following procedures.

4.1.2.2.1 In order to maintain a cut size of 10 μm in the sizing device, the flow rate through the sizing device must be maintained at a constant, discrete value during the run. If the sizing device is a cyclone that meets the design specifications in Figure 3 of this method, use the equations in Figure 4 of this method to calculate three orifice heads (ΔH): one at the average stack temperature, and the other two at temperatures ±28 °C (±50 °F) of the average stack temperature. Use ΔH calculated at the average stack temperature as the pressure head for the sample flow rate as long as the stack temperature during the run is within 28 °C (50 °F) of the average stack temperature. If the stack temperature varies by more than 28 °C (50 °F), then use the appropriate ΔH.

4.1.2.2.2 If the sizing device is a cyclone that does not meet the design specifications in Figure 3 of this method, use the equations in Figure 4 of this method, except use the procedures in Section 5.3 of this method to determine Q_a, the correct cyclone flow rate for a 10 μm size.

4.1.2.2.3 To select a nozzle, use the equations in Figure 5 of this method to calculate Δp_{min} and Δp_{max} for each nozzle at all three temperatures. If the sizing device is a cyclone that does not meet the design specifications in Figure 3 of this method, the example worksheets can be used.

4.1.2.2.4 Correct the Method 2 pitot readings to Method 201A pitot readings by multiplying the Method 2 pitot readings by the square of a ratio of the Method 201A pitot coefficient to the Method 2 pitot coefficient. Select the nozzle for which Δp_{min} and Δp_{max} bracket all of the corrected Method 2 pitot readings. If more than one nozzle meets this requirement, select the nozzle giving the greatest symmetry. Note that if the expected pitot reading for one or more points is near a limit for a chosen nozzle, it may be outside the limits at the time of the run.

4.1.2.2.5 Vary the dwell time, or sampling time, at each traverse point proportionately with the point velocity. Use the equations in Figure 6 of this method to calculate the dwell time at the first point and at each subsequent point. It is recommended that the number of minutes sampled at each point be rounded to the nearest 15 seconds.

4.1.3 Preparation of Collection Train. Same as in Method 5, Section 4.1.3, except omit directions about a glass cyclone.

4.1.4 Leak-Check Procedure. The sizing device is removed before the post-test leak-check to prevent any disturbance of the collected sample prior to analysis.

4.1.4.1 Pretest Leak-Check. A pretest leak-check of the entire sampling train, including the sizing device, is required. Use the leak-check procedure in Method 5, Section 4.1.4.1 to conduct a pretest leak-check.

4.1.4.2 Leak-Checks During Sample Run. Same as in Method 5, Section 4.1.4.1.

4.1.4.3 Post-Test Leak-Check. A leak-check is required at the conclusion of each sampling run. Remove the cyclone before the leak-check to prevent the vacuum created by the cooling of the probe from disturbing the collected sample and use the procedure in Method 5, Section 4.1.4.3 to conduct a post-test leak-check.

4.1.5 Method 201A Train Operation. Same as in Method 5, Section 4.1.5, except use the procedures in this section for isokinetic sampling and flow rate adjustment. Maintain the flow rate calculated in Section 4.1.2.2.1 of this method throughout the run provided the stack temperature is within 28 °C (50 °F) of the temperature used to calculate ΔH. If stack temperatures vary by more than 28 °C (50 °F), use the appropriate ΔH value calculated in Section 4.1.2.2.1 of this method. Calculate the dwell time at each traverse point as in Figure 6 of this method.

4.1.6 Calculation of Percent Isokinetic Rate and Aerodynamic Cut Size (D_{50}). Calculate percent isokinetic rate and D_{50} (see Calculations, Section 6 of this method) to determine whether the test was valid or another test run should be made. If there was difficulty in maintaining isokinetic sampling rates within the prescribed range, or if the D_{50} is not in its proper range because of source conditions, the Administrator may be consulted for possible variance.

4.2 Sample Recovery. If a cascade impactor is used, use the manufacturer's recommended procedures for sample recovery. If a cyclone is used, use the same sample recovery as that in Method 5, Section 4.2, except an increased number of sample recovery containers is required.

4.2.1 *Container Number 1* (In-Stack Filter). The recovery shall be the same as that for Container Number 1 in Method 5, Section 4.2.

4.2.3 *Container Number 2* (Cyclone or Large PM Catch). This step is optional. The anisokinetic error for the cyclone PM is theoretically larger than the error for the PM_{10} catch. Therefore, adding all the fractions to get a total PM catch is not as accurate as Method 5 or Method 201. Disassemble the cyclone and remove the nozzle to recover the large PM catch. Quantitatively recover the PM from the interior surfaces of the nozzle and cyclone, excluding the "turn around" cup and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.4 *Container Number 3* (PM_{10}). Quantitatively recover the PM from all of the surfaces from the cyclone exit to the front half of the in-stack filter holder, including the "turn around" cup inside the cyclone and the interior surfaces of the exit tube. The recovery shall be the same as that for Container Number 2 in Method 5, Section 4.2.

4.2.6 *Container Number 4* (Silica Gel). The recovery shall be the same as that for Container Number 3 in Method 5, Section 4.2.

4.2.7 *Impinger Water*. Same as in Method 5, Section 4.2, under "Impinger Water."

4.3 Analysis. Same as in Method 5, Section 4.3, except handle Method 201A Container Number 1 like Container Number 1, Method 201A Container Numbers 2 and 3 like Container Number 2, and Method 201A Container Number 4 like Container Number 3. Use Figure 7 of this method to record the weights of PM collected. Use Figure 5–3 in Method 5, Section 4.3, to record the volume of water collected.

4.4 Quality Control Procedures. Same as in Method 5, Section 4.4.

5. Calibration

Maintain an accurate laboratory log of all calibrations.

5.1 Probe Nozzle, Pitot Tube, Metering System, Probe Heater Calibration, Temperature Gauges, Leak-check of Metering System, and Barometer. Same as in Method 5, Section 5.1 through 5.7, respectively.

5.2 Probe Cyclone and Nozzle Combinations. The probe cyclone and nozzle combinations need not be calibrated if both meet design specifications in Figures 2 and 3 of this method. If the nozzles do not meet design specifications, then test the cyclone and nozzle combinations for conformity with performance specifications (PS's) in Table 1 of this method. If the cyclone does not meet design specifications, then the cyclone and nozzle combination shall conform to the PS's and calibrate the cyclone to determine the relationship between flow rate, gas viscosity, and gas density. Use the procedures in Section 5.2 of this method to conduct PS tests and the procedures in Section 5.3 of this method to calibrate the cyclone. The purpose of the PS tests are to conform that the cyclone and nozzle combination has the desired sharpness of cut. Conduct the PS tests in a wind tunnel described in Section 5.2.1 of this method and particle generation system described in Section 5.2.2 of this method. Use five particle sizes and three wind velocities as listed in Table 2 of this method. A minimum of three replicate measurements of collection efficiency shall be performed for each of the 15 conditions listed, for a minimum of 45 measurements.

5.2.1 Wind Tunnel. Perform the calibration and PS tests in a wind tunnel (or equivalent test apparatus) capable of establishing and maintaining the required gas stream velocities within 10 percent.

5.2.2 Particle Generation System. The particle generation system shall be capable of producing solid monodispersed dye particles with the mass median aerodynamic diameters specified in Table 2 of this method. Perform the particle size distribution verification on an integrated sample obtained during the sampling period of each test. An acceptable alternative is to verify the size distribution of samples obtained before and after each test, with both samples required to meet the diameter and monodispersity requirements for an acceptable test run.

5.2.2.1 Establish the size of the solid dye particles delivered to the test section of the wind tunnel by using the operating parameters of the particle generation system, and verify them during the tests by microscopic examination of samples of the particles collected on a membrane filter. The particle size, as established by the operating parameters of the generation system, shall be within the tolerance specified in Table 2 of this method. The precision of the particle size verification technique shall be at least ±0.5 μm, and particle size determined by the verification technique shall not differ by more than 10 percent from that established by the

$$D_{50} = \beta_1 \left[\frac{T_s}{M_c P_s} \right]^{0.3091} \left[\frac{\mu_{mv}}{Q_s} \right]^{0.1091}$$

where $\beta_1 = 0.027754$ for metric units (0.15625 for English units).

6.3.5 Acceptable Results. The results are acceptable if two conditions are met. The first is that 9.0 μm < D_{50} < 11.0 μm. The second is that no sampling points are outside Δp_{min} and Δp_{max}, or that 80 percent < I < 120 percent and no more than one sampling point is outside Δp_{min} and Δp_{max}. If D_{50} is less than 9.0 μm, reject the results and repeat the test.

7. Bibliography

1. Same as Bibliography in Method 5.

2. McCain, J.D., J.W. Ragland, and A.D. Williamson. Recommended Methodology for the Determination of Particle Size Distributions in Ducted Sources, Final Report. Prepared for the California Air Resources Board by Southern Research Institute. May 1986.

3. Farthing, W.E., S.S. Dawes, A.D. Williamson, J.D. McCain, R.S. Martin, and J.W. Ragland. Development of Sampling Methods for Source PM_{10} Emissions. Southern Research Institute for the Environmental Protection Agency. April 1989. NTIS PB 89 190075, EPA/600/3–88–056.

4. *Application Guide for Source PM_{10} Measurement with Constant Sampling Rate.* EPA/600/3–88–057.

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Sampling Rate, for directions in the use of this equation for Q in the setup calculations.

5.4 Cascade Impactor. The purpose of calibrating a cascade impactor is to determine the empirical constant (STK_{50}), which is specific to the impactor and which permits the accurate determination of the cut size of the impactor stages at field conditions. It is not necessary to calibrate each individual impactor. Once an impactor has been calibrated, the calibration data can be applied to other impactors of identical design.

5.4.1 Wind Tunnel. Same as in Section 5.2.1 of this method.

5.4.2 Particle Generation System. Same as in Section 5.2.2 of this method.

5.4.3 Hardware Configuration for Calibrations. An impaction stage constrains an aerosol to form circular or rectangular jets which are directed toward a suitable substrate where the larger aerosol particles are collected. For calibration purposes, three stages of the cascade impactor shall be discussed and designated calibration stages 1, 2, and 3. The first calibration stage consists of the collection substrate of an impaction stage and all upstream surfaces up to and including the nozzle. This may include other *preceding impactor stages*. The second and third calibration stages consist of each respective collection substrate and all upstream surfaces up to but excluding the collection substrate of the preceding calibration stage. This may include intervening impactor stages which are not designated as calibration stages. The cut size, or D_{50}, of the adjacent calibration stages shall differ by a factor of not less than 1.5 and not more than 2.0. For example, if the first calibration stage has a D_{50} of 12 μm, then the D_{50} of the downstream stage shall be between 6 and 8 μm.

5.4.3.1 It is expected, but not necessary, that the complete hardware assembly will be used in each of the sampling runs of the calibration and performance determinations. Only the first calibration stage must be tested under isokinetic sampling conditions. The second and third calibration stages must be calibrated with the collection substrate of the preceding calibration stage in place, so that *gas flow patterns existing in field operation* will be simulated.

5.4.3.2 Each of the PM_{10} stages should be calibrated with the type of collection substrate, viscid material (such as grease) or glass fiber, used in PM_{10} measurements. Note that most materials used as substrates at elevated temperatures are not viscid at normal laboratory conditions. The substrate material used for calibrations should minimize particle bounce, yet be viscous enough to withstand erosion or deformation by the impactor jets and not interfere with the procedure for measuring the collected PM.

5.4.4 Calibration Procedure. Establish test particle generator operation and verify particle size microscopically. If monodispersity is to be verified by measurements at the beginning and the end of the run rather than by an integrated sample, these measurements shall be made at this time. Measure in triplicate the PM collected by the calibration stage (m) and the PM on all surfaces downstream of the respective calibration stage (m') for all of the flow rates and particle size combinations shown in Table 2 of this method. Techniques of mass measurement may include the use of a dye and spectrophotometer. Particles on the upstream side of a jet plate shall be included with the substrate downstream, except agglomerates of particles, which shall be included with the preceding or upstream substrate. Use the following formula to calculate the collection efficiency (E) for each stage.

5.4.4.1 Use the formula in Section 5.2.5.3 of this method to calculate the standard deviation (σ) for the replicate measurements. If σ exceeds 0.10, repeat the replicate runs.

5.4.4.2 Use the following formula to *calculate the average collection efficiency* (E_{ave}) for each set of replicate measurements.

$$E_{ave} = (E_1 + E_2 + E_3)/3$$

where E_1, E_2, and E_3 are replicate measurements of E.

5.4.4.3 Use the following formula to calculate Stk for each E_{ave}.

$$Stk = \frac{D^2 Q}{9 \mu A d_j}$$

where:

D = Aerodynamic diameter of the test particle, cm $(g/cm^3)^{1/2}$.
Q = Gas flow rate through the calibration stage at inlet conditions, cm^3/sec.
μ = Gas viscosity, micropoise.
A = Total cross-sectional area of the jets of the calibration stage, cm^2.
d_j = Diameter of one jet of the calibration stage, cm.

5.4.4.4 Determine Stk_{50} for each calibration stage by plotting E_{ave} versus Stk on log-log paper. Stk_{50} is the Stk number at 50 percent efficiency. Note that particle bounce can cause efficiency to decrease at high values of Stk. Thus, 50 percent efficiency can occur at multiple values of Stk. The calibration data should clearly indicate the value of Stk_{50} for minimum particle bounce. Impactor efficiency versus Stk with minimal particle bounce is characterized by a monotonically increasing function with constant or increasing slope with increasing Stk.

5.4.4.5 The Stk_{50} of the first calibration stage can potentially decrease with decreasing nozzle size. Therefore, calibrations should be performed with enough nozzle sizes to provide a measured value within 25 percent of any nozzle size used in PM_{10} measurements.

5.4.5 Criteria For Acceptance. Plot E_{ave} for the first calibration stage versus the square root of the ratio of Stk to Stk_{50} on Figure 9 of this method. Draw a smooth curve through all of the points. The curve shall be within the banded region.

6. Calculations

6.1 Nomenclature.

B_{ws} = Moisture fraction of stack, by volume, dimensionless.
C_1 = Viscosity constant, 51.12 micropoise for °K (51.05 micropoise for °R).
C_2 = Viscosity constant, 0.372 micropoise/°K (0.207 micropoise/°R).
C_3 = Viscosity constant, 1.05×10^{-3} micropoise/$°K^2$ (3.24×10^{-4} micropoise/$°R^2$).
C_4 = Viscosity constant, 53.147 micropoise/fraction O_2.
C_5 = Viscosity constant, 74.143 micropoise/fraction H_2O.
D_{50} = Diameter of particles having a 50 percent probability of penetration, μm.
f_c = Stack gas fraction O_2, by volume, dry basis.
K_1 = 0.3858 °K/mm Hg (17.64 °R/in. Hg).
M_d = Wet molecular weight of mixed gas through the PM_{10} cyclone, g/g-mole (lb/lb-mole).
M_s = Dry molecular weight of stack gas, g/g-mole (lb/lb-mole).
P_{bar} = Barometric pressure at sampling site, mm Hg (in. Hg).
P_s = Absolute stack pressure, mm Hg (in. Hg).
Q_s = Total cyclone flow rate at wet cyclone conditions, m^3/min (ft^3/min).
Q_{std} = Total cyclone flow rate at standard conditions, dscm/min (dscf/min).
T_m = Average absolute temperature of dry meter, °K (°R).
T_s = Average absolute stack gas temperature, °K (°R).
V_{wstd} = Volume of water vapor in gas sample (standard conditions), scm (scf).
θ = Total sampling time, min.
μ_{cyc} = Viscosity of mixed cyclone gas, micropoise.
μ_{std} = Viscosity of standard air, 180.1 micropoise.

6.2 Analysis of Cascade Impactor Data. Use the manufacturer's recommended procedures to analyze data from cascade impactors.

6.3 Analysis of Cyclone Data. Use the following procedures to analyze data from a single stage cyclone.

6.3.1 PM_{10} Weight. Determine the PM catch in the PM_{10} range from the sum of the weights obtained from Container Numbers 1 and 3 less the acetone blank.

6.3.2 Total PM Weight (optional). Determine the PM catch for greater than PM_{10} from the weight obtained from Container 2 less the acetone blank, and add it to the PM_{10} weight.

6.3.3 PM_{10} Fraction. Determine the PM_{10} fraction of the total particulate weight by dividing the PM_{10} particulate weight by the total particulate weight.

6.3.4 Aerodynamic Cut Size. Calculate the stack gas viscosity as follows:

$$\mu_{cyc} = C_1 + C_2 T_s + C_3 T_s^2 + C_4 f_{oc} - C_5 B_{ws}$$

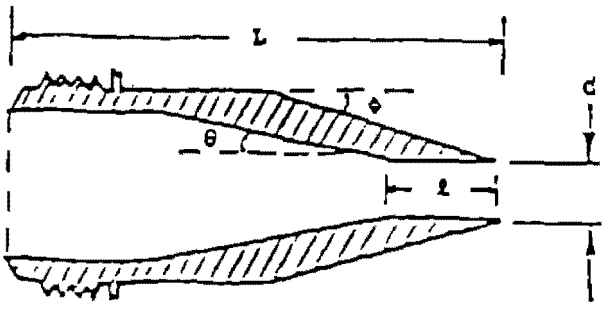
6.3.4.1 The PM_{10} flow rate, at actual cyclone conditions, is calculated as follows:

$$Q_s = \frac{T_s}{K_1 P_s}\left[Q_{std} + \frac{V_{wstd}}{\theta} \right]$$

6.3.4.2 Calculate the molecular weight on a wet basis of the stack gas as follows:

$$M_s = M_d(1 - B_{ws}) + 18.0(B_{ws})$$

6.3.4.3 Calculate the actual D_{50} of the cyclone for the given conditions as follows:



Nozzle Diameter (inches)	Cone Angle, θ (degrees)	Outside taper, ϕ (degrees)	Straight inlet length, ℓ (inches)	Total Length L (inches)
0.136	4	15	<0.05	2.653±0.05
0.150	4	15	<0.05	2.553±0.05
0.164	5	15	<0.05	1.970±0.05
0.180	6	15	<0.05	1.572±0.05
0.197	6	15	<0.05	1.491±0.05
0.215	6	15	<0.05	1.45 ±0.05
0.233	6	15	<0.05	1.45 ±0.05
0.264	5	15	<0.05	1.45 ±0.05
0.300	4	15	<0.05	1.48 ±0.05
0.342	4	15	<0.05	1.45 ±0.05
0.390	3	15	<0.05	1.45 ±0.05

Figure 2. Nozzle design specifications.

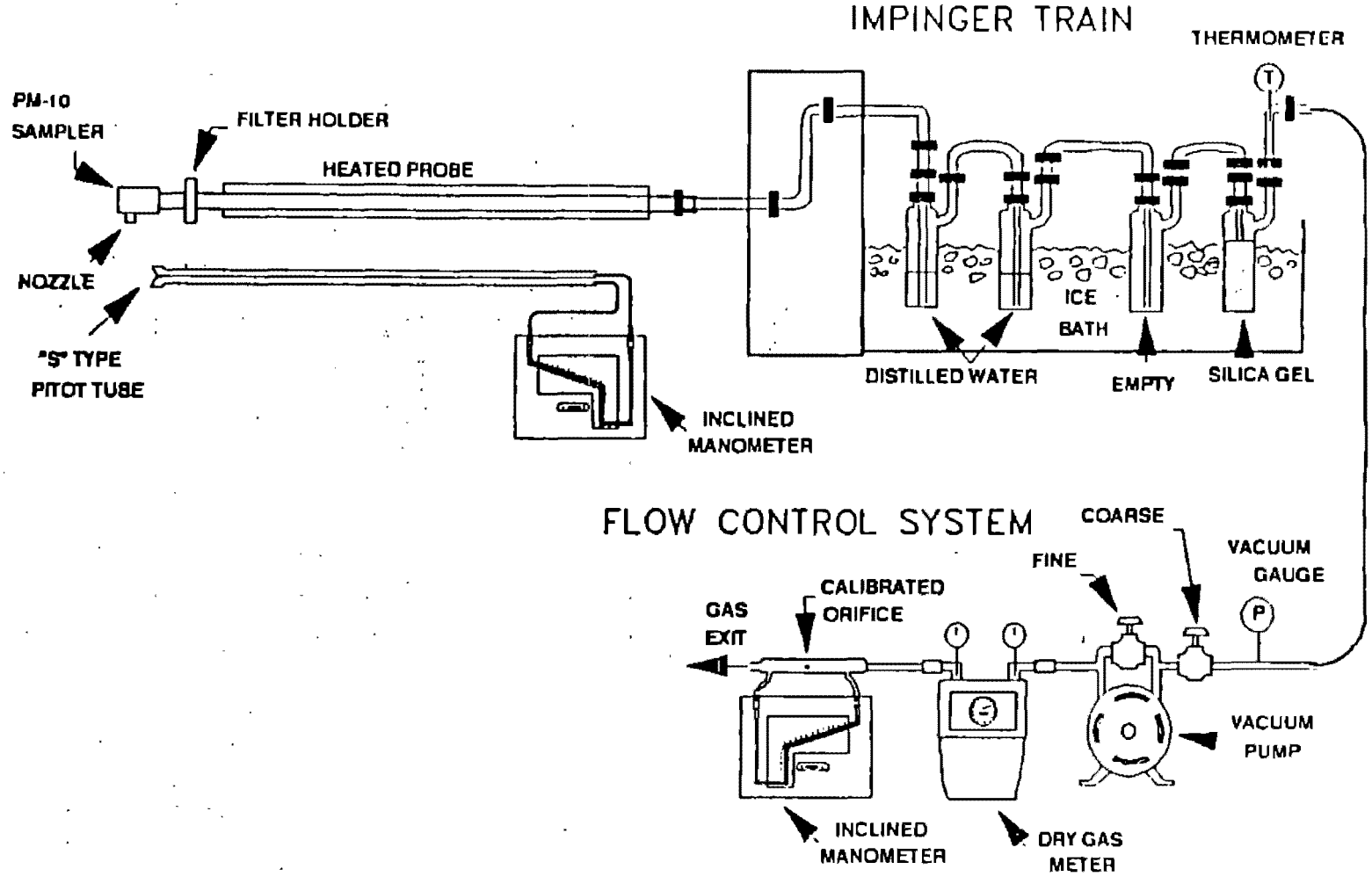


Figure 1. CSR Sampling Train

Barometric pressure, P_{bar}, in. Hg = _____
Stack static pressure, P_g, in. H_2O = _____
Average stack temperature, t_s, °F = _____
Meter temperature, t_m, °F = _____
Orifice $\Delta H_@$, in. H_2O = _____
Gas analysis:
$\%CO_2$ = _____
$\%O_2$ = _____
$\%N_2 + \%CO$ = _____
Fraction moisture content, B_{ws} = _____
Molecular weight of stack gas, dry basis:
$M_d = 0.44\ (\%CO_2) + 0.32\ (\%O_2) + 0.28\ (\%N_2 + \%CO)$ = _____ lb/lb mole
Molecular weight of stack gas, wet basis:
$M_s = M_d\ (1 - B_{ws}) - 18\ (B_{ws})$ = _____ lb/lb mole
Absolute stack pressure:

$$P_s = P_{bar} + \frac{P_g}{13.6} = _____ \text{ in. Hg}$$

Viscosity of stack gas:
$\mu_s = 152.418 + 0.2552\ t_s + 3.2355 \times 10^{-5}\ t_s^2 + 0.53147\ (\%O_2) - 74.143\ B_{ws}$ = _____ micropoise
Cyclone flow rate:

$$Q_s = 0.002837\ \mu_s \left[\frac{(t_s + 460)}{M_s\ P_s} \right]^{0.2949} = _____ \text{ ft}^3\text{/min}$$

Figure 4. Example worksheet 1, cyclone flow rate and ΔH.

Orifice pressure head (ΔH) needed for cyclone flow rate:

$$\Delta H = \left[\frac{Q_s\ (1 - B_{ws})\ P_s}{t_s + 460} \right]^2 \frac{t_m\ M_s\ 1.083\ \Delta H_@}{P_{bar}} = _____ \text{ in. } H_2O$$

Calculate ΔH for three temperatures:

t_s, °F			
ΔH, in. H_2O			

Stack viscosity, μ_s, micropoise = _____
Absolute stack pressure, P_s, in. Hg = _____
Average stack temperature, t_s, °F = _____
Meter temperature, t_m, °F = _____
Type S pitot coefficient, C_p = _____
Cyclone flow rate, ft³/min, Q_s = _____
Method 2 pitot coefficient, C_p' = _____
Molecular weight of stack gas, wet basis, M_s = _____
Nozzle diameter, D_n, in. = _____
Nozzle velocity:

$$v_n = \frac{3.056\ Q_s}{D_n^2} = _____ \text{ ft/sec}$$

Maximum and minimum velocities:

$$v_{min} = v_n \left[0.2457 + \left[0.3072 - \frac{0.2803\ Q_s^{1/3}\ \mu_s}{v_n^{1.5}} \right]^{1/3} \right] = _____ \text{ ft/sec}$$

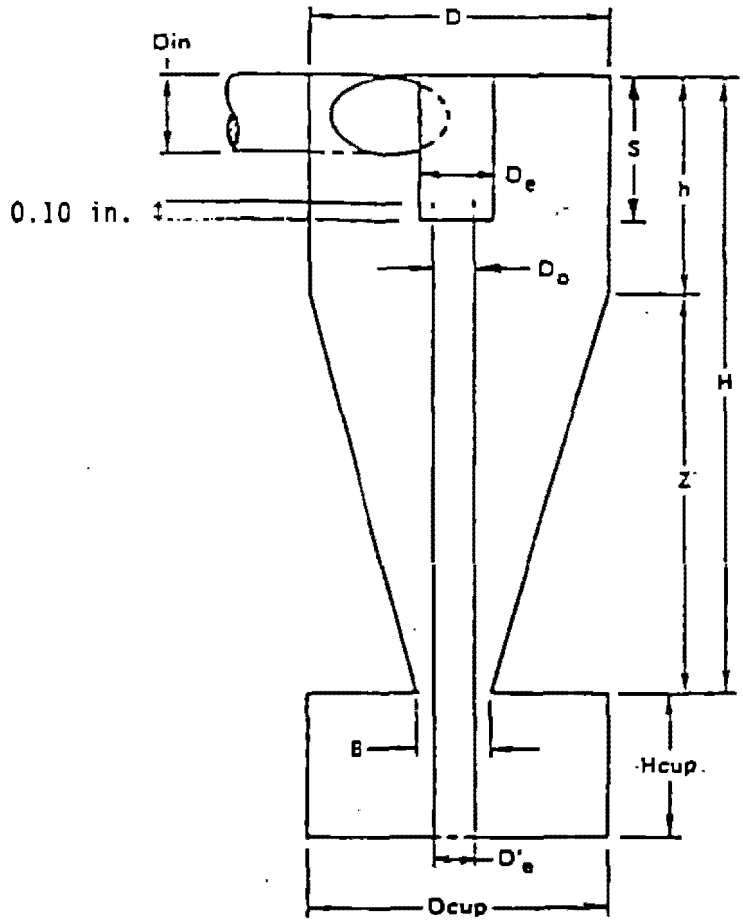
$$v_{max} = v_n \left[0.4457 + \left[0.5690 - \frac{0.2603\ Q_s^{1/3}\ \mu_s}{v_n^{1.5}} \right]^{1/3} \right] = _____ \text{ ft/sec}$$

Figure 5. Example worksheet 2, nozzle selection.

Maximum and minimum velocity head values:

$$\Delta p_{min} = 1.3636 \times 10^{-4} \frac{P_s\ M_s\ (v_{min})^2}{(t_s + 460)\ c_p^2} = _____ \text{ in. } H_2O$$

Cyclone Interior Dimensions



	Dimensions (±0.02 cm, ±0.01 in.)											
	Din	D	De	B	H	h	Z	S	Hcup	Dcup	D'_e	D_o
cm	1.27	4.47	1.50	1.88	6.95	2.24	4.71	1.57	2.25	4.45	1.02	1.24
inches	0.50	1.76	0.59	0.74	2.74	0.88	1.85	0.62	0.89	1.75	0.40	0.49

Figure 3. Cyclone design specifications.

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TABLE 2.—PARTICLE SIZES AND NOMINAL GAS VELOCITIES FOR EFFICIENCY

Particle size (μm)*	Target gas velocities (m/sec)		
	7 ± 1.0	15 ± 1.5	25 ± 2.5
5 ± 0.5			
7 ± 0.5			
10 ± 0.5			
14 ± 1.0			
20 ± 1.0			

* Mass median aerodynamic diameter.

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$$\Delta p_{max} = 1.3486 \times 10^{-4} \frac{P_s M_s (v_{max})^2}{(t_s + 460) C_p^2} = _____ \text{ in. } H_2O$$

Nozzle No.			
D_n, in.			
v_s, ft/sec			
v_{min}, ft/sec			
v_{max}, ft/sec			
Δp_{min}, in. H_2O			
Δp_{max}, in. H_2O			

Velocity traverse data:

$$\Delta p(\text{Method 201A}) = \Delta p(\text{Method 2}) \left[\frac{C_p}{C_p'} \right]^2$$

Total run time, minutes = _____
Number of traverse points = _____

$$t_1 = \left[\frac{\Delta p'_1}{\Delta p'_{avg}} \right] \times \frac{\text{(Total run time)}}{\text{(Number of points)}}$$

where:
t_1 = dwell time at first traverse point, minutes.
$\Delta p'_1$ = the velocity head at the first traverse point (from a previous traverse), in. H_2O.
$\Delta p'_{avg}$ = the square of the average square root of the Δp's (from a previous velocity traverse), in. H_2O.

At subsequent traverse points, measure the velocity Δp and calculate the dwell time by using the following equation:

$$t_n = \frac{t_1}{(\Delta p_1)^{1/2}} (\Delta p_n)^{1/2}, \; n = 2, 3, \ldots \text{total number of sampling points}$$

where:
t_n = dwell time at traverse point n, minutes.
Δp_n = measured velocity head at point n, in. H_2O.
Δp_1 = dwell time at first traverse point, minutes.

Figure 6. Example worksheet 3, dwell time.

Point No.	Port							
	Δp	t	Δp	t	Δp	t	Δp	t
1								
2								
3								
4								
5								
6								

Plant _____
Date _____
Run no. _____
Filter no. _____
Amount of liquid lost during transport _____
Acetone blank volume, ml _____
Acetone wash volume, ml (4) _____ (5) _____
Acetone blank conc., mg/mg (Equation 5–4, Method 5) _____
Acetone wash blank, mg (Equation 5–5, Method 5) _____

Container No.	Weight of PM_{10} (mg)		
	Final weight	Tare weight	Weight gain
1			
3			
Total			
Less acetone blank			
Weight of PM_{10}			

Figure 7. Method 201A analysis sheet.

TABLE 1.—PERFORMANCE SPECIFICATIONS FOR SOURCE PM_{10} CYCLONES AND NOZZLE COMBINATIONS

Parameter	Units	Specifications
1. Collection efficiency.	Percent	Such that collection efficiency falls within envelope specified by Section 5.2.6 and Figure 8.
2. Cyclone cut size (D_{50}).	μm	10 ± 1 μm aerodynamic diameter.

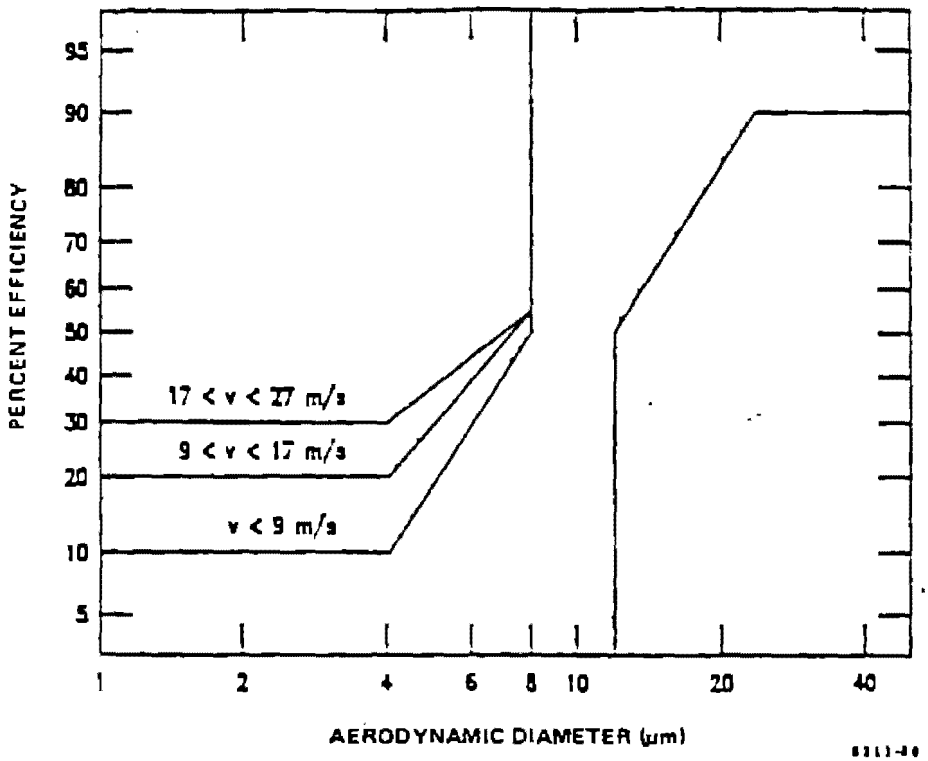

Figure 8. Efficiency envelope for the PM_{10} cyclone.

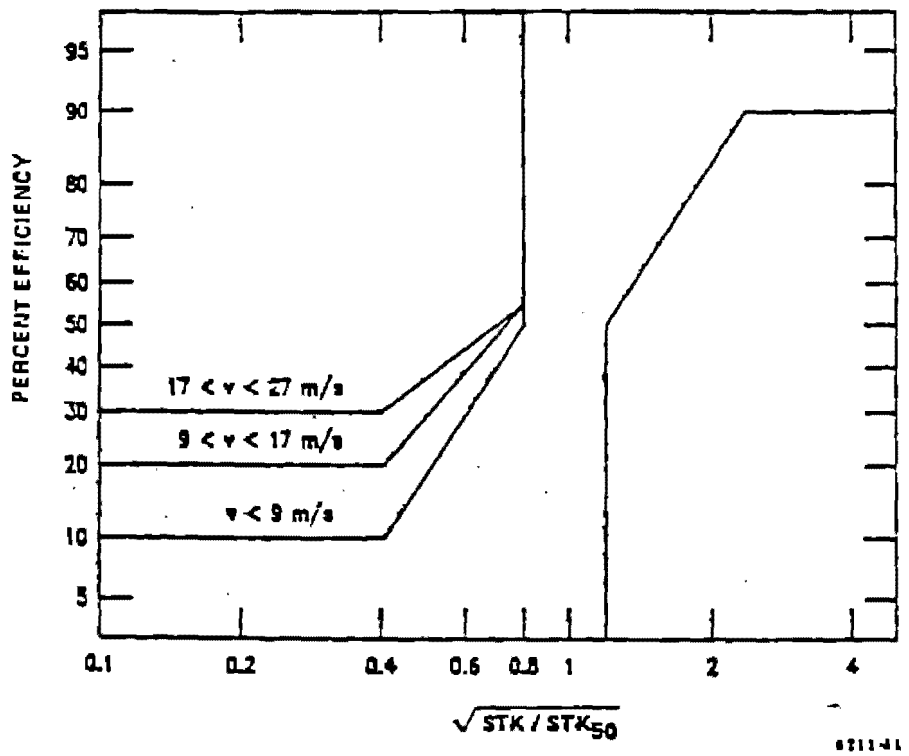

Figure 9. Efficiency envelope for first calibration stage.

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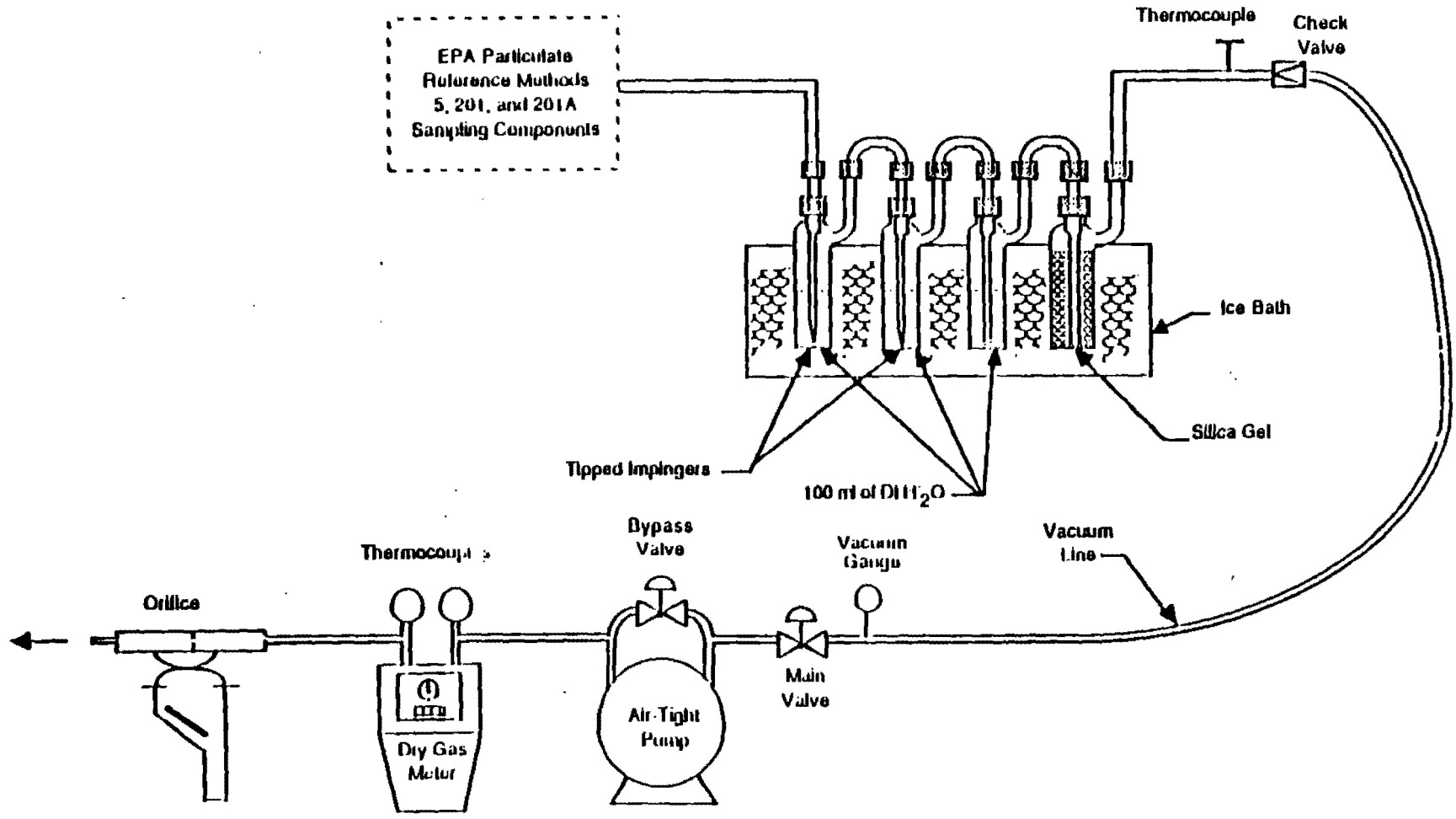

Figure 1. Schematic of condensible particulate sampling train.

Determination Of Condensible Emissions From Stationary Sources

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method applies to the determination of condensible particulate matter (CPM) emissions from stationary sources. It is intended to represent condensible matter as material that condenses after passing through an in-stack filter (Note: The filter catch can be analyzed according to Method 17 procedures). This method may be used in conjunction with Method 201 or 201A if the probes are glass lined. This method may also be modified to measure material that condenses at other temperatures by specifying the filter temperature.

1.2 Principle. The CPM is collected in the impinger portion of a Method 17 (Appendix A, 40 CFR Part 60) type sampling train. The impinger contents are immediately purged after the run with nitrogen (N_2) to remove dissolved sulfur dioxide (SO_2) gases from the impinger contents. The impinger solution is then extracted with methylene chloride ($MeCl_2$). The organic and aqueous fractions are then taken to dryness and the residues weighed. The total of both fractions represents the CPM.

2. PRECISION AND INTERFERENCE

2.1 Precision. The precisions based on method development tests at a wood waste burner and two coal-fired boilers are 13.0 $\pm$ 2.1 mg/m^3, 3.5 $\pm$ 1.1 mg/m^3, and 39.5 $\pm$ 9.0 mg/m^3, respectively.

2.2 Interference. Ammonia (e.g., in sources that use ammonia injection as a control technique) interferes by reacting with the hydrogen chloride (HCl) in the gas stream to form ammonium chloride (NH_4Cl) which would be measured as CPM. The sample may be analyzed for chloride and the equivalent amount of NH_4Cl can be subtracted from the CPM weight.

3. APPARATUS

3.1 Sampling Train. Same as in Method 17, Section 2.1, with the following exceptions noted below (see Figure 1). **Note:** Mention of trade names or specific products does not constitute endorsement by EPA.

3.1.1 The probe extension shall be glass-lined.

4.2.1 N_2 Gas. N_2 gas at delivery pressures high enough to provide a flow of 20 liters/min for 1 hour through the sampling train.

4.2.2 Methylene Chloride.

4.2.3 Water. Same as in Section 4.1.

4.3 Analysis. Same as in Method 5, Section 3.3, with the following additions:

4.3.1 Methylene Chloride.

4.3.2 Ammonium Hydroxide. Concentrated (14.8 M) NH_4OH.

4.3.3 Water. Same as in Section 4.1.

4.3.4 Phenolphthalein. The pH indicator solution, ~~1.0~~ .05 percent in ~~60~~ 50 percent alcohol.

5. PROCEDURE

5.1 Sampling. Same as in Method 5, Section 4.1, with the following exceptions:

5.1.1 Place 100 ml of water in the first three impingers.

5.1.2 The use of silicone grease in train assembly is not recommended. Teflon tape or similar means may be used to provide leak-free connections between glassware.

5.2 Sample Recovery. Same as in Method 17, Section 4.2 with the addition of a post-test N_2 purge and specific changes in handling of individual samples as described below.

5.2.1 Post-test N_2 Purge for Sources Emitting SO_2. (Note: This step is recommended, but is optional. When no or little SO_2 is present in the gas stream, i.e., the pH of the impinger solution is greater than 4.5, purging has been found to be unnecessary.) As soon as possible after the post-test leak check, detach the probe and filter from the impinger train. Leave the ice in the impinger box to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature below 20°C. With no flow of gas through the clean purge line and fittings, attach it to the input of the impinger train (see Figure 2). To avoid over- or under-pressurizing the impinger array, slowly commence the N_2 gas flow through the line while simultaneously opening the meter box pump valve(s). Adjust the pump bypass and N_2 delivery rates to obtain the following conditions: (1) 20 liters/min or $\Delta H_@$ and (2) an overflow rate through the rotameter of less than 2 liters/min. Condition (2) guarantees that the N_2 delivery system

3.1.2 A Teflon filter support shall be used.

3.1.3 Both the first and second impingers shall be of the Greenburg-Smith design with the standard tip.

3.1.4 All sampling train glassware shall be cleaned prior to the test with soap and tap water, water, and rinsed using tap water, water, acetone, and finally, $MeCl_2$. It is important to remove completely all silicone grease from areas that will be exposed to the $MeCl_2$ during sample recovery.

