Note: This is a reference cited in *AP 42, Compilation of Air Pollutant Emission Factors, Volume I Stationary Point and Area Sources.* AP42 is located on the EPA web site at www.epa.gov/ttn/chief/ap42/

The file name refers to the reference number, the AP42 chapter and section. The file name "ref02_c01s02.pdf" would mean the reference is from AP42 chapter 1 section 2. The reference may be from a previous version of the section and no longer cited. The primary source should always be checked.

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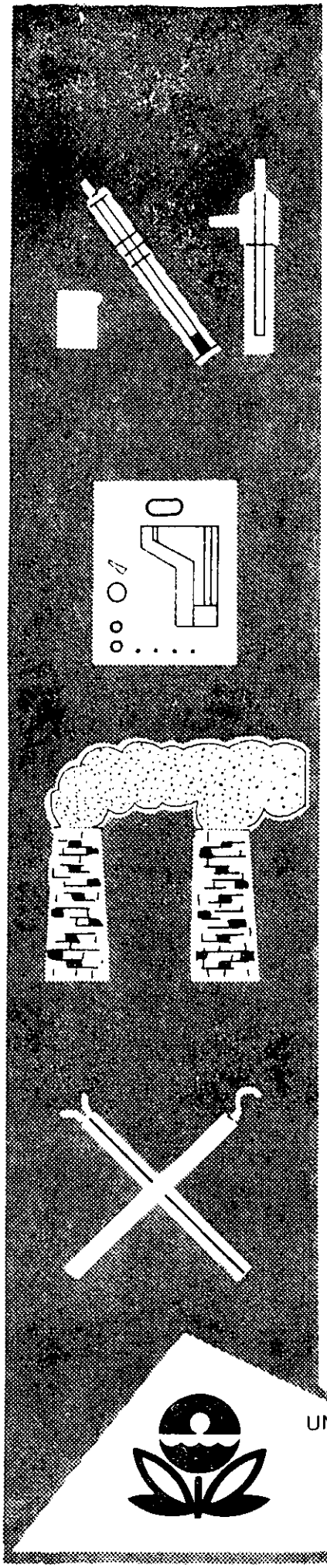
Background Chapter 4

Reference: 6

Title: Air Pollution Emission Test, Source Testing Methodology for the Asphalt Roofing Industry, Celotex Corporation, Cincinnati, Ohio,

EMB Report No. 75-ARM-10, U. S. Environmental Protection Agency, Research Triangle Park, NC,

May 1977.



Project Report No. 75-ARM-10

AIR POLLUTION EMISSION TEST

SOURCE TESTING METHODOLOGY
FOR THE
ASPHALT ROOFING INDUSTRY

CELOTEX CORPORATION
CINCINNATI, OHIO

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air and Waste Management
Office of Air Quality Planning and Standards
Emission Measurement Branch
Research Triangle Park, North Carolina

A MacQueen
SOTDAT?

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

SUBJECT: Source Test Report

DATE: MAR 8 1977

FROM: Roger T. Shigehara, Chief, Test Support Section RS
Emission Measurement Branch, ESED (MD-19)

TO: See Below

The enclosed final source test report is submitted for your information. Any questions regarding the test should be directed to the Project Officer (8/629-5276). Additional copies of this report are available from the Library, Research Triangle Park, North Carolina 27711.

Industry: Source Testing Methodology for the Asphalt Roofing Industry

Process: Saturator Lines and Blowing Still

Company: Celotex

Location: Cincinnati, Ohio

Project Report Number: 75 ARM 10

Project Officer: Tom Logan

Enclosure

Addressees:

John Nader (MD-59)
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Director, Air and Hazardous Materials Division, Region V
(copy enclosed for State Agency)
APTIC (MD-18)

FINAL REPORT

on

SOURCE TESTING METHODOLOGY FOR THE ASPHALT ROOFING INDUSTRY

to

ENVIRONMENTAL PROTECTION AGENCY

January 10, 1977

Contract No. 68-02-1409
Task No. 23

by

R. E. Barrett, P. R. Webb, W. C. Baytos,
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FINAL REPORT

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SOURCE TESTING METHODOLOGY FOR THE ASPHALT ROOFING INDUSTRY

to

ENVIRONMENTAL PROTECTION AGENCY
(Contract No. 68-02-1409, Task 23)

from

BATTELLE
Columbus Laboratories

January 10, 1977

INTRODUCTION

In early 1975 the U. S. Environmental Protection Agency initiated a program that would lead to development of New Source Performance Standards for asphalt roofing plants. The prime interest was to issue emissions regulations to cover emissions of particulate and gaseous hydrocarbons from modified asphalt roofing plants.

It was recognized that the majority of the emissions (on a weight basis) from the asphalt roofing plants would be hydrocarbon compounds. Because many hydrocarbon compounds are volatile at moderately low temperatures, it was determined that the standard Method 5 sampling train might not be appropriate for these measurements. The solution was to sample the heavier hydrocarbon compounds (solids and condensibles) as particulates using some modification of the Method 5 sampling train, and to sample gaseous hydrocarbons downstream of the Method 5 filter using a continuous hydrocarbon analyzer.

To permit a trial of proposed sampling methodology for asphalt roofing plants, EPA engaged Battelle-Columbus to conduct preliminary sampling at the Celotex Corporation plant in Cincinnati, Ohio.

The initial scope of this preliminary study and sampling activity included:

(1) Examining the suitability of 1,1,1-trichloroethane (TCE) as a solvent for washing the particulate train.

(2) Examining the nature of the particulate catch samples including determination of their susceptibility to evaporative loss.

(3) Examining the suitability of the flame ionization-detector (FID) hydrocarbon analyses for measuring gaseous hydrocarbon emissions, including estimating the FID response to the hydrocarbon compounds found in the sample gas stream.

(4) Comparing FID analyses with analyses of flask samples.

The scope was subsequently enlarged to include:

(5) Evaluating the possible loss of a portion of the particulate samples during the sample-drying procedure.

(6) Evaluating POM data obtained with a 175 F adsorbent column (instead of the 130 F normally used).

This report describes three activities related to the New Source Performance Standards (NSPS) emission measurements at asphalt plants. These activities are:

I. Sampling at the Celotex plant to establish procedures for the NSPS sampling at other asphalt plants.

II. Special drying studies of selected particulate samples from asphalt plants to establish procedures for drying the bulk of the samples.

III. A special POM run conducted at the Certain-Teed asphalt plant (Shakopee, Minnesota) to "calibrate" the 175 F POM runs made at the Elk Roofing plant (Stephens, Arkansas).

Each of these activities is described in a separate section of this report. The three sections are identified as Part I, II, and III, respectively.

PART I

EMISSION SAMPLING AT CELOTEX PLANT CINCINNATI, OHIO

As part of an overall effort to develop procedures for sampling emissions from asphalt roofing plants, experimental tests were conducted at the Celotex Plant in Cincinnati, Ohio. The purpose of the sampling effort was to test and evalute the proposed sampling methodology by sampling at one asphalt roofing plant prior to more extensive sampling efforts at other plants.

Paul R. Webb, Salo Miller, and Karl Turner of Battelle-Columbus Laboratories conducted the field test on June 12 and 13, 1975, under the direction of Tom Logan of EMB, ESED, OAQPS, EPA.

PROCESS DESCRIPTION

The felt used in the manufacture of common asphalt-saturated products is made from waste paper with either wood fibers or rags added for strength against tearing.

Organic felt base roofing shingles are manufactured by continuously passing a web of paper felt through a "line" or series of machines which include the following sequential operations: a felt unwind stand, dry looper, asphalt saturator, drying-in drums, wet looper, coater, granule applicator, backing applicator, press section, water-cooled rolls, finish floating looper, shingle cutter, and shingle stacker.

The temperature of the asphalt in the saturator dip section varies from 232 C (450 F) at the start to 218 C (425 F) at the finish. The temperature is maintained by continuously circulating the asphalt through an insulated, heated storage tank.

The Celotex plant obtains asphalt locally, the Gulf Oil Company being the major supplier. The asphalts are manufactured from the bottom products of crude petroleum distillation. The bottoms are air-blown and/or blended with purchased lube oil stocks to meet specifications on blown

asphalt and, thus, to enable products to meet roofing materials specifications. The blowing stills at Celotex use air at the rate of 2937 Nm^3/hr (1735 scfm), and the blowing times range from 1 to 4-1/2 hours, depending on the particular products. During normal blowing operations, Celotex collects approximately 200 kg (54 gallons) of oil per day from the cyclone, which is reclaimed to their fuel oil system. Approximately 0.75 percent of the raw material is lost during a blowing operation.

Figures I-1 and I-2 are schematic flow diagrams showing process components, control devices, and sample point locations. The air pollution control equipment at the Celotex plant consists of (1) a cyclone and afterburner which handle the effluent from the four asphalt blowing stills and (2) electrostatic precipitators (ESP), which handle effluents from the saturator enclosure.

The blowing stills have a cyclone collector to collect a portion of the heavy hydrocarbon emissions and an afterburner to burn the remaining hydrocarbons. The ESP's are Smog-Hog Units manufactured by United Air Specialists. The two precipitators (Units No. 1 and 2) are Model MS20AT cells, each containing two banks of cells and a fiber roll prefilter. The precipitators are also equipped with detergent/water spray systems to keep the plates clean.

Because most of the particulate emissions from asphalt roofing plants are hydrocarbons/organics, and are volatile, the temperature at which an emission control device operates may have an appreciable affect on the efficiency of the device. (This generally is true for filters and electrostatic precipitators, but not for afterburners.) Hence in EPA's effort to identify the "best technology" for controlling emissions from asphalt roofing plants, it was determined that control devices should be operated at the lowest practical temperature to insure maximum collection of condensible particulate. Thus, filter-type control devices (such as HEAF units - High Energy Air Filters) should be operated at 38 to 50 C (100 F to 120 F), rather than at some higher temperature. The emission measurement procedure should recognize this sensitivity of emissions to control device temperature and permit measurement of emissions on the basis of using the best control device technology. Hence, it was determined that the sampling procedure should permit collection of condensibles

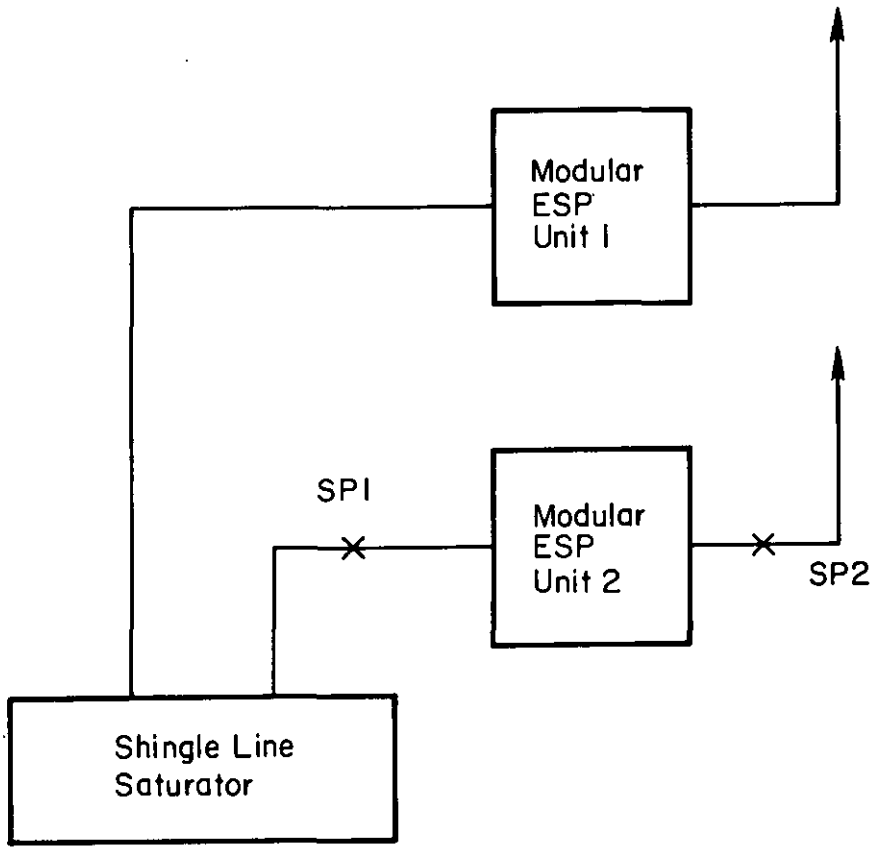


FIGURE I-1. LOCATION OF SAMPLING POINTS RELATIVE TO SATURATOR PROCESS COMPONENTS

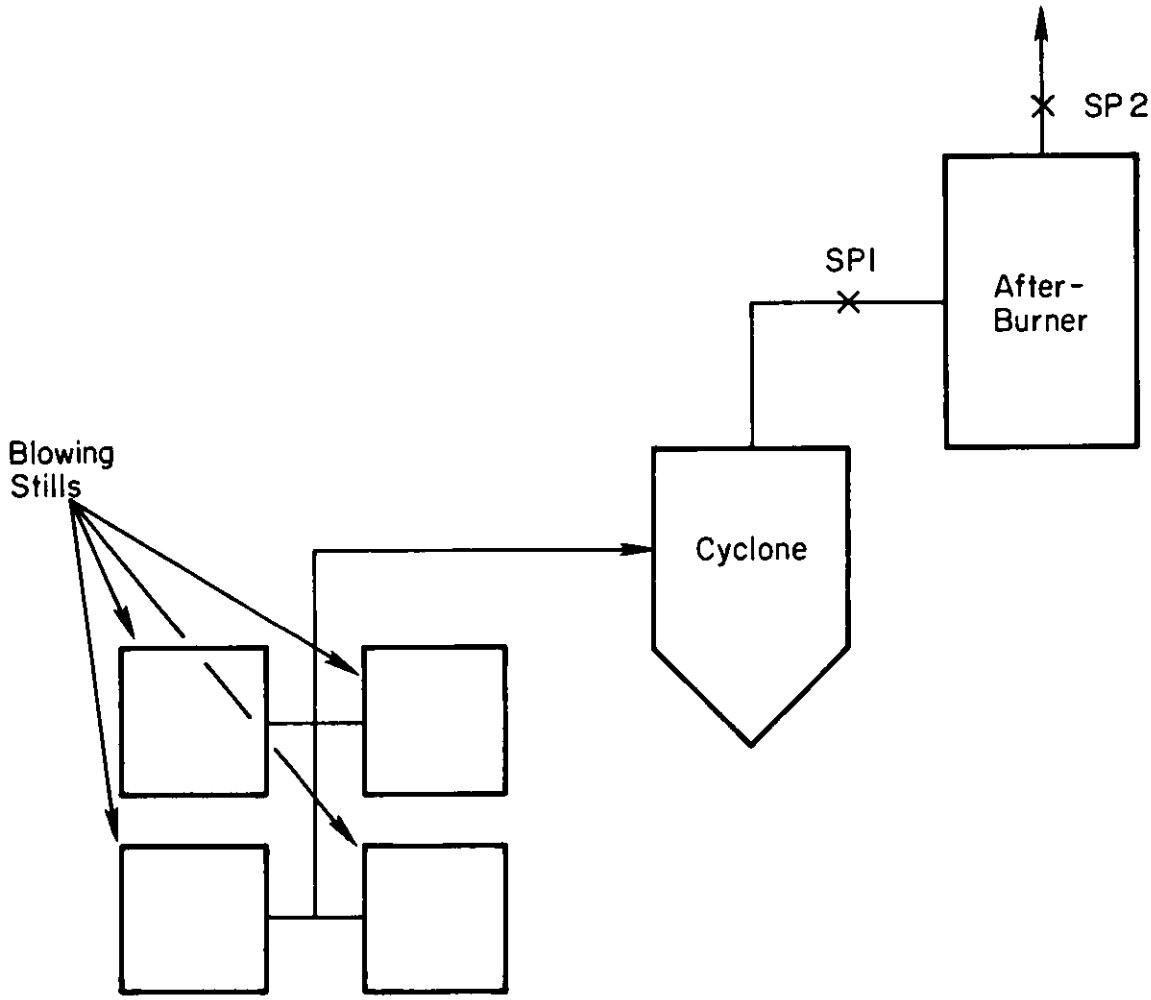


FIGURE I-2. LOCATION OF SAMPLING POINTS RELATIVE TO BLOWING STILL PROCESS COMPONENTS

at about 38 C (100 F), and vital sampling train components (prefilters and filters) were operated at this temperature.

SAMPLING POINT LOCATIONS

Figures I-3 and I-4 are schematic diagrams showing the locations of inlet and outlet sample points relative to process components for the blowing still and saturator operations, respectively. Single point sampling was used in lieu of traversing since the data would be used only as an aid in establishing sampling techniques. One duct diameter at each sampling location (inlet and outlet) was traversed to determine the best probe location to achieve sampling at the average stack velocity and to establish parameters necessary for isokinetic sampling. Sample point location has been noted on Figures I-3 and I-4 for the inlet and outlets respectively.

SAMPLING AND ANALYTICAL PROCEDURE

Standard Method 5 sampling and analytical procedures were judged not suitable due to the nature of the processes and the need to operate the filter at lower temperatures than are generally used for Method 5 sampling. Also, the relatively large amounts of oil and water associated with asphalt roofing plant operations plug the Method 5 filters.

Figure I-5 is a schematic of the sampling trains used for sampling at the Celotex plant. Figure I-5 also identifies cleanup and analysis procedures for each portion of the sampling train. To reduce and/or eliminate the water/oil problem for sampling at blowing stills, the Method 5 sampling train was rearranged so that the sampled gases passed through the cooled impingers upstream of the filter, thereby dropping out much of the water and oil. The impingers were placed in a water bath to maintain them at a temperature of 38 to 50 C (100 to 120 F). To further reduce the possibility of water and oil plugging the filters, a prefilter, fabricated from a glass column packed with fiber glass, was placed in the sampling train between the impingers and the conventional

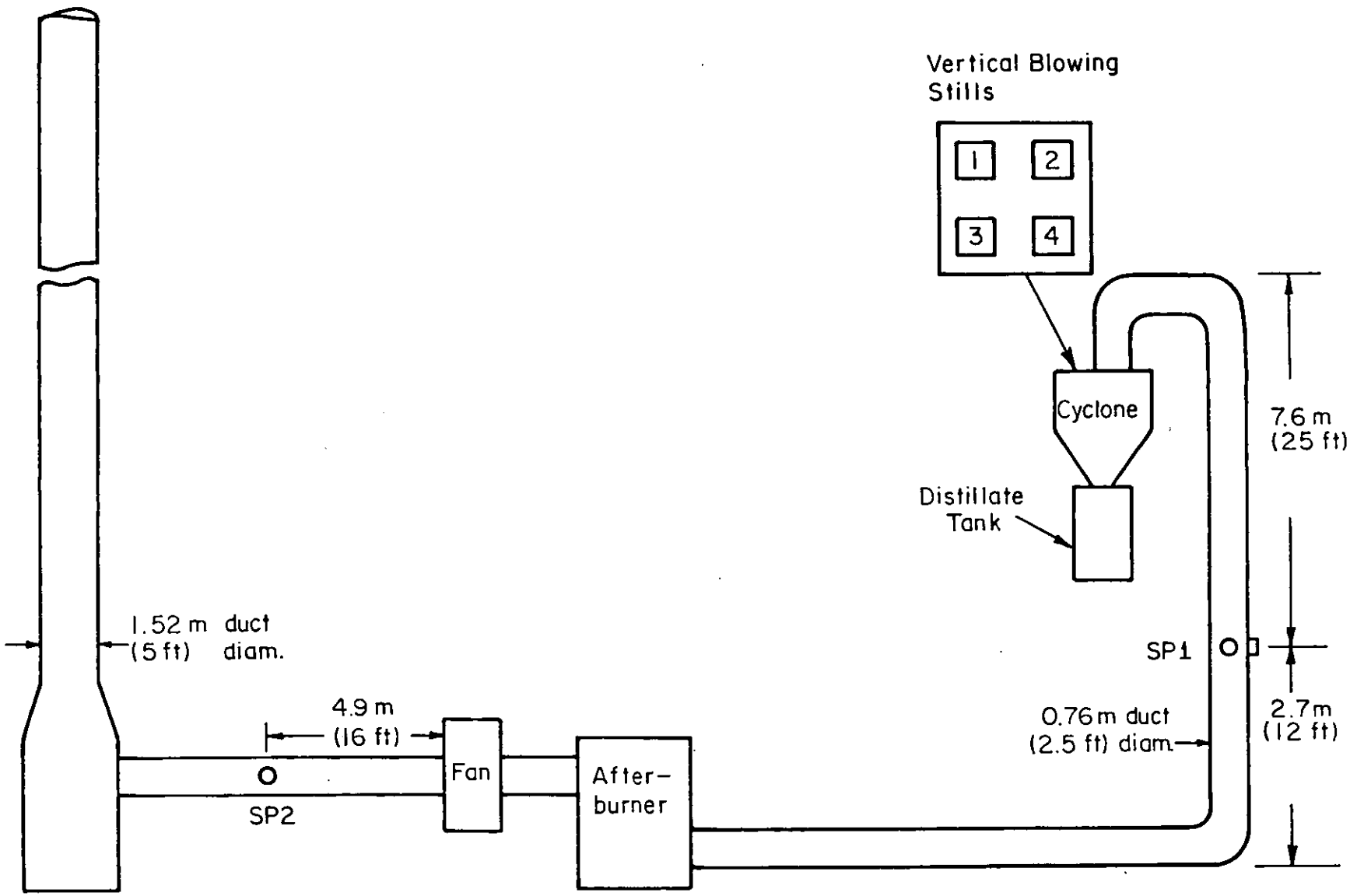


FIGURE I-3. DUCT GEOMETRY AND SAMPLING POINT LOCATIONS FROM BLOWING STILLS

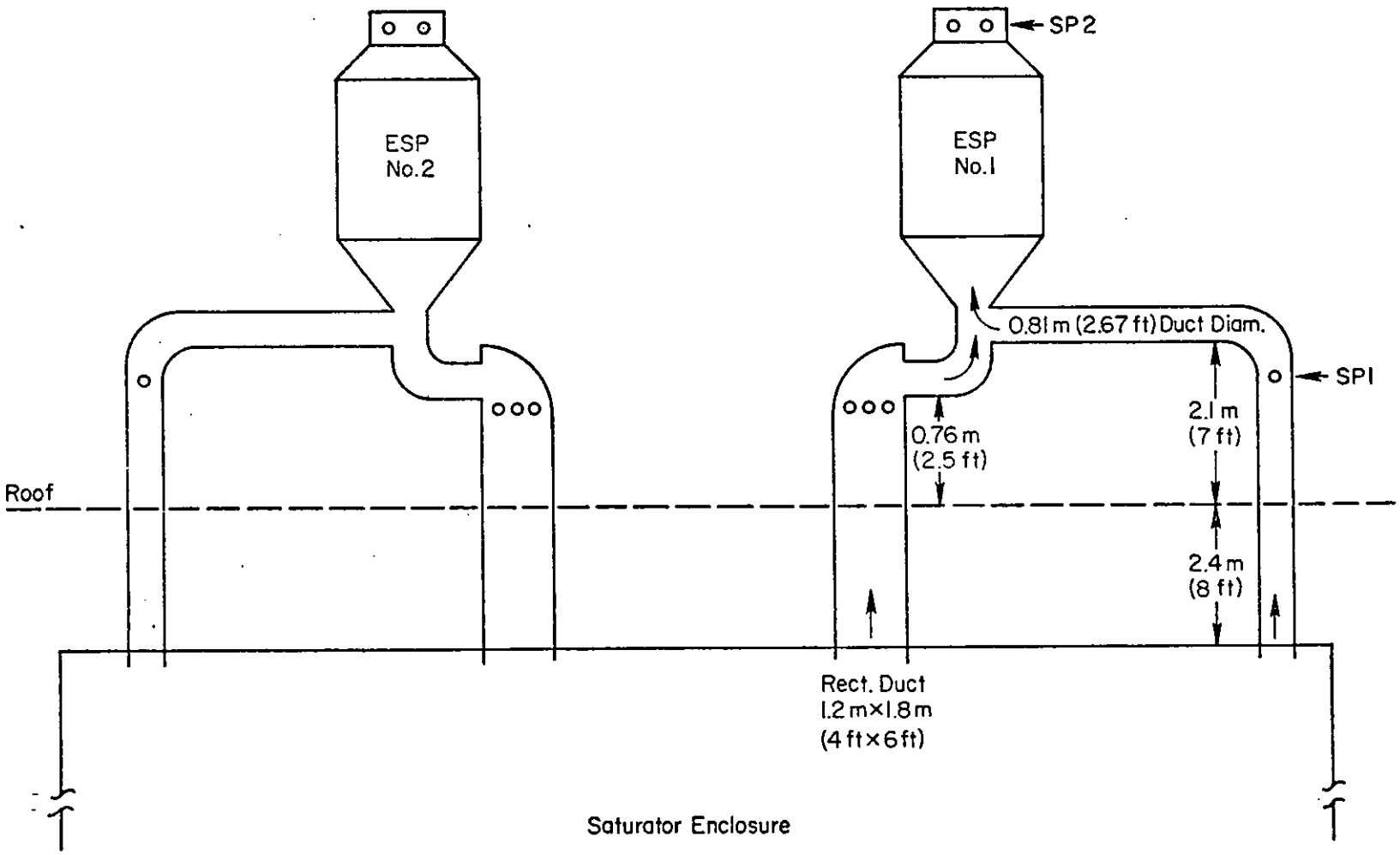
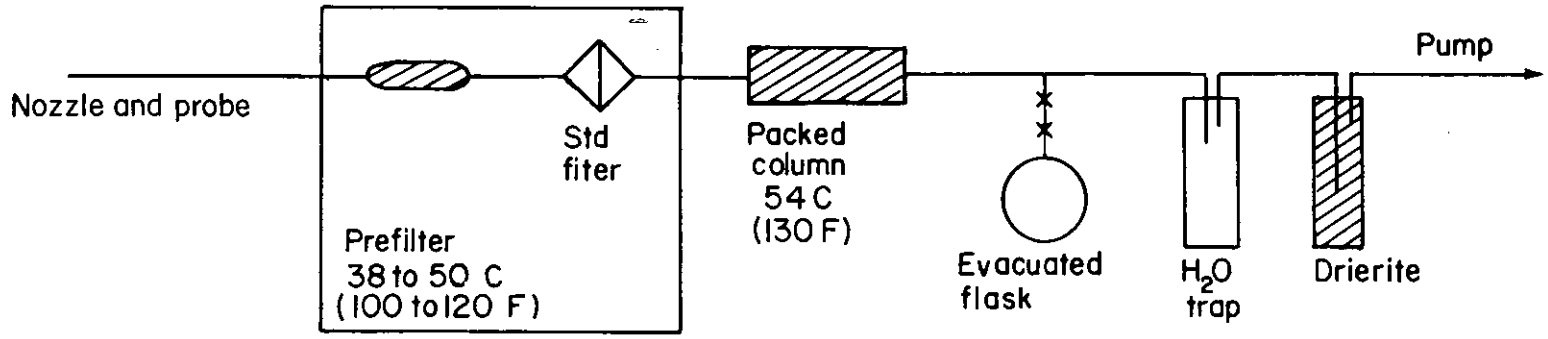


FIGURE I-4. DUCT GEOMETRY AND SAMPLING PORT LOCATIONS FROM SATURATOR LINE

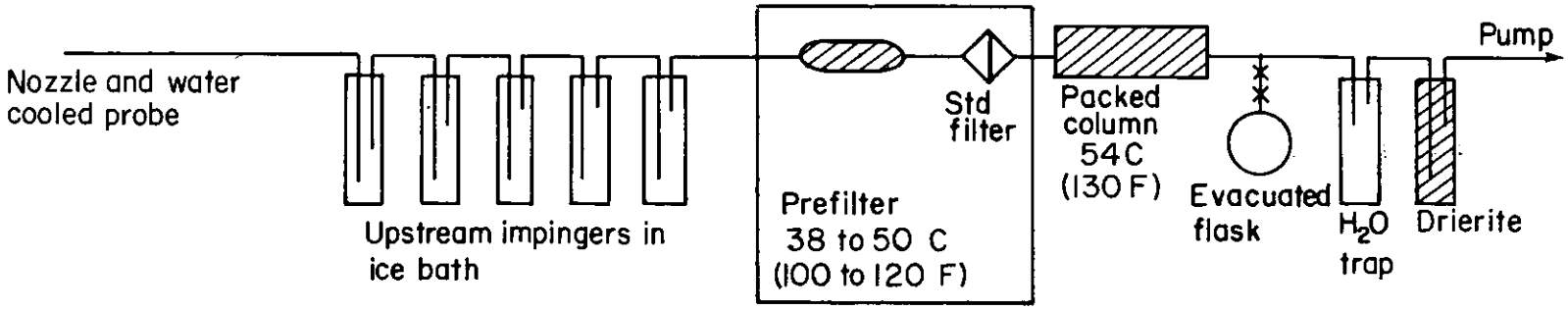
SATURATOR LINES



Cleanup Procedure

Probe of nozzle:	Wash with Trichloroethane (TCE), dry and weigh
Prefilter:	Wash fibreglass with Trichloroethane, dry and weigh
Filter:	Desiccate, weigh
Packed column:	Extract from packed column, separate HC's by liquid chromatography, weigh each fraction, analyze one sample by infrared analysis
Evacuated flask:	HC analysis by gas chromatography -- mass spectrometry
Moisture trap:	Determine accumulated moisture

BLOWING STILL



Cleanup Procedure

Analysis same as above except

Upstream inpinges: Evaporate H_2O, dessicate and weigh

FIGURE I-5. SAMPLING TRAINS AND CLEANUP PROCEDURES USED FOR SAMPLING THE SILOTEX LINES

filter. The large surface area associated with the use of glass wool as a prefilter supplied ample contact for impingement of oil and/or water droplets which may have passed through the impingers. The Method 5 prefilter and filter temperatures were kept at 38 to 50 C (100 to 120 F) by using a water cooled probe (depending on stack gas temperatures), by adjusting the sample probe temperature (if stack gas temperatures were relatively low), and/or by adding small amounts of ice to the impinger water bath. Impingers also were placed in an ice bath downstream of the filter to collect the remaining water for moisture determination and to dry the gas for metering.

