REVISED FINAL WORK PLAN

PERFORMANCE MONITORING OF THE VAPOR CONTROL SYSTEM LOCATED AT THE UNISYS GREAT NECK FACILITY

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TABLE	OF	CONTENTS
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NO. DESCRIPTION

PAGE NO.

1.0	Introd	luction
2.0	Site H 2.1	Iistory
3.0	Scope	of Work
	3.1	Gas Stream Sampling Locations and Characterization
		3.1.1 Inlet Sample Locations
		3.1.2 Outlet Sample Locations
		3.1.3 Gas Stream Characterization
	3.2	Determination of VOC Concentrations
		3.2.1 NYDEC Target Compound List
		3.2.2 Sampling Train
		3.2.3 Adsorbent Tubes
		3.2.4 Condensates
		3.2.5 Sample Identification
	3.3	Determination of HCL Concentrations
		3.3.1 Sampling Train
4.0	Samp	e Custody
5.0	Labor	atory Analysis
	5.1	Volatile Organic Analysis
		5.1.1 Desorption Efficiency Test
		5.1.2 TCL Analysis
	5.2	HCL Analysis
6.0	Final	Report
	6.1	Calculation of Gas Velocity and Flow Rate
	6.2	Calculation of Sample Volumes
		6.2.1 Calculation of Removal Efficiency
Apper	ndix A	U.S. EPA Test Methods 40 CFR Part 60, Appendix A A-1
Attacl	hment B	Preliminary Emission Monitoring Results

LIST OF FIGURES

NO. DESCRIPTION

PAGE NO.

2-1	VOC Control Schematic	4
2-2	Scrubber Control Schematic	5
3-1	VOC Inlet Sample Points	2
3-2	VOC and HCL Outlet Sample Points	3

LIST OF TABLES

Ν	Ю.	DESCRIPTION	ł	PA	GE	NO.
2.	-1	VOCs Detected During SVE Pilot Test			• •	. 2-2
2.	-2	Detected VOCs in Influent Gas Stream	•			. 2-3
3-	-1	Target Compound List	•			. 3-5
5	-1	Detection Limits for TCL Compounds				. 5-2

.

1.0 INTRODUCTION

Remediation Technologies, Inc. (RETEC) has been contracted to conduct a performance monitoring program on a newly installed soil vapor off-gas control system located at the Unisys Great Neck, New York facility. The program has been designed to validate the effectiveness of the system, which has been designed to achieve an organic removal efficiency of 99%.

The Unisys vapor control system is comprised of two major components: a catalytic oxidizer to control volatile organic compounds (VOCs); and a spray chamber scrubber to control hydrogen chloride (HCL) emissions. Details of the vapor control system are presented in Section 2.0 of the Work Plan.

RETEC will perform organic simultaneous sampling at the inlet and outlet vapor streams of the catalytic oxidizer to determine its efficiency. In addition, acid gas vapors will be monitored from the exhaust stream of the spray chamber scrubber. Three sample test runs will be performed as part of the emission monitoring program. Sampling procedures will follow methods established by the U.S. EPA under 40 CFR Part 60, *Standards of Performance for New Stationary Sources, Appendix A*. Details of the sampling and analytical procedures to be used during the program are presented in Sections 3.0 and 5.0 of this document.

2.0 SITE HISTORY

In December 1991, a pilot soil vapor extraction (SVE) test was performed by Unisys at their Great Neck facility. Data collected during the program was used to estimate VOC levels in the vapor stream to evaluate and select an appropriate vapor control system. Results indicated that six VOCs constituted the majority of VOCs detected in the vapor stream. Table 2-1 presents the individual VOCs of interest and their average concentration. Based on the results of the SVE program test, Unisys has chosen VOC oxidation as the vapor control system.

In June 1993, RETEC performed a preliminary stack test program on the inlet vapor stream to the catalytic oxider. At the request NYDEC, the full target compound list (TCL) for volatile and semi-volatile organics and PCBs were sampled for in the gas stream. Results indicated that fifteen VOCs constituted the majority of compounds detected in the vapor stream.

Only one semi-volaitle organic compound was detected during the preliminary stack test: naphthalene. PCB aroclors were not detected in the vapor stream at detection limits as low as 0.005 mg/m³. Table 2-2 presents the individual constituents and their concentration from the preliminary test. These sixteen compounds will comprise the new list of principal compounds for the compliance program.

2.1 VAPOR CONTROL SYSTEM

Treatment of the extracted vapors will be achieved by catalytic oxidation. Unisys has chosen a Chloro-Cat oxidizer, manufactured by Global Technologies, Inc. A schematic of the system is presented in Figure 2-1. The system was designed to achieve a 99% removal efficiency of VOCs at a flow rate of 500 scfm.

The VOC laden air is drawn into the system by way of a vacuum blower and passes through a heat exchanger prior to the natural gas fired burner. The burner raises the temperature of the vapors to the catalyzing temperature of approximately 650°F. The VOCs in the stream are converted to carbon dioxide, water vapor and inorganic acids. A spray chamber scrubber is used to neutralize the inorganic acids prior to the release to the atmosphere. A schematic of the scrubber system is presented in Figure 2-2.

The treated vapors will leave the scrubber and enter the discharge stack at a temperature of 135°F. The stack is eight inches in diameter, thirty-five feet high, and will terminate ten feet

TABLE 2-1

VOCs DETECTED DURING SVE PILOT TEST

Compound	Average Concentration (ppmv)
1,2 Dichloroethene	4,200
Trichloroethene (TCE)	1,700
Ethyl Benzene	36
Tetrachloroethene (PCE)	1,100
Toluene	1,200
Vinyl chloride	167
Total	8,403

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¹Source: Table 2.0 Contained in Unisys IRM Unit 11 Work Plan Appendix B 11/8/91

Table 2-2

Detected Compounds in Influent Gas Stream

Compound	Mass Detected (ug)	Corrected Mass Detected (ug)	Concentration (ppmv)	Percent of Flow
Benzene	181	206	2	0.013
2-Butanone	19	28	0.2	0.001
Chloroform	93	137	0.7	0.008
1,2-Dichlorobenzene	75	97	0.4	0.006
1,2-Dichloroethane	26	42	0.3	0.003
1,1-Dichloroethene	1,020	1,522	10	0.095
1,2-Dichloroethene (total)	58,075	105,591	666	6.57
Ethylbenzene	1,147	1,318	8	0.082
Methylene Chloride	995	880	6	0.055
Tetrachloroethene	225,225	214,500	790	13.34
Toluene	41,618	44,750	206	2.78
1,1,1-Trichloroethane	2,234	2,510	9	0.156
Trichloroethene	1,344,638	1,120,532	5,232	69.71
Xylene	6,063	9,624	55	0.598
Freon 113	105,740	105,740	610	6.58
Naphthalene	16	19	0.09	0.001
Total	1,787,149	1,607,477	7,596	100



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above the roof of the building. Exit velocity of the vapor stream is expected to be approximately 1,400 ft/min.

3.0 SCOPE OF WORK

RETEC will perform emission tests on the vapor control system to measure the removal efficiency for selected VOCs, SVOC and HCL. Sampling procedures will follow methods established by the U.S. EPA under 40 CFR Part 60, *Standards of Performance for New Stationary Sources, Appendix A*. This section details the sampling methodologies to be followed during the emission testing. Copies of each test method discussed in this section are presented in Appendix A of this Work Plan.

3.1 GAS STREAM SAMPLING LOCATIONS AND CHARACTERIZATION

RETEC will locate gas sampling ports on the vapor control system following the guidelines established by the U.S. EPA. A schematic of sampling ports on the vapor control system are presented in Figures 3-1 and 3-2.

3.1.1 Inlet Sample Locations

An inlet sampling port for VOC and SVOC measurement will be located in the exhaust pipe (8-inch diameter) from the soil vapor extraction system leading to the catalytic oxidizer. The port will be located downstream of any potential air dilution streams to characterize the gas stream as it enters the heat exchanger. Inlet HCL sampling will not be performed because the calculation of a removal efficiency is not required under NYDEC Regulation 212.

3.1.2 Outlet Sample Locations

The combined outlet sampling ports for VOC, SVOC and HCL measurements will be located in the exhaust stack (8-inch diameter) from the inorganic gas scrubber.

3.1.3 Gas Stream Characterization

Gas velocity and measurement will be performed at the inlet and outlet sampling ports. Field inspection of the catalytic oxidizer revealed the inlet sample location to be only two duct diameters downstream from the air dilution valve and the system blower. Engineer any design of the off-gas control system makes it impossible to increase the distance further. The combined outlet sampling ports for VOC, SVOC and HCL measurements will be located at least eight duct diameters downstream from any flow disturbances.



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Figure 3-2 VOC and HCL Outlet Sample Point

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Gas velocities will be measured with a standard pitot tube and manometer. The number of traverse points and the verification of absence of cyclonic flow will be performed following the details presented in Method 1A, Sample and Velocity Traverses for Stationary Sources with Small Stacks or Ducts and Method 2C, Determination of Stack Gas Velocity and Volumetric Flow Rate in Small Stacks or Ducts.

The inlet VOC sample location to the catalytic oxidizer will have a maximum of sixteen traverse points (Figure 1-2 of U.S. EPA Method 1) to measure gas velocities in the duct. The outlet location for VOC, SVOC and HCL measurements will have eight to twelve traverse points.

Gas velocities will be measured before and after each inlet and outlet sample run. Initial and final gas velocities will be reported separately and then averaged for each sample run.

Temperature of the inlet and outlet gas streams will be measured using a thermocouple capable of measuring temperatures to within 1.5 percent of the minimum absolute stack temperature. Barometric pressure will be obtained from a near by National Weather Service station or local airport, corrected for elevation. Any corrections to the barometric pressure will be applied at a rate of 0.1 inches of Mercury (Hg) per 100 foot elevation increase or decrease.

The dry molecular weight for the outlet gas stream will follow the procedures detailed in Method 3, *Gas Analysis for the Determination of Dry Molecular Weight*. The dry molecular weight of the vapor control off-gas will use 29.0 grams/gram mole (air).

The percent moisture of the gas stream will be calculated by condensation and/or wet bulb-dry bulb techniques, as per Method 4, *Determination of Moisture Content in Stack Gas*.

3.2 DETERMINATION OF VOC CONCENTRATIONS

The results from the Unisys pilot demonstration program indicated that nine VOCs (presented in Section 2.0, Table 2-1) were likely to be present in the vapor stream in significant concentrations. In a letter dated February 5, 1993, the NYDEC requested that the list be expanded to inlcude other volatile and semi-volatile organic constituents. The requested NYDEC target compound list (TCL) is presented in Table 3-1.

TABLE 3-1

NYDEC Target Compound List

Compound	Compound
1,1-Dichloroethane	Bromoform
1,1-Dichloroethylene	Bromomethane
1,1,2-Trichloroethane	Carbon Tetrachloride
1,1,2,2-Tetrachloroethane	Carbon Disulfide
1,2-Dibromo-3-chloropropane	Chlorobenzene
1,2-Dibromoethane	Chloroethane
1,2-Dichlorobenzene	Chloroform
1,2-Dichloroethylene(total)	Chloromethane
1,2-Dichloropropane	cis-1,3-Dichloropropene
1,3-Dichlorobenzene	Dibromochloromethane
1,4-Dichlorobenzene	Ethyl Benzene
2-Butanone	Styrene
2-Hexanone	Total Xylenes
4-Methyl-2-pentanone	trans-1,3-Dichloropropene
Bromodichloromethanex	Freon-113
Anenaphthene	Aroclor 1232
Acenaphthylene	Aroclor 1242
Anthracene	Aroclor 1248
Benzo(a)anthracene	Aroclor 1254
Benzo(b)fluoranthene	Aroclor 1260
Benzo(k)fluoranthene	Hexachlorobenzene
Benzoic Acid	Hexachlorobutadiene
Benzo(g,h,i)perylene	Hexachlorocyclopentadiene
Benzo(a)pyrene	Hexachloroethane
Benzyl alcohol	Indeno(1,2,3-cd)pyrene
Bis(2-chloroethyl)ether	Isophorone
Bis(2-chloroisopropyl)ether	2-MethInaphthalene
Bis(2-chloroethoxy)methane	Naphthalene
Bis(2-ethylexyl)pthalate	2-Nitroaniline
4-Bromophenyl-phenylether	3-Nitroaniline
Butylbenzylphthalate	4-Nitroaniline
4-Chloroaniline	Nitrobenzene
2-Chloronaphthalene	N-Nitrosodiphenylamine
4-Chlorophenyl-phenylether	N-Nitroso-di-n-propylamine

TABLE 3-1 con't

NYDEC Target Compound List

Chrysene	Phenanthrene				
Dibenz(a,h)anthracene	Pyrene				
Dibenzofuran	1,2,4-Trichlorobenzene				
1,2-Dichlorobenezene	2-Chlorophenol				
1,3-Dichlorobenzene	2,4-Dichlorophenol				
1,4-Dichlorobenzene	2,4-Dimethylphenol				
3,3'-Dichlorobenzidine	4,6-Dinitro-2-methyphenol				
Diethylphthalate	2,4-Dimitrophenol				
Dimethylphthalate	2-Methylphenol				
2,4-Dinitrotoluene	4-Methylphenol				
2,6-Dinitrotoluene	2-Nitrophenol				
Di(n)octyl-phthalate	4-Nitrophenol				
Fluoranthene	Pentachlorophenol				
Fluorene	Phenol				
Aroclor 1016	2,4,5-Trichlorophenol				
Aroclor 1221	2,4,6-Trichlorophenol				

In the interest of efficiency, a preliminary stack testing program was conducted by RETEC to quantitatively measure for compounds listed on the NYDEC TCL in the inlet vapor stream to the catalytic oxidizer. Results of the preliminary stack test would be used to modify the compliance emission monitoring program by adding compounds from the NYDEC list to the list of principal VOC compounds.

The decision to add compounds from the NYDEC TCL would involve their detection in the inlet vapor stream during the preliminary stack test. Results indicated that fifteen VOCs and two SVOCs were detected in the inlet vapor stream during the preliminary stack test. A list of fifteen VOC compounds detected and their concentrations were presented previously in Section 2.0, Table 2-2. The single SVOC detected during the stack test was napthalene.

These fifteen VOCs and the single SVOC will comprise the new list of principal compounds for the compliance monitoring program for organic destruction efficiency at the Unisys oxidizer. These compounds can be easily collected onto activated charcoal tubes. RETEC proposes to use activated charcoal tubes as the sample absorbent media for the collection of principal compounds.

3.2.1 Measurement of Principal Compounds

The concentration of principal compounds in the inlet and outlet gas streams from the catalytic oxidizer will be collected using a leak-less sample pump and carbon adsorption tubes in accordance with U.S. EPA Method 18, *Measurement of Gaseous Organic Compound Emission by Gas Chromatography* and NIOSH Method 1501, *Determination of Organic Solvents in Air*. The carbon tubes will be sent to the analytical laboratory, subsequently desorbed, and analyzed for the specific compounds of interest by gas chromatography.

A preliminary sample analysis survey will be performed on the inlet gas stream with a portable organic vapor analyzer before and during the sample run with the carbon adsorption tubes. The analyzer will measure total VOCs in the gas stream, on a ppmv basis, using a photo ionization detector. The portable analyzer will be calibrated prior to use with a standard concentration of 100 ppmv isobutylene mixed with air. This qualitative analysis will provide an appropriate loading rate and subsequent total sample volume for the charcoal adsorbent tubes to prevent constituent break-through. However, a minimum sample duration of 60 minutes will be used to account for possible variation in exhaust stream conditions.

The measured ppmv concentration (volume/volume) can be converted to a mg/m^3 concentration (mass/volume) using the relationship of 1 ppmv is equal to 2.29 mg/m^3 (at standard conditions).

The flow rate of the sample train will be calculated using the following formula:

$$Q_T = \frac{M_C \times 0.20}{(2.5 \times C_A) \times 2.29 \times \theta}$$

where:

 $Q_T = Sample train flow rate (m³/min)$ $M_C = Mass of carbon (mg)$ $C_A = Concentration of gas stream (ppmv)$ $\Theta = Total run time (minutes)$

The mass of carbon in the tube is assumed to be only 20% efficient in collecting VOCs, so the mass of carbon in the formula is multiplied by 0.20. A relative response factor of 2.5 is used to account for photoionization detector sensitivity.

The flow rate used during the sample collection run will be corrected to dry standard conditions using the following formula:

$$V_{sb}(std) = V_{sb}\left(\frac{T_{std}P_s}{T_sP_{std}}\right)$$

$$Q_{std} = (\frac{V_{sb}(std)}{\theta})$$

where:

V _{ab} (std)	=	Volume of gas as measured by the soap bubble meter corrected to standard conditions (scm)
V _{sb}	=	Volume of gas as measured by the soap bubble meter (cm ³)
Qstd	=	Volumetric flow rate through critical orifice (scm/min)
θ	=	Time (minutes)
Tstd	=	Absolute Temperature (298 ^k)
T,	=	Ambient Temperature $(273^{k} + C^{\circ})$
P _{std}	=	Absolute Pressure (760 mm Hg)

 $P_s = Ambient Pressure (mm Hg)$

The measured concentrations will be used to calculate an appropriate inlet and outlet sampling flow rate for the two sample trains. All inlet and outlet sample trains will operate simultaneously. This procedure will be performed three times to comply with U.S. EPA and NTDEC requirements.

3.2.2 Sampling Train

The sampling trains used for inlet and outlet sampling will consist of a stainless steel probe (approximately ¼ inches in diameter), midget glass impingers to condense water vapor, inert (Teflon) flexible tubing to connect the probe to the midget impingers, and charcoal adsorption tubes. Sample gas will be pulled through the system using either a electric vacuum pump equipped with a limiting flow orifice (high flow conditions) or a battery operated personnel pump (low flow conditions).

The electric vacuum pump, equipped with the limiting flow orifice, has a flow range (in whole units) of 1 to 4 liters per minute ($\pm 15\%$). The personnel pump, equipped with a pressure sensitive flow control needle valve, has a variable flow range of 0.05 to 5.0 liters per minute ($\pm 5\%$).

The calibration of the pump flow rate through the adsorption tubes will be performed with a bubble tube flow meter prior to and at the conclusion of each sample run. Ambient temperature and barometric pressure will be recorded. During sampling, an in-line rotameter and vacuum gauge will be used to verify that the pump flow rate remains constant.

The train will be leak checked before and immediately after each sampling run by plugging the probe and running the sample train beyond its operational vacuum. Leakage rates in excess of 4 percent are unacceptable. Leaks detected before the sample run will be corrected. Completed sample runs with leakage rates in excess of 4 percent with be voided.

The sample probe will be located at the centroid of the duct. The midget impingers will be placed in an ice bath and allowed to cool before sampling. Total sample time, sample flow rate, barometric pressure and temperature will be recorded.

3.2.3 Adsorbent Tubes

The adsorbent tubes, activated charcoal, will be obtained commercially and have the following general specifications: glass tube, 11 cm long, 1 cm OD, 8 mm ID, flamed sealed ends, containing two sections of activated (60° C) coconut shell charcoal (front = 800 mg, back

= 200 mg) separated by a 4 mm urethane foam plug. A glass wool plug precedes the front section and a 4 mm urethane foam plug follows the back section.

Two tubes will be used in series during the monitoring to detect possible constituent breakthrough. The back tube will be connected in the reverse direction such that the 200 mg section is first. This configuration will allow the contents of the first tube (1,000 mg) and the back-half of the second tube (200 mg) to be combined as one sample (1,200 mg). The remaining 800 mg of charcoal in the back tube will be analyzed for break-through at a frequency of 20% of the collected samples. If concentrations of constituents detected in the backup portion exceed 20 percent of the total amount, then break-through has occurred and the run is voided.

3.2.4 Condensates

Liquid condensates collected in the impingers during the sample run will be poured into 40 ml VOA vials. Any available space in the vials will be filled with distilled water before sealing the cap. Sample will be stored and shipped at 4° C.