3.2 Sample Recovery. Same as in Method 5, Section 2.2, with the following additions:

3.2.1 N_2 Purge Line. Inert tubing and fittings capable of delivering 0 to 28 liters/min of N_2 gas to the impinger train from a standard gas cylinder (see Figure 2). Standard 0.95 cm (3/8-inch) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

3.2.2 Rotameter. Capable of measuring gas flow at 20 liters/min.

3.3 Analysis. The following equipment is necessary in addition to that listed in Method 5, Section 2.3:

3.3.1 Separatory Funnel. Glass, 1-liter.

3.3.2 Weighing Tins. 350-ml.

3.3.3 Drying Equipment. Hot plate and oven with temperature control.

~~3.3.4 Burette. 5-ml size with 0.01-ml graduations.~~

3.3.5 Pipets. 5-ml.

3.3.6 Ion Chromatograph. Same as in Method 5F, Section 2.1.6.

4. REAGENTS

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

4.1 Sampling. Same as in Method 5, Section 3.1, with the addition of deionized distilled water to conform to the American Society for Testing and Materials Specification D 1193-74, Type II.

4.2 Sample Recovery. Same as in Method 5, Section 3.2, with the following additions:

(Note: Do not use this aliquot to determine chlorides since the HCl will be evaporated during the first drying step; Section 8.2 details a procedure for this analysis.)

5.3.2.1 Extraction. Separate the organic fraction of the sample by adding the contents of Container No. 5 ($MeCl_2$) to the contents of Container No. 4 in a 1000-ml separatory funnel. After mixing, allow the aqueous and organic phases to fully separate, and drain off most of the organic/$MeCl_2$ phase. Then add 75 ml of $MeCl_2$ to the funnel, mix well, and drain off the lower organic phase. Repeat with another 75 ml of $MeCl_2$. This extraction should yield about 250 ml of organic extract. Each time, leave a small amount of the organic/$MeCl_2$ phase in the separatory funnel ensuring that no water is collected in the organic phase. Place the organic extract in a tared 350-ml weighing tin.

5.3.2.2 Organic Fraction Weight Determination (Organic Phase from Container Nos. 4 and 5). Evaporate the organic extract at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg.

5.3.2.3 Inorganic Fraction Weight Determination. Using a hot plate, or equivalent, evaporate the aqueous phase to approximately 50 ml; then evaporate to dryness in a 105°C oven. Redissolve the residue in 100 ml of water. Add five drops of phenolphthalein to this solution, then add concentrated (14.8 M) NH_4OH until the sample turns pink. Any excess NH_4OH will be evaporated during the drying step. Evaporate the sample to dryness in a 105°C oven, desiccate the sample for 24 hours, weigh to a constant weight, and record the results to the nearest 0.1 mg. (Note: The addition of NH_4OH is recommended, but is optional when no or little SO_2 is present in the gas stream, i.e., when the pH of the impinger solution is greater than 4.5, the addition of NH_4OH is not necessary.)

5.3.2.4 Analysis of Sulfate by IC to Determine Ammonium Ion (NH_4^+) Retained in the Sample. (Note: If NH_4OH is not added, omit this step.) Determine the amount of sulfate in the aliquot taken from Container No. 4 earlier as described in Method 5F (Appendix A, 40 CFR Part 60). Based on the IC $SO_4^=$ analysis of the aliquot, calculate the correction factor to delete the NH_4^+ retained in the sample and to add the combined water removed by the acid-base reaction (see Section 7.2).

5.3.3 Analysis of Water and $MeCl_2$ Blanks (Container Nos. 6 and 7). Analyze these sample blanks as described above in Sections 5.3.2.3 and 5.3.2.2, respectively. The sum of the values for the water blank and the $MeCl_2$ blank must be less than 2 mg or 5 percent of the mass of the CPM ($m_s + m_r$), whichever is greater. If the sum of the actual blank values is greater, then subtract 2 mg or 5 percent of the mass of the CPM, whichever is greater.

is operating at greater than ambient pressure and prevents that possibility of passing ambient air (rather than N_2) through the impingers. Continue the purge under these conditions for 1 hour, checking the rotameter and ΔH value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

5.2.2 Sample Handling.

5.2.2.1 Container Nos. 1, 2, and 3. If filter catch is to be determined, as detailed in Method 5, Section 4.2.

5.2.2.2 Container No. 4 (Impinger Contents). Measure the liquid in the first three impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas. Quantitatively transfer this liquid into a clean sample bottle (glass or plastic); rinse each impinger and the connecting glassware, including probe extension, twice with water, recover the rinse water and add it to the same sample bottle. Mark the liquid level on the bottle.

5.2.2.3 Container No. 5 ($MeCl_2$ Rinse). Follow the water rinses of each impinger and the connecting glassware, including the probe extension with two rinses of $MeCl_2$; save the rinse products in a clean, glass sample jar. Mark the liquid level on the jar.

5.2.2.4 Container No. 6 (Water Blank). Once during each field test, place 500 ml of water in a separate sample container.

5.2.2.5 Container No. 7 ($MeCl_2$ Blank). Once during each field test, place in a separate glass sample jar a volume of $MeCl_2$ approximately equivalent to the volume used to conduct the $MeCl_2$ rinse of the impingers.

5.2.2.6 Container No. 8 (Acetone Blank). As described in Method 5, Section 4.2.

5.3 Analysis. Record the data required on a sheet such as the one shown in Figure 3. Handle each sample container as follows:

5.3.1 Container Nos. 1, 2, and 3. If filter catch is analyzed, as detailed in Method 5, Section 4.3.

5.3.2 Container Nos. 4 and 5. Note the level of liquid in the containers and confirm on the analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container No. 4 either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Remove a 5-ml aliquot and set aside for later ion chromatographic (IC) analysis of sulfates.

7.3 Mass of Inorganic CPM.

$$m_{rc} = m_r \left[\frac{V_{ic}}{V_{ic} - V_b}\right] - m_b + m_c \qquad \text{Eq. 2}$$

7.4 Concentration of CPM.

$$c_{cpm} = \frac{m_o + m_i}{V_{m\ std}} \qquad \text{Eq. 3}$$

8. ALTERNATIVE PROCEDURES

8.1 Determination of NH_4^+ Retained in Sample by Titration.

8.1.1 An alternative procedure to determine the amount of NH_4^+ added to the inorganic fraction by titration may be used. After dissolving the inorganic residue in 100 ml of water, titrate the solution with 0.1 N NH_4OH to a pH of 7.0, as indicated by a pH meter. The 0.1 N NH_4OH is made as follows: Add 7 ml of concentrated (14.8 M) NH_4OH to 1 liter of water. Standardize against standardized 0.1 N H_2SO_4 and calculate the exact normality using a procedure parallel to that described in Section 5.5 of Method 6 (Appendix A, 40 CFR Part 60). Alternatively, purchase 0.1 N NH_4OH that has been standardized against a National Institute of Standards and Technology reference material.

8.1.2 Calculate the concentration of $SO_4^=$ in the sample using the following equation.

$$C_{SO4} = \frac{48.03\ V_t\ N}{100} \qquad \text{Eq. 4}$$

where:

N = Normality of the NH_4OH, mg/ml.

V_t = Volume of NH_4OH titrant, ml.

48.03 = mg/meq.

100 = Volume of solution, ml.

8.1.3 Calculate the CPM as described in Section 7.

5.3.4 Analysis of Acetone Blank (Container No. 8). Same as in Method 5, Section 4.3.

6. CALIBRATION

Same as in Method 5, Section 5, except calibrate the IC according to the procedures in Method 5F, Section 5.

7. CALCULATIONS

Same as in Method 5, Section 6, with the following additions:

7.1 Nomenclature. Same as in Method 5, Section 6.1 with the following additions.

c_{cpm} = Concentration of the CPM in the stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

C_{SO4} = Concentration of $SO_4^=$ in the sample, mg/ml.

m_b = Sum of the mass of the water and $MeCl_2$ blanks, mg.

m_c = Mass of the NH_4^- added to sample to form ammonium sulfate, mg.

m_i = Mass of inorganic CPM matter, mg.

m_o = Mass of organic CPM, mg.

m_r = Mass of dried sample from inorganic fraction, mg.

m_{rc} = Mass of dried sample from inorganic fraction corrected for volume of aliquot taken for IC analysis, mg.

V_b = Volume of aliquot taken for IC analysis, ml.

V_{ic} = Volume of impinger contents sample, ml.

7.2 Correction for NH_4^- and H_2O. Calculate the correction factor to delete the NH_4^- retained in the sample and to add the combined water removed by the acid-base reaction based on the IC $SO_4^=$.

$$m_c = K\ C_{SO4}\ V_{ic} \qquad \text{Eq. 1}$$

where:

K = 0.020502

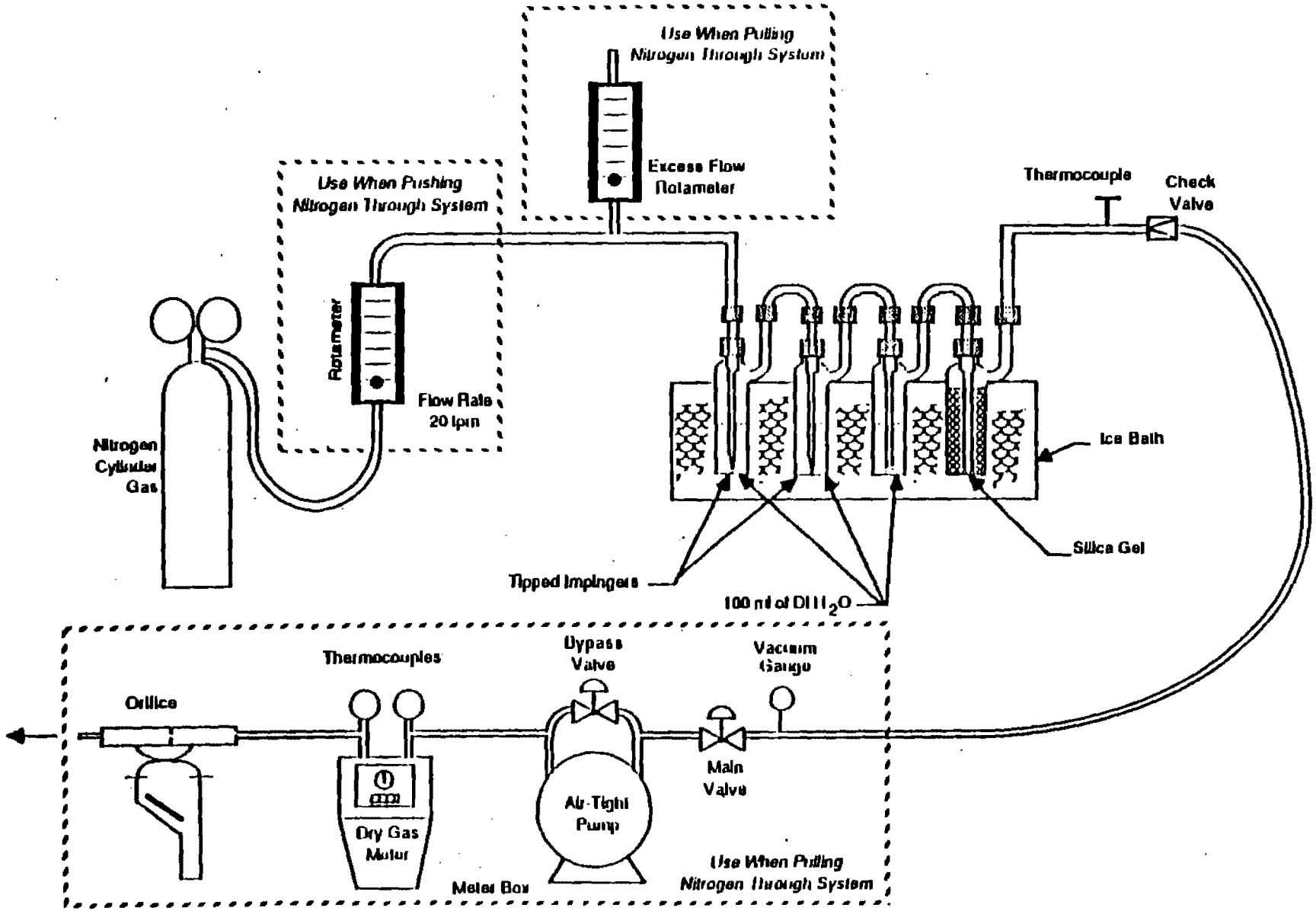

Figure 2. Schematic of post-test nitrogen purge system.

8.2 Analysis of Chlorides by IC. At the conclusion of the final weighing as described in Section 5.3.2.3, redissolve the inorganic fraction in 100 ml of water. Analyze an aliquot of the redissolved sample for chlorides by IC using techniques similar to those described in Method 5F for sulfates. Previous drying of the sample should have removed all HCl. Therefore, the remaining chlorides measured by IC can be assumed to be NH_4Cl, and this weight can be subtracted from the weight determined for CPM.

8.3 Air Purge to Remove SO_2 from Impinger Contents. As an alternative to the post-test N_2 purge described in Section 5.2.1, the tester may opt to conduct the post-test purge with air at 20 liter/min. Note: The use of an air purge is not as effective as a N_2 purge.

9. BIBLIOGRAPHY

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Moisture Determination

Volume or weight of liquid in impingers _____ ml or g
Weight of moisture in silica gel _____ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport _____ ml
Final volume _____ ml
pH of sample prior to analysis _____
Addition of NH_4OH required? _____
Sample extracted 2X with 75 ml $MeCl_2$? _____

For Titration of Sulfate

Normality of NH_4OH _____ N
Volume of sample titrated _____ ml
Volume of titrant _____ ml

Sample Analysis

Container number	Weight of Condensible Particulate, mg		
	Final Weight	Tare Weight	Weight Gain
4 (Inorganic) 4 & 5 (Organic)			

Total	_____
Less Blank	_____
Weight of Condensible Particulate	_____

Figure 3. Analytical data sheet.

APPENDIX J.3

ALDEHYDES

3.5 Sampling for Aldehyde and Ketone Emissions from Stationary Sources (Method 0011)

3.5.1 Scope and Application

This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of formaldehyde, CAS Registry number 50-00-0, and possibly other aldehydes and ketones from stationary sources as specified in the regulations. The methodology has been applied specifically to formaldehyde, however, many laboratories have extended the application to other aldehydes and ketones. Compounds derivatized with 2,4-dinitrophenyl-hydrazine can be detected as low as 6.4×10^{-8} lbs/cu ft (1.8 ppbv) in stack gas over a 1 hr sampling period, sampling approximately 45 cu ft.

3.5.2 Summary of Method

3.5.2.1 Gaseous and particulate pollutants are withdrawn isokinetically from an emission source and are collected in aqueous acidic 2,4-dinitrophenyl-hydrazine. Formaldehyde present in the emissions reacts with the 2,4-dinitrophenyl-hydrazine to form the formaldehyde dinitrophenylhydrazone derivative. The dinitrophenylhydrazone derivative is extracted, solvent-exchanged, concentrated, and then analyzed by high performance liquid chromatography.

3.5.3 Interferences

3.5.3.1 A decomposition product of 2,4-dinitrophenyl-hydrazine, 2,4-dinitroaniline, can be an analytical interferant if concentrations are high. 2,4-Dinitroaniline can coelute with 2,4-dinitrophenylhydrazone of formaldehyde under high performance liquid chromatography conditions, which may be used for the analysis. High concentrations of highly-oxygenated compounds, especially acetone, that have the same retention time or nearly the same retention time as the dinitrophenylhydrazone of formaldehyde, and that also absorb at 360 nm, will interfere with the analysis.

Formaldehyde, acetone, and 2,4-dinitroaniline contamination of the aqueous acidic 2,4-dinitrophenyl-hydrazine (DNPH) reagent is frequently encountered. The reagent must be prepared within five days of use in the field and must be stored in an uncontaminated environment both before and after sampling in order to minimize blank problems. Some concentration of acetone contamination is unavoidable, because acetone is ubiquitous in laboratory and field operations. However, the acetone contamination must be minimized.

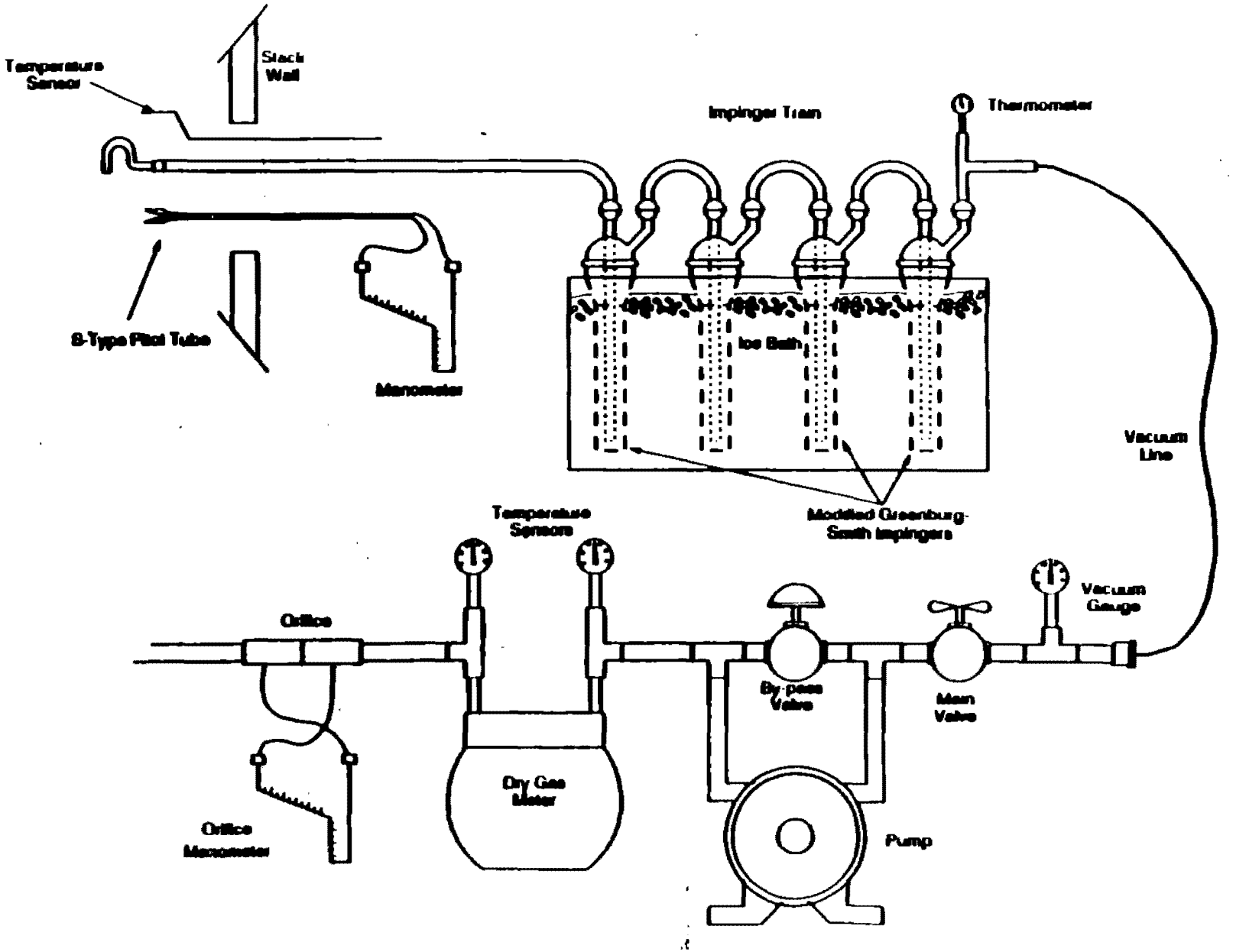
3.5.4 Apparatus and Materials

3.5.4.1 A schematic of the sampling train is shown in Figure 3.5-1. This sampling train configuration is adapted from EPA Method 4 procedures. The sampling train consists of the following components: Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment.

3.5.4.1.1 Probe Nozzle: Quartz or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant inner diameter. The nozzle shall be buttonhook or elbow design. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.15 cm(1/16 in), e.g., 0.32 to 1.27 cm (1/8 to 1/2 in), of larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Section 3.5.8.1

3.5.4.1.2 Probe Liner: Borosilicate glass or quartz shall be used for the probe liner. The tester should not allow the temperature in the probe to exceed 120 ± 14°C (248 ± 25°F).

3.5.4.1.3 Pitot Tube: The Pitot tube shall be Type S, as described in Section 2.1 of EPA Method 2, or any other appropriate device. The pitot tube shall be attached to the probe to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plan (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.



Formaldehyde Sampling Train

3.5.4.1.4 Differential Pressure Gauge: The differential pressure gauge shall be an inclined manometer or equivalent device as described in Section 2.2 of EPA Method 2. One manometer shall be used for velocity-head reading and the other for orifice differential pressure readings.

3.5.4.1.5 Impingers: The sampling train requires a minimum of four impingers, connected as shown in Figure 3.5-1, with ground glass (or equivalent) vacuum-tight fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm inside diameter (1/2 in) glass tube extending to 1.3 cm (1/2 in) from the bottom of the flask. For the second impinger, use a Greenburg-Smith impinger with the standard tip. Place a thermometer capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger for monitoring purposes.

3.5.4.1.6 Metering System: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment as shown in Figure 3.5-1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems may be used which are capable of maintaining sample volumes to within 2%. The metering system may be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates.

3.5.4.1.7 Barometer: The barometer may be mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service Station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in Hg) per 30 m (100 ft) elevation increases (vice versa for elevation decrease).

3.5.4.1.8 Gas Density Determination Equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.3 of EPA Method 2), and

gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot openings (see EPA Method 2, Figure 2-7). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

3.5.4.2 Sample Recovery

3.5.4.2.1 Probe Liner: Probe nozzle and brushes; Teflon bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel, Teflon, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the impingers.

3.5.4.2.2 Wash Bottles: Three wash bottles are required. Teflon or glass wash bottles are recommended; polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

3.5.4.2.3 Graduate Cylinder and/or Balance: A graduated cylinder or balance is required to measure condensed water to the nearest 1 ml or 1 g. Graduated cylinders shall have division not >2 ml. Laboratory balances capable of weighing to ± 0.5 g are required.

3.5.4.2.4 Amber Glass Storage Containers: One-liter wide-mouth amber flint glass bottles with Teflon-lined caps are required to store impinger water samples. The bottles must be sealed with Teflon tape.

3.5.4.2.5 Rubber Policeman and Funnel: A rubber policeman and funnel are required to aid in the transfer of material into and out of containers in the field.

3.5.4.3 Reagent Preparation

3.5.4.3.1 Bottles/Caps: Amber 1- or 4-L bottles with Teflon-lined caps are required for storing cleaned DNPH solution. Additional 4-L bottles are required to collect waste organic solvents.

3.5.4.3.2 Large Glass Container: At least one large glass (8 to 16 L) is required for mixing the aqueous acidic DNPH solution.

3.5.4.3.3 Stir Plate/Large Stir Bars/Stir Bar Retriever: a magnetic stir plate and large stir bar are required for the mixing of aqueous acidic DNPH solution. A stir bar retriever is needed for removing the stir bar from the large container holding the DNPH solution.

3.5.4.3.4 Buchner Filter/Filter Flask/Filter Paper: A large filter flask (2-4 L) with a buchner filter, appropriate rubber stopper, filter paper, and connecting tubing are required for filtering the aqueous acidic DNPH solution prior to cleaning.

3.5.4.3.5 Separatory Funnel: At least one large separatory funnel (2 L) is required for cleaning the DNPH prior to use.

3.5.4.3.6 Beakers: Beakers (150 ml, 250 ml, and 400 ml) are useful for holding/measuring organic liquids when cleaning the aqueous acidic DNPH solution and for weighing DNPH crystals.

3.5.4.3.7 Funnels: At lease one large funnel is needed for pouring the aqueous acidic DNPH into the separator funnel.

3.5.4.3.8 Graduated Cylinders: At least one large graduated cylinder (1 to 2 L) is required for measuring organic-free reagent water and acid when preparing the DNPH solution.

3.5.4.3.9 Top-Loading Balance: A one-place top loading balance is needed for weighing out the DNPH crystals used to prepare the aqueous acidic DNPH solution.

3.5.4.3.10 Spatulas: Spatulas are needed for weighing out DNPH when preparing the aqueous DNPH solution.

3.5.4.4 Crushed Ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending upon ambient temperature. Samples which have been taken must be stored and shipped cold; sufficient ice for this purpose must be allowed.

3.5.5 **Reagents**

3.5.5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.5.5.2 Organic-free reagent water: All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

3.5.5.3 Silica Gel: Silica gel shall be indicating type, 6-16 mesh. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

3.5.5.4 2,4-dinitrophenylhydrazine (DNPH), [2,4-$(O_2N)_2C_6H_3$]$NHNH_2$ - The quantity of water may vary from 10 to 30%.

3.5.5.4.1 The 2,4-dinitrophenylhydrazine reagent must be prepared in the laboratory within five days of sampling use in the field. Preparation of DNPH can also be done in the field, with consideration of appropriate procedures required for safe handling of solvent in the field. When a container of prepared DNPH reagent is opened in the field, the contents of the opened container should be used within 48 hours. All laboratory glassware must be washed with detergent and water and rinsed with water, methanol, and methylene chloride prior to use.

NOTE: DNPH crystals or DNPH solution should be handled with plastic gloves at all times with prompt and extensive use of running water in case of skin exposure.

3.5.5.4.2 Preparation of Aqueous Acidic DNPH Derivatizing Reagent: Each batch of DNPH reagent should be prepared and purified within five days of sampling, according to the procedures described below.

NOTE: Reagent bottles for storage of cleaned DNPH derivatizing solution must be rinsed with acetonitrile and dried before use. Baked glassware is not essential for preparation of DNPH reagent. The glassware must not be rinsed with acetone or an unacceptable concentration of acetone contamination will be introduced. If field preparation of DNPH is performed, caution must be exercised in avoiding acetone contamination.

3.5.5.4.2.1 Place an 8 L container under a fume hood on a magnetic stirrer. Add a large stir bar and fill the container half full of organic-free reagent water. Save the empty bottle from the organic-free reagent water. Start the stirring bar and adjust the stir rate to be as fast as possible. Using a graduated cylinder, measure 1.4 ml of concentrated hydrochloric acid. Slowly pour the acid into the stirring water. Fumes may be generated and the water may become warm. Weight the DNPH crystals on a

one-place balance (see Table 3.5-1 for approximate amounts) and add to the stirring acid solution. Fill the 8-L container to the 8-L mark with organic-free reagent water and stir overnight. If all of the DNPH crystals have dissolved overnight, add additional DNPH and stir for two more hours. Continue the process of adding DNPH with additional stirring until a saturated solution has been formed. Filter the DNPH solution using vacuum filtration. Gravity filtration may be used, but a much longer time is required. Store the filtered solution in an amber bottle at room temperature.

3.5.5.4.2.2 Within five days of proposed use, place about 1.6 L of the DNPH reagent in a 2-L separatory funnel. Add approximately 200 ml of methylene chloride and stopper the funnel. Wrap the stopper of the funnel with paper towels to absorb any leakage. Invert and vent the funnel. Then shake vigorously for 3 minutes. Initially, the funnel should be vented frequently (every 10 -15 sec). After the layers have separated, discard the lower (organic) layer.

3.5.5.4.2.3 Extract the DNPH a second time with methylene chloride and finally with cyclohexane. When the cyclohexane layer has separated from the DNPH reagent, the cyclohexane layer will be the top layer in the separatory funnel. Drain the lower layer (the cleaned extract DNPH reagent solution) into an amber bottle that has been rinsed with acetonitrile and allowed to dry.

3.5.5.4.3 Quality Control: Take two aliquots of the extracted DNPH reagent. The size of the aliquots is dependent upon the exact sampling procedure used, but 100 ml is reasonably representative. To ensure that the background in the reagent is acceptable for field use, analyze one aliquot of the reagent according to the procedure of Method 8315. Save the other aliquot of aqueous acidic DNPH for use as a method blank when the analysis is performed.

3.5.5.4.4 Shipment to the Field: Tightly cap the bottle containing extracted DNPH reagent using a Teflon-lined cap. Seal the bottle with Teflon

Table 3.5-1

APPROXMIATE AMOUNT OF CRYSTALLINE DNPH USED
TO PREPARE A SATURATED SOLUTION

Amount of Moisture in DNPH	Weight Required per 8 L of Solution
10 weight percent	31 g
15 weight percent	33 g
30 weight percent	40 g

Table 3.5-2

INSTRUMENT DETECTION LIMITS AND REAGENT CAPACITY
FOR FORMALDEHYDE ANALYSIS[1]

Analyte	Detection Limit, ppbv[2]	Reagent Capacity, ppmv
Formaldehyde	1.8	66
Acetaldehyde	1.7	70
Acrolein	1.5	75
Acetone/Propionaldehyde	1.5	75
Butyraldehyde	1.5	79
Methyl ethyl ketone	1.5	79
Valeraldehyde	1.5	84
Isovaleraldehyde	1.4	84
Hexaldehyde	1.3	88
Benzaldehyde	1.4	84
o-/m-/p-Tolualdehyde	1.3	89
Dimethylbenzaldehyde	1.2	93

[1]Oxygenated compounds in addition to formaldehyde are included for comparison with formaldehyde; extension of the methodology to other compounds is possible.

[2]Detection limits are determined in solvent. These values therefore represent the optimum capability of the methodology.

tape. After the bottle is labeled, the bottle may be placed in a friction-top can (paint can or equivalent) containing a 1-2 inch layer of granulated charcoal and stored at ambient temperature until use.

3.5.5.4.4.1 If the DNPH reagent has passed the Quality Control criteria, the reagent may be packaged to meet necessary shipping requirements and sent to the sampling area. If the Quality Control criteria are not met, the reagent solution may be re-extracted or the solution may be re-prepared and the extraction sequence repeated.

3.5.5.4.4.2 If the DNPH reagent is not used in the field within five days of extraction, an aliquot may be taken and analyzed as described in Method 0011A. If the reagent meets the Quality Control requirements, the reagent may be used. If the reagent does not meet the Quality Control requirements, the reagent must be discarded and new reagent must be prepared and tested.

3.5.5.4.5 Calculation of Acceptable Concentrations of Impurities in DNPH Reagent: The acceptable impurity concentration (AIC, µg/ml) is calculated from the expected analyte concentration in the sampled gas (EAC, ppbv), the volume of air that will be sampled at standard conditions (SVOL, L), the formula weight of the analyte (FW, g/mol), and the volume of DNPH reagent that will be used in the impingers (RVOL, ml):

$$AIC = 0.1 \times [EAC \times SVOL \times FW/22.4 \times (FW + 180)/FW](RVOL \times 1{,}000)$$

where:

0.1 is the acceptable contaminant concentration,
22.4 is a factor relating ppbv to g/L,
180 is a facto relating underivatized to derivatized analyte
1,000 is a unit conversion factor.

3.5.5.4.6 Disposal of Excess DNPH Reagent: Excess DNPH reagent may be returned to the laboratory and recycled or treated as aqueous waste for

disposal purposes. 2,4-dinitrophenylhydrazine is a flammable solid when dry, so water should not be evaporated from the solution of the reagent.

3.5.5.5 Field Spike Standard Preparation: To prepare a formaldehyde field spiking standard at 4.01 mg/ml, use a 500 µl syringe to transfer 0.5 ml to 37% by weight of formaldehyde (401 mg/ml) to a 50 ml volumetric flask containing approximately 50 ml of methanol. Dilute to 50 ml with methanol.

3.5.5.6 Hydrochloric Acid, HCL: Reagent grade hydrochloric acid (approximately 12N) is required for acidifying the aqueous DNPH solution.

3.5.5.7 Methylene Chloride, CH_2Cl_2: Methylene chloride (suitable for residue and pesticide analysis, GC/MS, HPLC, GC, Spectrophotometry or equivalent) is required for cleaning the aqueous acidic DNPH solution, rinsing glassware, and recovery of sample trains.

3.5.5.8 Cyclohexane, C_6H_{12}: Cyclohexane (HPLC grade) is required for cleaning the aqueous acidic DNPH solution.

NOTE: Do not use spectroanalyzed grades of cyclohexane if this sampling methodology is extended to aldehydes and ketones with four or more carbon atoms.

3.5.5.9 Methanol, CH_3OH: Methanol (HPLC grade or equivalent) is required for rinsing glassware.

3.5.5.10 Acetonitrile, CH_3CN: Acetonitrile (HPLC grade or equivalent) is required for rinsing glassware.

3.5.5.11 Formaldehyde, HCHO: Analytical grade or equivalent formaldehyde is required for preparation of standards. If other aldehydes or ketones are used, analytical grade or equivalent is required.

3.5.6 Sample Collection, Preservation, and Handling

3.5.6.1 Because of the complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

3.5.6.2 Laboratory Preparation:

3.5.6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified.

3.5.6.2.2 Weigh several 200 to 300 g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

3.5.6.3 Preliminary Field Determinations:

3.5.6.3.1 Select the sampling site and the minimum number of sampling point according to EPA Method 1 or other relevant criteria. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. A leak-check of the pitot lines according to EPA Method 2, Section 3.1, must be performed. Determine the stack gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

3.5.6.3.2 Select a nozzle size based on the range of velocity heads so that is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 L/min (1.0 cfm). During the run, do not

change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2. of EPA Method 2).

3.5.6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

3.5.6.3.4 A minimum of 45 ft^3 of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of formaldehyde from incineration systems (45 ft^3 is equivalent to one hour of sampling at 0.75 dscf). Additional sample volume shall be collected as necessitated by the capacity of the DNPH reagent and analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.

3.5.6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus 0.5 min.

3.5.6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-volume samples. In these cases, careful documentation must be maintained in order to allow accurate calculation of concentrations.

3.5.6.4 Preparation of Collection Train:

3.5.6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.

3.5.6.4.2 Place 100 ml of cleaned DNPH solution in each of the first two impingers, and leave the third impinger empty. If additional capacity is required for high expected concentrations of formaldehyde in the stack gas, 200 ml of DNPH per impinger may be used or additional impingers may be used for sampling. Transfer approximately 200 to 300 g of pre-weighed silica gel from its container to the fourth impinger. Care should be taken to ensure that the silica gel is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place or later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

3.5.6.4.3 With a glass or quartz liner, install the selected nozzle using a Viton-A O-ring with stack temperatures are <260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connection systems utilizing either 316 stainless steel or Teflon ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

3.5.6.4.4 Assemble the train as shown in Figure 3.5-1. During assembly, do not use any silicone grease on ground-glass joints upstream of the impingers. Use Teflon tape, if required. A very light coating of silicone grease may be used on ground-glass joints downstream of the impingers, but the silicone grease should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone grease contamination. If necessary, Teflon tape may be used to seal leaks. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperatures.

3.5.6.4.5 Place crushed ice all around the impingers.

3.5.6.4.6 Turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize.

3.5.6.5 Leak-Check Procedures:

3.5.6.5.1 Pre-test Leak Check

3.5.6.5.1.1 After the sampling train has been assembled, turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize. If a Viton-A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 381 mm Hg (15 in Hg) vacuum.

NOTE: A lower vacuum may be used, provided that the lower vacuum is not exceeded during the test.

3.5.6.5.1.2 If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first attaching a carbon-filled leak check impinger to the inlet and then plugging the inlet and pulling a 381 mm Hg (15 in Hg) vacuum. (A lower vacuum any be used if this lower vacuum is not exceeded during the test.) Next connect the probe to the train and leak-check at about 25 mm Hg (1 in Hg) vacuum. Alternatively, leak-check the probe with the rest of the sampling train in one step at 381 mm Hg (15 in Hg) vacuum. Leakage rates in excess of (a) 4% of the average sampling rate or (b) >0.00057 m^3/min (0.02 cfm), are unacceptable.

3.5.6.5.1.3 The following leak check instructions for the sampling train described in ADPT-0576 and APTD-0581 may be helpful. Start the pump with the fine-adjust valve fully open and coarse-valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do <u>not</u> reverse direction of the fine-adjust valve, as liquid will back up into the train. If the desired vacuum is exceeded, either perform the leak check at this higher vacuum or end the leak check, as shown below, and start over.

3.5.6.5.1.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe. When the vacuum drops to 127 mm (5 in) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed to prevent the liquid in the impingers from being forced backward in the sampling line and silica gel from being entrained backward into the third impinger.

3.5.6.5.2 Leak Checks During Sampling Run:

3.5.6.5.2.1 If, during the sampling run, a component change (i.e., impinger) becomes necessary, a leak check shall be conducted immediately after the interruption of sampling and before the change is made. The leak check shall be done according to the procedure described in Section 3.5.6.5.1, except that is shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm or 4% of the average sampling rate (whichever is less), the results are acceptable. If a higher leakage rate is obtained, the tester must void the sampling run.

NOTE: Any correction of the sample volume by calculation reduces the integrity of the pollutant concentration data generated and must be avoided.

3.5.6.5.2.2 Immediately after a component change and before sampling is reinitiated, a leak check similar to a pre-test leak check must also be conducted.

3.5.6.5.3 Post-test Leak Check:

3.5.6.5.3.1 A leak check is mandatory at the conclusion of each sampling run. The leak check shall be done with the same procedures as the pre-test leak check, except that the post-test leak check shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057

m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable. If, however, a higher leakage rate is obtained, the tester shall record the leakage rate and void the sampling run.

3.5.6.6 Sampling Train Operation:

3.5.6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, below 20 L/min (1.0 cfm). Maintain a temperature around the probe of 120°C (248° ± 25°F).

3.5.6.6.2 For each run, record the data on a data sheet such as the one shown in Figure 3.5-2. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 2 at least once at each sample point during each time increment and additional readings when significant adjustments 20% variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse

3.5.6.6.3 Clean the stack access ports prior to the test run to eliminate the change of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot tube coefficient is 0.84 ± 0.02 and the stack gas equivalent density (dry molecular weight) is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If the stack gas molecular weight and the pitot tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps are taken to compensate for the deviations.

Plant _____

Location _____

Operator _____

Date _____

Run No. _____

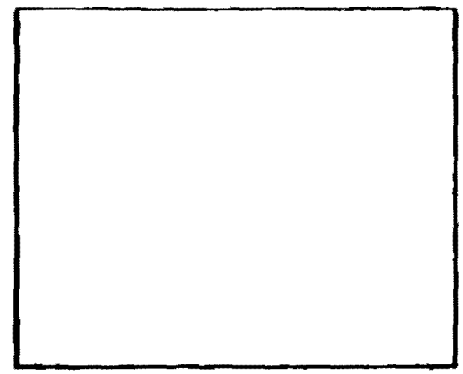
Sample Box No. _____

Meter Box No. _____

Meter H@ _____

C Factor _____

Pitot Tube Coefficient C_p _____



Schematic of Stack Cross Section

Ambient Temperature _____

Barometric Pressure _____

Assumed Moisture % _____

Probe Length, m(ft) _____

Nozzle Identification No. _____

Average Calibrated Nozzle Diameter, cm (in) _____

Probe Heating Setting _____

Leak Rate, m^3/min. (cfm) _____

Probe Liner Material _____

Static Pressure, mm Hg (in. Hg) _____

Filter No. _____

Traverse Point Number	Sampling Time (θ) Min.	Vacuum mm Hg (in. Hg)	Stack Temperature (Ts) °C (°F)	Velocity Head (P_s) mm (in) H_2O	Pressure Differential Across Orifice Meter mm (H_2O) (in H_2O)	Gas Sample Volume m^3 (ft^3)	Gas Sample Temp. at Dry Gas Meter		Filter Holder Temperature °C(°F)	Temperature of Gas Leaving Last Impinger °C(°F)
							Inlet °C(°F)	Outlet °C(°F)		
Total							Avg	Avg		
Average										

Figure 1.5-2 Field Data Sheet

3.5.6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack in order to prevent liquid from backing up through the train. If necessary, the pump may be turned on with the coarse-adjust valve closed.

3.5.6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent unrepresentative dilution of the gas stream.

3.5.6.6.6 Traverse the stack cross section, as required by EPA Method 1, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

3.5.6.6.7 During the test run, make periodic adjustments to keep the temperature around the probe at the proper levels. Add more ice and, if necessary, salt, to maintain a temperature of <20°C (68°F) at the silica gel outlet. Also, periodically check the level and zero of the manometer.

3.5.6.6.8 A single train shall be used for the entire sampling run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. An additional train or additional trains may also be used for sampling when the capacity of a single train is exceeded.

3.5.6.6.9 When two or more trains are used, separate analyses of components from each train shall be performed. If multiple trains have been used because the capacity of a single train would be exceeded, first impingers from each train may be combined, and second impingers from each train may be combined.

3.5.6.6.10 At the end of the sampling run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak check. Also, leak check the pitot lines as described in EPA Method 2. The lines must pass this leak check in order to validate the velocity-head data.

3.5.6.6.11 Calculate percent isokineticity (see Method 2) to determine whether the run was valid or another test should be made.

3.5.7 Sample Recovery

3.5.7.1 Preparation:

3.5.7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be handled safely, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling because a vacuum will be created, drawing liquid from the impingers back through the sampling train.

3.5.7.1.2 Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet, being careful not to lose any condensate that might be present. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used, let any condensed water or liquid drain into the impingers. Cap off any open impinger inlets and outlets. Ground glass stoppers, Teflon caps or caps of other inert materials may be used to seal all openings.

3.5.7.1.3 Transfer the probe and impinger assembly to an area that is clean and protected from wind so that the chances of contaminating or losing the sample are minimized.

3.5.7.1.4 Inspect the train before and during disassembly, and note any abnormal conditions.

3.5.7.1.5 Save a portion of all washing solution (methylene chloride, water) used for cleanup as a blank. Transfer 200 ml of each solution directly from the wash bottle being used and place each in a separate, prelabeled sample container.

3.5.7.2 Sample Containers:

3.5.7.2.1 Container 1: Probe and Impinger Catches. Using a graduated cylinder, measure to the nearest ml, and record the volume of the solution in the first three impingers. Alternatively, the solution may be weighed to the nearest 0.5 g. Include any condensate in the probe in this determination. Transfer the impinger solution from the graduated cylinder into the amber flint glass bottle. Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all surfaces to which the sample is exposed (including the probe nozzle, probe fitting, probe liner, first impinger, and impinger connector) with methylene chloride. Use less than 500 ml for the entire wash (250 ml would be better, if possible). Add the washing to the sample container.

3.5.7.2.1.1 Carefully remove the probe nozzle and rinse the inside surface with methylene chloride from a wash bottle. Brush with a Teflon bristle brush, and rinse until the rinse shows no visible particles or yellow color, after which make a final rinse of the inside surface. Brush and rinse the inside parts of the Swagelok fitting with methylene chloride in a similar way.

3.5.7.2.1.2 Rinse the probe liner with methylene chloride. While squirting the methylene chloride into the upper end of the probe, tilt and rotate the probe so that all inside surfaces will be wetted with methylene chloride. Let the methylene chloride drain from the lower end into the sample container. The tester may use a funnel (glass or polyethylene) to aid in transferring the liquid washes to the container. Follow the rinse with a Teflon brush. Hold the probe in an inclined position, and squirt methylene chloride into the upper end as the probe brush is being pushed with a twisting action through the probe. Hold the sample container underneath the lower end

of the probe, and catch any methylene chloride, water, and particulate matter that is brushed from the probe. Run the brush through the probe three times or more. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since there may be small crevices in which particulate matter can be entrapped. Rinse the brush with methylene chloride or water, and quantitatively collect these washing in the sample container. After the brushing, make a final rinse of the probe as described above.

NOTE: Two people should clean the probe in order to minimize sample losses. Between sampling runs, brushes must be kept clean and free from contamination.

3.5.7.2.1.3 Rinse the inside surface of each of the first three impingers (and connecting tubing) three separate times. Use a small portion of methylene chloride for each rinse, and brush each surface to which the sample is exposed with a Teflon bristle brush to ensure recovery of fine particulate matter. Water will be required for the recovery of the impingers in addition to the specified quantity of methylene chloride. There will be at least two phases in the impingers. This two-phase mixture does not pour well, and a significant amount of the impinger catch will be left on the walls. The use of water as a rinse makes the recovery quantitative. Make a final rinse of each surface and of the brush, using both methylene chloride and water.

3.5.7.2.1.4 After all methylene chloride and water washing and particulate matter have been collected in the sample container, tighten the lid so the solvent, water, and DNPH reagent will not leak out when the container is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Seal the container with Teflon tape. Label the container clearly to identify its contents.