Saturator plant samplings were accomplished without the impingers upstream of the filter (because the moisture in the gas sampling is relatively low). However, the prefilter is still recommended (at least for sampling upstream of control devices) due to possible high particulate (oil) loading of the gas stream.

Outlet stack sampling from incinerator control devices generally requires a water cooled probe because of the relatively high stack gas temperature. Also, in addition to the conventional filter to collect particulate emissions (as in Method 5), the prefilter or upstream impingers may be necessary to remove condensed moistrue resulting from the afterburner combustion.

After passing through the filter, the sample gases were passed through a packed column of Tenox* adsorbent material. This material would adsorb certain hydrocarbon vapors that would pass through the filter. A portion of the gaseous hydrocarbons which passed through the 38 to 50 C (100 to 120 F) filter was collected in a 3 litre evacuated flask by means of a glass tee placed in the sample line just after the conventional filter. The packed column and flask samples were analyzed by gas chromotography - mass spectroscopy (GC-MS) techniques to determine the nature of the constituents and to determine if these gaseous constituents could be measured by FID techniques.

Field and laboratory data sheets and calculations related to this sampling effort are reported in Appendix A.

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* Trademark

Analysis Methodology

Method 5 analysis laboratory procedures were used to determine mass values of the catch. For later asphalt roofing plant samples, special drying studies were conducted at Battelle-Columbus Laboratories to establish a methodology for drying possibly volatile samples. These studies are reported in Part II of this report.

For the material adsorbed on the column and collected in the flasks, the samples were analyzed and an evaluation was made to determine the response of an FID instrument to the components of the samples.

SUMMARY OF EMISSION RESULTS

Since the emission sampling at the Celotex Plant in Cincinnati, Ohio, was conducted only as a means to establish sampling methodology, no attempt was made to determine stack gas molecular weight. Table I-1 is a summary of the analytical results for the saturator and blowing still samples obtained during this field test. For the most part, the samples appeared to be comprised of water and oil. Because of single point and probable unrepresentative sampling, mass values and concentrations cannot be considered as valid emission data.

Figures I-6 through I-12 are thermogravimetric-analysis (TGA) runs of the particulate catch from sampling at the Cincinnati asphalt plant. Specifically, the figures are as follows:

Figure I-6	Blowing Still Inlet (Run 1)
Figure I-7	Blowing Still Inlet (Run 1)
Figure I-8	Blowing Still Outlet (Run 1)
Figure I-9	Saturator Line Inlet, Probe Wash
Figure I-10	Saturator Line Inlet, Filter Extract
Figure I-11	Saturator Line Outlet
Figure I-12	Blank or Residue from Evaporating Technical Grade TCE.

Data for Figures I-6, I-7, and I-8 include probe wash, impinger catch, and filter catch. Figure I-11 includes probe and filter catch.

TABLE I-1. WEIGHT DATA FOR PARTICULATE CATCH AT CELOTEX PLANT, CINCINNATI, OHIO
(Weights in mg)

Sample	Saturator Line		Blowing Still			
			Afterburner Inlet		Afterburner Outlet	
	ESP Inlet	ESP Outlet	Run #1	Run #2	Run #1	Run #2
Probe, gross	57.3	61.1	832.7	987.7	125.6	103.3
net [a]	-22.3	-24.6	716.4	761.3	-0.9	-33.4
Impinger, waterwash						
TCE wash	None	None	549.2	Not Done	12.6	4.1
Acetone wash			8687.1		8.2	4.9
			17.8		4.2	1.8
Total			9254.1		25.0	10.8
Glass wood prefitter, gross	145.7	126.6	262.7	70.6	145.8	109.7
net [a]	77.9	61.6	177.3	3.0	75.7	48.5
Glass fiber filter, net wt. gain	32.7	6.5	6.5	0	2.1	2.4
Total	110.6	68.1	10,154.3	-	102.8	61.7
Percent caught on						
Probe	0	0	7	-	0	0
Impingers	--	--	91	-	24	18
Prefilter	70	90	2	-	74	79
Filter	30	10	0	-	2	4

[a] Net = Corrected for TCE blank. (Technical grade TCE was used; it had a large blank value.)

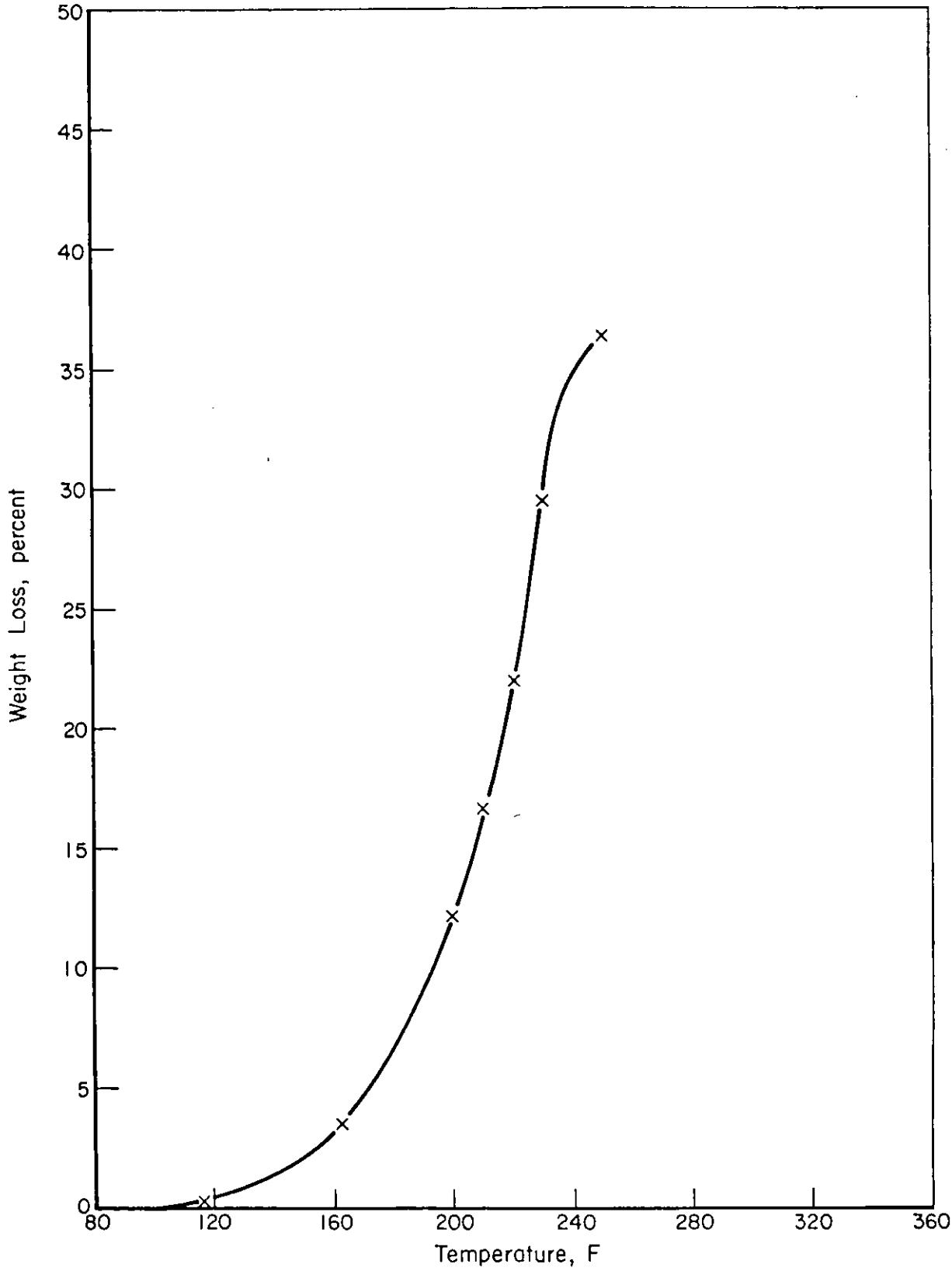


FIGURE I-6. TGA DATA FOR PARTICULATE CATCH FROM BLOWING STILL INLET (Run 1)

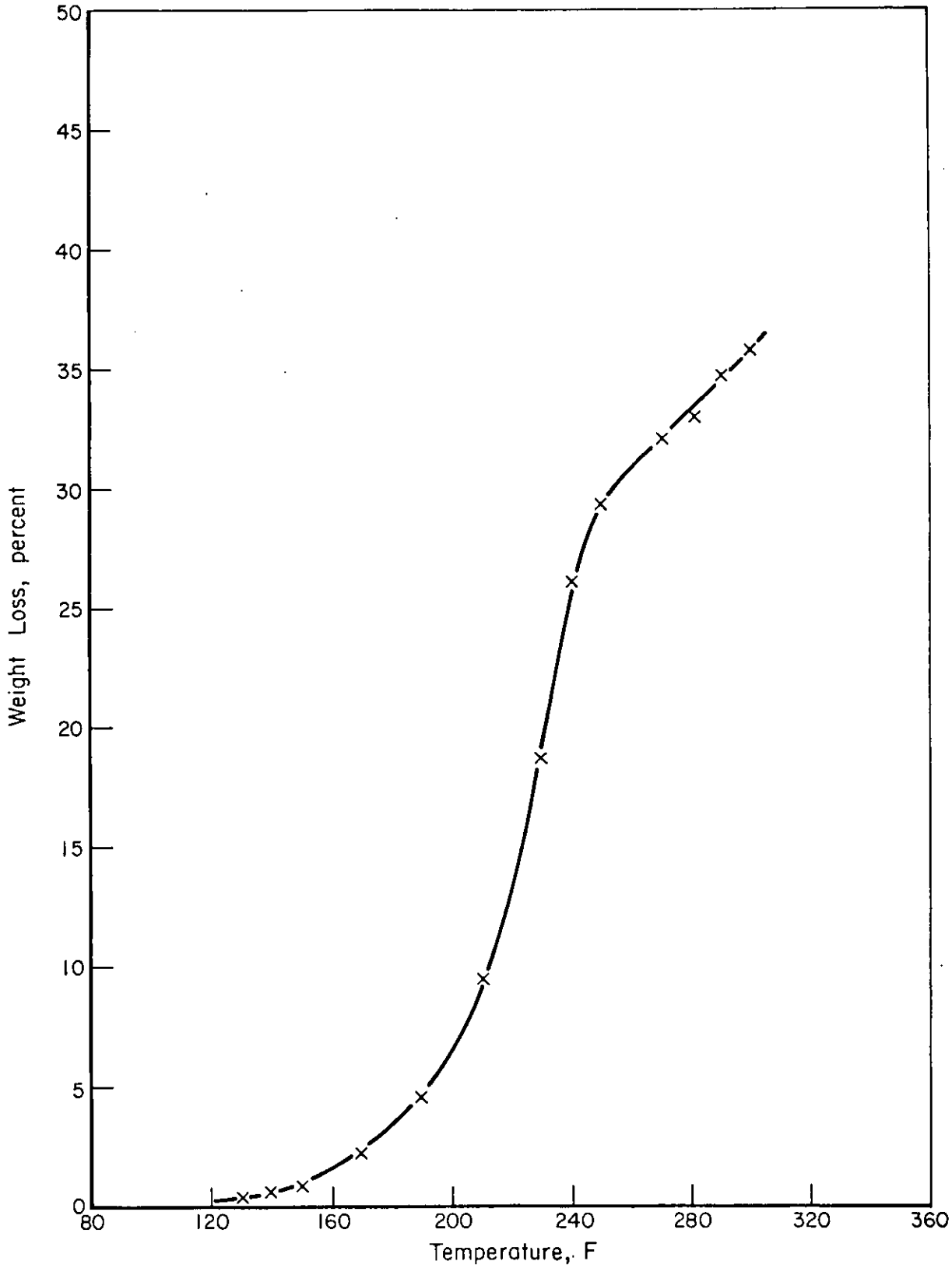


FIGURE I-7. TGA DATA FOR PARTICULATE CATCH FROM BLOWING STILL INLET (Run 1)

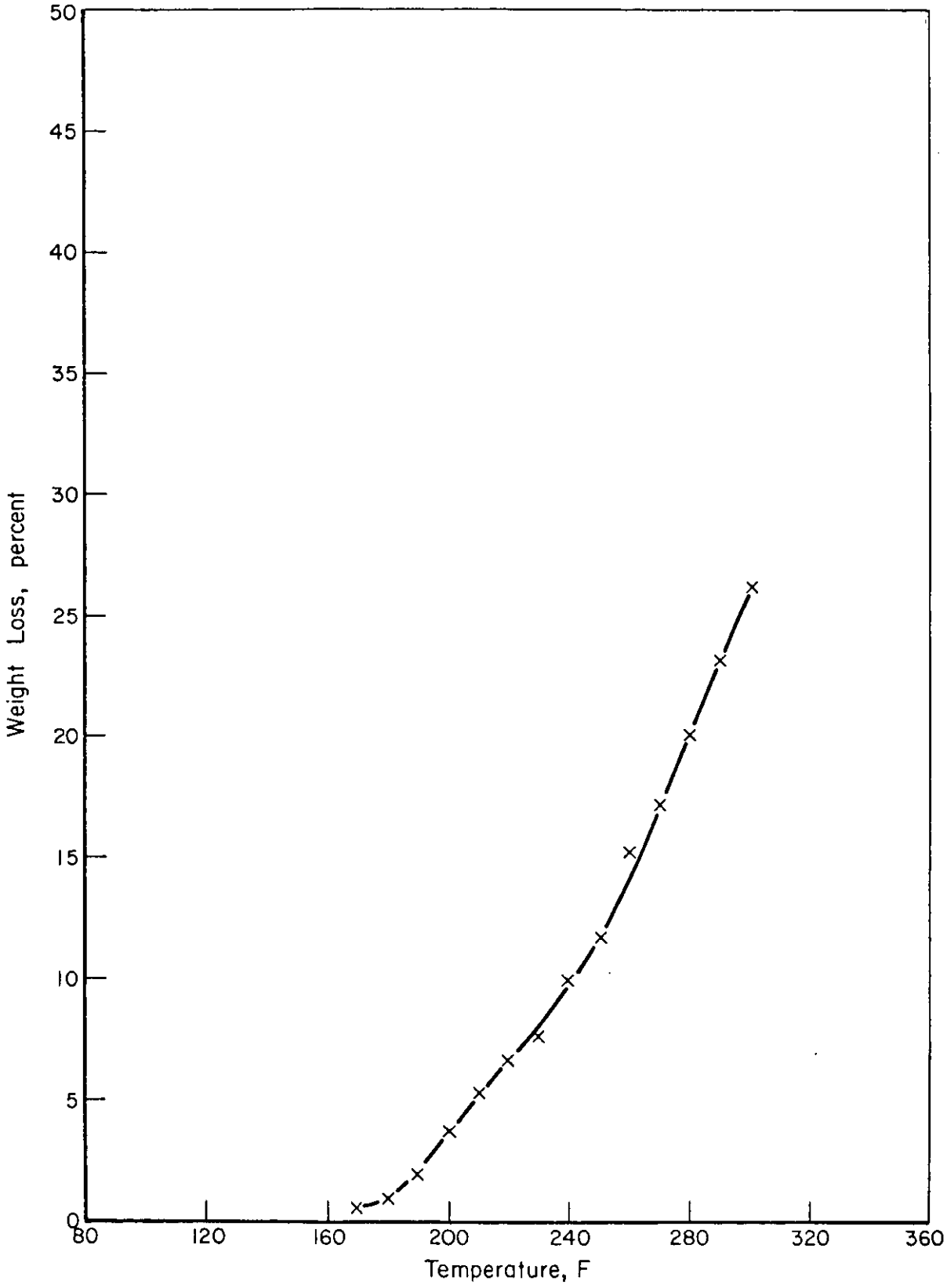


FIGURE I-8. TGA DATA FOR PARTICULATE CATCH FROM BLOWING STILL OUTLET (Run 1)

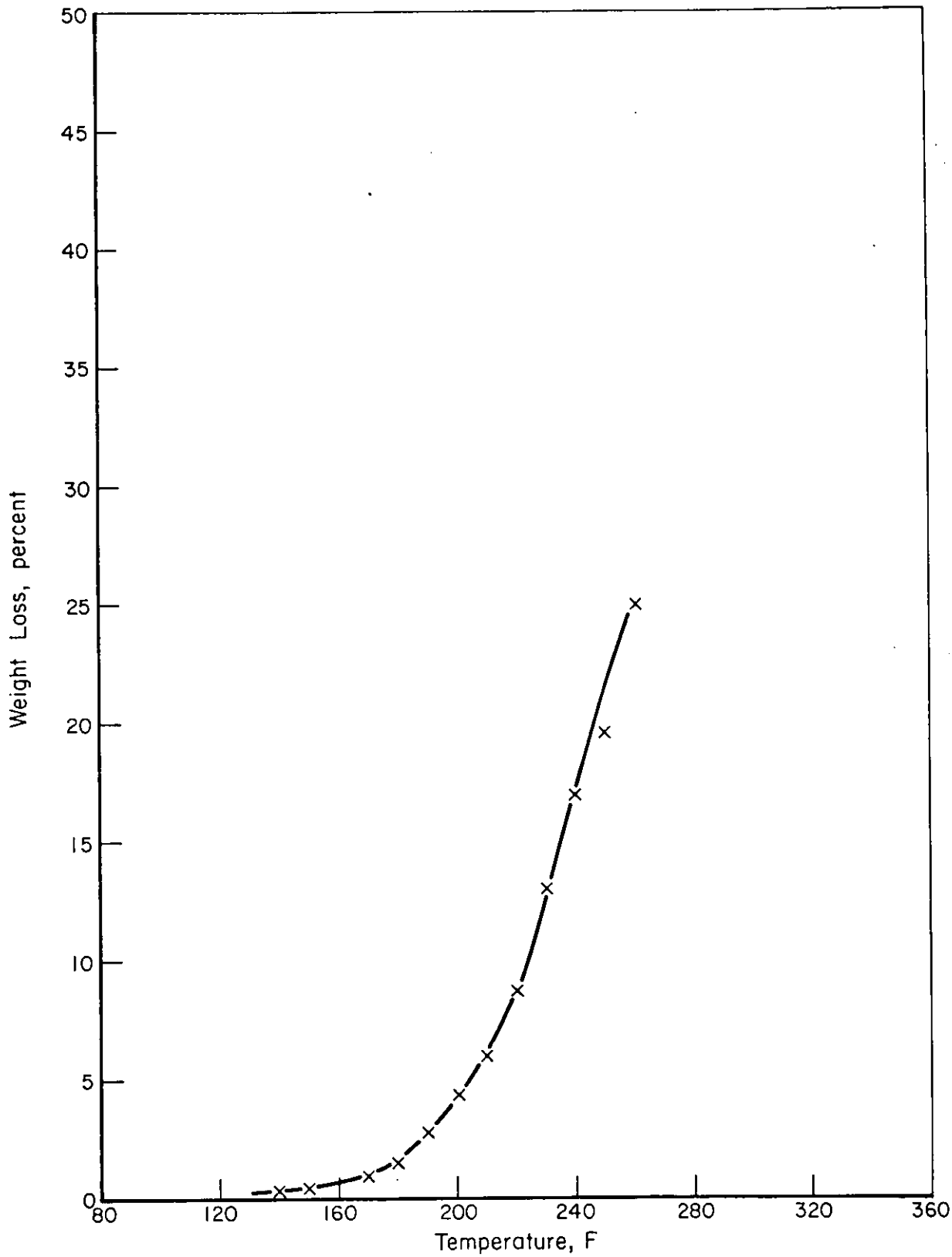


FIGURE I-9. TGA DATA FOR PROBE WASH FROM SATURATOR LINE INLET

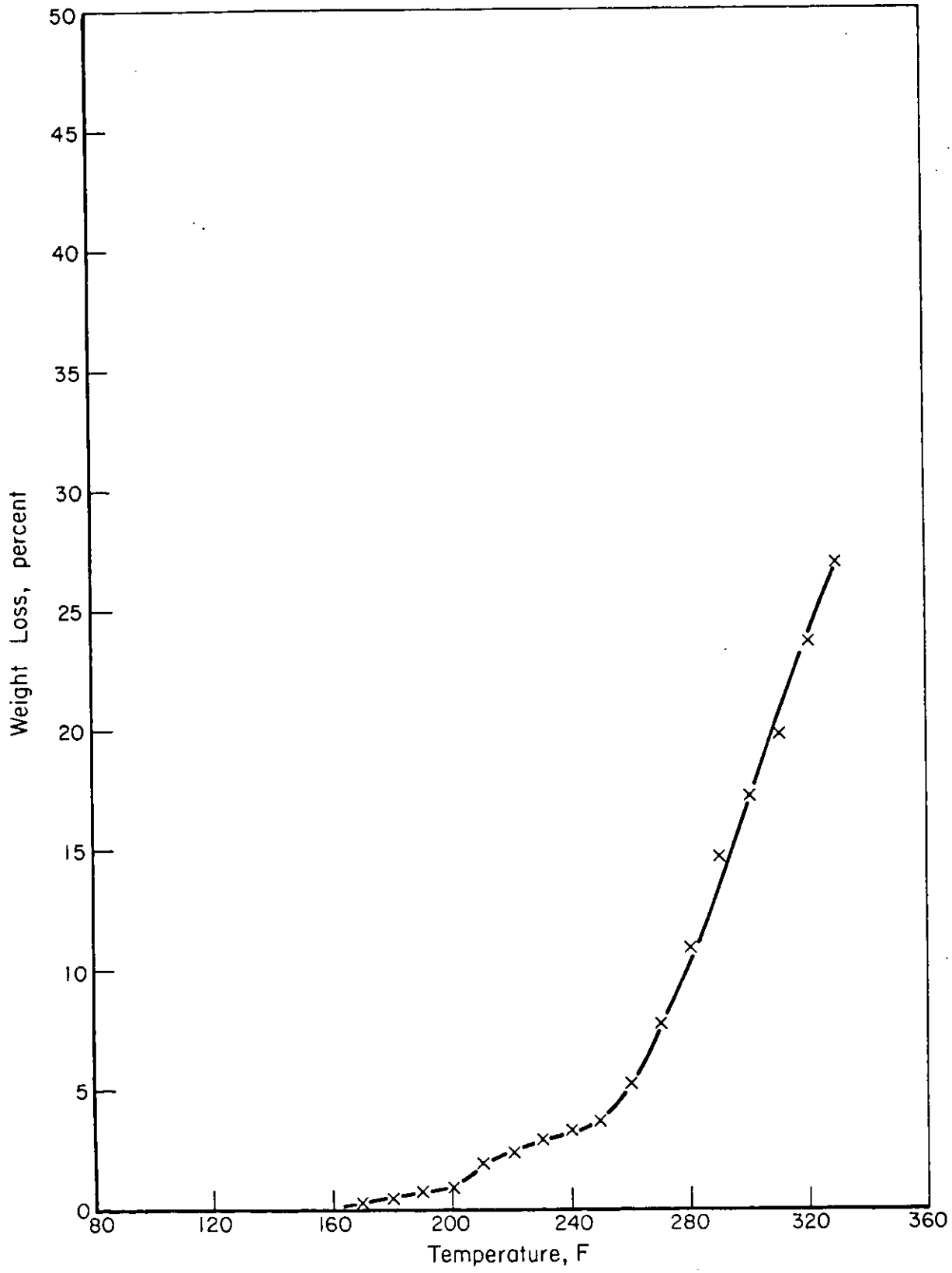


FIGURE I-10. TGA DATA FOR FILTER EXTRACT FROM SATURATOR LINE INLET

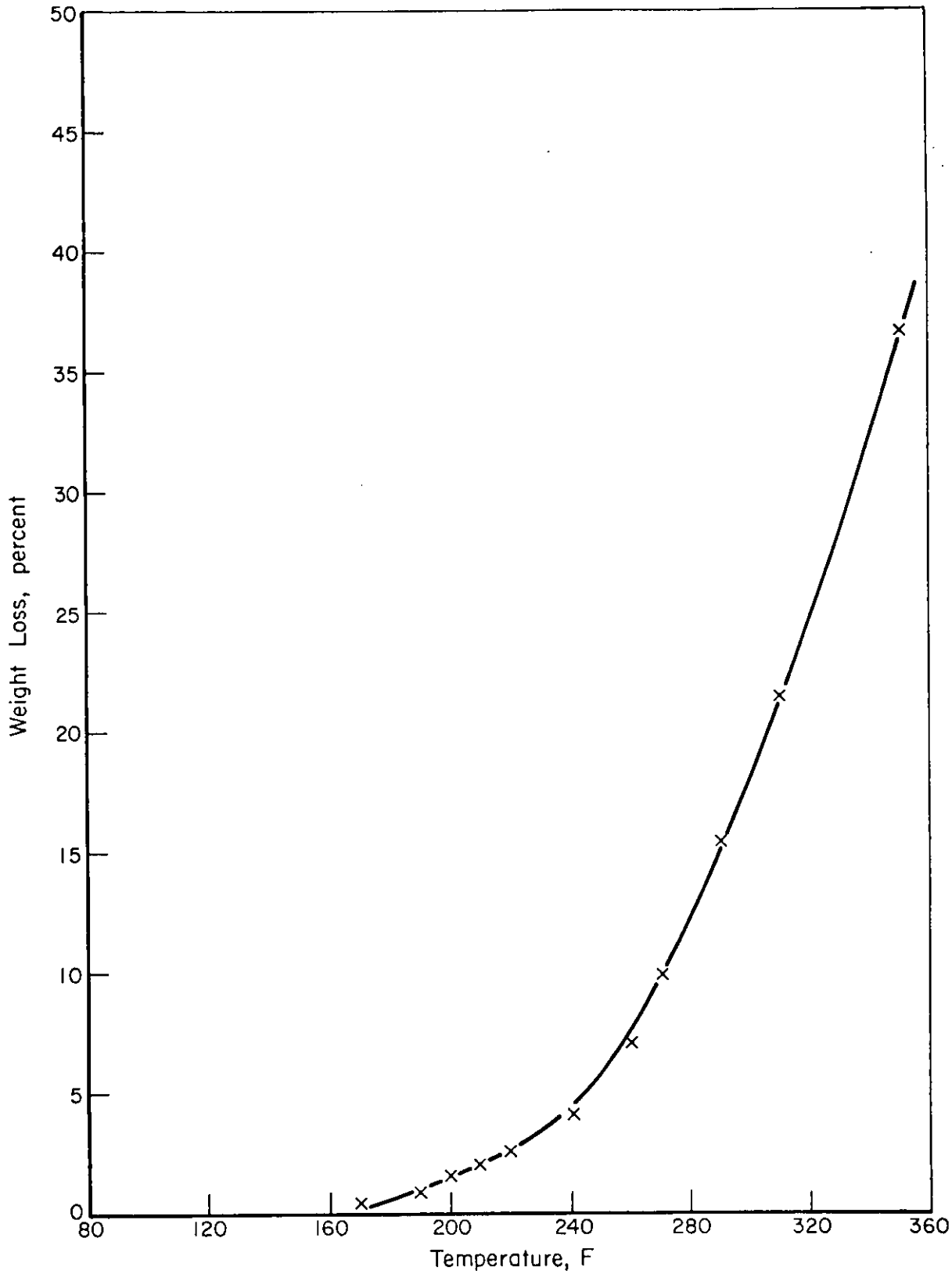


FIGURE I-11. TGA DATA FOR PROBE WASH AND FILTER EXTRACT FROM SATURATOR LINE OUTLET

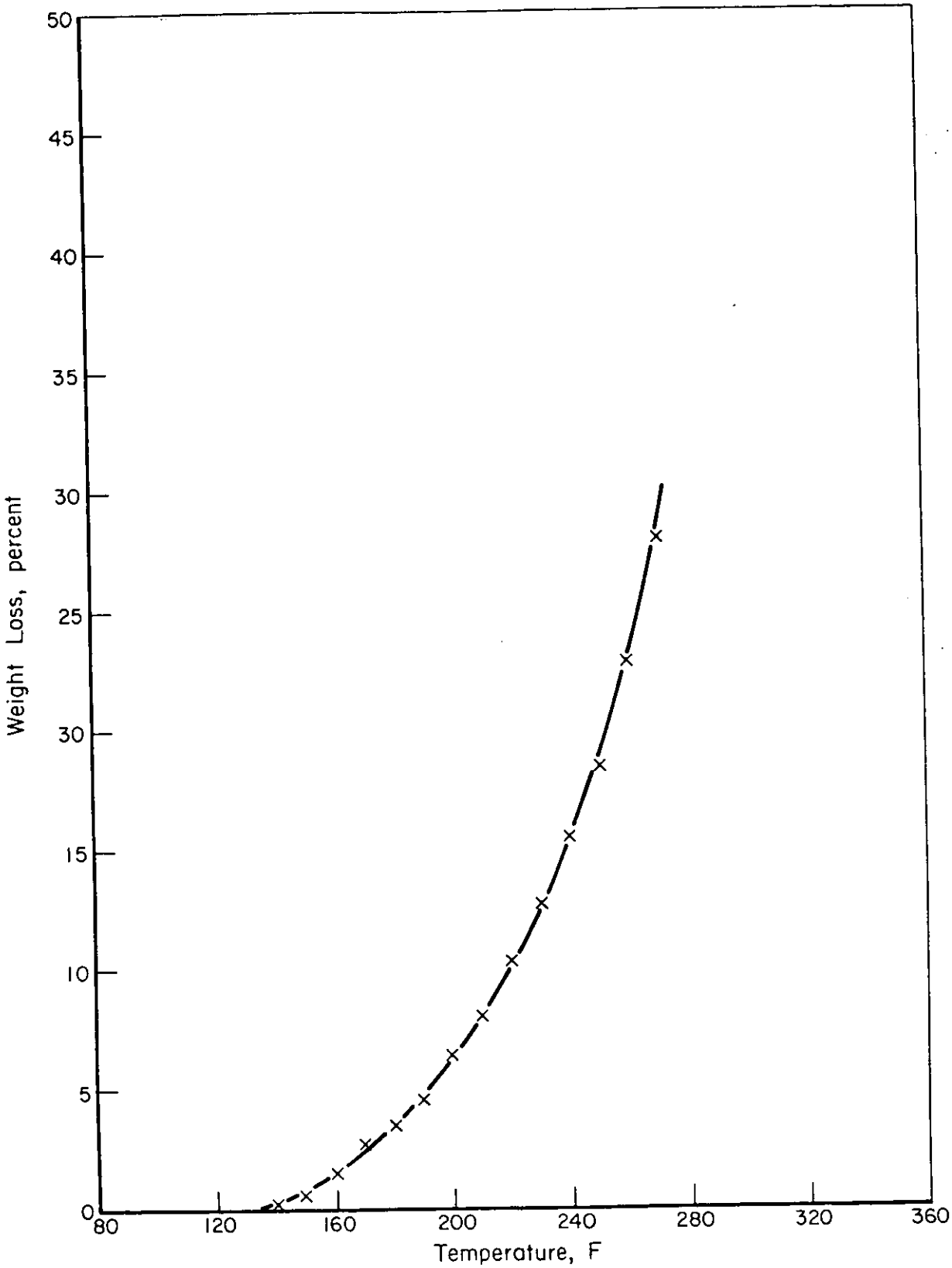


FIGURE I-12. TGA DATA FOR RESIDUE FROM EVAPORATION OF TECHNICAL GRADE TCE

There was concern about the absolute accuracy of the mass data as the long time (about 4 weeks) required for reducing some of the samples to their final form would give sufficient time to lose volatiles. However, the conclusions that were reached from examing these data were:

(1) The particulate collected from the saturator runs did not contain appreciable light ends. In the TGA runs these samples reached 96 C (205 F), or above, before a 5 percent weight loss was recorded. This suggests that a filter temperature of about 38 C (100 F) should collect all of the "particulate" and that results may not be sensitive to minor variations in filter temperature.