3.2.5 Sample Identification

Charcoal sample tubes and condensates will be labeled with a unique sample code detailing the location, its position in the sample train (front or back half), sample flow rate, the sample date, and sample run number (1, 2, or 3).

Three sample runs will be conducted on the inlet and outlet vapor stream to the VOC oxidizer. This would correspond to twelve adsorbent tubes (2 trains x 2 sample points x 3 runs) and six condensates to be analyzed for principal compounds. Twenty percent of the collected adsorbent tubes (2) will be analyzed for constituent break-through. Field blanks for the charcoal tubes and condensates will also be collected on-site at a frequency of 20% (2), to raise the number of analysis of the program to eighteen (10 tubes + 8 condensates). Analytical procedures are detailed in Section 5.0.

3.3 DETERMINATION OF HCL CONCENTRATIONS

The concentration of hydrogen chloride in the outlet gas stream at the spray chamber scrubber will be performed using U.S EPA Method 26, *Determination of Hydrogen Chloride Emission* from Stationary Sources. The method involves the passage of sample gas through a dilute sulfuric acid solution by means of a midget impinger. The solution is then measured for chloride ions using ion chromatography.

Three sample runs will be conducted simultaneously on the outlet gas stream at the conclusion of the VOC monitoring program.

3.3.1 Sampling Train

The sampling train will include a probe lined with borosilicate glass, six 30 ml midget impingers with leak free glass connectors, sample line, and a sample pump equipped with a 2 lpm limiting orifice.

The impingers will be connected in series with the knockout impinger first. The first two impingers will contain 15 ml of absorbing solution (0.1 N sulfuric acid). The third and fourth impingers will contain 15 ml of scrubber solution (0.1 N sodium hydroxide). The sixth impinger will act as the drying tube containing fresh silica gel.

The sampling train will be leaked checked before and after each sample run. The probe will be plugged and the sample pump turned on to pull a vacuum of at least 15 inches Hg. Leakage will be detected by the rotameter. Leakage rates in excess of four percent are unacceptable. Leaks detected before the sample run will be corrected. Completed sample runs with leakage rate in excess of four percent will be voided.

The probe will be placed at the centroid of the duct. Sample run times will be 60 minutes. Total sample time, flow rate, barometric pressure, and atmospheric temperature will be recorded.

Sample recovery will be performed by quantitatively transferring the contents of the first three impingers (knockout impinger and the two absorbing solution impingers) to a leak-free storage bottle. Water rinses of each of these impingers and connecting glassware will be added to the storage bottle. The sample bottles will be labeled and the fluid level marked so that if any sample is lost during shipment, a correction proportional to the lost volume could be applied.

Three sample runs will be conducted on the outlet vapor stream from the inorganic vapor scrubber. A field blank will also be performed on-site by placing fresh absorbing solution into a sample jar. This would raise the number of samples to be analyzed to four. Analytical procedures are detailed further in Section 5.0.

4.0 SAMPLE CUSTODY

The purpose of chain-of-custody procedure is to document the identity of the sample and its handling from its first existence as a sample until analysis and data reduction are completed. Custody records trace a sample from its collection through all transfers of custody until it's receipt in the analytical laboratory. Internal laboratory records then document the custody of the sample through its final disposition. RETEC utilizes a three-part carbonless copy custody record following the NEIC format.

All samples submitted to the laboratory will be brought to the Sample Bank Manager who will continue the chain of custody by assigning an appropriate identification number to each sample on receipt; this number identifies the sample through all further handling within the laboratory. The Sample Bank Manager will continue the documented chain of custody and ensure that each handling of the sample is appropriately documented. Copies of the chain of custody form will accompany the samples to the laboratory for sample preparation and analysis.

5.0 LABORATORY ANALYSIS

Samples collected in the field will be shipped back to the analytical laboratory at the end of the sampling program. RETEC will use the analytical services of: New England Testing Laboratory, 1254 Douglas Avenue, North Providence, RI 02904. NY Lab ID: 11382. The following sections detail the analytical procedures to be used on the sample for the constituents of interest.

5.1 PRINCIPAL COMPOUNDS

Upon receipt at the laboratory, the adsorbent tubes will be desorbed using either carbon disulfide or a toluene in accordance with NIOSH Method 1501 and U.S. EPA SW-846 Method 8240. The resulting extract will be analyzed by GC/MS for the constituents of interest.

5.1.1 Desorption Efficiency Test

The laboratory will perform a desorption efficiency (DE) test on the activated carbon for the constituents of interest using carbon disulfide and toluene solvents. The desorption efficiency test will follow the guidelines detailed in NIOSH Method 1501. The DE will be determined by preparing three carbon tubes at five TCL levels plus three media blanks. The desorption test will be performed using the same batch of activated charcoal to be used in the field.

The use of toluene as a desorption solvent will enable the laboratory to detect the following compounds without the elution interference caused by carbon disulfide:

- 2-Butanone
- Chloroform
- Methylene Chloride

Results of the test will reveal the desorption efficiency of carbon disulfide, in addition to toluene to desorb TCL compounds from the charcoal tubes.

5.1.2 TCL Analysis

In general, the contents of both sorbent tubes will be combined in a single vial for analysis. Samples submitted for constituent breakthrough analysis, however, will require a different preparation scheme. In these instances, the contents of the first tube (1000 mg) and the primary

Dectection Limits for Principal Compounds

•

Compound	Detection Limit ug/tube						
Benzene	1						
2-Butanone	10						
Chloroform	1						
1,2-Dichlorobenzene	1						
1,2-Dichloroethane	1						
1,2-Dichloroethene (total)	1						
Ethylbenzene	1						
Methylene Chloride	3						
Tetrachloroethane	1						
Toluene	1						
1,1,1-Trichloroethene	1						
Trichloroethene	1						
Xylene	1						
Freon 113	1						
Napthalene	1						

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section of the second tube (200mg), will be combined and analyzed. The remaining portion of the second tube (800mg) will be analyzed separately to detect breakthrough.

An appropriate volume of solvent (1-3 ml) will be added to the contents of the vial(s) which will be sealed immediately using a crimp cap. The covered vial will be set aside for a minimum of thirty minutes, with occasional agitation.

The extracted samples will be analyzed by GC/MS. The GC will be calibrated daily with a minimum of five working standards over the range of anticipated constituent concentrations. The results of these analysis will provide concentration data (ug/tube) for the specific indicator compounds. Detection limits for TCL compounds are listed in Table 5-1.

Liquid condensates will be analyzed by GC/MS following the procedures detailed in U.S EPA SW-846 Method 8240. Volatile compounds will be introduced into the gas chromatograph by purge and trap method. An inert gas is bubbled through the solution at ambient temperature were the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapors are swept through a sorbent column where the volatile components are trapped. After the purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components into the GC. The gas chromatograph separates the components of interest which are quantified by the mass spectrometer. The final result will be presented in μg /sample.

Detected sample compounds in the sample charcoal tube and corresponding condensate will be summed to obtain a total compound specific concentration in milligrams (ug) for the sample run. A final concentration, in ug/m³, will be calculated by dividing the detected mass by the total volume sampled, corrected to dry standard conditions as detailed in Section 6.0.

5.2 HCL ANALYSIS

Samples received for HCL analysis will be performed using ion chromatography. The instrument will be calibrated with standards covering an appropriate concentration range, starting with the lowest concentration standard. Duplicate injections of QC sample, followed by a water blank and the field sample will be performed. A final injection of the calibration standard will be made to allow for compensation of any drift in the instrument during analysis of the field samples.

The peak Cl area will be measured for all sample injections. The average response from duplicate samples will be used to determine the field sample concentrations by comparing it to

a linear calibration curve generated from the standards. The concentration of Cl⁻ in the sample will be reported in μ g/ml.

The mass of HCl in the sample (μg) will be calculated by multiplying the Cl⁻ concentration (blank corrected) by the molecular weight of HCl ($\mu g/\mu g$ -mole) and then dividing the product by the atomic weight of Cl ($\mu g/\mu g$ -mole). A final concentration, in mg/m³, will be calculated by dividing the mass the total volume sampled, corrected to dry standard conditions.

6.0 FINAL REPORT

A final report will be generated within forty-five days after sample collection. The report will include all field data sheets concerning sample flow rates and duration, ambient temperature and barometer pressure. Calculations concerning gas flow rates will also be presented.

The final report will focus on the ability of the vapor control system to remove the principal compounds and HCL from the SVE stream. All field calibrations, analytical results, and recorded field data will be presented in an appropriate format (tabular form), or in an associated appendix to the report.

6.1 CALCULATION OF GAS VELOCITY AND FLOW RATE

The calculation to determine the gas velocities within the inlet and outlet gas streams is as follows:

$$V_{s} = K_{p}C_{p}(\sqrt{\Delta P}) \frac{\sqrt{T_{s}(avg)}}{P_{s}M_{s}}$$

where:

 V_s = Average stack velocity (ft/sec)

 K_{p} = Pitot tube constant (.99)

 C_p = Molecular weight of stack gas (lb/lb mole)

 $\Delta P = Velocity$ head of stack gas (inches H₂O)

 $T_s = Stack$ temperature (°C)

 $P_s = Absolute stack pressure (inches Hg)$

 $M_s = Molecular$ weight of stack gas (lb/lb mole)

The equation used to determine the gas flow rates will be as follows:

$$Q_{sd} = 3,600 (1-Bws) V_s A \left(\frac{Tstd P_s}{Ts(avg) Pstd} \right)$$

where:

Q_{sd}

Dry volumetric stack gas flow rate corrected to standard conditions (dscf/hr)

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Bws	=	Water vapor in the gas stream, proportion by volume
V _s	=	Average stack velocity (fl/sec)
Α	=	Cross sectional area of stack
Tstd	=	Standard absolute temperature (293°K)
T,	=	Absolute stack temperature (°C)
P _s	=	Absolute stack pressure (in.Hg)
Pstd	=	Standard absolute pressure (29.92 in.Hg)

6.2 CALCULATION OF SAMPLE VOLUMES

Mass concentrations of constituents, reported from the analytical results, will be combined with their respective total sample volumes to be presented in mg/m³. Measured flow rates of the inlet and outlet stack gases will also be used to present constituent emission rates in lbs/hr. The final flow rate used during VOC and HCL sample runs will be corrected to dry standard conditions using the formula detailed in Section 3.2.2. Total volume sampled will be computed as:

6.2.1 Calculation of Removal Efficiency

Removal efficiencies of the system will be calculated by the following equation:

$$RE = \frac{C_i - C_o}{C_i} \times 100$$

where:

RE = removal efficiency in percent

 C_i = emission rate (lb/hr) of constituent in the inlet stream

 $C_o =$ emission rate (lb/hr) of constituent in the outlet stream

Appendix A U.S. EPA Test Methods 40 CFR Part 60, Appendix A

40 CFR Ch. I (7-1-90 Edition)

the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section title "Test Methods and Procedures" is provided to: (1) Identify the test methods to be used as reference methods to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a test method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a test method as a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the test methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the test methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods. (2) alternative methods; and (3) minor changes in the methodology of the test methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the test methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the test methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as

"subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the test methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the the degree of detail should be similar to the detail contained in the test methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

METHOD 1--SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle, To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m^{3} (113 in.²) cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

2. Procedure

2.1 Selection of Measurement Site, Sampling or velocity measurement is performed at a site located at least eight stack or duct

Environmental Protection Adency

diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_i) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_{\bullet} = \frac{2LW}{(L+W)}$$

where L =length and W =width.

An alternative procedure is available for determining the acceptability of a measurement location not meeting the criteria above. This precedure, determination of gas flow angles at the sampling points and comparing the results with acceptability criteria, is described in Section 2.5.

2.2 Determining the Number of Traverse Points.

DUCT DIAMETERS UPSTREAM FROM FLOW DISTURBANCE (DISTANCE A)



Figure 1-1. Minimum number of traverse points for particulate traverses.

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however determine the distances from the choser measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter of equivalent diameter, to determine the dis tance in terms of the number of duct diame ters. Then, determine from Figure 1-1 the minimum number of traverse points tha corresponds: (1) to the number of duct di ameters upstream; and (2) to the number o diameters downstream. Select the higher o

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the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

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TABLE 1-1. CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

Number of traverse points	Matrix layout
9) 3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7





Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples,

see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition for stacks having diameters greater than 0.61 m (24 in.) no traverse

Environmental Protection Agency

points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) : distance equal to the nozzle inside diameter whichever is larger. These relocated traverse points (on each end of a diameter shall be the "adjusted" traverse points.

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Whenever two successive traverse point are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sam pling (or velocity measurement) procedure and in recording the data.





TABLE 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

[Percent of stack diameter from inside wall to traverse point]

Traverse point number on a diameter	Number of traverse points on a diameter-											
	2	4	6	8	10	12	14	16	18	20	22	2
1	14.6	6,7	4,4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1 3
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	:
3		75.0	29.6	19.4	14.6	11.B	9.9	8.5	7.5	6.7	6.0	1 5
•		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10
5			95.6	80.6	65.8	35.6	26.9	22.0	18,8	16.5	14.6	13
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16
B				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23
10			[1	97.4	88.2	79.9	71.7	61.8	38.8	31.5	27
11						93.3	85.4	78.0	70 4	61.2	39.3	32
12						97.9	90.1	83.1	76.4	69.4	60.7	39
13							94 3	87.5	A1 2	75.0	68.5	60
14							0.00	01.5	954	70.6	72.9	62
16							50.2	06.1	80.1	19.0	79.0	70
4.4				1				00.4	0.00	07.1	10.2	22
• 7	*********		•••••		•••••			30.4	92.5	07.1	02.0	
· ·									95.0	90.3	05.4	
18	••••••	•••••						••••••	98.6	93.3	88.4	83
19			• • • • • • • • • • • • • • • •	J				·····		96.1	91.3	86
20	•••••	•••••						•••••		98.7	94.0	89
21								·····			96.5	92
22		[[[[[98.9	94
23												96
24									•••••			96

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points. expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4x3"minimum number of points" matrix were expanded to 36 points, the final matrix could be 9x4 or 12x3, and would not necessarily have to be 6x6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal arca.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.





Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0" reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0' reference, rotate the pitot tube (up to $\pm 90^{\circ}$ yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α ; assign a values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of a is greater than 20°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

The alternative procedure described in Section 2.5 may be used to determine the rotation angles in lieu of the procedure described above.

2.5 Alternative Measurement Site Selection Procedure. This alternative applies to sources where measurement locations are less than 2 equivalent stack or duct diameters downstream or less than ½ duct diameter upstream from a flow disturbance. The alternative should be limited to ducts larger than 24 in. in diameter where blockage and wall effects are minimal. A directional flowsensing probe is used to measure pitch and yaw angles of the gas flow at 40 or more traverse points; the resultant angle is calculated and compared with acceptable criteria for mean and standard deviation.



Note: Both the pitch and yaw angles are measured from a line passing through the traverse point and parallel to the stack axis. The pitch angle is the angle of the gas flow component in the plane that INCLUDES the traverse line and is parallel to the stack axis. The yaw angle is the angle of the gas flow component in the plane PERPENDIC-ULAR to the traverse line at the traverse point and is measured from the line passing through the traverse point and parallel to the stack axis.

2.5.1 Apparatus.

2.5.1.1 Directional Probe. Any directional probe, such as United Sensor Type DA Three-Dimensional Directional Probe, capable of measuring both the pitch and yaw angles of gas flows is acceptable. (Note: Mention of trade name or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Assign an identification number to the directional probe, and permanently mark or engrave the number on the body of the probe. The pressure holes of directional probes are susceptible to plugging when used in particulate-laden gas streams. Therefore, a system for cleaning the pressure holes by "backpurging" with pressurized air is required.

2.5.1.2 Differential Pressure Gauges. Inclined manometers, U-tube manometers, or other differential pressure gauges (e.g., magnehelic gauges) that meet the specifications described in Method 2, section 2.2.

NOTE: If the differential pressure gauge produces both negative and positive readings, then both negative and positive pressure readings shall be calibrated at a minimum of three points as specified in Method 2, section 2.2.

2.5.2 Traverse Points. Use a minimum of 40 traverse points for circular ducts and 42 points for rectangular ducts for the gas flow angle determinations. Follow section 2.3 and Table 1-1 or 1-2 for the location and layout of the traverse points. If the measurement location is determined to be acceptable according to the criteria in this alternative procedure, use the same traverse point number and locations for sampling and velocity measurements.

2.5.3 Measurement Procedure.

2.5.3.1 Prepare the directional probe and differential pressure gauges as recommended by the manufacturer. Capillary tubing or surge tanks may be used to dampen pressure fluctuations. It is recommended, but not required, that a pretest leak check be conducted. To perform a leak check, pressurize or use suction on the impact opening until a reading of at least 7.6 cm (3 in.) H_1O registers on the differential pressure gauge, then plug the impact opening. The pressure of a leak-free system will remain stable for at least 15 seconds.

2.5.3.2 Level and zero the manometers. Since the manometer level and zero may drift because of vibrations and temperature changes, periodically check the level and zero during the traverse.

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2.5.3.3 Position the probe at the appropriate locations in the gas stream, and rotate until zero deflection is indicated for the yaw angle pressure gauge. Determine and record the yaw angle. Record the pressure gauge readings for the pitch angle, and determine the pitch angle from the calibration curve. Repeat this procedure for each traverse point. Complete a "back-purge" of the pressure lines and the impact openings prior to measurements of each traverse point.

A post-test check as described in section 2.5.3.1 is required. If the criteria for a leak-free system are not met, repair the equipment, and repeat the flow angle measurements.

2.5.4 Calculate the resultant angle at each traverse point, the average resultant angle, and the standard deviation using the following equations. Complete the calculations retaining at least one extra significant figure beyond that of the acquired data. Round the values after the final calculations.

2.5.4.1 Calculate the resultant angle at each traverse point:

 $\mathbf{R}_i = \operatorname{arc} \operatorname{cosine} \left[(\operatorname{cosine} Y_i) (\operatorname{cosine} P_i) \right]$

Eq. 1-2

Where:

R_i=Resultant angle at traverse point i, degree.

 $Y_i = Yaw$ angle at traverse point i, degree.

 P_i = Pitch angle at traverse point i, degree.

2.5.4.2 Calculate the average resultant for the measurements:

$$\hat{R} = \frac{\Sigma R_i}{n} \qquad \qquad Eq. 1-3$$

Where:

R=Average resultant angle, degree.
n=Total number of traverse points.
2.5.4.3 Calculate the standard deviations:



Eq. 1-4

Where:

a=S rd def n, def 2.5.5 The measurement location is acentable if $R < 20^{\circ}$ and $S < 10^{\circ}$.

2.5.6 Calibration. Use a flow system as escribed in Sections 4.1.2.1 and 4.1.2.2 of fethod 2. In addition, the flow system shall ave the capacity to generate two test-secon velocities: one between 365 and 730 m/ lin (1200 and 2400 ft/min) and one beween 730 and 1100 m/min (2400 and 3600 t/min).

2.5.6.1 Cut two entry ports in the test action. The axes through the entry ports hall be perpendicular to each other and tersect in the centroid of the test section. 'he ports should be elongated slots parallel o the axis of the test section and of suffilent length to allow measurement of pitch ngles while maintaining the pitot head poition at the test-section centroid. To faciliate alignment of the directional probe uring calibration, the test section should e constructed of plexiglass or some other ransparent material. All calibration measrements should be made at the same point a the test section, preferably at the cenrold of the test-section.

2.5.6.2 To ensure that the gas flow is parllel to the central axis of the test section, ollow the procedure in Section 2.4 for cylonic flow determination to measure the as flow angles at the centroid of the test ection from two test ports located 90° .part. The gas flow angle measured in each ort must be $\pm 2^{\circ}$ of 0°. Straightening vanes hould be installed, if necessary, to meet his criterion.