3.5.7.2.1.5 If the first two impingers are to be analyzed separately to check for breakthrough, separate the contents and rinses of the two impingers into individual containers. Care must be taken to avoid physical carryover from the first impinger to the second. The formaldehyde

hydrazone is a solid which floats and froths on top of the impinger solution. Any physical carryover of collected moisture into the second impinger will invalidate a breakthrough assessment.

3.5.7.2.2 Container 2: Sample Blank. Prepare a blank by using an amber flint glass container and adding a volume of DNPH reagent and methylene chloride equal to the total volume in Container 1. Process the blank in the same manner as Container 1.

3.5.7.2.3 Container 3: Silica Gel. Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. The impinger containing the silica gel may be used as a sample transport container with both ends sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon caps maybe used. The silica gel impinger should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If the silica gel is removed from the impinger, the tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use water or other liquids to transfer the silica gel. If a balance is available in the field, the spent silica gel (or silica gel plus impinger) maybe weighed to the nearest 0.5 g.

3.5.7.2.4 Sample containers should be placed in a cooler, cooled by (although not in contact with) ice. Sample containers must be placed vertically and, since they are glass, protected from breakage during shipment. Samples should be cooled during shipment so they will be received cold at the laboratory.

3.5.8 Calibration

3.5.8.1 Probe Nozzle: Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in). When the nozzles become nicked or corroded, they shall be replaced and calibrated before use. Each nozzle must be permanently and uniquely identified.

3.5.8.2 Pitot Tube: The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of EPA Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked or corroded and if it meets design and intercomponent spacing specifications.

3.5.8.3 Metering System

3.5.8.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm pumps, the normal leak check procedure will not detect leakages with the pump. For these cases, the following leak check procedure will apply: make a ten-minute calibration run at 0.00057 m^3/min (0.02 cfm). At the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

3.5.8.3.2 After each field use, check the calibration of the metering system by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). Set the vacuum at the maximum value reached during the test series. To adjust the vacuum, insert a

valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more the 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

3.5.8.3.3 Leak check of metering system: The portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. Use the following procedure: Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 - 18 cm (5 - 7 in) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series must be voided or calculations for test series must be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

3.5.8.4 Probe Heater: The probe heating system must be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

3.5.8.5 Temperature gauges: Each thermocouple must be permanently and uniquely marked on the casting. All mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at the ambient air temperatures, with and without the extension lead, must be noted

and recorded. Correction is necessary if the use of an extension lead produces a change >1.5%.

3.5.8.5.1 Impinger and dry-gas meter thermocouples: For the thermocouples used to measure the temperature of the gas leaving the impinger train, three-point calibration at ice water, room air, and boiling water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2C (3.6°F) with those of the absolute value of the reference thermometer.

3.5.8.5.2 Probe and stack thermocouple: for the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice water, boiling water, and hot oil bath temperatures must be performed. Use of a point at room air temperature is recommended. The thermometer and thermocouple must agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

3.5.8.6 Barometer: Adjust the barometer initially and before each test series to agree to within ±2.5 mm Hg (0.1 in Hg) of the mercury barometer or the correct barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

3.5.8.7 Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class S standard weights. The weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

3.5.9 **Calculations**

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

3.5.9.1 Calculation of Total Formaldehyde: To determine the total formaldehyde in mg, use the following equation:

$$\text{Total mg formaldehyde} = C_d \times V \times DF \times \frac{[\text{g/mole aldehyde}]}{[\text{g/mole DNPH derivative}]} \times 10^3 \text{ mg}/\mu\text{g}$$

where:

C_d = measured concentration of DNPH - formaldehyde derivative, μg/ml
V = organic extract volume ml
DF = dilution factor

3.5.9.2 Formaldehyde concentration in stack gas:

Determine the formaldehyde concentration in the stack gas using the following equation:

$$C_f = K\,[\text{total formaldehyde, mg}]\,V_{m(std)}$$

where:

K = 35.31 ft^3/m^3 if $V_{m(std)}$ is expressed in English units
= 1.00 m^3/m^3 if $V_{m(std)}$ is expressed in metric units
$V_{m(std)}$ = volume of gas sample a measured by dry gas meter, corrected to standard conditions, dscm (dscf)

3.5.9.3 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop are obtained from the data sheet.

3.5.9.4 Dry Gas Volume: Calculate $V_{m(std)}$ and adjust for leakage, if necessary, using the equation in Section 6.3 of EPA Method 5.

3.5.9.5 Volume of Water Vapor and Moisture Content: Calculate the volume of water vapor and moisture content from equations 5-2 and 5-3 of EPA Method 5.

3.5.10 Determination of Volume to be Sampled

To determine the minimum sample volume to be collected, use the following sequence of equations.

3.5.10.1 From prior analysis of the waste feed, the concentration of formaldehyde (FORM) introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the amount of FORM allowed to be present in the effluent. This amount may be expressed as:

Max FORM Mass = [(WF) (FORM conc) (100 - %DRE)] /100

where:

WF = mass flow rate of waste feed per h, g/h (lb/h)

FORM = concentration of FORM (wt %) introduced into the combustion process

DRE = percent Destruction and Removal Efficiency required

Max FORM = mass flow rate (g/h [lb/]) of FORM emitted from the combustion sources

3.5.10.2 The average discharge concentration of the FORM in the effluent gas is determined by comparing the Max FORM with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary EPA Method 1 - 4 determinations:

Max FORM conc = [Max FORM Mass] / $DV_{eff(std)}$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf)

FORM conc = anticipated concentration of the FORM in the exhaust gas stream, g/dscm (lb/dscf)

3.5.10.3 In making this calculation, it is recommended that a safety margin of at least ten be included.

$$[LDL_{FORM} \times 10 / \text{FORM conc}] = V_{tbo}$$

where:

LDL_{FORM} = detectable amount of FORM in entire sampling train

V_{tbo} = minimum dry standard volume to be collected at dry-gas meter

3.5.10.4 The following analytical detection limits and DNPH Reagent Capacity (based on a total volume of 200 ml in two impingers) must also be considered in determining a volume to be sampled.

3.5.11 Quality Control

3.5.11.1 Sampling: See EPA Manual 600/4-77-02b for Method 5 quality control.

3.5.11.2 Analysis: The quality assurance program required for this method includes the analysis of the field and method blanks, procedure validations, and analysis of field spikes. The assessment of combustion data and positive identification and quantitation of formaldehyde are dependant on the integrity of the samples received and the precision and accuracy of the analytical methodology. Quality assurance procedures for this method are designed to monitor the performance of the analytical methodology and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

3.5.11.2.1 Field Blanks: Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, methylene chloride and water, and unused DNPH reagent. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling are, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual sampling train). The probe of the blank train must be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the blank sampling train.

3.5.11.2.2 Method Blanks: A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

3.5.11.2.3 Field Spike: A field spike is performed by introducing 200 µL of the Field Spike Standard into an impinger containing 200 ml of DNPH solution. Standard impinger recovery procedures are followed and the spike is used as a check on field handling and recovery procedures. An aliquot of the field spike standard is retained in the laboratory for derivatization and comparative analysis.

3.5.12 Method Performance

3.5.12.1 Method performance evaluation: The expected method performance parameters for precision, accuracy, and detection limits are provided in Table 3.5-3.

Addition of a Filter to the Formaldehyde Sampling Train

As a check on the survival of particulate material through the impinger system, a filter can be added to the impinger train either after the second impinger or after the third impinger. Since the impingers are in an ice bath there is no reason to heat the filter at this point.

Any suitable medium (e.g., paper, organic membrane) may be used for the filter if the material conforms to the following specifications:

1) the filter has at least 95% collection efficiency (<5% penetration) for 3 µm dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

2) the filter has a low aldehyde blank value (<0.015 mg formaldehyde/cm^2 of filter area). Before the test series, determine the average formaldehyde blank value of at least three filters (from the lot to be used for sampling) using the applicable analytical procedures.

Table 3.5-3

EXPECTED METHOD PERFORMANCE FOR FORMALDEHYDE

Parameter		Precision[1]	Accuracy[2]	Detection Limit[3]
Matrix:	Dual trains	±15% RPD	±20%	1.5×10^{-7} lb/ft^3 (1.8 ppbv)

[1]Relative percent difference limit for dual trains.

[2]Limit for field spike recoveries.

[3]The lower reporting limit having less than 1% probability of false positive detection.

Recover the exposed filter into a separate clean container and return the container over ice to the laboratory for analysis. If the filter is being analyzed for formaldehyde, the filter may be recovered into a container or DNPH reagent for shipment back to the laboratory. If the filter is being examined for the presence of particulate material, the filter may be recovered into a clean dry container and returned to the laboratory.

3.6 Analysis for Aldehydes and Ketones by High Performance Liquid Chromatography (HPLC) (Method 0011A)

3.6.1 Scope and Application

3.6.1.1 Method 0011A covers the determination of free formaldehyde in the aqueous samples and leachates and derived aldehydes/ketones collected by Method 0011.

Compound Name	CAS No.*
Formaldehyde	50-00-0
Acetaldehyde	75-07-0

* Chemical Abstract Services Registry Number

3.6.1.2 Method 0011A is a high performance liquid chromatographic (HPLC) method optimized for the determination of formaldehyde and acetaldehyde in aqueous environmental matrices and leachates of solid samples and stack samples collected by Method 0011. When this method is used to analyze unfamiliar sample matrices, compound identification should be supported by at least one additional qualitative technique. A gas chromatograph/mass spectrometer (GC/MS) may be used for the qualitative confirmation of results from the target analytes, using the extract produced by this method.

3.6.1.3 The method detection limits (MDL) are listed in Tables 3.6-1 and 3.6-2. The MDL for a specific sample may differ from that listed, depending upon the nature of interferences in the sample matrix and the amount of sample used in the procedure.

3.6.1.4 The extraction procedure for solid samples is similar to that specified in Method 1311 (1). Thus, a single sample may be extracted to measure the analytes included in the scope of other appropriate methods. The analyst is allowed the flexibility to select chromatographic conditions

Table 3.6-1

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS USING SOLID SORBENT EXTRACTION

Analyte	Retention Time (minutes)	MDL (µg/L)*
Formaldehyde	7.1	7.2

HPLC conditions: Reverse phase C18 column, 4.6 x 250 mm; isocratic elution using methanol/water (75:25, v/v); flow rate 1.0 mL/min.; detector 360 nm.

* After correction for laboratory blank.

Table 3.6-2

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS USING METHYLENE CHLORIDE EXTRACTION

Analyte	Retention Time (minutes)	MDL (µg/L)*
Formaldehyde	7.1	7.2
Acetaldehyde	8.6	171*

HPLC conditions: Reverse phase C18 column, 4.6 x 250 mm; isocratic elution using methanol/water (75:25, v/v); flow rate 1.0 mL/min.; detector 360 nm.

- These values include reagent blank concentrations of approximately 13 µg/L formaldehyde and 130 µg/L acetaldehyde.

appropriate for the simultaneous measurement of contaminations of these analytes.

3.6.1.5 This method is restricted to use by, or under the supervision of analysts experienced in the use of chromatography and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

3.6.1.6 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available.

3.6.1.7 Formaldehyde has been tentatively classified as a known or suspected, human or mammalian carcinogen.

3.6.2 Summary of Method

3.6.2.1 Environmental Liquids and Solid Leachates

3.6.2.1.1 For wastes comprised of solids or for aqueous wastes containing significant amounts of solid material, the aqueous phase, if any, is separated from the solid phase and stored for later analysis. If necessary, the particle size of the solids in the waste is reduced. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase of the waste. A special extractor vessel is used when testing for volatiles. Following extraction, the aqueous extract is separated from the solid phase by filtration employing 0.6 to 0.8 μm glass fiber filters.

3.6.2.1.2 If compatible (i.e., multiple phases will not form on combination), the initial aqueous phase of the waste is added to the aqueous extract, and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume weighted average concentration.

3.6.2.1.3 A measured volume of aqueous sample or an appropriate amount of solids leachate is buffered to pH 5 and derivatized with 2,4-dinitrophenylhydrazine (DNPH), using either the solid sorbent or the methylene derivatization/extraction option. If the solid sorbent option is used, the derivative is extracted using solid sorbent cartridges, followed by elution with ethanol. If the methylene chloride option is used, the derivative is extracted with methylene chloride. The methylene chloride extracts are concentrated using the Kuderna-Danish (K-D) procedure and solvent exchanged into methanol prior to HPLC analysis. Liquid chromatographic conditions are described which permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

3.6.2.2 Stack Gas Samples Collected by Method 0011

3.6.2.2.1 The entire sample returned to the laboratory is extracted with methylene chloride and the methylene chloride extract is brought up to a known volume. An aliquot of the methylene chloride extract is solvent exchanged and concentrated or diluted as necessary.

3.6.2.2.2 Liquid chromatographic conditions are described that permit the separation and measurement of formaldehyde in the extract by absorbance detection at 360 nm.

3.6.3 Interferences

3.6.3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of

these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks.

3.6.3.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used This should be followed by detergent washing with hot water, and rinses with tap water and distilled water. It should then be drained, dried, and heated in a laboratory oven at 130°C for several hours before use. Solvent rinses with methanol may be substituted for the oven heating. After drying and cooling, glassware should be stored in a clean environment to prevent any accumulation of dust or other contaminants.

3.6.3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.6.3.2 Analysis for formaldehyde is especially complicated by its ubiquitous occurrence in the environment.

3.6.3.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the matrix being sampled. No interferences have been observed in the matrices studied as a result of using solid sorbent extraction as opposed to liquid extraction. If interferences occur in subsequent samples, some additional cleanup may be necessary.

3.6.3.4 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the HPLC conditions described allow for a resolution of the specific compounds covered by this method, other matrix components may interfere.

3.6.4 <u>Apparatus and Materials</u>

3.6.4.1 Reaction vessel - 250 ml Florence flask.

3.6.4.2 Separatory funnel - 205 ml, with Teflon stopcock

3.6.4.3 Kuderna-Danish (K-D) apparatus.

3.6.4.3.1 Concentrator tube - 10 ml graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

3.6.4.3.2 Evaporation flask - 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent

3.6.4.3.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

3.6.4.3.4 Snyder column - Two ball macro (Kontes K-569001-0219 or equivalent).

3.6.4.3.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

3.6.4.4 Vials - 10, 25 ml, glass with Teflon lined screw caps or crimp tops.

3.6.4.5 Boiling chips - Solvent extracted with methylene chloride, approximately 10/40 mesh (silicon carbide or equivalent).

3.6.4.6 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

3.6.4.7 pH meter - Capable of measuring to the nearest 0.01 units.

3.6.4.8 High performance liquid chromatograph (modular)

3.6.4.8.1 Pumping system - Isocratic, with constant flow control capable of 1.00 ml/min.

3.6.4.8.2 High pressure injection valve with 20 µL loop.

3.6.4.8.3 Column - 250 mm x 4.6 mm ID, 5 µm particle size, C18 (or equivalent).

3.6.4.8.4 Absorbance detector - 360 nm.

3.6.4.8.3 Strip-chart recorder compatible with detector - Use of a data system for measuring peak areas and retention times is recommended.

3.6.4.9 Glass fiber filter paper.

3.6.4.10 Solid sorbent cartridges - Packed with 500 mg C18 (Baker or equivalent).

3.6.4.11 Vacuum manifold - Capable of simultaneous extraction of up to 12 samples (Supelco or equivalent).

3.6.4.12 Sample reservoirs - 60 ml capacity (Supelco or equivalent).

3.6.4.13 Pipet - Capable of accurately delivering 0.10 ml solution (Pipetman or equivalent).

3.6.4.14 Water bath - Heated, with concentric ring cover, capable of temperature control ((±) 2°C). The bath should be used under a hood.

3.6.4.15 Volumetric Flasks - 250 or 500 ml.

3.6.5 Reagents

3.6.5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.6.5.2 Organic-free water - All references to water in this method refer to organic-free reagent water, as defined in Chapter I SW-846.

3.6.5.3 Methylene chloride, CH_2Cl_2 - HPLC grade or equivalent.

3.6.5.4 Methanol, CH_3OH - HPLC grade or equivalent.

3.6.5.5 Ethanol (absolute), CH_3CH_2OH - HPLC grade or equivalent.

3.6.5.6 2,4-Dinitrophenylhydrazine (DNPH) (70% (W/W)), $(2,4\text{-}(O_2N)_2C_6H_3)NHNH_2$, in organic-free reagent water.

3.6.5.7 Formalin (37.6 percent (w/w)), formaldehyde in organic-free reagent water.

3.6.5.8 Acetic acid (glacial), CH_3CO_2H.

3.6.5.9 Sodium hydroxide solutions NaOH, 1.0 N and 5 N.

3.6.5.10 Sodium chloride, NaCl.

3.6.5.11 Sodium sulfite solution, Na_2SO_3, 0.1 M.

3.6.5.12 Hydrochloric Acid, HCl, 0.1 N.

3.6.5.13 Extraction fluid - Dilute 64.3 ml of 1.0 N NaOH and 5.7 ml glacial acetic acid to 900 ml with organic-free reagent water. Dilute to 1 liter with organic-free reagent water. The pH should be 4.93 ± 0.02.

3.6.5.14 Stock standard solutions

3.6.5.14.1 Stock formaldehyde (approximately 1.00 mg/ml) - Prepare by diluting 265 µl formalin to 100 ml with organic-free reagent water.

3.6.5.14.1.1 Standardization of formaldehyde stock solution - Transfer a 25 ml aliquot of a 0.1 M Na_2SO_3 solution to a beaker and record the pH. Add a 25.0 ml aliquot of the formaldehyde stock solution (Section 3.6.5.14.1) and record the pH. Titrate this mixture back to the original pH

using 0.1 N HCl. The formaldehyde concentration is calculated using the following equation:

Concentration (mg/ml) = 30.03 x (N HCl) x (ml HCl) 25.0

where:

N HCl = Normality of HCl solution used

ml HCl = ml of standardized HCl solution used

30.03 = MW of formaldehyde

3.6.5.14.2 Stock formaldehyde and acetaldehyde - Prepare by adding 265 µL formalin and 0.1 g acetaldehyde to 90 ml of water and dilute to 100 ml. The concentration of acetaldehyde in this solution is 1.00 mg/ml. Calculate the concentration of formaldehyde in this solution using the results of the assay performed in Section 3.6.5.14.1.1.

3.6.5.14.3 Stock standard solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

3.6.5.15 Reaction Solutions

3.6.5.15.1 DNPH (1.00 µg/L) - Dissolve 142.9 mg of 70% (w/w) reagent in 100 ml absolute ethanol. Slight heating or sonication may be necessary to effect dissolution.

3.6.5.15.2 Acetate buffer (5 N) Prepare by neutralizing glacial acetic acid to pH 5 with 5 N NaOH solution. Dilute to standard volume with water.

3.6.5.15.3 Sodium chloride solution (saturated) Prepare by mixing of the reagent grade solid with water.

3.6.6 Sample Collection, Preservation, and Handling

3.6.6.1 See the introductory material to this Chapter, Organic Analytes, Section 4.1 of SW-846.

3.6.6.2 Environmental liquid and leachate samples must be refrigerated at 4°C, and must be derivatized within 5 days of sample collection and analyzed within 3 days of derivatization.

3.6.6.3 Stack gas samples collected by Method 0011 must be refrigerated at 4°C. It is recommended that samples be extracted within 30 days of collection and that extracts be analyzed within 30 days extraction.

3.6.7 Procedure

3.6.7.1 Extraction of Solid Samples

3.6.7.1.1 All solid samples should be homogeneous. When the sample is not dry, determine the dry weight of the sample, using a representative aliquot.

3.6.7.1.1.1 Determination of dry weight - In certain cases, sample results are desired based on a dry weight basis. When such data is desired, or required, a portion of sample for dry weight determination should be weighed out at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample.

3.6.7.1.1.2 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the % dry weight of the sample by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

3.6.7.1.2 Measure 25 g of solid into a 500 ml bottle with a Teflon lined screw cap or crimp top, and add 500 ml of extraction fluid (Section 3.6.5.13). Extract the solid by rotating the bottle at approximately 30 rpm

for 18 hours. Filter the extract through glass fiber paper and store in sealed bottles at 4°C. Each ml of extract represents 0.050 g solid.

3.6.7.2 Cleanup and Separation

3.6.7.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. The cleanup procedures recommended in this method have been used for the analysis of various sample types. If particular circumstances demand the use of an alternative cleanup procedure, the analyst must determine the elution profile and demonstrate that the recovery of formaldehyde is no less then 85% of recoveries specified in Table 3.6-3. Recovery may be lower for samples which form emulsions.

3.6.7.2.2 If the sample is not clean, or the complexity is unknown, the entire sample should be centrifuged at 2500 rpm for 10 minutes. Decant the supernatant liquid from the centrifuge bottle, and filter through glass fiber filter paper into a container which can be tightly sealed.

3.6.7.3 Derivatization

3.6.7.3.1 For aqueous samples, measure a 50 to 100 ml aliquot of the sample. Quantitatively transfer the sample aliquot to the reaction vessel (Section 3.6.4.1).

3.6.7.3.2 For solid samples, 1 to 10 ml of leachate (Section 3.6.7.1) will usually be required. The amount used for a particular sample must be determined through preliminary experiments.

Table 3.6-3

SINGLE OPERATOR ACCURACY AND PRECISION
USING SOLID SORBENT EXTRACTION

Analyte	Matrix Type	Average Percent Recovery	Standard Deviation Percent	Spike Range (µg/L)	Number of Analyses
Formaldehyde	Reagent Water	86	9.4	15-1430	39
	Final Effluent	90	11.0	46.8-1430	16
	Phenol formaldehyde Sludge	93	12.0	457-1430	15

Note: For all reactions, the total volume of the aqueous layer should be adjusted to 100 ml with water.

3.6.7.3.3 Derivatization and extraction of the derivative can be accomplished using the solid sorbent (Section 3.6.7.3.4) or methylene chloride option (Section 3.6.7.3.5).

3.6.7.3.4 Solid Sorbent Option

3.6.7.3.4.1 Add 4 ml of acetate buffer and adjust the pH to 5.0 ± 0.1 with glacial acetic acid or 5 N NaOH. Add 6 ml of DNPH reagent, seal the container, and place on a wrist-action shaker for 30 minutes.

3.6.7.3.4.2 Assemble the vacuum manifold and connect to a water aspirator or vacuum pump. Assemble solid sorbent cartridges containing a minimum of 1.5 g of C18 sorbent, using connectors supplied by the manufacturer, and attach the sorbent train to the vacuum manifold. Condition each cartridge by passing 10 ml dilute acetate buffer (10 ml 5 N acetate buffer dissolved in 250 ml water) through the sorbent cartridge train.

3.6.7.3.4.3 Remove the reaction vessel from the shaker and add 10 ml saturated NaCl solution to the vessel.

3.6.7.3.4.4 Add the reaction solution to the sorbent train and apply a vacuum so that the solution is drawn through the cartridges at a rate of 3 to 5 ml/min. Release the vacuum after the solution has passed through the sorbent.

3.6.7.3.4.5 Elute each cartridge train with approximately 9 ml of absolute ethanol, directly into a 10 ml volumetric flask. Dilute the solution to volume with absolute ethanol, mixed thoroughly, and place in a tightly sealed vial until analyzed.

3.6.7.3.5 Methylene Chloride Option

3.6.7.3.5.1 Add 5 ml of acetate buffer and adjust the pH to 5.0 ± 0.5 with glacial acetic acid or 5 N NaOH. Add 10 ml of DNPH reagent, seal the container, and place on a wrist-action shaker for 1 hour.

3.6.7.3.5.2 Extract the solution with three 20 ml portions of methylene chloride, using a 250 ml separatory funnel, and combine the methylene chloride layers. If an emulsion forms upon extraction, remove the entire emulsion and centrifuge at 2000 rpm for 10 minutes. Separate the layers and proceed with the next extraction.

3.6.7.3.5.3 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporator flask. Wash the K-D apparatus with 25 ml of extraction solvent to complete the quantitative transfer.

3.6.7.3.5.4 Add one to two clean boiling chips to the evaporative flask and attach a three ball Snyder column. Preset the Snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 10 ml, remove the K-D apparatus and allow it to drain and cool for a least 10 min.

3.6.7.3.5.5 Prior to liquid chromatographic analysis, the solvent must be exchanged to methanol. The analyst must ensure quantitative transfer of the extract concentrate. The exchange is performed as follows:

3.6.7.3.5.5.1 Following K-D concentration of the methylene chloride extract to < 10 ml using the macro Snyder column, allow the apparatus to cool and drain for at least 10 minutes.

3.6.7.3.5.5.2 Momentarily remove the Snyder column, add 5 ml of the methanol, a [illegible] oed, or boiling chip, and attach the micro Snyder column. Concentrate the extract using 1 ml of methanol to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration. At the proper [illegible] of distillation the balls of the column will actively chatter, [illegible] chambers will not flood. When the apparent volume of liquid reac[illegible], remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

3.6.7.3.5.5.3 Remove the Snyder column and rinse the flask and its lower joint with 1-2 ml of methanol and add to concentrator tube. A 5-ml syringe is recommended for this operation. Adjust the extract volume to 10 ml. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon-lined screw cap or crimp top. Proceed with liquid chromatographic analysis if further cleanup is not required.

3.6.7.4 Extraction of Stack Gas Samples Collected by Method 0011

3.6.7.4.1 Measure the aqueous volume of the sample prior to extraction (for moisture determination in case the volume was not measured in the field). Pour the sample into a separatory funnel and drain the methylene chloride into a volumetric flask.

3.6.7.4.2 Extract the aqueous solution with two or three aliquots of methylene chloride. Add the methylene chloride extracts to the volumetric flask.

3.6.7.4.3 Fill the volumetric flask to the line with methylene chloride. Mix well and remove an aliquot.

3.6.7.4.4 If high levels of formaldehyde are present, the extract can be diluted with mobile phase, otherwise the extract must be solvent exchanged as described in Section 3.6.7.5.3.3. If low levels of formaldehyde are present, the sample should be concentrated during the solvent exchange procedure.

3.6.7.5 Chromatographic Conditions

Column:	C18, 250 mm x 4.6 mm ID, 5 μm particle size
Mobile Phase:	methanol/water, 75:25 (v/v), isocratic
Flow Rate:	1.0 ml/min
UV Detector:	360 nm
Injection Volume:	20 μl

3.6.7.6 Calibration

3.6.7.6.1 Establish liquid chromatographic operating parameters to produce a retention time equivalent to that indicated in Table 3.6-1 for the solid sorbent options, or in Table 3.6-2 for methylene chloride option. Suggested chromatographic conditions are provided in Section 3.6.7.5. Prepare derivatized calibration standards according to the procedure in Section 3.6.7.6.1.1. Calibrate the chromatographic system using the external standard technique (Section 3.6.7.6.1.2).

3.6.7.6.1.1 Preparation of calibration standards

3.6.7.6.1.1.1 Prepare calibration standard solutions of formaldehyde and acetaldehyde in water from the stock standard (Section 3.6.5.14.2). Prepare these solutions at the following concentrations (in μg/ml) by serial dilution of the stock standard solution: 50, 20, 10. Prepare additional calibration standard solutions at the following concentrations, by dilution of the appropriate 50, 20, or 10 μg/ml standard: 5, 0.5, 2, 0.2, 1, 0.1.

3.6.7.6.1.1.2 Process each calibration standard solution through the derivatization option used for sample processing (Section 3.6.7.3.4 or 3.6.7.3.5).

3.6.7.6.1.2 External standard calibration procedure

3.6.7.6.1.2.1 Analyze each derivatized calibration standard using the chromatographic conditions listed in Tables 3.6-1 and 3.6-2, and tabulate peak area against concentration injected. The results may be used to prepare calibration curves for formaldehyde and acetaldehyde.

3.6.7.6.1.2.2 The working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any analyte varies from the previously established responses by more the 10%, the test must be repeated using a fresh calibration standard after it is verified that the analytical system is in control. Alternatively, a new calibration curve may be prepared for that compound. If an autosampler is available, it is convenient to prepare a calibration curve daily by analyzing standards along with test samples.

3.6.7.7 Analysis

3.6.7.7.1 Analyze samples by HPLC, using conditions established in Section 3.6.7.6.1. Tables 3.6-1 and 3.6-2 list the retention times and MDLs that were obtained under these conditions. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements for Section 3.6.8.1 are met, or if the data are within the limits described in Tables 3.6-1 and 3.6-2.

3.6.7.7.2 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

3.6.7.7.3 If the peak area exceeds the linear range of the calibration curve, a smaller sample volume should be used. Alternatively, the final solution may be diluted with ethanol and reanalyzed.

3.6.7.7.4 If the peak area measurement is prevented by the presence of observed interferences, further cleanup is required. However, none of the 3600 method series have been evaluated for this procedure.

3.6.7.8 Calculations

3.6.7.8.1 Calculate each response factor as follows (mean value based on 5 points):

$$RF = \frac{\text{concentration of standard}}{\text{area of the signal}}$$

$$\text{mean} = RF = \overline{RF} = \frac{(\sum_{i}^{5} RF_i)}{5}$$

3.6.7.8.2 Calculate the concentration of formaldehyde and acetaldehyde as follows:

$$\mu g/ml = (\overline{RF})\ (\text{area of signal})\ (\text{concentration factor})$$

where:

$$\text{concentration factor} = \frac{\text{Final volume of extract}}{\text{Initial sample (or leachate) volume}}$$

NOTE: For solid samples, a dilution factor must be included in the equation to account for the weight of the sample used.

3.6.7.8.3 Calculate the total weight of formaldehyde in the stack gas sample as follows:

total µg/ml = (RF) (area of signal) (concentration factor)

where:

$$\text{concentration factor} = \frac{\text{Final Volume of Extract}}{\text{Initial Extract Volume}}$$

3.6.8 Quality Control

3.6.8.1 Refer to Chapter One of SW-846 for guidance on quality control procedures.

3.6.9 Method Performance

3.6.9.1 The MDL concentrations listed in Table 3.6-1 were obtained using organic-free water and solid sorbent extraction. Similar results were achieved using a final effluent and sludge leachate. The MDL concentrations listed in Table 3.6-2 were obtained using organic-free water and methylene chloride extraction. Similar results were achieved using representative matrices.

3.6.9.2 This method has been tested for linearity of recovery from spiked organic-free water and has been demonstrated to be applicable over the range from 2 x MDL to 200 x MDL.

3.6.9.3 In a single laboratory evaluation using several spiked matrices, the average recoveries presented in Tables 3.6-3 and 3.6-4 were obtained using solid sorbent and methylene chloride extraction, respectively. The standard deviations of the percent recovery are also included in Tables 3.6-3 and 3.6-4.

3.6.9.4 A representative chromatogram is presented in Figure 3.6-1.

3.6.10 References

1. Federal Register, 1986, 51, 40643-40652; November 7.

2. EPA Methods 6010, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency. Washington, D.C. 20460.

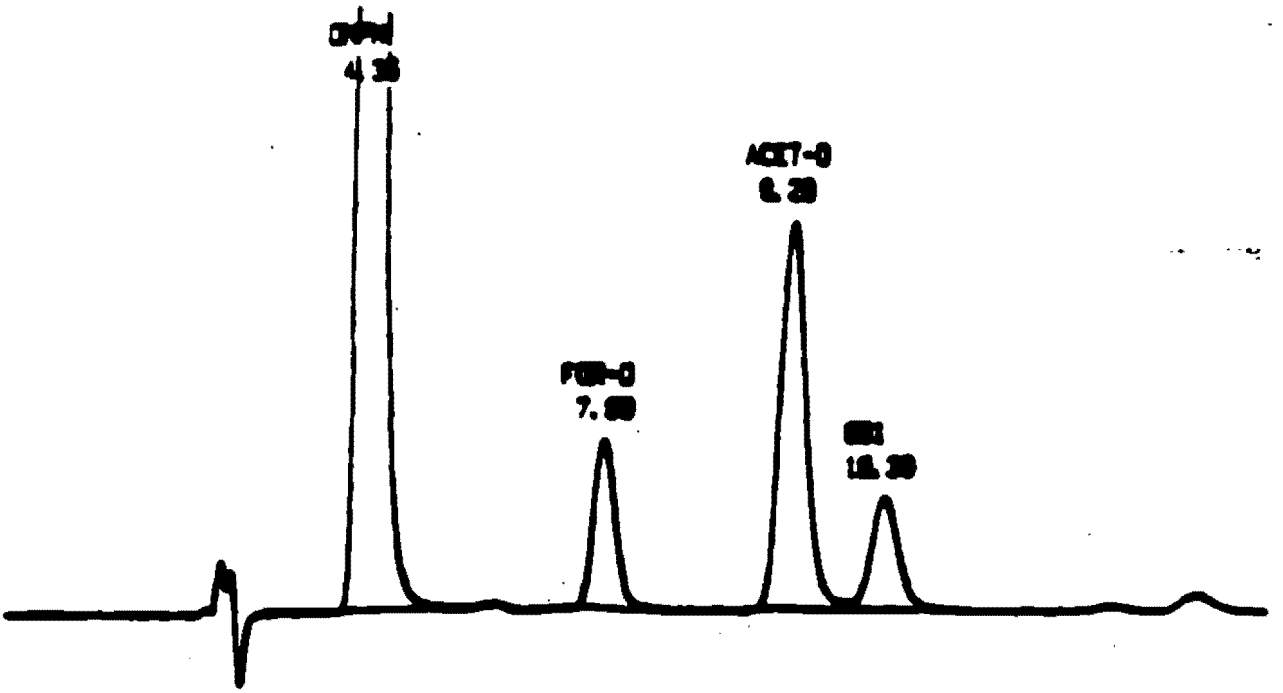
Table 3.6-4

SINGLE OPERATOR ACCURACY AND PRECISION
USING METHYLENE CHLORIDE EXTRACTION

Analyte	Matrix Type	Average Percent Recovery x	Standard Deviation Percent p	Spike Range (µg/L)	Number of Analyses
Formaldehyde	Reagent Water	91	2.5	50-1000	9
	Ground-water	92.5	8.2	50	6
	Liquids	69.6	16.3	250	12
Acetaldehyde	Reagent Water	60.3	3.2	50-1000	9
	Ground-water	63.6	10.9	50	12
	Liquids (2 types)	44.0	20.2	250	12
	Solids	58.4	2.7	0.10-1.0[a]	12

[a] Spike range in units of mg/g.
x = Average recovery expected for this method
p = Average standard deviation expected for this method.

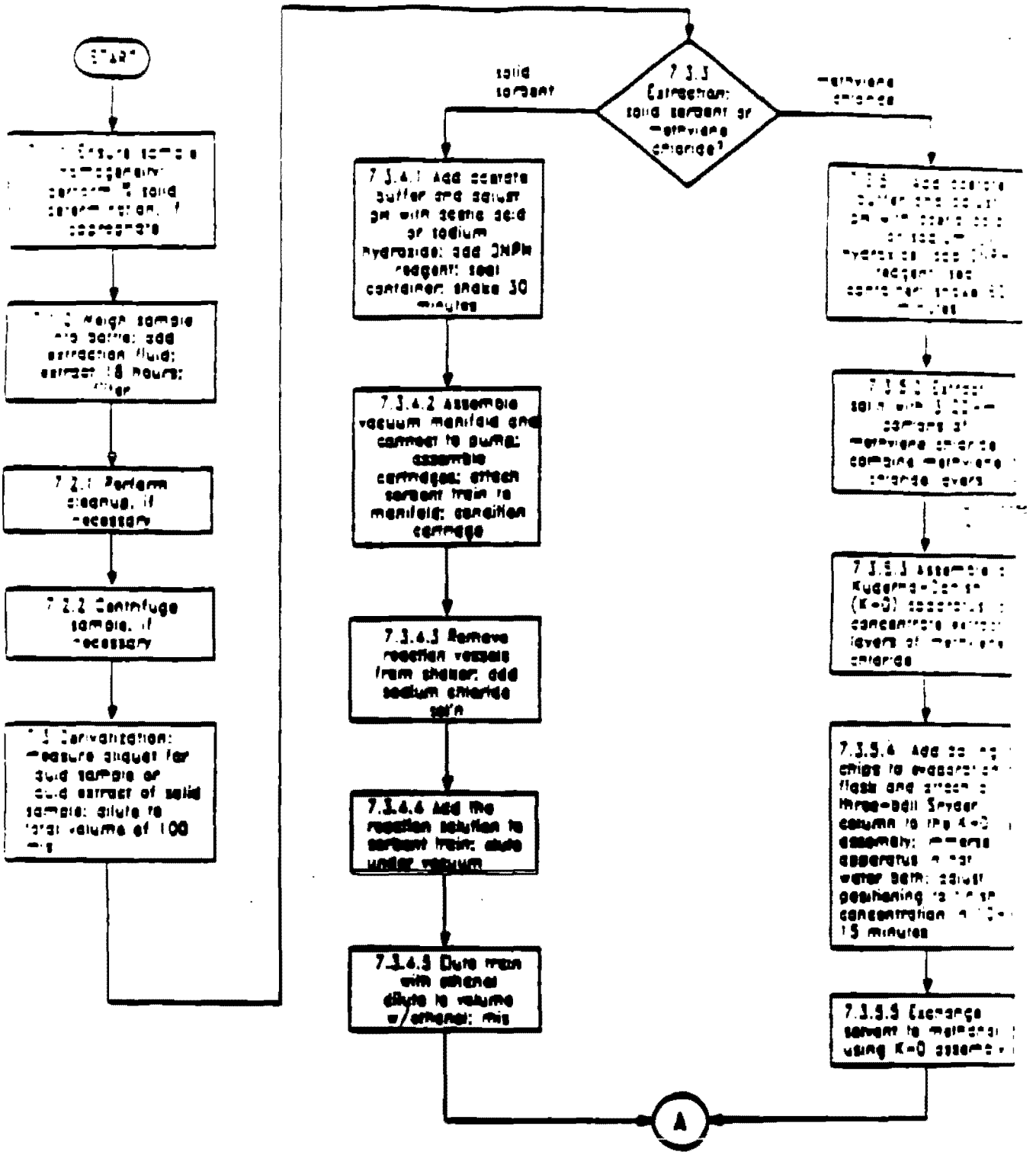
Figure 3.6-1
REPRESENTATIVE CHROMATOGRAM OF A 50 µg/L SOLUTION OF FORMALDEHYDE

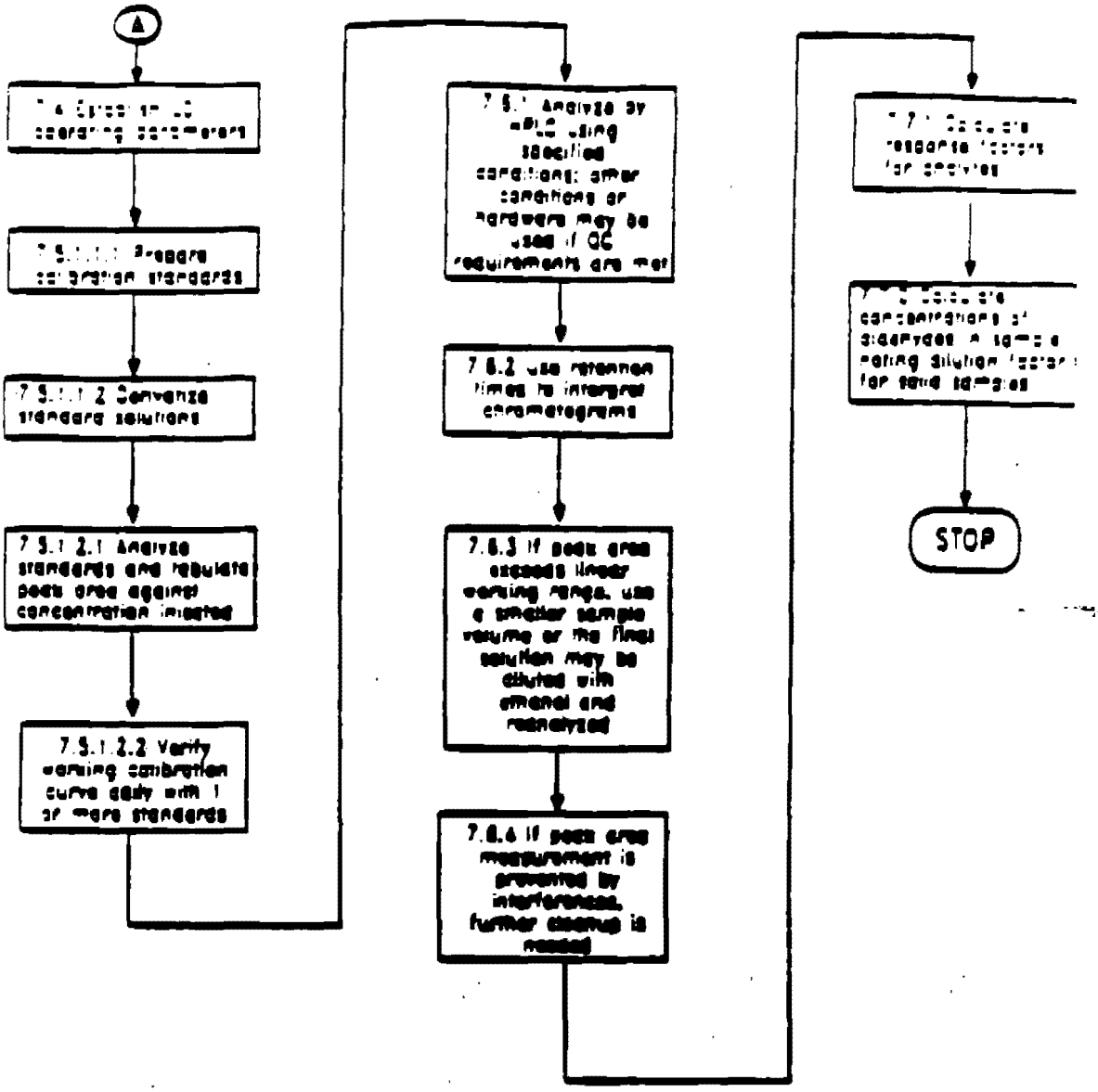


FOR-D = Formaldehyde derivative
ACET-D = Acetaldehyde derivative

METHOD 0011A

FORMALDEHYDE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)




STOP

APPENDIX J.4

PAH

METHOD 0010

MODIFIED METHOD 5 SAMPLING TRAIN

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of semivolatile Principal Organic Hazardous Compounds (POHCs) from incineration systems (PHS, 1967). This method also may be used to determine particulate emission rates from stationary sources as per EPA Method 5 (see References at end of this method).

2.0 SUMMARY OF METHOD

2.1 Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high-efficiency glass- or quartz-fiber filter and a packed bed of porous polymeric adsorbent resin. The filter is used to collect organic-laden particulate materials and the porous polymeric resin to adsorb semivolatile organic species. Semivolatile species are defined as compounds with boiling points >100°C.

2.2 Comprehensive chemical analyses of the collected sample are conducted to determine the concentration and identity of the organic materials.

3.0 INTERFERENCES

3.1 Oxides of nitrogen (NO_x) are possible interferents in the determination of certain water-soluble compounds such as dioxane, phenol, and urethane; reaction of these compounds with NO_x in the presence of moisture will reduce their concentration. Other possibilities that could result in positive or negative bias are (1) stability of the compounds in methylene chloride, (2) the formation of water-soluble organic salts on the resin in the presence of moisture, and (3) the solvent extraction efficiency of water-soluble compounds from aqueous media. Use of two or more ions per compound for qualitative and quantitative analysis can overcome interference at one mass. These concerns should be addressed on a compound-by-compound basis before using this method.