(2) The blowing still afterburner outlet run (Figure I-8) showed similar results as the saturator runs; i.e., a temperature of 98 C (209 F) was reached before 5 percent of the "particulate" was distilled from the sample. Again, this suggests a 38 C (100 F) filter can be used to collect this sample. Of course, the filter must be preceded by cooled impingers to reduce the water dew point to below 38 C (100 F).

    These data do show that use of the conventional Method 5 sampling train with a 121 C (250 F) filter temperature would result in collection of 12 percent less particulate than with a filter temperature below about 66 C (150 F).

(3) Finally, the blowing still afterburner inlet runs show the largest content of light hydrocarbons--in the TGA runs 5 percent of the material distilled off at between 78 and 89 C (172 and 192 F), and 29 to 32 percent had distilled off by the time the sample was heated to 121 C (250 F). If anything, more light hydrocarbons are actually present than shown by this analysis (due to loss during processing of samples). In general, it appears that a 38 C (100 F) filter should be used for sampling at this point to collect as much "particulate" as possible.

Tables I-2 and I-3 show analytical data for gaseous hydrocarbons, Table 1 for the flask samples and Table 2 for the packed column samples. Also shown are FID responses (known or estimated) for each component or fraction. The FID response for each total sample was estimated using the given FID responses for each component. Generally, all FID responses are in excess of 90 percent; that is, the FID gives a reading

TABLE I-2. ANALYSIS OF FLASK SAMPLES

Sample	Saturator ESP Outlet	Blowing Still Afterburner Inlet	Blowing Still Afterburner Outlet	FID Response Factor For Each Component, Percent
	Volume percent			
Oxygen	20.3	8.07	13.2	
Carbon monoxide	0.03	0.85	3.30	
Carbon dioxide	<0.01	0.45	<0.01	
Argon	0.96	1.11	0.99	
Nitrogen	78.1	89.1	82.4	
Hydrogen	0.06	0.03	0.05	
	Parts per million			
Sulfur dioxide, SO_2	<0.2	800	2	-
Formaldehyde, HCHO	<0.2	<50	<0.2	50 ?
Nitrogen oxides, NO_x	<0.2	<50	<0.2	-
Carbonyl sulfide, COS	<0.2	<50	<0.2	-
Ethanol, C_2H_5OH	<0.2	200	<0.2	90
Methanol, CH_3OH	<0.5	<50	<0.5	80
Acetone, CH_3COCH_3	1.0	900	<0.2	67
Benzene, C_6H_6	<0.2	100	<0.2	100
Hexane, C_6H_{14}	0.2	400	<0.2	100
Pentane, C_5H_{12}	0.2	300	<0.2	100
Butane, C_4H_{10}	<0.1	100	<0.1	100
Butene, C_4H_8	<0.1	100	<0.1	100
Propane, C_3H_8	0.4	200	<0.2	100
Propene, C_3H_6	0.2	90	<0.1	100
Ethane, C_2H_6	0.2	300	<0.2	100
Ethene, C_2H_4	0.1	200	<0.1	100
Methane, CH_4	<1.0	500	<1.0	100
Total Hydrocarbons, Max.	4.6	3490	3.5	
Min.	2.3	3390	0.0	
FID Response for Total Sample, percent	86-91	91	Undefined	

TABLE I-3. ANALYSIS OF PACKED COLUMN SAMPLES
(Weight in mg)

Sample	Saturator		Blowing Still				FID Response Factor for Each Sample Fraction, percent
	ESP Inlet	ESP Outlet	Afterburner Inlet #1	Afterburner Inlet #2	Afterburner Outlet #1	Afterburner Outlet #2	
Fraction #1	14.1	26.4	47.2	40.4	0.5	0.1	100
Alkanes Alkenes							
Fraction #2	6.3	14.9	22.4	17.4	0.2	0.1	100
Alkylbenzenes							
Fractions #3 & #4	1.5	3.5	6.0	1.4	0.1	0.0	96
POM's Thiophenes							
Fraction #5	1.9	2.6	28.9	24.6	0.3	0.2	90
Carbazoles Esters							
Fractions #6 & #7	2.0	6.1	63.2	46.3	11.5	9.7	90
Benzaldehydes Tolualdehydes Phalaldehydes Ketones							
Fraction #8	7.1	18.3	45.6	32.6	10.5	10.6	90
Alcohols Phenols Amines Acids Oxygenated diphatic compounds							
Total	32.9	72.3	213.3	162.7	23.1	20.7	
FID Response for Total Sample, percent	96	96	93	94	91	90	

equivalent to 90+ percent of the number of carbon atoms present. The only response that might be below 90 percent was the saturator outlet sample collected in the flask. For this sample, if the worst combination of values is assumed where "less than" values are reported, an 86 percent response is estimated.

Therefore, it was concluded that an FID measuring gaseous hydrocarbons exhausted from a particulate train sampling an asphalt plant would give a response of 90 percent or more of the true emission of hydrocarbons and that the FID is suitable for such use. (Alternative methods to achieve more exact numbers would probably require field collection of samples followed by costly analysis.)

CONCLUSIONS

The modified Method 5 sampling methodology used in sampling the blowing still and saturator process at the Celotex plant appeared to be suitable for such sampling. Whenever volatile materials are sampled, there is uncertainty about the effectiveness of the sampling methodology unless some proven reference method exists. In the absence of a reference method (such as is the case with asphalt roofing plants) the adequacy of the sampling method must be inferred from available data. When the sampling train was operated properly, there was no visible evidence that significant quantities of heavy hydrocarbons were passing through the sampling train without being collected. However, if the filter was allowed to get too heavily loaded, some material appeared to pass through it and FID hydrocarbon readings increased. Furthermore, the analysis of the flask samples (taken downstream of the filter) did not show the presence of hydrocarbons above about C_6 compounds. Thus, the available data suggests that the sampling procedure used at the Celotex plant is suitable for making emission measurements at asphalt roofing plants.

Of course, any sampling methodology is only as good as the methodology for recovering and analyzing or weighing the samples. Processing of volatile samples raises uncertainties and it might be desired to design a new sampling procedure to make sample processing more exact. However, the design of a sampling train to accomplish this is not simple.

EPA has considered the results of the Celotex sampling and proposed a sampling methodology based on this work as the standard method for sampling at asphalt plants.

The problem of drying and weighing asphalt roofing samples is discussed in Part II of this report.

PART II

SPECIAL DRYING STUDY OF SAMPLES COLLECTED AT FIVE ASPHALT PLANTS

Upon completion of the sampling effort described in Part I of this report, EPA engaged Battelle to participate in conducting New Source Performance Standards (NSPS) testing at five asphalt roofing plants having "best available technology" emission control devices. The nature of the asphalt roofing plant processes that were sampled was such that the majority of sample mass was expected to be organic materials. Also, the samples were collected at much lower temperatures than are normally used for Method 5 sampling trains (filter temperatures of 32 to 38 C (90 to 100 F) instead of 121 C (250 F). Thus, the samples would be more likely to contain organic compounds which would evaporate during the drying process if standard Method 5 drying procedures were used.

To examine the sample drying problem, Battelle designed and carried out a series of experiments to quantify the loss of particulate mass associated with evaporation of organics during the drying process. These experiments and the results and conclusions of this study are presented below.

SAMPLES COLLECTED

Two different processes were included in the five asphalt roofing plants for which NSPS sampling was conducted. The processes were blowing stills (1 plant) and saturator lines (4 plants). The blowing still process posed a problem in that estimated moisture content of the flue gas was about 40 percent, giving a dew point temperature of about 74 C (165 F). Because it was desired to operate the particulate sampling trains with glass fiber filter temperatures in the 38 to 50 C (100 to 120 F) range, this high moisture content would cause condensation on the filter. To avoid this condensation, a water dropout element was added upstream of the filter for sampling at blowing stills. Thus, different particulate sampling trains were used for sampling the blowing stills and the saturator lines.

Figure II-1 and II-2 show the configuration of the particulate sampling trains used for sampling the blowing still and saturator lines, respectively.

Cleanup of the particulate sampling trains resulted in collection of 7 or 9 samples from each train, as listed in Table II-1. The impinger samples from the two types of processes were quite different. For the blowing stills, a set of impingers preceded the prefilter and glass fiber filter and, thus, collected much of the liquid and solid particulates that would normally collect in the prefilter and filter. This was not so for the saturator lines, where impingers were used only downstream of the filter.

For some runs prefilters were not used (for example, in outlet runs for saturator lines where the particulate loading of the gas streams was low) and/or some samples were combined. Still, the list in Table II-1 defines the samples that had to be considered for the purpose of establishing acceptable sample drying methodology.

Only samples collected upstream of the filters were dried and weighed to determine particulate mass. Impinger water samples collected downstream of the filter (Containers 8 and 9 for blowing still runs and Containers 5, 6, and 7 for saturator line runs) were not treated further.

SPECIAL DRYING STUDIES OF SELECTED SAMPLES

The samples that were washed from nozzle and probes and from impingers were considered the most difficult for obtaining accurate particulate mass weights as there were no absolutes with which to gage the true particulate mass. The particulate deposited on the probe and glassware and collected in the impingers was combined with the washing solution during recovery and, upon drying these samples it was impossible to accurately determine how much particulate was evaporated along with the washing solution.

The initial experiment at sample drying involved drying a single sample from a preliminary run at the blowing still using conventional Method 5 procedures. To assess particulate volatility, particulate mass values were recorded for a number of days after the samples would have normally been considered dry. Table II-2 shows the results of this effort; further weight losses after the samples were initially dry were minor, about 1 to

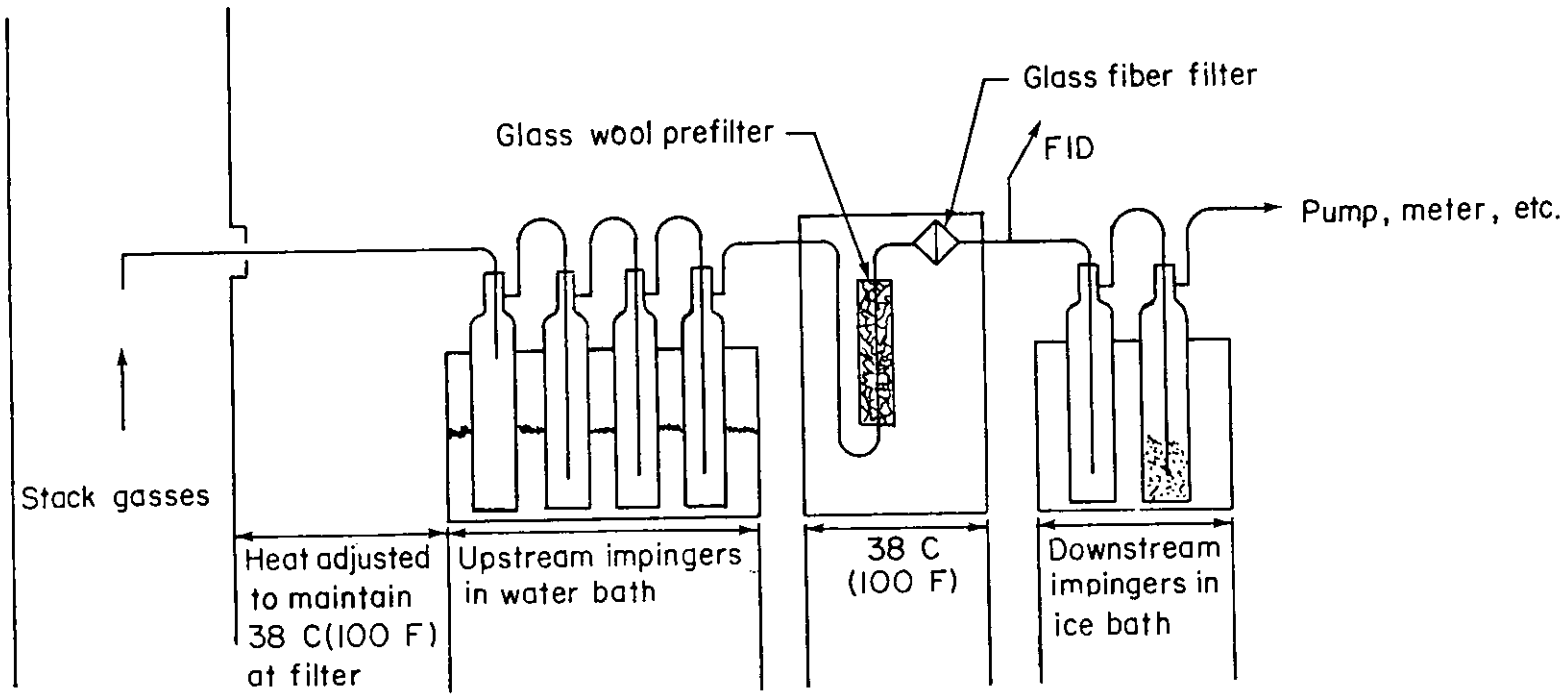
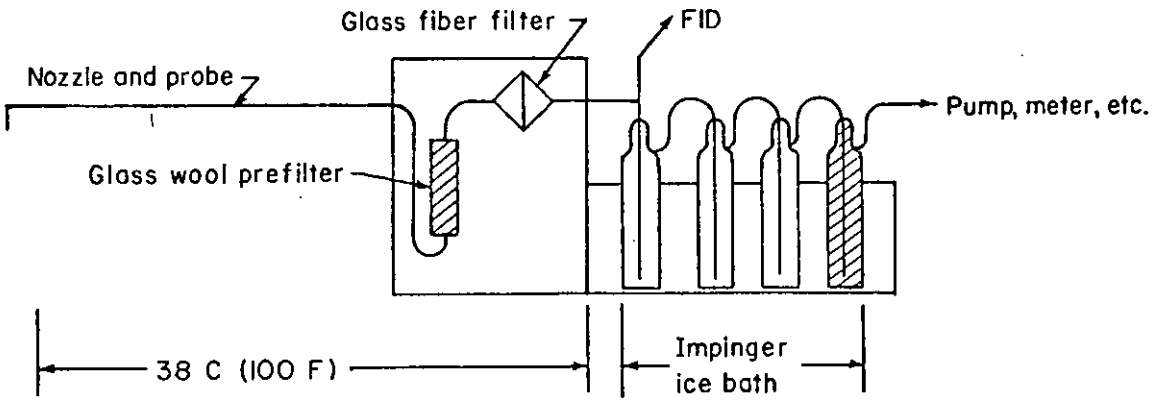
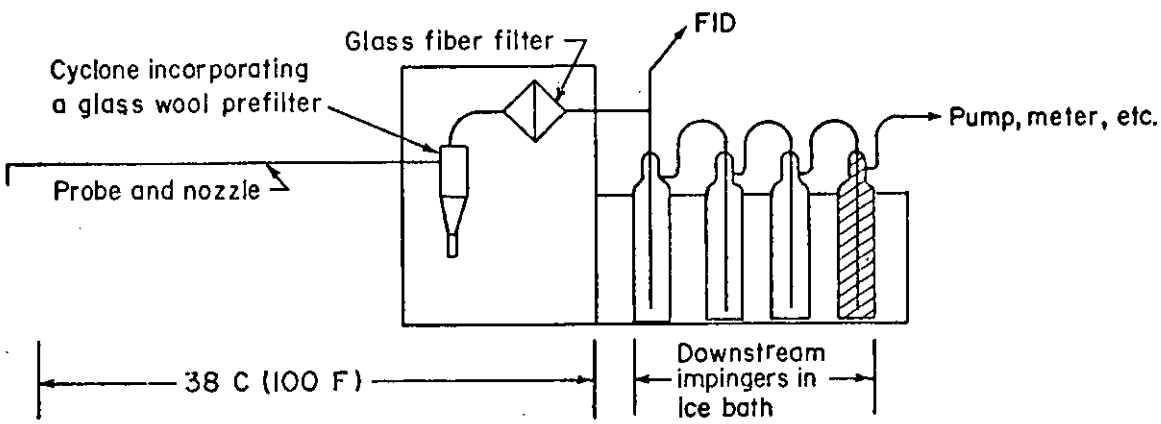


FIGURE II-1. SAMPLE TRAIN CONFIGURATION USED FOR PARTICULATE SAMPLING ON BLOWING STILLS



Sampling Train Used at Inlet Sampling Points



Sampling Train Used at Outlet Sampling Points

FIGURE II-2. SAMPLING TRAIN CONFIGURATION USED FOR PARTICULATE SAMPLING ON SATURATOR LINES

TABLE II-1. SAMPLES OBTAINED FROM PARTICULATE SAMPLING AT ASPHALT ROOFING PLANTS

	Blowing Stills	
Sampling Train Components	Sample	Sample Identification
Nozzle, probe, and front-half glassware*	TCE wash	Container 1
Nozzle, probe, and front-half glassware*	Acetone wash	Container 2
Impingers	Water & oil (bulk)	Container 3
Impingers	TCE wash	Container 4
Impingers	Acetone wash	Container 2
Prefilter	Prefilter & TCE wash	Container 5
Prefilter	Acetone wash	Container 6
Filter	--	Container 7
Back-half glassware and impingers	Water	Container 8
Back-half glassware and impingers	TCE wash	Container 9
	Saturator Lines	
Sampling Train Components	Sample	Sample Identification
Nozzle, probe, and front-half glassware*	TCE wash	Container 1
Nozzle, probe, and front-half glassware*	Acetone wash	Container 2
Prefilter	Prefilter & TCE wash	Container 3
Prefilter	Acetone wash	Container 2
Filter	--	Container 4
Back-half glassware and impingers	Water	Container 5
Back-half glassware and impingers	TCE wash	Container 6
Back-half glassware and impingers	Acetone wash	Container 7

* Except prefilter glassware.

TABLE II-2. ADDITIONAL DATA ON WEIGHT CHANGES WITH TIME FOR PRELIMINARY RUN (INLET) FROM ELK ROOFING, SAMPLE (weights in mg)

Time, days[a]	TCE & Acetone Wash of Nozzle and Probe	TCE Extract of 1/2 Impinger Water	Impinger Water (after TCE extract)	Chloroform/Ethyl Ether Extract of 1/2 Impinger Water	Impinger Water (after chlor./ether extract)	TCE Wash of Impingers	Acetone Wash of Impingers	Filter
0	1140.4	175.3	121.9	98.1	151.2	4731.9	28.2	49.0
1	1135.1	173.1	122.2	96.7	150.4	4661.2	27.2	45.2
2	1134.2	172.6	123.3	96.8	151.6	4663.2	27.3	50.4
3	--	172.1	124.1	96.7	150.7	--	--	--
5	1131.6 (-0.8%)	--	--	--	--	4586.0 (-3.1%)	27.0 (-4.3%)	51.1 (7 days)
6	--	170.5 (-2.7%)	124.6 (+2.2%)	95.7 (-2.4%)	150.8 (-0.3%)	--	--	--
19	1128.1 (-1.1%)	163.4 (-6.8%)	124.4 (+2.1%)	90.9 (-7.3%)	--	4495.8 (-5.0%)	25.9 (-8.2%)	49.7 (28 days) (+1.4%)

(a) First weights were after sample had been dried (washing solution evaporated) and sample had been desiccated for 24 hours. Samples were stored in the desiccator between subsequent weighings.

8 percent. However, these data do not indicate the magnitude of weight loss of particulate during the initial drying period (when washing solution was still evaporating). It is during this initial drying period that the most volatile portion of the particulate would be lost.

Following the above effort, Battelle conducted a brief study to assess the extent of sample loss during the initial drying period. This sample drying study was conducted using a system providing for recondensation and recovery of the washing solution and any volatiles that were carried off during the evaporation of the washing solution.

Figure II-3 shows the apparatus that were used for conducting these experiments. The samples containing TCE (or acetone) as the washing solution were dried with equipment set up as shown in Figure II-3a. TCE (or acetone) was evaporated at about 30 C (86 F) with a steady steam of air passing over the sample (to represent a sample drying under a hood at ambient temperature). The evaporated TCE was collected in a condenser maintained at -79 C (-110 F) by keeping it in dry ice. Each drying required about 48 actual hours for evaporation of all the TCE. In each case, the mass of the sample collected in the condenser flask was over 99 percent of the weight loss from the evaporation flask--suggesting a minimal sample loss during this evaporation/condensation process.

The samples containing appreciable water (impinger samples) were dried using the apparatus shown in Figure II-3b. The water was evaporated at about 99 C (210 F) and recondensed with cooling water (temperature about 10 to 16 C or 50 to 60 F). Again, over 99 percent of the evaporated material was collected in the condenser flask. Evaporation of the water required 80 to 120 hours for each sample. Thus, the drying required 2 to 3 weeks per sample.

In both cases the evaporation was assumed to be complete when no visual evidence existed of further washing solution carryover (drops of condensation could not be seen forming on the discharge tube in the condenser flask).

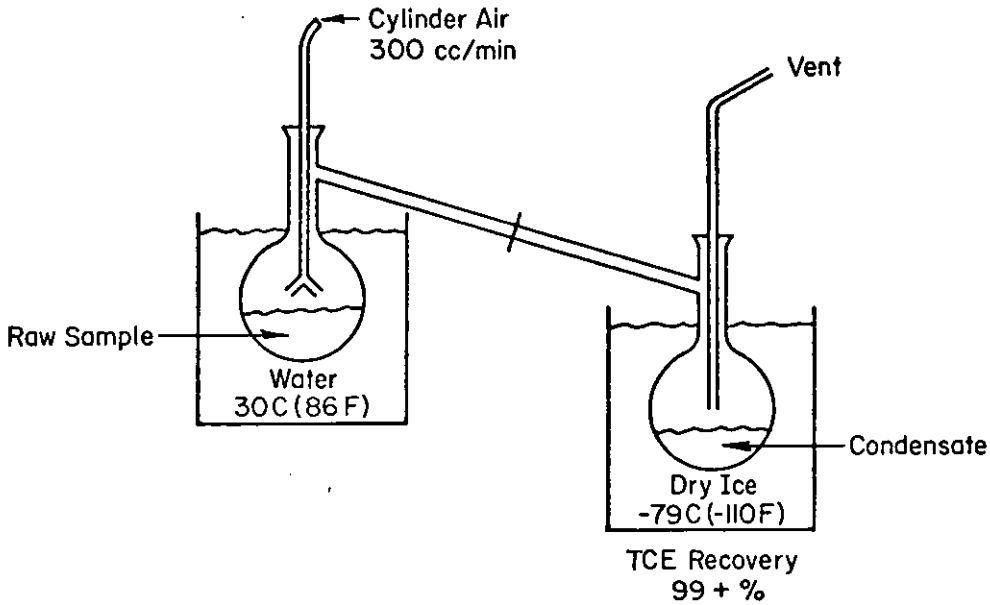


FIGURE II-3a. TCE SAMPLES

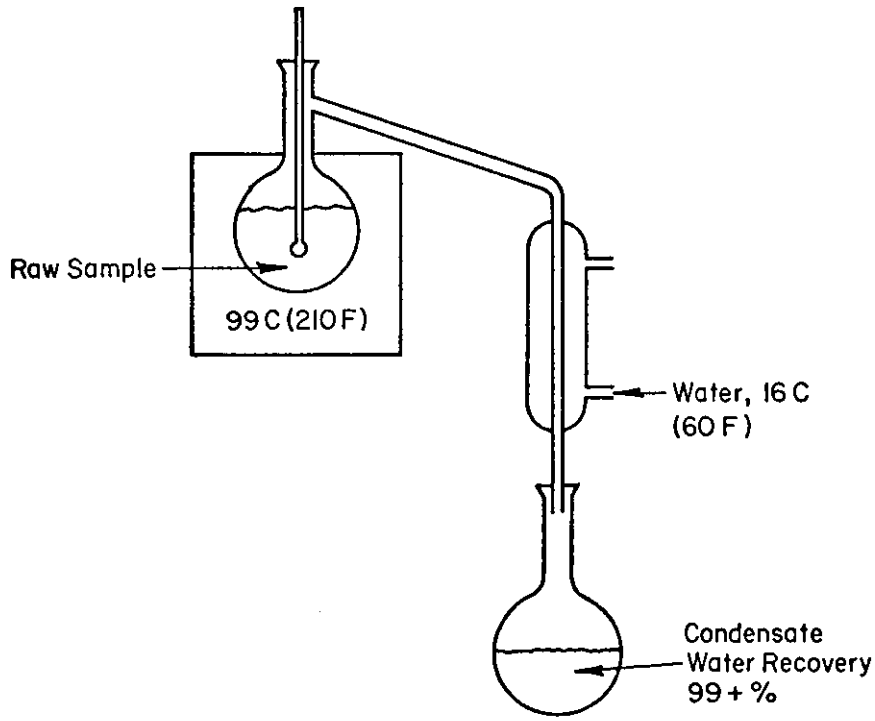


FIGURE II-3b. WATER SAMPLES

FIGURE II-3. APPARATUS FOR SPECIAL DRYING OF ASPHALT SAMPLES

Results of Special Drying Studies of Samples Containing TCE and Water

Saturant Line Samples. TCE washes of the nozzle, probe, and front-half glassware for two saturant line outlet samples were selected for the special drying procedures as shown in Figure II-3a. These samples were outlet run samples for plants controlled with a High Energy Air Filter (HEAF) unit (Celotex, Loss Angeles--EPA Sample S75-002-015) and an electrostatic precipitator (Celotex, Fairfield, Alabama--EPA Sample S75-006-402). To determine if a significant amount of the particulate catch from these samples was being evaporated during evaporation of the TCE, gas chromatographic analyses of the condensate were performed.

Chromatographic analysis of the condensate showed no straight-chain hydrocarbons above C11* for condensate from Sample S75-002-015 and no evidence of straight-chain hydrocarbons for condensate from Sample S75-006-402 (when compared to TCE blanks in both cases). Thus, there was no evidence that significant particulate catch from these samples was being evaporated while the TCE was being evaporated.

Blowing Still Samples. Selected samples from the inlet and outlet runs at the blowing still were examined using the cold trap techniques described above. Samples examined were:

	Inlet	Outlet
TCE wash of nozzle and probe	S75-006-037	S75-006-043
Bulk oil and water collected in impingers upstream of the filter	S75-006-038	S75-006-044
--oil (removed with separatory funnel)	S75-006-038a	S75-006-044a
	(Oil samples were not dried)	
--TCE extract of water portion	S75-006-038b	S75-006-044b
--Water remaining after oil removal and TCE extraction	S75-006-038c	S75-006-044c
TCE wash of impingers	S75-006-039	S75-006-045

* C11, C12, etc., refers to hydrocarbons with 11, 12, etc., carbon atoms. Detection limit of the GC was about 20 ppm of each compound.