2.5.6.3 Pitch Angle Calibration. Perform calibration traverse according to the manifacturer's recommended protocol in 5° inrements for angles from -60° to $+60^{\circ}$ at me velocity in each of the two ranges speciled above. Average the pressure ratio alues obtained for each angle in the two low ranges, and plot a calibration curve with the average values of the pressure atio (or other suitable measurement factor is recommended by the manufacturer) /ersus the pitch angle. Draw a smooth line hrough the data points. Plot also the data /alues for each traverse point. Determine the differences between the measured data values and the angle from the calibration curve at the same pressure ratio. The difference at each comparison must be within 2° (or angles between 0° and 40° and within 3° for angles between 40° and 60°.

2.5.6.4 Yaw Angle Calibration. Mark the three-dimensional probe to allow the determination of the yaw position of the probe. This is usually a line extending the length of the probe and aligned with the impact opening. To determine the accuracy of measurements of the yaw angle, only the zero or null position need be calibrated as follows. Place the directional probe in the test section. and rotate the probe until the hosible 'f found With a protector of other angle measuring device, however the angle indicated by the yaw angle indicator on the three-dimensional probe. This should be within 2' of 0'. Repeat this measurement for any other points along the length of the pitot where yaw angle measurements could be read in order to account for variations in the pitot markings used to indicate pitot head positions.

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METHOD 1A-SAMPLE AND VELOCITY TRA-VERSES FOR STATIONARY SOURCES WITH SMALL STACKS OR DUCTS

1. Applicability and Principle

1.1 The applicability and principle of this method are identical to Method 1, except this method's applicability is limited to stacks or ducts less than about 0.30 meter (12 in.) in diameter or 0.071 m^2 (113 in.²) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter or 0.0081 m^2 (12.57 in.²) in cross-sectional area. 1.2 In these small diameter stacks or ducts, the conventional Method 5 stack assembly (consisting of a Type S pitot tube attached to a sampling probe, equipped with a nozzle and thermocouple) blocks a signifi-

an ause accur mean ents. Therefore, for particulate matter (FM) sampling in small stacks or ducts, the gas velocity is measured using a standard pitot tube downstream of the actual emission sampling site. The straight run of duct between the PM sampling and velocity measurement sites allows the flow profile, temporarily disturbed by the presence of the sampling probe, to redevelop and stabilize.

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1.3 The cross-sectional layout and location of traverse points and the verification of the absence of cyclonic flow are the same as in Method 1, Sections 2.3 and 2.4, respectively. Differences from Method 1, except as noted, are given below.

2. Procedure

2.1 Selection of Sampling and Measurement Sites.

2.1.1 PM Measurements. Select a PM sampling site located preferably at least 8 equivalent stack or duct diameters downstream and 10 equivalent diameters upstream from any flow disturbances such as bends, expansions, or contractions in the stack, or from a visible flame. Next, locate the velocity measurement site 8 equivalent diameters downstream of the PM sampling site. See Figure 1A-1. If such locations are not available, select an alternative PM sampling site that is at least 2 equivalent stack or duct diameters downstream and 2½ diameters upstream from any flow disturbance. Then, locate the velocity measurement site 2 equivalent diameters downstream from the PM sampling site. Follow Section 2.1 of Method 1 for calculating equivalent diameters for a rectangular cross section.



2.2 Determining the Number of Traverse Points.

2.2.1 PM Sampling. Use Figure 1-1 of Method 1 to determine the number of traverse points to use at both the velocity measurement and PM sampling locations. Before referring to the figure, however, determine the distances between both the velocity measurement and PM sampling sites to the nearest upstream and downstream disturbances. Then divide each distance by the stack diameter or equivalent diameter to express the distances in terms of the number of duct diameters. Next, determine the number of traverse points from Figure 1-1 of Method 1 corresponding to each of these four distances. Choose the highest of the four numbers of traverse points (or a greater number) so that, for circular ducts, the number is a multiple of four, and for rectangular ducts, the number is one of those shown in Table 1-1 of Method 1. When the optimum duct diameter location criteria can be satisfied, the minimum number of traverse points required is eight for circular ducts and nine for rectangular ducts,

2.2.2 PM Sampling (Steady Flow) or Velocity Measurements. Use Figure 1-2 of Method 1 to determine the number of traverse points, following the same procedure used for PM sampling traverses as described in Section 2.2.1 of Method 1. When the optimum duct diameter location criteria can be satisfied, the minimum number of traverse

3. Bibliography

1. Same as in Method 1, Section 3, Citations 1 through 6.

2. Vollaro, Robert F. Recommended Procedure for Sample Traverses in Ducts Smaller Than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, North Carolina. January 1977.

METHOD 2-DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. Principle and Applicability

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausschelbe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1, Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

641



2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter (dimension D_r Figure 2-2b) be between 0.48 and 0.95 centimeter (\aleph_0 and ϑ_i inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_A and P_B Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external

tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.







(c)

Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

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Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of Cp(s) so long as $\alpha 1$ and $\alpha 2 < 10^{\circ}$, $\beta 1$ and $\beta 2 < 5^{\circ}$, z < 0.32 cm (1/8 in.) and w < 0.08 cm (1/32 in.) (citation 11 in Section 6).

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at the final 'traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (± 5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative Δp readings shall be taken, as above, for the last two back purges at which suitably high Δp readings are observed.

Environmental Protection Agency

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. HrO divisions on the 0-to 1-in, inclined scale, and 0.1-in. HO divisions on the 1- to 10-in, vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H₂O. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H₂O; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H₂O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H₁O. Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:



Where:

 $\Delta p_i =$ Individual velocity head reading at a traverse point, mm H₂O (in, H₂O).

n =Total number of traverse points.

K=0.13 mm H₂O when metric units are used and 0.005 in. H₂O when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE: If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Ap values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge, capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced. the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A plezonieter tube and mercury- or water-filled Utube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pilot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube
designed according to the criteria given ln 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01 .

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 D), equally spaced in a piezometer ring configuration.

40 CFR Ch. I (7-1-90 Edition)

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the singlevelocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H₂O (0.605 in. H₂O). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O) for Δp values between 1.3 and 25 mm H₂O (0.05 and 1.0 in. H₂O), and to the nearest 1.3 mm H₂O (0.05 in. H_1O) for Δp values above 25 mm H_2O (1.0 in. H_1O). A special, more sensitive gauge will be required to read Δp values below 1.3 mm H₂O [0.05 in. H₂O] (see Citation 18 in Section 6).



Pt. 60, App. A, Meth. 2

3. Procedure

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot. tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H₂O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds: (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H.O. Other leakcheck procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may 40 CFR Ch. I (7-1-90 Edition)

drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

Environmental Protection Agency

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PLANT	
DATE	RUN NO
STACK DIAMETE	R OR DIMENSIONS, m(in.)
BAROMETRIC PR	ESSURE, mm Hg (in. Hg)
CROSS SECTIONA	L AREA, m ² (ft ²)
OPERATORS	·
PITOT TUBE L.D. I	
AVG. COEFFIC	IENT, Cp =

LAST DATE CALIBRATED___

SCHEMATIC OF STACK CROSS SECTION

Transie	Val Med An	st Md. An			
Pt. No.	mm (in.) H20	ts, °C (°F)	T ₅ , ^e K (^e R)	mm Hg (in.Hg)	VΔp
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	4	Average			



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3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO₃, O₃, CO, and N₃, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4. Calibration

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_i , Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B , Figure 2-2b). If D_f is between 0.48 and 0.95 cm (%, and % in.) and if P. and P. are equal and between 1.05 and 1.50 D_i , there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (Isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).

40 CFR Ch. I (7-1-90 Edition)

If D_i , P_A , and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

4.1.1 Type S Pitct Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used: in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6): therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (% and % in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitotprobe sheath) shall be measured and record-

ed. NOTE: Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features: Environmental Protection Agency

Pt. 60, App. A, Meth. 2







Figure 2-6. Proper pitot tube-sampling nozzle configuration to prevent aerodynamic interfer ence; buttonhook-type nozzle; centers of nozzle and pitot opening aligned; D_i between 0.4*i* and 0.95 cm (γ_{16} and γ_{6} in.).



Environmental Protection Agency

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$D = \frac{2LW}{2LW}$	Eg. 2-1
L^{+} (L+W)	•

Where: $D_{t} = Equivalent diameter$ L = LengthW = W i d t h

prevent interference;

separation needed to

Figure 2-8. Minimum pitot-sample probe separati $D_{\rm f}$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

Figure 2-8.

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To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

NOTE: The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 ft/min) will generally be valid to within ± 3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to within ± 5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 ft/min). If a more precise correlation between C, and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/ min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downPt. 60, App. A, Meth. 2

stream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read Δp_{std} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_{t} and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

Pt. 60, App. A, Meth. 2

40 CFR Ch. I (7-1-90 Edition)

PITOT TUBE IDENTIFICATION NUMBER: ______ DATE: _____ DATE: _____

CALIBRATED BY: __

	"A" SI			
RUN NO.	△ Pstd cm H20 (in. H20)	△ p(s) cm H20 (in. H20)	Cp(s)	DEVIATION $C_{p(s)} \cdot \overline{C}_{p}(A)$
1				
2				
3				
	·	Ēp (SIDE A)		

	"B" SIDE CALIBRATION			
RUN NO.	△ Pstd cm H20 (in, H20)	△ ₽(s) cm H20 (in. H20)	C _{p(s)}	OEVIATION $C_{p(s)} \cdot \overline{C}_{p}(B)$
1				
2	··	 		
3	<u>-</u>	1		
		Cp (SIDE B)		



 $\left| \widetilde{C}_{p} (SIDE A) - \widetilde{C}_{p} (SIDE B) \right| \rightarrow MUST BE \leq 0.01$



Environmental Protection Agency

$$C_{p(s)} = C_{p(s)(s)} \sqrt{\frac{\Delta p_{s(s)}}{\Delta p_{s}}}$$

Equation 2-2

Where:

- $C_{P(u)}$ =Type S pitot tube coefficient $C_{P(u)}$ =Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and
- the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.
- $\Delta p_{,id}$ = Velocity head measured by the standard pitot tube, cm H₂O (in. H₂O)
- Δp_e=Velocity head measured by the Type S pitot tube, cm H₃O (in. H₃O)

4.1.4.2 Calculate C_p (side A), the mean Aside coefficient, and C_p (side B), the mean Bside coefficient: calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{\mu\nu}$, from \tilde{C}_{ρ} (side A), and the deviation of each B-side value of $C_{\rho\nu}$, from \tilde{C}_{ρ} (side B). Use the following equation:

Deviation =
$$C_{p(r)} - \overline{C}_p(A \text{ or } B)$$

Equation 2-3

4.1.4.4 Calculate σ , the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma \text{ (side A or B)} = \frac{\sum_{1}^{3} |C_{p(s)} - \overline{C}_{p}(A \text{ or } B)|}{3}$$
Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the values of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between C_p (A) and C_b (B) is 0.01 or less.

4.1.5 Special considerations.4.1.5.1 Selection of calibration point.

4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., C_p (side A) and C_p (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the

pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projectedarea model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of freespace between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or $\frac{1}{4}$ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3.000 ft/ min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 4.1.4.4). Eq. 2-6 P_{att}=Standard absolute pressure, 760 mm Hg (29.92 in, Hg).

Q_M = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

L = Stack temperature. 'C ('F).

 T_s = Absolute stack temperature, 'K, ('R). = 273 + 4 for metric.

=460+4 for English.

Eq. 2-8 T_{sis} =Standard absolute temperature, 293 'K (528' R).

v.=Average stack gas velocity, m/sec (ft/ sec).

 Δp =Velocity head of stack gas, mm H₂O (in. H₂O).

3.600 = Conversion factor, sec/hr.

18.0=Molecular weight of water, g/g-mole (lb/lb-mcle).

5.2 Average Stack Gas Velocity.

 $v_{s} = K_{p}C_{p}(\sqrt{\Delta p})_{a^{\star}e}\sqrt{\frac{T_{s(a^{\star}e)}}{P_{s}M_{s}}}$

Equation 2-9

Eq. 2-7

5.3 Average Stack Gas Dry Volumetric Flow Rate.

$$Q_{id} = 3,600(1 - B_{ics})v_i A = \frac{T_{ild}}{T_r(avg)} = \frac{P_i}{P_{ild}}$$

To convert Q_{id} from dscm/hr (dscf/hr) to dscm/min (dscf/min), divide Q_{id} by 60.

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Environmental Protection Agency

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METHOD 2A-DIRECT MEASUREMENT OF GAS VOLUME THROUGH PIPES AND SMALL DUCTS

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of gas flow rates in pipes and small ducts, either in-line or at exhaust positions, within the temperature range of 0 to 50° C.

1.2 Principle. A gas volume meter is used to measure gas volume directly. Temperature and pressure measurements are made to correct the volume to standard conditions.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Gas Volume Meter. A positive displacement meter, turbine meter, or other direct volume measuring device capable of measuring volume to within 2 percent. The meter shall be equipped with a temperature gauge (± 2 percent of the minimum absolute temperature) and a pressure gauge (± 2.5 mm Hg). The manufacturer's recommended capacity of the meter shall be sufficient for the expected maximum and minimum flow rates at the sampling conditions. Temperature, pressure, corrosive characteristics, and pipe size are factors necessary to consiler in choosing a suitable gas meter.

2.2 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg. In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested, and an adjustPt. 60, App. A, Meth. 2A

ment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg per 30-meter elevation increase, or viceversa for elevation decrease.

2.3 Stopwatch. Capable of measurement to within 1 second.

3. Procedure

3.1 Installation. As there are numerous types of pipes and small ducts that may be subject to volume measurement, it would be difficult to describe all possible installation schemes. In general, flange fittings should be used for all connections wherever possible. Gaskets or other seal materials should be used to assure leak-tight connections. The volume meter should be located so as to avoid severe vibrations and other factors that may affect the meter calibration.

3.2 Leak Test. A volume meter installed at a location under positive pressure may be leak-checked at the meter connections by using a liquid leak detector solution containing a surfactant. Apply a small amount of the solution to the connections. If a leak exists, bubbles will form, and the leak must be corrected.

A volume meter installed at a location under negative pressure is very difficult to test for leaks without blocking flow at the inlet of the line and watching for meter movement. If this procedure is not possible visually check all connections and assure tight seals.

3.3 Volume Measurement.

3.3.1 For sources with continuous, steady emission flow rates, record the initial meter volume reading, meter temperature(s) meter pressure, and start the stopwatch Throughout the test period, record the meter temperature(s) and pressure so that average values can be determined. At the end of the test, stop the timer and record the elapsed time, the final volume reading meter temperature(s), and pressure. Record the barometric pressure at the beginning and end of the test run. Record the data or a table similar to Figure 2A-1.

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Environmental Protection Agency

4.1.6 Field Use and Recalibration. 4.1.6.1 Field Use.

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field. the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the E side coefficient shall be used when the B side faces the flow: alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section. causing a reduction in the effective value of C_{n(1)}. Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration.

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed. restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocou-

ple-potentiometer systems, and oth gauges at a temperature within 10 perce of the average absolute stack temperatu: For temperatures up to 405' C (761' F), u an ASTM mercury-in-glass reference the mometer, or equivalent, as a reference; : ternatively, either a reference thermocour and potentiometer (calibrated by NBS) thermometric fixed points, e.g., ice bath at boiling water (corrected for barometric pre sure) may be used. For temperatures abo 405° C (761° F), use an NBS-calibrated refe ence thermocouple-potentiometer system an alternate reference, subject to the a proval of the Administrator.

If, during calibration, the absolute ter peratures measured with the gauge bei calibrated and the reference gauge agr within 1.5 percent, the temperature da taken in the field shall be considered val Otherwise, the pollutant emission test sh: either be considered invalid or adjustmer (if appropriate) of the test results shall made, subject to the approval of the Admi istrator.

4.4 Barometer. Calibrate the baromet used against a mercury barometer.

5. Calculations

Carry out calculations, retaining at lea one extra decimal figure beyond that of th acquired data. Round off figures after fin calculation.

5.1 Nomenclature.

A = Cross-sectional area of stack. m^{2} (ft²).

 $B_{\rm ex} = Water$ vapor in the gas stream (fro Method 5 or Reference Method 4), pr portion by volume.

 C_{0} = Pitot tube coefficient, dimensionless. $K_{s} =$ Pitot tube constant.

34.97
$$\frac{m}{\text{sec}} \left[\frac{(g/g \cdot \text{mole})(\text{mm Hg})}{(^{\circ}\text{K})(\text{mm H}_2\text{O})} \right]^{1/2}$$

for the metric system and

85.49
$$\frac{ft}{sec} \left[\frac{(lb/lb-mole)(in, llg)}{(^{\circ}R)(in, H_2O)} \right]^{1/2}$$

for the English system.

 M_d = Molecular weight of stack gas, dry bas (see Section 3.6) g/g-mole (lb/lb-mole).

 M_{\star} =Molecular weight of stack gas, we basis, g/g-mole (lb/lb-mole). $= M_d (1 - B_{ws}) + 18.0 B_s$

Eq. 2-

- $P_{\rm bar}$ =Barometric pressure at measuremen site, mm Hg (in, Hg).
- P_g =Stack static pressure, mm Hg (in. Hg).
- P.= Absolute stack gas pressure, mm Hg (ir Hg).

 $= P_{bu} + P_{\theta}$

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Environmental Protection Agency

3.3.2 For sources with noncontinuous, non-steady emission flow rates, use the procedure in 3.3.1 with the addition of the following: Record all the meter parameters and the start and stop times corresponding to each process cyclical or noncontinuous event.

4. Calibration

4.1 Volume Meter. The volume meter is calibrated against a standard reference meter prior to its initial use in the field. The reference meter is a spirometer or liquid displacement meter with a capacity consistent with that of the test meter.

Alternately, a calibrated, standard pitot may be used as the reference meter in conjunction with a wind tunnel assembly. Attach the test meter to the wind tunnel so that the total flow passes through the test meter. For each calibration run, conduct a 4-point traverse along one stack diameter at a position at least eight diameters of straight tunnel downstream and two diameters upstream of any bend, inlet, or air mover. Determine the traverse point locations as specified in Method 1. Calculate the reference volume using the velocity values following the procedure in Method 2, the wind tunnel cross-sectional area, and the run time.

Set up the test meter in a configuration similar to that used in the field installation (i.e., in relation to the flow moving device). Connect the temperature and pressure gauges as they are to be used in the field. Connect the reference meter at the inlet of the flow line, if appropriate for the meter, and begin gas flow through the system to condition the meters. During this conditioning operation, check the system for leaks.

The calibration shall be run over at least three different flow rates. The calibration flow rates shall be about 0.3, 0.6, and 0.9 times the test meter's rated maximum flow rate.

For each calibration run, the data to be collected include: reference meter initial and final volume readings, the test meter initial and final volume reading, meter average temperature and pressure, barometric pressure, and run time. Repeat the runs at each flow rate at least three times.

Calculate the test meter calibration coefficient, Υ_m , for each run as follows:

$$Y_{m} = \frac{(V_{rr} - V_{rr})(t_{r} + 273)}{(V_{mr} - V_{ms})(t_{m} + 273)} \frac{P_{b}}{(P_{b} + P_{s})}$$

Where:

Ym=Test volume meter calibration coefficlent, dimensionless. Pt. 60, App. A, Meth. 2A

V,≃Reference meter volume reading, m³. V_m=Test meter volume reading, m³.

t,=Reference meter average temperature. *C.

 $t_m =$ Test meter average temperature, 'C.

 P_b =Barometric pressure, mm Hg.

 P_{g} = Test meter average static pressure, mm Hg.

f = Final reading for run.

i=Initial reading for run.

Compare the three Y_m values at each of the flow rates tested and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra runs may be required to complete this requirement. If this specification cannot be met in six successive runs, the test meter is not suitable for use. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications are met at all the flow rates, average all the Y_m values from runs meeting the specifications to obtain an average meter calibration coefficient, Y_m .