4.0 APPARATUS AND MATERIALS

4.1 Sampling train:

4.1.1 A schematic of the sampling train used in this method is shown in Figure 1. This sampling train configuration is adapted from EPA Method 5 procedures, and, as such, the majority of the required equipment

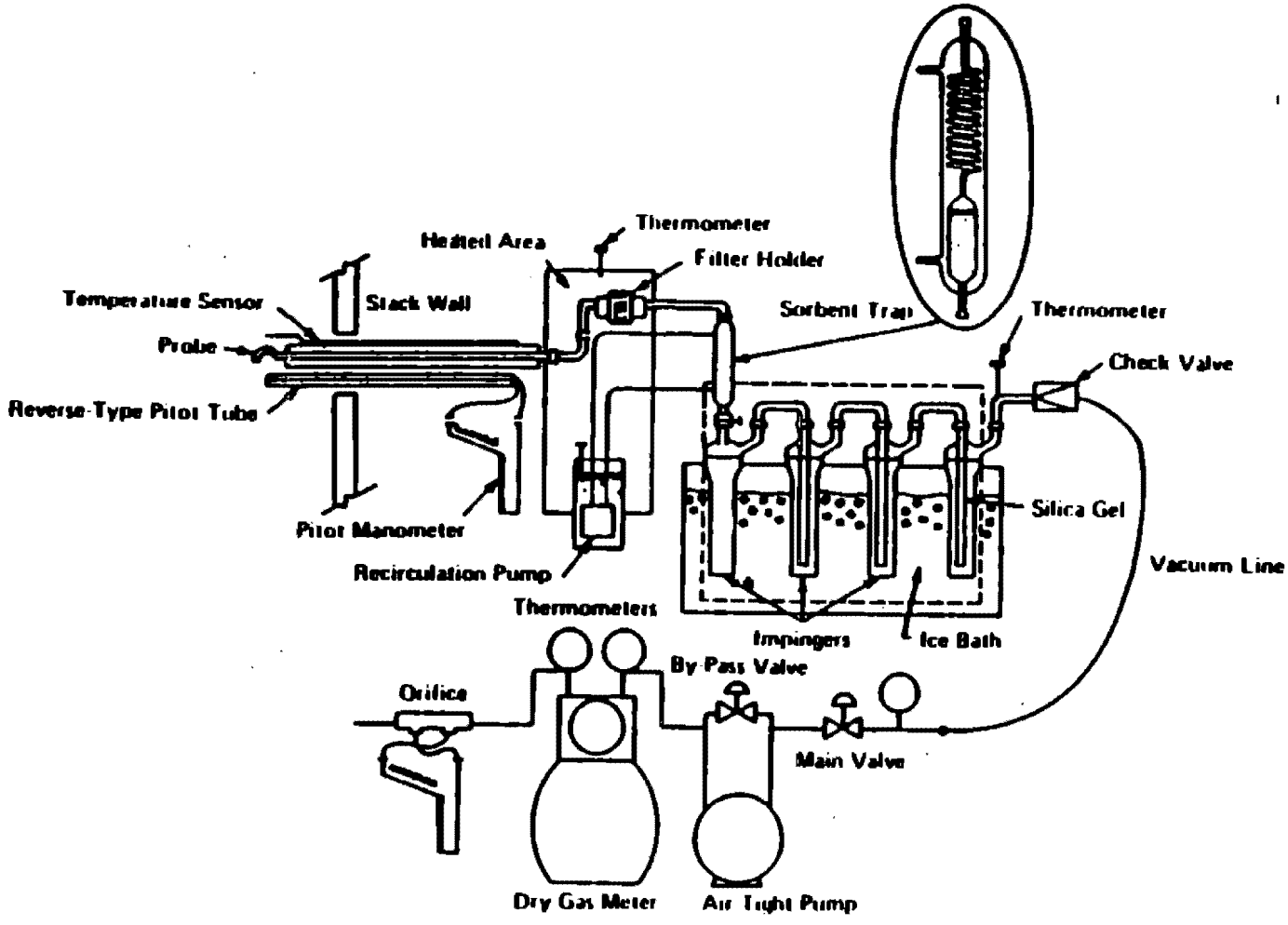


Figure 1. Modified Method 5 Sampling Train.

is identical to that used in EPA Method 5 determinations. The new components required are a condenser coil and a sorbent module, which are used to collect semivolatile organic materials that pass through the glass- or quartz-fiber filter in the gas phase.

4.1.2 Construction details for the basic train components are given in APTD-0581 (see Martin, 1971, in Section 13.0, References); commercial models of this equipment are also available. Specifications for the sorbent module are provided in the following subsections. Additionally, the following subsections list changes to APTD-0581 and identify allowable train configuration modifications.

4.1.3 Basic operating and maintenance procedures for the sampling train are described in APTD-0576 (see Rom, 1972, in Section 13.0, References). As correct usage is important in obtaining valid results, all users should refer to APTD-0576 and adopt the operating and maintenance procedures outlined therein unless otherwise specified. The sampling train consists of the components detailed below.

4.1.3.1 Probe nozzle: Stainless steel (316) or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant I.D. The nozzle shall be buttonhook or elbow design and constructed from seamless tubing (if made of stainless steel). Other construction materials may be considered for particular applications. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g., 0.32-1.27 cm (1/8-1/2 in.), or larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Paragraph 9.1.

4.1.3.2 Probe liner: Borosilicate or quartz-glass tubing with a heating system capable of maintaining a gas temperature of 120 ± 14°C (248 ± 25°F) at the exit end during sampling. (The tester may opt to operate the equipment at a temperature lower than that specified.) Because the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) are considered acceptable. Either borosilicate or quartz-glass probe liners may be used for stack temperatures up to about 480°C (900°F). Quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). (The softening temperature for borosilicate is 820°C (1508°F), and for quartz 1500°C (2732°F).) Water-cooling of the stainless steel sheath will be necessary at temperatures approaching and exceeding 500°C.

4.1.3.3 Pitot tube: Type S, as described in Section 2.1 of EPA Method 2, or other appropriate devices (Vollaro, 1976). The pitot tube shall be attached to the probe to allow constant monitoring of the stack-gas velocity. The impact (high-pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.

4.1.3.4 Differential pressure gauge: Inclined manometer or equivalent device as described in Section 2.2 of EPA Method 2. One manometer shall be used for velocity-head (ΔP) readings and the other for orifice differential pressure (ΔH) readings.

4.1.3.5 Filter holder: Borosilicate glass, with a glass frit filter support and a sealing gasket. The sealing gasket should be made of materials that will not introduce organic material into the gas stream at the temperature at which the filter holder will be maintained. The gasket shall be constructed of Teflon or materials of equal or better characteristics. The holder design shall provide a positive seal against leakage at any point along the filter circumference. The holder shall be attached immediately to the outlet of the cyclone or cyclone bypass.

4.1.3.6 Filter heating system: Any heating system capable of maintaining a temperature of 120 ± 14°C (248 ± 25°F) around the filter holder during sampling. Other temperatures may be appropriate for particular applications. Alternatively, the tester may opt to operate the equipment at temperatures other than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

4.1.3.7 Organic sampling module: This unit consists of three sections, including a gas-conditioning section, a sorbent trap, and a condensate knockout trap. The gas-conditioning system shall be capable of conditioning the gas leaving the back half of the filter holder to a temperature not exceeding 20°C (68°F). The sorbent trap shall be sized to contain approximately 20 g of porous polymeric resin (Rohm and Haas XAD-2 or equivalent) and shall be jacketed to maintain the internal gas temperature at 17 ± 3°C (62.5 ± 5.4°F). The most commonly used coolant is ice water from the impinger ice-water bath, constantly circulated through the outer jacket, using rubber or plastic tubing and a peristaltic pump. The sorbent trap should be outfitted with a glass well or depression, appropriately sized to accommodate a small thermocouple in the trap for monitoring the gas entry temperature. The condensate knockout trap shall be of sufficient size to collect the condensate following gas conditioning. The organic module components shall be oriented to direct the flow of condensate formed vertically downward from the conditioning section, through the adsorbent media, and into the condensate knockout trap. The knockout trap is usually similar in appearance to an empty impinger directly underneath the sorbent module; it may be oversized but should have a shortened center stem (at a minimum, one-half the length of the normal impinger stems) to collect a large volume of condensate without bubbling and overflowing into the impinger train. All surfaces of the organic module wetted by the gas sample shall be fabricated of borosilicate glass, Teflon, or other inert materials. Commercial versions of the

complete organic module are not currently available, but may be assembled from commercially available laboratory glassware and a custom-fabricated sorbent trap. Details of two acceptable designs are shown in Figures 2 and 3 (the thermocouple well is shown in Figure 2).

4.1.3.8 Impinger train: To determine the stack-gas moisture content, four 500-mL impingers, connected in series with leak-free ground-glass joints, follow the knockout trap. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (1/2-in.) I.D. glass tube extending about 1.3 cm (1/2 in.) from the bottom of the outer cylinder. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of water or appropriate trapping solution. The third shall be empty or charged with a caustic solution, should the stack gas contain hydrochloric acid (HCl). The fourth shall contain a known weight of silica gel or equivalent desiccant.

4.1.3.9 Metering system: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment, as shown in Figure 1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry-gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems capable of maintaining sampling rates within 10% of isokineticity and of determining sample volumes to within 2% may be used. The metering system must be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates. Sampling trains using metering systems designed for flow rates higher than those described in APTD-0581 and APTD-0576 may be used, provided that the specifications of this method are met.

4.1.3.10 Barometer: Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30-m (100 ft) elevation increase (vice versa for elevation decrease).

4.1.3.11 Gas density determination equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.4 of EPA Method 2), and gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal.

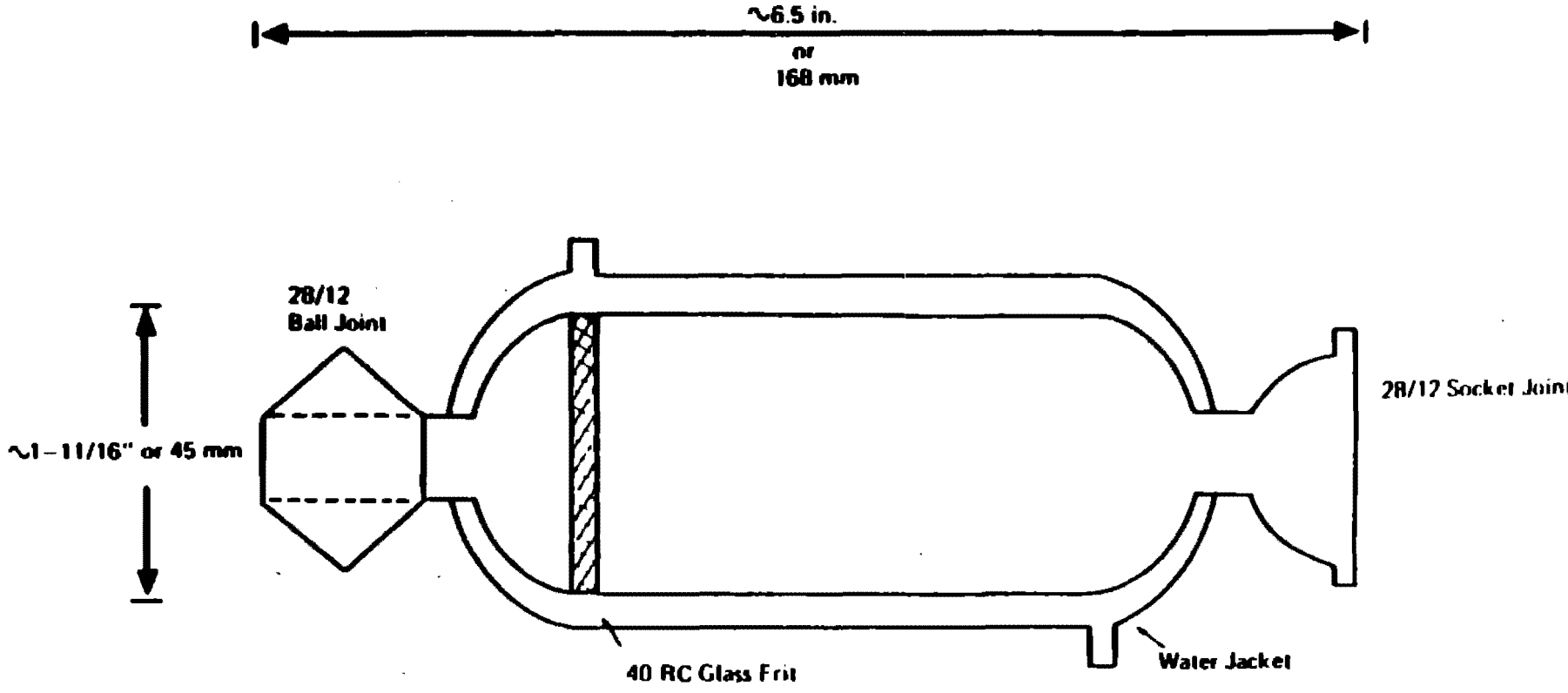


Figure 2. Adsorbent Sampling System.

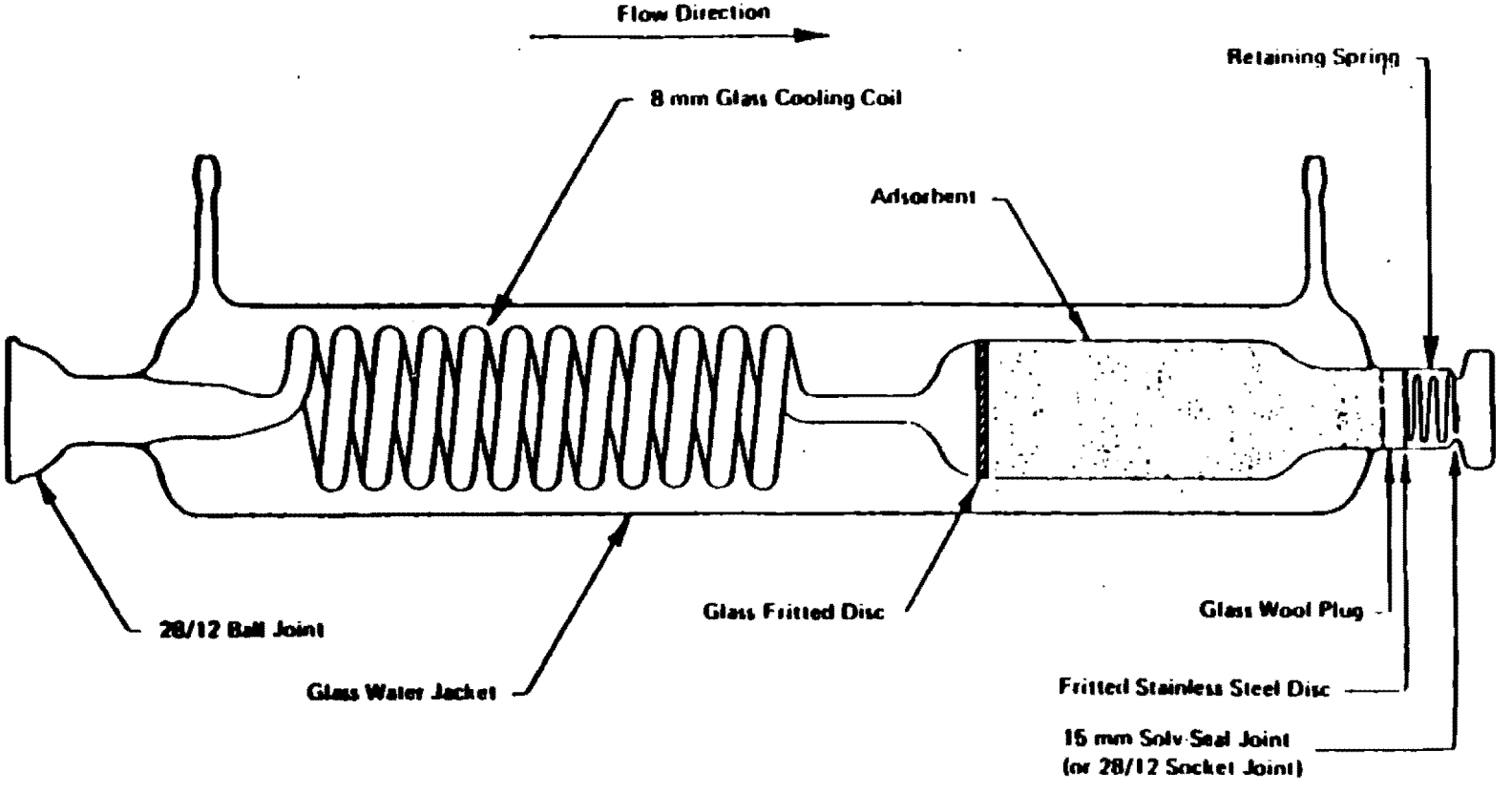


Figure 3. Adsorbent Sampling System.

Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see EPA Method 2, Figure 2-7). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

4.1.3.12 Calibration/field-preparation record: A permanently bound laboratory notebook, in which duplicate copies of data may be made as they are being recorded, is required for documenting and recording calibrations and preparation procedures (i.e., filter and silica gel tare weights, clean XAD-2, quality assurance/quality control check results, dry-gas meter, and thermocouple calibrations, etc.). The duplicate copies should be detachable and should be stored separately in the test program archives.

4.2 Sample Recovery:

4.2.1 **Probe liner:** Probe nozzle and organic module conditioning section brushes; nylon bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel, Teflon, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the organic module conditioning section.

4.2.2 **Wash bottles:** Three. Teflon or glass wash bottles are recommended; polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

4.2.3 **Glass sample storage containers:** Chemically resistant, borosilicate amber and clear glass bottles, 500-mL or 1,000-mL. Bottles should be tinted to prevent action of light on sample. Screw-cap liners shall be either Teflon or constructed so as to be leak-free and resistant to chemical attack by organic recovery solvents. Narrow-mouth glass bottles have been found to exhibit less tendency toward leakage.

4.2.4 **Petri dishes:** Glass, sealed around the circumference with wide (1-in.) Teflon tape, for storage and transport of filter samples.

4.2.5 **Graduated cylinder and/or balances:** To measure condensed water to the nearest 1 mL or 1 g. Graduated cylinders shall have subdivisions not >2 mL. Laboratory triple-beam balances capable of weighing to ±0.5 g or better are required.

4.2.6 **Plastic storage containers:** Screw-cap polypropylene or polyethylene containers to store silica gel.

4.2.7 **Funnel and rubber policeman:** To aid in transfer of silica gel to container (not necessary if silica gel is weighed in field).

4.2.8 Funnels: Glass, to aid in sample recovery.

4.3 Filters: Glass- or quartz-fiber filters, without organic binder, exhibiting at least 99.95% efficiency (<0.05% penetration) on 0.3-um dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Reeve Angel 934 AH or Schleicher and Schwell #3 filters work well under these conditions.

4.4 Crushed ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending on ambient air temperature.

4.5 Stopcock grease: Solvent-insoluble, heat-stable silicone grease. Use of silicone grease upstream of the module is not permitted, and amounts used on components located downstream of the organic module shall be minimized. Silicone grease usage is not necessary if screw-on connectors and Teflon sleeves or ground-glass joints are used.

4.6 Glass wool: Used to plug the unfritted end of the sorbent module. The glass-wool fiber should be solvent-extracted with methylene chloride in a Soxhlet extractor for 12 hr and air-dried prior to use.

5.0 REAGENTS

5.1 Adsorbent resin: Porous polymeric resin (XAD-2 or equivalent) is recommended. These resins shall be cleaned prior to their use for sample collection. Appendix A of this method should be consulted to determine appropriate precleaning procedure. For best results, resin used should not exhibit a blank of higher than 4 mg/kg of total chromatographable organics (TCO) (see Appendix B) prior to use. Once cleaned, resin should be stored in an airtight, wide-mouth amber glass container with a Teflon-lined cap or placed in one of the glass sorbent modules tightly sealed with Teflon film and elastic bands. The resin should be used within 4 wk of the preparation.

5.2 Silica gel: Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hr before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

5.3 Impinger solutions: Distilled organic-free water (Type II) shall be used, unless sampling is intended to quantify a particular inorganic gaseous species. If sampling is intended to quantify the concentration of additional species, the impinger solution of choice shall be subject to Administrator approval. This water should be prescreened for any compounds of interest. One hundred mL will be added to the specified impinger; the third impinger in the train may be charged with a basic solution (1 N sodium hydroxide or sodium acetate) to protect the sampling pump from acidic gases. Sodium acetate should be used when large sample volumes are anticipated because sodium hydroxide will react with carbon dioxide in aqueous media to form sodium carbonate, which may possibly plug the impinger.

5.4 Sample recovery reagents:

5.4.1 Methylene chloride: Distilled-in-glass grade is required for sample recovery and cleanup (see Note to 5.4.2 below).

5.4.2 Methyl alcohol: Distilled-in-glass grade is required for sample recovery and cleanup.

NOTE: Organic solvents from metal containers may have a high residue blank and should not be used. Sometimes suppliers transfer solvents from metal to glass bottles; thus blanks shall be run prior to field use and only solvents with low blank value (<0.001%) shall be used.

5.4.3 Water: Water (Type II) shall be used for rinsing the organic module and condenser component.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Because of complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

6.2 Laboratory preparation:

6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified.

6.2.2 Weigh several 200- to 300-g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

6.2.3 Check filters visually against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass Petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

6.2.4 Desiccate the filters at 20 ± 5.6°C (68 ± 10°F) and ambient pressure for at least 24 hr, and weigh at intervals of at least 6 hr to a constant weight (i.e., <0.5-mg change from previous weighing), recording results to the nearest 0.1 mg. During each weighing the filter must not be exposed for more than a 2-min period to the laboratory atmosphere and relative humidity above 50%. Alternatively (unless otherwise specified by the Administrator), the filters may be oven-dried at 105°C (220°F) for 2-3 hr, desiccated for 2 hr, and weighed.

6.3 Preliminary field determinations:

6.3.1 Select the sampling site and the minimum number of sampling points according to EPA Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. It is recommended that a leak-check of the pitot lines (see EPA Method 2, Section 3.1) be performed. Determine the stack-gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack-gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

6.3.2 Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of EPA Method 2).

6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

6.3.4 A minimum of 3 dscm (105.9 dscf) of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of POHCs from incineration systems. Additional sample volume shall be collected as necessitated by analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.0.

6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus one-half min.

6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-sample volumes. In these cases, the Administrator's approval must first be obtained.

6.4 Preparation of collection train:

6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.

6.4.2 Fill the sorbent trap section of the organic module with approximately 20 g of clean adsorbent resin. While filling, ensure that the trap packs uniformly, to eliminate the possibility of channeling. When freshly cleaned, many adsorbent resins carry a static charge, which will cause clinging to trap walls. This may be minimized by filling the trap in the presence of an antistatic device. Commercial antistatic devices include Model-204 and Model-210 manufactured by the 3M Company, St. Paul, Minnesota.

6.4.3 If an impinger train is used to collect moisture, place 100 mL of water in each of the first two impingers, leave the third impinger empty (or charge with caustic solution, as necessary), and transfer approximately 200-300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

6.4.4 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

6.4.5 When glass liners are used, install the selected nozzle using a Viton-A O-ring when stack temperatures are <260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connecting systems utilizing either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above, or by a leak-free direct mechanical connection. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

6.4.6 Set up the train as in Figure 1. During assembly, do not use any silicone grease on ground-glass joints that are located upstream of the organic module. A very light coating of silicone grease may be used on all ground-glass joints that are located downstream of the organic module, but it should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone-grease contamination. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and the filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack. The organic module condenser must be maintained at a temperature of 17 ± 3°C. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperature.

6.4.7 Place crushed ice around the impingers and the organic module condensate knockout.

6.4.8 Turn on the sorbent module and condenser coil coolant recirculating pump and begin monitoring the sorbent module gas entry temperature. Ensure proper sorbent module gas entry temperature before proceeding and again before any sampling is initiated. It is extremely important that the XAD-2 resin temperature never exceed 50°C (122°F), because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20°C (68°F) for efficient capture of the semivolatile species of interest.

6.4.9 Turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize.

6.5 Leak-check procedures

6.5.1 Pre-test leak-check:

6.5.1.1 Because the number of additional intercomponent connections in the Semi-VOST train (over the M5 Train) increases the possibility of leakage, a pre-test leak-check is required.

6.5.1.2 After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 381-mm Hg (15-in. Hg) vacuum.
(NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.)

6.5.1.3 If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first attaching a carbon-filled leak-check impinger (shown in Figure 4) to the inlet of the filter holder (cyclone, if applicable) and then plugging the inlet and pulling a 381-mm Hg (15-in. Hg) vacuum. (Again, a lower vacuum may be used, provided that it is not exceeded during the test.) Then, connect the probe to the train and leak-check at about 25-mm Hg (1-in. Hg) vacuum; alternatively, leak-check the probe with the rest of the sampling train in one step at 381-mm Hg (15-in. Hg) vacuum. Leakage rates in excess of 4% of the average sampling rate or >0.00057 m^3/min (0.02 cfm), whichever is less, are unacceptable.

6.5.1.4 The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with fine-adjust valve fully open and coarse-adjust valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do not reverse direction of the fine-adjust valve; this will cause water to back up into the organic module. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check, as shown below, and start over.

CROSS SECTIONAL VIEW

Leak Testing Apparatus

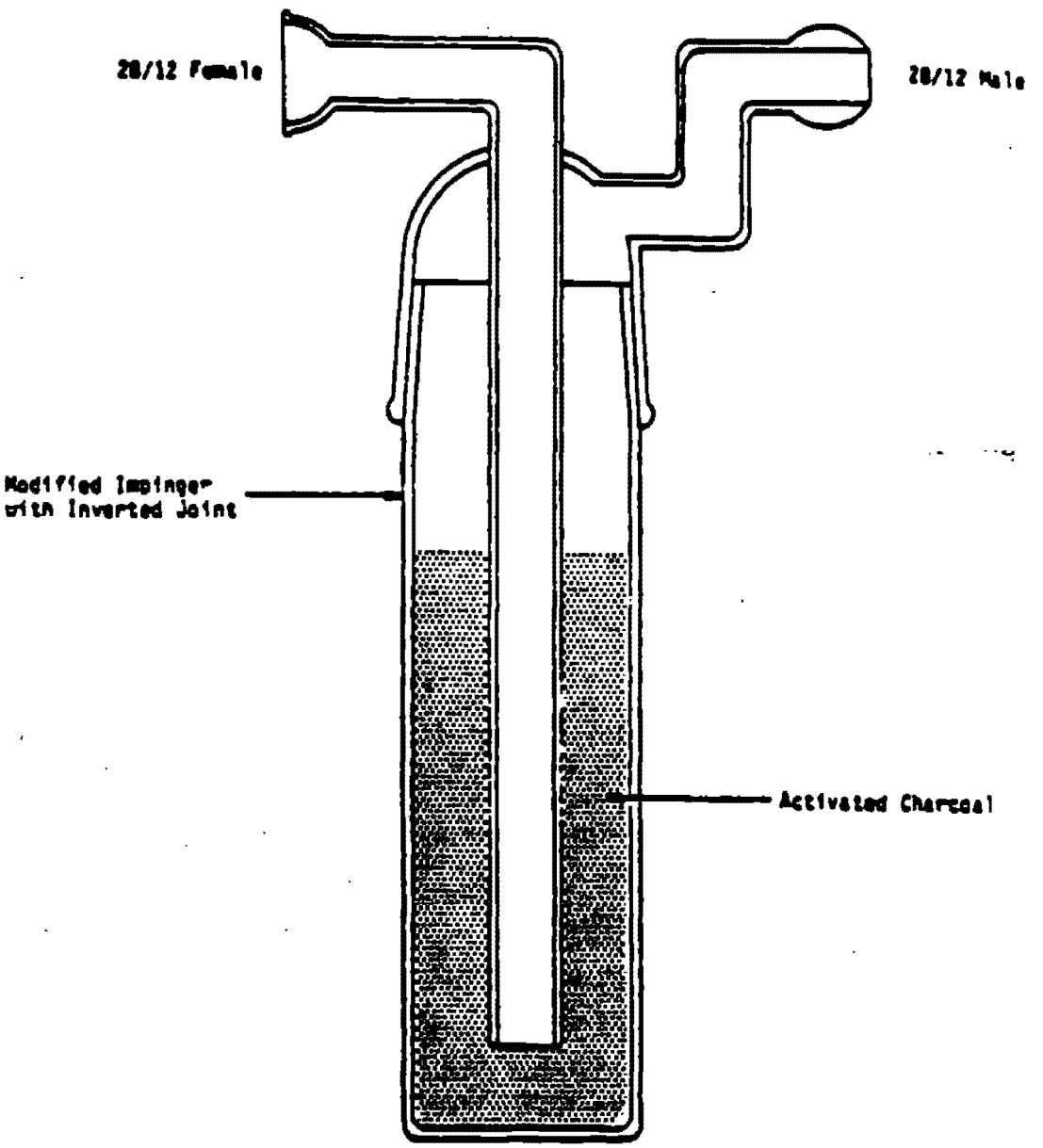


Figure 4. Leak-check impinger.

6.5.1.5 When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable). When the vacuum drops to 127 mm (5 in.) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed. This prevents the water in the impingers from being forced backward into the organic module and silica gel from being entrained backward into the third impinger.

6.5.2 Leak-checks during sampling run:

6.5.2.1 If, during the sampling run, a component (e.g., filter assembly, impinger, or sorbent trap) change becomes necessary, a leak-check shall be conducted immediately after the interruption of sampling and before the change is made. The leak-check shall be done according to the procedure outlined in Paragraph 6.5.1, except that it shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, the tester shall void the sampling run. (It should be noted that any "correction" of the sample volume by calculation by calculation reduces the integrity of the pollutant concentrations data generated and must be avoided.)

6.5.2.2 Immediately after a component change, and before sampling is reinitiated, a leak-check similar to a pre-test leak-check must also be conducted.

6.5.3 Post-test leak-check:

6.5.3.1 A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done with the same procedures as those with the pre-test leak-check, except that it shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate, correct the sample volume (as shown in the calculation section of this method), and consider the data obtained of questionable reliability, or void the sampling run.

6.6 Sampling-train operation:

6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, unless otherwise specified by the Administrator. Maintain a temperature around the filter of 120 ± 14°C (248 ± 25°F) and a gas temperature entering the sorbent trap at a maximum of 20°C (68°F).

6.6.2 For each run, record the data required on a data sheet such as the one shown in Figure 5. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5 at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity-head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

6.6.3 Clean the stack access ports prior to the test run to eliminate the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot-tube coefficient is 0.84 $\pm$ 0.02 and the stack-gas equivalent density (dry molecular weight) is equal to 29 $\pm$ 4. APTD-0576 details the procedure for using the nomographs. If the stack-gas molecular weight and the pitot-tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps (Shigehara, 1974) are taken to compensate for the deviations.

6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack, to prevent water from backing into the organic module. If necessary, the pump may be turned on with the coarse-adjust valve closed.

6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent unrepresentative dilution of the gas stream.

6.6.6 Traverse the stack cross section, as required by EPA Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

6.6.7 During the test run, make periodic adjustments to keep the temperature around the filter holder and the organic module at the proper levels; add more ice and, if necessary, salt to maintain a temperature of <20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

Plant _____

Location _____

Operator _____

Date _____

Run No. _____

Sample Box No. _____

Meter Box No. _____

Meter ΔH _____

C Factor _____

Pitot Tube Coefficient C_p _____

Schematic of Stack Cross Section

Ambient Temperature _____

Barometric Pressure _____

Assumed Moisture % _____

Probe Length, m (ft) _____

Nozzle Identification No. _____

Average Calibrated Nozzle Diameter, cm (in) _____

Probe Heater Setting _____

Leak Rate, m^3/min, (cfm) _____

Probe Liner Material _____

Static Pressure, mm Hg (in. Hg) _____

Filter No. _____

Traverse Point Number	Sampling Time (θ) min.	Vacuum mm Hg (in. Hg)	Stack Temperature (T_s) °C(°F)	Velocity Head (ΔP_s) mm (in) H_2O	Pressure Differential Across Orifice Meter mm (H_2O) (in H_2O)	Gas Sample Volume m_3 (ft_3)	Gas Sample Temp. at Dry Gas Meter Inlet °C(°F)	Gas Sample Temp. at Dry Gas Meter Outlet °C(°F)	Filter Holder Temperature °C(°F)	Temperature of Gas Entering Sorbet Trap °C(°F)	Temperature of Gas Leaving Condenser or Last Impinger
Total							Avg.	Avg.			
Average											

Figure 5. Particulate field data.

6.6.8 If the pressure drop across the filter or sorbent trap becomes too high, making isokinetic sampling difficult to maintain, the filter/sorbent trap may be replaced in the midst of a sample run. Using another complete filter holder/sorbent trap assembly is recommended, rather than attempting to change the filter and resin themselves. After a new filter/sorbent trap assembly is installed, conduct a leak-check. The total particulate weight shall include the summation of all filter assembly catches.

6.6.9 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

6.6.10 Note that when two or more trains are used, separate analysis of the front-half (if applicable) organic-module and impinger (if applicable) catches from each train shall be performed, unless identical nozzle sizes were used on all trains. In that case, the front-half catches from the individual trains may be combined (as may the impinger catches), and one analysis of front-half catch and one analysis of impinger catch may be performed.

6.6.11 At the end of the sample run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry-gas meter reading, and conduct a post-test leak-check. Also, leak-check the pitot lines as described in EPA Method 2. The lines must pass this leak-check in order to validate the velocity-head data.

6.6.12 Calculate percent isokineticity (see Section 10.8) to determine whether the run was valid or another test run should be made.

7.0 SAMPLE RECOVERY

7.1 Preparation:

7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling down because this will create a vacuum in the filter holder, drawing water from the impingers into the sorbent module.

7.1.2 Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the open outlet, being careful not to lose any condensate that might be present. Cap the filter inlet.

Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the organic module and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the organic module.

7.1.3 Cap the filter-holder outlet and the inlet to the organic module. Separate the sorbent trap section of the organic module from the condensate knockout trap and the gas-conditioning section. Cap all organic module openings. Disconnect the organic-module knockout trap from the impinger train inlet and cap both of these openings. Ground-glass stoppers, Teflon caps, or caps of other inert materials may be used to seal all openings.

7.1.4 Transfer the probe, the filter, the organic-module components, and the impinger/condenser assembly to the cleanup area. This area should be clean and protected from the weather to minimize sample contamination or loss.

7.1.5 Save a portion of all washing solutions (methanol/methylene chloride, Type II water) used for cleanup as a blank. Transfer 200 mL of each solution directly from the wash bottle being used and place each in a separate, prelabeled glass sample container.

7.1.6 Inspect the train prior to and during disassembly and note any abnormal conditions.

7.2 Sample containers:

7.2.1 **Container no. 1:** Carefully remove the filter from the filter holder and place it in its identified Petri dish container. Use a pair or pairs of tweezers to handle the filter. If it is necessary to fold the filter, ensure that the particulate cake is inside the fold. Carefully transfer to the Petri dish any particulate matter or filter fibers that adhere to the filter-holder gasket, using a dry nylon bristle brush or sharp-edged blade, or both. Label the container and seal with 1-in.-wide Teflon tape around the circumference of the lid.

7.2.2 **Container no. 2:** Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components first with methanol/methylene chloride (1:1 v/v) into a glass container. Distilled water may also be used. Retain a water and solvent blank and analyze in the same manner as with the samples. Perform rinses as follows:

7.2.2.1 Carefully remove the probe nozzle and clean the inside surface by rinsing with the solvent mixture (1:1 v/v methanol/-methylene chloride) from a wash bottle and brushing with a nylon bristle brush. Brush until the rinse shows no visible particles; then make a final rinse of the inside surface with the solvent mix. Brush and rinse the inside parts of the Swagelok fitting with the solvent mix in a similar way until no visible particles remain.

7.2.2.2 Have two people rinse the probe liner with the solvent mix by tilting and rotating the probe while squirting solvent into its upper end so that all inside surfaces will be wetted with solvent. Let the solvent drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container.

7.2.2.3 Follow the solvent rinse with a probe brush. Hold the probe in an inclined position and squirt solvent into the upper end while pushing the probe brush through the probe with a twisting action; place a sample container underneath the lower end of the probe and catch any solvent and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the solvent or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above-prescribed manner at least six times (metal probes have small crevices in which particulate matter can be entrapped). Rinse the brush with solvent and quantitatively collect these washings in the sample container. After the brushing, make a final solvent rinse of the probe as described above.

7.2.2.4 It is recommended that two people work together to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

7.2.2.5 Clean the inside of the front half of the filter holder and cyclone/cyclone flask, if used, by rubbing the surfaces with a nylon bristle brush and rinsing with methanol/methylene chloride (1:1 v/v) mixture. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone and cyclone flask (if applicable). Brush and rinse any particulate material adhering to the inner surfaces of these components into the front-half rinse sample. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents.

7.2.3 **Container no. 3:** The sorbent trap section of the organic module may be used as a sample transport container, or the spent resin may be transferred to a separate glass bottle for shipment. If the sorbent trap itself is used as the transport container, both ends should be sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon caps may be used. The sorbent trap should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If a separate bottle is used, the spent resin should be quantitatively transferred from the trap into the clean bottle. Resin that adheres to the walls of the trap should be recovered using a rubber policeman or spatula and added to this bottle.

7.2.4 **Container no. 4:** Measure the volume of condensate collected in the condensate knockout section of the organic module to within +1 mL by using a graduated cylinder or by weighing to within +0.5 g using a triple-beam balance. Record the volume or weight of liquid present and note any discoloration or film in the liquid catch. Transfer this liquid to a prelabeled glass sample container. Inspect the back half of the filter housing and the gas-conditioning section of the organic module. If condensate is observed, transfer it to a graduated or weighing bottle and measure the volume, as described above. Add this material to the condensate knockout-trap catch.

7.2.5 **Container no. 5:** All sampling train components located between the high-efficiency glass- or quartz-fiber filter and the first wet impinger or the final condenser system (including the heated Teflon line connecting the filter outlet to the condenser) should be thoroughly rinsed with methanol/methylene chloride (1:1 v/v) and the rinsings combined. This rinse shall be separated from the condensate. If the spent resin is transferred from the sorbent trap to a separate sample container for transport, the sorbent trap shall be thoroughly rinsed until all sample-wetted surfaces appear clean. Visible films should be removed by brushing. Whenever train components are brushed, the brush should be subsequently rinsed with solvent mixture and the rinsings added to this container.

7.2.6 **Container no. 6:** Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere strongly to the impinger wall. Because the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the container and its contents to 0.5 g or better.

7.3 Impinger water:

7.3.1 Make a notation of any color or film in the liquid catch. Measure the liquid in the first three impingers to within ±1 mL by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

7.3.2 Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Paragraph 4.1.3.7). Amber glass containers should be used for storage of impinger catch, if required.

7.3.3 If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

7.4 Sample preparation for shipment: Prior to shipment, recheck all sample containers to ensure that the caps are well secured. Seal the lids of all containers around the circumference with Teflon tape. Ship all liquid samples upright on ice and all particulate filters with the particulate catch facing upward. The particulate filters should be shipped unrefrigerated.

8.0 ANALYSIS

8.1 Sample preparation:

8.1.1 General: The preparation steps for all samples will result in a finite volume of concentrated solvent. The final sample volume (usually in the 1- to 10-mL range) is then subjected to analysis by GC/MS. All samples should be inspected and the appearance documented. All samples are to be spiked with surrogate standards as received from the field prior to any sample manipulations. The spike should be at a level equivalent to 10 times the MDL when the solvent is reduced in volume to the desired level (i.e., 10 mL). The spiking compounds should be the stable isotopically labeled analog of the compounds of interest or a compound that would exhibit properties similar to the compounds of interest, be easily chromatographed, and not interfere with the analysis of the compounds of interest. Suggested surrogate spiking compounds are: deuterated naphthalene, chrysene, phenol, nitrobenzene, chlorobenzene, toluene, and carbon-13-labeled pentachlorophenol.

8.1.2 Condensate: The "condensate" is the moisture collected in the first impinger following the XAD-2 module. Spike the condensate with the surrogate standards. The volume is measured and recorded and then transferred to a separatory funnel. The pH is to be adjusted to pH 2 with 6 N sulfuric acid, if necessary. The sample container and graduated cylinder are sequentially rinsed with three successive 10-mL aliquots of the extraction solvent and added to the separatory funnel. The ratio of solvent to aqueous sample should be maintained at 1:3. Extract the sample by vigorously shaking the separatory funnel for 5 min. After complete separation of the phases, remove the solvent and transfer to a Kuderna-Danish concentrator (K-D), filtering through a bed of precleaned, dry sodium sulfate. Repeat the extraction step two additional times. Adjust the pH to 11 with 6 N sodium hydroxide and reextract combining the acid and base extracts. Rinse the sodium sulfate into the K-D with fresh solvent and discard the desiccant. Add Teflon boiling chips and concentrate to 10 mL by reducing the volume to slightly less than 10 mL and then bringing to volume with fresh solvent. In order to achieve the necessary detection limit, the sample volume can be further reduced to 1 mL by using a micro column K-D or nitrogen blow-down. Should the sample start to exhibit precipitation, the concentration step should be stopped and the sample redissolved with fresh solvent taking the volume to some finite amount. After adding a standard (for the purpose of quantitation by GC/MS), the sample is ready for analysis, as discussed in Paragraph 8.2.

8.1.3 **Impinger:** Spike the sample with the surrogate standards; measure and record the volume and transfer to a separatory funnel. Proceed as described in Paragraph 8.1.2.

8.1.4 **XAD-2:** Spike the resin directly with the surrogate standards. Transfer the resin to the all-glass thimbles by the following procedure (care should be taken so as not to contaminate the thimble by touching it with anything other than tweezers or other solvent-rinsed mechanical holding devices). Suspend the XAD-2 module directly over the thimble. The glass frit of the module (see Figure 2) should be in the up position. The thimble is contained in a clean beaker, which will serve to catch the solvent rinses. Using a Teflon squeeze bottle, flush the XAD-2 into the thimble. Thoroughly rinse the glass module with solvent into the beaker containing the thimble. Add the XAD-2 glass-wool plug to the thimble. Cover the XAD-2 in the thimble with a precleaned glass-wool plug sufficient to prevent the resin from floating into the solvent reservoir of the extractor. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. If a question arises concerning the completeness of the extraction, a second extraction, without a spike, is advised. The thimble is placed in the extractor and the rinse solvent contained in the beaker is added to the solvent reservoir. Additional solvent is added to make the reservoir approximately two-thirds full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle 5-6 times per hr. Extract the resin for 16 hr. Transfer the solvent and three 10-mL rinses of the reservoir to a K-D and concentrate as described in Paragraph 8.1.2.

8.1.5 **Particulate filter (and cyclone catch):** If particulate loading is to be determined, weigh the filter (and cyclone catch, if applicable). The particulate filter (and cyclone catch, if applicable) is transferred to the glass thimble and extracted simultaneously with the XAD-2 resin.

8.1.6 **Train solvent rinses:** All train rinses (i.e., probe, impinger, filter housing) using the extraction solvent and methanol are returned to the laboratory as a single sample. If the rinses are contained in more than one container, the intended spike is divided equally among the containers proportioned from a single syringe volume. Transfer the rinse to a separatory funnel and add a sufficient amount of organic-free water so that the methylene chloride becomes immiscible and its volume no longer increases with the addition of more water. The extraction and concentration steps are then performed as described in Paragraph 8.1.2.

8.2 Sample analysis:

8.2.1 The primary analytical tool for the measurement of emissions from hazardous waste incinerators is GC/MS using fused-silica capillary GC columns, as described in Method 8270 in Chapter Four of this manual. Because of the nature of GC/MS instrumentation and the cost associated

with sample analysis, prescreening of the sample extracts by gas chromatography/flame ionization detection (GC/FID) or with electron capture (GC/ECD) is encouraged. Information regarding the complexity and concentration level of a sample prior to GC/MS analysis can be of enormous help. This information can be obtained by using either capillary columns or less expensive packed columns. However, the FID screen should be performed with a column similar to that used with the GC/MS. Keep in mind that GC/FID has a slightly lower detection limit than GC/MS and, therefore, that the concentration of the sample can be adjusted either up or down prior to analysis by GC/MS.

8.2.2 The mass spectrometer will be operated in a full scan (40-450) mode for most of the analyses. The range for which data are acquired in a GC/MS run will be sufficiently broad to encompass the major ions, as listed in Chapter Four, Method 8270, for each of the designated POHCs in an incinerator effluent analysis.

8.2.3 For most purposes, electron ionization (EI) spectra will be collected because a majority of the POHCs give reasonable EI spectra. Also, EI spectra are compatible with the NBS Library of Mass Spectra and other mass spectral references, which aid in the identification process for other components in the incinerator process streams.