The samples containing TCE were treated as shown in Figure II-3a. These samples included (1) TCE rinses of the nozzle and probe, (2) TCE extracts of the impinger water, and (3) TCE rinses of the impingers. The water portions from the impinger (remaining after physical separation of the oil and extraction with TCE) were dried as shown in Figure II-3b.

Table II-3 gives weight and time data for the drying of these blowing still samples. The blowing still outlet particulate weights were stable with time: weight losses after the samples appeared to be dry (washing solution carryover terminated as judged by eye) were less than 1 percent per day. However, for the inlet samples, particulate mass changes occurred after the time at which the samples appeared dry. Much of the weight loss beyond the visually dry point accurred during the desiccation period and may have been due to evaporation of water. However, this suggests that the visually dry samples contained up to 20 percent water; this seemed unlikely judging from their appearance.

Analysis of the condensate samples by gas chromatograph and comparison to similar analysis of the raw samples showed that little organic appeared to evaporate and recondense when drying the samples by using the procedures shown in Figure II-3. Table II-4 presents the results of the GC analysis. The more volatile portions of the particulate could be expected to be lost during the initial period, and thus, the recovery of only small quantities of organic in the condensate suggested particulate loss was small. Hence, since these procedures were intended to duplicate the evaporation mechanisms occurring in modified Method 5 drying procedures as much as possible, while still collecting the condensate, these results suggest that methods similar to Method 5 drying procedures could be used to dry blowing still samples without significantly affecting the weight data. To further confirm this conclusion and to explain differences in the results shown in Table II-3 and Table II-4, more controlled experiments could be conducted. Such tests were not within the budget or time constraints of this task.

Issuance of the Draft Procedure. At this point in the program EPA issued a draft of the procedure for "Determination of Particulate and Total Gaseous Hydrocarbon Emissions from the Asphalt Roofing Industry".*

* The draft procedure is reproduced in Appendix B.

TABLE II-3. WEIGHTS VERSUS TIME FOR SPECIAL DRYING OR SELECTED BLOWING STILL SAMPLES (weights are in mg)

	Date	Nozzle & Probe TCE Wash	Date	TCE Extract of Impinger Water	Date	Water, After TCE Extract	Date	TCE Wash of Impinger
Elk Inlet (Run B-5)		Sample S75-006-037		Sample S75-006-038b		Sample S75-006-038c		Sample S75-006-039
	2/19/76	16,766(a)	12/18/75	1,277.3(a)	12/29/75	1,705.3(a)	1/5/76	8,327.0(a)
	2/24/76	8,388(b)	12/19/75	1,084.8(b)	1/9/76	1,650.5(b)	1/9/76	5,182.5(b)*
			12/29/75	984.8(c)	1/13/76	1,647.2(c)	1/12/76	4,134.6(c, d)
			1/19/76	979.8(c)	1/19/76	1,639.4(c)	1/13/76	3,832.5(c)
							1/13/76	**
							1/19/76	2,822.5(c)***
Elk Outlet (a) weights		Sample S75-006-043		Sample S75-006-044b		Sample S75-006-044c		Sample S75-006-045
	12/29/75	180.6(a)	1/9/76	12.3(b)	12/15/75	309.8(b)	1/9/76	496.0(b)
	1/9/76	190.3(b,e)	1/19/76	9.3(c)	12/17/75	303.8(c)	1/13/76	490.0(c)
	1/19/76	180.3(c)			12/19/75	296.8(c)	1/19/76	496.8(c)
					12/22/75	294.8(c)		
					12/29/75	288.3(c)		
					1/9/76	281.1(c)		
					1/13/76	279.5(c)		
					1/19/76	273.8(c)		

* Arm broke off flash prior to this weighing.
** 250 mg removed for analysis.
*** Includes 250 mg correction for portion of sample removed on 1/13/76.

(a) Solvent or water evaporated by visual examination, sample not desiccated.
(b) After sample was desiccated for at least 24 hours.
(c) Sample desiccated between weighings.
(d) GC Analysis showed significant (20 to 50 percent) of sample was TCE.
(e) Assume 10 mg error in weighing.

TABLE II-4. ANALYSIS OF RAW SAMPLES AND CONDENSATE FOR ORGANICS

Sample		Results of GC Analysis (Specifically detects straight-chain hydrocarbons)	
		Raw Sample	Condensate
Inlet Samples			
S75-006-037	TCE probe and nozzle wash	C11 to C32 organics present; peak in C13 to C16 range	Nil organics, organics less than 1 percent of raw sample
S75-006-038b	TCE extract of impinger water	Organic present, peak concentration in C16 to C18 range	Contained C10 and C12 compounds equally to 10 percent and 4 percent of raw sample, respectively
S75-006-038c	Impinger water (after extraction)	Slight organic content; some TCE evident	C10 to C25 organics contained in a 3 ml TCE bubble; trace of raw organics in water portion
S75-006-039	TCE wash of impingers	C11 to C32 organics present; peak in C13 to C16 range	Contained C10, C12, and C14 compounds equal to 5 percent, 2 percent, and less than 1 percent of raw sample, respectively
Outlet Samples			
S75-006-043	TCE probe and nozzle wash	Nil organic content	Trace of C10 to C15 organics
S75-006-044b	TCE extract of impinger water	Not analyzed	Trace of C10 and C11 compounds
S75-006-044c	Impinger water (after extraction	Not analyzed	Trace of organics
S75-006-045	TCE wash of impingers	Nil organic content	Nil organic content

The draft procedure is similar to the Method 5 procedure but provides for:

(1) Collection of sample with a filter at a temperature of not over 50 C (122 F) and, for sampling moist streams, includes impingers upstream of the filter.

(2) Drying samples as in Method 5. However, in recognition of the possible volatility of samples, the particulate mass is to be followed until the weight loss over 24 hours does not exceed 10 percent. The mass value to be used in emission calculations is the average of the last two particulate mass values--the two values that do not differ by more than 10 percent.

Drying Certain-Teed Products Plant Samples With Draft Procedure. As a final attempt to evaluate the loss of particulate when drying solvent-laden saturator line samples, the draft procedure was used to dry samples from the Certain-Teed Products Plant (Shakopee, Minnesota). These samples were air-dried in a hood as per the draft procedures and dessicated. A time-record of sample weights was kept for these samples. Table II-5 gives particulate mass values for desiccation periods of up to about 500 hours. The weights used in particulate emissions calculations were the average of two readings taken 24 hours apart and where the latter reading was not less than 10 percent below the earlier reading. For all samples except some TCE washes of the nozzle and probe, the average of the first two readings given in Table II-5 was used to calculate the particulate mass.

Evaporative Loss from Oil Samples

An attempt was made to evaluate the volatility of the oil collected in the upstream impingers during the blowing still runs by measuring weight loss of the bulk oil when exposed to conditions similar to those recommended for drying asphalt roofing plant samples. Approximately one gram of this oil was removed from each of two samples and placed on a tared drying dish in a hood.

TABLE II-5. DRYING DATA FOR PARTICULATE SAMPLES COLLECTED AT CERTAIN-TEED PRODUCTS

Time, hrs	Sampling Point and Run																
	TP1-1	TP1-2	TP1-3	TP2-1	TP2-2	TP2-3	TP3-1	TP3-2	TP3-3	TP4-1	TP4-2	TP4-3	TP5-1	TP5-2	TP5-3	TP5-4
TCE Wash of Nozzle, Probe, and Front-Half Glassware (except prefilter)																
0[a]	222.2	251.8	471.4	102.8	78.2	104.7	19,993.2	157.2	454.9	26.6	15.6	12.3	49.8		219.7	50.9
24	171.2	206.3	340.4	88.1	71.3	86.8	16,909.5	128.8	347.5	27.1	16.0	12.8	47.7		218.0[c]	51.0[c]
48	170.2	183.2	310.6	86.9	70.1	84.0	15,279.4	131.2	322.8	26.9	15.5	13.1	45.0		213.2[d]	--
144	155.1	165.1	282.2	86.2	69.4	80.3	12,564.6	124.3	294.2	--	--	11.6	42.7		209.6[f]	48.6[f]
336	150.6	160.7	265.9	86.9	71.0	78.7	--	118.5	278.4	--	--	--	42.7			
504	145.9	154.4	256.6	--	69.7	76.0	11,403.9	112.6	266.1	--	--	--	--			
TCE Rinse of Prefilter																
0[a]	730.2	518.5	793.8		42.5	72.7	1119.3	336.3	880.6	28.3	12.8	24.1		985.1	2783.1	1087.0
24	729.4	--	--		41.7	71.9	1108.1	--	--	28.0	12.0	23.6		--	--	--
72	--	518.2	790.1		--	--	1098.6	335.2	877.1	--	--	--		977.2	2755.4	1079.3
120	728.0	--	--		40.4	70.6	--	--	--	--	11.0	--		--	--	--
264	--	518.6	787.4		--	--	--	334.3	871.5	27.8	--	--		967.1	2726.0	1068.2
312	727.1	--	--		40.9	71.5	--	--	--	--	12.2	23.8		--	--	--
432	--	--	786.2		--	--	--	333.8	867.9	--	--	--		959.7	2705.5	1061.5
480	724.7	--	--		40.4	70.0	--	--	--	--	11.2	--		--	--	--
Acetone Wash of Nozzle, Probe, and Front-Half Glassware (including prefilter)																
0[a]			7.4		6.2	5.3			19.6		19.9	45.6			1.8	3.4
24			7.2		6.4	5.9			19.5		19.6	44.2			2.8[c]	3.9[c]
336						7.3						43.4[e]			2.1[d]	3.8[d]
504						6.1						41.1[g]			2.6[f]	4.1[f]
Filter																
0[b]	313.7	185.1	200.0	49.9	29.7	22.7	70.5	147.5	977.6	5.6	8.1	13.5	1.1	926.8	85.0	58.2

(a) Time zero weighing if after apparent evaporation of all TCE followed by 24 in desiccator.
(b) After 24 hr in desiccator
(c) At 72 hr.
(d) At 264 hr.
(e) At 312 hr.
(f) At 432 hr.
(g) At 480 hr.

Note: Sampling points TP1 and TP3 were on the control device inlet; TP2 and TP4 on the control device outlet; TP5 was a side stream.

Weight data was observed for periods of up to 118 hours. These data are tabulated and shown graphically in Figure II-4. A large initial weight loss was observed--about 25 percent in 16 hours followed by a small additional weight loss. Another set of 1 gram oil samples was mixed with 10 ml of TCE and air dried to determine the effect of TCE on evaporative loss of sample. Within 16 hours the TCE had evaporated and the sample had lost about 30 percent of its mass (a slightly greater loss than for the samples with no TCE present). To preclude the possibility that the mass decrease was water, an oil sample was dried (with $MgSO_4$) and then exposed to air (as during evaporation of washing solutions). Again, the rate of mass loss for the "dried" sample was the same as for the oil alone. Thus, we concluded that the oil portion of the impingers catch was evaporating upon exposure to air. Of all sample drying data, these data were the most suggestive that a particulate weight loss problem could occur during drying of the asphalt plant samples.

Due to the high weight loss measured for the above samples, it was decided to expose recovery oil samples to air in the same manner and determine weight loss with time. Table II-6 contains results of these determinations. None of the recovery oil samples experienced a significant weight loss when one gram was exposed to air for 87 hours. Because the recovery oil settles out of the gas stream within the gas duct and control device, it is expected to be more condensible and less volatile than the oil sampled as a vapor and caught in the sampling train. Thus, it is not surprising that these oil samples were less volatile than those reported in Figure II-4.

Long-Term Drying of Samples Collected of Glass Fiber Filters

The long-term weight loss of the sample collected on glass fiber filters from saturant line runs was also examined. Results of these observations are reported in Table II-7. Based on drying of filter samples over a period of 3 to 4 months, weight loss of filter samples was very low. Of course, some organic could have been lost during the desiccation period, prior to the initial weighings. However, the best interpretation of these data suggests only a small weight loss of the filters over a long-time period.

FIGURE II-4. AIR DRYING OF OIL SAMPLES FROM BLOWING STILL IMPINGERS (weight in mg)

Time, hrs	Sample: Oil From Impingers A	Oil From Impingers B	Oil From Impingers & TCE C	Oil From Impingers & TCE D	Dried Oil From Impingers
0	1000.3	1000.0	1005.8	1004.1	760
16	752.5 (-25%)	762.7 (-24%)	716.8 (-29%)	684.4 (-32%)	571.5 (-25%)
42	708.0	720.2	682.9	652.9	530.8
64	701.3	714.0	676.1	646.5	--
87	694.5	706.4	670.0	640.7	--
118	678.8 (-32%)	686.0 (-31%)	661.9 (-34%)	634.0 (-37%)	--
192	--	--	--	--	485.5 (-36%)

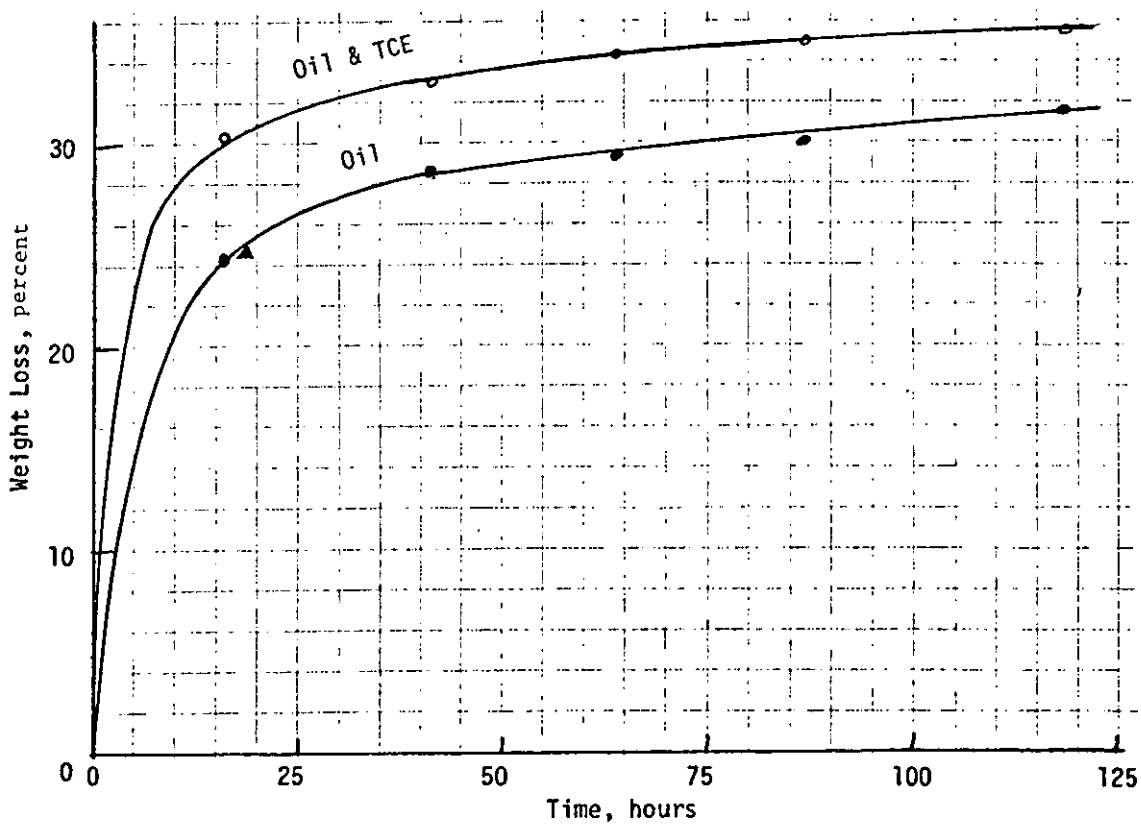


TABLE II-6. AIR DRYING OF RECOVERY OIL SAMPLES (weight in mg)

Time, hrs	Recovery Oil Elk	Recovery Oil Elk	Recovery Oil Celotex (L.A.)	Recovery Oil Johns-Manville	Recovery Oil Celotex(Ala.)	Recovery Oil Certain-Teed	Recovery Oil Certain-Teed
0	1083.3	1017.6	1003.2	1001.0	1120.0	1021.0	1025.4
16	1078.6	1017.7	998.0	1001.4	1119.1	1020.7	1026.3
42	1075.5	1017.1	991.7	1001.1	1118.8	1020.4	1026.3
64	1074.1	1016.0	987.5	1000.9	1118.0	1019.6	1026.1
87	1072.4 (-1.0%)	1016.3 (-0.1%)	985.9 (-1.7%)	1000.9 (-0.0%)	1117.5 (-0.2%)	1019.6 (-0.1%)	1026.1 (-0.0%)

TABLE II-7. LONG-TERM CHANGES IN FILTER WEIGHTS-OUTLET SAMPLES FROM SATURANT PLANT

Asphalt Plant	Plant Location	Sample No.	Initial Filter Data		Final Filter Data		Change, percent
			Mass, mg	Date	Mass, mg	Date	
Johns-Manville	Waukegan, Ill.	S75-002-025	31.2	9/24/75	29.3	1/19/75	-6.1
		-032	39.8	"	37.4	"	-6.0
		-039	50.5	"	47.3	"	-6.3
Celotex	Fairfield, Ala	S75-006-404	27.5	10/15/75	25.1	"	-8.7
		-422	24.4	"	22.2	"	-9.0
		-410	6.8	"	6.1	"	-10.3
		-428	4.1	"	3.6	"	-12.2
		-416	8.7	"	8.1	"	-6.9
		-434	9.6	"	9.0	"	-6.3
Celotex	Los Angeles, Cal.	S75-007-018	44.0	10/29/75	41.9	"	-4.8
		-042	10.6	"	10.3	"	-2.8
		-087	24.5	"	33.0	"	-4.3

CONCLUSIONS

Saturator Lines

Based on results from studying the drying of outlet samples from The Certain-Teed Products Plant in Shakopee, Minnesota, and the Johns-Manville Plant in Waukegan, Illinois, the bulk of the outlet particulate samples consisted of material that was not rapidly lost during normal Method 5 sample drying procedures. Therefore, it was concluded that procedures recommended in the draft asphalt roofing sampling method (referred to as the draft procedure) could be used for drying and weighing outlet samples from saturator lines.

The relative volatility of the inlet and outlet samples used in the special drying studies had to be considered to determine if similar drying procedures could be utilized for drying inlet samples from saturator lines. If one considers the volatile content of inlet and outlet samples collected from HEAF filter or electrostatic precipitator collectors, it can be reasoned that the outlet samples would contain the highest percentage of volatiles as the more volatile materials would be expected to pass through these control devices while the less volatile materials would tend to condense within the control device. (A similar comparison cannot be made for afterburner control devices because of the higher control device temperature.) Thus, it was concluded that the data from the special drying studies of the outlet samples was considering the most volatile sample portions and, it was further reasoned that, since the sampling drying method defined in the draft procedure was concluded to be suitable for outlet samples, they would also be suitable for the less volatile inlet samples.

Because there was no basis for believing that saturator line samples collected at the Certain-Teed Products and the Johns-Manville plants would be substantially different from other saturator line samples (including afterburner-controlled plants), it was concluded that samples from other saturator lines also could be dried using the draft procedure.

Thus, all particulate samples collected from saturator lines were dried using the draft procedures which are:

For the wash of nozzle, probe, and prefilter:

- Evaporate the solvent by placing the sample in a tared evaporating dish and in a hood at ambient temperature.
- When all of the sample solvent appears to have been evaporated, place the sample in a desiccator for 24 hours and then weigh to determine the particulate mass.
- If the sample lost substantial mass during desiccation (over 10 percent in 24 hours), place in desiccator for an additional 24 hours and weigh again. Repeat until stable mass (weight change of less than 10 percent in 24 hours) is obtained.

For glass fiber filters:

- Desiccate for a minimum of 24 hours and weigh.

Battelle experience on drying saturator line samples suggested that one 24-hour period in the desiccator was sufficient to obtain stable mass data.

Blowing Stills

The data from special drying studies of blowing still samples were more complex and less definitive. Initial drying of samples by conventional Method 5 techniques suggested no problems (Table II-2). However, exposure to air of the oil recovered from the bulk oil and water mixture lected in the impingers showed a high potential for particulate loss during sample drying (Figure II-4). Gas chromatograph analysis of samples dried using the cold trap procedure suggested only minor loss of organics upon drying.

Reviewing the preliminary particulate mass data, it was obvious that the bulk oil collected in the impingers was the major contributor to inlet particulate mass. Since this material was physically separated from the impinger water, it was assumed to be dry and no further drying was considered necessary. For inlet samples, the nozzle and probe wash appeared

to be the next largest particulate portion and appeared to be greatly affected by drying (Table II-3). For blowing still afterburnor outlet samples there was little evidence of particulate mass being affected by drying procedure.

Thus, it was finally concluded that all nozzle-probe wash samples, from the afterburner inlet would be dried using the cold trap procedure (Figure II-3a), and that the remaining samples would be dried using the draft procedures.

PART III

SAMPLING FOR POLYCYCLIC ORGANIC MATTER WITH A HIGH TEMPERATURE ADSORBENT COLUMN

As one aspect of the NSPS sampling program at several asphalt plants, polycyclic organic matter (POM) emissions were measured. POM emissions are considered important because some POM compounds are known to be carcinogenic. The procedure for sampling for POM from the saturator lines was essentially the same as the procedure Battelle has used for POM sampling at power plants, boiler, carbon black plants, etc. An extensive discussion of details of the sampling train, its operation, sample recovery, and analysis are included in Appendix C.

The sampling methodology used for this work is state-of-the-art. However, it is still undergoing validation studies and its accuracy and reliability for all sampling conditions is not proven. The absorbent sampler, the crucial component of the POM sampling train, has been validated for a limited number of "spiked" laboratory samples.

Because of the unusually high water content of the blowing still process (estimated to be up to 40 percent), it was necessary to modify the sampling train to keep the water in the vapor phase until after passing through the adsorbent sampler (to avoid plugging). Figure III-1 depicts the modified train component configuration. The probe and filter were kept at near stack temperature (about 77 C or 350 F). Two extra impingers (not normally used) were placed upstream of the adsorbent columns to collect any water droplets. The temperature in the first two impingers and adsorbent sampler was maintained at 79 C (175 F) (instead of the usual 54 C or 130 F for the adsorbent column) to keep the water in the gas phase; the estimated dewpoint was 74 C (165 F). The deviations from normal POM sampling train configuration and operation necessary for sampling at blow stills are noted in more detail in Appendix B. Table III-1 gives results of the blowing still runs with the modified sampling train.

An uncertainty was introduced into the POM measurements at the blowing still because of the temperature of the adsorbent sampler. It is

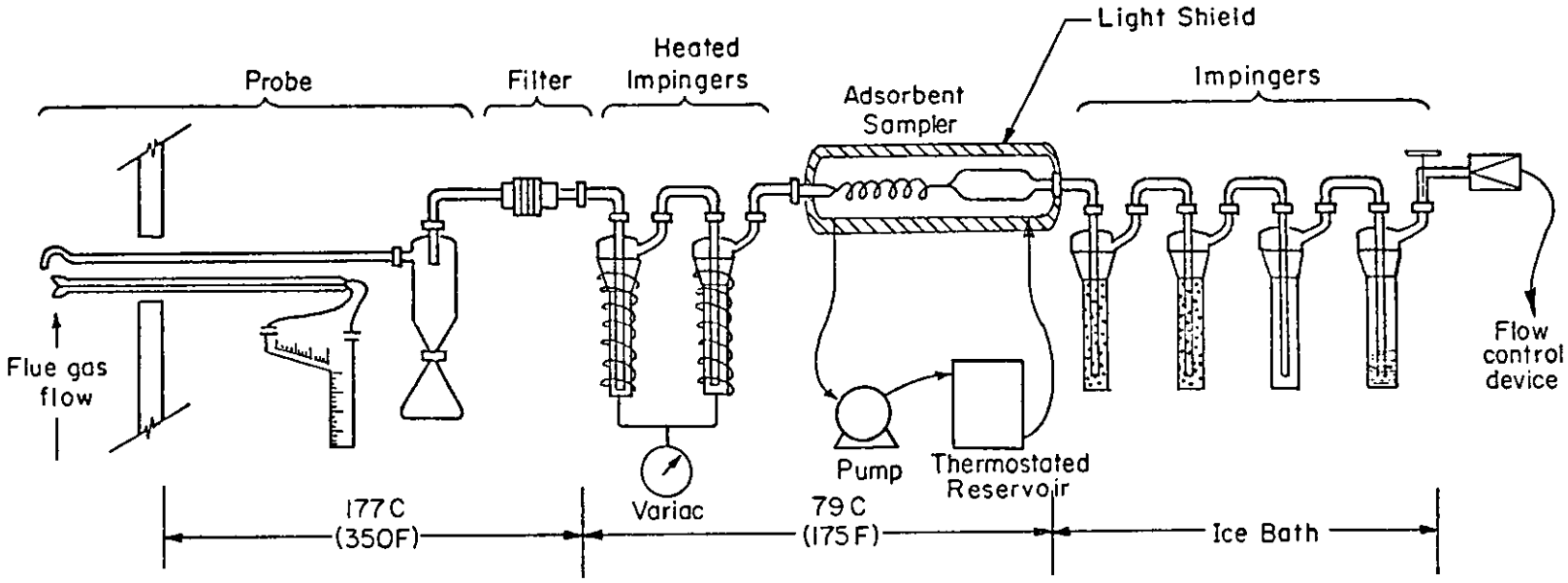


FIGURE III-1. POM SAMPLE TRAIN CONFIGURATION FOR ASPHALT BLOWING STILL PROCESS

TABLE III-1. SUMMARY OF POM DATA FOR ELK ROOFING PLANT
(With 175 F Adsorbent Column Temperature)

Component	NAS Notation	Raw Data (POM by GC-MS, No Correction for Blanks,)ug: Inlet (EPA Sample S75-006-112 & 113 BCL Sample 2-1)	Raw Data: Outlet (EPA Sample S 75-006-106 BCL Sample I5)	POM in Blank, ug (EPA Sample S 75-006-155; BCL Sample I6)	POM in Sample (Corrected for blank), ug: Inlet	POM in Sample: Outlet	POM, Loading in Gas Stream, ug/Nm^3 (a): Inlet	POM, Loading: Outlet
Anthracene/Phenanthrene		41,152	79.0	0.45	41,152	78.5	19,052	25.4
Methyl anthracenes		112,128	69.3	<0.1	112,128	69.3	51,911	22.4
Fluoranthene		1,920	6.0	0.1	1,920	5.9	888	1.91
Pyrene		2,816	7.8	0.1	2,816	7.7	1,304	2.49
Methyl Pyrene/Fluoranthene		44,096	51.8	<0.1	44,096	51.8	20,414	16.8
Benzo(c)phenanthrene	***	ND	1.0	ND	--	1.0	--	0.32
Chrysene/Benz(a)anthracene	*	12,032	4.0	<0.1	12,032	4.0	5,517	1.29
Methyl chrysenes	*	39,360	3.5	<0.1	39,360	3.5	18,222	1.13
Benzo fluoranthenes	**	1,152	4.7	<0.1	1,152	4.7	533	1.52
Benz(a)pyrene	***	512	(4.5)	(0.1)	512	(4.5)	237	(1.46)
Benz(e)pyrene		896			896		415	
Perylene		ND	ND	ND	--	--	--	--
3-Methylcholanthrene	****							
Indeno(1,2,3,-cd)pyrene	*							
Benzo(ghi)perylene								
Dibenzo(a,h)anthracene	***							
Diebenzo(c,g)carbazole	***							
Dibenz(ai and ah)pyrenes	***							
Coronene								
TOTAL							118,493	74.72

(a) Sample volume, Nm^3: Inlet Run = 2.16
Outlet Run = 3.09

normally operated at 54 C (130 F), and has been tested in laboratory validation runs at that temperature. No data were available on the performance of the adsorbent sampler at the 79 C (175 F) temperature necessary to avoid water condensation at the blowing still. Thus, to calibrate the 79 C (175 F) runs at the blowing still, Battelle conducted sampling with parallel POM sampling trains at a saturator line with the adsorbent column temperatures for the two trains being 54 C and 79 C (130 F and 175 F). Table III-2 gives results of these runs. (Field data sheets, laboratory reports, and emissions calculations for the blowing still runs and calibration runs are included in Appendix D.)

From Table III-2, it can be seen that the adsorbent column does not appear to be nearly as effective at "catching" POM at 79 C (130 F) as it does at 54 C (130 F).

The overall ratio of POM collected by the 54 C (130 F) train to POM collected by the 79 C (175 F) train is 74.7. However, for the POM of molecular weight 228 and above (which includes all the NAS starred compounds), the equivalent ratio is only 4.0. In fact, for several of the heavier compounds listed in Table III-2, the 54 C (130 F) and 79 C (175 F) sampling trains gave a nearly identical loading.

Thus, it is concluded that the overall POM data from the blowing still sampling are minimum values, and that the actual total POM emissions may have been many times the measured values. However, the data on specific heavy POM compounds may be fairly accurate.

Pending completion of additional method validation runs at higher adsorbent column temperatures (now in process under EPRI sponsorship), we are unable to recommend a suitable sampling train for sampling POM from high moisture blowing still exhaust gas.