The procedure above shall be performed at least once for each volume meter. Thereafter, an abbreviated calibration check shall be completed following each field test. The calibration of the volume meter shall be checked by performing three calibration runs at a single, intermediate flow rate (based on the previous field test) with the meter pressure set at the average value encountered in the field test. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of flow as described above.

Nore.—If the volume meter calibration coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the greater value of pollutant emission rate.

4.2 Temperature Gauge. After each test series, check the temperature gauge at ambient temperature. Use an American Society for Testing and Materials (ASTM) mercuryin-glass reference thermometer, or equivalent, as a reference. If the gauge being checked agrees within 2 percent (absolute temperature) of the reference, the temperature data collected in the field shall be considered valid. Otherwise, the test data shall be considered invalid or adjustments of the test results shall be made, subject to the approval of the Administrator.

4.3 Barometer. Calibrate the barometer used against a mercury barometer prior to the field test.

5. Calculations

Eq. 2A-1

out calcules, reing an least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

5.1 Nomenclature.

P.=Barometric pressure, mm Hg.

P_g=Average static pressure in volume meter, mm Hg.

Q.=Gas flow rate, m³/min, standard conditions.

 $T_m = Average$ absolute meter temperature, 'K.

 $V_m =$ Meter volume reading, m³.

 $Y_m = Average meter calibration coefficient, dimensionless.$

f=Final reading for test period.

i=Initial reading for test period.

- s=Standard conditions, 20° C and 760 mm Hg.
- Θ =Elapsed test period time, min.

5.2 Volume.

$$\mathbf{V}_{me} = 0.3853 \ \mathbf{Y}_{m} \ (\mathbf{V}_{mf} - \mathbf{V}_{mi})$$

5.3 Gas Flow Rate.

$$Q_t = \frac{V_{ms}}{\Theta}$$
 Eq. 2A-3

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METHOD 2B-DETERMINATION OF EXHAUST GAS VOLUME FLOW RATE FROM GASOLINE VAPOR INCINERATORS

1. Applicability and Principle

1.1 A stability while makes applies the measurement of exhaust volume how rate from inclnerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). It is assumed that the amount of auxiliary fuel is negligible.

1.2 Principle. The incinerator exhaust flow rate is determined by carbon balance. Organic carbon concentration and volume flow rate are measured at the incinerator inlet. Organic carbon, carbon dioxide (CO_2), and carbon monoxide (CO) concentrations are measured at the outlet. Then the ratio of total carbon at the incinerator inlet and outlet is multiplied by the inlet volume to determine the exhaust volume and volume flow rate.

2. Apparatus

2.1 Volume Meter. Equipment described in Method 2A.

2.2 Organic Analyzers (2). Equipment described in Method 25A or 25B.

2.3 CO Analyzer. Equipment described in Method 10.

2.4 CO₂ Analyzer. A nondispersive infrared (NDIR) CO₂ analyzer and supporting equipment with comparable specifications as CO analyzer described in Method 10.

3. Procedure

 $(\mathbf{P}_{b} + \mathbf{P}_{f})$

T_

Eq. 2A-2
3.1 Inlet Installation. Install a volume meter in the vapor line to incinerator inlet according to the procedure in Method 2A. At the volume meter inlet, install a sample probe as described in Method 25A. Connect to the probe a leak-tight, heated (if necessary to prevent condensation) sample line (stainless steel or equivalent) and an organic analyzer system as described in Method 25A or 25B.

3.2 Exhaust Installation. Three sample analyzers are required for the incinerator exhaust: CO_2 , CO, and organic analyzers. A sample manifold with a single sample probe may be used. Install a sample probe as described in Method 25A. Connect a leak-tight heated sample line to the sample probe. Heat the sample line sufficiently to prevent any condensation.

3.3 Recording Requirements. The output of each analyzer must be permanently recorded on an analog strip chart, digital recorder, or other recording device. The chart speed or number of readings per time unit must be similar for all analyzers so that data can be correlated. The minimum data recording requirement for each analyzer is one measurement value per minute.

3.4 Preparation. Prepare and calibrate all equipment and analyzers according to the procedures in the respective methods. For the CO, analyzer, follow the procedures described in Method 10 for CO analysis substituting CO, calibration gas where the method calls for CO calibration gas. The part when for the CO, analyzer shall be 15 percent by volume. All analyzer shall be 15 must be introduced at the connection between the probe and the sample line. If a manifold system is used for the exhaust analyzers, all the analyzers and sample pumps must be operating when the calibrations are done. Note: For the purposes of this test, methane should not be used as an organic calibration gas.

3.5 Sampling. At the beginning of the test period, record the initial parameters for the inlet volume meter according to the procedures in Method 2A and mark all of the recorder strip charts to indicate the start of the test. Continue recording inlet organic and exhaust CO2, CO, and organic concentrations throughout the test. During periods of process interruption and halting of gas flow, stop the timer and mark the recorder strip charts so that data from this interruption are not included in the calculations. At the end of the test period, record the final parameters for the inlet volume meter and mark the end on all of the recorder strip charts.

3.6 Post Test Calibrations. At the conclusion of the sampling period, introduce the calibration gases as specified in the respective reference methods. If an analyzer output does not meet the specifications of the method, invalidate the test data for the period. Alternatively, calculate the volume results using initial calibration data and using final calibration data and report both resulting volumes. Then, for emissions calculations, use the volume measurement resulting in the greatest emission rate or concentration.

4. Calculations

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

4.1 Nomenclature.

CO.= Mean carbon monoxide concentration in system exhaust, ppmv.

- CO2e = Mean carbon dioxide concentration in system exhaust, ppmv.
- HC,=Mean organic concentration in system exhaust as defined by the calibration gas, ppmv.
- HC,=Mean organic concentration in system inlet as defined by the calibration gas, ppmv.
- K=Calibration gas factor
- = 2 for ethane calibration gas.
- =3 for propane calibration gas.
- =4 for butane calibration gas.
- = Appropriate response factor for other calibration gas.
- V_{es}=Exhaust gas volume, m².
- V_{is}=Inlet gas volume, m³.
- Q_{es}=Exhaust gas volume flow rate, m³/min.
- Q_µ=Inlet gas volume flow rate, m³/min.

Standard conditions: 2010, 760 mm Hg.

 CO_3 , ppmv. (CO_3 concentration in the ambient air may be measured during the test period using an NDIR).

4.2 Concentrations. Determine mean concentrations of inlet organics, outlet CO_3 , outlet CO_3 and outlet organics according to the procedures in the respective methods and the analyzers' calibration curves, and for the time intervals specified in the applicable regulations. Concentrations should be determined on a parts per million by volume (ppmv) basis.

4.3 Exhaust Gas Volume. Calculate the exhaust gas volume as follows:

$$V_{e_{i}} = V_{i_{i}} - \frac{K(HC_{i})}{K(HC_{e}) + CO_{i_{i}} + CO_{i_{i}} - 300}$$

Eq. 2B-1

4.4 Exhaust Gas Volume Flow Rate. Calculate the exhaust gas volume flow rate as follows:

$$Q_{ii} = V_{ii}/\theta$$

Eq. 2B-2

5. Bibliography

5.1 Measurement of Volatile Organic Compounds. U.S. Environmental Protection Agency. Office of Air Quality Planning and Standards, Research Triangle Park, NC 27711. Publication No. EPA-450/2-78-041. October 1978. 55 p.

METHOD 2C-DETERMINATION OF STACK GAS Velocity and Volumetric Flow Rate in Small Stacks or Ducts (Standard Pitot Tube)

1. Applicability and Principle

1.1 Applicability.

1.1.1 The applicability of this method is identical to Method 2, except this method is limited to stationery source stacks or ducts less than about 0.30 meter (12 in.) in diameter or 0.071 m^2 (113 in.²) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter or 0.0081 m² (12.57 in.³) in cross-sectional area.

1.1.2 The apparatus, procedure, calibration, calculations, and billography are the same as in Method 2, Sections 2, 3, 4, 5, and 6, except as noted in the following sections.

1.2 Principle. The average gas velocity in a stack or duct is determined from the gas density and from measurement of velocity heads with a standard pitot tube.

 $[\]Theta$ =Sample run time, min.

Pt. 60, App. A, Meth. 2C

2. Apparatus

2.1 Standard Pitot Tube (instead of Type S). Use a standard pitot tube that meets the specifications of Section 2.7 of Method 2. Use a coefficient value of 0.99 unless it is calibrated against another standard pitot tube with an NBS-traceable coefficient. 40 CFR Ch. 1 (7-1-90 Edition)

2.2 Alternative Pitot Tube. A modified hemispherical-nosed pitot tube (see Figure 2C-1), which features a shortened stem and enlarged impact and static pressure holes, may be used. This pitot tube is useful in liquid drop-laden gas streams when a pitot "back purge" is ineffective. Use a coefficient value of 0.99 unless the pitot is calibrated as mentioned in Section 2.1 above.

Environmental Protection Agency

Figure 2C-1. Modified hemispherical-nosed pitot tube.



3. Procedure

Follow the general procedures in Section 3 of Method 2, except conduct the measurements at the traverse points specified in Method 1A. The static and impact pressure holes of standard pitot tubes are susceptible to plugging in PM-laden gas streams. Therefore, the tester must furnish adequate proof that the openings of the pitot tube have not plugged during the traverse period; this proof can be obained by first recording the velocity head (Δp) reading at the final traverse point, then cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and finally by recording another Δp reading at the final traverse point. If the Ap reading made after the air purge is within 5 percent of the reading during the traverse, then the traverse is acceptable. Otherwise, reject the run. Note that if the Δp at the final traverse point is so low as to make this determination too difficult, then another traverse point may be selected. If "back purging" at regular intervals is part of the procedure, then take comparative Ap readings, as above, for the last two back purges at which suitable high Δp readings are observed.

METHOD 2D-MEASUREMENT OF GAS VOLU-METRIC FLOW RATES IN SMALL PIPES AND DUCTS

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of gas flow rates in small pipes and ducts, either before or after emission control devices.

1.2 Principle. To measure flow rate or pressure drop, all the stack gas is directed through a rotameter, orifice plate or similar flow rate measuring device. The measuring device has been previously calibrated in a manner that insures its proper calibration for the gas or gas mixture being measured. Absolute temperature and pressure measurements are also made to calculate volumetric flow rates at standard conditions.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Flow Rate Measuring Device. A rotameter, orifice plate, or other flow rate measuring device capable of measuring all the stack flow rate to within 5 percent of its true value. The measuring device shall be equipped with a temperature gauge accurate to within 2 percent of the minimum absolute stack temperature and a pressure gauge accurate to within 5 mm Hg. The capacity of the measuring device shall be suf-

40 CFR Ch. I (7-1-90 Edition)

ficient for the expected maximum and minimum flow rates at the stack gas conditions. The magnitude and variability of stack gas flow rate, molecular weight, temperature, pressure, compressibility, dew point, corrosiveness, and pipe or duct size are all factors to consider in choosing a suitable measuring device.

2.2 Barometer. Same as in Method 2, Section 2.5.

2.3 Stopwatch. Capable of incremental time measurement to within 1 second.

3. Procedure

3.1 Installation. Use the procedure in Method 2A, Section 3.1.

3.2 Leak Check. Use the procedure in Method 2A, Section 3.2.

3.3 Flow Rate Measurement.

3.3.1 Continuous, Steady Flow. At least once an hour, record the measuring device flow rate reading, and the measuring device temperature and pressure. Make a minimum of twelve equally spaced readings of each parameter during the test period. Record the barometric pressure at the beginning and end of the test period. Record the data on a table similar to Figure 2D-1.

Date:				
Date	Run	number		
Sample loca	.clon		<u> </u>	** -
Barometric	pressure,	mm	(in.)	нg
Start	Finish			
Operators _				
Measuring	device nur	nber	C	alibra-
tion coeffici	ent			
Calibration	gas	_		
Last date ca	librated			

Time Flow read	Elou toto	Static	Tempe	erature
	reading	mm (in.) Hg	°C (°F)	°K (°R)
			<u> </u>	
			i	
	·			
	Average			

Figure 2D-1. Flow rate measurement data.

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Environmental Protection Agency

3.3.2 Noncontinuous and Nonsteady Flows. Use flow rate measuring devices with particular caution. Calibration will be affected by variation in stack gas temperature, pressure, compressibility, and molecular weight. Use the procedure in Section 3.3.1. Record all the measuring device parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

4. Calibration

4.1 Flow Rate Measuring Device. Use the procedure in Method 2A, Section 4, and

 $Q_{,=}$ reference meter flow rate reading, $m^{3}/$

 Q_m = measuring device flow rate reading.

T.=reference meter average absolute tem-

 T_m = measuring device average absolute

 P_{her} = barometric pressure, mm Hg (in. Hg).

P,=measuring device average static pres-

For measuring devices that do not have a

Q.

readout as flow rate, refer to the manufac-

$$Y_{m} = \frac{(Q_{r})(T_{r})P_{bar}}{(Q_{m})(T_{m})(P_{bar} + P_{r})} \qquad Eq. 2D-1$$

turer's instructions to calculate the Q_m corresponding to each Q_m .

Pt. 60, App. A. Meth. 3

apply the same performance standards.

Calibrate the measuring device with the

principal stack gas to be measured (e.g., air,

nitrogen) against a standard reference

meter. A calibrated dry gas meter is an ac-

ceptable reference meter. Ideally, calibrate

the measuring device in the field with the

actual gas to be measured. For measuring

devices that have a volume rate readout,

calculate the measuring device calibration

coefficient, Y_m , for each run as follows:

4.2 Temperature Gauge. Use the procedure and specifications in Method 2A, Section 4.2. Perform the calibration at a temperature that approximates field test conditions.

4.3 Barometer. Calibrate the barometer to be used in the field test with a mercury barometer prior to the field test.

5. Gas Flow Rate Calculation

Calculate the stack gas flow rate, Q_{*} , as follows:

$$= K_1 Y_m Q_m \frac{\frac{(P_{bar} + P_s)}{P_s}}{T_m} \qquad \text{Eq. 2D-2}$$

where:

where'

min (ft²/min).

ni³/min (ft³/min).

perature. 'K ('R).

temperature, 'K ('R).

sure, mm Hg (in. Hg).

 $K_1 = 0.3858$ for international system of units (SI); 17.64 for English units.

6. Bibliography

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METHOD 3—GAS ANALYSIS FOR THE DETERMINATION OF DRY MOLECULAR WEIGHT

1. Applicability and Principle

1.1 Applicability.

1.1.1 This method is applicable for determining carbon dloxide (CO_2) and oxygen (O_2) concentrations and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2 , O_2 , carbon monoxide (CO), and nitrogen (N_2) are not present Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

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METHOD 18-MEASUREMENT OF GASEOUS OR-GANIC COMPOUND EMISSIONS BY GAS CHROMATOGRAPHY

Introduction

This method should not be attempted by persons unfamiliar with the performance characteristics of gas chromatography, nor by those persons who are unfamiliar with source sampling. Particular care should be exercised in the area of safety concerning choice of equipment and operation in potentially explosive atmospheres.

1. Applicability and Principle

1.1 Applicability. This method applies to the analysis of approximately 90 percent of the total gaseous organics emitted from an industrial source. It does not include techniques to identify and measure trace amounts of organic compounds, such as those found in building air and fugitive emission sources.

This method will not determine compounds that (1) are polymeric (high molecular weight), (2) can polymerize before analysis, or (3) have very low vapor pressures at stack or instrument conditions.

1.2 Principle.

40 CFR Ch. 1 (7-1-90 Edition)

The major organic components of a gas mixture are separated by gas chromatography (GC) and individually quantified by flame ionization, photoionization, electron capture, or other appropriate detection principles.

The retention times of cach separated component are compared with those of known compounds under identical conditions. Therefore, the analyst confirms the identity and approximate concentrations of the organic emission components beforehand. With this information, the analyst then prepares or purchases commercially available standard mixtures to calibrate the GC under conditions identical to those of the samples. The analyst also determines the need for sample dilution to avoid detector saturation, gas stream filtration to eliminate particulate matter, and prevention of moisture condensation.

2. Range and Sensitivity

2.1 Range. The range of this method is from about 1 part per million (ppm) to the upper limit governed by GC detector saturation or column overloading. The upper limit can be extended by diluting the stack gases with an inert gas or by using smaller gas sampling loops.

2.2 Sensitivity. The sensitivity limit for a compound is defined as the minimum detectable concentration of that compound, or the concentration that produces a signal-tonoise ratio of three to one. The minimum detectable concentration is determined during the presurvey calibration for each compound.

3. Precision and Accuracy

Gas chromatographic techniques typically provide a precision of 5 to 10 percent relative standard deviation (RSD), but an experienced GC operator with a reliable instrument can readily achieve 5 percent RSD. For this method, the following combined GC/operator values are required.

(a) Precision. Duplicate analyses are within 5 percent of their mean value.

(b) Accuracy. Analysis results of prepared audit samples are within 10 percent of preparation values.

4. Interferences

Resolution interferences that may occur can be eliminated by appropriate GC column and detector choice or by shifting the retention times through changes in the column flow rate and the use of temperature programming.

The analytical system is demonstrated to be essentially free from contaminants by periodically analyzing blanks that consist of hydrocarbon-free air or nitrogen.

Sample cross-contamination that occurs when high-level and low-level samples or standards are analyzed alternately, is best Environmental Protection Agency

dealt with by thorough purging of the GC sample loop between samples.

To assure consistent detector response, calibration gases are contained in dry air. To adjust gaseous organic concentrations when water vapor is present in the sample, water vapor concentrations are determined for those samples, and a correction factor is applied.

5. Presurvey and Presurvey Sampling.

Perform a presurvey for each source to be tested. Refer to Figure 18-1. Some of the information can be collected from literature surveys and source personnel. Collect gas samples that can be analyzed to confirm the identities and approximate concentrations of the organic emissions.

5.1 Apparatus. This apparatus list also applies to Sections 6 and 7.

5.1.1 Teflon Tubing. (Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Diameter and length determined by connection requirements of cylinder regulators and the GC. Additional tubing is necessary to connect the GC sample loop to the sample.

5.1.2 Gas Chromatograph. GC with suitable detector, columns, temperature-controlled sample loop and valve assembly, and temperature programable oven, if necessary. The GC shall achieve sensitivity requirements for the compounds under study.

5.1.3 Pump. Capable of pumping 100 ml/ min. For flushing sample loop.

5.1.4 Flowmeters. To measure flow rates. 5.1.5 Regulators. Used on gas cylinders for GC and for cylinder standards.

5.1.6 Recorder. Recorder with linear strip chart is minimum acceptable. Integrator (optional) is recommended.

5.1.7 Syringes. 0.5-ml, 1.0- and 10-microliter sizes, calibrated, maximum accuracy (gas tight), for preparing calibration standards. Other appropriate sizes can be used.

5.1.8 Tubing Fittings. To plumb GC and gas cylinders.

5.1.9 Septums. For syringe injections.

5.1.10 Glass Jars. If necessary, clean-colored glass jars with Teflon-lined lids for condensate sample collection. Size dependon volume of condensate.

5.1.11 Soap Film Flow Meter. To determine flow rates.

5.1.12 Tedlar Bags. 10- and 50-liter capacity, for preparation of standards.

5.1.13 Dry Gas Meter with Temperature and Pressure Gauges. Accurate to ± 2 percent, for perparation of gas standards.

5.1.14 Midget Impinger/Hot Plate Assembly. For preparation of gas standards.

5.1.15 Sample Flasks. For presurvey samples, must have gas-tight seals.

5.1.16 Adsorption Tubes. If necessary, blank tubes filled with necessary adsorbent

(charcoal, Tenax, XAD-2, etc.) for presurvey samples.

5.1.17 Personnel Sampling Pump. Calibrated, for collecting adsorbent tube presurvey samples.

5.1.18 Dilution System. Calibrated, the dilution system is to be constructed following the specifications of an acceptable method.