8.2.4 To clarify some identifications, chemical ionization (CI) spectra using either positive ions or negative ions will be used to elucidate molecular-weight information and simplify the fragmentation patterns of some compounds. In no case, however, should CI spectra alone be used for compound identification. Refer to Chapter Four, Method 8270, for complete descriptions of GC conditions, MS conditions, and quantitative and quantitative identification.

9.0 CALIBRATION

9.1 Probe nozzle: Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

9.2 Pitot tube: The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of EPA Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked, dented, or corroded and if it meets design and intercomponent spacing specifications.

9.3 Metering system:

9.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: Make a 10-min calibration run at 0.00057 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

9.3.2 After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). The vacuum shall be set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

9.3.3 Leak-check of metering system: That portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 6): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13-18 cm (5-7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks, if present, must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series shall be voided or calculations for test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

9.4 Probe heater: The probe-heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

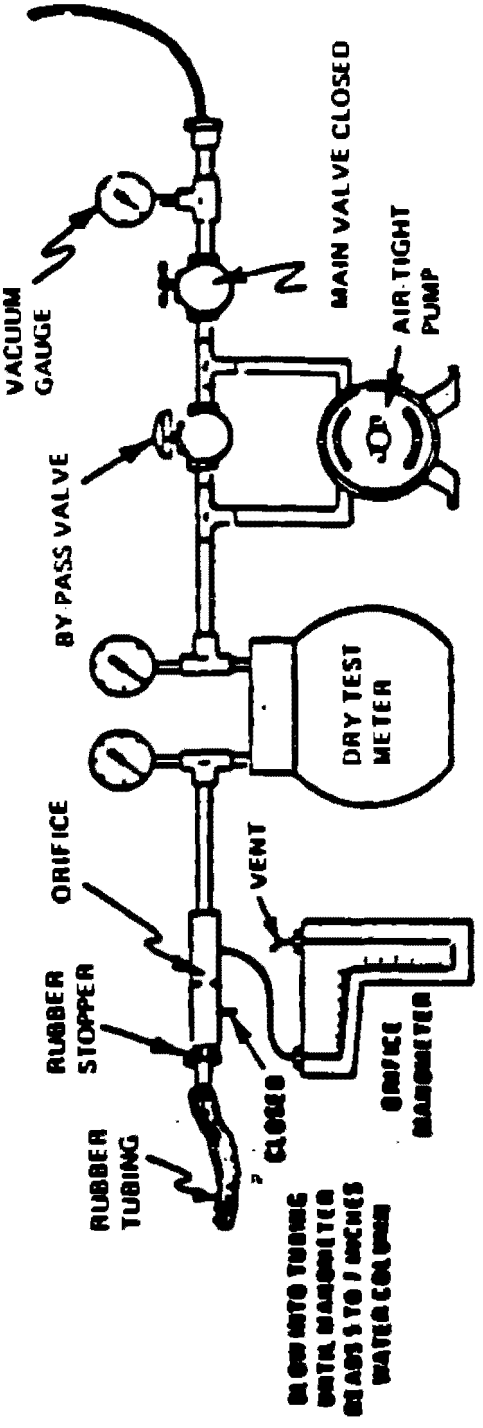


Figure 6. Leak-check of meter box.

9.5 Temperature gauges: Each thermocouple must be permanently and uniquely marked on the casting; all mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change >1.5%.

9.5.1 Impinger, organic module, and dry-gas meter thermocouples: For the thermocouples used to measure the temperature of the gas leaving the impinger train and the XAD-2 resin bed, three-point calibration at ice-water, room-air, and boiling-water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2°C (3.6°F) with those of the absolute value of the reference thermometer.

9.5.2 Probe and stack thermocouple: For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice-water, boiling-water, and hot-oil-bath temperatures must be performed; it is recommended that room-air temperature be added, and that the thermometer and the thermocouple agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

9.6 Barometer: Adjust the barometer initially and before each test series to agree to within ±25 mm Hg (0.1 in. Hg) of the mercury barometer or the corrected barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

9.7 Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class-S standard weights; the weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

10.0 CALCULATIONS

10.1 Carry out calculations. Round off figures after the final calculation to the correct number of significant figures.

10.2 Nomenclature:

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_d = Type S pitot tube coefficient (nominally 0.84 ± 0.02), dimensionless.

I = Percent of isokinetic sampling.

L_a = Maximum acceptable leakage rate for a leak-check, either pre-test or following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak-check conducted prior to the "i^{th}" component change (i = 1, 2, 3...n) m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

M_d = Stack-gas dry molecular weight, g/g-mole (lb/lb-mole).

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack-gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m^3/K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).

T_m = Absolute average dry-gas meter temperature (see Figure 6), K (°R).

T_s = Absolute average stack-gas temperature (see Figure 6), K (°R).

T_{std} = Standard absolute temperature, 293K (528°R).

V_{lc} = Total volume of liquid collected in the organic module condensate knockout trap, the impingers, and silica gel, mL.

V_m = Volume of gas sample as measured by dry-gas meter, dscm (dscf).

$V_{m(std)}$ = Volume of gas sample measured by the dry-gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

V_s = Stack-gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

γ = Dry-gas-meter calibration factor, dimensionless.

ΔH = Average pressure differential across the orifice meter (see Figure 2), mm H_2O (in. H_2O).

ρ_w = Density of water, 0.9982 g/mL (0.002201 lb/mL).

θ = Total sampling time, min.

θ_1 = Sampling time interval from the beginning of a run until the first component change, min.

θ_i = Sampling time interval between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p = Sampling time interval from the final (n^{th}) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

10.3 Average dry-gas-meter temperature and average orifice pressure drop: See data sheet (Figure 5, above).

10.4 Dry-gas volume: Correct the sample measured by the dry-gas meter to standard conditions (20°C, 760 mm Hg [68°F, 29.92 in. Hg]) by using Equation 1:

$$V_{m(std)} = V_m \gamma \frac{T_{std}}{T_m} \frac{P_{bar} + \Delta H/13.6}{P_{std}} = K_1 V_m \gamma \frac{P_{bar} + \Delta H/13.6}{T_m} \qquad (1)$$

where:

K_1 = 0.3858 K/mm Hg for metric units, or
K_1 = 17.64°R/in. Hg for English units.

It should be noted that Equation 1 can be used as written, unless the leakage rate observed during any of the mandatory leak-checks (i.e., the post-test leak-check or leak-checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 1 must be modified as follows:

a. Case I (no component changes made during sampling run): Replace V_m in Equation 1 with the expression:

$$V_m - (L_p - L_a)$$

b. Case II (one or more component changes made during the sampling run): Replace V_m in Equation 1 by the expression:

$$V_m - (L_1 - L_a)\theta_1 \quad - \sum_{i=2}^{n} (L_i - L_a)\theta_i \quad - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_i or L_p) that exceed L_a.

10.5 Volume of water vapor:

$$V_{w(std)} = V_{lc} \frac{P_w}{M_w} \frac{RT_{std}}{P_{std}} = K_2 V_{lc} \qquad (2)$$

where:

$K_2 = 0.001333\ m^3/mL$ for metric units, or
$K_2 = 0.04707\ ft^3/mL$ for English units.

10.6 Moisture content:

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad (3)$$

NOTE: In saturated or water-droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 3) and a second from the assumption of saturated conditions. The lower of the two values of B_w shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note to Section 1.2 of Method 4. For the purposes of this method, the average stack-gas temperature from Figure 1 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ±1°C (2°F).

10.7 Conversion factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

10.8 Isokinetic variation:

10.8.1 Calculation from raw data:

$$I = \frac{100\ T_s[K_3 F_{1c} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{60\theta V_s P_s A_n} \qquad (4)$$

where:

K_3 = 0.003454 mm Hg-m^3/mL-K for metric units, or
K_3 = 0.002669 in. Hg-ft^3/mL-°R for English units.

10.8.2 Calculation for intermediate values:

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(1-B_{ws})} \qquad (5)$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (1-B_{ws})}$$

where:

K_4 = 4.320 for metric units, or
K_4 = 0.09450 for English units.

10.8.3 Acceptable results: If 90% $\leq$ I $\leq$ 110%, the results are acceptable. If the results are low in comparison with the standard and I is beyond the acceptable range, or if I is less than 90%, the Administrator may opt to accept the results.

10.9 To determine the minimum sample volume that shall be collected, the following sequence of calculations shall be used.

10.9.1 From prior analysis of the waste feed, the concentration of POHCs introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the maximum amount of POHC allowed to be present in the effluent. This may be expressed as:

$$\frac{(WF)\ (POHC_1\ conc)}{100} \quad \frac{(100-\%DRE)}{100} = \text{Max } POHC_1 \text{ Mass} \qquad (6)$$

where:

WF = mass flow rate of waste feed per hr, g/hr (lb/hr).

$POHC_1$ = concentration of Principal Organic Hazardous Compound (wt %) introduced into the combustion process.

DRE = percent Destruction and Removal Efficiency required.

Max POHC = mass flow rate (g/hr [lb/hr]) of POHC emitted from the combustion source.

10.9.2 The average discharge concentration of the POHC in the effluent gas is determined by comparing the Max POHC with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary Method 1-4 determinations:

$$\frac{\text{Max POHC}_1 \text{ Mass}}{DV_{eff(std)}} = \text{Max POHC}_1 \text{ conc} \qquad (7)$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf).

$POHC_1$ conc = anticipated concentration of the POHC in the exhaust gas stream, g/dscm (lb/dscf).

10.9.3 In making this calculation, it is recommended that a safety margin of at least ten be included:

$$\frac{LDL_{POHC} \times 10}{POHC_1 \text{ conc}} = V_{TBC} \qquad (8)$$

where:

LDL_{POHC} = detectable amount of POHC in entire sampling train.
NOTE: The whole extract from an XAD-2 cartridge is seldom if ever, injected at once. Therefore, if aliquoting factors are involved, the LDL_{POHC} is not the same as the analytical (or column) detection limit.

V_{TBC} = minimum dry standard volume to be collected at dry-gas meter.

10.10 Concentration of any given POHC in the gaseous emissions of a combustion process:

1) Multiply the concentration of the POHC as determined in Method 8270 by the final concentration volume, typically 10 mL.

C_{POHC} (ug/mL) x sample volume (mL) = amount (ug) of POHC in sample (9)

where:

C_{POHC} = concentration of POHC as analyzed by Method 8270.

2) Sum the amount of POHC found in all samples associated with a single train.

Total (ug) = XAD-2 (ug) + condensate (ug) + rinses (ug) + impinger (ug) (10)

3) Divide the total ug found by the volume of stack gas sampled (m^3).

(Total ug)/(train sample volume) = concentration of POHC (ug/m^3) (11)

11.0 QUALITY CONTROL

11.1 Sampling: See EPA Manual 600/4-77-027b for Method 5 quality control.

11.2 Analysis: The quality assurance program required for this study includes the analysis of field and method blanks, procedure validations, incorporation of stable labeled surrogate compounds, quantitation versus stable labeled internal standards, capillary column performance checks, and external performance tests. The surrogate spiking compounds selected for a particular analysis are used as primary indicators of the quality of the analytical data for a wide range of compounds and a variety of sample matrices. The assessment of combustion data, positive identification, and quantitation of the selected compounds are dependent on the integrity of the samples received and the precision and accuracy of the analytical methods employed. The quality assurance procedures for this method are designed to monitor the performance of the analytical method and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

11.2.1 **Field Blanks:** Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters, and resin cartridges. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling area, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual test train). The filter housing and probe of the blank train will be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train.

11.2.2 **Method blanks:** A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

11.2.3 Refer to Method 8270 for additional quality control considerations.

12.0 METHOD PERFORMANCE

12.1 Method performance evaluation: Evaluation of analytical procedures for a selected series of compounds must include the sample-preparation procedures and each associated analytical determination. The analytical procedures should be challenged by the test compounds spiked at appropriate levels and carried through the procedures.

12.2 Method detection limit: The overall method detection limits (lower and upper) must be determined on a compound-by-compound basis because different compounds may exhibit different collection, retention, and extraction efficiencies as well as instrumental minimum detection limit (MDL). The method detection limit must be quoted relative to a given sample volume. The upper limits for the method must be determined relative to compound retention volumes (breakthrough).

12.3 Method precision and bias: The overall method precision and bias must be determined on a compound-by-compound basis at a given concentration level. The method precision value would include a combined variability due to sampling, sample preparation, and instrumental analysis. The method bias would be dependent upon the collection, retention, and extraction efficiency of the train components. From evaluation studies to date using a dynamic spiking system, method biases of -13% and -16% have been determined for toluene and 1,1,2,2-tetrachloroethane, respectively. A precision of 19.9% was calculated from a field test data set representing seven degrees of freedom which resulted from a series of paired, unspiked Semivolatile Organic Sampling trains (Semi-VOST) sampling emissions from a hazardous waste incinerator.

13.0 REFERENCES

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PREPARATION OF XAD-2 SORBENT RESIN

1.0 SCOPE AND APPLICATION

1.1 XAD-2 resin as supplied by the manufacturer is impregnated with a bicarbonate solution to inhibit microbial growth during storage. Both the salt solution and any residual extractable monomer and polymer species must be removed before use. The resin is prepared by a series of water and organic extractions, followed by careful drying.

2.0 EXTRACTION

2.1 Method 1: The procedure may be carried out in a giant Soxhlet extractor. An all-glass thimble containing an extra-coarse frit is used for extraction of XAD-2. The frit is recessed 10-15 mm above a crenellated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass-wool plug and stainless steel screen because it floats on methylene chloride. This process involves sequential extraction in the following order.

Solvent	Procedure
Water	Initial rinse: Place resin in a beaker, rinse once with Type II water, and discard. Fill with water a second time, let stand overnight, and discard.
Water	Extract with H_2O for 8 hr.
Methyl alcohol	Extract for 22 hr.
Methylene chloride	Extract for 22 hr.
Methylene chloride (fresh)	Extract for 22 hr.

2.2 Method 2:

2.2.1 As an alternative to Soxhlet extraction, a continuous extractor has been fabricated for the extraction sequence. This extractor has been found to be acceptable. The particular canister used for the apparatus shown in Figure A-1 contains about 500 g of finished XAD-2. Any size may be constructed; the choice is dependent on the needs of the sampling programs. The XAD-2 is held under light spring tension between a pair of coarse and fine screens. Spacers under the bottom screen allow for even distribution of clean solvent. The three-necked flask should be of sufficient size (3-liter in this case) to hold solvent

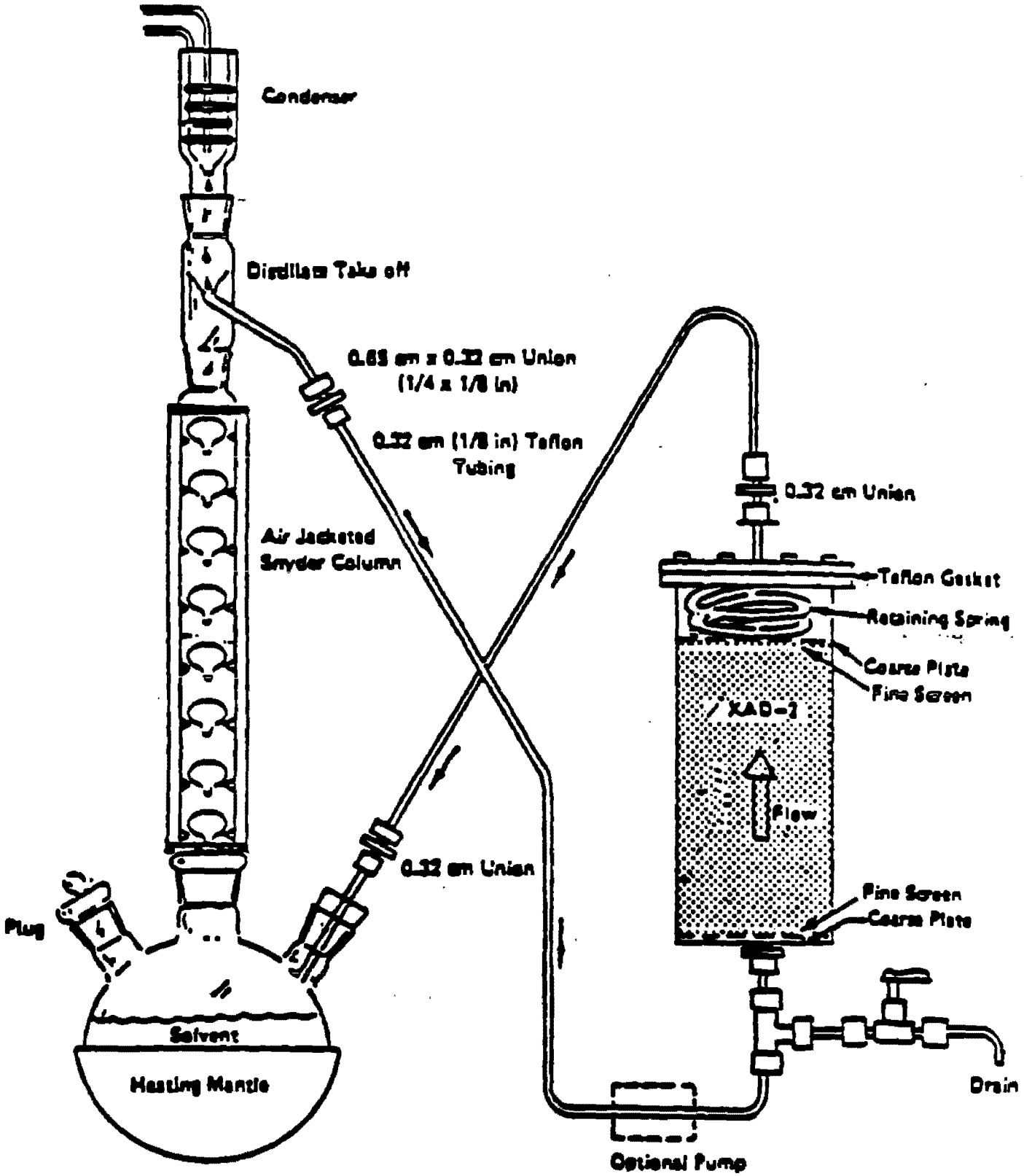

Figure A-1. XAD-2 cleanup extraction apparatus.

equal to twice the dead volume of the XAD-2 canister. Solvent is refluxed through the Snyder column, and the distillate is continuously cycled up through the XAD-2 for extraction and returned to the flask. The flow is maintained upward through the XAD-2 to allow maximum solvent contact and prevent channeling. A valve at the bottom of the canister allows removal of solvent from the canister between changes.

2.2.2 Experience has shown that it is very difficult to cycle sufficient water in this mode. Therefore the aqueous rinse is accomplished by simply flushing the canister with about 20 liters of distilled water. A small pump may be useful for pumping the water through the canister. The water extraction should be carried out at the rate of about 20-40 mL/min.

2.2.3 After draining the water, subsequent methyl alcohol and methylene chloride extractions are carried out using the refluxing apparatus. An overnight or 10- to 20-hr period is normally sufficient for each extraction.

2.2.4 All materials of construction are glass, Teflon, or stainless steel. Pumps, if used, should not contain extractable materials. Pumps are not used with methanol and methylene chloride.

3.0 DRYING

3.1 After evaluation of several methods of removing residual solvent, a fluidized-bed technique has proved to be the fastest and most reliable drying method.

3.2 A simple column with suitable retainers, as shown in Figure A-2, will serve as a satisfactory column. A 10.2-cm (4-in.) Pyrex pipe 0.6 m (2 ft) long will hold all of the XAD-2 from the extractor shown in Figure A-1 or the Soxhlet extractor, with sufficient space for fluidizing the bed while generating a minimum resin load at the exit of the column.

3.3 Method 1: The gas used to remove the solvent is the key to preserving the cleanliness of the XAD-2. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has routinely proved to be a reliable source of large volumes of gas free from organic contaminants. The liquid nitrogen cylinder is connected to the column by a length of precleaned 0.95-cm (3/8-in.) copper tubing, coiled to pass through a heat source. As nitrogen is bled from the cylinder, it is vaporized in the heat source and passes through the column. A convenient heat source is a water bath heated from a steam line. The final nitrogen temperature should only be warm to the touch and not over 40°C. Experience has shown that about 500 g of XAD-2 may be dried overnight by consuming a full 160-liter cylinder of liquid nitrogen.

3.4 Method 2: As a second choice, high-purity tank nitrogen may be used to dry the XAD-2. The high-purity nitrogen must first be passed through a bed

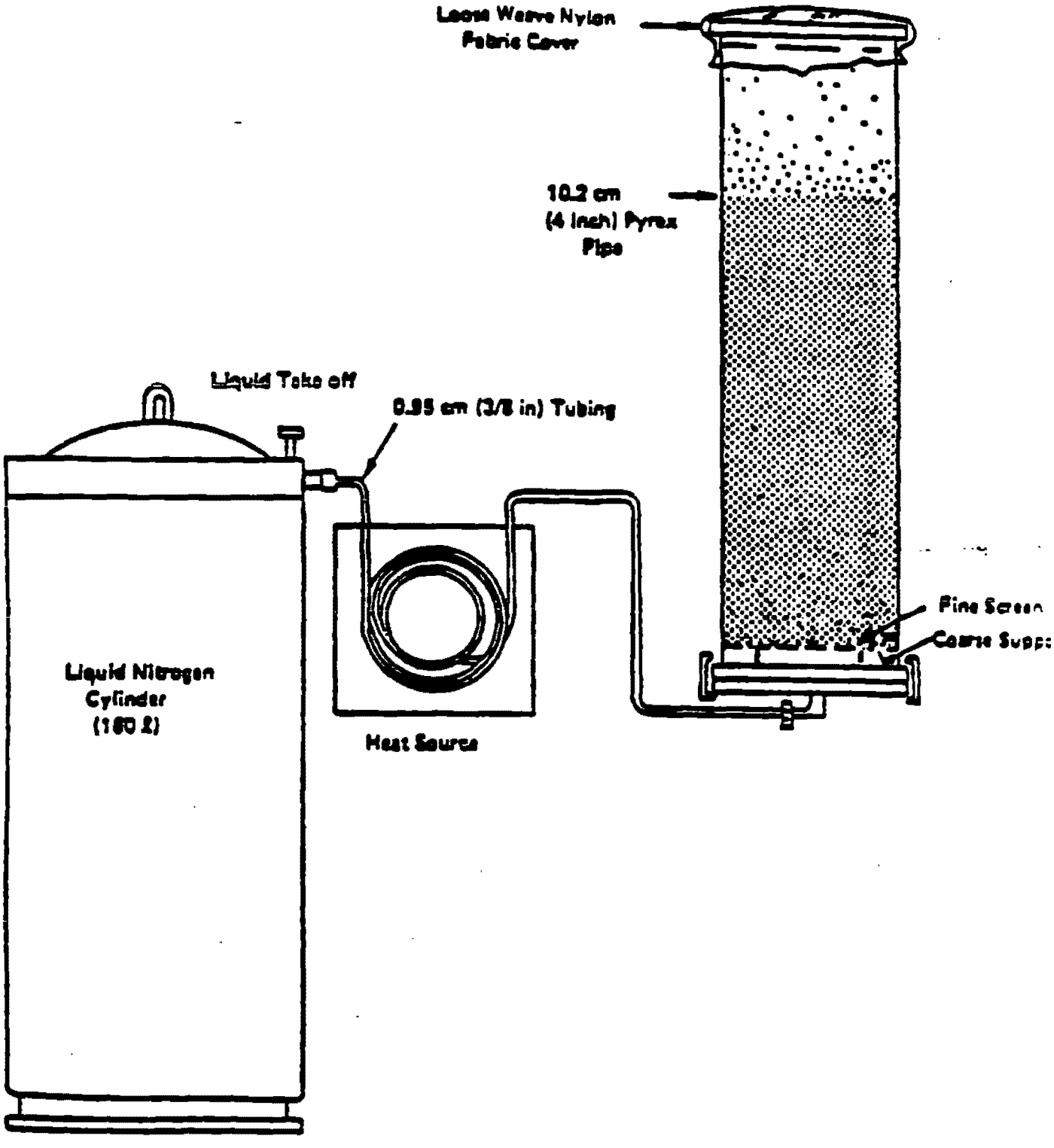

Figure A-2. XAD-2 fluidized-bed drying apparatus.

of activated charcoal approximately 150 mL in volume. With either type of drying method, the rate of flow should gently agitate the bed. Excessive fluidization may cause the particles to break up.

4.0 QUALITY CONTROL PROCEDURES

4.1 For both Methods 1 and 2, the quality control results must be reported for the batch. The batch must be reextracted if the residual extractable organics are >20 ug/mL by TCO analysis or the gravimetric residue is >0.5 mg/20 g XAD-2 extracted. (See also section 5.1, Method 0010.)

4.2 Four control procedures are used with the final XAD-2 to check for (1) residual methylene chloride, (2) extractable organics (TCO), (3) specific compounds of interest as determined by GC/MS, as described in Section 4.5 below, and (4) residue (GRAV).

4.3 Procedure for residual methylene chloride:

4.3.1 **Description:** A 1±0.1-g sample of dried resin is weighed into a small vial, 3 mL of toluene are added, and the vial is capped and well shaken. Five uL of toluene (now containing extracted methylene chloride) are injected into a gas chromatograph, and the resulting integrated area is compared with a reference standard. The reference solution consists of 2.5 uL of methylene chloride in 100 mL of toluene, simulating 100 ug of residual methylene chloride on the resin. The acceptable maximum content is 1,000 ug/g resin.

4.3.2 **Experimental:** The gas chromatograph conditions are as follows:

6-ft x 1/8-in. stainless steel column containing 10% OV-101 on 100/120 Supelcoport;

Helium carrier at 30 mL/min;

FID operated on 4 x 10^{-11} A/mV;

Injection port temperature: 250°C;

Detector temperature: 305°C;

Program: 30°C(4 min) 40°C/min 250°C (hold); and

Program terminated at 1,000 sec.

4.4 Procedure for residual extractable organics:

4.4.1 **Description:** A 20±0.1-g sample of cleaned, dried resin is weighed into a precleaned alundum or cellulose thimble which is plugged with cleaned glass wool. (Note that 20 g of resin will fill a thimble, and the

resin will float out unless well plugged.) The thimble containing the resin is extracted for 24 hr with 200-mL of pesticide- grade methylene chloride (Burdick and Jackson pesticide-grade or equivalent purity). The 200-mL extract is reduced in volume to 10-mL using a Kuderna-Danish concentrator and/or a nitrogen evaporation stream. Five uL of that solution are analyzed by gas chromatography using the TCO analysis procedure. The concentrated solution should not contain >20 ug/mL of TCO extracted from the XAD-2. This is equivalent to 10 ug/g of TCO in the XAD-2 and would correspond to 1.3 mg of TCO in the extract of the 130-g XAD-2 module. Care should be taken to correct the TCO data for a solvent blank prepared (200 mL reduced to 10 mL) in a similar manner.

4.4.2 **Experimental:** Use the TCO analysis conditions described in the revised Level 1 manual (EPA 600/7-78-201).

4.5 GC/MS Screen: The extract, as prepared in paragraph 4.4.1, is subjected to GC/MS analysis for each of the individual compounds of interest. The GC/MS procedure is described in Chapter Four, Method 8270. The extract is screened at the MDL of each compound. The presence of any compound at a concentration >25 ug/mL in the concentrated extract will require the XAD-2 to be recleaned by repeating the methylene chloride step.

4.6 Methodology for residual gravimetric determination: After the TCO value and GC/MS data are obtained for the resin batch by the above procedures, dry the remainder of the extract in a tared vessel. There must be <0.5 mg residue registered or the batch of resin will have to be extracted with fresh methylene chloride again until it meets this criterion. This level corresponds to 25 ug/g in the XAD-2, or about 3.25 mg in a resin charge of 130 g.

METHOD 0010, APPENDIX B

TOTAL CHROMATOGRAPHABLE ORGANIC MATERIAL ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 In this procedure, gas chromatography is used to determine the quantity of lower boiling hydrocarbons (boiling points between 90° and 300°C) in the concentrates of all organic solvent rinses, XAD-2 resin and LC fractions - when Method 1 is used (see References, Method 0010) - encountered in Level 1 environmental sample analyses. Data obtained using this procedure serve a twofold purpose. First, the total quantity of the lower boiling hydrocarbons in the sample is determined. Then whenever the hydrocarbon concentrations in the original concentrates exceed 75 ug/m^3, the chromatography results are reexamined to determine the amounts of individual species.

The extent of compound identification is limited to representing all materials as normal alkanes based upon comparison of boiling points. Thus the method is not qualitative. In a similar manner, the analysis is semiquantitative; calibrations are prepared using only one hydrocarbon. They are replicated but samples routinely are not.

1.2 Application: This procedure applies solely to the Level 1 C7-C16 gas chromatographic analysis of concentrates of organic extracts, neat liquids, and of LC fractions. Throughout the procedure, it is assumed the analyst has been given a properly prepared sample.

1.3 Sensitivity: The sensitivity of this procedure, defined as the slope of a plot of response versus concentration, is dependent on the instrument and must be verified regularly. TRW experience indicates the nominal range is of the order of 77 uV·V·sec·uL/ng of n-heptane and 79 uV·sec·ul/ng of n-hexadecane. The instrument is capable of perhaps one hundredfold greater sensitivity. The level specified here is sufficient for Level 1 analysis.

1.4 Detection limit: The detection limit of this procedure as written is 1.3 ng/uL for a 1 uL injection of n-decane. This limit is arbitrarily based on defining the minimum detectable response as 100 uv·sec. This is an easier operational definition than defining the minimum detection limit to be that amount of material which yields a signal twice the noise level.

1.5 Range: The range of the procedure will be concentrations of 1.3 ng/uL and greater.

1.6 Limitations

1.6.1 Reporting limitations: It should be noted that a typical environmental sample will contain compounds which: (a) will not elute in the specified boiling ranges and thus will not be reported, and/or (b)

will not elute from the column at all and thus will not be reported. Consequently, the organic content of the sample as reported is a lower bound and should be regarded as such.

1.6.2 **Calibration limitations:** Quantitation is based on calibration with n-decane. Data should therefore be reported as, e.g., mg C8/m^3 as n-decane. Since response varies linearly with carbon number (over a wide range the assumption may involve a 20% error), it is clear that heptane (C7) detected in a sample and quantitated as decane will be overestimated. Likewise, hexadecane (C16) quantitated as decane will be underestimated. From previous data, it is estimated the error involved is on the order of 6-7%.

1.6.3 **Detection limitations:** The sensitivity of the flame ionization detector varies from compound to compound. However, n-alkanes have a greater response than other classes. Consequently, using an n-alkane as a calibrant and assuming equal responses of all other compounds tends to give low reported values.

2.0 SUMMARY OF METHOD

2.1 A mL aliquot of all 10-mL concentrates is disbursed for GC-TCO analysis. With boiling point-retention time and response-amount calibration curves, the data (peak retention times and peak areas) are interpreted by first summing peak areas in the ranges obtained from the boiling point-retention time calibration. Then, with the response-amount calibration curve, the area sums are converted to amounts of material in the reported boiling point ranges.

2.2 After the instrument is set up, the boiling point-retention time calibration is effected by injecting a mixture of n-C7 through n-C16 hydrocarbons and operating the standard temperature program. Response-quantity calibrations are accomplished by injecting n-decane in n-pentane standards and performing the standard temperature program.

2.3 Definitions

2.3.1 **GC:** Gas chromatography or gas chromatograph.

2.3.2 **C7-C16 n-alkanes:** Heptane through hexadecane.

2.3.3 **GCA temperature program:** 4 min isothermal at 60°C, 10°C/min from 60° to 220°C.

2.3.4 **TRW temperature program:** 5 min isothermal at room temperature, then program from 30°C to 250°C at 15°C/min.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: This procedure is intended for use on a Varian 1860 gas chromatograph, equipped with dual flame ionization detectors and a linear temperature programmer. Any equivalent instrument can be used provided that electrometer settings, etc., be changed appropriately.

4.2 Gases:

4.2.1 Helium: Minimum quality is reactor grade. A 4A or 13X molecular sieve drying tube is required. A filter must be placed between the trap and the instrument. The trap should be recharged after every third tank of helium.

4.2.2 Air: Zero grade is satisfactory.

4.2.3 Hydrogen: Zero grade.

4.3 Syringe: Syringes are Hamilton 701N, 10 uL, or equivalent.

4.4 Septa: Septa will be of such quality as to produce very low bleed during the temperature program. An appropriate septum is Supelco Microsep 138, which is Teflon-backed. If septum bleed cannot be reduced to a negligible level, it will be necessary to install septum swingers on the instrument.

4.5 Recorder: The recorder of this procedure must be capable of not less than 1 mV full-scale display, a 1-sec time constant and 0.5 in. per min chart rate.

4.6 Integrator: An integrator is required. Peak area measurement by hand is satisfactory but too time-consuming. If manual integration is required, the method of "height times width at half height" is used.

4.7 Columns:

4.7.1 Preferred column: 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-101 on 100/120 mesh Supelcoport.

4.7.2 Alternate column: 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-1 (or other silicon phase) on 100/120 mesh Supelcoport.

4.8 Syringe cleaner: Hamilton syringe cleaner or equivalent connected to a suitable vacuum source.

5.0 REAGENTS

5.1 Pentane: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for standards and for syringe cleaning.

5.2 Methylene chloride: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for syringe cleaning.

6.0 SAMPLING HANDLING AND PRESERVATION

6.1 The extracts are concentrated in a Kuderna-Danish evaporator to a volume less than 10 mL. The concentrate is then quantitatively transferred to a 10-mL volumetric flask and diluted to volume. A 1-mL aliquot is taken for both this analysis and possible subsequent GC/MS analysis and set aside in the sample bank. For each GC-TCO analysis, obtain the sample sufficiently in advance to allow it to warm to room temperature. For example, after one analysis is started, return that sample to the sample bank and take the next sample.

7.0 PROCEDURES

7.1 Setup and checkout: Each day, the operator will verify the following:

7.1.1 That supplies of carrier gas, air and hydrogen are sufficient, i.e., that each tank contains > 100 psig.

7.1.2 That, after replacement of any gas cylinder, all connections leading to the chromatograph have been leak-checked.

7.1.3 That the carrier gas flow rate is 30 ± 2 mL/min, the hydrogen flow rate is 30 ± 2 mL/min, and the air flow rate is 300 ± 20 mL/min.

7.1.4 That the electrometer is functioning properly.

7.1.5 That the recorder and integrator are functioning properly.

7.1.6 That the septa have been leak-checked (leak-checking is effected by placing the soap bubble flow meter inlet tube over the injection port adaptors), and that no septum will be used for more than 20 injections.

7.1.7 That the list of samples to be run is ready.

7.2 Retention time calibration:

7.2.1 To obtain the temperature ranges for reporting the results of the analyses, the chromatograph is given a normal boiling point-retention time calibration. The n-alkanes, their boiling points, and data reporting ranges are given in the table below:

	NBP, °C	Reporting Range, °C	Report As
n-heptane	98	90-110	C7
n-octane	126	110-140	C8
n-nonane	151	140-160	C9
n-decane	174	160-180	C10
n-undecane	194	180-200	C11
n-dodecane	214	200-220	C12
n-tridecane	234	220-240	C13
n-tetradecane	252	240-260	C14
n-pentadecane	270	260-280	C15
n-hexadecane	288	280-300	C16

7.2.2 **Preparation of standards:** Preparing a mixture of the C7-C16 alkanes is required. There are two approaches: (1) use of a standards kit (e.g., Polyscience Kit) containing bottles of mixtures of selected n-alkanes which may be combined to produce a C7-C16 standard; or (2) use of bottles of the individual C7-C16 alkanes from which accurately known volumes may be taken and combined to give a C7-C16 mixture.

7.2.3 **Procedure for retention time calibration:** This calibration is performed at the start of an analytical program; the mixture is chromatographed at the start of each day. To attain the required retention time precision, both the carrier gas flow rate and the temperature program specifications must be observed. Details of the procedure depend on the instrument being used. The general procedure is as follows:

7.2.3.1 Set the programmer upper limit at 250°C. If this setting does not produce a column temperature of 250°C, find the correct setting.

7.2.3.2 Set the programmer lower limit at 30°C.

7.2.3.3 Verify that the instrument and samples are at room temperature.

7.2.3.4 Inject 1 uL of the n-alkane mixture.

7.2.3.5 Start the integrator and recorder.

7.2.3.6 Allow the instrument to run isothermally at room temperature for five min.

7.2.3.7 Shut the oven door.

7.2.3.8 Change the mode to Automatic and start the temperature program.

7.2.3.9 Repeat Steps 1-9 a sufficient number of times so that the relative standard deviation of the retention times for each peak is <5%.

7.3 Response calibration:

7.3.1 For the purposes of a Level 1 analysis, response-quantity calibration with n-decane is adequate. A 10-uL volume of n-decane is injected into a tared 10 mL volumetric flask. The weight injected is obtained and the flask is diluted to the mark with n-pentane. This standard contains about 730 ng n-decane per uL n-pentane. The exact concentration depends on temperature, so that a weight is required. Two serial tenfold dilutions are made from this standard, giving standards at about 730, 73, and 7.3 ng n-decane per uL n-pentane, respectively.

7.3.2 Procedure for response calibration: This calibration is performed at the start of an analytical program and monthly thereafter. The most concentrated standard is injected once each day. Any change in calibration necessitates a full calibration with new standards. Standards are stored in the refrigerator locker and are made up monthly.

7.3.2.1 Verify that the instrument is set up properly.

7.3.2.2 Set electrometer at 1 x 10^{-10} A/mV.

7.3.2.3 Inject 1 uL of the highest concentration standard.

7.3.2.4 Run standard temperature program as specified above.

7.3.2.5 Clean syringe.

7.3.2.6 Make repeated injections of all three standards until the relative standard deviations of the areas of each standard are ≤5%.

7.4 Sample analysis procedure:

7.4.1 The following apparatus is required:

7.4.1.1 Gas chromatograph set up and working.

7.4.1.2 Recorder, integrator working.

7.4.1.3 Syringe and syringe cleaning apparatus.

7.4.1.4 Parameters: Electrometer setting is 1 x 10^{-10} A/mV; recorder is set at 0.5 in./min and 1 mV full-scale.

7.4.2 Steps in the procedure are:

7.4.2.1 Label chromatogram with the data, sample number, etc.

7.4.2.2 Inject sample.

7.4.2.3 Start integrator and recorder.

7.4.2.4 After isothermal operation for 5 min, begin temperature program.

7.4.2.5 Clean syringe.

7.4.2.6 Return sample; obtain new sample.

7.4.2.7 When analysis is finished, allow instrument to cool. Turn chromatogram and integrator output and data sheet over to data analyst.

7.5 Syringe cleaning procedure:

7.5.1 Remove plunger from syringe.

7.5.2 Insert syringe into cleaner; turn on aspirator.

7.5.3 Fill pipet with pentane; run pentane through syringe.

7.5.4 Repeat with methylene chloride from a separate pipet.

7.5.5 Flush plunger with pentane followed by methylene chloride.

7.5.6 Repeat with methylene chloride.

7.6 Sample analysis decision criterion: The data from the TCO analyses of organic extract and rinse concentrates are first used to calculate the total concentration of C7-C16 hydrocarbon-equivalents (Paragraph 7.7.3) in the sample with respect to the volume of air actually sampled, i.e., ug/m^3. On this basis, a decision is made both on whether to calculate the quantity of each n-alkane equivalent present and on which analytical procedural pathway will be followed. If the total organic content is great enough to warrant continuing the analysis -- >500 ug/m^3 -- a TCO of less than 75 ug/m^3 will require only LC fractionation and gravimetric determinations and IR spectra to be obtained on each fraction. If the TCO is greater than 75 ug/m^3, then the first seven LC fractions of each sample will be reanalyzed using this same gas chromatographic technique.

7.7 Calculations:

7.7.1 **Boiling Point - Retention Time Calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.1.1 Average the retention times and calculate relative standard deviations for each n-hydrocarbon.

7.7.1.2 Plot average retention times as abscissae versus normal boiling points as ordinates.

7.7.1.3 Draw in calibration curve.

7.7.1.4 Locate and record retention times corresonding to boiling ranges 90-100, 110-140, 140-160, 160-180, 180-200, 200-220, 220-240, 240-260, 260-280, 280-300°C.

7.7.2 **Response-amount calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.2.1 Average the area responses of each standard and calculate relative standard deviations.

7.7.2.2 Plot response (uv·sec) as ordinate versus ng/uL as abscissa.

7.7.2.3 Draw in the curve. Perform least squares regression and obtain slope (uV·sec·uL/ng).

7.7.3 **Total C7-C16 hydrocarbons analysis:** The required data for this calculation are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.3.1 Sum the areas of all peaks within the retention time range of interest.

7.7.3.2 Convert this area (uV·sec) to ng/uL by dividing by the weight response for n-decane (uV·sec.uL/ng).

7.7.3.3 Multiply this weight by the total concentrate volume (10 mL) to get the weight of the C7-C16 hydrocarbons in the sample.

7.7.3.4 Using the volume of gas sampled or the total weight of sample acquired, convert the result of Step 7.7.3.3 above to ug/m^3.

7.7.3.5 If the value of total C7-C16 hydrocarbons from Step 7.7.3.4 above exceeds 75 ug/m^3, calculate individual hydrocarbon concentrations in accordance with the instructions in Paragraph 7.7.5.5 below.

7.7.4 **Individual C7-C16 n-Alkane Equivalent Analysis:** The required data from the analyses are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.4.1 Sum the areas of peaks in the proper retention time ranges.

7.7.4.2 Convert areas (uV·sec) to ng/uL by dividing by the proper weight response (uV·sec·uL/ng).

7.7.4.3 Multiply each weight by total concentrate volume (10 mL) to get weight of species in each range of the sample.

7.7.4.4 Using the volume of gas sampled on the total weight of sample acquired, convert the result of Step 7.7.4.3 above to ug/m^3.

8.0 QUALITY CONTROL

8.1 Appropriate QC is found in the pertinent procedures throughout the method.

9.0 METHOD PERFORMANCE

9.1 Even relatively comprehensive error propagation analysis is beyond the scope of this procedure. With reasonable care, peak area reproducibility of a standard should be of the order of 1% RSD. The relative standard deviation of the sum of all peaks in a fairly complex waste might be of the order of 5-10%. Accuracy is more difficult to assess. With good analytical technique, accuracy and precision should be of the order of 10-20%.

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APPENDIX J.5

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Method 18—Measurement of Gaseous Organic Compound Emissions by Gas Chromatography

Introduction

This method should not be attempted by persons unfamiliar with the performance characteristics of gas chromatography, nor by those persons who are unfamiliar with source sampling. Particular care should be exercised in the area of safety concerning choice of equipment and operation in potentially explosive atmospheres.

1. *Applicability and Principle*

1.1 Applicability. This method applies to the analysis of approximately 90 percent of the total gaseous organics emitted from an industrial source. It does not include techniques to identify and measure trace amounts of organic compounds, such as those found in building air and fugitive emission sources.

This method will not determine compounds that (1) are polymeric (high molecular weight), (2) can polymerize before analysis, or (3) have very low vapor pressures at stack or instrument conditions.

1.2 Principle.

The major organic components of a gas mixture are separated by gas chromatography (GC) and individually quantified by flame ionization, photoionization, electron capture, or other appropriate detection principles.

The retention times of each separated component are compared with those of known compounds under identical conditions. Therefore, the analyst confirms the identity and approximate concentrations of the organic emission components beforehand. With this information, the analyst then prepares or purchases commercially available standard mixtures to calibrate the GC under conditions identical to those of the samples. The analyst also determines the need for sample dilution to avoid detector saturation, gas stream filtration to eliminate particulate matter, and prevention of moisture condensation.

2. *Range and Sensitivity*

2.1 Range. The range of this method is from about 1 part per million (ppm) to the upper limit governed by GC detector saturation or column overloading. The upper limit can be extended by diluting the stack gases with an inert gas or by using smaller gas sampling loops.

2.2 Sensitivity. The sensitivity limit for a compound is defined as the minimum detectable concentration of that compound, or the concentration that produces a signal-to-noise ratio of three to one. The minimum detectable concentration is determined during the presurvey calibration for each compound.