TABLE III-2. SUMMARY OF POM DATA FOR 130 F and 175 F SIMULTANEOUS RUNS ON SATURATOR LINE

Component	NAS Notation	Raw Data (POM by GC-MS, No Correction for Blanks, μg) 130 F Run (EPA Sample S75-006-329; BCL Sample 2-3)	175 F Run (EPA Sample S75-006-335; BCL Sample 2-4)	POM in Blank, μg (EPA Sample S75-006-155, BCL Sample 16)	POM in Sample (Corrected for blank), μg 130 F Run	175 F Run	POM, Loading in Gas Stream g/Nm3 (a) 130 F Run	175 F Run	Ratio 130 F Run / 175 F Run
Anthracene/Phenanthrene		631.	10.0	0.45	630.6	9.55	255.	4.48	57.
Methyl Anthracenes		1359.	2.0	0.1	1359.	2.0	550.	0.94	585.
Fluoranthene		8.	0.15	0.1	7.9	0.05	3.20	0.02	160.
Pyrene		60.	0.85	0.1	59.9	0.75	24.3	0.35	69.
Methyl Pyrene/Fluoranthene		185.	2.7	0.1	185.	2.7	74.9	1.27	59.
Benzo(c)Phenanthrene	***	ND	ND	ND	--	--	--	--	--
Chrysene/Benz(a) Anthracene	*	35.	1.2	0.1	35.	1.2	14.2	0.56	25.4
Methyl Chrysenes	*	13.	4.8	0.1	13.	4.8	5.26	2.25	2.34
Benzo Fluoranthenes	**	1.9	1.75	0.1	1.9	1.75	0.77	0.82	0.94
Benz(a)Pyrene	***	1.0	0.7		1.0	0.7	0.41	0.33	1.24
Benz(e)Pyrene		1.3	1.2	(0.1)	1.3	1.2	0.53	0.56	0.95
Perylene		0.1	1.6	ND	0.1	1.6	0.04	0.75	0.053
3-Methylcholanthrene	****								
Indeno(1,2,3,-cd)Pyrene	*								
Benzo(ghi)Perylene									
Dibenzo(a,h)Anthracene	***								
Dibenzo(c,g)Carbazole	***								
Dibenz(ai and ah)Pyrenes	***								
Coronene									
TOTAL							928.61	12.33	75.31

(a) Sample volume, Nm^3: 130 F Run = 2.47
175 F Run = 2.13

CONCLUSIONS

Based on the results of one run, it may be concluded that:

1. The sampling train with the 79 C (175 F) adsorbent column is not suitable for collecting POM compounds of molecular weights below about 228 as, apparently, most of these lighter compounds pass through the adsorbent column.

2. The 79 C (175 F) adsorbent column appears suitable for collecting POM compounds of molecular weight 228 and above with nearly the efficiency of the 54 C (130 F) column.

Of course, when contemplating such a complex subject as POM sampling, it is dangerous to base conclusions on one run. Therefore, if extensive POM sampling in high-moisture streams is required, some additional laboratory studies must be made to better define the effect of column temperature on collection efficiency.

APPENDIX A

FIELD AND LABORATORY DATA FOR PARTICULATE RUNS AT CELOTEX PLANT, CINCINNATI, OHIO

- Preliminary Data
- Sample Run Data
- Laboratory Data

PRELIMINARY DATA

PRELIMINARY VELOCITY TRAVERSE

PLANT [illegible]

DATE [illegible]

LOCATION [illegible]

STACK I.D. 32"

BAROMETRIC PRESSURE, in. Hg [illegible]

STACK GAUGE PRESSURE, in. H_2O 0.01

OPERATORS [illegible]

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
5"	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
12"	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
AVERAGE		

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
AVERAGE		

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PRELIMINARY VELOCITY TRAVERSE

PLANT [illegible]

DATE [illegible]

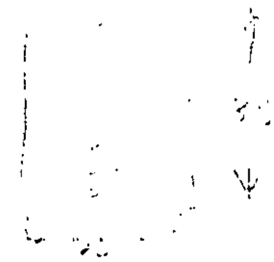
LOCATION [illegible]

STACK I.D. [illegible]

BAROMETRIC PRESSURE, in. Hg 29.[illegible]

STACK GAUGE PRESSURE, in. H_2O [illegible]

OPERATORS [illegible]



SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
3	0.3(0.5) = .15	145
6	1.3 " = .7	145
[illegible]	2.0 " = 1.0	[illegible]
12	[illegible] = 1.0	[illegible]
[illegible]	[illegible] = 1.2	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
AVERAGE		

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
AVERAGE		

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PRELIMINARY VELOCITY TRAVERSE

PLANT [illegible]
DATE [illegible]
LOCATION [illegible]
STACK I.D. [illegible]
BAROMETRIC PRESSURE, in. Hg [illegible]
STACK GAUGE PRESSURE, in. H_2O
OPERATORS [illegible]

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
[illegible]	[illegible]	[illegible]
[illegible]		
[illegible]		
[illegible]		
[illegible]		
[illegible]		
[illegible]		
[illegible]	V	
AVERAGE		

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
AVERAGE		

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A-4

PRELIMINARY VELOCITY TRAVERSE

[illegible]

PLANT [illegible]

DATE 6/3/75

LOCATION [illegible]

STACK I.D. ____

BAROMETRIC PRESSURE, in. Hg 29.3

STACK GAUGE PRESSURE, in. H_2O ____

OPERATORS [illegible]

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
1 3	[illegible]	650
2 5	0.5	!
3 9		
4 16	0.9	!
5 20	0.5	
6 23	0.5	
7 26	[illegible]	
8 30	0.4	V
AVERAGE		

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
AVERAGE		

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NOMOGRAPH DATA

PLANT [illegible]

DATE [illegible]

SAMPLING LOCATION [illegible]

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	1.72
AVERAGE METER TEMPERATURE (AMBIENT + 20 °F), °F	$T_{m_{avg.}}$	[illegible]
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	[illegible]
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	[illegible]
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	[illegible]
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	1.0
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	150
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	1.4
C FACTOR	1.0	
CALCULATED NOZZLE DIAMETER, in.	0.195	
ACTUAL NOZZLE DIAMETER, in.	[illegible]	
REFERENCE Δp, in. H_2O	[illegible]	

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NOMOGRAPH DATA

PLANT [illegible]

DATE [illegible]

SAMPLING LOCATION [illegible]

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	1.83
AVERAGE METER TEMPERATURE (AMBIENT + 20 °F), °F	$T_{m_{avg.}}$	110
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	5
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	[illegible]
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	[illegible]
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	[illegible]
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	155
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	.13
C FACTOR	[illegible]	
CALCULATED NOZZLE DIAMETER, in.	[illegible]	
ACTUAL NOZZLE DIAMETER, in.	[illegible]	
REFERENCE Δp, in. H_2O	.115	

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NOMOGRAPH DATA

PLANT [illegible]

DATE [illegible]

SAMPLING LOCATION [illegible]

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	[illegible]
AVERAGE METER TEMPERATURE (AMBIENT + 20 °F), °F	$T_{m_{avg.}}$	[illegible]
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	[illegible]
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	[illegible]
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	[illegible]
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	[illegible]
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	[illegible]
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	[illegible]
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	
C FACTOR		[illegible]
CALCULATED NOZZLE DIAMETER, in.		0.59
ACTUAL NOZZLE DIAMETER, in.		[illegible]
REFERENCE Δp, in. H_2O		[illegible]

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NOMOGRAPH DATA

PLANT [illegible]

DATE [illegible]

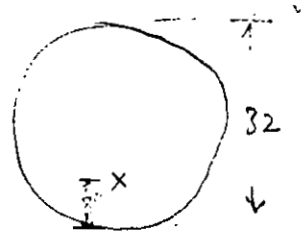
SAMPLING LOCATION [illegible]

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H_2O	$\Delta H_@$	1.72
AVERAGE METER TEMPERATURE (AMBIENT + 20 °F), °F	$T_{m_{avg.}}$	110
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	30
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	[illegible]
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073$ x STACK GAUGE PRESSURE in in. H_2O)	P_s	[illegible]
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	1.0
AVERAGE STACK TEMPERATURE, °F	$T_{s_{avg.}}$	650
AVERAGE VELOCITY HEAD, in. H_2O	$\Delta p_{avg.}$	0.7
MAXIMUM VELOCITY HEAD, in. H_2O	$\Delta p_{max.}$	
C FACTOR	0.60	
CALCULATED NOZZLE DIAMETER, in.	[illegible]	
ACTUAL NOZZLE DIAMETER, in.	0.27	
REFERENCE Δp, in. H_2O	1.15	

EPA (Dur) 234
4/72

FIELD DATA

PLANT [illegible]
DATE [illegible]
SAMPLING LOCATION inlet, [illegible]
SAMPLE TYPE [illegible]
RUN NUMBER 1
OPERATOR Webb, [illegible]
AMBIENT TEMPERATURE [illegible]
BAROMETRIC PRESSURE 27.24
STATIC PRESSURE, (P_s) [illegible]
FILTER NUMBER (s) 0.3776



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE [illegible]
NOZZLE I.D. 0.36
ASSUMED MOISTURE, % 5
SAMPLE BOX NUMBER 1
METER BOX NUMBER A
METER $\Delta H_@$ 1.83
C FACTOR 1.08
PROBE HEATER SETTING [illegible]
HEATER BOX SETTING [illegible]
REFERENCE Δp [illegible]

READ AND RECORD ALL DATA EVERY [illegible] MINUTES

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 DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	~~SAMPLE BOX~~ TEMPERATURE, °F [illegible]	IMPINGER TEMPERATURE, °F
		21.1									
1	[illegible]	[illegible]	0.13		2.1						
	Stop 1635	Probe down									
		[illegible]									
	Start 1718	61.6	0.13		2.1	140	104	96	11	58	66
	1729	[illegible]	0.14		2.2	110 ?	105	105	12	105	82
	1850	133.6	0.12		1.7	107 ?	95	100	11	100	73
	1920	[illegible]									
		72.5 ft³									
	Dried gross	400.3 g	Impinger gross			620					
	tare	475.0 g	initial			0					
		15.3 g				62.0					
		62.0									
	total	77.3 g									

COMMENTS:

Probe [illegible] 1820

EPA (Dur) 235
4 72

Pre Sim C

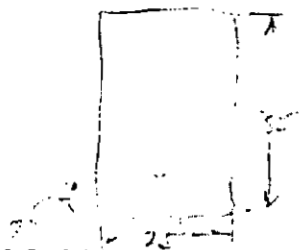
T_{23} 6.60179

T_{23} 7.[illegible]

Probe Temp F

FIELD DATA

PLANT Colstrip
DATE 6/12/75
SAMPLING LOCATION outlet, Scrubber
SAMPLE TYPE Point
RUN NUMBER 1
OPERATOR [illegible]
AMBIENT TEMPERATURE 90 F
BAROMETRIC PRESSURE [illegible]
STATIC PRESSURE, (P_s) [illegible]
FILTER NUMBER (s) [illegible]



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 4' Glass
NOZZLE I.D. .175
ASSUMED MOISTURE, % 5
SAMPLE BOX NUMBER 1
METER BOX NUMBER 5
METER $\Delta H_@$ [illegible]
C FACTOR 1.0
PROBE HEATER SETTING –
HEATER BOX SETTING –
REFERENCE Δp [illegible]

READ AND RECORD ALL DATA EVERY – MINUTES

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 DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	~~SAMPLE BOX~~ TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
		– 57.5									
1	1010	54.2	1.5		1.35						
	Stop 1633	Process down									
		54.2									
	Start 1717	55.0	1.5		1.35	145	105	102	7	84	66
	1753	115.7	1.5		1.35	140	104	104	7	89	72
	1848	139.[illegible]	1.5		1.35	140	104	104	7	92	65
	Stop [illegible]	→ [illegible]									
		– 101.[illegible] ft^3									

Drierite gross 489.2 g
Tare 475.0 g
14.2
2.0
Total 22.2 grams

Impinger Vol gross 9 ml
Tare 0 ml
9 ml

COMMENTS:

[illegible]

EPA (Out) 235
4 72

A-10

PLANT
DATE
SAMPLING LOCATION
SAMPLE TYPE
RUN NUMBER
OPERATOR
AMBIENT TEMPERATURE
BAROMETRIC PRESSURE
STATIC PRESSURE, (P_s)
FILTER NUMBER (s)

PROBE LENGTH AND TYPE
NOZZLE I.D.
ASSUMED MOISTURE, % 10
SAMPLE BOX NUMBER
METER BOX NUMBER
METER $\Delta H_@$ 1.83
C FACTOR
PROBE HEATER SETTING
HEATER BOX SETTING
REFERENCE Δp

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY _____ MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24-hr CLOCK) / SAMPLING TIME, min	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	~~SAMPLE BOX~~ TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
		133.9									
	1451		.02		0.9	210		85	5.0		
	1521										
	1220		.02		0.9				5.0		

COMMENTS:

EPA (Dur) 235
4 72

FIEL ATA

PLANT
DATE
SAMPLING LOCATION
SAMPLE TYPE
RUN NUMBER 2-B
OPERATOR
AMBIENT TEMPERATURE 95
BAROMETRIC PRESSURE
STATIC PRESSURE, (P_s)
FILTER NUMBER (s)

PROBE LENGTH AND TYPE
NOZZLE I.D. 0.27
ASSUMED MOISTURE, %
SAMPLE BOX NUMBER
METER BOX NUMBER
METER $\Delta H_@$
C FACTOR
PROBE HEATER SETTING
HEATER BOX SETTING
REFERENCE Δp

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY _____ MINUTES

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 DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\ in}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m\ out}$), °F	PUMP VACUUM, in. Hg	~~SAMPLE BOX~~ TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
		1[illegible]2									
	1400	170.2	[illegible]		1.3	[illegible]	[illegible]	[illegible]	9	[illegible]	
	Stop 1530	139.2									
		[illegible] ft^3									
	Start 1[illegible]	1[illegible]0.[illegible]									
			[illegible]		1.3		[illegible]	[illegible]			
	1[illegible]42	[illegible]									
	Beginning	Reverse [illegible]3									
		Turn [illegible]									
		Tot 7.[illegible]									
	End [illegible]										
		Tot [illegible]									

COMMENTS: Blower stopped approximately 1315

EPA (Dur) 235
4 72

LABORATORY DATA

SAMPLE RUN DATA

Work Performed by: Project No. Date of Work: 6/2/75

Title or Purpose: Run C-Sa-In-1 Continued from:

C-Sa-In-1

Saturator Outlet.

	EPA No.	Final Weight grams	Tare grams	Weight Change mg	Remarks
(1) Nozzle & Probe catch Evap. dish Number C-Sa-In-1 Total vol 78 ml		40.3773	40.3200	57.3	
(2) Impinger catch condensibles, Evap. dish Number____ Total vol____ml		none used	—		Moisture fraction
					Trichloroethane soluble fraction
(3) Acetone rinse of impingers Evap. dish Number____ Total vol____ml		not used	—		
(4) Glass wool filter Evap. dish Number C-Sa-In-4 Total vol 266 ml		37.0273	37.8816	145.7	1st wad catch + 2 combined
(5) Glass fiber filter No.____		0.4103	0.3776	32.7	

Recorded by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by:

Project No.

Date of Work:

Continued from:

Title or Purpose:

TCE Tech grade contains
since 1.02 mg/ml contaminating
Then 78 ml of TCE
contains

79.5 mg = 22.3 mg

if corrected
for blank in TCE, Technical –

8/4 –
combined.
soxhlet wash from Glass Fiber Filter,
with Glass wool prefilter
dish - C-Sa-IN 1/4
final wt = 38.0587 g
tare =

assume that 25% TCE Tech grade was used.
Then 66 ml x 1.02 mg = 67.8 mg
145.7 - 67.8 = 77.9 mg catch

Entered by:

Date:

Continued to:

Performances of this work observed by:

Date:

Disclosed to and understood by me:

Date:

Date:

Date:

Work Performed by: Project No. Date of Work: 6/12/75

Title or Purpose: C-Sa-Our-1 Continued from:

	EVAP DISH ~~EPA No.~~	Final Weight grams	Tare grams	Weight Change mg	Remarks
1) Nozzle & Probe catch Evap. dish Number C-Sa-Our 1 Total vol 84 ml		42.6468	42.5857	61.1	
2) Impinger catch condensibles, Evap. dish Number____ Total vol____ml					Moisture fractio
					Trichloroethane soluble fraction
3) Acetone rinse of impingers Evap. dish Number____ Total vol____ml					
4) Glass wool filter Evap. dish Number C-Sa-Our 1/4 Total vol 255 ml		42.9588	42.8322	126.6	1st wad catch heavy dep amber residue
5) Glass fiber filter No.____		0.3683	0.3618	6.5	

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: Continued from:

8/4
combined soxhlet extraction of glass fiber filter with glass wool pre-filter - in dish - C-Sa-Ovr. + residue wt - tare 42.9532 g

background
85.6 - 24.6 mg

assume 25% TCE-Tech used.
63.75 ml x 1.02 mg/ml = 65.03 mg - background due to solvent blank.
126.6 - 65.03 = 61.58 mg.

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work: 6/13/75

Title or Purpose: C-BS-In-1 C-BS-In-1 Continued from:

Blowing Still Inlet
Run #1

	EPA No.	Final Weight grams	Tare grams	Weight Change mg	Remarks
(1) Nozzle & Probe catch Evap. dish Number C-BS-In-1 Total vol 114 ml		45.3744 ~~45.5687~~	44.5417	0.8327 ~~1.0265~~	has 2 phase
(2) Impinger catch condensibles, 94 ml Evap. dish Number 320 Total vol ~~320~~ ml	C-BS-IP 1/2a	has water phase ~~40.7032~~ 40.0842 7/30	39.5350	~~2.1682~~ g 0.5492 g	Moisture fraction harvest
	C-BS-IN 1/2B	41.9308 ~~42.2587~~	7/30. one phase. 33.2437	8.6871 g ~~7.0150~~ g	Trichloroethane soluble fraction
(3) Acetone rinse of impingers Evap. dish Number Total vol 209 ml	C-BS-IN 1/3	44.4780	44.4602	17.8 mg	
(4) Glass wool filter Evap. dish Number Total vol 335 ml	C-BS-IN 1/4	44.3571 7/8	44.0944	262.7	1st wad catch heavy amber residue
(5) Glass fiber filter No.		0.3730	0.3665	6.5	

0.2262 2590
83 ml
Tech TCE.
ARCE

Note: On 7/27/75 - Begin Drying Impinger Catch over P_2O_5.

Reviewed by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: Continued from:

7/30/75

Background

114 ml x 1.02 mg/ml
background

116.3 mg = 716.4 mg

Have Jake run
I.R. trace (spectrum.)

CBS-in-1
CBS-in 1/2a
CBS-in 1/2b
CBS-in 1/3
CBS-in 1/4
+ Methylene Chloride into CBS-in-1

(Background)

= 85.4 mg
~~= 4.2 mg~~
~~55.0~~

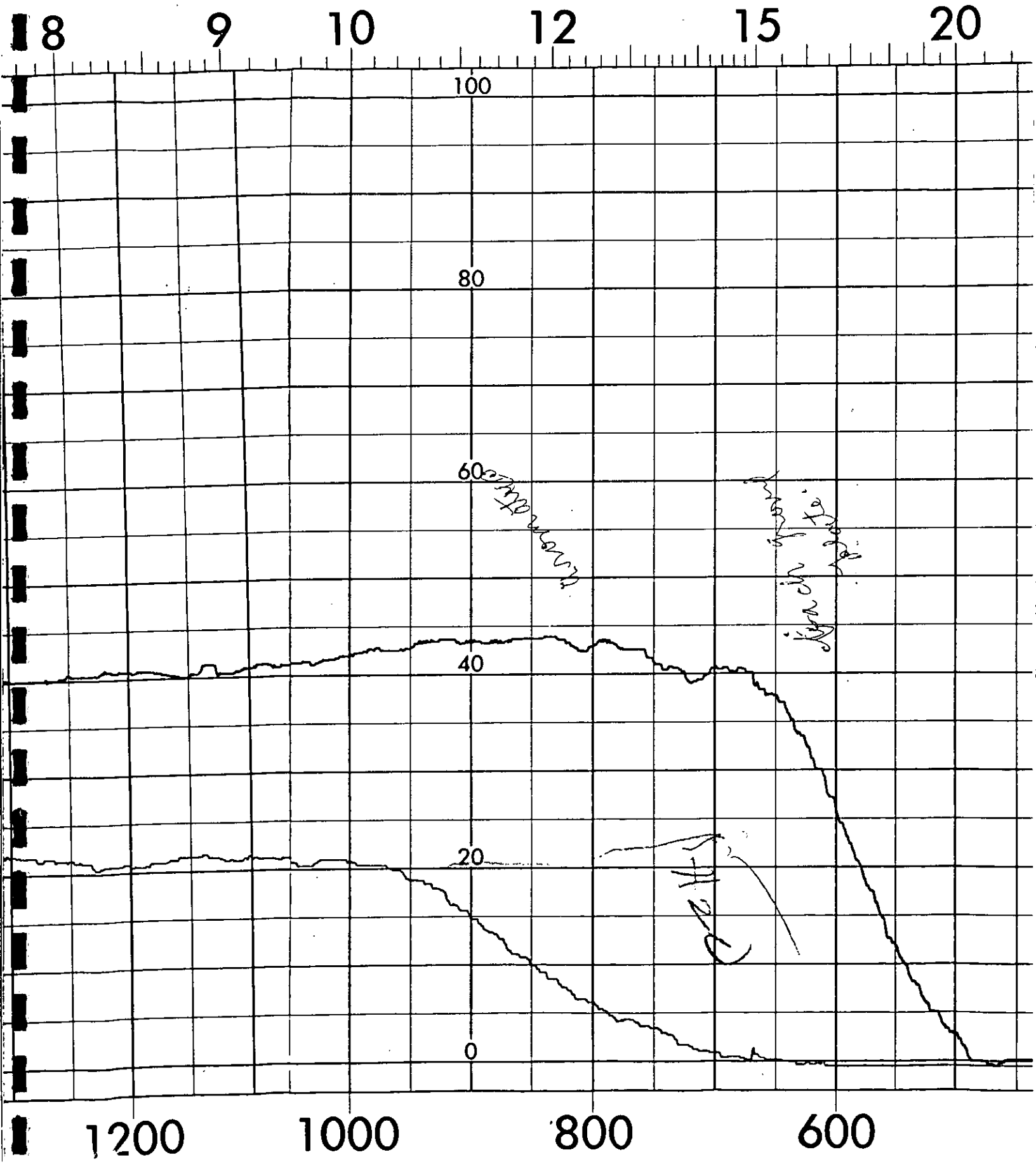
177.3 mg - corrected for Background -

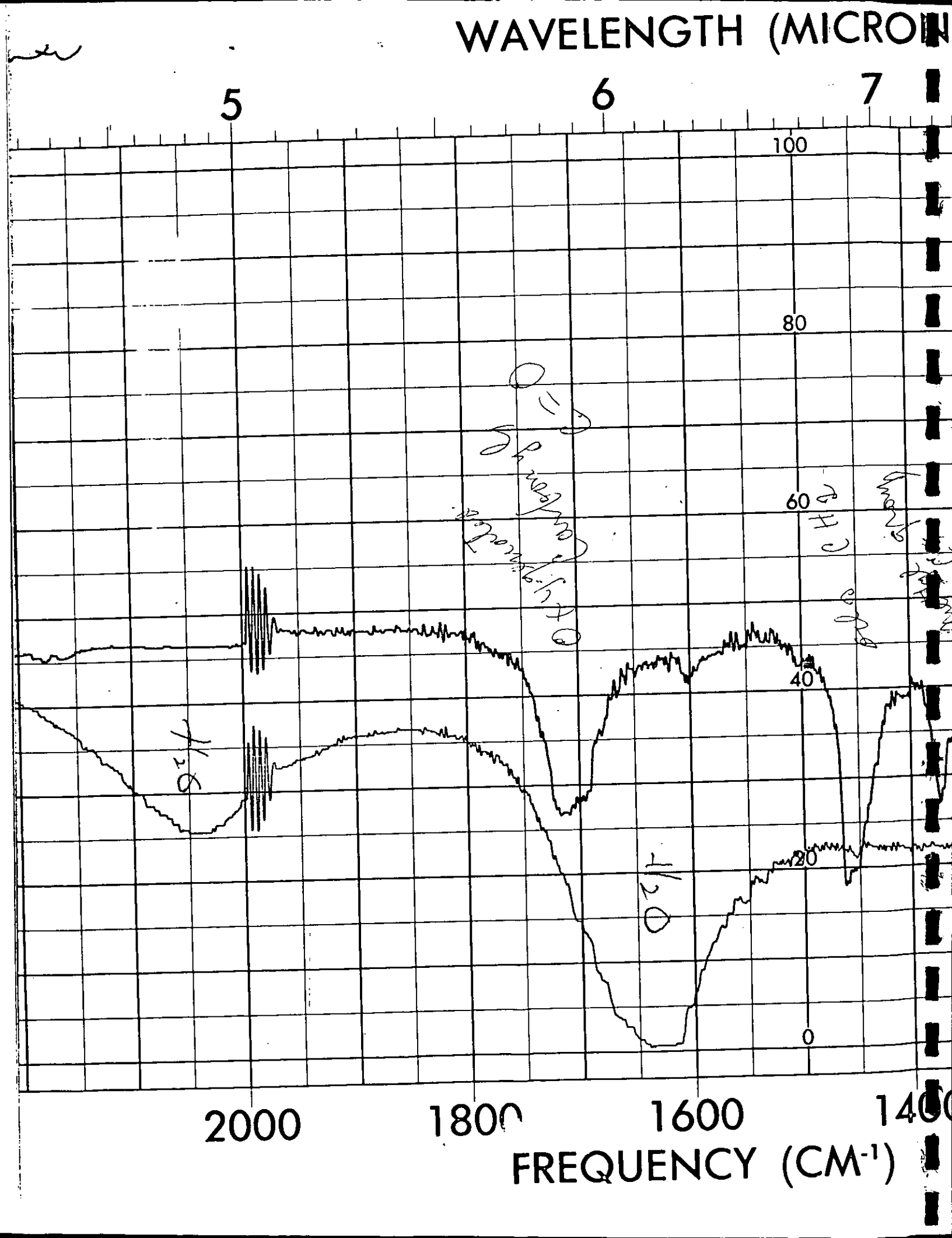
Entered by: Date: Continued to:

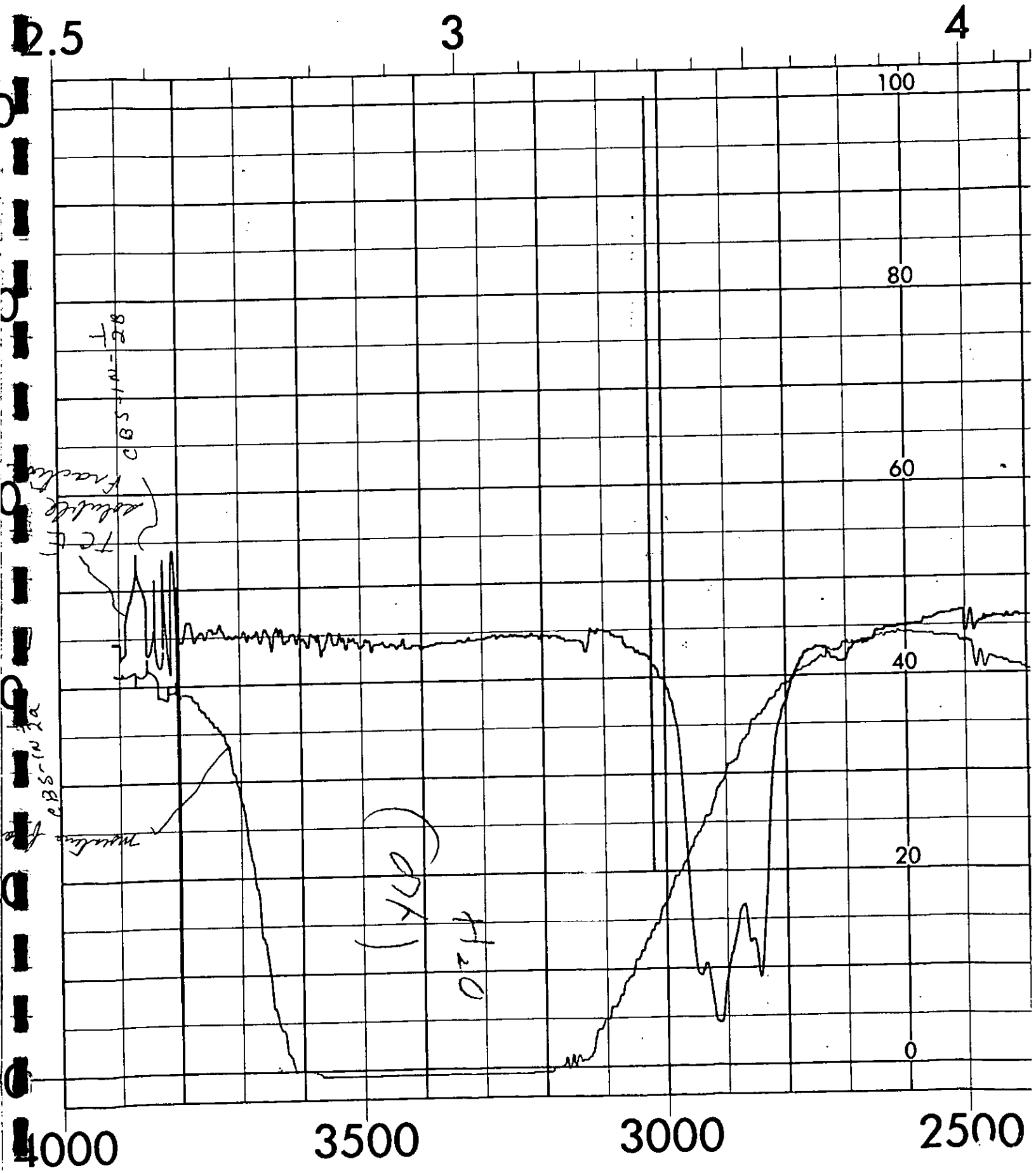
Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:


8
9
10
12
15
20
100
80
60
40
20
0
1200
1000
800
600


WAVELENGTH (MICRON
5
6
7
100
80
60
40
20
0
2000
1800
1600
1400
FREQUENCY (CM-1)


2.5
3
4
100
80
60
40
20
0
4000
3500
3000
2500
CBS-IN-28
CBS-IN-2a
TCE
methylene chloride
methanol
H2O

14

Work Performed by: Bayless / Miller Project No. 2872.2310 Date of Work:

Title or Purpose: C-BS-OUT-1 Continued from:

	Dish EPA No.	Final Weight grams	Tare grams	Weight Change mg	Remarks
(1) Nozzle & Probe catch Evap. dish Number C-BS-OUT 1 Total vol 124 ml		34.8934 7/8	34.7678	125.6	
(2) Impinger catch condensibles, Evap. dish Number ____ Total vol ____ ml	C-BS-OUT 1/20	42.4900 7/17	42.~~48~~ 47 74	12.6	Moisture fraction
	C-BS-OUT 1/20	37.5765 7/17	37.5683	8.2	Trichloroethane soluble fraction
(3) Acetone rinse of impingers Evap. dish Number ____ Total vol 220 ml		38.6542	38.6500	4.2	
			~~41.62~~		
(4) Glass wool filter Evap. dish Number ____ Total vol 275 ml	CBS-OUT 1/4	41.7675	41.6217	145.8	1st wad catch Heavy amber residue. backgrind
(5) Glass fiber filter No. ____		0.3573	0.3552	2.1	

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: Continued from:

7/30/73

C-BS-out 1/2B
C-BS-out 1/3
CBS-out 1/4
} + Methylene Chloride into CBS-out 1/2A

Final wt. 42.6191 g.