5.1.19 Sample Probes. Pyrex or stainless steel, of sufficient length to reach centroid of stack, or a point no closer to the walls than 1 m.

5.1.20 Barometer. To measure barometric pressure.

5.2 Reagents.

t

5.2.1 Deionized Distilled Water.

5.2.2 Methylene Dichloride,

5.2.3 Calibration Gases. A series of standards prepared for every compound of interest.

5.2.4 Organic Compound Solutions. Pure (99.9 percent), or as pure as can reasonably be obtained, liquid samples of all the organic compounds needed to prepare calibration standards.

5.2.5 Extraction Solvents. For extraction of adsorbent tube samples in preparation for analysis.

5.2.6 Fuel. As recommended by the manufacturer for operation of the GC.

5.2.7 Carrier Gas. Hydrocarbon free, as recommended by the manufacturer for operation of the detector and compatability with the column.

5.2.8 Zero Gas. Hydrocarbon free air or nitrogen, to be used for dilutions, blank preparation, and standard preparation.

5.3 Sampling.

5.3.1 Collection of Samples with Glass Sampling Flasks. Presurvey samples can be collected in precleaned 250-ml double-ended glass sampling flasks. Teflon stopcocks, without grease, are preferred. Flasks should be cleaned as follows: Remove the stopcocks from both ends of the flasks, and wipe the parts to remove any grease. Clean the stopcocks, barrels, and receivers with methylene dichloride. Clean all glass ports with a soap solution, then rinse with tap and deionized distilled water. Place the flask in a cool glass annealing furnace and apply heat up to 500° C. Maintain at this temperature for 1 hour. After this time period, shut off and open the furnace to allow the flask to cool. Grease the stopcocks with stopcock grease and return them to the flask receivers. Purge the assembly with high-purity nitrogen for 2 to 5 minutes. Close off the stopcocks after purging to maintain a slight positive nitrogen pressure. Secure the stopcocks with tape.

Presurvey samples can be obtained either by drawing the gases into the previously evacuated flask or by drawing the gases into and purging the flask with a rubber suction bulb.

5.3.1.1 Evacuated Flask Procedure. Use a high-vacuum pump to evacuate the flask to the capacity of the pump; then close off the stopcock leading to the pump. Attach a 6mm outside diameter (OD) glass tee to the flask inlet with a short piece of Teflon tubing. Select a 6-mm OD borosilicate sampling probe, enlarged at one end to a 12-mm OD and of sufficient length to reach the centroid of the duct to be sampled. Insert a glass wool plug in the enlarged end of the probe to remove particulate matter. Attach the other end of the probe to the tee with a short piece of Tefion tubing. Connect a rubber suction bulb to the third leg of the tee. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and purge the probe with the rubber suction bulb. After the probe is completely purged and filled with duct gases, open the stopcock to the grab flask until the pressure in the flask reaches duct pressure. Close off the stopcock, and remove the probe from the duct. Remove the tee from the flask and tape the stopcocks to prevent leaks during shipment. Measure and record the duct temperature and pressure.

5.3.1.2 Purged Flask Procedure. Attach one end of the sampling flask to a rubber suction bulb. Attach the other end to a 6mm OD glass probe as described in Section 5.3.1.1. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and apply suction with the bulb to completely purge the probe and flask. After the flask has been purged, close off the stopcock near the suction bulb, and then close the stopcock near the probe. Remove the probe from the duct. and disconnect both the probe and suction bulb. Tape the stopcocks to prevent leakage during shipment. Measure and record the duct temperature and pressure.

5.3.2 Flexible Bag Procedure. Tedlar or aluminized Mylar bags can also be used to obtain the presurvey sample. Use new bags, and leak check them before field use. In addition, check the bag before use for contamination by filling it with nitrogen or air, and analyzing the gas by GC at high sensitivity. Experience indicates that it is desirable to allow the inert gas to remain in the bag about 24 hours or longer to check for desorption of organics from the bag. Follow the leak check and sample collection procedures given in Section 7.1.

5.3.3 Determination of Moisture Content. For combustion or water-controlled processes, obtain the moisture content from plant personnel or by measurement during the presurvey. If the source is below 59° C, measure the wet bulb and dry bulb temperatures, and calculate the moisture content using a psychrometric chart. At higher temperatures, use Method 4 to determine the moisture content.

5.4 Determination of Static Pressure. Obtain the static pressure from the plant personnel or measurement. If a type S pitot tube and an inclined manometer are used, take care to align the pitot tube 90° from the direction of the flow. Disconnect one of the tubes to the manometer, and read the static pressure; note whether the reading is positive or negative.

5.5 Collection of Presurvey Samples with Adsorption Tube. Follow Section 7.4 for presurvey sampling.

6. Analysis Development

6.1 Selection of GC Parameters.

6.1.1 Column Choice. Based on the initial contact with plant personnel concerning the plant process and the anticipated emissions, choose a column that provides good resolution and rapid analysis time. The choice of an appropriate column can be aided by a literature search, contact with manufacturers of GC columns, and discussion with personnel at the emission source.

Most column manufacturers keep excellent records of their products. Their technical service departments may be able to recommend appropriate columns and detector type for separating the anticipated compounds, and they may be able to provide information on interferences, optimum operating conditions, and column limitations.

Plants with analytical laboratories may also be able to provide information on appropriate analytical procedures.

6.1.2 Preliminary GC Adjustment. Using the standards and column obtained in Section 6.1.1, perform initial tests to determine appropriate GC conditions that provide good resolution and minimum analysis time for the compounds of interest.

6.1.3 Preparation of Presurvey Samples. If the samples were collected on an adsorbent, extract the sample as recommended by the manufacturer for removal of the compounds with a solvent suitable to the type of GC analysis. Prepare other samples in an appropriate manner.

6.1.4 Presurvey Sample Analysis. Before analysis, heat the presurvey sample to the duct temperature to vaporize any condensed material. Analyze the samples by the GC procedure, and compare the retention times against those of the calibration samples that contain the components expected to be in the stream. If any compounds cannot be identified with certainty by this procedure, identify them by other means such as GC/ mass spectroscopy (GC/MS) or GC/infrared techniques. A GC/MS system is recom-

Use the GC conditions determined by the procedures of Section 6.1.2 for the first injection. Vary the GC parameters during subsequent injections to determine the opti-

Environmental Protection Agency

mum settings. Once the optimum settings have been determined, perform repeat injections of the sample to determine the retention time of each compound. To inject a sample, draw sample through the loop at a constant rate (100 ml/min for 30 seconds). Be careful not to pressurize the gas in the loop. Turn off the pump and allow the gas in the sample loop to come to ambient pressure. Activate the sample valve, and record injection time, loop temperature, column temperature, carrier flow rate, chart speed. and attenuator setting. Calculate the retention time of each peak using the distance from injection to the peak maximum divided by the chart speed. Retention times should be repeatable within 0.5 seconds.

If the concentrations are too high for appropriate detector response, a smaller sample loop or dilutions may be used for gas samples, and, for ilquid samples, dilution with solvent is appropriate. Use the standard curves (Section 6.3) to obtain an estimate of the concentrations.

Identify all peaks by comparing the known retention times of compounds expected to be in the retention times of peaks in the sample. Identify any remaining unidentified peaks which have areas larger than 5 percent of the total using a GC/MS, or estimation of possible compounds by their retention times compared to known compounds, with confirmation by further GC analysis.

6.2 Calibration Standards. Prepare or obtain enough calibration standards so that there are three different concentrations of each organic compound expected to be measured in the source sample. For each organic compound, select those concentrations that bracket the concentrations expected in the source samples. A calibration standard may contain more than one organic compound. If available, commercial cylinder gases may be used if their concentrations have been certified by direct analysis.

If samples are collected in adsorbent tubes (charcoal, XAD-2, Tenax, etc.), prepare or obtain standards in the same solvent used for the sample extraction procedure. Refer to Section 7.4.3.

Verify the stability of all standards for the time periods they are used. If gas standards are prepared in the laboratory, use one or more of the following procedures.

6.2.1 Preparation of Standards from High Concentration Cylinder Standards. Obtain enough high concentration cylinder standards to represent all the organic compounds expected in the source samples. Pt. 60, App. A, Meth. 18

Use these high concentration standards to prepare lower concentration standards by dilution, as shown by Figures 18-5 and 18-6.

To prepare the diluted calibration samples, calibrated rotameters are normally used to meter both the high concentration calibration gas and the diluent gas. Other types of flowmeters and commercially available dilution systems can also be used.

Calibrate each flowmeter before use by placing it between the diluent gas supply and suitably sized bubble meter, spirometer, or wet test meter. Record all data shown on Figure 18-4. While it is desirable to calibrate the cylinder gas flowmeter with cylinder gas, the available quantity and cost may preclude it. The error introduced by using the diluent gas for calibration is insignificant for gas mixtures of up to 1,000 to 2,000 ppm of each organic component.

Once the flowmeters are calibrated, connect the flowmeters to the calibration and diluent gas supplies using 6-mm Teflon tubing. Connect the outlet side of the flowmeters through a connector to a leak-free Tedlar bag as shown in Figure 18-5, (See Section 7.1 for bag leak-check procedures.) Adjust the gas flow to provide the desired dilution, and fill the bag with sufficient gas for GC calibration. Be careful not to overfill and cause the bag to apply additional pressure on the dilution system. Record the flow rates of both flowmeters, and the laboratory temperature and atmospheric pressure. Calculate the concentration C, in ppm of each organic in the diluted gas as follows:

$$C_{s} = \frac{10^{6} (\bar{x} q_{c})}{q_{c} + q_{d}}$$

Eq. 18-1

10^s = Conversion to ppm.

where:

X = Mole or volume fraction of the organic in the calibration gas to be diluted.

q.=Flow rate of the calibration gas to be diluted.

 $q_d = Diluent$ gas flow rate.

Single-stage dilutions should be used to prepare calibration mixtures up to about 1:20 dilution factor.

For greater dilutions, a double dilution system is recommended, as shown in Figure 18-6. Fill the Tediar bag with the dilute gas from the second stage. Record the laboratory temperature, barometric pressure, and static pressure readings. Correct the flow reading for temperature and pressure. Calculate the concentration C, in ppm of the organic in the final gas mixture as follows:

$$C_{s} = 10^{6} \overline{X} \left(\frac{q_{c1}}{q_{c1} + q_{d1}} \right) \left(\frac{q_{c2}}{q_{c2} + q_{d2}} \right)$$
 Eq. 18-2

Where:

- 10°=Conversion to ppm.
- X = Mole or volume fraction of the organic in the calibration gas to be diluted.
- $q_{ei} = Flow$ rate of the calibration gas to be diluted in stage 1.
- q_a=Flow rate of the calibration gas to be diluted in stage 2.
- q_{d1}=Flow rate of diluent gas in stage 1.
- q_{a} = Flow rate of diluent gas in stage 2.

Further details of the calibration methods for flowmeters and the dilution system can be found in Citation 21 in the Bibliography.

6.2.2 Preparation of Standards from Volatile Materials. Record all data shown on Figure 18-3.

6.2.2.1 Gas Injection Technique. This procedure is applicable to organic compounds that exist entirely as a gas at ambient conditions. Evacuate a 10-liter Tedlar bag that has passed a leak-check (see Section 7.1), and meter in 5.0 liters of air or ni-

Calculate each organic standard concentration C, in ppm as follows:

$$C_{s} = \frac{G_{v} \times 10^{6} \frac{293}{T_{s}} \frac{P_{s}}{760}}{V_{m} Y \frac{293}{T_{m}} \frac{P_{m}}{760} 1000}$$
$$= \frac{G_{v} \times 10^{3} \frac{P_{s}}{T_{s}} \frac{T_{m}}{T_{s}}}{V_{m} Y}$$

where:

- G_{*}=Gas volume or organic compound iniected. ml.
- ¹⁰ = Conversion to ppm.
- P.=Absolute pressure of syringe before injection, mm Hg.
- T.=Absolute temperature of syringe before injection, 'K.
- $V_m = Gas$ volume indicated by dry gas meter, liters.
- Y=Dry gas meter calibration factor, dimensionless.
- $P_m = Absolute pressure of dry gas meter, mm Hg.$

1000=Conversion factor, ml/liter.

6.2.2.2 Liquid Injection Technique. Use the equipment shown in Figure 18-8. Calibrate the dry gas meter as described in Section 6.2.2.1 with a wet test meter or a spirometer. Use a water manometer for the pressure gauge and glass, Teflon, brass, or stainless steel for all connections. Connect a valve to the inlet of the 50-liter Tedlar bag.

Eq. 18-3

To prepare the standards, assemble the equipment as shown in Figure 18-8, and leak-check the system. Completely evacuate the bag. Fill the bag with hydrocarbon-free air, and evacuate the bag again. Close the inlet valve.

Turn on the hot plate, and allow the water to reach bolling. Connect the bag to the impinger outlet. Record the initial meter reading, open the bag inlet valve, and open the cylinder. Adjust the rate so that the bag will be completely filled in approximately 15 minutes. Record meter pressure and temperature, and local barometric pressure.

Allow the liquid organic to equilibrate to room temperature. Fill the 1.0- or 10-microliter syringe to the desired liquid volume

Environmental Protection Agency

with the organic. Place the syringe needle into the impinger inlet using the septum provided, and inject the liquid into the flowing air stream. Use a needle of sufficient length to permit injection of the liquid below the air inlet branch of the tee. Remove the syringe.

When the bag is filled, stop the pump, and close the bag inlet valve. Record the final meter reading, temperature, and pressure.

Disconnect the bag from the impinger outlet, and either set it aside for at least 1 hour, or massage the bag to insure complete mixing.

$C_{s} = \frac{\frac{L_{v}}{M}\rho}{V_{m} + \frac{293}{T_{m}}} \frac{P_{m}}{760} = 6.24 \times 10^{4} \frac{L_{v}\rho}{M} \frac{T_{m}}{V_{m} + P_{m}} = 6.24 \times 10^{4} \frac{L_{v}\rho}{M} \frac{V_{m}}{V_{m}} \frac{V_{m}}{V_{m}} = 1000$

where:

L.= Liquid volume of organic injected, µl. µl=Liquid organic density as determined, g/ ml.

M=Molecular weight of organic, g/g-mole. 24.055=Ideal gas molar volume at 293 'K

and 760 mm Hg, liters/g-mole. 10⁴=Conversion to ppm.

 $1000 = Conversion factor, \mu l/ml.$

6.3 Preparation of Calibration Curves. Establish proper GC conditions, then flush the sampling loop for 30 seconds at a rate of 100 ml/min. Allow the sample loop pressure to equilibrate to atmospheric pressure, and activate the injection valve. Record the standard concentration, attenuator factor, injection time, chart speed, retention time, peak area, sample loop temperature, column temperature, and carrier gas flow rate. Repeat the standard injection until two consecutive injections give area counts within 5 percent of their average. The average value multipled by the attenuator factor is then the calibration area value for the concentration.

Repeat this procedure for each standard. Prepare a graphical plot of concentration (C_i) versus the calibration area values. Perform a regression analysis, and draw the least squares line.

6.4 Relative Response Factors. The calibration curve generated from the standards for a single organic can usually be related to each of the individual GC response curves that are developed in the laboratory for all the compounds in the source. In the field, standards for that single organic can then be used to "calibrate" the GC for all the organics present. This procedure should first

ical balance to the nearest 1.0 milligram. A

Pt. 60, App. A, Meth. 18

ground-glass stoppered 25-mil volumetric flask or a glass-stoppered specific gravity bothe is suitable for weighing. Calculate the result in terms of g/ml. As an alternative, literature values of the density of the liquid at 20 °C may be used.

Measure the solvent liquid density at

room temperature by accurately weighing a

known volume of the material on an analyt-

Calculate each organic standard concentration C, in ppm as follows:

be confirmed in the laboratory by preparing and analyzing calibration standards containing multiple organic compounds.

6.5 Quality Assurance for Laboratory Procedures. Immediately after the preparation of the calibration curves and prior to the presurvey sample analysis, the analysis audit described in 40 CFR Part 61, Appendix C. Procedure 2: "Procedure for Field Auditing GC Analysis." should be performed. The information required to document the analysis of the audit samples has been included on the example data sheets shown in Figures 18-3 and 18-7. The audit analyses should agree with the audit concentrations within 10 percent. When available, the tester may obtain audit cylinders by contacting: U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711, Audit cylinders obtained from a commercial gas manufacturer may be used provided that (a) the gas manufacturer certifies the audit cylinder in a manner similar to the procedure described in 40 CFR Part 61, Appendix B, Method 106, Section 5.2.3.1, and (b) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined as an analysis performed by an individual other than the individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis

concentration is within 5 percent of the gas manufacturer's concentration.

7. Final Sampling and Analysis Procedure Considering safety (flame hazards) and the source conditions, select an appropriate sampling and analysis procedure (Section 7.1, 7.2, 7.3, or 7.4). In situations where a hydrogen flame is a hazard and no intrinsically safe GC is suitable, use the flexible bag collection technique or an adsorption technique. If the source temperature is below 100°C, and the organic concentrations are suitable for the detector to be used, use the direct interface method. If the source gases require dilution, use a dilution interface and either the bag sample or adsorption tubes. The choice between these two techniques will depend on the physical layout of the site, the source temperature, and the storage stability of the compounds if collected in the bag. Sample polar compounds by direct interfacing or dilution interfacing to prevent sample loss by adsorption on the bag.

7.1 Integrated Bag Sampling and Analysis.

7.1.1 Evacuated Container Sampling Procedure. In this procedure, the bags are filled by evacuating the rigid air-tight containers that hold the bags. Use a field sample data sheet as shown in Figure 18-10. Collect triplicate sample from each sample location.

7.1.1.1 Apparatus.

7.1.1.1.1 Probe. Stainless steel, Pyrex glass, or Teflon tubing probe, according to the duct temperature, with 6.4-mm OD Teflon tubing of sufficient length to connect to the sample bag. Use stainless steel or Teflon unions to connect probe and sample line.

7.1.1.1.2 Quick Connects. Male (2) and female (2) of stainless steel construction.

7.1.1.1.3 Needle Valve. To control gas flow.

7.1.1.1.4 Pump. Leakless Teflon-coated diaphragm-type pump or equivalent. To deliver at least 1 liter/min.

7.1.1.1.5 Charcoal Adsorption Tube. Tube filled with activated charcoal, with glass wool plugs at each end, to adsorb organic vapors.

7.1.1.1.6 Flowmeter. 0 to 500-ml flow range; with manufacturer's calibration curve.

7.1.1.2 Sampling Procedure. To obtain a sample, assemble the sample train as shown in Figure 18-9. Leak check both the bag and the container. Connect the vacuum line from the needle valve to the Teflon sample line from the probe. Place the end of the probe at the centroid of the stack, or at a point no closer to the walls than 1 m, and start the pump with the needle valve adjusted to yield a flow of 0.5 liter/minute. After allowing sufficient time to purge the line several times, connect the vacuum line to the bag, and evacuate until the rotameter

40 CFR Ch. 1 (7-1-90 Edition)

indicates no flow. Then position the sample and vacuum lines for sampling, and begin the actual sampling, keeping the rate proportional to the stack velocity. As a precaution, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container, Record the source temperature, barometric pressure, ambient temperature, sampling flow rate, and initial and final sampling time on the data sheet shown in Figure 18-10. Protect the Tedlar bag and its container from sunlight. When possible, perform the analysis within 2 hours of sample collection.

7.1.2 Direct Pump Sampling Procedure. Follow 7.1.1, except place the pump and needle valve between the probe and the bag. Use a pump and needle valve constructed of stainless steel or some other material not affected by the stack gas. Leak check the system, and then purge with stack gas before the connecting to the previously evacuated bag.

7.1.3 Explosion Risk Area Bag Sampling Procedure. Follow 7.1.1 except replace the pump with another evacuated can (see Figure 18-9a). Use this method whenever there is a possibility of an explosion due to pumps, heated probes, or other flame producing equipment.