3. *Precision and Accuracy*

Gas chromatographic techniques typically provide a precision of 5 to 10 percent relative standard deviation (RSD), but an experienced GC operator with a reliable instrument can readily achieve 5 percent RSD. For this method, the following combined GC/operator values are required.

(a) Precision. Duplicate analyses are within 5 percent of their mean value.

(b) Accuracy. Analysis results of prepared audit samples are within 10 percent of preparation values.

4. *Interferences*

Resolution interferences that may occur can be eliminated by appropriate GC column and detector choice or by shifting the retention times through changes in the column flow rate and the use of temperature programming.

The analytical system is demonstrated to be essentially free from contaminants by periodically analyzing blanks that consist of hydrocarbon-free air or nitrogen.

Sample cross-contamination that occurs when high level and low level samples or standards are analyzed alternately, is best

dealt with by thorough purging of the GC sample loop between samples.

To assure consistent detector response, calibration gases are contained in dry air. To adjust gaseous organic concentrations when water vapor is present in the sample, water vapor concentrations are determined for those samples, and a correction factor is applied.

5 *Presurvey and Presurvey Sampling.*

Perform a presurvey for each source to be tested. Refer to Figure 18-1. Some of the information can be collected from literature surveys and source personnel. Collect gas samples that can be analyzed to confirm the identities and approximate concentrations of the organic emissions.

5.1 Apparatus. This apparatus list also applies to Sections 6 and 7.

5.1.1 Teflon Tubing. (Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Diameter and length determined by connection requirements of cylinder regulators and the GC. Additional tubing is necessary to connect the GC sample loop to the sample.

5.1.2 Gas Chromatograph. GC with suitable detector, columns, temperature-controlled sample loop and valve assembly, and temperature programmable oven, if necessary. The GC shall achieve sensitivity requirements for the compounds under study.

5.1.3 Pump. Capable of pumping 100 ml/min. For flushing sample loop.

5.1.4 Flowmeters. To measure flow rates.

5.1.5 Regulators. Used on gas cylinders for GC and for cylinder standards.

5.1.6 Recorder. Recorder with linear strip chart is minimum acceptable. Integrator (optional) is recommended.

5.1.7 Syringes. 0.5-ml, 1.0- and 10-microliter sizes, calibrated, maximum accuracy (gas tight), for preparing calibration standards. Other appropriate sizes can be used.

5.1.8 Tubing Fittings. To plumb GC and gas cylinders.

5.1.9 Septums. For syringe injections.

5.1.10 Glass Jars. If necessary, clean-colored glass jars with Teflon-lined lids for condensate sample collection. Size depends on volume of condensate.

5.1.11 Soap Film Flow Meter. To determine flow rates.

5.1.12 Tedlar Bags. 10- and 50-liter capacity, for preparation of standards.

5.1.13 Dry Gas Meter with Temperature and Pressure Gauges. Accurate to ±2 percent, for perparation of gas standards.

5.1.14 Midget Impinger/Hot Plate Assembly. For preparation of gas standards.

5.1.15 Sample Flasks. For presurvey samples, must have gas-tight seals.

5.1.16 Adsorption Tubes. If necessary, blank tubes filled with necessary adsorbent (charcoal, Tenax, XAD-2, etc.) for presurvey samples.

5.1.17 Personnel Sampling Pump. Calibrated, for collecting adsorbent tube presurvey samples.

5.1.18 Dilution System. Calibrated, the dilution system is to be constructed following the specifications of an acceptable method.

5.1.19 Sample Probes. Pyrex or stainless steel, of sufficient length to reach centroid of stack, or a point no closer to the walls than 1 m.

5.1.20 Barometer. To measure barometric pressure.

5.2 Reagents.

5.2.1 Deionized Distilled Water.

5.2.2 Methylene Dichloride.

5.2.3 Calibration Gases. A series of standards prepared for every compound of interest.

5.2.4 Organic Compound Solutions. Pure (99.9 percent), or as pure as can reasonably be obtained, liquid samples of all the organic compounds needed to prepare calibration standards.

5.2.5 Extraction Solvents. For extraction of adsorbent tube samples in preparation for analysis.

5.2.6 Fuel. As recommended by the manufacturer for operation of the GC.

5.2.7 Carrier Gas. Hydrocarbon free, as recommended by the manufacturer for operation of the detector and compatability with the column.

5.2.8 Zero Gas. Hydrocarbon free air or nitrogen, to be used for dilutions, blank preparation, and standard preparation.

5.3 Sampling.

5.3.1 Collection of Samples with Glass Sampling Flasks. Presurvey samples can be collected in precleaned 250-ml double-ended glass sampling flasks. Teflon stopcocks, without grease, are preferred. Flasks should be cleaned as follows: Remove the stopcocks from both ends of the flasks, and wipe the parts to remove any grease. Clean the stopcocks, barrels, and receivers with methylene dichloride. Clean all glass ports with a soap solution, then rinse with tap and deionized distilled water. Place the flask in a cool glass annealing furnace and apply heat up to 500° C. Maintain at this temperature for 1 hour. After this time period, shut off and open the furnace to allow the flask to cool. Grease the stopcocks with stopcock grease and return them to the flask receivers. Purge the assembly with high-purity nitrogen for 2 to 5 minutes. Close off the stopcocks after purging to maintain a slight positive nitrogen pressure. Secure the stopcocks with tape.

Presurvey samples can be obtained either by drawing the gases into the previously evacuated flask or by drawing the gases into

and purging the flask with a rubber suction bulb.

5.3.1.1 Evacuated Flask Procedure. Use a high-vacuum pump to evacuate the flask to the capacity of the pump, then close off the stopcock leading to the pump. Attach a 6-mm outside diameter (OD) glass tee to the flask inlet with a short piece of Teflon tubing. Select a 6-mm OD borosilicate sampling probe, enlarged at one end to a 12-mm OD and of sufficient length to reach the centroid of the duct to be sampled. Insert a glass wool plug in the enlarged end of the probe to remove particulate matter. Attach the other end of the probe to the tee with a short piece of Teflon tubing. Connect a rubber suction bulb to the third leg of the tee. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and purge the probe with the rubber suction bulb. After the probe is completely purged and filled with duct gases, open the stopcock to the grab flask until the pressure in the flask reaches duct pressure. Close off the stopcock, and remove the probe from the duct. Remove the tee from the flask and tape the stopcocks to prevent leaks during shipment. Measure and record the duct temperature and pressure.

5.3.1.2 Purged Flask Procedure. Attach one end of the sampling flask to a rubber suction bulb. Attach the other end to a 6-mm OD glass probe as described in Section 5.3.1.1. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and apply suction with the bulb to completely purge the probe and flask. After the flask has been purged, close off the stopcock near the suction bulb, and then close the stopcock near the probe. Remove the probe from the duct, and disconnect both the probe and suction bulb. Tape the stopcocks to prevent leakage during shipment. Measure and record the duct temperature and pressure.

5.3.2 Flexible Bag Procedure. Tedlar or aluminized Mylar bags can also be used to obtain the presurvey sample. Use new bags, and leak check them before field use. In addition, check the bag before use for contamination by filling it with nitrogen or air, and analyzing the gas by GC at high sensitivity. Experience indicates that it is desirable to allow the inert gas to remain in the bag about 24 hours or longer to check for desorption of organics from the bag. Follow the leak check and sample collection procedures given in Section 7.1.

5.3.3 Determination of Moisture Content. For combustion or water-controlled processes, obtain the moisture content from plant personnel or by measurement during the presurvey. If the source is below 59° C, measure the wet bulb and dry bulb temperatures, and calculate the moisture content using a psychrometric chart. At higher temperatures, use Method 4 to determine the moisture content.

5.4 Determination of Static Pressure. Obtain the static pressure from the plant personnel or measurement. If a type S pitot tube and an inclined manometer are used, take care to align the pitot tube 90° from the direction of the flow. Disconnect one of the tubes to the manometer, and read the static pressure; note whether the reading is positive or negative.

5.5 Collection of Presurvey Samples with Adsorption Tube. Follow Section 7.4 for presurvey sampling.

6. *Analysis Development*

6.1 Selection of GC Parameters.

6.1.1 Column Choice. Based on the initial contact with plant personnel concerning the plant process and the anticipated emissions, choose a column that provides good resolution and rapid analysis time. The choice of an appropriate column can be aided by a literature search, contact with manufacturers of GC columns, and discussion with personnel at the emission source.

Most column manufacturers keep excellent records of their products. Their technical service departments may be able to recommend appropriate columns and detector type for separating the anticipated compounds, and they may be able to provide information on interferences, optimum operating conditions, and column limitations.

Plants with analytical laboratories may also be able to provide information on appropriate analytical procedures.

6.1.2 Preliminary GC Adjustment. Using the standards and column obtained in Section 6.1.1, perform initial tests to determine appropriate GC conditions that provide good resolution and minimum analysis time for the compounds of interest.

6.1.3 Preparation of Presurvey Samples. If the samples were collected on an adsorbent, extract the sample as recommended by the manufacturer for removal of the compounds with a solvent suitable to the type of GC analysis. Prepare other samples in an appropriate manner.

6.1.4 Presurvey Sample Analysis. Before analysis, heat the presurvey sample to the duct temperature to vaporize any condensed material. Analyze the samples by the GC procedure, and compare the retention times against those of the calibration samples that contain the components expected to be in the stream. If any compounds cannot be identified with certainty by this procedure, identify them by other means such as GC/mass spectroscopy (GC/MS) or GC/infrared techniques. A GC/MS system is recommended.

Use the GC conditions determined by the procedures of Section 6.1.2 for the first injection. Vary the GC parameters during subsequent injections to determine the optimum settings. Once the optimum settings have been determined, perform repeat injections of the sample to determine the retention time of each compound. To inject a sample, draw sample through the loop at a constant rate (100 ml/min for 30 seconds). Be careful not to pressurize the gas in the loop. Turn off the pump and allow the gas in the sample loop to come to ambient pressure. Activate the sample valve, and record injection time, loop temperature, column temperature, carrier flow rate, chart speed, and attenuator setting. Calculate the retention time of each peak using the distance from injection to the peak maximum divided by the chart speed. Retention times should be repeatable within 0.5 seconds.

If the concentrations are too high for appropriate detector response, a smaller sample loop or dilutions may be used for gas samples, and, for liquid samples, dilution with solvent is appropriate. Use the standard curves (Section 6.3) to obtain an estimate of the concentrations.

Identify all peaks by comparing the known retention times of compounds expected to be in the retention times of peaks in the sample. Identify any remaining unidentified peaks which have areas larger than 5 percent of the total using a GC/MS, or estimation of possible compounds by their retention times compared to known compounds, with confirmation by further GC analysis.

6.2 Calibration Standards. Prepare or obtain enough calibration standards so that there are three different concentrations of each organic compound expected to be measured in the source sample. For each organic compound, select those concentrations that bracket the concentrations expected in the source samples. A calibration standard may contain more than one organic compound. If available, commercial cylinder gases may be used if their concentrations have been certified by direct analysis.

If samples are collected in adsorbent tubes (charcoal, XAD 2, Tenax, etc.), prepare or obtain standards in the same solvent used for the sample extraction procedure. Refer to Section 7.4.3.

Verify the stability of all standards for the time periods they are used. If gas standards are prepared in the laboratory, use one or more of the following procedures.

6.2.1 Preparation of Standards from High Concentration Cylinder Standards. Obtain enough high concentration cylinder standards to represent all the organic compounds expected in the source samples.

Use these high concentration standards to prepare lower concentration standards by dilution, as shown by Figures 18-5 and 18-6.

To prepare the diluted calibration samples, calibrated rotameters are normally used to meter both the high concentration calibration gas and the diluent gas. Other types of flowmeters and commercially available dilution systems can also be used.

Calibrate each flowmeter before use by placing it between the diluent gas supply and suitably sized bubble meter, spirometer, or wet test meter. Record all data shown on Figure 18-4. While it is desirable to calibrate the cylinder gas flowmeter with cylinder gas, the available quantity and cost may preclude it. The error introduced by using the diluent gas for calibration is insignificant for gas mixtures of up to 1,000 to 2,000 ppm of each organic component.

Once the flowmeters are calibrated, connect the flowmeters to the calibration and diluent gas supplies using 6 mm Teflon tubing. Connect the outlet side of the flowmeters through a connector to a leak-free Tedlar bag as shown in Figure 18-5. (See Section 7.1 for bag leak-check procedures.) Adjust the gas flow to provide the desired dilution, and fill the bag with sufficient gas for GC calibration. Be careful not to overfill and cause the bag to apply additional pressure on the dilution system. Record the flow rates of both flowmeters, and the laboratory temperature and atmospheric pressure. Calculate the concentration C_s in ppm of each organic in the diluted gas as follows:

$$C_s = \frac{10^6 \ (\bar{X} \ q_c)}{q_c + q_d} \qquad \text{Eq. 18-1}$$

where:

10^6 = Conversion to ppm.

$\bar{X}$ = Mole or volume fraction of the organic in the calibration gas to be diluted.

q_c = Flow rate of the calibration gas to be diluted.

q_d = Diluent gas flow rate.

Single-stage dilutions should be used to prepare calibration mixtures up to about 1:20 dilution factor.

For greater dilutions, a double dilution system is recommended, as shown in Figure 18-6. Fill the Tedlar bag with the dilute gas from the second stage. Record the laboratory temperature, barometric pressure, and static pressure readings. Correct the flow reading for temperature and pressure. Calculate the concentration C_s in ppm of the organic in the final gas mixture as follows:

$$C_s = 10^6 X \left(\frac{q_{c1}}{q_{c1} + q_{d1}}\right)\left(\frac{q_{c2}}{q_{c2} + q_{d2}}\right) \qquad \text{Eq. 18-2}$$

Where:

10^6 = Conversion to ppm.

X = Mole or volume fraction of the organic in the calibration gas to be diluted.

q_{c1} = Flow rate of the calibration gas to be diluted in stage 1.

q_{c2} = Flow rate of the calibration gas to be diluted in stage 2.

q_{d1} = Flow rate of diluent gas in stage 1.

q_{d2} = Flow rate of diluent gas in stage 2.

Further details of the calibration methods for flowmeters and the dilution system can be found in Citation 21 in the Bibliography.

6.2.3 Preparation of Standards from Volatile Materials. Record all data shown on Figure 18-3.

6.2.3.1 Gas Injection Technique. This procedure is applicable to organic compounds that exist entirely as a gas at ambient conditions. Evacuate a 10-liter Tedlar bag that has passed a leak-check (see Section 7.1), and meter in 5.0 liters of air or nitrogen through a dry gas meter that has been calibrated in a manner consistent with the procedure described in Section 5.1.1 of Method 6. While the bag is filling use a 0.5-ml syringe to inject a known quantity of "pure" gas of the organic compound through the wall of the bag, or through a septum-capped tee at the bag inlet. Withdraw the syringe needle, and immediately cover the resulting hole with a piece of masking tape. In a like manner, prepare dilutions having other concentrations. Prepare a minimum of three concentrations. Place each bag on a smooth surface, and alternately depress opposite sides of the bag 50 times to mix the gases. Record the average meter temperature and pressure, the gas volume and the barometric pressure. Record the syringe temperature and pressure before injection.

Calculate each organic standard concentration C_s in ppm as follows:

$$C_s = \frac{G_v \times 10^6 \dfrac{293}{T_s} \dfrac{P_s}{760}}{V_m Y \dfrac{293}{T_m} \dfrac{P_m}{760} 1000} = \frac{G_v \times 10^3 P_s T_m}{V_m Y T_s P_m} \qquad \text{Eq. 18-3}$$

where:

G_v = Gas volume of organic compound injected, ml.

10^6 = Conversion to ppm.

P_s = Absolute pressure of syringe before injection, mm Hg.

T_s = Absolute temperature of syringe before injection, °K.

V_m = Gas volume indicated by dry gas meter, liters.

Y = Dry gas meter calibration factor, dimensionless.

P_m = Absolute pressure of dry gas meter, mm Hg.

T_m = Absolute temperature of dry gas meter, °K.

1000 = Conversion factor, ml/liter

6.2.3.2 Liquid Injection Technique. Use the equipment shown in Figure 18-8. Calibrate the dry gas meter as described in Section 6.2.3.1 with a wet test meter or a spirometer. Use a water manometer for the pressure gauge and glass, Teflon, brass, or stainless steel for all connections. Connect a valve to the inlet of the 50-liter Tedlar bag.

To prepare the standards, assemble the equipment as shown in Figure 18-8, and leak-check the system. Completely evacuate the bag. Fill the bag with hydrocarbon-free air, and evacuate the bag again. Close the inlet valve.

Turn on the hot plate, and allow the water to reach boiling. Connect the bag to the impinger outlet. Record the initial meter reading, open the bag inlet valve, and open the cylinder. Adjust the rate so that the bag will be completely filled in approximately 15 minutes. Record meter pressure and temperature, and local barometric pressure.

Allow the liquid organic to equilibrate to room temperature. Fill the 1.0- or 10-microliter syringe to the desired liquid volume with the organic. Place the syringe needle into the impinger inlet using the septum provided, and inject the liquid into the flowing air stream. Use a needle of sufficient length to permit injection of the liquid below the air inlet branch of the tee. Remove the syringe.

When the bag is filled, stop the pump, and close the bag inlet valve. Record the final meter reading, temperature, and pressure.

Disconnect the bag from the impinger outlet, and either set it aside for at least 1 hour, or massage the bag to insure complete mixing.

Measure the solvent liquid density at room temperature by accurately weighing a known volume of the material on an analytical balance to the nearest 1.0 milligram. A ground-glass stoppered 25-ml volumetric flask or a glass-stoppered specific gravity bottle is suitable for weighing. Calculate the result in terms of g/ml. As an alternative, literature values of the density of the liquid at 20 °C may be used.

Calculate each organic standard concentration C_s in ppm as follows:

$$C_s = \frac{\dfrac{L_v \rho}{M}(24.055 \times 10^6)}{V_m Y \dfrac{293}{T_m} \dfrac{P_m}{760} 1000} = 6.24 \times 10^4 \frac{L_v \rho T_m}{M V_m Y P_m} \qquad \text{Eq. 18-4}$$

where:

L_v = Liquid volume of organic injected, µl.

ρ = Liquid organic density as determined, g/ml.

M = Molecular weight of organic, g/g-mole.

24.055 = Ideal gas molar volume at 293 °K and 760 mm Hg, liters/g-mole.

10^6 = Conversion to ppm.

1000 = Conversion factor, µl/ml.

6.3 Preparation of Calibration Curves. Establish proper GC conditions, then flush the sampling loop for 30 seconds at a rate of 100 ml/min. Allow the sample loop pressure to equilibrate to atmospheric pressure, and activate the injection valve. Record the standard concentration, attenuator factor, injection time, chart speed, retention time, peak area, sample loop temperature, column temperature, and carrier gas flow rate. Repeat the standard injection until two consecutive injections give area counts within 5 percent of their average. The average value multiplied by the attenuator factor is then the calibration area value for the concentration.

Repeat this procedure for each standard. Prepare a graphical plot of concentration (C_s) versus the calibration area values. Perform a regression analysis, and draw the least squares line.

6.4 Relative Response Factors. The calibration curve generated from the standards for a single organic can usually be related to each of the individual GC response curves that are developed in the laboratory for all the compounds in the source. In the field, standards for that single organic can then be used to "calibrate" the GC for all the organics present. This procedure should first be confirmed in the laboratory by preparing and analyzing calibration standards containing multiple organic compounds.

6.5 *Quality Assurance for Laboratory Procedures.* Immediately after the preparation of the calibration curves and prior to the presurvey sample analysis, the analysis audit described in 40 CFR Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis," should be performed. The information required to document the analysis of the audit samples has been included on the example data sheets shown in Figures 18-3 and 18-7. The audit analyses *should agree with the audit concentrations* within 10 percent. When available, the tester may obtain audit cylinders by contacting: U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711. Audit cylinders obtained from a commercial gas manufacturer may be used provided that (a) the gas manufacturer certifies the audit cylinder in a manner similar to the procedure described in 40 CFR Part 61, Appendix B, Method 106, Section 6.3.3.1, and (b) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined as an analysis performed by an individual other than the individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis

concentration is within 5 percent of the gas manufacturer's concentration.

1. Final Sampling and Analysis Procedure

Considering safety (flame hazards) and the source conditions, select an appropriate sampling and analysis procedure (Section 7.1, 7.2, 7.3, or 7.4). In situations where a hydrogen flame is a hazard and no intrinsically safe GC is suitable, use the flexible bag collection technique or an adsorption technique. If the source temperature is below 100°C, and the organic concentrations are suitable for the detector to be used, use the direct interface method. If the source gases require dilution, use a dilution interface and either the bag sample or adsorption tubes. The choice between these two techniques will depend on the physical layout of the site, the source temperature, and the storage stability of the compounds if collected in the bag. Sample polar compounds by direct interfacing or dilution interfacing to prevent sample loss by adsorption on the bag.

7.1 Integrated Bag Sampling and Analysis.

7.1.1 Evacuated Container Sampling Procedure. In this procedure, the bags are filled by evacuating the rigid air-tight containers that hold the bags. Use a field sample data sheet as shown in Figure 18-10. Collect triplicate sample from each sample location.

7.1.1.1 Apparatus.

7.1.1.1.1 Probe. Stainless steel, Pyrex glass, or Teflon tubing probe, according to the duct temperature, with 6.4-mm OD Teflon tubing of sufficient length to connect to the sample bag. Use stainless steel or Teflon unions to connect probe and sample line.

7.1.1.1.2 Quick Connects. Male (2) and female (2) of stainless steel construction.

7.1.1.1.3 Needle Valve. To control gas flow.

7.1.1.1.4 Pump. Leakless Teflon-coated diaphragm-type pump or equivalent. To deliver at least 1 liter/min.

7.1.1.1.5 Charcoal Adsorption Tube. Tube filled with activated charcoal, with glass wool plugs at each end, to adsorb organic vapors.

7.1.1.1.6 Flowmeter. 0 to 500-ml flow range; with manufacturer's calibration curve.

7.1.1.2 Sampling Procedure. To obtain a sample, assemble the sample train as shown in Figure 18-9. Leak check both the bag and the container. Connect the vacuum line from the needle valve to the Teflon sample line from the probe. Place the end of the probe at the centroid of the stack, or at a point no closer to the walls than 1 m, and start the pump with the needle valve adjusted to yield a flow of 0.5 liter/minute. After allowing sufficient time to purge the line several times, connect the vacuum line to the bag, and evacuate until the rotameter indicates no flow. Then position the sample and vacuum lines for sampling, and begin the actual sampling, keeping the rate proportional to the stack velocity. As a precaution, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Record the source temperature, barometric pressure, ambient temperature, sampling flow rate, and initial and final sampling time on the data sheet shown in Figure 18-10. Protect the Tedlar bag and its container from sunlight. When possible, perform the analysis within 2 hours of sample collection.

7.1.2 Direct Pump Sampling Procedure. Follow 7.1.1, except place the pump and needle valve between the probe and the bag. Use a pump and needle valve constructed of stainless steel or some other material not affected by the stack gas. Leak check the system, and then purge with stack gas before the connecting to the previously evacuated bag.

7.1.3 Explosion Risk Area Bag Sampling Procedure. Follow 7.1.1 except replace the pump with another evacuated can (see Figure 18-9a). Use this method whenever there is a possibility of an explosion due to pumps, heated probes, or other flame producing equipment.

7.1.4 Other Modified Bag Sampling Procedures. In the event that condensation is observed in the bag while collecting the sample and a direct interface system cannot be used, heat the bag during collection, and maintain it at a suitably elevated temperature during all subsequent operations. (Note: Take care to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.) As an alternative, collect the sample gas, and simultaneously dilute it in the Tedlar bag.

In the first procedure, heat the box containing the sample bag to the source temperature, provided the components of the bag and the surrounding box can withstand this temperature. Then transport the bag as rapidly as possible to the analytical area while maintaining the heating, or cover the box with an insulating blanket. In the analytical area, keep the box heated to source temperature until analysis. Be sure that the method of heating the box and the control for the heating circuit are compatible with the safety restrictions required in each area.

To use the second procedure, prefill the Tedlar bag with a known quantity of inert gas. Meter the inert gas into the bag according to the procedure for the preparation of gas concentration standards of volatile liquid materials (Section 6.2.2.2), but eliminate the midget impinger section. Take the partly filled bag to the source, and meter the source gas into the bag through heated sampling lines and a heated flowmeter, or Teflon positive displacement pump. Verify the dilution factors periodically through dilution and analysis of gases of known concentration.

7.1.5 Analysis of Bag Samples.

7.1.5.1 Apparatus. Same as Section 5. A minimum of three gas standards are required.

7.1.5.2 Procedure. Establish proper GC operating conditions as described in Section 6.3, and record all data listed in Figure 18-7. Prepare the GC so that gas can be drawn through the sample valve. Flush the sample loop with gas from one of the three calibration mixtures, and activate the valve. Obtain at least two chromatograms for the mixture. The results are acceptable when the peak areas from two consecutive injections agree to within 5 percent of their average. If they do not, run additional analyses or correct the analytical technique until this requirement is met. Then analyze the other two calibration mixtures in the same manner. Prepare a calibration curve as described in the same manner. Prepare a calibration curve as described in Section 6.3.

Analyze the source gas samples by connecting each bag to the sampling valve with a piece of Teflon tubing identified for that bag. Follow the specifications on replicate analyses specified for the calibration gases. Record the data listed in Figure 18-11. If certain items do not apply, use the notation "N.A." After all samples have been analyzed, repeat the analyses of the calibration gas mixtures, and generate a second calibration curve. Use an average of the two curves to determine the sample gas concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then report the final results by comparison to both calibration curves.

7.1.6 Determination of Bag Water Vapor Content. Measure and record the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.) If the bag has been maintained at an elevated temperature as described in Section 7.1.4, determine the stack gas water content by Method 4.

7.1.7 Quality Assurance. Immediately prior to the analysis of the stack gas samples, perform audit analyses as described in Section 6.5. The audit analyses must agree with the audit concentrations within 10 percent. If the results are acceptable, proceed with the analyses of the source samples. If they do not agree within 10 percent, then determine the reason for the discrepancy, and take corrective action before proceeding.

7.1.8 Emission Calculations. From the average calibration curve described in Section 7.1.5, select the value of C_c that corresponds to the peak area. Calculate the concentration C_c, in ppm, dry basis, of each organic in the sample as follows:

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1 - B_{ws})} \qquad \text{Eq. 18-5}$$

where:

C_c = Concentration of the organic from the calibration curve, ppm.

P_r = Reference pressure, the barometric pressure or absolute sample loop pressure recorded during calibration, mm Hg.

T_i = Sample loop temperature at the time of sample analysis, °K.

P_i = Relative response factor (if applicable, see Section 6.4).

P_i = Barometric or absolute sample loop pressure at time of sample analysis, mm Hg.

T_r = Reference temperature, the temperature of the sample loop recorded during calibration, °K.

B_{ws} = Water vapor content of the bag sample or stack gas, proportion by volume.

7.2 Direct Interface Sampling and Analysis Procedure. The direct interface procedure can be used provided that the moisture content of the gas does not interfere with the analysis procedure, the physical requirements of the equipment can be met at the site, and the source gas concentration is low enough that detector saturation is not a problem. Adhere to all safety requirements with this method.

7.2.1 Apparatus.

7.2.1.1 Probe. Constructed of stainless steel, Pyrex glass, or Teflon tubing as required by duct temperature, 6.4-mm OD, enlarged at duct end to contain glass wool plug. If necessary, heat the probe with heating tape or a special heating unit capable of maintaining duct temperature.

7.2.1.2 Sample Lines. 6.4-mm OD Teflon lines, heat-traced to prevent condensation of material.

7.2.1.3 Quick Connects. To connect sample line to gas sampling valve on GC instrument and to pump unit used to withdraw source gas. Use a quick connect or equivalent on the cylinder or bag containing calibration gas to allow connection of the calibration gas to the gas sampling valve.

7.2.1.4 Thermocouple Readout Device. Potentiometer or digital thermometer, to measure source temperature and probe temperature.

7.2.1.5 Heated Gas Sampling Valve. Of two position, six port design, to allow sample loop to be purged with source gas or to direct source gas into the GC instrument.

7.2.1.6 Needle Valve. To control gas sampling rate from the source.

7.2.1.7 Pump. Leakless Teflon-coated diaphragm-type pump or equivalent, capable of at least 1 liter/minute sampling rate.

7.2.1.8 Flowmeter. Of suitable range to measure sampling rate.

7.2.1.9 Charcoal Adsorber. To adsorb organic vapor collected from the source to prevent exposure of personnel to source gas.

7.2.1.10 Gas Cylinders. Carrier gas (helium or nitrogen), and oxygen and hydrogen for a flame ionization detector (FID) if one is used.

7.2.1.11 Gas Chromatograph. Capable of being moved into the field, with detector, heated gas sampling valve, column required to complete separation of desired components, and option for temperature programming.

7.2.1.12 Recorder/Integrator. To record results.

7.2.2 Procedure. To obtain a sample, assemble the sampling system as shown in Figure 18-12. Make sure all connections are tight. Turn on the probe and sample line heaters. As the temperature of the probe and heated line approaches the source temperature as indicated on the thermocouple readout device, control the heating to maintain a temperature of 0 to 3°C above the source temperature. While the probe and heated line are being heated, disconnect the sample line from the gas sampling valve, and attach the line from the calibration gas mixture. Flush the sample loop with calibration gas and analyze a portion of that gas. Record the results. After the calibration gas sample has been flushed into the GC instrument, turn the gas sampling valve to flush position, then reconnect the probe sample line to the valve. Place the inlet of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and draw source gas into the probe, heated line, and sample loop. After thorough flushing, analyze the sample using the same conditions as for the calibration gas mixture. Repeat the analysis on an additional sample. Measure the peak areas for the two samples, and if they do not agree to within 5 percent of their mean value, analyze additional samples until two consecutive analyses meet this criteria. Record the data. After consistent results are obtained, remove the probe from the source and analyze a second calibration gas mixture. Record this calibration data and the other required data on the data sheet shown in Figure 18-11, deleting the dilution gas information.

(NOTE: Take care to draw all samples, calibration mixtures, and audits through the sample loop at the same pressure.)

7.2.3 Determination of Stack Gas Moisture Content. Use Method 4 to measure the stack gas moisture content.

7.2.4 Quality Assurance. Same as Section 7.1.7. Introduce the audit gases in the sample line immediately following the probe.

7.2.5 Emission Calculations. Same as Section 7.1.8.

7.3 Dilution Interface Sampling and Analysis Procedure. Source samples that contain a high concentration of organic materials may require dilution prior to analysis to prevent saturating the GC detector. The apparatus required for this direct interface procedure is basically the same as that described in the Section 7.2, except a dilution system is added between the heated sample line and the gas sampling valve. The apparatus is arranged so that either a 10:1 or 100:1 dilution of the source gas can be directed to the chromatograph. A pump of larger capacity is also required, and this pump must be heated and placed in the system between the sample line and the dilution apparatus.

7.3.1 Apparatus. The equipment required in addition to that specified for the direct interface system is as follows:

7.3.1.1 Sample Pump. Leakless Teflon-coated diaphragm-type that can withstand being heated to 120°C and deliver 1.5 liters/minute.

7.3.1.2 Dilution Pumps. Two Model A-150 Komhyr Teflon positive displacement type delivering 150 cc/minute, or equivalent. As an option, calibrated flowmeters can be used in conjunction with Teflon-coated diaphragm pumps.

7.3.1.3 Valves. Two Teflon three-way valves, suitable for connecting to 6.4-mm OD Teflon tubing.

7.3.1.4 Flowmeters. Two, for measurement of diluent gas, expected delivery flow rate to be 1,350 cc/min.

7.3.1.5 Diluent Gas with Cylinders and Regulators. Gas can be nitrogen or clean dry air, depending on the nature of the source gases.

7.3.1.6 Heated Box. Suitable for being heated to 120°C, to contain the three pumps, three-way valves, and associated connections. The box should be equipped with quick connect fittings to facilitate connection of: (1) The heated sample line from the probe, (2) the gas sampling valve, (3) the calibration gas mixtures, and (4) diluent gas lines. A schematic diagram of the components and connections is shown in Figure 18-13.

(NOTE: Care must be taken to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.)

The heated box shown in Figure 18-13 is designed to receive a heated line from the probe. An optional design is to build a probe unit that attaches directly to the heated box. In this way, the heated box contains the controls for the probe heaters, or, if the box is placed against the duct being sampled, it may be possible to eliminate the probe heaters. In either case, a heated Teflon line is used to connect the heated box to the gas sampling valve on the chromatograph.

7.3.2 Procedure. Assemble the apparatus by connecting the heated box, shown in Figure 18-13, between the heated sample line from the probe and the gas sampling valve on the chromatograph. Vent the source gas from the gas sampling valve directly to the charcoal filter, eliminating the pump and rotameter. Heat the sample probe, sample line, and heated box. Insert the probe and source thermocouple to the centroid of the duct, or to a point no closer to the walls than 1 m. Measure the source temperature, and adjust all heating units to a temperature 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds. Verify the operation of the dilution system by analyzing a high concentration gas of known composition through either the 10:1 or 100:1 dilution stages, as appropriate. (If necessary, vary the flow of the diluent gas to obtain other dilution ratios.) Determine the concentration of the diluted calibration gas using the dilution factor and the calibration curves prepared in the laboratory. Record the pertinent data on the data sheet shown in Figure 18-11. If the data on the diluted calibration gas are not within 10 percent of the expected values, determine whether the chromatograph or the dilution system is in error, and correct it. Verify the GC operation using a low concentration standard by diverting the gas into the sample loop, bypassing the dilution system. If these analyses are not within acceptable limits, correct the dilution system to provide the desired dilution factors. Make this correction by diluting a high-concentration standard gas mixture to adjust the dilution ratio as required.

Once the dilution system and GC operations are satisfactory, proceed with the analysis of source gas, maintaining the same dilution settings as used for the standards. Repeat the analyses until two consecutive values do not vary by more than 5 percent from their mean value are obtained.

Repeat the analysis of the calibration gas mixtures to verify equipment operation. Analyze the two field audit samples using either the dilution system, or directly connect to the gas sampling valve as required. Record all data and report the results to the audit supervisor.

7.3.3 Determination of Stack Gas Moisture Content. Same as Section 7.2.3.

7.3.4 Quality Assurance. Same as Section 7.2.4.

7.3.5 Emission Calculations. Same as Section 7.2.5, with the dilution factor applied.

7.4 Adsorption Tube Procedure (Alternative Procedure). It is suggested that the tester refer to the National Institute of Occupational Safety and Health (NIOSH) method for the particular organics to be sampled. The principal interferent will be water vapor. If water vapor is present at concentrations above 3 percent, silica gel should be used in front of the charcoal. Where more than one compound is present in the emissions, then develop relative adsorptive capacity information.

7.4.1 Additional Apparatus. In addition to the equipment listed in the NIOSH method for the particular organic(s) to be sampled, the following items (or equivalent) are suggested.

7.4.1.1 Probe (Optional). Borosilicate glass or stainless steel, approximately 6-mm ID, with a heating system if water condensation is a problem, and a filter (either in-stack or out-stack heated to stack temperature) to remove particulate matter. In most instances, a plug of glass wool is a satisfactory filter.

7.4.1.2 Flexible Tubing. To connect probe to adsorption tubes. Use a material that exhibits minimal sample adsorption.

7.4.1.3 Leakless Sample Pump. Flow controlled, constant rate pump, with a set of limiting (sonic) orifices to provide pumping rates from approximately 10 to 100 cc/min.

7.4.1.4 Bubble-Tube Flowmeter. Volume accuracy within ± 1 percent, to calibrate pump.

7.4.1.5 Stopwatch. To time sampling and pump rate calibration.

7.4.1.6 Adsorption Tubes. Similar to ones specified by NIOSH, except the amounts of adsorbent per primary/backup sections are 800/200 mg for charcoal tubes and 1040/200 mg for silica gel tubes. As an alternative, the tubes may contain a porous polymer adsorbent such as Tenax GC or XAD-2.

7.4.1.7 Barometer. Accurate to 5 mm Hg, to measure atmospheric pressure during sampling and pump calibration.

7.4.1.8 Rotameter. 0 to 100 cc/min, to detect changes in flow rate during sampling.

7.4.2 Sampling and Analysis. It is suggested that the tester follow the sampling and analysis portion of the respective NIOSH method section entitled "Procedure." Calibrate the pump and limiting orifice flow rate through adsorption tubes with the bubble tube flowmeter before sampling. The sample system can be operated as a "recirculating loop" for this operation. Record the ambient temperature and barometric pressure. Then, during sampling, use the ro-

lameter to verify that the pump and orifice sampling rate remains constant.

Use a sample probe, if required, to obtain the sample at the centroid of the duct, or at a point no closer to the walls than 1 m. Minimize the length of flexible tubing between the probe and adsorption tubes. Several adsorption tubes can be connected in series, if the extra adsorptive capacity is needed. Provide the gas sample to the sample system at a pressure sufficient for the limiting orifice to function as a sonic orifice. Record the total time and sample flow rate (or the number of pump strokes), the barometric pressure, and ambient temperature. Obtain a total sample volume commensurate with the expected concentration(s) of the volatile organic(s) present, and recommended sample loading factors (weight sample per weight adsorption media). Laboratory tests prior to actual sampling may be necessary to predetermine this volume. When more than one organic is present in the emissions, then develop relative adsorptive capacity information. If water vapor is present in the sample at concentrations above 2 to 3 percent, the adsorptive capacity may be severely reduced. Operate the gas chromatograph according to the manufacture's instructions. After establishing optimum conditions, verify and document these conditions during all operations. Analyze the audit samples (see Section 7.4.4.3), then the emission samples. Repeat the analysis of each sample until the relative deviation of two consecutive injections does not exceed 5 percent.

7.4.3 Standards and Calibration. The standards can be prepared according to the respective NIOSH method. Use a minimum of three different standards; select the concentrations to bracket the expected average sample concentration. Perform the calibration before and after each day's sample analyses. Prepare the calibration curve by using the least squares method.

7.4.4 Quality Assurance.

7.4.4.1 Determination of Desorption Efficiency. During the testing program, determine the desorption efficiency in the expected sample concentration range for each batch of adsorption media to be used. Use an internal standard. A minimum desorption efficiency of 50 percent shall be obtained. Repeat the desorption determination until the relative deviation of two consecutive determinations does not exceed 5 percent. Use the average desorption efficiency of these two consecutive determinations for the correction specified in Section 7.4.4.5. If the desorption efficiency of the compound(s) of interest is questionable under actual sampling conditions, use of the Method of Standard Additions may be helpful to determine this value.

7.4.4.2 Determination of Sample Collection Efficiency. For the source samples, analyze the primary and backup portions of the adsorption tubes separately. If the backup portion exceeds 10 percent of the total amount (primary and backup), repeat the sampling with a larger sampling portion.

7.4.4.3 Analysis Audit. Immediately before the sample analyses, analyze the two audits in accordance with Section 7.4.2. The analysis audit shall agree with the audit concentration within 10 percent.

7.4.4.4 Pump Leak Checks and Volume Flow Rate Checks. Perform both of these checks immediately after sampling with all sampling train components in place. Perform all leak checks according to the manufacturer's instructions, and record the results. Use the bubble-tube flowmeter to measure the pump volume flow rate with the orifice used in the test sampling, and the result. If it has changed by more than 5 but less than 20 percent, calculate an average flow rate for the test. If the flow rate has changed by more than 20 percent, recalibrate the pump and repeat the sampling.

7.4.4.5 Calculations. All calculations can be performed according to the respective NIOSH method. Correct all sample volumes to standard conditions. If a sample dilution system has been used, multiply the results by the appropriate dilution ratio. Correct all results by dividing by the desorption efficiency (decimal value). Report results as ppm by volume, dry basis.

7.5 Reporting of Results. At the completion of the field analysis portion of the study, ensure that the data sheets shown in Figure 18-11 have been completed. Summarize this data on the data sheets shown in Figure 18-15.

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I. Name of company _____ Date _____
Address _____
_____
Contacts _____ Phone _____
_____ _____
Process to be sampled _____
_____
_____
Duct or vent to be sampled _____
_____

II. Process description _____
_____
_____
_____
_____
Raw material _____
_____
_____
Products _____
_____
_____
Operating cycle
Check: Batch _____ Continuous _____ Cyclic _____
Timing of batch or cycle _____
Best time to test _____

Figure 18-1. Preliminary survey data sheet.

III. Sampling site
A. Description
Site description _____
Duct shape and size _____
Material _____
Wall thickness _____ inches
Upstream distance _____ inches _____ diameter
Downstream distance _____ inches _____ diameter
Size of port _____
Size of access area _____
Hazards _____ Ambient temp. _____ °F

B. Properties of gas stream
Temperature _____ °C _____ °F, Data source _____
Velocity _____, Data source _____
Static pressure ___ inches H_2O, Data source _____
Moisture content _____ %, Data source _____
Particulate content _____, Data source _____
Gaseous components

N_2	_____ %	Hydrocarbons	_____ ppm
O_2	_____ %	_____	_____
CO	_____ %	_____	_____
CO_2	_____ %	_____	_____
SO_2	_____ %	_____	_____

Hydrocarbon components

_____	_____ ppm
_____	_____ ppm
_____	_____ ppm
_____	_____ ppm
_____	_____ ppm
_____	_____ ppm

Figure 18-1 (continued). Preliminary survey data sheet.

C. Sampling considerations

Location to set up GC _____

_____

Special hazards to be considered _____

_____

Power available at duct _____

Power available for GC _____

Plant safety requirements _____

_____

Vehicle traffic rules _____

_____

Plant entry requirements _____

_____

Security agreements _____

_____

Potential problems _____

_____

D. Site diagrams. (Attach additional sheets if required).

Figure 18-1 (continued). Preliminary survey data sheet.

Components to be analyzed	Expected concentration

Suggested chromatographic column _____

_____

Column flow rate ____ ml/min Head pressure____ mm Hg

Column temperature:

Isothermal _____°C

Programmed from _____°C to ____°C at ____°C/min

Injection port/sample loop temperature _____°C

Detector temperature _____°C

Detector flow rates: Hydrogen _____ml/min.

head pressure____ mm Hg

Air/Oxygen _____ml/min,

head pressure____ mm Hg

Chart speed _____ inches/minute

Compound data:

Compound	Retention time	Attenuation

Figure 18-2. Chromatographic conditions data sheet.

Preparation of Standards in Tedlar Bags and Calibration Curve

Standards Preparation Data:	Standards Mixture #1	Standards Mixture #2	Standards Mixture #3
Organic: _____			
Bag number or identification			
Dry gas meter calibration factor			
Final meter reading (liters)			
Initial meter reading (liters)			
Metered volume (liters)			
Average meter temperature (°K)			
Average meter pressure, gauge (mm Hg)			
Average atmospheric pressure (mm Hg)			
Average meter pressure, absolute (mm Hg)			
Syringe temperature (°K) (Section 6.2.2.1)			
Syringe pressure, absolute (mm Hg) (Section 6.2.2.1)			
Volume of gas in syringe (ml) (Section 6.2.2.1)			
Density of liquid organic (g/ml) (Section 6.2.2.2)			
Volume of liquid in syringe (µl) (Section 6.2.2.2)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Carrier gas flow rate (ml/min)			
Column temperature			
Initial (°C)			
Rate change (°C/min)			
Final (°C)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24-hr clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Organic retention time (min)			
Attenuation factor			
Peak height (mm)			
Peak area (mm^2)			
Peak area × attenuation factor (mm^2)			
Calculated concentration (ppm) (Equation 18-3 or 18-4)			

Plot peak area × attenuation factor against calculated concentration to obtain calibration curve.

Figure 18-3. Standards prepared in Tedlar bags and calibration curve.

Flowmeter Calibration

Flowmeter number or identification _____
Flowmeter type
Calibration device (x): Bubble meter ___ Spirometer ___ Wet test meter ___
Readings at laboratory conditions:
Laboratory temperature (T_{lab}) _____ °K
Laboratory barometric pressure (P_{lab}) _____ mm Hg
Flow data:

Flowmeter reading (as marked)	Flowmeter temp. (°K)	Flowmeter pressure (absolute)	Calibration device time (min)	Calibration device gas volume[a]	Calibration device flow rate[b]

a = Volume of gas measured by calibration device, corrected to standard conditions (liters).

b = Calibration device gas volume/time.