126.5 = -.9

% of 275 = 68.8 ml - Tech TCE used.

weight 70.1 mg = 75.7 mg "Particulate recovered.

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: C-BS-IN-2 Continued from:

	Dish ~~EPA~~ No.	Final Weight grams	Tare grams (7.3.75)	Weight Change mg	Remarks
(1) Nozzle & Probe catch Evap. dish Number___ Total vol. ___ ml	C-BS-IN 2/1	47.6460 ~~47.6444~~ 47.7014 (7/20/75)	46.7142 (8/5/75)	987.7	Weighed after dessicating
(2) Impinger catch condensibles, 2222 Evap. dish Number___ Total vol. 244 ml	C-BS-IN 2/2a	has been weighed 7/17 47.2178 (7/17) ~~47.4983~~ (7/20) - has moisture.	44.7278 (8/5/75)	2.7705	Moisture fraction weighed after dessicating
	C-BS-IN 2/2b	~~48.7350~~ (7/17) ~~48.1953~~ (7/20) - has 2 phases. 47.9830 - 8/5/75	44.7806	~~3.7544~~ 3.4147	Trichloroethane soluble fraction — has 2 phases — might be water
(3) Acetone rinse of impingers Evap. dish Number___ Total vol. 108 ml 112	ACETONE C-BS-IN 2/3a	7/8 38.5206	38.4466	74.0	
	from filter C-BS-IN 2/3b	METHYLENE CHLORIDE 7/8 41.7572	41.7543	2.9	
(4) Glass wool filter Evap. dish Number___ Total vol. 265 ml	C-BS-IN 2/4	43.4047 (7/8)	43.3341	70.6	1st wad catch + 2d has amber resin
(5) Glass fiber filter No.___		0.3558	0.3561	-0.3	
"0" Bl. (empty dish) p.16		41.2620	41.2623		

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: Continued from:

correction factor
background in Technetium

226.4 = 761.3 mg

assume 25% in Technetium
bacon

66.25 ml × 1.02 mg/ml = 67.6 mg = 3.0 mg.

tered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: C-BS-OUT-2 Continued from:

	DISH EPA No.	Final Weight grams	Tare grams	Weight Change mg	Remarks
(1) Nozzle & Probe catch Evap. dish Number____ Total vol 134 ml	C-BS-OUT 2/1	41.5574	41.4541	103.3	amber residue
(2) Impinger catch condensibles, Evap. dish Number____ Total vol 225 ml (31 ml)	C-BS-OUT 2/2a	44.7797	44.7716	~~44~~ 4.1	Moisture fraction
	C-BS-OUT 2/2b	46.5386	46.5337	4.9	Trichloroethane soluble fraction
(3) Acetone rinse of impingers Evap. dish Number____ Total vol 192 ml	BS-OUT 2/3	49.7957	49.7939	1.8	
(4) Glass wool filter Evap. dish Number____ Total vol 240 ml	BS-OUT 2/4	48.9945	48.8848	109.7	1st wad catch amber residue similar to CBS-OUT-2/1
(5) Glass fiber filter No.____		0.3549	0.3525	2.4	
BL-1	TRICHLOROETHYLENE TECH. 65 ml 100 ml	40.9221	40.8557	66.4	1.02 mg/ml heavy amber residues
BL-2	TCE A.R. 100 200 ml	47.5889	47.5875	1.4	14.0 ug/ml

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Project No. Date of Work:

Title or Purpose: Continued from:

6.7 mg background = -33.4 mg

Assume 25% TCC in technical grade

60.0 ml X 1.02 mg/ml = 61.2 mg

109.7 mg - 61.2 mg = 48.5 mg

Entered by: Date: Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

Work Performed by: Costant / Baytos Project No. J2872-B10 Date of Work: August 4, 6, 7

Title or Purpose: Thermal Gravimeter Analyses — (TGA) Continued from:

"TGA"'s were run on the following ~~composite~~ composited samples — over a temperature range of 70F to 300F, programmed at 2½ °F/min —

Sample.

C-Sa IN-1/4 } glass wool wadding + Soxhlet extraction of filter —

C-Sa OUT-1/4 } glass wool wadding + Soxhlet extraction of filter —

C-Sa-IN-1/1 — Probe wash with TGE, Tech-grade —

Blank — Residue resulting upon evaporation from Tri chloro-ethane, technical grade —

C-BS-IN-1/4 — combined contents of 5 dishes (no filter extract) see pg 13

C-BS-OUT 1/4 — combined contents of 3 dishes — (no filter extract) see p. 15

data sheets, graphs, and work sheets kept in minutefolder in project.

Entered by: Wm C Baytos Date: August 7, 1975 Continued to:

Performances of this work observed by: Disclosed to and understood by me:

Date: Date:

Date: Date:

APPENDIX B

EPA DRAFT METHOD FOR DETERMINATION OF PARTICULATE AND TOTAL GASEOUS HYDROCARBON EMISSIONS FROM THE ASPHALT ROOFING INDUSTRY

DRAFT
DO NOT QUOTE OR CITE

5 MAY 1976

APPENDIX B

EPA DRAFT METHOD FOR DETERMINATION OF PARTICULATE AND TOTAL GASEOUS HYDROCARBON EMISSIONS FROM THE ASPHALT ROOFING INDUSTRY

1. Principle and Applicability

1.1 Principle. Particulate matter and gaseous hydrocarbons are withdrawn isokinetically from the source and collected on glass fiber filter maintained at temperatures of no greater than 50°C (122°F). The particulate mass is determined gravimetrically after removal of uncombined water and solvent. Hydrocarbons are determined from a small side stream extracted after the filter and analyzed by a flame ionization detector (FID) analyzer.

1.2 Applicability. This method is applicable for the determination of particulate and gaseous hydrocarbon emissions from the asphalt roofing industry.

2. Apparatus

2.1 Sampling train. A schematic of the sampling train used in this method is shown in Figure B-1. Commercial models of the particulate train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure B-1, see the following subsections. Commercial FID analyzer systems are also available.

The operating and maintenance procedures for the particulate sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance

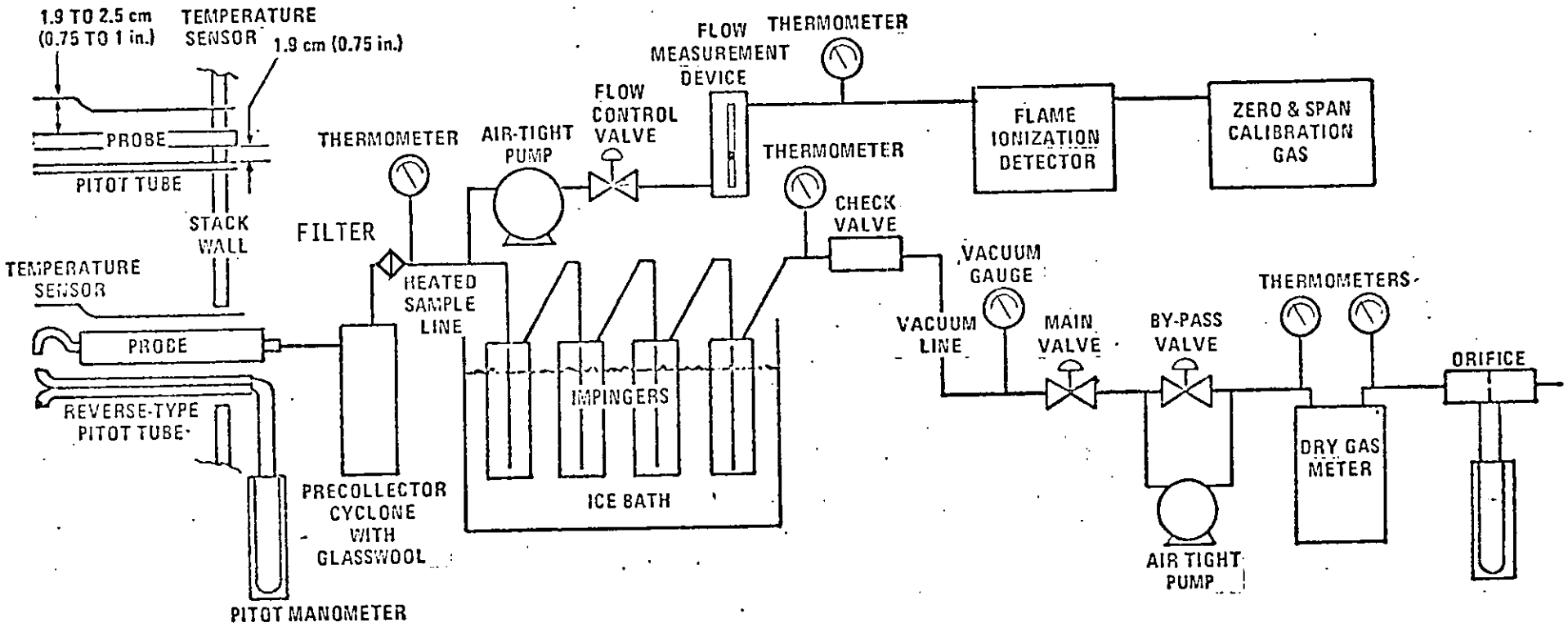


Figure 20.1. Particulate and gaseous hydrocarbon sampling train.

procedures outlined in it, unless otherwise specified herein. Manufacturers' instructions should be followed for the gaseous hydrocarbon measurement system, unless otherwise specified herein.

2.1.1 Probe nozzle--Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be $\leq 30^{\circ}$ and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise approved by the Administrator. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used subject to approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (1/8 in.) up to 1.27 cm (1/2 in.) inside diameter (ID) nozzles (or larger if higher volume sampling trains are used) in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

2.1.2 Probe--Borosilicate or quartz glass tubing with a heating or cooling system capable of maintaining a gas temperature at the exit end of the filter during sampling of no greater than 50°C (122°F). In most cases, ambient cooling of the probe is sufficient to maintain the desired temperature. After incinerators, it may be necessary to use water cooled probes. In this case, stainless steel (316) probes may be used.

2.1.3 Pitot tube--Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the largest sized nozzle shall be used to set the free space.

The pitot tube must also meet the criteria specified in Method 2 and calibrated according to the procedure in the calibration section of that method.

2.1.4 Differential pressure gauge--Inclined manometer capable of measuring velocity head to within 10% of the minimum measured value or ± 0.013 mm (0.0005 in.), whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.5 Particulate and moisture cyclone precollector--Optional for use when stack gas oil and moisture concentrations

are high and would cause liquids to seep through the glass filter mat. Above 10% moisture content water vapor will condense and saturate the filter mat to prevent liquid carry-over, a cyclone collector is recommended (other devices may be used with approval from the Administrator). The collector shall be constructed of borosilicate glass. The top section of the cyclone (see Figure B-1) contains a known weight of glass wool to trap the condensed oil and/or water. A 125 ml, or larger, Erlenmyer collecting flask of borosilicate glass is connected to the cyclone to hold any condensate.

2.1.6 Filter holder--Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

2.1.7 Filter temperature control system--Any system capable of maintaining a temperature of gas exiting the filter holder during sampling of no greater than 50°C (122°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed (with sensor tip within 2.5 cm or 1 in. of the filter frit) such that the gas temperature after the filter can be regulated and monitored during sampling.

2.1.8 Total gaseous hydrocarbon measurement system--To remove and analyze a portion of the filtered gas for total hydrocarbons (see Figure B-1).

2.1.8.1 Heated Sample line--FEP fluorocarbon tubing heated

to maintain gas temperature entering the FID analyzer to slightly above filter exit temperature. The tubing length should be kept to a minimum to reduce transport delay time.

2.1.8.2 Flame ionization detector analyzer--Commercially available systems with a gas pump and flow regulation device for conveying a known amount of sample gas to the detector cell. The operating temperature is about 50°C (122°F); therefore, either ambient or heated cell FID instruments may be used. Operating instructions and calibration procedures are given in later sections.

2.1.8.3 Data Recorder--That portion of the measurement system that provides a permanent record of the output signal in terms of concentration units.

2.1.9 Condenser--Any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser (1) by monitoring the temperature and pressure at the exit of the condenser and using Dalton's law or (2) by passing the sample gas stream through a tared silica gel trap with exit gases kept below 20°C (68°F) and below 380 mm Hg (15 in. Hg) vacuum and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices.

Unless otherwise specified by the Administrator, flexible vacuum lines may be used to connect the filter holder to the condenser.

2.1.10 Metering system--Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications in section 2 of this method are met. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.11 Barometer--Mercury, aneroid, or other barometers 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 weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.12 Gas density determination equipment--Temperature and pressure gauges and gas analyzer as described in Methods 2 and 3.

2.1.13 Temperature and pressure gauges--If Dalton's law is used, to monitor temperature and pressure at condenser outlet. The temperature gauge shall have an accuracy of 1°C (2°F). The pressure gauge shall be capable of measuring pressure to within 2.5 mm Hg (0.1 in. Hg). If silica gel is used in the condenser system the temperature and pressure must be measured before the silica gel component.

2.2 Sample recovery.

2.2.1 Probe liner and probe nozzle brushes--Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, nylon, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass wash bottles--Two.

2.2.3 Glass sample storage containers--Chemically resistant, borosilicate glass bottles, for 1,1,1-trichloroethane washes, 500 ml or 1,000 ml. Screw cap closures shall be teflon rubber-backed liners or of such construction so as to be leak free and prevent chemical attack from the 1,1,1-trichloroethane. (Narrow mouth glass bottles have been found to be less prone to leakage.) Other types of containers must be approved by the Administrator.

2.2.4 Petri dishes--For filter samples, glass, unless otherwise specified by the Administrator.

2.2.5 Graduated cylinder and/or balance--To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most labora-

balances are capable of weighing to the nearest 0.5 g or less. Any of these balances are suitable for use here and in section 2.3.4.

2.2.6 Plastic storage containers--Air tight containers to store silica gel.

2.2.7 Funnel and rubber policeman--To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance--To measure to within 0.1 mg.

2.3.4 Balance--To measure to within 0.5 g.

2.3.5 Beakers--250 ml.

2.3.6 Hygrometer--To measure the relative humidity of the laboratory environment.

2.3.7 Temperature gauge--To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling.

3.1.1 Filters--Glass fiber filters, without organic binder exhibiting at least 99.95% efficiency ($\leq$ 0.05% penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program is sufficient for this purpose.

3.1.2 Precollector glass wool--Glass fibers, No. 7220, Pyrex brand, or equivalent.

3.1.3 Silica gel--Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

3 .1.4 Water.

3.1.5 Crushed ice.

3.1.6 Zero Gas--A grade of compressed zero air containing less than 1 ppm hydrocarbon as methane.

3.1.7 Calibration Gas--A compressed gas mixture of a known concentration of methane or propane in air. Nominal concentrations of 50% and 90% of the instrument full scale range are required. The higher concentration gas mixture is used to set and check the instrument span and is referred to as the span gas.

3.2 Sample recovery.

3.2.1 1,1,1-Trichloroethane (TCE)--Reagent grade, $\leq$ 0.001% residue, in glass bottles. TCE blanks shall be run prior to field use and only TCE with low blank values ($\leq$ 0.001%) shall be used.

3.3 Analysis.

3.3.1 1,1,1-Trichloroethane--Same as 3.2.1.

3.3.2 Desiccant--Anhydrous calcium sulfate, indicating type.

4. Procedure

4.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

4.1.1 Pretest preparation--All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label a filter of proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping container and keep the filter in this container at all times except during sampling and weighing.

Desiccate the filters and glass wool at 20 $\pm$ 5.6°C (68 + 10°F) and ambient pressure for at least 24 hours and weigh at 6 or more hour intervals to a constant weight, i.e., $\leq$ 0.5 mg change from previous weighing, and record results to the nearest 0.1 mg. During each weighing the filter or glass wool must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50%. Clean and preweigh the precollector (cylcone) to the nearest 0.1 mg. Install a preweighed amount of glass wool and seal the openings of precollector.

4.1.2 Preliminary determinations--Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test. Note: A portion of flow will go to the FID analyzer. This flow rate must be included when establishing isokinetic sampling rates. See section 6.3.1 for this calculation.

Select a nozzle size based on the range of velocity heads such 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 size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10%, or as specified by the Administrator.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or some greater time interval as specified by the Administrator and the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that 1/2 or an integral number of minutes be sampled at each point in order to avoid timekeeping errors.

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.

4.1.3 Preparation of collection train--During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200-300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container to the nearest 0.5 g. Place the container in a clean place for later use in the sample recovery.

Using a tweezer or clean disposable surgical gloves, place the labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed..

Assemble the filter holder and attach the precollector cyclone ahead of the filter when the precollector is to be used.

When glass liners are used, install selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C

(500°F) or an asbestos string gasket when temperatures are higher. The Viton A O-ring and asbestos string gasket are installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners or water cooled probes are used, install the nozzle as above or by a leak free direct mechanical connection. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Set up the train as in Figure B-1, using no silicone grease on ground glass joints ahead of the filter. Silicone grease is soluble in TCE, therefore, clean glassware with only water seals shall be used.

Place crushed ice around the impingers.

4.1.4 Leak check procedure--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 380 mm Hg (15 in. Hg) vacuum. (Note: A lower vacuum may be used provided that it is not exceeded during the test.) If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train as above by first plugging the inlet to the

the filter holder. Then connect the probe to the train and leak check at about 25 mm Hg (1 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.00057 m^3/min. (0.02 cfm), whichever is less, is unacceptable. The gaseous hydrocarbon system may be shut off for the leak check. However, all fittings on the analyzer system should be checked to insure they are properly tightened.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g., for silica gel or filter

changes during the test, prior to each test run, and at the completion of each test run. Close the flow control valve of the gaseous hydrocarbon measurement system. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes at the highest vacuum reading drawn during that sampling traverse.

4.1.5 Particulate train operation--During the sampling run, isokinetic sampling rate to within 10%, or as specified by the Administrator, of true isokinetic and the temperature of the gas exiting the filter of no greater than 50°C (122°F) shall be maintained.

For each run, record the data required on the example data sheet shown in Figure B-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, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity head readings) necessitate addtional adjustments in flow rate. Level and zero the manometer.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. Allow time for

hydrocarbon analyzer operation to stabilize and for the heated hydrocarbon sample line to reach the required temperature. To begin sampling, remove the nozzle cap, verify that the filter and probe are at the proper temperature, and 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 are available for sampling trains using type S pitot tubes with 0.85 $\pm$ 0.02 coefficient and when sampling in air or a stack gas with equivalent density (molecular weight equal to 29 + 4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomograph unless appropriate steps (see Reference 7.7) are taken to compensate for the deviations.

After starting and adjusting the flow of the sampler pump, start the flow of sample gas through the hydrocarbon analyzer and allow a constant regulated amount of gas to go to the hydrocarbon analyzer. The flow rate of the hydrocarbon system should be preset to no more than 1.0 liters per minute. A correction for the flow of gas not passing through the sample train meter should be made. To permit accurate isokinetic flow rate settings the nomograph should be corrected by converting the flow rate of gas through the hydrocarbon analyzer to a psuedo moisture content and

add this amount to the actual moisture content when setting the nomograph reference points, see section 6.3.1.

When the stack is under significant negative stack pressure take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by 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 portholes to minimize chance of extracting deposited material. Note the hydrocarbon value for each traverse point, both on the analyzer chart recorder and on a form.

During the test run, make periodic adjustments to keep the temperature of gas exiting the filter holder at the proper temperature and add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the condenser/silica gel outlet to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If a yellow-brown color forms on the filter or the condensed moisture begins to fill the precollector, the precollector and the filter shall be replaced in the midst of a sample run.

It is recommended that another complete precollector and filter assembly be used rather than attempting to change the filter itself. After the new assembly is installed conduct a leak check. The particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except for precollector, filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The results shall be the total of all sampling train catches.

At the end of the sample run shut off the flow of sample gas to the hydrocarbon system, turn off the sample train pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check at a vacuum equal to or greater than the maximum reached during sampling. Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 *Hydrocarbon analyzer operation.*

4.2.1 Install the hydrocarbon analyzer system as close as possible to the probe and filter apparatus. Heat the fluorocarbon sample line to above the filter temperature to prevent condensation

of hydrocarbons. Note: Due to the design of most FID analyzers, it will be necessary to protect the instrument from the ambient environment (rain, dust, extreme heat or cold, etc.). Check for stable electrical power; voltage fluctuations can cause instrument drift in some analyzers. Calibrate the analyzer using a span concentration, zero air and one other upscale concentration of methane in air to check linearity of system. Refer to calibration section 5.2 for details of this procedure.

After the particulate sampling train is operating at the first traverse point, the gaseous hydrocarbon sample pump shall be started and the flow regulated so that the analyzer functions properly. The average hydrocarbon analyzer reading shall be recorded for each traverse point. A strip chart or other data recorded can also be used to monitor the analyzer output.

At the conclusion of the test shut off the hydrocarbon system before stopping the particulate sampling train pump. The hydrocarbon analyzer shall be recalibrated after the test so that zero and calibration gas drift can be determined.

4.2.2 "Zero drift" is the change in analyzer output during the performance of the test when the input to the measurement system is a zero grade air (zero gas). The maximum zero drift for measurement systems used in this method is $\pm$ 2% of the specified instrument span. The zero drift calculation is to be made for each test. It is determined from the zero concentration values measured at the start and finish of each test.

Calculate the difference between these readings expressed in concentration units. Record the data and report zero drift as a percentage of the instrument span, using a form similar to Figure B-3.

4.2.3 "Span drift" is the change in analyzer output during the performance of the test when the input to the measurement system is span gas. The maximum span drift for measurement systems used in this method is ± 2% of the specified instrument span. The span drift calculation is to be made for each gas for each test. It is determined by measuring the response of the instrument when span gas mixture is introduced into the system at the start and finish of each test. Calculate the difference between these readings expressed in concentration units. These values should be corrected for the corresponding zero drift during that period. The correction for zero drift is explained in Figure B-3 Record the data and report span drift as a percentage of the instrument span, using a form similar to Figure B-3.

4.2.4 "Response time" is defined as the time interval from a step change in pollutant concentration at the system inlet to the time at which 95% of the final value is reached as displayed on output recording system. The maximum response time for a hydrocarbon analyzer meeting the requirements of this method

is one minute. To determine response time, first introduce zero gas to the system until the reading is stable and then introduce the span gas to the system. The time to the 95% final value is recorded as the upscale response time. Zero gas is then introduced and the time to 95% final response is recorded as the downscale response time. This procedure is to be followed three times, the readings averaged and the upscale or downscale response time, whichever is greater, shall be reported as the "response time." Record this data on a form similar to Figure B-4. This test shall be conducted prior to the initial test and repeated if changes are made to the measurement system.

4.3 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external oil particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter.

Before moving the sample train to the cleanup site, disconnect the hydrocarbon analyzer system, remove the probe from the sample train, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers

or condenser. Cap off the filter holder outlet and impinger inlet. Either Teflon or ground glass stoppers may be used to close these openings. If plastic or rubber caps are used, they must not react with TCE.

Transfer the probe and filter impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the TCE used for cleanup as a blank. Place about 200 ml of this TCE taken directly from the wash bottle being used in a glass sample container labeled "TCE blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Quantitatively remove any particulate matter and/or filter which adheres to the filter holder by carefully using a dry nylon bristle brush and/or a sharp-edged blade and place into this container. Seal the container.

Container No. 2. Remove the collector flask from the cyclone using glass or other nonreactive caps, seal ends of cyclone and store for shipment to the laboratory.

Container No. 3. Taking care to see that material 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, cyclone collector flask and front half of the filter holder by washing these components with TCE and placing the wash into a glass container in the following manner.

Carefully remove the probe nozzle and clean the inside surface with a nylon bristle brush. Brush until TCE rinse shows no visible particles, or discoloration after which make a final rinse of the inside surface with TCE.

Brush and rinse with TCE the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with TCE by tilting the probe and squirting TCE into its upper end, while rotating the probe so that all inside surfaces will be rinsed with TCE. Let the TCE drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washes to the container. Follow the TCE rinse with a probe brush. Hold the probe in an inclined position, squirt TCE into the upper end as the probe brush is being pushed with a twisting action through the probe, hold a sample container underneath the lower end of the probe,

and catch any TCE and particulate matter which 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 TCE or 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 since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with TCE and quantitatively collect these washings in the sample container. After the brushing make a final acetone (reagent grade) rinse of the probe as described above. This rinse is to promote drying of the equipment and is not retained for analysis. It is recommended that two people be used to clean the probe to minimize losing the sample. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the cyclone collection flask and the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with TCE. Rinse each surface three times or more if needed to remove visible particulate and condensed moisture. Make a final rinse of the brush, flask and filter holder. After all TCE washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that TCE 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 container to clearly identify its contents. To promote the drying of the glassware a final rinse of the components with acetone is recommended. This rinse should not be saved for analysis.

Container No. 4. Note color of 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 the 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 to the walls and are difficult to remove. Since 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, follow the procedure under analysis.

Impinger water. Treat the impingers or condenser as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ± 1 ml by using a graduated cylinder or, if available, to within ± 0.5 mg by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

4.4 Analysis. Record the data required on the example sheet shown in Figure B-5. Handle each sample container as follows:

(For the purpose of this section 4.4, the term "constant weight" means a difference of no more than 10% between two weighings 24 hours apart. The "final weight" to be used in calculations is the average of these two values.)

Container No 1. Leave in shipping container or transfer the filter from the sample container to a tared glass weighing dish and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Rinse the filter container with TCE and combine this rinse with the contents of Container 3. Weigh to a constant weight and report the results to the nearest 0.1 mg.

Container No. 2. Clean the outside of the cyclone, remove caps, and desiccate for 24 hours or until any condensed water has evaporated. Weigh the cyclone with the oil and glass wool plug inplace. Subtract the weight of the cyclone and the tare weight of glass wool. Clean out the oil and glass wool with TCE into a tared weighing dish. Desiccate the cyclone for 24 hours and reweigh the cyclone. if the final weight of the clean cyclone is within 10 mg of the initial weight of the clean cyclone, the calculated oil weight will be considered valid. If the weight difference is greater, then the cyclone will be assumed damaged and the oil must be extracted from the glass wool and this oil solution added to Container 3. Note: This procedure is subject to error if the glass wool fibers are not kept out of the oil solution.

Container No. 3. Note level of liquid in container and confirm on analysis sheet whether or not leakage occurred during transport. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. If the volume of condensed water appears larger than 5 ml it will be necessary to separate the oil-TCE fraction from the water. This should be done with a separatory funnel. Additional rinses of the water fraction shall be made with 25 ml portions of TCE until, by visual observation, the TCE does not remove, any additional organic material. The remaining water fraction shall be evaporated to dryness at 200°C, dessicated for 25 hours and weighed to the nearest 0.1 mg. Treat the TCE fraction with the following procedure. Transfer the TCE and oil to a tared 250 ml beaker, and evaporate at ambient temperature and pressure. The terms dryness and constant weight are not appropriate for liquid oil samples which continue to loose weight through evaporation. The volume of TCE required to remove the oil from the sample train should be estimated. When it appears that its volume has been evaporated the sample should be desiccated and weighed at 24 hour intervals to obtain a "constant weight." The "final weight" is the sum of the evaporated particulate weight fractions for TCE-oil and water. Report the results to the nearest 0.1 mg.