7.1.4 Other Modified Bag Sampling Procedures. In the event that condensation is observed in the bag while collecting the sample and a direct interface system cannot be used, heat the bag during collection, and maintain it at a suitably elevated temperature during all subsequent operations. (Note: Take care to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.) As an alternative, collect the sample gas, and simultaneously dilute it in the Tedlar bag.

In the first procedure, heat the box containing the sample bag to the source temperature, provided the components of the bag and the surrounding box can withstand this temperature. Then transport the bag as rapidly as possible to the analytical area while maintaining the heating, or cover the box with an insulating blanket. In the analytical area, keep the box heated to source temperature until analysis. Be sure that the method of heating the box and the control for the heating circuit are compatible with the safety restrictions required in each area. To use the second procedure, prefill the

Tedlar bag with a known quantity of inert gas. Meter the inert gas into the bag according to the procedure for the preparation of gas concentration standards of volatile liquid materials (Section 6.2.2.2), but eliminate the midget impinger section. Take the partly filled bag to the source, and meter

Invironmental Protection Agency

the source gas into the bag through heated ampling lines and a heated flowmeter, or Teflon positive displacement pump. Verify the dilution factors periodically through diation and analysis of gases of known concentration.

7.1.5 Analysis of Bag Samples.

1.1.5.1 Apparatus, Same as Section 5. A minimum of three gas standards are reguired.

7.1.5.2 Procedure, Establish proper GC operating conditions as described in Section 13, and record all data listed in Figure 18-7. Prepare the GC so that gas can be drawn through the sample valve. Flush the sample kop with gas from one of the three calibration mixtures, and activate the valve, Obtain at least two chromatograms for the mixture. The results are acceptable when the peak areas from two consecutive injec-Mons agree to within 5 percent of their averwe If they do not, run additional analyses or correct the analytical techniques until this requirement is met. Then analyze the ther two calibration mixtures in the same manner. Prepare a calibration curve as dewibed in the same manner. Prepare a calibration curve as described in Section 6.3.

Analyze the source gas samples by connecting each bag to the sampling valve with iplece of Teflon tubing identified for that bar. Follow the specifications on replicate malyses specified for the calibration gases. Record the data listed in Figure 18-11. If artain items do not apply, use the notation "N.A." After all samples have been anabred, repeat the analyses of the calibration iss mixtures, and generate a second calibration curve. Use an average of the two curves to determine the sample gas concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then moort the final results by comparison to both calibration curves.

1.6 Determination of Bag Water Vapor Content. Measure and record the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the netr vapor content as a decimal figure. (Assume the relative humidity to be 100 perent unless a lesser value is known.) If the high as been maintained at an elevated temperature as described in Section 7.1.4, determine the stack gas water content by Method

1.1.7 Quality Assurance. Immediately prior to the analysis of the stack gas.samples, perform audit analyses as described in Section 6.5. The audit analyses must agree with the audit concentrations within 10 perent. If the results are acceptable, proceed with the analyses of the source samples. If they do not agree within 10 percent, then determine the reason for the discrepancy, and take corrective action before proceed ing. Pt. 60, App. A, Meth. 18

7.1.8 Emission Calculations. From the average calibration curve described in Section 7.1.5., select the value of C, that corresponds to the peak area. Calculate the concentration C, in ppm, dry basis, of each organic in the sample as follows:

$$C_{c} = \frac{C_{s}P_{r}T_{i}F_{r}}{P_{i}T_{r}(1-B_{ws})} E_{q}, 18-5$$

whe**re**:

- C_i=Concentration of the organic from the calibration curve, ppm.
- P,=Reference pressure, the barometric pressure or absolute sample loop pressure recorded during calibration, mm Hg.
- •T_i a Sample loop temperature at the time of sample analysis, 'K.
- F.=Relative response factor (if applicable, see Section 6.4).
- Pi=Barometric or absolute sample loop pressure at time of sample analysis, mm Hg.
- T_r=Reference temperature, the termperature of the sample loop recorded during calibration, K.
- B_w=Water vapor:content of the bag sample or stack gas; proportion by volume.

7.2 Direct Interface Sampling and Analysis Procedure. The direct interface procedure can be used provided that the moisture content of the gas does not interfere with the analysis procedure, the physical requirements of the equipment can be met at the site, and the source gas concentration is low enough that detector saturation is not a problem. Adhere to all safety requirements with this method.

7.2.1 Apparatus.

7.2.1.1 Probe. Constructed of stainless steel, Pyrex glass, or Eeflon tubing as required by duct temperature, 6.4-mm OD, enlarged at duct end to contain glass wool plug. If necessary, heat the probe with heating tape or a special heating unit capable of maintaining duct temperature.

. 7.2.1.2 Sample Lines. 6.4-mm OD Teflon lines, heat-traced to prevent condensation of material.

7.2.1.3 Quick Connects. To connect sample line to gas sampling valve on GC instrument and to pump unit used to withdraw source.rgas. Use a quick connect or sequivalent on the cylinder or bag containing calibration gas to allow connection of the calibration gas to the gas sampling valve.

7.2.1.4 Thermocouple Readout Device. Potentiometer or digital thermometer, to measure source temperature and probe temperature.

bey do not agree within 10 percent, then determine the reason for the discrepancy,... two-position, six-port design, to allow and take corrective action before proceedsample loop to be purged with source gas or to direct source gas into the GC instrument.

- - -

7.2.1.6 Needle Valve. To control gas sampling rate from the source.

7.2.1.7 Pump. Leakless Teflon-coated diaphragm-type pump or equivalent, capable of at least 1 liter/minute sampling rate.

7.2.1.8 Flowmeter. Of suitable range to measure sampling rate.

7.2.1.9 Charcoal Adsorber. To adsorb organic vapor collected from the source to prevent exposure of personnel to source gas.

7.2.1.10 Gas Cylinders. Carrier gas (helium or nitrogen), and oxygen and hydrogen for a flame ionization detector (FID) if one is used.

7.2.1.11 Gas Chromatograph. Capable of being moved into the field, with detector, heated gas sampling valve, column required to complete separation of desired components, and option for temperature programming.

7.2.1.12 Recorder/Integrator. To record results.

7.2.2 Procedure. To obtain a sample, assemble the sampling system as shown in Figure 18-12. Make sure all connections are tight. Turn on the probe and sample line heaters. As the temperature of the probe and heated line approaches the source temperature as indicated on the thermocouple readout device, control the heating to maintain a temperature of 0 to 3°C above the source temperature. While the probe and heated line are being heated, disconnect the sample line from the gas sampling valve. and attach the line from the calibration gas mixture. Flush the sample loop with calibration gas and analyze a portion of that gas. Record the results. After the calibration gas sample has been flushed into the GC instrument, turn the gas sampling valve to flush position, then reconnect the probe sample line to the valve. Place the inlet of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and draw source gas into the probe, heated line, and sample loop. After thorough flushing. analyze the sample using the same conditions as for the calibration gas mixture. Repeat the analysis on an additional sample. Measure the peak areas for the two samples, and if they do not agree to within 5 percent of their mean value, analyze additional samples until two consecutive analyses meet this criteria. Record the data. After consistent results are obtained, remove the probe from the source and analyze a second calibration gas mixture. Record this calibration data and the other required data on the data sheet shown in Figure 18-11, deleting the dilution gas information.

(Note: Take care to draw all samples, calibration mixtures, and audits through the sample loop at the same pressure.) 7.2.3 Determination of Stack Gas Molsture Content. Use Method 4 to measure the stack gas moisture content.

40 CFR Ch. 1 (7-1-90 Edition)

7.2.4 Quality Assurance. Same as Section 7.1.7. Introduce the audit gases in the sample line immediately following the probe.

7.2.5 Emission Calculations. Same as Section 7.1.8.

7.3 Dilution Interface Sampling and Analysis Procedure, Source samples that contain a high concentration of organic materials may require dilution prior to analysis to prevent saturating the GC detector. The apparatus required for this direct interface procedure is basically the same as that described in the Section 7.2, except a dilution system is added between the heated sample line and the gas sampling valve. The apparatus is arranged so that either a 10:1 or 100:1 dilution of the source gas can be directed to the chromatograph. A pump of larger capacity is also required, and this pump must be heated and placed in the system between the sample line and the dilution apparatus.

7.3.1 Apparatus. The equipment required in addition to that specified for the direct interface system is as follows:

7.3.1.1 Sample Pump. Leakless Tefloncoated diaphragm-type that can withstand being heated to 120°C and deliver 1.5 liters/ minute.

7.3.1.2 Dilution Pumps. Two Model A-150 Komhyr Teflon positive displacement type delivering 150 cc/minute, or equivalent. As an option, calibrated flowmeters can be used in conjunction with Teflon-coated diaphragm pumps.

7.3.1.3 Valves. Two Teflon three-way valves, suitable for connecting to 6.4-mm OD Teflon tubing.

7.3.1.4 Flowmeters. Two, for measurement of diluent gas, expected delivery flow rate to be 1.350 cc/min.

7.3.1.5 Diluent Gas with Cylinders and Regulators. Gas can be nitrogen or clean dry air, depending on the nature of the source gases.

7.3.1.6 Heated Box. Suitable for being heated to 120 °C, to contain the three pumps, three-way valves, and associated connections. The box should be equipped with quick connect fittings to facilitate connection of: (1) The heated sample line from the probe, (2) the gas sampling valve, (3) the calibration gas mixtures, and (4) diluent gas lines. A schematic diagram of the components and connections is shown in Figure 18-13.

(Note: Care must be taken to leak check the system prior to the dilutions so as not to create a potentially explosive atmosphere.)

The heated box shown in Figure 18-13 is designed to receive a heated line from the

Environmental Protection Agency

probe. An optional design is to build a probe unit that attaches directly to the heated box. In this way, the heated box contains the controls for the probe heaters, or, if the box is placed against the duct being sampled, it may be possible to eliminate the probe heaters. In either case, a heated Teflon line is used to connect the heated box to the gas sampling valve on the chromatograph.

7.3.2 Procedure. Assemble the apparatus by connecting the heated box, shown in Figure 18-13, between the heated sample line from the probe and the gas sampling valve on the chromatograph. Vent the source gas from the gas sampling valve directly to the charcoal filter, eliminating the pump and rotameter. Heat the sample probe, sample line, and heated box. Insert the probe and source thermocouple to the centroid of the duct, or to a point no closer to the walls than 1 m. Measure the source temperature, and adjust all heating units to a temperature 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds. Verify the operation of the dilution system by analyzing a high concentration gas of known composition through either the 10:1 or 100:1 dilution stages, as appropriate. (If necessary, vary the flow of the diluent gas to obtain other dilution ratios.) Determine the concentration of the diluted calibration gas using the dilution factor and the calibration curves prepared in the laboratory. Record the pertinent data on the data sheet shown in Figure 18-11. If the data on the diluted calibration gas are not within 10 percent of the expected values, determine whether the chromatograph or the dilution system is in error, and correct it. Verify the GC operation using a low concentration standard by diverting the gas into the sample loop, bypassing the dilution system. If these analyses are not within acceptable limits, correct the dilution system to provide the desired dilution factors. Make this correction by diluting a high-concentration standard gas mixture to adjust the dilution ratio as required.

Once the dilution system and GC operations are satisfactory, proceed with the analysis of source gas, maintaining the same dilution settings as used for the standards. Repeat the analyses until two consecutive values do not vary by more than 5 percent from their mean value are obtained.

Repeat the analysis of the calibration gas mixtures to verify equipment operation. Analyze the two field audit samples using either the dilution system, or directly connect to the gas sampling valve as required. Record all data and report the results to the audit supervisor. Pt. 60, App. A, Meth. 18

7.3.3 Determination of Stack Gas Moisture Content. Same as Section 7.2.3.

7.3.4 Quality Assurance. Same as Section 7.2.4.

7.3.5 Emission Calculations. Same as Section **7:2.5**, with the dilution factor applied.

7.4 Adsorption Tube Procedure (Alternative Procedure). It is suggested that the tester refer to the National Institute of Occupational Safety and Health (NIOSH) method for the particular organics to be sampled. The principal interferent will be water vapor. If water vapor is present at concentrations above 3 percent, silica gel should be used in front of the charcoal. Where more than one compound is present in the emissions, then develop relative adsorptive capacity information.

7.4.1 Additional Apparatus. In addition to the equipment listed in the NIOSH method for the particular organic(s) to be sampled, the following items (or equivalent) are suggested.

7.4.1.1 Probe (Optional). Borosilicate glass or stainless steel, approximately 6-mm ID, with a heating system if water condensation is a problem, and a filter (either instack or out-stack heated to stack temperature) to remove particulate matter. In most instances, a plug of glass wool is a satisfactory filter.

7.4.1.2 Flexible Tubing. To connect probe to adsorption tubes. Use a material that exhibits minimal sample adsorption.

7.4.1.3 Leakless Sample Pump. Flow controlled, constant rate pump, with a set of limiting (sonic) orifices to provide pumping rates from approximately 10 to 100 cc/min.

7.4.1.4 Bubble-Tube Flowmeter. Volume accuracy within ± 1 percent, to calibrate pump.

7.4.1.5 Stopwatch. To time sampling and pump rate calibration.

7.4.1.6 Adsorption Tubes. Similar to ones specified by NIOSH, except the amounts of adsorbent per primary/backup sections are 800/200 mg for charcoal tubes and 1040/260 mg for silica gel tubes. As an alternative, the tubes may contain a porous polymer adsorbent such as Tenax GC or XAD-2.

7.4.1.7 Barometer. Accurate to 5 mm Hg, to measure atmospheric pressure during sampling and pump calibration.

7.4.1.8 Rotameter. 0 to 100 cc/min, to detect changes in flow rate during sampling.

7.4.2 Sampling and Analysis. It is suggested that the tester follow the sampling and analysis portion of the respective NIOSH method section entitled "Procedure." Calibrate the pump and limiting orifice flow rate through adsorption tubes with the bubble tube flowmeter before sampling. The sample system can be operated as a "recirculating loop" for this operation. Record the ambient temperature and barometric pressure. Then, during sampling, use the rotameter to verify that the pump and orifice sampling rate remains constant.

Use a sample probe, if required, to obtain the sample at the centroid of the duct, or at a point no closer to the walls than 1 m. Minimize the length of flexible tubing between the probe and adsorption tubes. Several adsorption tubes can be connected in series, if the extra adsorptive capacity is needed. Provide the gas sample to the sample system at a pressure sufficient for the limiting orifice to function as a sonic orifice. Record the total time and sample flow rate (or the number of pump strokes), the barometric pressure, and ambient temperature. Obtain a total sample volume commensurate with the expected concentration(s) of the volatile organic(s) present, and recommended sample loading factors (weight sample per weight adsorption media). Laboratory tests prior to actual sampling may be necessary to predetermine this volume. When more than one organic is present in the emissions. then develop relative adsorptive capacity information. If water vapor is present in the sample at concentrations above 2 to 3 percent, the adsorptive capacity may be severely reduced. Operate the gas chromatograph according to the manufacture's instructions. After establishing optimum conditions. verify and document these conditions during all operations. Analyze the audit samples (see Section 7.4.4.3), then the emission samples. Repeat the analysis of each sample until the relative deviation of two consecutive injections does not exceed 5 percent.

7.4.3 Standards and Calibration. The standards can be prepared according to the respective NIOSH method. Use a minimum of three different standards; select the concentrations to bracket the expected average sample concentration. Perform the calibration before and after each day's sample analyses. Prepare the calibration curve by using the least squares method.

7.4.4 Quality Assurance.

7.4.4.1 Determination of Desorption Efficiency. During the testing program, determine the desorption efficiency in the expected sample concentration range for each batch of adsorption media to be used. Use an internal standard. A minimum desorption efficiency of 50 percent shall be obtained. Repeat the desorption determination until the relative deviation of two consecutive determinations does not exceed 5 percent. Use the average desorption efficiency of these two consecutive determinations for the correction specified in Section 7.4.4.5. If the desorption efficiency of the compound(s) of interest is questionable under actual sampling conditions, use of the Method of Standard Additions may be helpful to determine this value.

7.4.4.2 Determination of Sample Collection Efficiency. For the source samples, ana-

lyze the primary and backup portions of the adsorption tubes separately. If the backup portion exceeds 10 percent of the total amount (primary and backup), repeat the sampling with a larger sampling portion.

7.4.4.3 Analysis Audit. Immediately before the sample analyses, analyze the two audits in accordance with Section 7.4.2. The analysis audit shall agree with the audit concentration within 10 percent.

7.4.4.4 Pump Leak Checks and Volume Flow Rate Checks. Perform both of these checks immediately after sampling with all sampling train components in place. Perform all leak checks according to the manufacturer's instructions, and record the results. Use the bubble-tube flowmeter to measure the pump volume flow rate with the orifice used in the test sampling, and the result. If it has changed by more than 5 but less than 20 percent, calculate an average flow rate for the test. If the flow rate has changed by more than 20 percent, recalibrate the pump and repeat the sampling.

7.4.4.5 Calculations. All calculations can be performed according to the respective NIOSH method. Correct all sample volumes to standard conditions. If a sample dilution system has been used, multiply the results by the appropriate dilution ratio. Correct all results by dividing by the desorption efficiency (decimal value). Report results as ppm by volume, dry basis.

7.5 Reporting of Results. At the completion of the field analysis portion of the study, ensure that the data sheets shown in Figure 18-11 have been completed. Summarize this data on the data sheets shown in Figure 18-15.

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5770 Federal Register / Vol. 56, No. 30 / Wednesday, February 13, 1991 / Rules and Regulations

TABLE 5.—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RE-SPONSE FACTORS—Continued

1	Relative response factors		
Compound	Initial calibration RSD	Daily calibration % difference	
Alternate Standard 13C12- 1,2,3,7,8,9- HxCDF	25 ;	25	

Method 28-Determination of Hydrogen

Chloride Emissions From Stationary Sources

1. Applicability, Principle, Interferences. Precision, Bias, and Stability

 Applicability. This method is
 applicable for determining hydrogen chloride (HC1) emissions from stationary sources.
 Principle. An integrated sample is

extracted from the stack and passed through dilute sulfuric acid. In the dilute acid. the HC1 gas is dissolved and forms chloride (C1⁻) ions. The C1⁻ is analyzed by ion chromatography (IC).

1.3 Interferences. Volatile materials which produce chloride ions upon dissolution during sampling are obvious interferences. Another likely interferent is diatomic chlorine (C1:) gas which reacts to form HC1 and hypochlorous acid (HOC1) upon dissolving in water. However, C12 gas exhibits a low solubility in water and the use of acidic. rather than neutral or basic collection solutions, greatly reduces the chance of dissolving any chlorine present. This method does not experience a significant bias when sampling a 400 ppm HC1 gas stream containing 50 ppm C12. Sampling a 220 ppm HC1 gas stream containing 180 ppm C1₃ results in a positive bias of 3.4 percent in the HC1 measurement.

1.4 Precision and Bias. The withinlaboratory relative standard deviations are 6.2 and 3.2 percent at HC1 concentrations of 3.9 and 15.3 ppm, respectively. The method does not exhibit a bias to C1₂ when sampling at concentrations less than 50 ppm.

1.5 Stability. The collected samples can be stored for up to 4 weeks before analysis.
1.6 Detection Limit. The enalytical

detection limit of the method is $0.1 \mu g/ml$.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 26–1, and component parts are discussed below.

2.1.1 Probe. Borgsilicate gless. approximately %-in. (9-mm) I.D. with a heating system to prevent moisture condensation. A 3/s-in I.D. Tellon elbow should be attached to the inlet of the probe and a 1-in. (25-mm) length of 3%-in. I.D. Teflon tubing should be attached to the open end of the elbow to permit the opening of the probe to be turned away from the gas stream. This reduces the amount of particulate entering the train. This probe configuration should be used when the concentration of particulate matter in the emissions is high. When high concentrations are not present, the Teflon elbow is not necessary, and the probe inlet may be perpendicular to the gas stream. A glass wool plug should not be used to remove particulate matter since a negative bias in the data could result. Instead, a Teflon filter (see Section 2.1.5) should be installed at the inlet (for stack temperatures <300 'F) or outlet (for stack temperatures >300 °F) of the probe.