Plot flowmeter reading against flow rate (standard conditions), and draw a smooth curve. If the flowmeter being calibrated is a rotameter or other flow device that is viscosity dependent, it may be necessary to generate a "family" of calibration curves that cover the operating pressure and temperature ranges of the flowmeter.

While the following technique should be verified before application, it may be possible to calculate flow rate readings for rotameters at standard conditions Q_{std} as follows:

$$Q_{std} = Q_{lab}\left(\frac{760 \times T_{lab}}{P_{lab} \times 293}\right)^{1/2}$$

Flow rate (laboratory conditions)	Flow rate (standard conditions)

Figure 18-4. Flowmeter calibration.

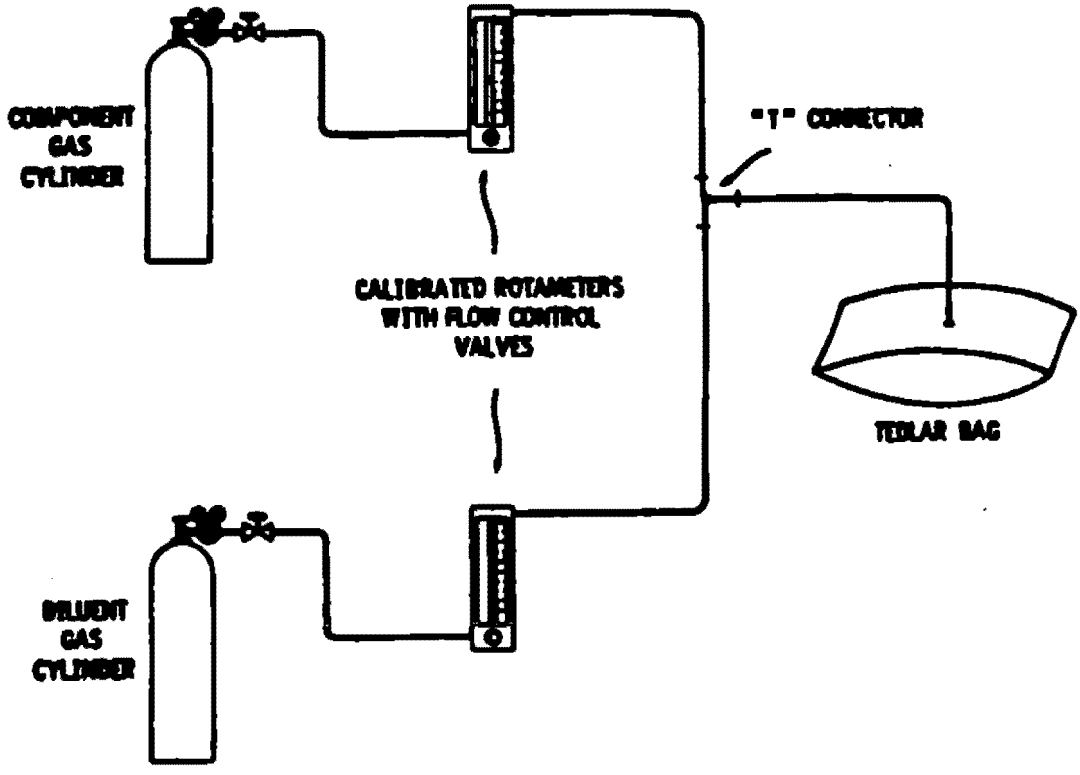

Figure 18-6. Single-stage calibration gas dilution system.

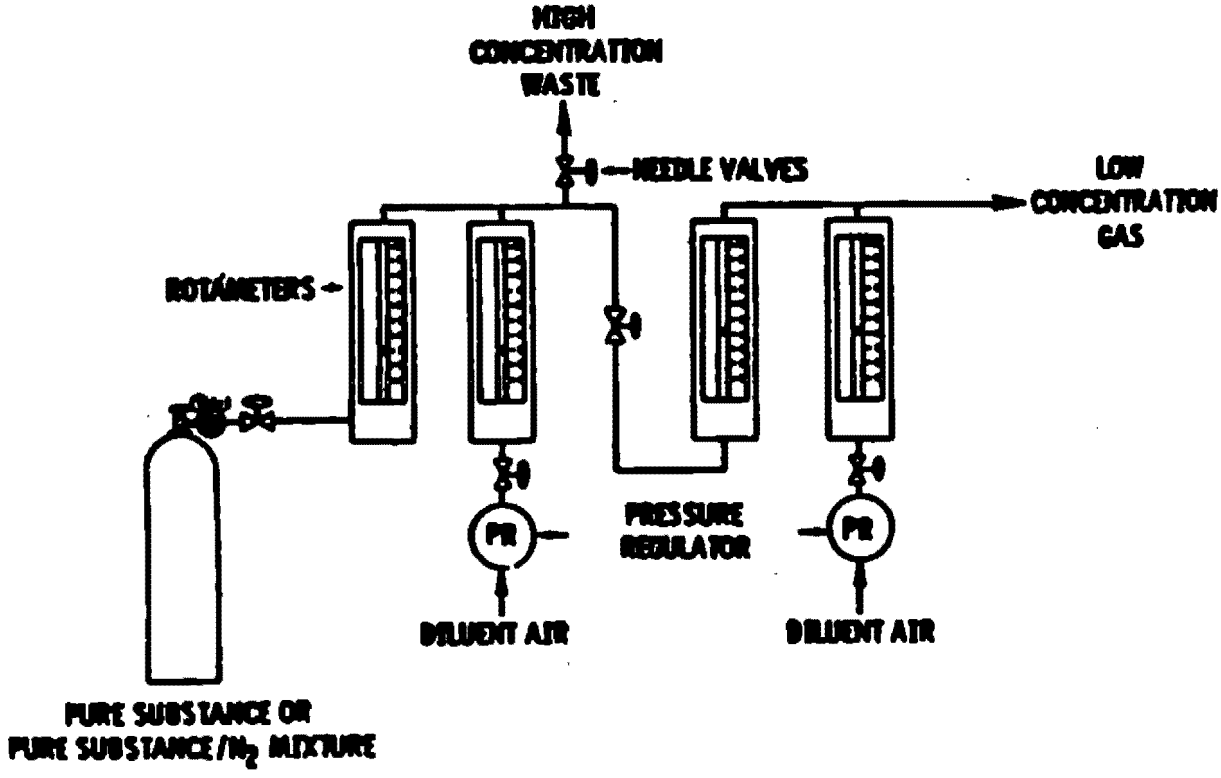

Figure 18-6. Two-stage dilution apparatus.

Preparation of Standards by Dilution of Cylinder Standard

Cylinder standard: Organic _____ Certified concentration _____ ppm

Standards Preparation Data: Date _____

Stage 1	Mixture 1	Mixture 2	Mixture 3
Standard gas flowmeter reading			
Diluent gas flowmeter reading			
Laboratory temperature (°K)			
Barometric pressure (mm Hg)			
Flowmeter gage pressure (mm Hg)			
Flow rate cylinder gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions (ml/min)			
Calculated concentration (ppm)			
Stage 2 (if used)			
Standard gas flowmeter reading			
Diluent gas flowmeter reading			
Flow rate stage 1 gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions (ml/min)			
Calculated concentration (ppm)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°K)			
Carrier gas flow rate (ml/min)			
Column temperature:			
Initial (°K)			
Program rate (°K/min)			
Final (°K)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24-hr clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Retention time (min)			
Attenuation factor			
Peak area (cm^2)			
Peak area x attenuation factor			

Plot peak area x attenuation factor against calculated concentration to obtain calibration curve.

Figure 18-7. Standards prepared by dilution of cylinder standard.

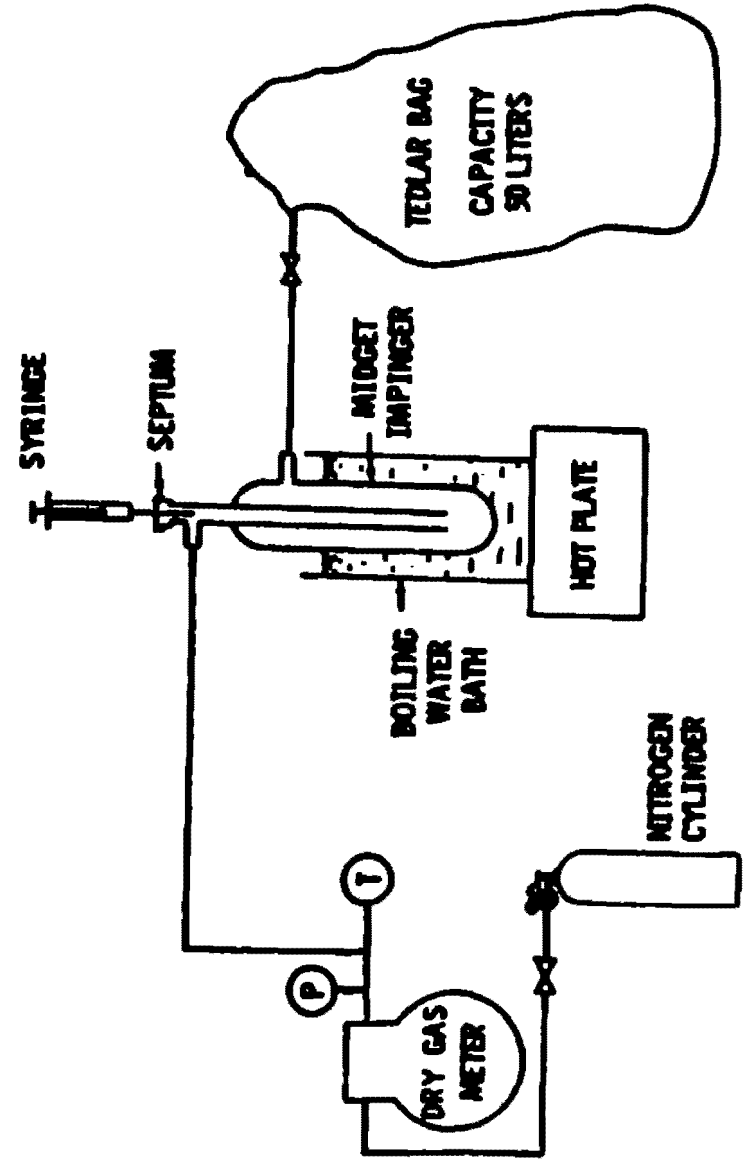

Figure 18-8. Apparatus for preparation of liquid materials.

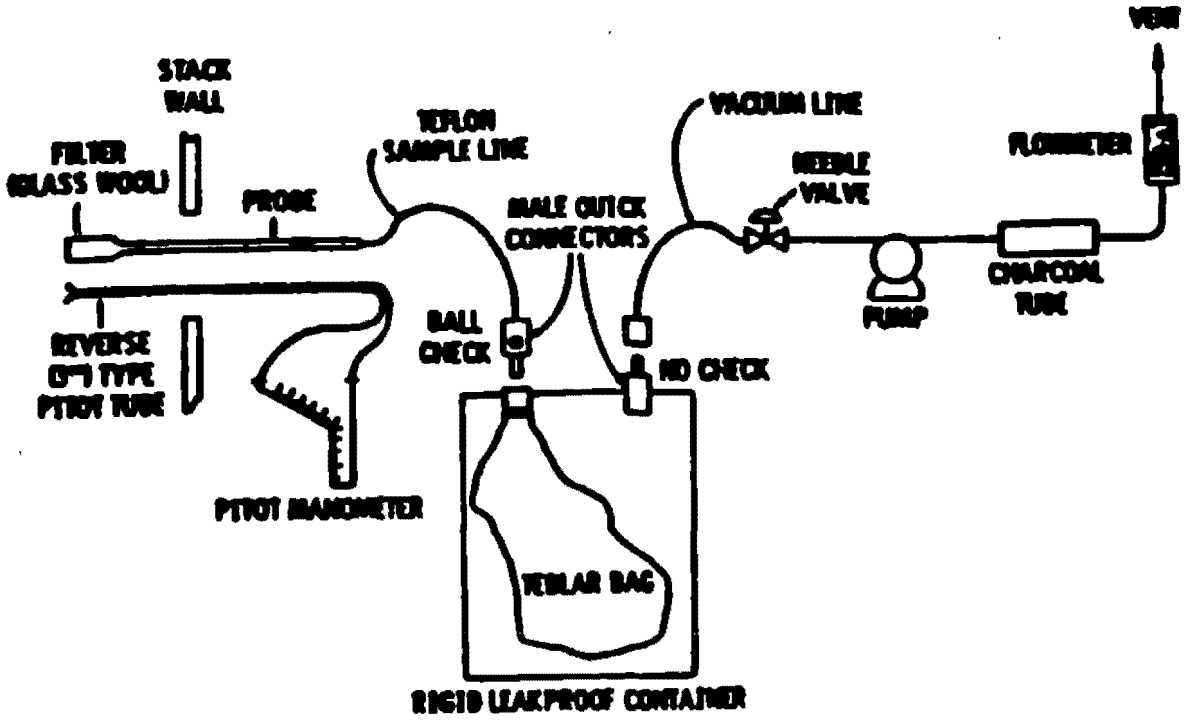

Figure 18-9. Integrated bag sampling train.

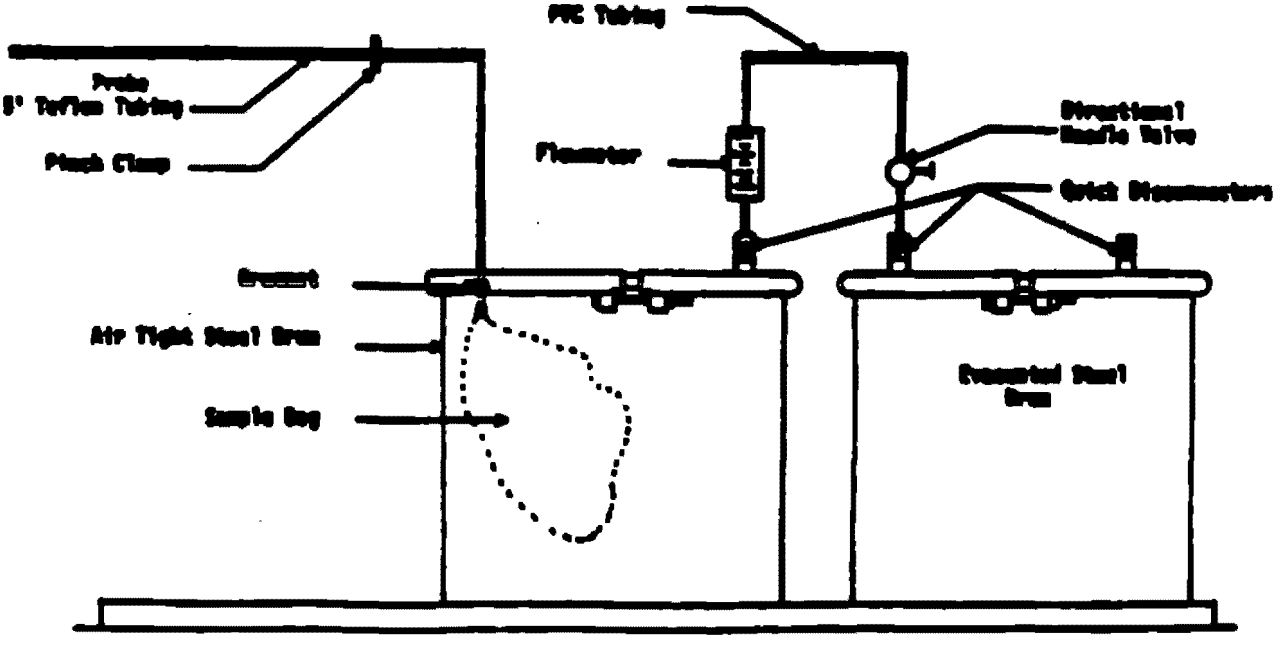

Figure 18-9a. Explosion risk gas sampling method.

Plant _____ Date _____

Site _____

	Sample 1	Sample 2	Sample 3
Source temperature (°C)			
Barometric pressure (mm Hg)			
Ambient temperature (°C)			
Sample flow rate (appr.)			
Bag number			
Start time			
Finish time			

Figure 18-10. Field sample data sheet - Tedlar bag collection method.

Plant _____ Date _____

Location _____

1. General information

Source temperature (°C)	
Probe temperature (°C)	
Ambient temperature (°C)	
Atmospheric pressure (mm)	
Source pressure (°Hg)	
Absolute source pressure (mm)	
Sampling rate (liter/min)	
Sample loop volume (ml)	
Sample loop temperature (°C)	
Columnar temperature:	
Initial (°C)/time (min)	
Program rate (°C/min)	
Final (°C)/time (min)	
Carrier gas flow rate (ml/min)	
Detector temperature (°C)	
Injection time (24-hour basis)	
Chart speed (mm/min)	
Dilution gas flow rate (ml/min)	
Dilution Gas used (symbol)	
Dilution ratio	

Figure 18-11. Field analysis data sheets.

3. Field Analysis Data - Calibration Gas

Run No._____ Time_____

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Run No._____ Time_____

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Run No._____ Time_____

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Figure 18-11 (continued). Field analysis data sheets.

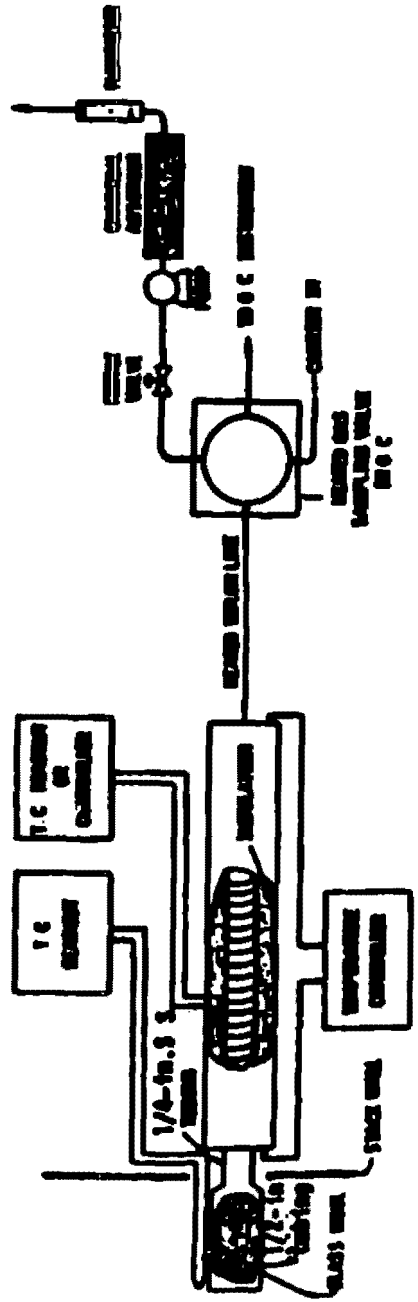

Figure 18-12. Direct interface sampling system.

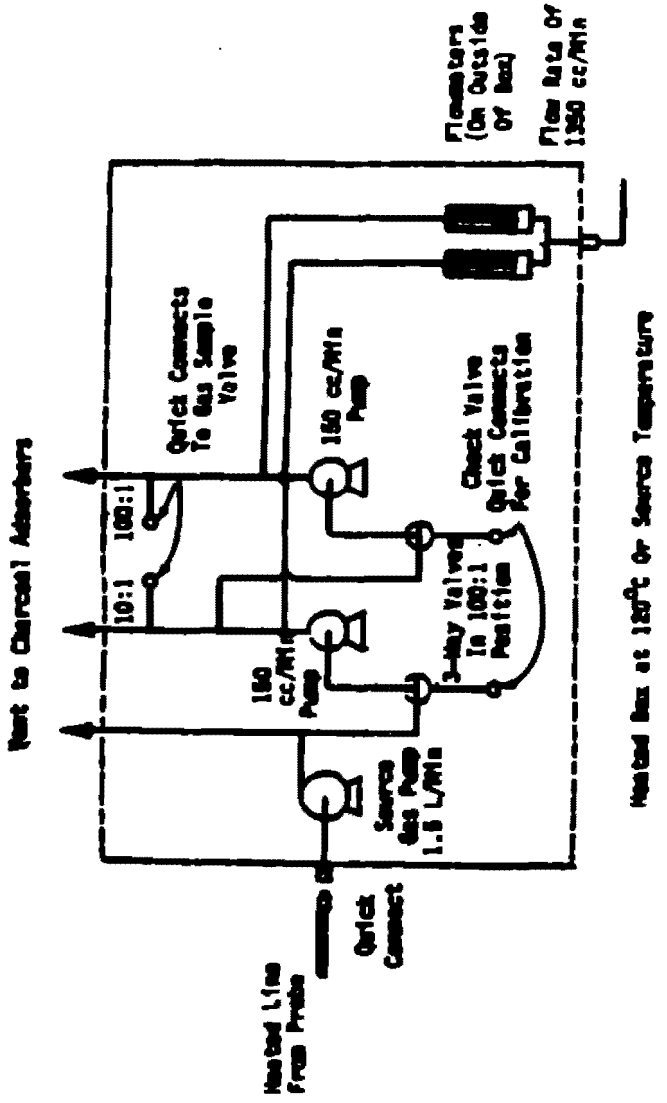

Figure 18-13. Schematic diagram of the heated box required for dilution of sample gas.

GASEOUS ORGANIC SAMPLING AND ANALYSIS CHECK LIST

(Respond with initials or number as appropriate)

	Date	
1. Preservey data:		
A. Grab sample collected	O	
B. Grab sample analyzed for composition	O	
Method GC	O	O
GC/MS	O	O
Other	O	O
C. GC-FID analysis performed	O	
2. Laboratory calibration data:		
A. Calibration curves prepared	O	
Number of components	O	O
Number of concentrations/component (3 required)	O	O
B. Audit samples (optional):		
Analysis completed	O	
Verified for concentration	O	
CE obtained for field work	O	
3. Sampling procedures:		
A. Method		
Bag sample	O	O
Direct interface	O	O
Dilution interface	O	O
B. Number of samples collected	NO	
4. Field analysis:		
A. Total hydrocarbon analysis performed	O	
B. Calibration curve prepared	O	
Number of components	O	O
Number of concentrations per component (3 required)	O	O

Figure 18-14. Sampling and analysis check.

GASEOUS ORGANIC SAMPLING AND ANALYSIS DATA

Plant _____

Date _____

Location _____

	Source sample 1	Source sample 2	Source sample 3
1. General information:			
Source temperature (°C)			
Probe temperature (°C)			
Ambient temperature (°C)			
Atmospheric pressure (mm Hg)			
Source pressure (mm Hg)			
Sampling rate (ml/min)			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Sample collection time (24-hr basis)			
Column temperature:			
Initial (°C)			
Program rate (°C/min)			
Final (°C)			
Carrier gas flow rate (ml/min)			
Detector temperature (°C)			
Chart speed (cm/min)			
Dilution gas flow rate (ml/min)			
Diluent gas used (symbol)			
Dilution ratio			

Performed by (signature): _____

Date: _____

Figure 18-14. Sampling and analysis sheet.

EPA METHOD 3A

particular caution. Calibration will be affected by variation in stack gas temperature, pressure, compressibility, and molecular weight. Use the procedure in Section 3.3.1. Record all the measuring device parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

4. Calibration

4.1 Flow Rate Measuring Device. Use the procedure in Method 2A, Section 4, and apply the same performance standards. Calibrate the measuring device with the principal stack gas to be measured (e.g., air, nitrogen) against a standard reference meter. A calibrated dry gas meter is an acceptable reference meter. Ideally, calibrate the measuring device in the field with the actual gas to be measured. For measuring devices that have a volume rate readout, calculate the measuring device calibration coefficient, Y_m, for each run as follows:

$$Y_m = \frac{(Q_r)(T_m)P_{bm}}{(Q_m)(T_r)\left(P_{bm} + \frac{P_g}{P_s}\right)} \qquad \text{Eq. 2D-1}$$

where:

Q_r = reference meter flow rate reading, m³/min (ft³/min).

Q_m = measuring device flow rate reading, m³/min (ft³/min).

T_r = reference meter average absolute temperature, °K (°R).

T_m = measuring device average absolute temperature, °K (°R).

P_{bm} = barometric pressure, mm Hg (in. Hg).

P_g = measuring device average static pressure, mm Hg (in. Hg).

For measuring devices that do not have a readout as flow rate, refer to the manufacturer's instructions to calculate the Q_m corresponding to each Q_r.

4.2 Temperature Gauge. Use the procedure and specifications in Method 2A, Section 4.2. Perform the calibration at a temperature that approximates field test conditions.

4.3 Barometer. Calibrate the barometer to be used in the field test with a mercury barometer prior to the field test.

5. Gas Flow Rate Calculation

Calculate the stack gas flow rate, Q_s, as follows:

$$Q_s = K_1 Y_m Q_m \frac{\left(P_{bm} + \frac{P_g}{P_s}\right)}{T_m} \qquad \text{Eq. 2D-2}$$

Where:

K_1 = 0.2669 for International system of units (SI); 17.64 for English units.

6. Bibliography

1. Spink, L.K. Principles and Practice of Flowmeter Engineering. The Foxboro Company. Foxboro, MA. 1967.

2. Benedict, Robert P. Fundamentals of Temperature, Pressure, and Flow Measurements. John Wiley and Sons, Inc. New York, NY. 1969.

3. Orifice Metering of Natural Gas. American Gas Association. Arlington, VA. Report No. 3. March 1978. 88 p.

Method 3—Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO_2), percent oxygen (O_2), and, if necessary, percent carbon monoxide (CO). If a *dry molecular weight* determination is to be made, either an Orsat or a Fyrite[1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO_2 and O_2 concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, CO, and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency.

2. Apparatus

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials inert to O_2, CO_2, CO, and N_2, and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

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

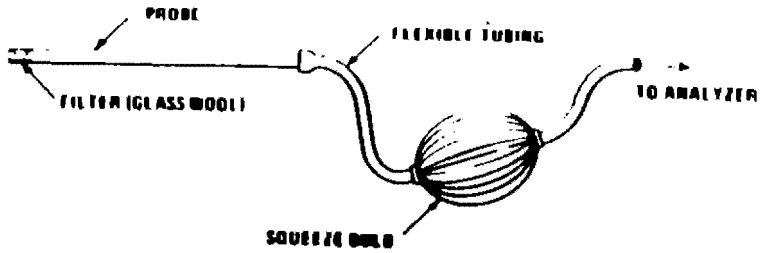

Figure 3-1 Grab sampling train

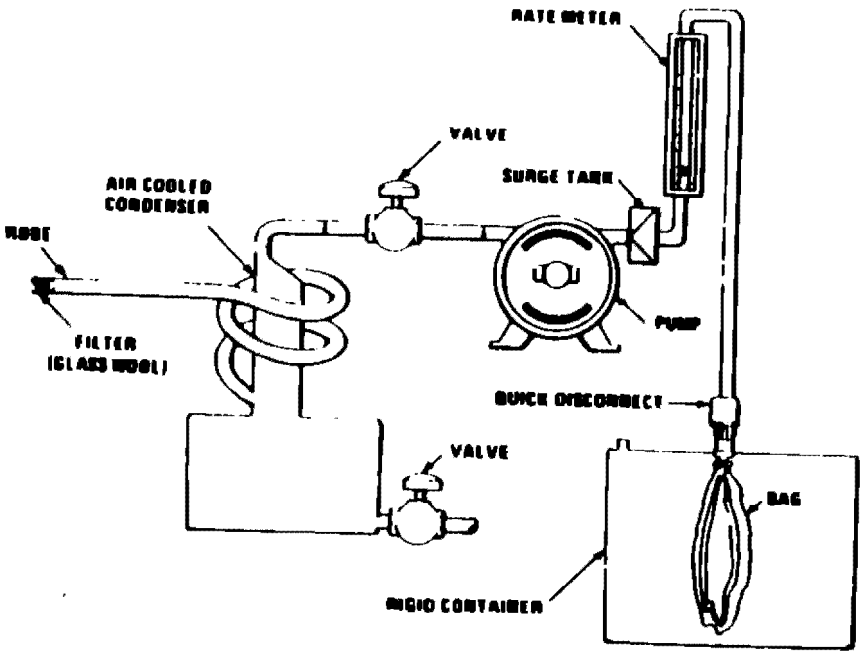

Figure 3-2 Integrated gas sampling train

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2, CO_2, CO, and N_2, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ±2 percent of the selected flow rate. A flow rate range of 500 to 1000 cm^3/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. *Dry Molecular Weight Determination*

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leak-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2, using either an Orsat analyzer or a Fyrite-type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional.

Determine the percentage of the gas that is N, and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.6 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-2.

4. *Emission Rate Correction Factor or Excess Air Determination*

NOTE: A Pyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

FIGURE 3-3—SAMPLING RATE DATA

Time	Traverse pt	Q 1pm	% dev.*
Average			

*% Dev. = $(Q - Q_{avg})/Q_{avg} \times 100$ (Must be ≤ ±10%.)

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N, by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.3.

4.1.4 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis.

NOTE: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 4.4 be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.1.5. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft³) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N, by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.3.

4.2.5 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more that (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.2 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before an after the analysis.

NOTE: Although in most instances only CO, or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 4.4.1 be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4.4 Quality Control Procedures.

4.4.1 Data Validation When Both CO_2 and O_2 Are Measured. Although in most instances, only CO_2 or O_2 measurement is required, it is recommended that both CO_2 and O_2 be measured to provide a check on the quality of the data. The following quality control procedure is suggested.

NOTE: Since the method for validating the CO_2 and O_2 analyses is based on combustion of organic and fossil fuels and dilution of the gas stream with air, this method does not apply to sources that (1) remove CO_2 or O_2 through processes other than combustion, (2) add O_2 (e.g., oxygen enrichment) and N, in proportions different from that of air, (3) add CO_2 (e.g., cement or lime kilns), or (4) have no fuel factor, F_o values obtainable (e.g., extremely variable waste mixtures). This method validates the measured proportions of CO_2 and O_2 for the fuel type, but the method does not detect sample dilution resulting from leaks during or after sample collection. The method is applicable for samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO_2 added or removed from the gas stream is not significant in relation to the total CO_2 concentration. The CO_2 concentrations from other types of scrubbers using only water or basic slurry can be significantly affected and would render the F_o check minimally useful.

4.4.1.1 Calculate a fuel factor, F_o, using the following equation:

$$F_c = \frac{20.9 - \%O_2}{\%CO_2}$$

Eq. 3-3

Where:

$\%O_2$ = Percent O_2 by volume (dry basis).
$\%CO_2$ = Percent CO_2 by volume (dry basis).
20.9 = Percent O_2 by volume in ambient air.

If CO is present in quantities measurable by this method, adjust the O_2 and CO_2 values before performing the calculation for F_c as follows:

$$\%CO_2(adj) = \%CO_2 + \%CO$$

$$\%O_2(adj) = \%O_2 - 0.5\ \%CO$$

Where:

%CO = Percent CO by volume (dry basis).

4.4.1.3 Compare the calculated F_c factor with the expected F_c value. The following table may be used in establishing acceptable ranges for the expected F_c if the fuel being burned is known. When fuels are burned in combination, calculate the combined fuel F_o and F_c factors (as defined in Method 19) according to the procedure in Method 19 Section 5.2.3. Then calculate the F_c factor as follows:

$$F_c = \frac{0.209\ F_o}{F_c}$$

Eq. 3-4

Fuel type	F_c range
Coal	
Anthracite and lignite	1.016-1.130
Bituminous	1.083-1.230
Oil	
Distillate	1.260-1.413
Residual	1.210-1.370
Gas	
Natural	1.000-1.120
Propane	1.426-1.586
Butane	1.405-1.553
Wood	1.000-1.120
Wood bark	1.003-1.130

Calculated F_c values beyond the acceptable ranges shown in this table should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ±10 percent is appropriate for the F_c factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate, i.e., if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time-consuming and costly.

6. *Leak-Check Procedure for Orsat Analyzers*

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

6.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

6.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

6.3 Record the meniscus position.

6.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

6.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

6.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

6.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

6.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. Calculations

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).
%EA = Percent excess air.
$\%CO_2$ = Percent CO_2 by volume (dry basis).
$\%O_2$ = Percent O_2 by volume (dry basis).
%CO = Percent CO by volume (dry basis).
$\%N_2$ = Percent N_2 by volume (dry basis).
0.264 = Ratio of O_2 to N_2 in air, v/v.
0.280 = Molecular weight of N_2 or CO, divided by 100.
0.320 = Molecular weight of O_2, divided by 100.
0.440 = Molecular weight of CO_2 divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of percent O_2, CO, and N_2 (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA = \frac{\%O_2 - 0.5\%\ CO}{0.264\ \%N_2 - (\%O_2 - 0.5\ \%CO)}$$

Eq. 3-1

NOTE: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO)$$

Eq. 3-2

NOTE: The above equation does not consider argon in air (about 0.9 percent, molecular weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. *Bibliography*

1. Altshuller, A. P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. 6:75-81. 1963.

2. Conner, William D. and J. S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. 25:291-297. 1964.

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4. Mitchell, W. J. and M. R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association 26:491-495. May 1976.

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Method 3A—Determination of Oxygen and Carbon Dioxide Concentrations in Emissions From Stationary Sources (Instrumental Analyzer Procedure)

1. *Applicability and Principle*

1.1 Applicability. This method is applicable to the determination of oxygen (O_2) and carbon dioxide (CO_2) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A sample is continuously extracted from the effluent stream, a portion of the sample stream is conveyed to an instrumental analyzer(s) for determination of O_2 and CO_2 concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

Same as Method 6C, Sections 2.1 and 2.2, except that the span of the monitoring system shall be selected such that the average O_2 or CO_2 concentration is not less than 20 percent of the span.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of the O_2 or CO_2 concentration. The measurement system consists of the same major subsystems as defined in Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, Response Time, and Calibration Curve. Same as Method 6C, Sections 3.3 through 3.9, and 3.10.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. *Measurement System Performance Specifications*

Same as Method 6C, Sections 4.1 through 4.4.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for O_2 or CO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe. A leak-free probe, of sufficient length to traverse the sample points.

5.1.2 Sample Line. Tubing, to transport the sample gas from the probe to the moisture removal system. A heated sample line is not required for systems that measure the O_2 or CO_2 concentration on a dry basis, or transport dry gases.

5.1.3 Sample Transport Line, Calibration Value Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.3 through 5.1.9, and 5.1.11, except that the requirements to use stainless steel, Teflon, and nonreactive glass filters do not apply.

5.1.4 Gas Analyzer. An analyzer to determine continuously the O_2 or CO_2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g.,

Method 3 - Gas Analysis for the Determination of Dry Molecular Weight

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability.

1.1.1 This method is applicable for determining carbon dioxide (CO_2) and oxygen (O_2) concentrations and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, carbon monoxide (CO), and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

1.1.2 Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight; and (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency (EPA).

1.1.3 **Note.** Mention of trade names or specific products does not constitute endorsement by EPA.

1.2 **Principle.** A gas sample is extracted from a stack by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2, percent O_2, and if necessary, for percent CO. For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.

2. APPARATUS

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. Stainless steel or borosilicate glass tubing equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials, inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions, may be used for the probe. Examples of such materials are aluminum, copper, quartz glass, and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. Same as in Section 2.1.1.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser no greater than 250 ml that will not remove O_2, CO_2, CO, and N_2, to remove excess moisture which would interfere with the operation of the pump and flowmeter.

2.2.3 Valve. A needle valve, to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. A rotameter, or equivalent rate meter, capable of measuring flow rate to within 2 percent of the selected flow rate. A flow rate range of 500 to 1000 cc/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run. A capacity in the range of 55 to 90 liters is suggested. To leak check the bag, connect it to a water manometer, and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm (2 to 4 in.) H_2O and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.), for the flexible bag leak check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm (30 in.) Hg, for the sampling train leak check.

2.3 Analysis. An Orsat or Fyrite type combustion gas analyzer. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

3. SINGLE-POINT, GRAB SAMPLING AND ANALYTICAL PROCEDURE

3.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight. If an Orsat analyzer is used, it is recommended that the analyzer be leak checked by following the procedure in Section 6; however, the leak check is optional.

3.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer, and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 7.2.

3.4 Repeat the sampling, analysis, and calculation procedures until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

4. SINGLE-POINT, INTEGRATED SAMPLING AND ANALYTICAL PROCEDURE

4.1 The sampling point in the duct shall be located as specified in Section 3.1.

4.2 Leak check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just before sampling, leak check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag, and make sure that all connections are tight.

4.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

4.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that Orsat leak check described in Section 6, be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 7.2.

4.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

5. MULTI-POINT, INTEGRATED SAMPLING AND ANALYTICAL PROCEDURE

5.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

5.2 Follow the procedures outlined in Sections 4.2 through 4.5, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

6. LEAK-CHECK PROCEDURE FOR ORSAT ANALYZER

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak checked on site before the flue gas sample is introduced into it. The procedure for leak checking an Orsat analyzer is as follows:

6.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.

6.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.

6.3 Record the meniscus position.

6.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

6.5 For the Orsat analyzer to pass the leak check, two conditions must be met:

6.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

6.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

6.6 If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease leaking stopcocks. Replace leaking rubber connections. After the analyzer is reassembled, repeat the leak-check procedure.

7. CALCULATIONS

7.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).

$\%CO_2$ = Percent CO_2 by volume, dry basis.

$\%O_2$ = Percent O_2 by volume, dry basis.

%CO = Percent CO by volume, dry basis.

$\%N_2$ = Percent N_2 by volume, dry basis.

0.280 = Molecular weight of N_2 or CO, divided by 100.

0.320 = Molecular weight of O_2 divided by 100.

0.440 = Molecular weight of CO_2 divided by 100.

7.2 Dry Molecular Weight. Use Equation 3-I to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO) \qquad \text{Eq. 3-1}$$

Note: The above equation does not consider argon in air (about 0.9 percent, molecular weight of 39.9). A negative error of about 0.4 percent is introduced. The tester may choose to include argon in the analysis using procedures subject to approval of the Administrator.

8. BIBLIOGRAPHY

1. Altshuller, A.P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. 6:75-81. 1963.

2. Conner, William D. and J.S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. 25:291-297. 1964.

3. Burrell Manual for Gas Analysts, Seventh edition. Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, PA. 15219. 1951.

4. Mitchell, W.J. and M.R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association. 26:491-495. May 1976.

5. Shigehara, R.T., R.M. Neulicht, and W.S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. 4(2):21-26. August 1976.

Time	Traverse pt.	Q, liter/min	% dev.[a]
Average			

[a] % dev. = $(Q - Q_{avg})/Q_{avg}$ x 100 (Must be $\leq|10\%|$)

Figure 3-3. Sampling rate data.

EPA METHOD 10

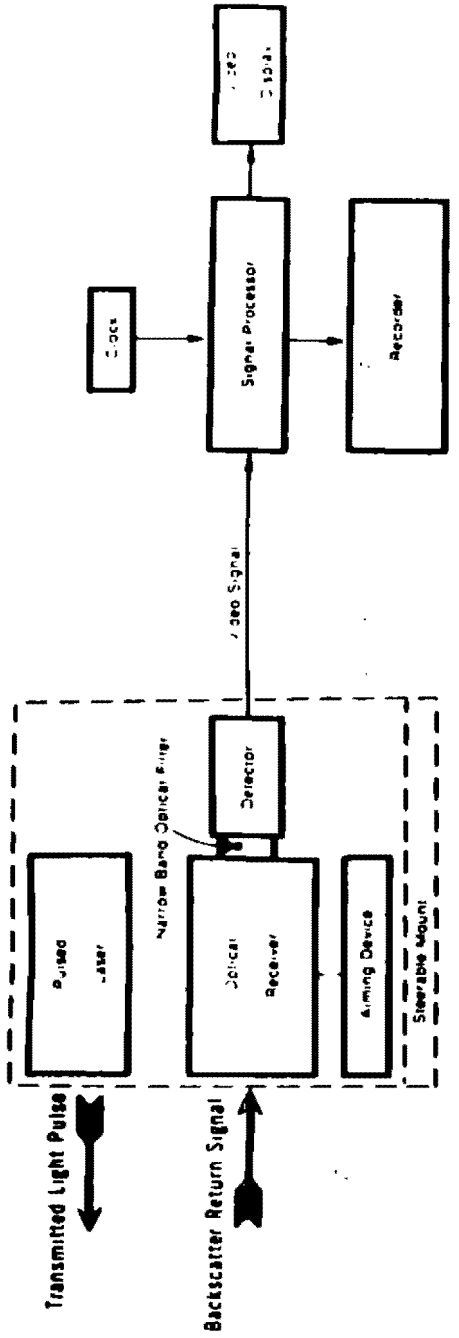

Figure AMI-VII. Functional Block Diagram of a Basic Lidar System

4.2 Performance Evaluation Tests. The owner of a lidar system shall subject such a lidar system to the performance verification tests described in Section 3, prior to first use of this method. The annual calibration shall be performed for three separate, complete runs and the results of each should be recorded. The requirements of Section 3.3.1 must be fulfilled for each of the three runs.

Once the conditions of the annual calibration are fulfilled the lidar shall be subjected to the routine verification for three separate complete runs. The requirements of Section 3.3.2 must be fulfilled for each of the three runs and the results should be recorded. The Administrator may request that the results of the performance evaluation be submitted for review.

5. *References*

5.1 The Use of Lidar for Emissions Source Opacity Determination, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA-330/1-79-003-R, Arthur W. Dybdahl, current edition [NTIS No. PB81-246662].

5.2 Field Evaluation of Mobile Lidar for the Measurement of Smoke Plume Opacity, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA/NEIC-TS-128, February 1976.

5.3 Remote Measurement of Smoke Plume Transmittance Using Lidar, C. S. Cook, G. W. Bethke, W. D. Conner (EPA/RTP). Applied Optics 11, pg 1742, August 1972.

5.4 Lidar Studies of Stack Plumes in Rural and Urban Environments, EPA-650/4-73-002, October 1973.

5.5 American National Standard for the Safe Use of Lasers ANSI Z 136.1-170, March 8, 1976.

5.6 U.S. Army Technical Manual TB MED 279, Control of Hazards to Health from Laser Radiation, February 1969.

5.7 Laser Institute of America Laser Safety Manual, 4th Edition.

5.8 U.S. Department of Health, Education and Welfare, Regulations for the Administration and Enforcement of the Radiation Control for Health and Safety Act of 1968, January 1976.

5.9 Laser Safety Handbook, Alex Mallow, Leon Chabot, Van Nostrand Reinhold Co., 1978.

Method 10—Determination of Carbon Monoxide Emissions From Stationary Sources

1. *Principle and Applicability*

1.1 Principle. An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 Applicability. This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and Sensitivity*

2.1 Range. 0 to 1,000 ppm.

2.2 Sensitivity. Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences*

Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H_2O) and carbon dioxide (CO_2) are 3.5 percent H_2O per 7 ppm CO and 10 percent CO_2 per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H_2O per 25 ppm CO and 10 percent CO_2 per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and Accuracy*

4.1 Precision. The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 Accuracy. The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus*

5.1 Continuous Sample (Figure 10-1).

5.1.1 Probe. Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2 Integrated Sample (Figure 10-2).

5.2.1 Probe. Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

5.2.3 Valve. Needle valve, or equivalent, to to adjust flow rate.

5.2.4 Pump. Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 Rate Meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min (0.035 cfm).

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

5.3.6 Flexible Bag. Tedlar, or equivalent, with a capacity of 80 to 90 liters (2 to 3 ft³). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.3.7 Pitot Tube. Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 Analysis (Figure 10-3).

5.3.1 Carbon Monoxide Analyzer. Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 Drying Tube. To contain approximately 200 g of silica gel.

5.3.3 Calibration Gas. Refer to section 6.1.

5.3.4 Filter. As recommended by NDIR manufacturer.

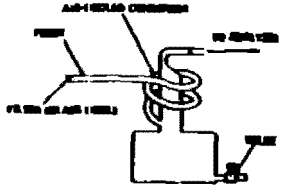

Figure 10-1. Continuous sampling train.