Container No. 4. Weigh the spent silica gel to the nearest 0.5 g using a balance. This step may be conducted in the field.

"TCE Blank" Container. Measure TCE in this container either volumetrically or gravimetrically. Transer the TCE to a tared 250 ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a "constant weight." Report the results to the nearest 0.1 mg.

5. Calibration

5.1 Particulate Sampling Equipment. Maintain a laboratory log of all calibrations.

5.1.1 Probe nozzle--Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time 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.

5.1.2 Pitot tube. The pitot tube shall be calibrated according to the procedure outlined in Method 2.

5.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When a diaphragm pump is used, assure that there is no leak.

5.1.4 Probe heater calibration. The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.1.5 Temperature gauges. Calibrate dial and liquid filled bulb thermometers and thermocouple-potentiometer systems against mercury-in-glass thermometers. Ice bath and boiling water (corrected for barometric pressure) are acceptable reference points. For other devices, check with the Administrator.

5.2 Calibration of Gaseous Hydrocarbon System. Prior to the conduct of the test, the hydrocarbon measurement system shall be calibrated according to the procedures described in this paragraph. The manufacturer's operation and calibration instructions are also to be followed as required.

5.2.1 The measurement system shall be put into operation and allowed to warm up until stable conditions are achieved. Zero gas, span gas, and a mid-scale gas mixture corresponding to approximately 50% of span shall be introduced into the analyzer. The response to these gases shall be used to establish a calibration curve or verify the manufacturer's calibration curve. The data obtained in these procedures shall be recorded on a form similar to Figure B-6. If the manufacturer's calibration curve, or the expected response curve cannot be attained at the mid-scale point with an accuracy of less than 2% of full scale, the calibration shall be considered invalid and corrective measures shall be taken. The calibration procedure shall be repeated using only zero gas and span gas at the conclusion of test to calculate the instrument zero and span drift.

5.2.2 Hydrocarbon (HC) calibration gas mixtures shall be certified accurate by the gas manufacturer to within $\pm$ 2% of

the indicated concentration. The calibration analysis shall be certified traceable to a National Bureau of Standards certified gas concentration.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature

A_n = Cross sectional area of nozzle, m^2 (ft^2)

A_s = Cross sectional area of stack, m^2 (ft^2)

B_{ws} = Water vapor in the gas stream, proportion by volume

B_{wt} = water vapor in the sample gas stream, proportion by volume

C_a = TCE blank residue concentration, mg/g

c_s = Concentration of particulate matter in stack gas, corrected to standard conditions, g/dscm (g/dscf)

I = Percent of isokinetic sampling

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)

m_a = Mass of residue of TCE after evaporation, mg.

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg)

P_{mr} = Pollutant mass rate, g/hr (lb/hr)

ppm_v = Parts per million by volume

P_s = Absolute stack gas pressure, mm Hg (in. Hg)

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)

Q_h = Flow rate through hydrocarbon analyzer, cm/min (cfm)

R = Ideal gas constant, 0.06236 mm $Hgm^3/^oK$-mole (21.83 in. Hg-$ft^3/^oR$-lb-mole)

T_m = Absolute average dry gas meter temperature (see Figure B-2), ^oK (^oR)

T_h = Absolute temperature of hydrocarbon sample at flow meter, ^oK (^oR)

T_s = Absolute average stack gas temperature (see Figure B-2), ^oK (^oR).

T_{std} = Standard absolute temperature, 293^oK (528^oR)

V_a = Volume of TCE, blank ml

V_{aw} = Volume of TCE used in wash, ml

V_h = Volume of hydrocarbon sample gas, cm (cf)

V_{lc} = Total volume of liquid collected in impingers and silca gel (see Figure B-5, ml.)

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf)

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

V_{pc} = Volume of water collected in particulate catch, ml

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).

$V_{wt(std)}$ = Volume of water vapor in sample gas corrected to standard conditions, scm, (scf)

v_s = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained by this method, m/sec (ft/sec)

W_a = Weight of residue in TCE wash, mg

ΔH = Average pressure differential across the orifice meter (see Figure B-2), mm H_2O (in. H_2O)

ρ_a = Density of TCE, mg/ml (see label on bottle)

ρ_w = Density of water, g/ml (0.00220 lb/ml)

θ = Total sampling time, min.

13.6 = Specific gravity of mercury

60 = sec/min

100 = Conversion to percent

6.2 Particulate Calculations.

6.2.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure B-2).

6.2.2 Hydrocarbon Analyzer Sample Volume

$$V_h = (Q_h)\ (\Theta)\ \left(\frac{T_{std}}{T_h}\right)\ \left(\frac{P_{bar}}{P_{std}}\right)\ (1-B_{ws})$$

$$= Z\ (Q_h)(\Theta)\ \left(\frac{P_{bar}}{T_h}\right)\ (1-B_{ws})$$ *Equation B-1

where:

Z = .386 metric units

= 17.65 english units

*Note: Equation B-1. The term B_{ws} must be estimated. Use Equation B-4-1 for B_{ws} and assume V_h to be dry gas.

6.2.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation B-1.

$$V_{m(std)} = V_m \left(\frac{T_{std}}{T_m}\right) \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right) + V_h$$

$$= K\ V_m \left(\frac{P_{bar} + \Delta H/13.6}{T_m}\right) + V_h$$ Equation B-2

where:

K = 0,3856 ^{o}K/mm Hg for metric units

= 17.65 ^{o}R/in. Hg for English units

6.2.4 Volume of water vapor.

$$V_{m(std)} = V_{lv} \left(\frac{P_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K\ V_{lc}$$

Equation B-3-1

$$V_{wt(std)} = (V_{lc} + V_{fh}) \left(\frac{\rho_w}{M_w}\right) RT \quad \text{*Equation B-3-1}$$

where:

$K = 0.00134\ m^3/ml$ for metric units

$= 0.0472\ ft^3/ml$ for English units

*Note: Equation B-3-2 for use when there is water condensation in the particulate catch.

6.2.5 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + (V_{w(std)} - V_n)} \quad \text{Equation B-4-1}$$

$$B_{wt} = \frac{V_{wt(std)}}{V_{m(std)} + V_{wt}} \quad \text{*Equation B-4-2}$$

6.2.6 TCE blank concentration.

$$C_a = \frac{m_a}{V_a \rho_a} \quad \text{Equation B-5}$$

6.2.7 TCE wash blank.

$$W_a = (C_a)(V_{aw})(\rho_a) \quad \text{Equation B-6}$$

6.2.8 Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the TCE blank (see Figure B-5).

6.2.9 Particulate concentration.

$$c_s = (0.001\ g/mg)\ (m_n/V_{m(std)}) \quad \text{Equation B-7}$$

*Note: Equation B-4-2 for use when there is water condensation in the particulate catch.

6.2.10 Conversion factors:

From	To	Multiply by
scf	m^3	0.0283
g/ft^3	gr/ft^3	15.4
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

6.2.11 Isokinetic variation.

6.2.1.1 Calculations from raw data.

$$I = \frac{100 \; T_s \; [K \; V_{lc} + (V_m/T_m) \; (P_{bar} + \Delta H/13.6)]}{60 \; \Theta \; v_s \; P_s \; A_n}$$

Equation B-8

where:

$K = 0.00346$ mm Hg-m^3/ml-°K for metric units

$= 0.00267$ in. Hg-ft^3/ml-°R for English units

6.2.11.2 Calculations from intermediate values.

$$I = \frac{T_s \; V_{m(std)} \; P_{std} \; 100}{T_{std} \; v_s \; \Theta \; A_n \; P_s \; 60 \; (1-B_w)}$$

$$= K \frac{T_s \; V_{m(std)}}{P_s \; v_s \; A_n \; \Theta \; (1-B_w)}$$

Equation B-9

where:

$K = 4.323$ for metric units

$= 0.0944$ for English units

$B_w = B_{ws}$ or B_{wt}, as appropriate

6.2.11.3 Acceptable results. If 90% $\leq$ I $\leq$ 110%, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 to make judgments. Otherwise, reject the results and repeat the test.

6.3 Gaseous Hydrocarbon Calculations.

6.3.1 Correction to set isokinetic flow is necessary because the hydrocarbon sample gas does not pass through the orifice meter in the sampling train. The orifice meter volume must be compensated for or the isokinetic sampling velocity will not be correct. The simplest approach is to determine the volume to be sampled by the hydrocarbon system (V_h) and to add this volume to the volume of moisture to be collected by the sampling train. The hydrocarbon sample gas, similar to moisture content, does not pass through the orifice meter. The resulting pseudo water volume is used in the calculation to determine the isokinetic sampling rate. If the nomograph is used, the pseudo moisture content used in C factor nomograph (Figure 22 of APTD-0576) can be calculated. This estimated moisutre content can be improved upon after an initial test.

6.3.2 Average the ppm by volume readings of gaseous hydrocarbons for each traverse point and calculate the average concentration. The stack area and the velocity data from Method 2 can be used to calculate the mass emission rate of hydrocarbons as methane by using Equation B-8.

$$pmr = 0.67 \times 10^6 \ (\overline{ppm}_v) \ (v_s) \ (A_s) \qquad \text{Equation B-10}$$

7. References

7.1 Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

7.2 Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

7.3 Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD0576.

7.4 Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 1419, 1970.

7.5 Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67119, 1967.

7.6 Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

7.7 Shigehara, R. T., Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights, Stack Sampling News 2:411, Oct. 1974.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH@ _____
F FACTOR _____
PITOT TUBE COEFFICIENT, Cp _____

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 _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE 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 TEMPERATURE AT DRY GAS METER		FILTER GAS EXIT TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)	TOTAL GASEOUS HYDROCARBON ppm
							INLET °C (°F)	OUTLET °C (°F)			
							AVG.	AVG.			
TOTAL							AVG.				
AVERAGE											

Figure B-2. Particulate and gaseous hydrocarbon field data.

Zero and Span Drift Data

Site Location _____ Operator _____

Date: _____

Test No.: _____

Analyzer: Type _____ S/N _____

	Initial Calibration ppm or %	Final Calibration ppm or %	Difference Initial-Final ppm or %	% of Span
Zero Gas				
High Calibration Gas (Span Gas)			*	

$$\% \text{ of Span} = \frac{\text{Absolute Value of Difference}}{\text{Instrument Span}} \times 100$$

*Corrected for zero drift, i.e., if zero drift over test period is +2 ppm then 2 ppm shall be subtracted from the difference between the initial and final readings.

Figure B-3.

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RESPONSE TIME

Date of Test _____

Analyzer Type _____ S/N _____

Span Gas Concentration _____ ppm

Analyzer Span Setting _____ ppm

Upscale
1 _____ seconds
2 _____ seconds
3 _____ seconds

Average upscale response _____ seconds

Downscale
1 _____ seconds
2 _____ seconds
3 _____ seconds

Average downscale response _____ seconds

System response time = slower average time = _____ seconds.

Figure B-4.

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Plant _____

Date _____

Run No. _____

Relative Humidity _____

Amount liquid lost during transport _____

blank volume, ml _____

wash volume, ml _____

blank concentration, mg/mg (equation B-5) _____

wash blank, mg (equation B-6) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
3			
Total			
	Less acetone blank		
	Weight of particulate matter		

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml.	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure B-5. Analytical data.

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CALIBRATION DATA

Date _____

Analyzer Type _____ S/N _____

High Range Gas Conc. _____ % Full Scale _____

Mid Range Gas Conc. _____ % Full Scale _____

Low Range Gas Conc. _____ % Full Scale _____

Zero Gas _____ % Full Scale _____

Figure B-6.

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APPENDIX C

POM SAMPLING AND ANALYSIS USING THE SPECIAL POM SAMPLING TRAIN

APPENDIX C

POM SAMPLING AND ANALYSIS USING THE SPECIAL POM SAMPLING TRAIN

Background

Several years ago, the need for sampling and analysis of polycyclic organic material (POM) arose on several programs at Battelle. At that time the best available technology for sampling for POM appeared to be some version of the EPA Method 5 particulate sampling train. Use of the Method 5 sampling train produced inconsistent POM emission data and led Battelle staff to question the suitability of using this train for POM sampling. As a result, Battelle developed a POM sampling train that retains many of the features and components of the Method 5 train, but which incorporates an additional element that serves to collect the majority of the POM in the gas sample.

Sampling for POM with the POM Sampling Train should be a straightforward procedure for those already familiar and skilled with the operation of a Method 5 train for particulate collection. The POM sampling procedure utilizes the existing Method 5 train, but also includes a chromatographic adsorbent device (referred to as the adsorbent sampler). This additional component, and its auxiliary equipment, increases the time required for setup, and requires some additional precautionary measures over those associated with particulate sampling alone, but the additional effort and skill required to obtain a meaningful POM sample is not appreciable and the reliability of the POM data obtained is significantly increased.

Sampling System

The POM Sampling Train, as shown in Figure C-1, consists of a Method 5 train with an adsorbent sampler located between the filter and the impingers. Immediately after leaving the hot filter, the gas sample passes into the cooling coil (120 x 0.8 cm) of the adsorbent sampler, and then passes through a Pyrex frit and into a cylindrical column of Tenax adsorbent (7 x 3-cm diameter). The cooling coil and Tenax adsorbent are maintained above the water dewpoint by means of a thermostated circulating water bath. Thus, the incoming gases are cooled to maintain adsorbent efficiency, yet the adsorbent is maintained at a temperature which precludes condensation of water vapor present in all combustion effluents. The gases leaving the sampler are drawn through impingers and a Drierite trap, dry gas meter, and leakless vacuum pump (as in Method 5 sampling).

With the system, POM emissions can be determined from the analysis of the probe wash, filter catch, and adsorbent sampler catch. The impingers are only used to cool and dry the stack gases before they enter the dry-gas meter. Laboratory tests, reported later, have shown that the probe, filter, and adsorbent sampler retain all the POM, and that the POM can be recovered during analysis.

Special Modifications for POM Sampling at Blowing Stills. Because of the expected high moisture content of exhaust gas at the blowing still, the POM Sampling Train configuration

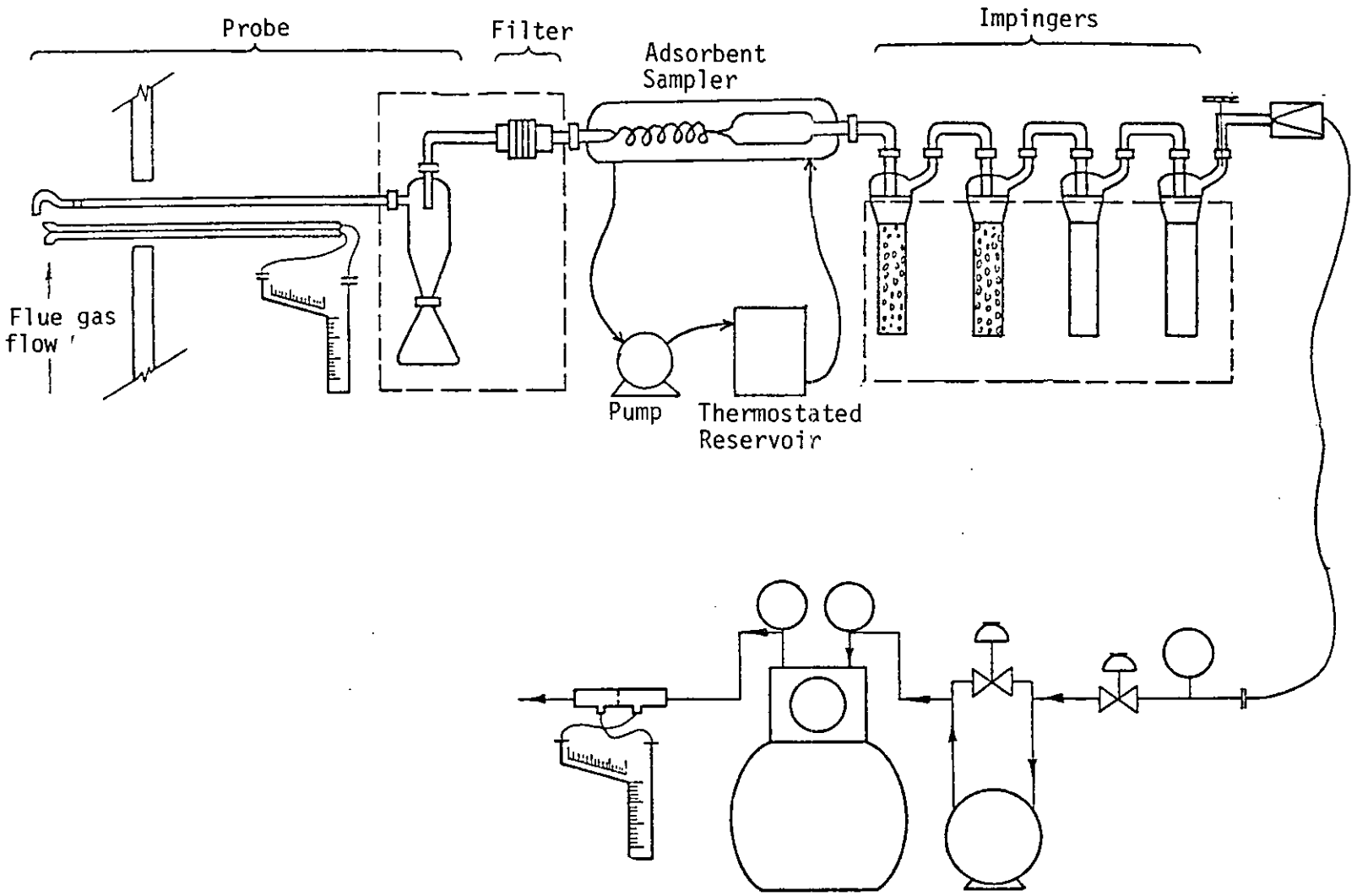


FIGURE C-1. BATTELLE POM SAMPLING TRAIN FIGURE

(shown in Figure C-1) was modified, as shown in Figure C-2, for use in measuring blowing still POM emissions. In order to prevent the large amount of water present in this process from condensing in the adsorption sampler, it was considered necessary to (1) pass the effluent gases through two heated impingers at a temperature (175 F) which would allow condensation to occur prior to the gases entering the absorbent sampler, and (2) operate the adsorbent sampler at 175 F (above the expected dewpoint temperature), instead of the 125 F normally used.

Thus, immediately after leaving the hot filter, the gas sample passed into two empty, heated impingers, then into the coil of the adsorbent sampler, and then through a Pyrex frit and into a cyclindrical column of Tenax adsorbent. The cooling coil was also maintained at 175 F by means of a thermostated circulating water both in hopes of keeping the remaining moisture in the gas phase. The gases leaving the sampler were drawn through cooled impingers and a Drierite trap, dry gas meter, and leakless vacuum pump.

With this system, POM emissions can be determined from the analysis of the nozzle and probe wash, filter catch and filter holder wash, any catch and wash from the hot impingers, and adsorbent sampler catch.

Details of POM Absorber

A schematic representation of the adsorbent sampler is shown in Figure C-3. The heat exchanger section consists of 120 cm (4 feet) of 8-mm Pyrex tubing wound in approximately eight coils. The adsorbent is retained by an extra coarse Pyrex frit and a spring loaded glass wool plug, as shown: Tenax (35/60 mesh) is routinely used in the adsorbent trap of the adsorbent sampler. The dimensions of the adsorbent section are 15-mm radius and 70-mm length. The 28/12 Pyrex joint on the inlet to the sampler is compatible with the fittings commonly used in commercial EPA Method 5 sampling trains; the 15-mm Solv-Seal joint at the sampler outlet provides an efficient vacuum seal during sampling. (A 28/12 Pyrex joint could be used at the sampler outlet.) A vacuum hose coupling between the front sampler outlet and the impinger inlet is sufficient.

Adsorbent Sampler Temperature

The collection efficiency of the adsorbent sampler is dependent on the temperature of the adsorbent material. The optimum temperature of the adsorbent should be as low as possible without condensing large quantities of water vapor (present in most stack gases) and plugging the adsorbent device. Generally, this temperature is 20 to 25 F (10 to 15 C) above the dewpoint of the stack gases. For sampling gases that would consist primarily of vented air or products of combustion of fossil fuels it has been found that 125 F to 130 F (52 to 55 C) is a satisfactory adsorbent temperature. Of course, the adsorbent temperature is to be maintained at the predetermined temperature throughout the run to insure a continuous high POM collection efficiency.

Special Modifications for POM Sampling at Blowing Stills. Because of the high moisture content of the gas to be sampled at the blowing still, it was necessary to maintain the adsorbent sampler temperature at a higher temperature than the 125 to 130 F normally used. The expected dewpoint was 165 F and so the adsorbent sampler was maintained at 175 F to avoid condensation in the unit.

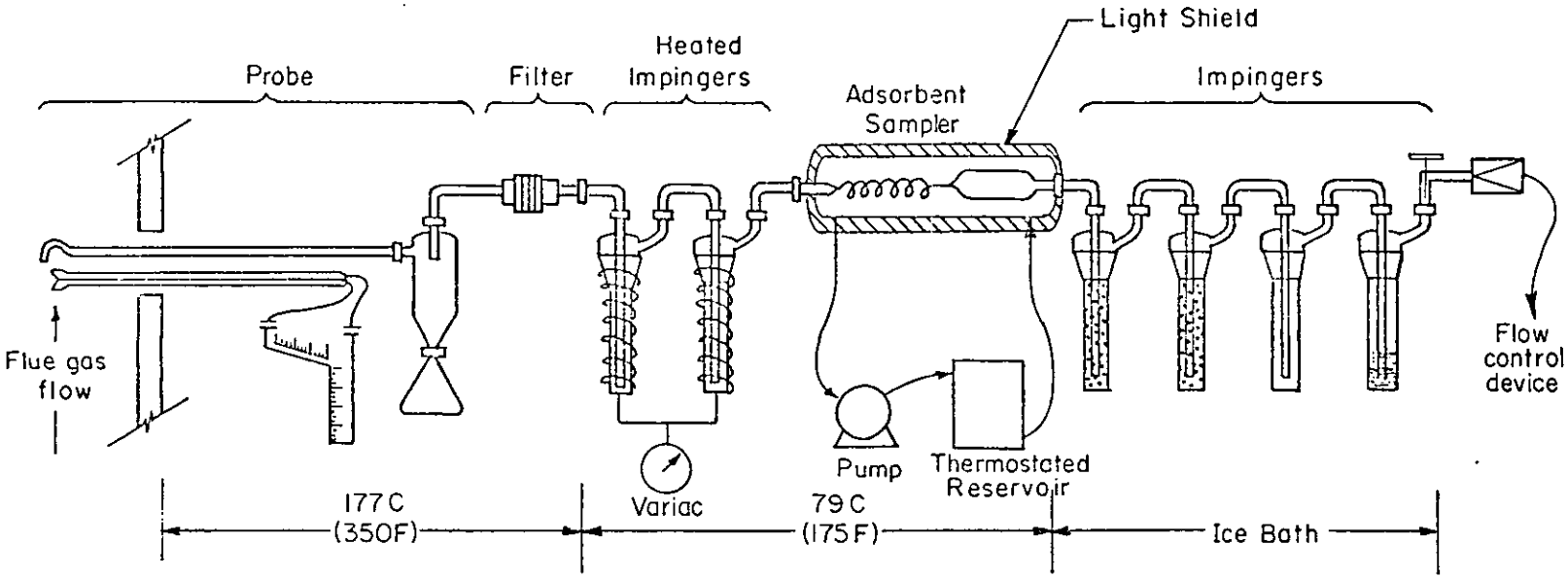


FIGURE C-2. POM SAMPLE TRAIN CONFIGURATION FOR ASPHALT BLOWING STILL PROCESS

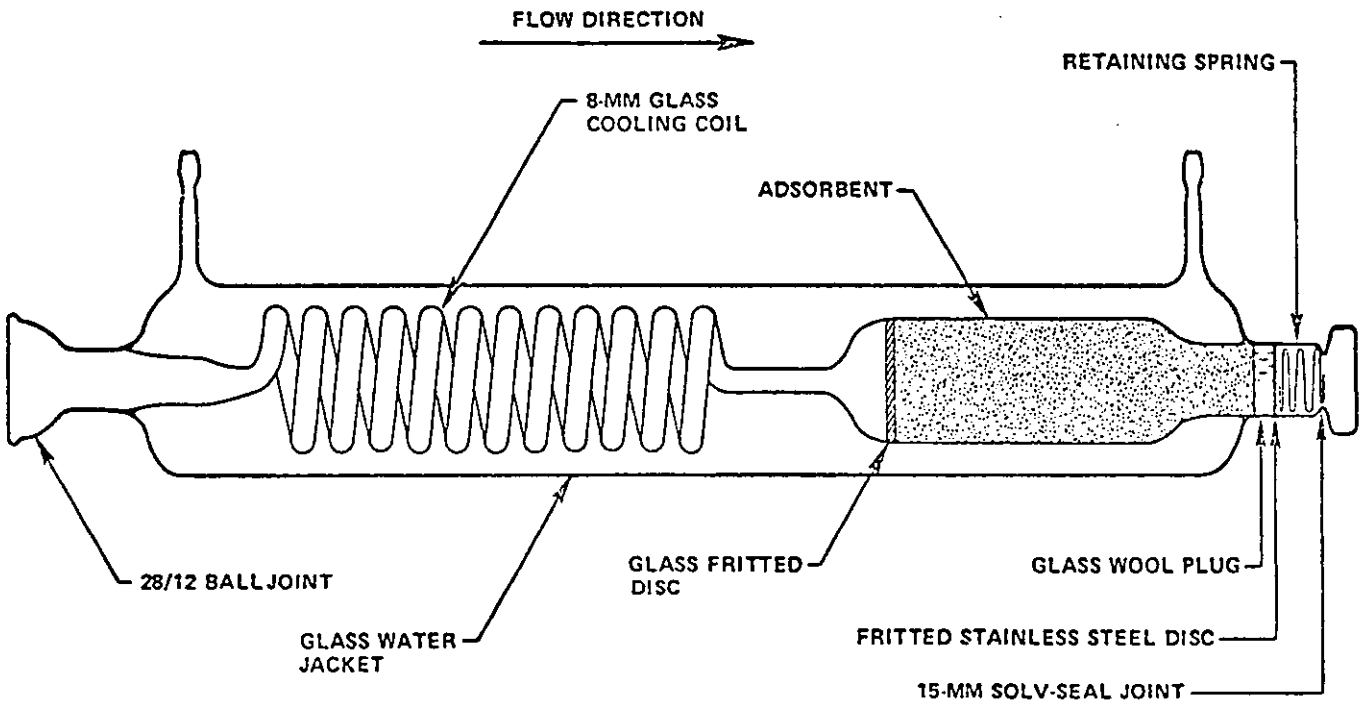


FIGURE C-3. ADSORBENT SAMPLING SYSTEM

Auxiliary Equipment

To maintain the adsorber at a temperature above the dewpoint, a thermostated controlled water bath is used. A 10-ℓ/min pump circulates water to the absorbent sampler from a 4-ℓ reservoir. An acquastat is used to control a 450-watt heating element to maintain the water temperature. For stack gas temperatures up to at least 550 F (300 C) and sampling rates of 0.6 scfm (0.017 Nm^3/min) to 0.75 scfm (0.021 Nm^3/min), the heat loss from the water circulating loop to the ambient surroundings generally is greater than the heat gain from the stack gases to the water, so it usually is necessary to supply auxiliary heat to the water loop. Although unlikely, depending upon the conditions under which a sample is collected (at high stack temperatures and/or high ambient temperature), it may be necessary to cool the circulating water to maintain the desired temperature. A simple calculation of the heat transfer between the gas sample and the circulating water will determine whether or not cooling is needed. If cooling is needed, an additional cooling coil could be inserted between the adsorbent sampler and the reservoir.

Sampling

Sampling with the POM Sampling Train is conducted in essentially the same manner as sampling with the EPA Method 5 sampling train. The one difference between operation of these two sampling trains is that, in using the Battelle POM Sampling Train, it is desired to maintain the probe and filter temperature at 350 F (versus the 250 F when using the EPA sampling train). Maintaining the probe and filter at 350 F prevents condensation and/or adsorption of SO_3 and POM on these components (followed by destructive reaction of SO_3 with POM). Instead of being caught by the probe and filter, the POM passes through these components and is retained in the absorbent sampler.

When sampling for POM, the stack gases are sampled isokinetically as described in Method 5. (Isokinetic sampling is desired because some of the POM may be physically associated with particulate in the gas stream.) The pressure drop associated with the flow of stack gases through the adsorbent device may interfere with maintaining an isokinetic sampling rate throughout the run, therefore it is usually a good idea to reduce the calculated nozzle size in order to reduce the flow rate and, thus, the pressure differential across the sampler.

Sample Recovery in the Field and Sample Preservation

Polycyclic organic materials are readily photooxidized in the presence of ultraviolet light (and possible visible light). Thus, the sampling train should be protected from sunlight and all other ultraviolet sources, both during and after sample collection. Also, some organic compounds which are collected by the adsorbent sample may have an appreciable vapor pressure, and care must be exercised to minimize losses of such materials. To prevent loss of POM by photooxidation, a heavy dark cloth is placed over the exposed sampling train glassware during sampling. Immediately after sample collection is completed, the adsorbent sampler is sealed with ball-joint and Solv-Seal stoppers and the filter and adsorbent sampler are stored in a cool light-free container for transport to the analytical lab.

The probe and glassware up to the filter (including the filter holder) are washed with acetone followed by methylene chloride*; these solvents are 'Distilled-in-Glass' quality or better. The solution and particulate matter from the probe rinse are stored in a dark (amber) glass bottle prior to analysis and kept cool.