2.1.2 Three-way Stopcock. A borosilicate, three-way glass stopcock with a heating system to prevent moisture condensation. The heated stopcock should connect directly to the outlet of the probe and the inlet of the first impinger. The heating system should be capable of preventing condensation up to the inlet of the first impinger. Silicone grease may be used, if necessary, to prevent leakage.

BILLING CODE 6560-50-M



Figure 26-1. Sampling train.

BILLING CODE 6460-50-C

5771

 2.1.3 Impingers. Four 30-ml midget impingers with leak-free glass connectors.
 Silicone grease may be used, if necessary, to prevent leakage. For sampling at high moisture sources or for sampling times

greater than 1 hour, a midget imping times shortened stem (such that the gas sample does not bubble through the collected condensate) should be used in front of the first impinger.

2.1.4 Drying Tube or Impinger. Tube or impinger, of Mae West design, filled with 6to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the dry gas meter and pump. If the silica gel has been used previously, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

2.1.5 Filter. A 25-mm Teflon mat, Pallflex TX40H175 or equivalent. Locate between the probe liner and Teflon elbow in a glass or quartz filter holder in a filter box heated to 250 °F.

2.1.6 Sample Line. Leak-free, with compatible fittings to connect the last impinger to the needle valve.

2.1.7 Rate Meter. Rotameter. or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of 2 liters/min.

2.1.8 Purge Pump, Purge Line, Drying Tube, Needle Valve, and Rate Meter. Pump capable of purging the sampling probe at 2 liters/min, with drying tube, filled with silica gel or equivalent, to protect pump, and a rate meter capable of measuring 0 to 5 liters/min.

2.1.9 Stopcock Grease, Valve, Pump. Volume Meter, Barometer, and Vacuum Gauge. Same as in Method 6, Sections 2.1.4, 2.1.7, 2.1.8, 2.1.10, 2.1.11, and 2.1.12.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500-ml or larger, two.

2.2.2 Storage Bottles. 100-ml glass, with Teflon-lined lids, to store impinger samples (two per sampling run). During clean-up, the two front impinger contents $(0.1 \text{ N H}_2\text{ SO}_4)$ should be combined. The contents of the two rear impinger (0.1 N NaOH) may be discarded, as these solutions are included only to absorb C1₂, and thus protect the pump.

2.3 Sample Preparation and Analysis. The materials required for volumetric dilution and chromatographic analysis of samples are described below.

2.3.1 Volumetric Flasks. Class A, 100-ml size.

2.3.2 Volumetric Pipets. Class A, assortment. To dilute samples into the calibration range of the instrument.

2.3.3 Ion Chromatograph. Suppressed or nonsuppressed, with a conductivity detector

and electronic integrator operating in the peak area mode. Other detectors, strip chart recorders, and peak height measurements may be used provided the 5 percent repeatability criteria for sample analysis and the linearity criteria for the calibration curve can be met.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS reagent grade). When such specifications are not available, the best available grade shall be used.

3.1. Sampling.

3.1.1 Water. Deionized, distilled water that conforms to ASTM Specification D 1193– 77, Type 3.

3.1.2 Absorbing solution, 0.1 N Sulfuric Acid (H_2SO_4) . To prepare 100 ml of the absorbing solution for the front impinger pair, slowly add 0.28 ml of concentrated H_2SO_4 to about 90 ml of water while stirring, and adjust the final volume to 100 ml using additional water. Shake well to mix the solution.

3.1.3. Chlorine Scrubber Solution, 0.1 N Sodium Hydroxide (NaOH). To prepare 100 ml of the scrubber solution for the back pair - of impingers, dissolve 0.40 g of solid NaOH in about 90 ml of water, and adjust the final solution volume to 100 ml using additional water. Shake well to mix the solution.

3.2 Sample Preparation and Analysis.

3.2.1 Water. Same as in Section 3.1.1. 3.2.2 Blank Solution. A separate blank solution of the absorbing reagent should be prepared for analysis with the field samples. Dilute 30 ml of absorbing solution to 100 ml with water in a separate volumetric flask.

3.2.3 Sodium Chloride (NaCl) Stock Standard Solution. Solutions containing a nominal certified concentration of 1000 mg/l are commercially available as convenient stock solutions from which working standards can be made by appropriate volumetric dilution. Alternately, concentrated stock solutions may be produced from reagent grade NaCl. The NaCl should be dried at 100 °C for 2 or more hours and cooled to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g of the dried NaCl to within 0.1 mg, dissolve in water, and dilute to 1 liter. The exact Cl concentration can be calculated using Eq. 26-1.

 $\mu g \operatorname{Cl}^{-}/\mathrm{ml} = g \text{ of } \operatorname{NaCl} \times 10^{3} \times 35.453/58.44$

Eq. 26-1

Refrigerate the stock standard solution and store no longer than 1 month.

3.2.4 Chromatographic Eluent. Effective eluents for nonsuppressed IC using a resin- or silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the absorbing solution into each of the first two impingers, and add 15 ml of scrubber solution to the third and fourth impingers. Connect the impingers in series with the knockout impinger first. followed by the two impingers containing absorbing solution and the two containing the scrubber solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or Mae West impinger.

4.1.2 Leak-Check Procedures. Leak-check the probe and three-way stopcock before inserting the probe into the stack. Connect the stopcock to the outlet of the probe, and connect the sample line to the needle valve. Plug the probe inlet, turn on the sample pump, and pull a vacuum of at least 250 mm Hg (10 in. Hg). Turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Place the probe in the stack at the sampling location, and adjust the probe and stopcock:

$$\frac{\mu g Cl^{-}}{ml} = \frac{g of NoCl \times 10^{3} \times 35.453}{58.44}$$

Eq. 26-1

Refrigerate the stock standard solution and store no longer than 1 month.

3.2.4 Chromatographic Eluent. Effective eluents for nonsuppressed IC using a resin- or silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the aborbing solution into each of the first two impingers, and add 15 ml of scrubber solution to the third and fourth impingers. Connect the impingers in series with the knockout impinger first, followed by the two impingers containing absorbing solution and the two containing the scrubber solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or Mae West impinger.

4.1.2 Leak-Check Procedures. Leak-check the probe and three-way stopcock before inserting the probe into the stack. Connect the stopcock to the outlet of the probe, and connect the sample line to the needle valve. Plug the probe inlet, turn on the sample pump, and pull a vacuum of at least 250 mm Hg (10 in. Hg]. Turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Place the probe in the stack at the sampling location, and adjust the probe and stopcock heating system to a temperature sufficient to prevent water condensation. Connect the first impinger to the stopcock, and connect the sumple line to the last impinger and the needle valve. Upon completion of a sampling run, remove the probe from the stack and leak-check as described above. If a leak has occurred, the sampling run must be voided. Alternately, the portion of the train behind the probe may be leak-checked between multiple runs at the same site as follows: Close the stopcock to the first impinger [see Figure 1A of Figure 26-1), and turn on the sampling pump. Pull a vacuum of at least 250 mm Hg. turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Release the vacuum on the impinger train by turning the stopcock to the vent position to permit ambient air to enter (see Figure 1B of Figure 26-2). If this procedure is used, the full train leak-check described above must be conducted following the final run, and all preceding sampling runs must be voided if a leak has occurred.

4.1.3 Purge Procedure. Immediately before sampling. connect the purge line to the stopcock, and turn the stopcock to permit the purge pump to purge the probe (see Figure 1A of Figure 26-1). Turn on the purge pump, and adjust the purge rate to 2 liters/min. Purge for at least 5 minutes before sampling.

4.1.4 Sample Collection. Turn on the sampling pump, pull a slight vacuum of approximately 25 mm Hg (1 in. Hg) on the impinger train, and turn the stopcock to permit stack gas to be pulled through the impinger train (see Figure 1C of Figure 26-3). Adjust the sampling rate to 2 liters/min, as indicated by the rate meter, and maintain this rate to within 10 percent during the entire sampling run. Take readings of the dry gas meter volume and temperature, rate meter, and vacuum gauge at least once every 5 minutes during the run. A sampling time of 1 hour is recommended. Shorter sampling times may introduce a significant negative bias in the HCI concentration. At the conclusion of the sampling run, remove the train from the stack_cool_ and perform a leak-check as described in section 4.1.2.

4.2 Sample Recovery. Disconnect the impingers after sampling. Quantitatively transfer the contents of the first three impingers (the knockout impinger and the two absorbing solution impingers) to a leakfree storage bottle. Add the water rinses of each of these impingers and connecting glassware to the storage bottle. The contents of the scrubber impingers and connecting glassware rinses may be discarded. The sample bottle should be sealed, shaken to mix, and labeled. The fluid level should be marked so that if any sample is lost during transport, a correction proportional to the lost volume can be applied.

4.3 Sample Preparation for Analysis. Check the liquid level in each sample, and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, the volume lost can be determined from the difference between the initial and final solution levels, and this volue can be used to correct the analytical results. Quantitatively transfer the sample solution to a 100-ml volumetric flask, and dilute the solution to 100 ml with water.

4.4 Sample Analysis.

4.4.1 The IC conditions will depend upon analytical column type and whether suppressed or nonsuppressed IC is used. An example chromatogram from a nonsuppressed system using a 150-mm Hamilton PRP-X100 anion column, a * * . 2 I/min flow rate of a 4 mM 4-hydroxy benzoate solution adjusted to a pH of 8.0 using 1 N NaOH, a 50 μ I sample loop, and a conductivity detector set on 1.0 μ S full scale is shown in Figure 26-2.

4.4.2 Before sample analysis, establish a stable baseline. Next, inject a sample of water, and determine if any Cl⁻ appears in the chromatogram. If Cl⁻ is present, repeat ¹ the load/injection procedure until no Cl⁻ is present. At this point, the instrument is ready for use.

4.4.3 First, inject the calibration standards covering an appropriate concentration range, starting with the lowest concentration standard. Next, inject in duplicate, a QC sample followed by a water blank and the field samples. Finally, repeat the injection of calibration standards to allow compensation for any drift in the instrument during analysis of the field samples. Measure the Cl⁻ peak areas or heights of the samples. Use the average response from the duplicate injections to determine the field sample concentrations using a linear calibration curve generated from the standards.

4.5 Audit Analysis. An audit sample must be analyzed, subject to availability.



Figure 26-2. Example Chromatogram

5. Calibration

5.1 Dry Gas Metering System, Thermometers, Rate Meter, and Burometer. Same as in Method 6, sections 5.1, 5.2, 5.3, and 5.4.

5.2 Calibration Curve for Ion Chromatograph. To prepare calibration standards, dilute given volumes (1.0 ml or greater) of the stock standard solution, with 0.1 N H₂SO₄ (section 3.1.2) to convenient volumes. Prepare at least four standards that are within the linear range of the instrument and which cover the expected concentration range of the field samples. Analyze the standards as instructed in section 4.4.3, beginning with the lowest concentration standard. Determine the peak measurements. and plot individual values versus Cl² concentration in $\mu g/ml$. Draw a smooth curve through the points. Use linear regression to

calculate a formula describing the resulting linear curve.

6. Quality Assurance

6.1 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, a set of two audit samples must be analyzed.

6.2 Audit Procedure. The audit sample are chloride solutions. Concurrently analyze the two audit samples and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit samples. If this condition is met,

auditing the subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both

enforcement agencies.

6.3 Audit Sample Availability. The audit samples may be obtained by writing or calling the EPA Regional Office or the appropriate enforcement agency. The request for the audit samples must be made at least 30 days prior to the scheduled compliance sample analyses.

6.4 Audit Results. Calculate the concentrations in mg/dscm using the specified sample volume in the audit instructions.

Note: Indication of acceptable results may be obtained immediately by reporting the audit results in mg/dscm and compliance results in total μ g HCl/ sample to the responsible enforcement agency. Include the results of both audit samples, their identification numbers, and the analyst's name with the results of the compliance determination samples in appropriate reports to the EPA Regional Office or the appropriate enforcement agency. Include this information with subsequent analyses for the same enforcement agency during the 30-day period.

The concentrations of the audit samples obtained by the analyst shall agree within 10 percent of the actual concentrations. If the 10 percent specification is not met, reanalyze the compliance samples and audit samples. and include initial and reanalysis values in the test report.

Failure to meet the 10 percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.

7. Calculations

Retain at least one extra decimal figure beyond those contained in the available data in intermediate calculations, and round off only the final answer appropriately.

7.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Calculate the sample volume using Eq. 6-1 of Method 8. 7.2 Total µg HCl Per Sample.

$$m = \frac{(S-B)(100)(36.46)}{(35.453)} = (102.84)(S-B)$$

Eq. 26-2

where:

- $m = Mass of Hcl in sample, \mu g.$
- $S = Concentration of sample, \mu g Cl /ml.$
- $B = Concentration of blank, \mu g Cl /ml.$
- 100 = Volume of filtered and diluted sample. mł.

 $36.46 = Molecular weight of HCl, \mu g/\mu g$ mole.

35.453 = Atomic weight of Cl, $\mu g/\mu g$ -mole. 7.3 Concentration of HCl in the Flue Gas.

$$C = \frac{K_{\rm m}}{V_{\rm c}}$$
 Eq. 26-3

where:

C = Concentration of HCl, dry basis, mg/dscm.

 $K = 10^{-3} mg/\mu g$

 $m = Mass of HCl in sample, \mu g.$

 $V_{m(std)} = Dry gas volume measured by the$ dry gas meter, corrected to standard conditions, dscm.

8. Bibliography

1. Steinsberger, S.C. and J.H. Margeson. "Laboratory and Field Evaluation of a Methodology for Determination of Hydrogen Chloride Emissions from Municipal and

Hazardous Waste Incinerators." U.S. Environmental Protection Agency, Office of Research and Development. Report No. 1989

2. State of California, Air Resources Board. Method 421. "Determination of Hydrochloric Acid Emissions from Stationary Sources." March 18, 1987.

3. Entropy Environmentalists Inc. "Laboratory Evaluation of a Sampling and Analysis Method for Hydrogen Chloride Emissions from Stationary Sources: Interim Report." EPA Contract No. 68-02-4442. Research Triangle Park. North Carolina. January 22, 1988.

Appendix A [Amended]

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3. Method 19 of appendix A part 60 is amended by adding paragraphs 4.3 and 5.4 as follows: Method 19-Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide, and Nitrogen Oxides Emission Rates

4.3 Daily Geometric Average Pollutant Rates from Hourly Values. The geometric average pollutant rate (E_{re}) is computed using the following equation:

$$\mathbf{E}_{\mathbf{rs}} = \mathbf{E} \mathbf{X} \mathbf{P} \left[(1/n) \quad \boldsymbol{\Sigma} \quad \{1n\{\mathbf{E}_{N}\}\} \right]$$
$$\mathbf{j} = 1$$

Eq. 19-20a

where:

- $E_{aa} = daily geometric average pollutant rate.$ ng/J (lbs/million Btu) or ppm corrected to 7 percent O2.
- E_{hl} = hourly arithmetic average pollutant rate for hour "j," ng/J (lb/million Btu) or ppm corrected to 7 percent O2.
- n = total number of hourly averages for which pollutant rates are available within the 24 hr midnight to midnight daily period.

ln = natural log of indicated value.

EXP = the natural logarithmic base (2.718) raised to the value enclosed by brackets. .

5.4 Daily Geometric Average Percent Reduction from Hourly Values. The geometric average percent reduction (%R_s) is computed using the following equation:

$$%R_{aa} = 109 \left[1 - EXP \left[(1/n) \sum_{j=1}^{n} \ln (E_{io}/E_{ji}) \right] \right]$$

243

where:

- %R_{sa} = daily geometric average percent reduction.
- E_{io} . E_{it} = matched pair hourly arithmetic average pollutant rate, outlet and inlet, respectively, ng/] (lb/million Btu) or ppm corrected to 7 percent O₂.
- n = total number of hourly averages for which paired inlet and outlet pollutant rates are available within the 24-hr midnight to midnight daily period.
 In = natural log of indicated value.
- EXP = the natural logarithmic base (2.718) raised to the value enclosed by brackets.
- Note: The calculation includes only paired data sets (hourly average) for the inlet and outlet pollutant measurements.

[FR Doc. 91-1399 Filed 2-12-91; 8:45 am] BILLING CODE 6560-50-M

DEPARTMENT OF COMMERCE

National Oceanic and Atmospheric Administration

50 CFR Parts 672 and 675

[Docket No. 90899-1019]

Groundfish of the Gulf of Alaska; Groundfish of the Bering Sea and Aleution Islands

AGENCY: National Marine Fisheries Service (NMFS), NOAA, Commerce. ACTION: Final rule, technical amendment.

SUMMARY: The Secretary of Commerce issues this final rule implementing a technical amendment to reinstate regulatory language that was unintentionally deleted by regulations implementing Amendment 13 to the Fishery Management Plan for the Bering Sea/Aleutian Islands Groundfish (Bering FMP) and Amendment 18 to the Fishery Management Plan for the Gulf of Alaska Groundfish Fishery (Gulf FMP) (54 FR 50386, December 6, 1989). EFFECTIVE DATE: February 8, 1991. FOR FURTHER INFORMATION CONTACT:

Patricia Peacock (Fishery Management Specialist), NMFS, Plans and Regulations Division, 1335 East-West Highway, Silver Spring, Maryland 20910, telephone 301-427-2343.

SUPPLEMENTARY INFORMATION: Groundfish fisheries in the Exclusive Economic Zone off Alaska are governed by Federal regulations at 50 CFR parts 611, 620, 672, and 675 that implement the Bering and the Gulf FMPs. These FMPs were prepared by the North Pacific Fishery Management Council and approved by the Secretary of Commerce (Secretary) under provisions of the Magnuson Fishery Conservation and Management Act.

This final rule implements a technical amendment that (1) reinstates language unintentionally deleted by amendatory language in 54 FR 50383 (December 6, 1989) and (2) retains the amendatory language of 56 FR 492 (January 7, 1991).

On December 6, 1989, a final rule was published in the Federal Register (54 FR 50386) that was intended only to amend the introductory language of §§ 672.20(a)(2) and 675.20(a)(2). Item 11 of this amendatory language states that * * paragraphs (a)[2] and (f)(1) are revised * * *; item 20 states that • • • paragraph (a)(2) is revised This amendatory language revised the introductory text of paragraph (a)(2) in these sections but deleted the remainder of the regulatory language in §§ 672.20(a)(2) and 675.20(a)(2). Paragraphs in § 672.20 that were unintentionally deleted were (a)(2)(i), (a)(2)(ii), (a)(2)(ii)(A), and (a)(2)(ii)(B). Table 1 in § 672.20 also was unintentionally deleted. Paragraphs that were unintentionally deleted in § 675.20 were as follows: [a](2)[i], (a)(2)(i)(A). and (a)(2)(i)(B).

On January 1, 1991, final rules implementing Amendment 14 to the Bering FMP and Amendment 19 to the Gulf FMP also revised text in §§ 672.20(a)(2) and 675.20(a)(2) (56 FR 492, January 7, 1991). This rule retains the changes to these sections resulting from Amendments 14 and 19.

The Gulf and Bering FMPs were implemented using procedures specified by the Magnuson Act and the Administrative Procedure Act. However, as explained above, the language currently in §§ 672.20(a) and 675.20(a) does not correctly reflect the Gulf or Bering FMPs and subsequent amendments. This final rule, technical amendment. is reinstating regulatory language needed to implement correctly the Bering and Gulf FMPs as amended.

Classification

This final rule, technical amendment, is issued under parts 672 and 675. Because this rule reinstates regulatory text that was inadvertently deleted during an earlier rulemeking proceeding and makes no substantive changes other than changes determined in rulemaking for Amendments 14 and 19, it is unnecessary under 5 U.S.C. 553(b)(B) to provide for prior public comment and there is good cause under 5 U.S.C. 553(d) not to delay for 30 days its effective date.