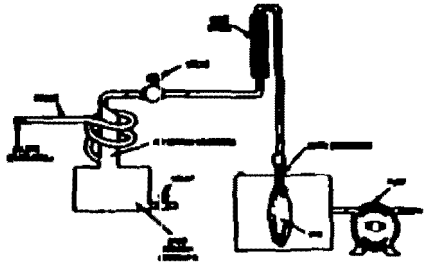

Figure 10-2. Integrated gas sampling train.

5.3.5 CO_2 Removal Tube. To contain approximately 500 g of ascarite.

5.3.6 Ice Water Bath. For ascarite and silica gel tubes.

5.3.7 Valve. Needle valve, or equivalent, to adjust flow rate

5.3.8 Rate Meter. Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min (0.035 cfm) through NDIR.

5.3.9 Recorder (*optional*). To provide permanent record of NDIR readings.

6. *Reagents*

6.1 Calibration Gases. Known concentration of CO in nitrogen (N_2) for instrument span, prepurified grade of N_2 for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

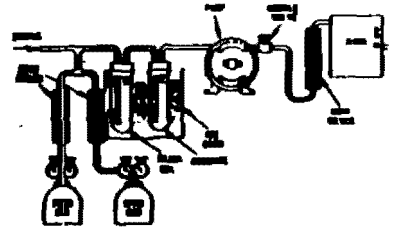

Figure 10-3. Analytical equipment.

6.2 Silica Gel. Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 Ascarite. Commercially available.

7. *Procedure*

7.1 Sampling.

7.1.1 Continuous Sampling. Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See section 7.2 and 8). CO_2 content of the gas may be determined by using the Method 3 integrated sample procedure, or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 Integrated Sampling. Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO_2 content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.2 CO Analysis. Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in section 8. Purge analyzer with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration*

Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of 1 hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO_2 removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1—FIELD DATA

	Comments
Location	
Test	
Date	
Operator	

Clock time	Rotameter setting, liters per minute (cubic feet per minute)

9. *Calculation*

Concentration of carbon monoxide. Calculate the concentration of carbon monoxide in the stack using Equation 10-1.

$$C_{CO,\,stack} = C_{CO,\,NDIR}(1 - F_{CO_2}) \qquad \text{Eq. 10-1}$$

Where:

$C_{CO,\,stack}$ = Concentration of CO in stack, ppm by volume (dry basis).

$C_{CO,\,NDIR}$ = Concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

F_{CO_2} = Volume fraction of CO_2 in sample, i.e., percent CO_2 from Orsat analysis divided by 100.

10. *Alternative Procedures*

10.1 Interference Trap. The sample conditioning system described in Method 10A, sections 2.1.2 and 4.2, may be used as an alternative to the silica gel and ascarite traps

11. *Bibliography*

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11.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, PA.

11.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, CA. October 1967.

11.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, NJ.

11.6 UNOR Infrared Gas Analyzers, Bendix Corp., Ronceverte, WV

ADDENDA

A. PERFORMANCE SPECIFICATIONS FOR NDIR CARBON MONOXIDE ANALYZERS

Range (minimum)	0-1000 ppm
Output (minimum)	0-10mV
Minimum detectable sensitivity	20 ppm
Rise time, 90 percent (maximum)	30 seconds
Fall time, 90 percent (maximum)	30 seconds
Zero drift (maximum)	10% in 8 hours
Span drift (maximum)	10% in 8 hours
Precision (minimum)	±2% of full scale
Noise (maximum)	±1% of full scale
Linearity (maximum deviation)	2% of full scale
Interference rejection ratio	CO_2—1000 to 1, H_2O—500 to 1

B. *Definitions of Performance Specifications.*

Range—The minimum and maximum measurement limits.

Output—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

Full scale—The maximum measuring limit for a given range.

Minimum detectable sensitivity—The smallest amount of input concentration that can be detected as the concentration approaches zero.

Accuracy—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

Time to 90 percent response—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

Rise Time (90 percent)—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

Fall Time (90 percent)—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

Zero Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

Span Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

Precision—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

Noise—Spontaneous deviations from a mean output not caused by input concentration changes.

Linearity—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

Method 10A—Determination of Carbon Monoxide Emissions in Certifying Continuous Emission Monitoring Systems at Petroleum Refineries

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of carbon monoxide (CO) at petroleum refineries. This method serves as the reference method in the relative accuracy test for nondispersive infrared (NDIR) CO continuous emission monitoring systems (CEMS's) that are required to be installed in petroleum refineries on fluid catalytic cracking unit catalyst regenerators [40 CFR Part 60.105(a)(2)].

1.2 Principle. An integrated gas sample is extracted from the stack, passed through an alkaline permanganate solution to remove sulfur and nitrogen oxides, and collected in a Tedlar bag. The CO concentration in the sample is measured spectrophotometrically using the reaction of CO with p-sulfaminobenzoic acid.

1.3 Range and Sensitivity.

1.3.1 Range. Approximately 3 to 1800 ppm CO. Samples having concentrations below 400 ppm are analyzed at 425 nm, and samples having concentrations above 400 ppm are analyzed at 660 nm.

1.3.2 Sensitivity. The detection limit is 3 ppm based on three times the standard deviation of the mean reagent blank values.

1.4 Interferences. Sulfur oxides, nitric oxide, and other acid gases interfere with the colorimetric reaction. They are removed by passing the sampled gas through an alkaline potassium permanganate scrubbing solution. Carbon dioxide (CO_2) does not interfere, but, because it is removed by the scrubbing solution, its concentration must be measured independently and an appropriate volume correction made to the sampled gas.

1.5 Precision, Accuracy, and Stability.

1.5.1 Precision. The estimated intralaboratory standard deviation of the method is 3 percent of the mean for gas samples analyzed in duplicate in the concentration range of 39 to 412 ppm. The interlaboratory precision has not been established.

1.5.2 Accuracy. The method contains no significant biases when compared to an NDIR analyzer calibrated with National Bureau of Standards (NBS) standards.

1.5.3 Stability. The individual components of the colorimetric reagent are stable for at least 1 month. The colorimetric reagent must be used within 2 days after preparation to avoid excessive blank correction. The samples in the Tedlar [1] bag should be stable for at least 1 month if the bags are leak-free.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 10A-1, and component parts are discussed below:

[1] Mention of trade names or commercial products in this publication does not constitute the endorsement or recommendation for use by the Environmental Protection Agency.

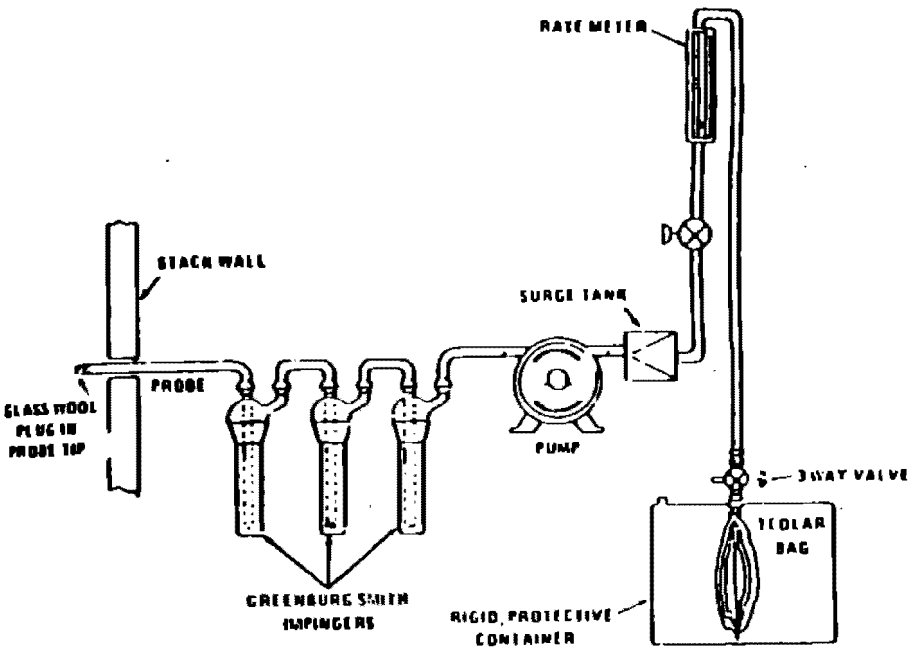

Figure 10A-1. Sampling train.

2.1.1 Probe. Stainless steel, sheathed Pyrex glass, or equivalent, equipped with a glass wool plug to remove particulate matter.

2.1.2 Sample Conditioning System. Three Greenburg-Smith impingers connected in series with leak-free connections.

2.1.3 Pump. Leak-free pump with stainless steel and Teflon parts to transport sample at a flow rate of 300 ml/min to the flexible bag.

2.1.4 Surge Tank. Installed between the pump and the rate meter to eliminate the pulsation effect of the pump on the rate meter.

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The sampling train is assembled as shown in Figure 6A-1, except the isopropanol bubbler is not included. The probe must be heated to a temperature sufficient to prevent water condensation and must include a filter (either in-stack, out-of-stack, or both) to prevent particulate entrainment in the peroxide impingers. The electric supply for the probe heat should be continuous and separate from the timed operation of the sample pump.

Adjust the timer-switch to operate in the "on" position from 2 to 4 minutes on a 2-hour repeating cycle or other cycle specified in the applicable regulation. Other timer sequences may be used with the restriction that the total sample volume collected is between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

Add cold water to the tank until the impingers and bubblers are covered at least two-thirds of their length. The impingers and bubbler tank must be covered and protected from intense heat and direct sunlight. If freezing conditions exist, the impinger solution and the water bath must be protected.

NOTE: Sampling may be conducted continuously if a low flow-rate sample pump (20 to 40 ml/min for the reagent volumes described in this method) is used. Then the timer-switch is not necessary. In addition, if the sample pump is designed for constant rate sampling, the rate meter may be deleted. The total gas volume collected should be between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

6.1.2 Leak Check Procedure. The leak-check procedure is the same as described in Method 6, Section 6.1.2.

6.1.3 Sample Collection. Record the initial dry gas meter reading. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the first impinger (or filter), and start the timer and the sample pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Ensure that the timer is operating as intended, i.e., in the "on" position for the desired period and the cycle repeats as required.

During the 24-hour sampling period, record the dry gas meter temperature one time between 9:00 a.m. and 11:00 a.m., and the barometric pressure.

At the conclusion of the run, turn off the timer and the sample pump, remove the probe from the stack, and record the final gas meter volume reading. Conduct a leak check as described in Section 6.1.2. If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for leakage. Repeat the steps in this section (6.1.3) for successive runs.

6.2 Sample Recovery. The procedures for sample recovery (moisture measurement, peroxide solution, and CO_2 absorber) are the same as in Method 6A, Section 6.2.

6.3 Sample Analysis. Analysis of the peroxide impinger solutions is the same as in Method 6, Section 6.3.

6.4 Quality Assurance (QA) Audit Samples. Only when this method is used for compliance determinations, obtain an audit sample set as directed in Section 3.3.6 of Method 6. Analyze the audit samples at least once for every 30 days of sample collection, and report the results as directed in Section 6.4 of Method 6. The analyst performing the sample analyses shall perform the audit analyses. If more than one analyst performed the sample analyses during the 30-day sampling period, each analyst shall perform the audit analyses and all audit results shall be reported. Acceptance criteria for the audit results are the same as in Method 6.

5. *Calibration*

5.1 Metering System.

5.1.1 Initial Calibration. The initial calibration for the volume metering system is the same as for Method 6, Section 5.1.1.

5.1.2 Periodic Calibration Check. After 30 days of operation of the test train, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (1) The leak check is not to be conducted, (2) three or more revolutions of the dry gas meter must be used, and (3) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor determined in Section 5.1.1, then the dry gas meter volumes obtained during the test series are acceptable and use of the train can continue. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1; and for the calculations for the preceding 30 days of data, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run. Use the latest calibration factor for succeeding tests.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers initially and at 30 day intervals.

5.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instructions.

5.4 Barometer. Calibrate against a mercury barometer initially and at 30 day intervals.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

6. *Calculations*

The nomenclature and calculation procedures are the same as in Method 6A with the following exceptions:

P_{bar} = Initial barometric pressure for the test period, mm Hg.

T_m = Absolute meter temperature for the test period, °K.

7. *Emission Rate Procedure*

The emission rate procedure is the same as described in Method 6A, Section 7, except that the timer is needed and is operated as described in this method. Only when this method is used for compliance determinations, perform the QA audit analyses as described in Section 6.4.

8. *Bibliography*

The bibliography is the same as described in Method 6A, section 7, with the addition of the following:

8.1 Butler, Frank E; J.E. Knoll, J.C. Suggs, M.R. Midgett, and W. Mason. The *Collaborative Test of Method 6B: Twenty-Four-Hour Analysis of SO_2 and CO_2*. JAPCA. Vol. 33, No. 10. October 1983.

Method 6C—Determination of Sulfur Dioxide Emissions From Stationary Sources (Instrumental Analyzer Procedure)

1. *Applicability and Principle*

1.1 *Applicability.* This method is applicable to the determination of sulfur dioxide (SO_2) concentrations in controlled and uncontrolled emissions from stationary sources only when specified within the *regulations.*

1.2 Principle. A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental analyzer for determination of SO_2 gas concentration using an ultraviolet (UV), nondispersive infrared (NDIR), or fluorescence analyzer. *Performance specifications and test procedures are provided to ensure reliable data.*

2. *Range and Sensitivity*

2.1 Analytical Range. The analytical range is determined by the instrumental design. For this method, a portion of the analytical range is selected by choosing the *span of the monitoring system.* The span of the monitoring system shall be selected such that the pollutant gas concentration equivalent to the emission standard is not less than 30 percent of the span. If at any time during a run the measured gas concentration exceeds the span, the run shall be considered invalid.

2.2 Sensitivity. The minimum detectable limit depends on the analytical range, span, and signal-to-noise ratio of the measurement system. For a well designed system, the minimum detectable limit should be less than 2 percent of the span.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface. That *portion of a* system used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the analyzers from the effects of the stack effluent.

3.1.2 Gas Analyzer. That portion of the system that senses the gas to be measured and generates an output proportional to its concentration.

3.1.3 Data Recorder. A strip chart recorder, analog computer, or digital recorder for recording measurement data from the analyzer output.

3.2 Span. The upper limit of the gas concentration *measurement range displayed* on the data recorder.

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

3.4 Analyzer Calibration Error. The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the calibration gas *when* the calibration gas is introduced directly to the analyzer.

3.5 Sampling System Bias. The difference between the gas concentrations exhibited by the measurement system when a known concentration gas is introduced at the outlet of the sampling probe and when the same gas is introduced directly to the analyzer.

3.6 Zero Drift. The difference in the measurement system output reading from the initial calibration response at the zero concentration level after a stated period of operation during which no unscheduled maintenance, repair, or *adjustment* took place.

3.7 Calibration Drift. The difference in the measurement system output reading from the initial calibration response at a mid-range calibration value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

3.8 Response Time. The amount of time required for the measurement system to display 95 percent of a step change in gas concentration on the data recorder.

3.9 Interference Check. A method for detecting analytical interferences and excessive *biases* (through direct comparison of gas concentrations provided by the measurement system and by a modified Method 6 procedure. For this check, the modified

Method 6 samples are acquired at the sample by pass discharge vent.

3.10 Calibration Curve. A graph or other systematic method of establishing the relationship between the analyzer response and the actual gas concentration introduced to the analyzer.

4. Measurement System Performance Specifications

4.1 Analyzer Calibration Error. Less than ±2 percent of the span for the zero, mid-range, and high-range calibration gases.

4.2 Sampling System Bias. Less than ±5 percent of the span for the zero, and mid- or high-range calibration gases.

4.3 Zero Drift. Less than ±2 percent of the span over the period of each run.

4.4 Calibration Drift. Less than ±3 percent of the span over the period of each run.

4.5 Interference Check. Less than ±7 percent of the modified Method 6 result for each run.

5. Apparatus and Reagents

5.1 Measurement System. Any measurement system for SO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1. The essential components of the measurement system are described below:

5.1.1 Sample Probe. Glass, stainless steel, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation.

5.1.2 Sample Line. Heated (sufficient to prevent condensation) stainless steel or Teflon tubing, to transport the sample gas to the moisture removal system.

5.1.3 Sample Transport Lines. Stainless steel or Teflon tubing, to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

5.1.4 Calibration Valve Assembly. A three-way valve assembly, or equivalent, for blocking the sample gas flow and introducing calibration gases to the measurement system at the outlet of the sampling probe when in the calibration mode.

5.1.5 Moisture Removal System. A refrigerator-type condenser or similar device (e.g., permeation dryer), to remove condensate continuously from the sample gas while maintaining minimal contact between the condensate and the sample gas. The moisture removal system is not necessary for analyzers that can measure gas concentrations on a wet basis; for these analyzers, (1) heat the sample line and all interface components up to the inlet of the analyzer sufficiently to prevent condensation, and (2) determine the moisture content and correct the measured gas concentrations to a dry basis using appropriate methods, subject to the approval of the Administrator. The determination of sample moisture content is not necessary for pollutant analyzers that measure concentrations on a wet basis when (1) a wet basis CO_2 analyzer operated according to Method 3A is used to obtain simultaneous measurements, and (2) the pollutant/CO_2 measurements are used to determine emissions in units of the standard.

5.1.6 Particulate Filter. An in-stack or heated (sufficient to prevent water condensation) out-of-stack filter. The filter shall be borosilicate or quartz glass wool, or glass fiber mat. Additional filters at the inlet or outlet of the moisture removal system and inlet of the analyzer may be used to prevent accumulation of particulate material in the measurement system and extend the useful life of the components. All filters shall be fabricated of materials that are nonreactive to the gas being sampled.

5.1.7 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The pump may be constructed of any material that is nonreactive to the gas being sampled.

5.1.8 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent.

(Note: The tester may elect to install a back-pressure regulator to maintain the sample gas manifold at a constant pressure in order to protect the analyzer(s) from overpressurization, and to minimize the need for flow rate adjustments.)

5.1.9 Sample Gas Manifold. A sample gas manifold, to divert a portion of the sample gas stream to the analyzer, and the remainder to the by-pass discharge vent. The sample gas manifold should also include provisions for introducing calibration gases directly to the analyzer. The manifold may be constructed of any material that is nonreactive to the gas being sampled.

5.1.10 Gas Analyzer. A UV or NDIR absorption or fluorescence analyzer, to determine continuously the SO_2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

(Note: Housing the analyzer(s) in a clean, thermally-stable, vibration-free environment will minimize drift in the analyzer calibration.)

5.1.11 Data Recorder. A strip chart recorder, analog computer, or digital recorder, for recording measurement data. The data recorder resolution (i.e., readability) shall be 0.5 percent of span. Alternatively, a digital or analog meter having a resolution of 0.5 percent of span may be used to obtain the analyzer responses and the readings may be recorded manually. If this alternative is used, the readings shall be obtained at equally spaced intervals over the duration of the sampling run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be obtained. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 60 measurements, whichever is less restrictive, shall be obtained.

5.2 Method 6 Apparatus and Reagents. The apparatus and reagents described in Method 6, and shown by the schematic of the sampling train in Figure 6C-2, to conduct the interference check.

5.3 SO_2 Calibration Gases. The calibration gases for the gas analyzer shall be SO_2 in N_2 or SO_2 in air. Alternatively, SO_2/CO_2, SO_2/O_2, or $SO_2/CO_2/O_2$ gas mixtures in N_2 may be used. For fluorescence-based analyzers, the O_2 and CO_2 concentrations of the calibration gases as introduced to the analyzer shall be within 1 percent (absolute) O_2 and 1 percent (absolute) CO_2 of the O_2 and CO_2 concentrations of the effluent samples as introduced to the analyzer. Alternatively, for fluorescence-based analyzers, use calibration blends of SO_2 in air and the nomographs provided by the vendor to determine the quenching correction factor (the effluent O_2 and CO_2 concentrations must be known). Use three calibration gases as specified below:

5.3.1 High-Range Gas. Concentration equivalent to 80 to 90 percent of the span.

5.3.2 Mid-Range Gas. Concentration equivalent to 50 to 60 percent of the span.

5.3.3 Zero Gas. Concentration of less than 0.25 percent of the span. Purified ambient air may be used for the zero gas by passing air through a charcoal filter, or through one or more impingers containing a solution of 3 percent H_2O_2.

6. Measurement System Performance Test Procedures

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. There are two alternatives for establishing the concentrations of calibration gases. Alternative Number 1 is preferred.

6.1.1 Alternative Number 1. Use of calibration gases that are analyzed following the Environmental Protection Agency Traceability Protocol Number 1 (see Citation 1 in the Bibliography). Obtain a certification from the gas manufacturer that Protocol Number 1 was followed.

6.1.2 Alternative Number 2. Use of calibration gases not prepared according to Protocol Number 1. If this alternative is chosen, obtain gas mixtures with a manufacturer's tolerance not to exceed ±2 percent of the tag value. Within 6 months before the emission test, analyze each of the calibration gases in triplicate using Method 6. Citation 3 in the Bibliography describes procedures and techniques that may be used for this analysis. Record the results on a data sheet (example is shown in Figure 6C-3). Each of the individual SO_2 analytical results for each calibration gas shall be within 5 percent (or 5 ppm, whichever is greater) of the triplicate set average; otherwise, discard the entire set, and repeat the triplicate analyses. If the average of the triplicate analyses is within 5 percent of the calibration gas manufacturer's cylinder tag value, use the tag value; otherwise, conduct at least three additional analyses until the results of six consecutive runs agree with 5 percent (or 5 ppm, whichever is greater) of their average. Then use this average for the cylinder value.

6.2 Measurement System Preparation. Assemble the measurement system by following the manufacturer's written instructions for preparing and preconditioning the gas analyzer and, as applicable, the other system components. Introduce the calibration gases in any sequence, and make all necessary adjustments to calibrate the analyzer and the data recorder. Adjust system components to achieve correct sampling rates.

6.3 Analyzer Calibration Error. Conduct the analyzer calibration error check by introducing calibration gases to the measurement system at any point upstream of the gas analyzer as follows:

6.3.1 After the measurement system has been prepared for use, introduce the zero, mid-range, and high-range gases to the analyzer. During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate at the analyzer. Record the analyzer responses to each calibration gas on a form similar to Figure 6C-4.

Note: A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

6.3.2 The analyzer calibration error check shall be considered invalid if the gas concentration displayed by the analyzer exceeds ±2 percent of the span for any of the calibration gases. If an invalid calibration is exhibited, take corrective action, and repeat

the analyzer calibration error check until acceptable performance is achieved.

6.4 Sampling System Bias Check. Perform the sampling system bias check by introducing calibration gases at the calibration valve installed at the outlet of the sampling probe. A zero gas and either the mid-range or high-range gas, whichever most closely approximates the effluent concentrations, shall be used for this check as follows:

6.4.1 Introduce the upscale calibration gas, and record the gas concentration displayed by the analyzer on a form similar to Figure 6C-5. Then introduce zero gas, and record the gas concentration displayed by the analyzer. During the sampling system bias check, operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to achieve proper calibration gas flow rates at the analyzer. Alternately introduce the zero and upscale gases until a stable response is achieved. The tester shall determine the measurement system response time by observing the times required to achieve a stable response for both the zero and upscale gases. Note the longer of the two times as the response time.

6.4.2 The sampling system bias check shall be considered invalid if the difference between the gas concentrations displayed by the measurement system for the analyzer calibration error check and for the sampling system bias check exceeds ±5 percent of the span for either the zero or upscale calibration gas. If an invalid calibration is exhibited, take corrective action, and repeat the sampling system bias check until acceptable performance is achieved. If adjustment to the analyzer is required, first repeat the analyzer calibration error check, then repeat the sampling system bias check.

7. *Emission Test Procedure*

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to Method 6.

7.2 Interference Check Preparation. For each individual analyzer, conduct an interference check for at least three runs during the initial field test on a particular source category. Retain the results, and report them with each test performed on that source category.

If an interference check is being performed, assemble the modified Method 6 train (flow control valve, two midget impingers containing 3 percent H_2O_2, and dry gas meter) as shown in Figure 6C-2. Install the sampling train to obtain a sample at the measurement system sample by-pass discharge vent. Record the initial dry gas meter reading.

7.3 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as for Method 6 plus twice the system response time. For each run, use only those measurements obtained after twice response time of the measurement system has elapsed, to determine the average effluent concentration. If an interference check is being performed, open the flow control valve on the modified Method 6 train concurrent with the initiation of the sampling period, and adjust the flow to 1 liter per minute (±10 percent).

(NOTE: If a pump is not used in the modified Method 6 train, caution should be exercised in adjusting the flow rate since overpressurization of the impingers may cause leakage in the impinger train, resulting in positively biased results).

7.4 Zero and Calibration Drift Tests. Immediately preceding and following each run, or if adjustments are necessary for the measurement system during the run, repeat the sampling system bias check procedure described in Section 6.4 (Make no adjustments to the measurement system until after the drift checks are completed.) Record and analyzer's responses on a form similar to Figure 6C-6.

7.4.1 If either the zero or upscale calibration value exceeds the sampling system bias specification, then the run is considered invalid. Repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before repeating the run.

7.4.2 If both the zero and upscale calibration values are within the sampling system bias specification, then use the average of the initial and final bias check values to calculate the gas concentration for the run. If the zero or upscale calibration drift value exceeds the drift limits, based on the difference between the sampling system bias check responses immediately before and after the run, repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before conducting additional runs.

7.5 Interference Check (If performed). After completing the run, record the final dry gas meter reading, meter temperature, and barometric pressure. Recover and analyze the contents of the midget impingers, and determine the SO_2 gas concentration using the procedures of Method 6. (It is not necessary to analyze EPA performance audit samples for Method 6.) Determine the average gas concentration exhibited by the analyzer for the run. If the gas concentrations provided by the analyzer and the modified Method 6 differ by more than 7 percent of the modified Method 6 result, the run is invalidated.

8. *Emission Calculation*

The average gas effluent concentration is determined from the average gas concentration displayed by the gas analyzer, and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with Section 7.4. The average gas concentration displayed by the analyzer may be determined by integration of the area under the curve for chart recorders, or by averaging all of the effluent measurements. Alternatively, the average may be calculated from measurements recorded at equally spaced intervals over the entire duration of the run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be used. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 98 measurements, whichever is less restrictive, shall be used. Calculate the effluent gas concentration using Equation 6C-1.

$$C_{ma} = (C - C_o)\frac{C_{ma}}{C_m - C_o}$$

Eq. 6C-1

Where:

C_{ma} = Effluent gas concentration, dry basis, ppm.

C = Average gas concentration indicated by gas analyzer, dry basis, ppm.

C_o = Average of initial and final system calibration bias check responses for the zero gas, ppm.

C_m = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.

C_{ma} = Actual concentration of the upscale calibration gas, ppm.

9. *Bibliography*

1. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors. Protocol Number 1. U.S. Environmental Protection Agency, Quality Assurance Division, Research Triangle Park, NC. June 1978.

2. Westlin, Peter R. and J. W. Brown. Methods for Collecting and Analyzing Gas Cylinder Samples. Source Evaluation Society Newsletter. 3(3):5-15. September 1978.

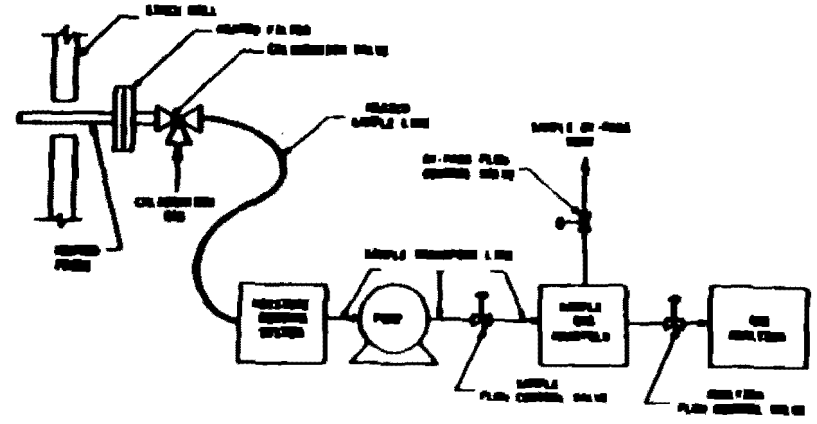

Figure 6C-1. [illegible]

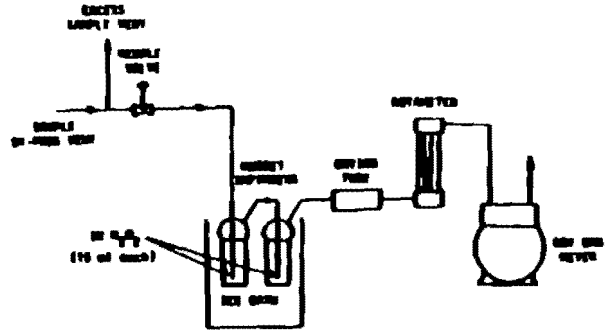

Figure 6C-2. Integrated stack sampling train.

FIGURE 6C-3—ANALYSIS OF CALIBRATION GASES

Date _____ Analytic method used _____

	Gas concentration (indicate units)		
	Zero [a]	Mid-range [b]	High-range [c]
Sample run			
1			
2			
3			
Average			
Maximum percent deviation			

[a] Average must be less than 0.25 percent of span
[b] Average must be 50 to 60 percent of span
[c] Average must be 80 to 90 percent of span

FIGURE 6C-4—ANALYZER CALIBRATION DATA

Source identification: _____ Analyzer calibration data for sampling
Test personnel: _____ runs: _____
Date: _____ Span: _____

	Cylinder value (indicate units)	Analyzer calibration response (indicate units)	Absolute difference (indicate units)	Difference (percent of span)
Zero gas				
Mid-range gas				
High-range gas				

FIGURE 6C-5—SYSTEM CALIBRATION BIAS AND DRIFT DATA

Source identification: _____ Run number: _____
Test personnel: _____ Span: _____
Date: _____

	Analyzer calibration response	Initial values: System calibration response	Initial values: System cal bias (percent of span)	Final values: System calibration response	Final values: System cal bias (percent of span)	Drift (percent of span)
Zero gas						
Upscale gas						

$$\text{System Calibration Bias} = \frac{\text{System Cal. Response} - \text{Analyzer Cal. Response}}{\text{Span}} \times 100$$

$$\text{Drift} = \frac{\text{Final System Cal. Response} - \text{Initial System Cal. Response}}{\text{Span}} \times 100$$

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO_2) per dry standard cubic meter, without having to dilute the sample.

2. Apparatus

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a

EPA METHOD 7E

values versus concentration in $\mu g NO_x$/ml. Do not force the curve through zero. Draw a smooth curve through the points. The curve should be linear. With the linear curve, use linear regression to determine the calibration equation.

8. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

8.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Same as in Method 7C, Section 8.1.

8.2 Total μg NO_x Per Sample.

$$m = (S - B) \times 250 \times \frac{1000}{50} \times \frac{46.01}{63.01} = 3710\,(S - B) \qquad \text{Eq. 7D-1}$$

Where:

m = Mass of NO_x, as NO_2, in sample, μg.
S = Analysis of sample, μg NO_x/ml.
B = Analysis of blank, μg NO_x/ml.
250 = Volume of prepared sample, ml.
46.01 = Molecular weight of NO_2.
63.01 = Molecular weight of NO_x.
1000 = Total volume of $KMnO_4$ solution, ml.
50 = Aliquot $KMnO_4$/NaOH solution, ml.

8.3 Sample Concentration.

$$C = K_1 \frac{m}{V_{m(std)}}$$

Where:

C = Concentration of NO_x, as NO_2, dry basis, mg/dscm.
$K_1 = 10^{-3}$ mg/μg.
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

8.4 Conversion Factors.

1.0 ppm NO = 1.341 mg NO/m³ at STP.
1.0 ppm NO_2 = 1.913 mg NO_2/m³ at STP.
1 ft³ = 2.833×10^{-2} m³.
1000 mg = 1 g.

7. Quality Control

Quality control procedures are specified in Sections 4.1.3 (flow rate accuracy) and 4.6 (audit analysis accuracy) of Method 7C.

8. Bibliography

1. Margeson, J.H., W.J. Mitchell, J.C. Suggs, and M.R. Midgett. Integrated Sampling and Analysis Methods for Determining NO_x Emissions at Electric Utility Plants. U.S. Environmental Protection Agency, Research Triangle Park, NC. Journal of the Air Pollution Control Association. 32:1216-1218. 1982.

2. Memorandum and attachment from J.R. Margeson, Source Branch, Quality Assurance Division, Environmental Monitoring Systems Laboratory, to The Record, EPA. March 30, 1983. NH$_3$ Interference in Methods 7C and 7D.

3. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III—Stationary Source Specific Methods. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-600/4-77-027b. August 1977.

4. Margeson, J.H., et al. An Integrated Method for Determining NO_x Emissions at Nitric Acid Plants. Manuscript submitted to Analytical Chemistry. April 1984.

Method 7E—Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure)

1. Applicability and Principle

1.1 Applicability. This method is applicable to the determination of nitrogen oxides (NO_x) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental chemiluminescent analyzer for determination of NO_x concentration. Performance specifications and test procedures are provided to ensure reliable data.

2. Range and Sensitivity

Same as Method 6C, Sections 2.1 and 2.2.

3. Definitions

3.1 Measurement System. The total equipment required for the determination of NO_x concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface, Gas Analyzer, and Data Recorder. Same as Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.1.2 NO_2 to NO Converter. A device that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO).

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, and Response Time. Same as Method 6C, Sections 3.3 through 3.8.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. Measurement System Performance Specifications

Same as Method 6C, Sections 4.1 through 4.4.

5. Apparatus and Reagents

5.1 Measurement System. Any measurement system for NO, that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe, Sample Line, Calibration Valve Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.1 through 5.1.9, and 5.1.11.

5.1.2 NO_2 to NO Converter. That portion of the system that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO). An NO_2 to NO converter is not necessary if data are presented to demonstrate that the NO_2 portion of the exhaust gas is less than 5 percent of the total NO_x concentration.

5.1.3 NO_x Analyzer. An analyzer based on the principles of chemiluminescence, to determine continuously the NO_x concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 6. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

5.2 NO_x Calibration Gases. The calibration gases for the NO_x analyzer shall be NO in N_2. Three calibration gases, as specified in Sections 5.2.1 through 5.2.3. of Method 6C, shall be used. Ambient air may be used for the zero gas.

6. Measurement System Performance Test Procedures

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 7, and change all 5 percent performance values to 10 percent (or ±0 ppm, whichever is greater).

6.2 Interference Response. Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response (e.g., changes in the gas detector). Conduct that interference response in accordance with Section 5.4 of Method 20.

6.3 Measurement System Preparation, Analyzer Calibration Error, and Sampling System Bias Check. Follow Sections 6.2 through 6.4 of Method 6C.

6.4 NO_2 to NO Conversion Efficiency. Unless data are presented to demonstrate that the NO_2 concentration within the sample stream is not greater than 5 percent of the NO_x concentration, conduct an NO_2 to NO conversion efficiency test in accordance with Section 5.5 of Method 20.

7. Emission Test Procedure

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 7.

7.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the system calibration drift test. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as the total time required to perform a run using Method 7, plus twice the system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed, to determine the average effluent concentration.

7.3 Zero and Calibration Drift Test. Follow Section 7.4 of Method 6C.

8. Emission Calculation

Follow Section 8 of Method 6C.

9. Bibliography

Same as bibliography of Method 6C.

Method 8—Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions From Stationary Sources

1. Principle and Applicability

1.1 Principle. A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03×10^{-6} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74×10^{-7} lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³)

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high-level calibration gas at the calibration valve assembly. Adjust the analyzer output to the appropriate levels, if necessary. Calculate the predicted response for the low-level and mid-level gases based on a linear response line between the zero and high-level responses. Then introduce low-level and mid-level calibration gases successively to the measurement system. Record the analyzer responses for low-level and mid-level calibration gases and determine the differences between the measurement system responses and the predicted responses. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and must be replaced or repaired prior to testing. No adjustments to the measurement system shall be conducted after the calibration and before the drift check (Section 7.2). If adjustments are necessary before the completion of the test series, perform the drift checks prior to the required adjustments and repeat the calibration following the adjustments. If multiple electronic ranges are to be used, each additional range must be checked with a mid-level calibration gas to verify the multiplication factor.

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

7. *Emission Measurement Test Procedure*

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

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

8. *Organic Concentration Calculations*

Determine the average organic concentration in terms of ppmv as propane or other calibration gas. The average shall be determined by the integration of the output recording over the period specified in the applicable regulation.

If results are required in terms of ppmv as carbon, adjust measured concentrations using Equation 25A-1.

$$C_c = K\,C_{meas} \qquad \text{Eq. 25A-1}$$

Where:

C_c = Organic concentration as carbon, ppmv.

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

K = Carbon equivalent correction factor.

K = 2 for ethane.

K = 3 for propane.

K = 4 for butane.

K = Appropriate response factor for other organic calibration gases.

9. *Bibliography*

9.1 Measurement of Volatile Organic Compounds—Guideline Series. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA-450/2-78-041, June 1978, p. 49-54.

9.2 Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol No. 1). U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Research Triangle Park, NC. June 1978.

9.3 Gasoline Vapor Emission Laboratory Evaluation—Part 2. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Research Triangle Park, NC. EMB Report No. 75-GAS-8. August 1978.

Method 25B—Determination of Total Gaseous Organic Concentration Using a Nondispersive Infrared Analyzer

1. *Applicability and Principle*

1.1 Applicability. This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes. (Other organic materials may be measured using the general procedure in this method, the appropriate calibration gas, and an analyzer set to the appropriate absorption band.) The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

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

2. *Definitions*

The terms and definitions are the same as for Method 25A.

3. *Apparatus*

The apparatus is the same as for Method 25A with the exception of the following:

3.1 Organic Concentration Analyzer. A nondispersive infrared analyzer designed to measure alkane organics and capable of meeting or exceeding the specifications in this method.

4. *Calibration Gases*

The calibration gases are the same as required for Method 25A, Section 4. No fuel gas is required for an NDIR.

5. *Measurement System Performance Specifications*

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

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

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

6. *Pretest Preparations*

6.1 Selection of Sampling Site. Same as in Method 25A, Section 6.1.

6.2 Location of Sample Probe. Same as in Method 25A, Section 6.2.

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

6.4 Calibration Error Test. Same as in Method 25A, Section 6.4.

6.5 Response Time Test Procedure. Same as in Method 25A, Section 6.5.

7. *Emission Measurement Test Procedure*

Proceed with [illegible] emission measurement immediately upon satisfactory completion of the calibration.

7.1 Organic Measurement. Same as in Method 25A, Section 7.1.

7.2 Drift Determination. Same as in Method 25A, Section 7.2.

8. *Organic Concentration Calculations*

The calculations are the same as in Method 25A, Section 8.

9. *Bibliography*

The bibliography is the same as in Method 25A, Section 9.

Method 27—Determination of Vapor Tightness of Gasoline Delivery Tank Using Pressure-Vacuum Test

1. *Applicability and Principle*

1.1 Applicability. This method is applicable for the determination of vapor tightness of a gasoline delivery tank which is equipped with vapor collection equipment.

1.2 Principle. Pressure and vacuum are applied alternately to the compartments of a gasoline delivery tank and the change in pressure or vacuum is recorded after a specified period of time.

2. *Definitions and Nomenclature*

2.1 Gasoline. Any petroleum distillate or petroleum distillate/alcohol blend having a Reid vapor pressure of 27.6 kilopascals or greater which is used as a fuel for internal combustion engines.

2.2 Delivery Tank. Any container, including associated pipes and fittings, that is attached to or forms a part of any truck, trailer, or railcar used for the transport of gasoline.

2.3 Compartment. A liquid-tight division of a delivery tank.

2.4 Delivery Tank Vapor Collection Equipment. Any piping, hoses, and devices on the delivery tank used to collect and route gasoline vapors either from the tank to a bulk terminal vapor control system or from a bulk plant or service station into the tank.

2.5 Time Period of the Pressure or Vacuum Test (t). The time period of the test, as specified in the appropriate regulation, during which the change in pressure or vacuum is monitored, in minutes.

2.6 Initial Pressure (P_i). The pressure applied to the delivery tank at the beginning of the static pressure test, as specified in the appropriate regulation, in mm H_2O.

2.7 Initial Vacuum (P_i). The vacuum applied to the delivery tank at the beginning of the static vacuum test, as specified in the appropriate regulation, in mm H_2O.

2.8 Allowable Pressure Change (Δp). The allowable amount of decrease in pressure during the static pressure test, within the time period t, as specified in the appropriate regulation, in mm H_2O.

2.9 Allowable Vacuum Change (Δv). The allowable amount of decrease in vacuum during the static vacuum test, within the time period t, as specified in the appropriate regulation, in mm H_2O.

3. *Apparatus*

3.1 Pressure Source. Pump or compressed gas cylinder of air or inert gas sufficient to pressurize the delivery tank to 500 mm H_2O above atmospheric pressure.

3.2 Regulator. Low pressure regulator for controlling pressurization of the delivery tank.

3.3 Vacuum Source. Vacuum pump capable of evacuating the delivery tank to 250 mm H_2O below atmospheric pressure.

3.4 Pressure-Vacuum Supply Hose.

3.5 Manometer. Liquid manometer, or equivalent instrument, capable of measuring up to 600 mm H_2O gauge pressure with ±2.5 mm H_2O precision.

3.6 Pressure-Vacuum Relief Valves. The test apparatus shall be equipped with an inline pressure-vacuum relief valve set to actu-

Method 25A—Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer

1. Applicability and Principle

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

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

2. Definitions

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

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

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

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

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

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

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

2.6 Response Time. The time interval from a step change in pollutant concentration at the inlet to the emission measurement system to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.

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

3. Apparatus

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

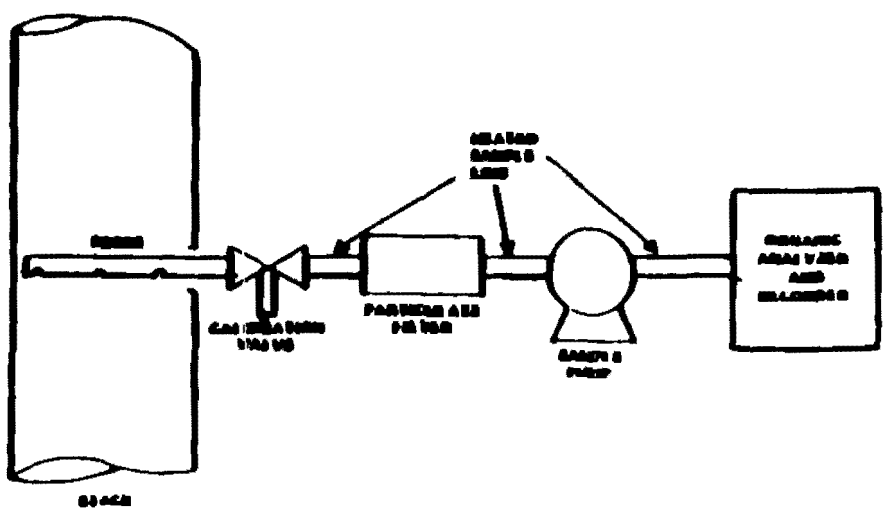

Figure 25A-1 Organic Concentration Measurement System

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

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

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

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

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

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

4. Calibration and Other Gases

Gases used for calibrations, fuel, and combustion air (if required) are contained in compressed gas cylinders. Preparation of calibration gases shall be done according to the procedure in Protocol No. 1, listed in Reference 9.2. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval of the Administrator.

Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value. Organic compounds other than propane can be used following the above guidelines and making the appropriate corrections for response factor.

4.1 Fuel. A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

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

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

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

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

5. Measurement System Performance Specifications

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

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

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

6. Pretest Preparations

6.1 Selection of Sampling Site. The location of the sampling site is generally specified by the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. The sample port shall be located at least 1.6 meters or 2 equivalent diameters upstream of the gas discharge to the atmosphere.

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

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

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

6.4 Calibration Error Test. Immediately prior to the test series, (within 2 hours of the start of the test) introduce zero gas and

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