Sample Extraction and Recovery

Sample recovery from the POM Sampling Train for POM analysis involves extraction of three separate portions of the total sampling train:

(1) Probe and glassware up to the filter
(2) Filter
(3) The adsorbent sampler.

Initial recovery of Item (1) is done in the field (i.e., the probe is washed as described above), while Items (2) and (3) are most conveniently extracted in the laboratory.

Sample Recovery from the Probe Wash

The probe wash (a solvent and particulate mixture) is agitated for 1 hour in an ultrasonic bath before filtering off the solvent with a Whatman No. 40 filter.

Sample Recovery from the Filter

Organic material is extracted from the filter by means of Soxhet extraction with methylene chloride ('Distilled-in-Glass'), or by ultrasonic agitation with methylene chloride followed by filtration using a Whatman No. 40 filter. While both methods have been found to be equally satisfactory, ultrasonic extraction is somewhat faster.

Sample Recovery from the Adsorbent Sampler

Great care must be taken not to expose the adsorbent sampler to polar solvents such as methylene chloride or acetone, since the Tenax adsorbent is readily soluble in these solvents. Our experience has shown that is it preferable to extract the adsorbent sampler with a low boiling point hydrocarbon such as pentane ('Distilled-in-Glass').

To extract the adsorbent sampler, the two stoppers are first removed, and the extraction apparatus assembled, as shown in Figure C-4, under yellow safe-lights. A double surface water cooled condenser is preferred, the distilling flask is of 250-ml capacity. The adsorbent sampler is extracted with 'Distilled-in-Glass' pentane. An initial volume of 180 ml is usually necessary since there is an appreciable solvent holdup during extraction. The samplers are extracted with the continuous extraction apparatus for 24 hours, and it is normal to experience a small loss of pentane during this period. The extraction apparatus is then disassembled and the pentane extract stoppered and stored in darkness.

*To minimize evaporative sample loss during solvent extraction, solvents with very low boiling points are used.

Thus, three extracts are obtained from the adsorbent sampler-Method 5 sampling train:

(1) Acetone and methylene chloride probe extract
(2) Methylene chloride filter extract
(3) Pentane adsorbent sampler extract.

These extracts are sealed and kept in darkness while awaiting analysis.

Reactivation of Adsorbent Sampler

Following extraction, air is drawn through the sampler with an aspirator to remove most of the remaining pentane solvent. The sampler is then dismounted by withdrawing the stainless steel spring with a hooked spatula, removing the stainless steel perforated disk, discarding the glass wool plug, and emptying the almost dry Tenax into a clean glass container. The adsorbent sampler body is cleaned by blowing with compressed air to remove any trace materials and then rinsed with the following solvents in the order given:

(1) Methylene chloride
(2) Chromic acid
(3) Water
(4) Acetone
(5) Methylene chloride
(6) Pentane.

The sampler is then sealed with clean stoppers prior to refilling with activated Tenax.

Used pentane extracted Tenas may be reactivated and thoroughly cleaned by placing it in an oven at 200 C under nitrogen flow in a glass tube for 24 hours. New Tenax may be similarly prepared by first Soxhlet extracting with pentane for 24 hours and then heating under nitrogen. It is generally desirable to maintain a small supply of activated Tenax, and to reactivate the adsorbent from six or more samplers at one time.

An adsorbent sampler is prepared by filling the adsorbent section with activated Tenax to within 3/4 inch of the top of the sampler while agitating the sampler with an electrical vibrator. A clean glass wool plug is then inserted into the neck, followed by a perforated stainless steel disk and the stainless steel retaining spring. The sampler is then sealed with the appropriate 28/12 ball-joint and 15-mm Solv-Seal stoppers and is ready to use.

Analysis of Extracts

The three extracts from the probe, filter, and absorbent samples, may be analyzed separately for POM compounds, or they may be combined and a single POM analysis performed on the total sample.

Internal standards are added to the combined extracts from each sampling train prior to volume reduction by rotary evaporation and Kuderna-Danish evaporation. The extract is subjected to a Rosen-type liquid chromatography separation[1] in order to isolate the POM fraction

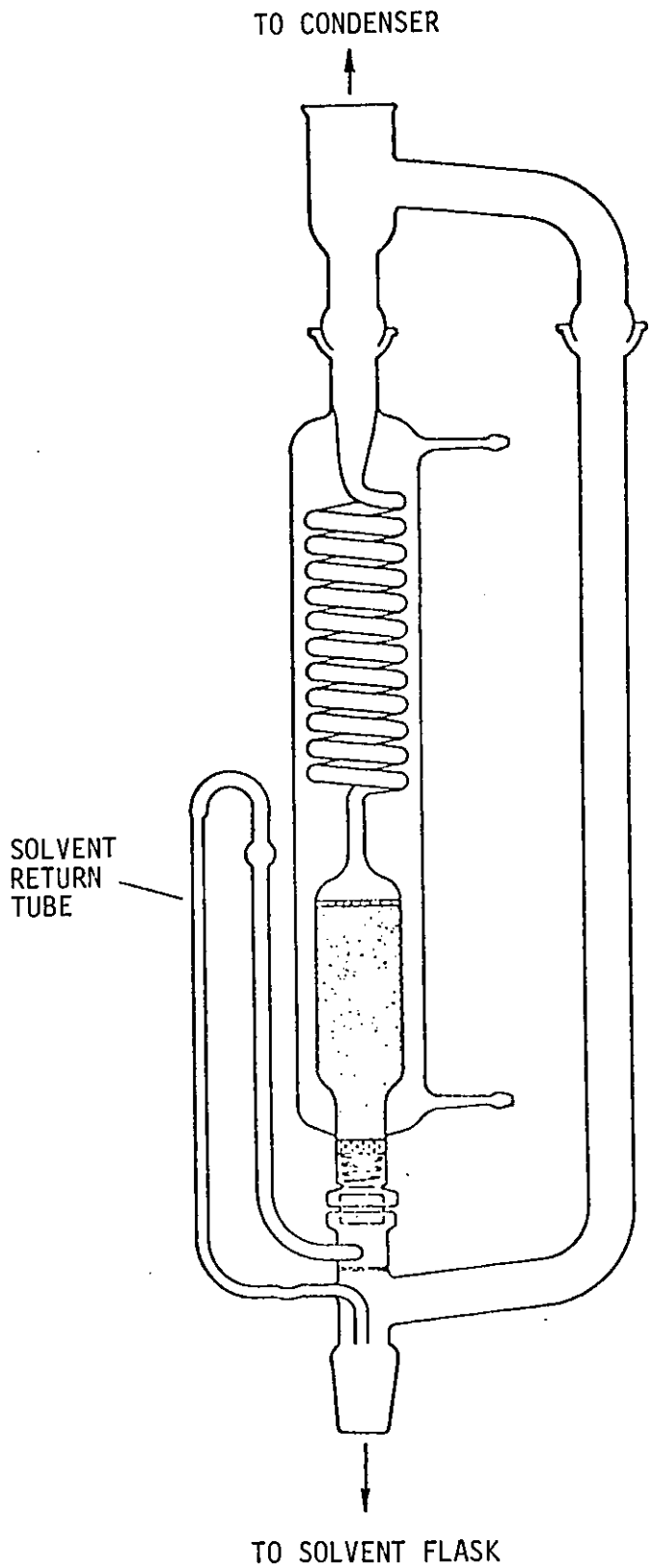


FIGURE C-4. CONTINUOUS EXTRACTION ASSEMBLY FOR ABSORBENT SAMPLER

before carrying out gas chromatographic-mass spectrometric (GC-MS) analysis. Gas chromatographic separation is achieved using a 14-foot x 2-mm, 2½ percent, Dexil 300 column programmed from 170 C to 350 C at 4 C min^{-1}. Separation of the benzpyrene isomers is routinely accomplished using a one foot 1% N,N'-Bix (p-methoxy-benzylidene)-α,α'-bi-p-toluidine column isothermal at 130 C. Mass spectrometric analysis is carried out with a Finnigan 1015 quadrupole mass spectrometer with a chemical ionization source; methane is routinely used as the carrier and reagent gas. Data acquisition is accomplished with a System Industries 150 data acquisition system, and quantification of the POM compounds present is accomplished using a Digital PDP8 computer.

This mass spectrometric-computer quantification procedure makes use of specific absolute ion currents. The bases for the quantification procedure is to initially obtain the computer reconstructed gas chromatogram and mass spectrum in the normal fashion; this reconstructed gas chromatogram is then displayed on the CRT terminal and an overlay for the protonated molecular ion of the POM of interest is superimposed. This overlay represents the ion current corresponding to that specific POM molecular weight plus 1 mass unit. If there is an area in the reconstructed gas chromatogram where the overlay indicates that this mass number is prevalent, the mass spectrum of this peak is displayed on the CRT unit, and the presence of the POM may be confirmed. If the POM is found to be present at a correct relative retention time to the internal standards, the computer then sums the ion current due to all important ions in the POM's mass spectrum which represents the area of the peak of interest. Quantification of each POM is achieved by ratioing its ion current to that of an internal standard of known concentration. The relative ionization efficiencies of the internal standards and many POM species were previously determined, and the appropriate factor is used in quantification.

Preliminary experiments were carried out in order to determine the most suitable solvent for extraction of the adsorbent sampler. Extraction was attempted with methylene chloride, acetone, methyl alcohol, p-dioxane, pentane, cyclohexane, benzene, and toluene; only saturated hydrocarbons proved entirely suitable, on account of partial Texan solubility in more polar solvents. Pentane was found to be the most suitable solvent, its high volatility minimized sample loss during extraction. The relatively low extraction efficiency of pentane is overcome by means of continuous solvent extraction for a period of over 24 hours, as described above.

Initial validation experiments involved sampling and recovery of measured quantities of anthracene; during this work the temperature of the sampler was allowed to rise to approximately 200 F (93 C), but quantitative anthracene recovery was always obtained. (Later field experiments have suggested that sampling with absorbent temperatures of 175 F (80 C) may not result in retention of a significant fraction of the POM compounds.)

Subsequent validation experiments were carried out with the sampler at 130 F (55 C) using pyrene, chrysene, perylene, benz(ghi)perylene, and coronene; these compounds are representative of commonly encountered POM species. Ten thousand ng of each of these compounds was separately sampled over a 2-hour time period; following pentane extraction and addition of internal standard, each POM compound was quantified by GC-MS using specific absolute ion current integration. The results of several representative laboratory validation experiments are given in Table C-1.

This quantification technique overcomes the problems associated with interfering or overlapping peaks and poor base-line separations since these interfering species usually have different molecular weights. Isomeric compounds such as pyrene and fluoranthrene which have the same

TABLE C-1. RECOVERY OF POM FROM ADSORBENT SAMPLER
Integration by GC-MS

POM	Spiked (ng)	Sample #1 Average % Recovery	Sample #2 Average % Recovery	Sample #3 Average % Recovery
Pyrene	10,000	91 ± 3	98 ± 4	104 ± 4
Chrysene	10,000	90 ± 5	92 ± 5	106 ± 5
Perylene	10,000	91 ± 4	105 ± 5	102 ± 6
Benz(ghi)perylene	10,000	101 ± 10	106 ± 10	103 ± 7
Coronene	10,000	80 ± 7	92 ± 8	100 ± 14

molecular weight can be quantified easily since they are very adequately separated by gas chromatography and their order of elution is known.

In order to obtain optimum sensitivity during the gas chromatographic-mass spectrometric analyses, the ionization voltage must be adjusted at various stages during the analysis. This adjustment necessitated the incorporation of three internal standards so that an internal standard would elute between each ionization adjustment. The internal standards chosen were 9-methylanthracene, 9-phenylanthracene, and 9,10-diphenylanthracene. It was fortuitous that 9,10-diphenylanthracene elutes almost coincident with the benz(a)pyrene/benz(e)pyrene isomers, giving a very accurate marker when searching for these important compounds. The relative retention times of other POM species to one or more of the internal standards are generally sufficiently well known to permit specific compound identification when the mass spectra are displayed.

This ion integration technique has proven to be far superior to GC for the analysis and quantification of POM due to its very high selectivity; it also offers a very significant advantage in speed of data handling.

Laboratory Validation Studies

Laboratory validation studies were performed on the absorbent sampler component of the POM Sampling Train. These validation studies involved setting up an adsorbent sampler, to collect air drawn through a 500 F (260 C) tube furnace. A precisely measured quantity of polynuclear compounds in a few microliters of methylene chloride solution was then injected into the inlet of the adsorbent sampler, and heated air was passed through the system for at least an hour. Following solvent extraction of the sampler, a suitable internal standard was added, and analysis for the spiked polynuclear compound was made by GC-MS analysis.

References

(1) Moore, G. G., Thomas, R. S., and Monkman, J. L., *J. Chromatog.*, **26**, 456 (1967).

APPENDIX D

POM FIELD DATA AND CALCULATIONS
175 F CALIBRATION RUNS

- POM Field Data
- POM Run Calculations

POM FIELD DATA

ANALYTICAL DATA

PLANT Certain-Teed, Shakopee Minn

DATE Sept 13 75

SAMPLING LOCATION

SAMPLE TYPE EPA #5 w/ POM

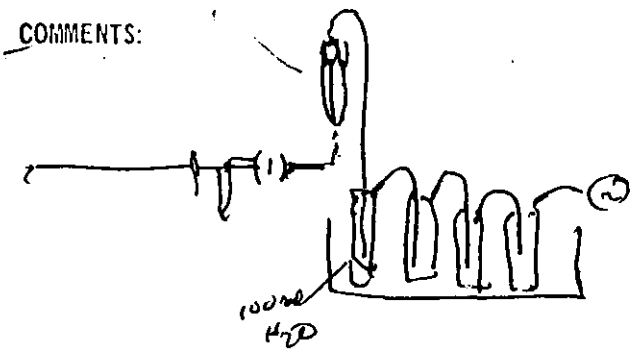
RUN NUMBER POM-TP-~~1~~-1

SAMPLE BOX NUMBER Loop #12, Glass Set #6

CLEAN-UP MAN MCN

HUPPS - 175 F

COMMENTS:



FRONT HALF

LABORATORY RESULTS

ACETONE WASH OF NOZZLE, PROBE, CYCLONE (BYPASS), FLASK, FRONT HALF OF FILTER HOLDER — CONTAINER _____ _____ mg

FILTER NUMBER 5a146 _____ — CONTAINER _____ _____ mg

tare 0.37824 g _____

net _____ _____

FRONT HALF SUBTOTAL _____ mg

BACK HALF

IMPINGER CONTENTS AND WATER WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER _____ _____ mg

ETHER-CHLOROFORM EXTRACTION _____ mg

ACETONE WASH OF IMPINGERS, CONNECTORS, AND BACK HALF OF FILTER HOLDER — CONTAINER _____ _____ mg

BACK HALF SUBTOTAL _____ mg

TOTAL WEIGHT _____ mg

Rinse volumes _____ ml _____ ml _____ ml _____ ml _____ ml _____

MOISTURE

IMPINGERS	1	2	3	4	5	6
FINAL VOLUME	92 ml	8 ml	ml	51 ml	ml	
INITIAL VOLUME	100 ml	0	0	51		
NET VOLUME	ml	ml	ml	41 ml	ml	ml

SILICA GEL			
FINAL WEIGHT	494.6 g	g	g
INITIAL WEIGHT	475 g	g	g
NET WEIGHT	g	g	g

TOTAL MOISTURE _____ g

EPA (Dur) 231
4/72

FIELD DATA

15 point
4
7.5 minutes/port

PPOM TP1-1

PLANT Certain Teed
DATE 9-13-13
SAMPLING LOCATION INLET TP-1 North Port
SAMPLE TYPE PPOM + Method 5
RUN NUMBER TP1-4 North Port
OPERATOR Don Hopp
AMBIENT TEMPERATURE 60
BAROMETRIC PRESSURE 29.60
STATIC PRESSURE, (P_s) -1.0
FILTER NUMBER (s)
(PPOM Temp set 175°F)

EAST
North

PROBE LENGTH AND TYPE 4 ft actual length
NOZZLE I.D. .245
ASSUMED MOISTURE, % 3
SAMPLE BOX NUMBER 12
METER BOX NUMBER 12
METER $\Delta H_@$ 1.82
C FACTOR 1.05
PROBE HEATER SETTING
HEATER BOX SETTING
REFERENCE Δp ~~.696~~ .6

38
32
70

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY 4 MINUTES

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 DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F OVEN	PORT	IMPINGER TEMPERATURE, °F	Probe °F
	0	220	370.0	.24	.24									
North Port 1 19	4	224		.28	.88	.88	235	71	71	8.7	305	176	56	
2 20	8	228		.3	.93	.93	232	75	80	8.8	300	176	56	200
3 21	12	232	376.3	.34	1.1	1.1	230	78	85	8.8	310	175	56	
4 22	16	236		.34	1.1	1.1	242	78	85	8.8	318	175	56	
5 23	20	240		.37	1.12	1.12	236	78	85	8.8	307	175	65	
6 24	24	244		.36	1.1	1.1	222	70	88	10.5	307	175	63	
7 25	28	248		.34	1.08	1.08	228	70	88	10.5	310	175	63	110
8 26	32	252	386.6	.3	.93	.93	236	72	94	9.5	310	176	66	300
9 27	36	256	389.7	.26	.8	.8	252	72	95	8.9	320	175	67	235
10 28	40	300	~~389.7~~	.26	.8	.8	222	72	98	8.9	328	176	67	278
11 29	44	304	393.6	.24	.74	.74	216	70	98	8.4	336	177	71	292
12 30	48	308		.27	.82	.82	226	70	98	8.8	345	177	72	313
13 31	52	312	397.4	.23	.72	.72	228	70	98	8.2	350	177	72	351
STOPPED 32	56	316	399.0	.23	.72	.72	230	70	98	8.2	360	177	75	360
STARTED 32	56	338		.23	.72	.72	220	70	98	8.2	300	180	60	
15 33	60	342		.28	.86	.86	220	70	98	8.2	300	180	60	
16 35	64	346		.3	.93	.93	225	79	91	9.2	306	175	63	340
17 36	68	350	405.7	.27	.83	.83	221	79	91	9.0	320	172	63	360
18 2	72	354	408.0	.27	.77	.77	216	79	91	8.2	327	174	65	370
END														

COMMENTS:

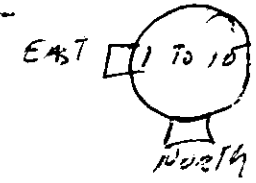
.3 Corrected

EPA (Dur) 235
1/72

620

72
67
25 min

PLANT Certain Teed
DATE 9-13-75
SAMPLING LOCATION INLET TP-1 EAST PORT
SAMPLE TYPE PDM+ Method 5 1 min
RUN NUMBER TP1-4
OPERATOR DON HUPP
AMBIENT TEMPERATURE 60
BAROMETRIC PRESSURE 29.60
STATIC PRESSURE, (P_s) -6.0
FILTER NUMBER (s)



SCHEMATIC OF TRAVERSE POINT LAYOUT

PROBE LENGTH AND TYPE 48" Octrin Lamp
NOZZLE I.D. .242
ASSUMED MOISTURE, % 3.0
SAMPLE BOX NUMBER 12
METER BOX NUMBER 12
METER $\Delta H_@$ 1.82
C FACTOR 1.05
PROBE HEATER SETTING
HEATER BOX SETTING
REFERENCE Δp .6

READ AND RECORD ALL DATA EVERY _____ MINUTES

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 DESIRED	ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m\,in}$), °F	OUTLET ($T_{m\,out}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE, °F OVEN	PDM	IMPINGER TEMPERATURE, °F	Probe
	0	400	408.0											
1	4	404	410.3	.32	1.0	1.0	223	70	86	9.5	340	173	60	228
2	8	408	410.9	.32	.99	.99	228	76	86	9.5	245	174	60	528
3	12	412	413.4	.36	1.1	1.1	160	65	68	10.0	195	174	60	175
4	13	415	414.6	.3	1.0	1.0	185	65	68	9.5	210	174	56	240
5	16	420		.30	1.0	1.0	185	65	70	9.5	215	174	56	
6	20	424	417.1	.35	1.1	1.1	185	65	70	9.5	225	174	56	
7	24	428		.35	1.1	1.1	194	70	82	10.2	280	174	60	300
8	28	432		.33	1.02	1.02	200	68	81	10.1	300	174	60	315
9	32	436	423.7	.3	.94	.94	195	68	91	9.5	312	176	65	
10	36	440		.3	.93	.93	300	70	95	9.5	285	170	68	270
11	40	444	428.1	.34	1.08	1.08	308	70	95	10.0	255	180	70	220
12	44	448		.26	.8	.8	306	70	93	10.0	257	176	70	265
13	48	452		.25	.78	.78	210	72	95	8.5	283	172	75	282
14	52	456		.28	.86	.86	214	68	98	8.5	300	173	75	320
15	56	460	436.24	.3	.93	.93	216	68	98	9.2	285	174	73	278
16	60	464	438.47	.33	1.05	1.05	217	68	98	9.5	262	172	73	249
17	64	468	440.0	.33	1.05	1.05	250	73	100	10.0	250	176	73	284
18	68	472	~~443.0~~	.35	1.1	1.1	303	73	95	10.0	277	170	65	
19	64	730	442.8	.35	1.1	1.1	305	73	95	10.0	338	170	65	262
20	68	741		.33	1.05	1.05	305	73	95	10.1	310	175	60	190
	72	749	447.35	.3	.95	.95	210	73	95	10.0	300	178	60	560
			END OF RUN											

Topped
Topped
Topped
Topped
Started
Stopped
Started

COMMENTS:

77.35 cf

$\frac{3.62086}{46}$ 0.91

$\frac{6318}{84}$ = 77

D-3

EPA (Dur) 235
4-72

IDENTIFICATION LOG OF SAMPLES COLLECTED

Plant CERTAIN-TEED, INC.
Location Shakopee, Minnesota
Industry Asphalt roofing
Project No. G-2872-3514

Battelle Record Book No 32148
Collected & Recorded By
Page 7 of 12.

EPA No. S75-006	Date 9/9/75	Run Number	Sample Description	Remarks
			POST-CLEAN-UP--AFTER RUN	
-199		CER-CU-1A	TCE Rinse of probe and nozzle	
-200		"	Impinger water	
-201		"	TCE rinse of impinger and connectors	
-202		"	Acetone rinse of impinger and connectors	
-203		CER-CU-2A	TCE rinse of probe and nozzle	
-204		"	impinger water (catch)	
-205		"	TCE rinse of impinger and connectors	
-206		"	Acetone rinse of impinger and connector	
-207		CER-CU-3A	TCE Rinse of probe and nozzle	
-208		"	Impinger water catch	
-209		"	TCE rinse of impinger and connectors	
-210		"	Acetone rinse of impinger and connectors	
-211		CER-CI-4A	TCE rinse of probe and nozzle	
-212		"	Impinger water catch	
-213		"	TCE rinse of impinger and connectors	
-214		"	Acetone rinse of impinger and connectors	
-215		CER-CU-5A	TCE rinse of probe and nozzle	
-216		"	Impinger water	
-217		"	TCE rinse of impinger and connectors	
-218		"	Acetone rinse of impinger and connectors	

POM RUN CALCULATIONS

CERTAIN-TEED PP04 (175 F) TP-1

	RUN NO.	1
	TEST DATE	9/13
	SAMPLING TIME, 24 HOUR CLOCK FROM	1420
	TO	1949
DN	SAMPLING NOZZLE DIAMETER, IN.	0.245
TT	NET TIME OF TEST, MIN.	144
PB	BAROMETRIC PRESSURE, IN. HG ABSOLUTE	29.60
PM	AVG. ORIFICE PRESSURE DROP, IN. H2O	0.91
VM	VOLUME OF DRY GAS SAMPLED AT METER CONDITIONS, DCF	77.3
TM	AVG. GAS METER TEMPERATURE, F	77
VMSTD	VOLUME OF DRY GAS SAMPLED AT STANDARD CONDITIONS, DSCF	75.6
VW	TOTAL H2O COLLECTED IN IMPINGERS AND SILICA GEL, ML	31.5
VWGAS	VOLUME OF H2O VAPOR COLLECTED, SCF	1.5
%M	PERCENT MOISTURE IN STACK GAS BY VOLUME	1.94
MD	MOLECULAR FRACTION OF DRY GAS	0.98
% CO2	VOLUME PERCENT DRY	0.1
% O2	VOLUME PERCENT DRY	20.8
% CO	VOLUME PERCENT DRY	0.1
% N2	VOLUME PERCENT DRY	79.0
% EA	PERCENT EXCESS AIR	37143
MWD	MOLECULAR WEIGHT OF STACK GAS, DRY BASIS	28.8
MW	MOLECULAR WEIGHT OF STACK GAS, WET BASIS	28.5
CP	PITOT TUBE COEFFICIENT	0.85
TS	AVG. STACK TEMPERATURE	214
NP	NET SAMPLING POINTS	40

CERTAIN-TEED PROM (174 F) TP-1

	RUN NO.	1
	TEST DATE	9/13
	SAMPLING TIME, 24 HOUR CLOCK FROM	1420
	TO	1949
PST	STATIC PRESSURE OF STACK GAS, IN. HG.	-0.07
PS	STACK GAS PRESSURE, IN. HG ABS.	29.53
VS	STACK GAS VELOCITY AT STACK CONDITIONS, FPM	2133
AS	STACK AREA, SQ. IN.	707
QS	DRY STACK GAS VOLUMETRIC FLOW RATE AT STANDARD CONDITIONS, DSCFM	8008
QA	STACK GAS VOLUMETRIC FLOW RATE AT STACK CONDITIONS, ACFM	10513
% I	PERCENT ISOKINETIC	98.6
% O	PERCENT OPACITY	
TC	UNIT FEED RATE, TONS/HR	

CERTAIN-TEED PROM (175 F) TP-1

RUN NO.	1
TEST DATE	9/13
VOLUME OF GAS SAMPLED, DSCF	75.6
PERCENT MOISTURE BY VOLUME	1.9
AVERAGE STACK TEMPERATURE, F	214
STACK VOLUMETRIC FLOW RATE, DSCFM	8008
STACK VOLUMETRIC FLOW RATE, ACFM	10513
PERCENT ISOKINETIC	98.6
PERCENT EXCESS AIR	37143
PERCENT OPACITY	
FEED RATE, TONS/HR	

CERTAIN-TEED PROM (175 F) TP-1

RUN NO.	1
TEST DATE	9/13
VOLUME OF GAS SAMPLED, NCM	2.18
PERCENT MOISTURE BY VOLUME	1.9
AVERAGE STACK TEMPERATURE, C	101
STACK VOLUMETRIC FLOW RATE, NCMM	225
STACK VOLUMETRIC FLOW RATE, CMM	236
PERCENT ISOKINETIC	98.6
PERCENT EXCESS AIR	37143
PERCENT OPACITY	
FEED RATE, MTON/HR	

TABLE I-3. ANALYSIS OF PACKED COLUMN SAMPLES
(Weights in mg)

Sample	Saturator		Blowing Still				FID Response Factor for Each Sample Fraction, percent
	ESP Inlet	ESP Outlet	Afterburner Inlet #1	Afterburner Inlet #2	Afterburner Outlet #1	Afterburner Outlet #2	
Fraction #1	14.1	26.4	47.2	40.4	0.5	0.1	100
Alkanes Alkenes							
Fraction #2	6.3	14.9	22.4	17.4	0.2	0.1	100
Alkylbenzenes							
Fractions #3 & #4	1.5	3.5	6.0	1.4	0.1	0.0	96
POM's Thiophenes							
Fraction #5	1.9	2.6	28.9	24.6	0.3	0.2	90
Carbazoles Esters							
Fractions #6 & #7	2.0	6.1	63.2	46.3	11.5	9.7	90
Benzaldehydes Tolualdehydes Phalaldehydes Ketones							
Fraction #8	7.1	18.3	45.6	32.6	10.5	10.6	90
Alcohols Phenols Amines Acids Oxygenated diphatic compounds							
Total	32.9	72.3	213.3	162.7	23.1	20.7	
FID Response for Total Sample, percent	96	96	93	94	91	90	

TABLE II-4. ANALYSIS OF RAW SAMPLES AND CONDENSATE FOR ORGANICS

Sample		Results of GC Analysis (Specifically detects straight-chain hydrocarbons)	
		Raw Sample	Condensate
Inlet Samples			
S75-006-037	TCE probe and nozzle wash	C11 to C32 organics present; peak in C13 to C16 range	Nil organics, organics less than 1 percent of raw sample
S75-006-038b	TCE extract of impinger water	Organic present, peak concentration in C16 to C18 range	Contained C10 and C12 compounds equally to 10 percent and 4 percent of raw sample, respectively
S75-006-038c	Impinger water (after extraction)	Slight organic content; some TCE evident	C10 to C25 organics contained in a 3 ml TCE bubble; trace of raw organics in water portion
S75-006-039	TCE wash of impingers	C11 to C32 organics present; peak in C13 to C16 range	Contained C10, C12, and C14 compounds equal to 5 percent, 2 percent, and less than 1 percent of raw sample, respectively
Outlet Samples			
S75-006-043	TCE probe and nozzle wash	Nil organic content	Trace of C10 to C15 organics
S75-006-044b	TCE extract of impinger water	Not analyzed	Trace of C10 and C11 compounds
S75-006-044c	Impinger water (after extraction	Not analyzed	Trace of organics
S75-006-045	TCE wash of impingers	Nil organic content	Nil organic content