Because this rule is being issued without prior comment, a regulatory flexibility analysis is not required under the Regulatory Flexibility Act and none has been prepared.

This rule reinstates language that has been determined not to be a major rule under Executive Order 12291, does not contain policies with federalism implications sufficient to warrant preparation of a federalism assessment under E.O. 12612, and does not contain a collection-of-information requirement for the purposes of the Paperwork Reduction Act. There is no change in the regulatory impacts previously reviewed and analyzed.

List of Subjects in 50 CFR Parts 672 and 675

Fisheries, General limitations.

Dated: February 7, 1991.

Michael F. Tillman,

Acting Assistant Administrator for Fisheries. National Marine Fisheries Service.

For reasons set out in the preamble, 50 CFR parts 672 and 675 are amended as follows:

PART 672—GROUNDFISH OF THE GULF OF ALASKA

1. The authority citation for part 672 continues to read as follows:

Authority: 16 U.S.C. 1801 *et seq.* 2. Section 672.20(a)(2) is revised and Table 1 is added to the section to read as follows:



TABLE OF CONTENTS

NO.	DESCRIPTION	PAGE NO.
1.0 INTE	ODUCTION	
2.0 SCO	PE OF WORK	
3.0 RESU 3.1 3.2	JLTS	
3.3	RECOMMENDATIONS	

LIST OF TABLES

NO	D. DESCRIPTION	
Table 2-1	Compound List - VOCs	
Table 3-2	Detected VOCs in Influent Gas Stream	

1.0 INTRODUCTION

Remediation Technologies, Inc. (RETEC) was contracted to conduct an emission monitoring program for a thermal catalytic oxidizer at the Unisys Corporation, Great Neck, New York facility. The catalytic oxidizer, a new emission source, would control organic vapors from a soil venting system. A Work Plan for the emission monitoring program was written by RETEC and submitted to the New York State Department of Environmental Conservation (NYDEC) for review.

Comments by NYDEC on the Work Plan included a request for sampling and analysis for additional organic constituents, specifically the Target Compound List (TCL) for volatile and semi-volatile organics.

The requested organic compounds were not detected in the off-gas during a pilot program of the soil vapor extraction system by Unisys. In the interest of efficiency, a preliminary emission monitoring program was written by RETEC that would quantitatively screen for the organic constituents requested by NYDEC. Results of the test would be used by RETEC to modify the original compliance emission monitoring work plan for the thermal oxidizer. The preliminary monitoring plan was approved by NYDEC and was subsequently performed by RETEC on June 24, 1993.

2.0 SCOPE OF WORK

The inlet gas stream to the catalytic oxidizer was sampled for the volatile and semivolatile organics of concern, presented in Table 2-1. RETEC used activated charcoal absorbent tubes and constant flow sampling pumps to collect the volatile organics of concern. Semivolatile organics were collected on to XAD-2 absorbent resin tubes using constant flow sampling pumps. Full details of the monitoring procedures are presented in the RETEC Preliminary Stack Test Plan (June 1993).

The inlet sampling port used during the program was located on the off-gas system prior to any air dilution values. This prevented any inclusion of air into the sample stream which might dilute the sample. A portable organic vapor analyzer, equipped with a photo-ionization detector (PID), was used by RETEC to measure the total concentration of volatile organics in the vapor stream. Results from the survey indicated a total VOC concentration of 2,700 ppmv. RETEC calculated a sample run time of 40 minutes (equation presented in the RETEC Preliminary Stack Test Plan) per charcoal tube, based on the total VOC concentration of the sample gas (mg/m³) and a mass of activated charcoal in the tubes (1,200 mg). Since NYDEC requested a total run time of 120 minutes, three charcoal tubes were used.

A single set of XAD-2 resin tubes were used for the entire 120 minute sampling run for semi-volatile organics.

TABLE 2-1

NYDEC Target Compound List

Compound	Compound
1,1-Dichloroethane	Bromoform
1,1-Dichloroethylene	Bromomethane
1,1,2-Trichloroethane	Carbon Tetrachloride
1,1,2,2-Tetrachloroethane	Carbon Disulfide
1,2-Dibromo-3-chloropropane	Chlorobenzene
1,2-Dibromoethane	Chloroethane
1,2-Dichlorobenzene	Chloroform
1,2-Dichloroethylene(total)	Chloromethane
1,2-Dichloropropane	cis-1,3-Dichloropropene
1,3-Dichlorobenzene	Dibromochloromethane
1,4-Dichlorobenzene	Ethyl Benzene
2-Butanone	Styrene
2-Hexanone	Total Xylenes
4-Methyl-2-pentanone	trans-1,3-Dichloropropene
Bromodichloromethanex	Freon-113
Anenaphthene	Aroclor 1232
Acenaphthylene	Aroclor 1242
Anthracene	Aroclor 1248
Benzo(a)anthracene	Aroclor 1254
Benzo(b)fluoranthene	Aroclor 1260
Benzo(k)fluoranthene	Hexachlorobenzene
Benzoic Acid	Hexachlorobutadiene
Benzo(g,h,i)perylene	Hexachlorocyclopentadiene
Benzo(a)pyrene	Hexachioroethane
Benzyl alcohol	Indeno(1,2,3-cd)pyrene
Bis(2-chloroethyl)ether	Isophorone
Bis(2-chloroisopropyl)ether	2-Methinaphthalene
Bis(2-chloroethoxy)methane	Naphthalene
Bis(2-ethylexyl)pthalate	2-Nitroaniline
4-Bromophenyl-phenylether	3-Nitroaniline

TABLE 2-1 con't

NYDEC Target Compound List

Butylbenzylphthalate	4-Nitroaniline		
4-Chloroaniline	Nitrobenzene		
2-Chloronaphthaiene	N-Nitrosodiphenylamine		
4-Chlorophenyl-phenylether	N-Nitroso-di-n-propylamine		
Chrysene	Phenanthrene		
Dibenz(a,h)anthracene	Pyrene		
Dibenzofuran	1,2,4-Trichlorobenzene		
1,2-Dichlorobenezene	2-Chlorophenol		
1,3-Dichlorobenzene	2,4-Dichlorophenol		
1,4-Dichlorobenzene	2,4-Dimethylphenol		
3,3'-Dichlorobenzidine	4,6-Dinitro-2-methyphenol		
Diethylphthalate	2,4-Dimitrophenol		
Dimethylphthalate	2-Methylphenol		
2,4-Dinitrotoluene	4-Methylphenol		
2,6-Dinitrotoluene	2-Nitrophenol		
Di(n)octyl-phthalate	4-Nitrophenol		
Fluoranthene	Pentachlorophenol		
Fluorene	Phenol		
Aroclor 1016	2,4,5-Trichlorophenol		
Aroclor 1221	2,4,6-Trichlorophenol		

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3.0 RESULTS

The preliminary stack test characterized the physical conditions of the soil vapor stream as well as the presence or absence of organic constituents of concern.

The characteristics of the soil vapor stream are presented below:

<u>Variables</u>

<u>Data</u>

Gas temperature:	80 ^{0F}
Barometric pressure:	29.89 inches Hg
Percent moisture of gas:	6.36%
Mole fraction of dry gas:	0.94%
Mole weight of dry gas:	29.00 g/mole
Stack area:	12.57 in ²
Gas velocity:	500 fpm
Gas flow rate:	39.91 dscfm
Total sample volume:	0.04 m ³

3.1 VOC RESULTS

A full listing of VOCs that were analyzer for are presented in the laboratory report, which is include as Appendix A of this report. Sample codes and descriptions for the carbon tubes and condensates analyzed are as follows:

- VOC-1F1 and VOC-2F1: are the front half carbon tubes from sample trains 1 and 2, respectively.
- VOC-1B1 and VOC-2B1: are the back half carbon tubes from sample trains 1 and 2, respectively.
- VOC-FB: is a charcoal tube field blank
- VOC-1W: is the liquid condensate collected in the impinger prior to the carbon tubes

• VOC-1WFB: is a condensate field blank

Charcoal tubes selected for analysis were desorbed using either carbon disulfide or toluene. Toluene was used as a desorption solvent for constituents that elude from the gas chromatograph at approximately the same time as carbon disulfide.

As part of the analytical program, a desorption efficiency (DE) test was performed by spiking the charcoal tubes with known concentrations of VOCs of concern. Results of the desorption efficiency test are presented in the laboratory report. Results indicated desorption efficiencies from 120 percent (trichloroethene) to 34 percent (vinyl chloride). Following EPA and NIOSH procedures, desorption efficiencies were used to correct the detected constituent masses from the carbon tubes (ug/tube). Table 3-2 presents the detected VOCs from the preliminary stack test program.

The mass detected per tube is the summation of the mass detected on the front tube, back tube, and in the liquid condensate. The corrected mass is the total mass divided by the desorption efficiency factor, presented in the laboratory report, for each constituent. Results indicate that trichloroethene is the major component of the VOC stream from the soil vapor system (69.7%). Additional compounds and their percent concentration in the vapor stream are: tetrachloroethene (13.3%); freon 113 (6.9%); and 1,2-dichloroethene (6.6%). The remaining detected VOCs compose less than one percent of soil vapor VOC stream.

The total VOC concentration from the preliminary test was 7,596 ppmv. This concentration is 2.5 times higher than what was recorded by the portable vapor analyzer (3,000 ppmv). Reasons for this discrepancy could be attributed to:

- The portable organic vapor analyzer is calibrated differently than the GC/MS. The vapor analyzer is calibrated with a single benzene equivalent standard of isobutylene. The GC/MS uses a range of VOCs to measure unknown in the sample; and
- The PID used as a detector in the organic vapor analyzer has different sensitivities for the detection of VOCs.

It was also detected during the charcoal tube analysis that compound break-through occurred for chloroform, 1,2-dichloroethane, 1,2-dichloroethene, methylene chloride, 1,1,1-trichloroethane and freon 113. High loadings of trichloroethene on the charcoal tubes may have contributed to the breakthrough of these contaminants.

Table 3-2

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Detected VOCs in Influent Gas Stream

Compound	Mass Detected (ug)	Corrected Mass Detected (ug)	Concentration (ppmv)	Percent of Flow
Benzene	181	206	2	0.013
2-Butanone	19	28	0.2	0.001
Chloroform	93	137	0.7	0.008
1,2-Dichlorobenzene	75	97	0.4	0.006
1,2-Dichloroethane	26	42	0.3	0.003
1,1-Dichloroethene	1,020	1,522	10	0.095
1,2-Dichloroethene (total)	58,075	105,591	666	6.57
Ethylbenzene	1,147	1,318	8	0.082
Methylene Chloride	995	880	6	0.055
Tetrachloroethene	225,225	214,500	790	13.34
Toluene	41,618	44,750	206	2.78
1,1,1-Trichloroethane	2,234	2,510	9	0.156
Trichloroethene	1,344,638	1,120,532	5,232	69.71
Xylene	6,063	9,624	55	0.598
Freon 113	105,740	105,740	610	6.58
Total	1,787,149	1,607,477	7,596	100

8-1153/UNISYS.PLN

July 29, 1993

3.2 SVOC RESULTS

Possible concentrations of SVOCs in the inlet vapor stream were sampled for using XAD-2 absorbent resin. Analytical results show that 1,2-dichlorobenzene and naphthalene were the only SVOCs detected during the preliminary stack test program. 1,2-Dichlorobenzene had a detectable concentration in the gas stream at 4.6 mg/m³. Naphthalene concentration in the gas stream at 4.6 mg/m³. Naphthalene concentration in the gas stream at detected in the vapor stream at detection limits as low as 0.005 mg/m³.

3.3 RECOMMENDATIONS

Results from the preliminary stack monitoring program indicated the presence of four major VOCs in the soil vapor off-gas: trichloroethene; tetrachloroethene; freon 113; and 1,2-dichloroethene. These results also confirm the conclusions presented by Unisys concerning their analysis of the off-gas vapors from the pilot study (Unisys October 1991).

Trichloroethene, tetrachloroethene, and 1,2-dichloroethene are three of the nine principal VOCs used in the generation of the original RETEC Emission Monitoring Work Plan (December 1992) for the thermal oxidizer. The semi-volatile compounds: 1,2-dichlorobenzene and naphthalene were detected at low concentrations in the gas stream.

RETEC recommends that the compliance emission monitoring program for the catalytic oxidizer use the detected VOC compounds from the preliminary stack test program. Sampling and analytical procedures originally presented in the RETEC Work Plan (December 1992) would be used to perform the compliance monitoring program. Semi-volatile compounds: 1,2-dichlorobenzene and naphthalene, would be added to the list of VOCs to be monitored during the compliance program since their are easily collected onto activated charcoal.



REPORT OF ANALYTICAL RESULTS

Case Number: D0625-07 D0625-08

Prepared for:

Remediation Technologies, Inc. 9 Pond Lane Concord, MA 01742 Attn: Rich Roat

Prepared by:

New England Testing Laboratory, Inc. 1254 Douglas Avenue North Providence, RI 02904

Date Reported: 21 JULY 1993

Reviewed By: Made Mark H. Bishop Laboratory Director

NEW ENGLAND TESTING LABORATORY, INC.

1254 Douglas Avenue, North Providence, Rhode Island 02904-5392 • 401-353-3420

Our letters and reports are for the exclusive use of the client to whom they are addressed, and their communication to any others, or the use of the name of the New England Testing Laboratory, Inc. must receive our prior written approval. Our letters and reports apply only to the sample tested and are not necessarily indicative of the qualities of apparently identical or similar products. Samples not destroyed in testing are retained a maximum of thirty (30) days.

Sample Description

The following samples were submitted to New England Testing Laboratory on 25 JUNE 1993:

"Unisys, NY - Desorption Efficiency Evaluation"

D0625-07:

Carbon tubes:

- 1. VOC 1F1
- 2. VOC 1B1 3. VOC 2F1
- 4. VOC 2B1
- 5. VOC FB
 - Impingers: 1. VOČ 1W
 - 2. VOC 1WFB
 - D0625-08:

XAD-2 tubes:

- 1. SVOC 1F
- 2. SVOC 1B
- 3. SVOC FB

The Custody record is included in this report. The samples were assigned internal identification codes (case numbers) for laboratory information management purposes. The case numbers for this sample submission are as follows:

> Case Numbers: D0625-07 D0625-08
Method

Carbon tubes:

Carbon tubes were prepared for analysis by NIOSH Method 1500. A carbon disulfide desorption was used for all compounds except those which elute before or during the elution of the carbon disulfide solvent. The remaining compounds were desorbed with toluene.

After desorption, the extracts were analyzed by EPA Method 8240.

XAD-2 tubes:

XAD-2 tubes were prepared for analysis by extraction with 1:1 methylene chloride:acetone. The extracts were analyzed by EPA Method 8270.

Impinger solutions:

Impinger solutions were analyzed by EPA Method 8240.

Quality Assurance/Control Statements

All samples were found to be properly preserved/cooled upon receipt. Procedure/calibration checks required by the designated protocols were within control limits.

New York Laboratory ID: 11382

Desorption Efficiency Evaluation

A desorption efficiency evaluation was performed for both the carbon disulfide and toluene extraction/analysis sequences. Also, a desorption efficiency evaluation was performed for the XAD-2 extraction/analysis. Results for this analysis are documented on the following three pages.

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Desorption efficiency

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Subject: Volatile Organic Compounds

Compound	<u>Recovery, %</u>
Acetone	61
Benzene	88
Bromodichloromethane	80
Bromoform	108
Bromomethane	38
2-Butanone	68
Carbon disulfide	27
Carbon tetrachloride	74
Chlorobenzene	94
Chlorodibromomethane	102
Chloroethane	60
Chloroform	68
Chloromethane	42
1,2-Dibromo-3-chloropropane	110
1,2-Dibromoethane	111
1,2-Dichlorobenzene	77
1,3-Dichlorobenzene	79
1,4-Dichlorobenzene	82
1,1-Dichloroethane	61
1,2-Dichloroethane	62
1,1-Dichloroethylene	67
1,2-Dichloroethylene (total)	55
1,2-Dichloropropane	92
cis-1,3-Dichloropropene	83
trans-1,3-Dichloropropene	84
Ethylbenzene	87
2-Hexanone	85
Methylene chloride	113
4-Methyl-2-pentanone	95
Styrene	46
1,1,2,2-Tetrachloroethane	89
Tetrachloroethene	105
Toluene	93
1,1,1-Trichloroethane	89
1,1,2-Trichloroethane	111
Trichloroethene	120
Vinyl chloride	34
Xylene, Total	63

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Desorption efficiency

Subject: Semivolatile Base/Neutral Extractable Compounds

•	Compound	Recovery, %
	Acenaphthelene	90
	Acenaphthalene	85
•	Anthracene Revise (s) anthracene	70
	Benzo (a) anthracene	78
	Benzo(b) fluoranthene	51
		78
	Benzoic acia	98
	Benzo(g,n,1) perylene	105 -
	Benzo(a) pyrene	80
	Benzyl alconol	81
	Bis(2-chloroethyl)ether	72
	Bis(2-chloroisopropyl)ether	54
•	Bis(2-chloroethoxy)methane	73
	Bis(2-ethylhexyl) phthalate	116
	4-Bromophenyl phenyl ether	77
-	Butyl benzyl phthalate	113
	4-Chloroaniline	66
	2-Chloronaphthalene	111
	4-Chlorophenyl phenyl ether	74
1	Chrysene	79
	Dibenz(a,h)anthracene	101
	Dibenzofuran	81
	Di-n-butylphthalate	79
	1,2-Dichlorobenzene	81
	1,3-Dichlorobenzene	77
	1,4-Dichlorobenzene	77
•	3,3'-Dichlorobenzidine	57
	Diethyl phthalate	81
	Dimethyl phthalate	85
•	2,4-Dinitrotoluene	76
	2,6-Dinitrotoluene	65
	Di(n)octyl phthalate	107
	Fluoranthene	50
	Fluorene	83
	Hexachlerobenzene	65
	Hexachlorobutadiene	75
	Hexachlorocyclopentadiene	27
	Hexachloroethane	63
	Indeno(1,2,3-cd)pyrene	109

Desorption efficiency

Compound	Recovery, %
Isophorone	66
2-Methylnaphthalene	83
Naphthalene	83
2-Nitroaniline	64
3-Nitroaniline	97
4-Nitroaniline	. 75
Nitrobenzene	69
N-Nitrosodiphenylamine	105
N-Nitroso-di-n-propylamine	53
Phenanthrene	72
Pyrene	106
1,2,4-Trichlorobenzene	84

Semivolatile Acid Extractable Compounds

<u>Compound</u>	<u>Recovery, %</u>
4-Chloro-3-methylphenol	69
2-Chlorophenol	77
2,4-Dichlorophenol •	80
2,4-Dimethylphenol	90
4,6-Dinitro-2-methylphenol	27
2,4-Dinitrophenol	0.27
2-Methylphenol	76
4-Methylphenol	71
2-Nitrophenol	70
4-Nitrophenol	40
Pentachlorophenol	30
Phenol	68
2,4,5-Trichlorophenol	81
2,4,6-Trichlorophenol	82

ANALYTICAL RESULTS

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Subject: Volatile Organic Compounds

	Concentration	Reporting
Compound	<u>ug/tube</u>	<u>Limit</u>
Acetone	N.D.	10
Benzene	172	1
Bromodichloromethane	N.D.	1
Bromoform	N.D.	1
Bromomethane	N.D.	20
2-Butanone	19	10
Carbon disulfide	N.D.	10
Carbon tetrachloride	N.D.	1
Chlorobenzene	N.D.	1
Chlorodibromomethane	N.D.	1
Chloroethane	N.D.	20
Chloroform	16	1
Chloromethane	N.D.	20
1,2-Dibromo-3-chloropropane	N.D.	1
1,2-Dibromoethane	N.D.	1
1,2-Dichlorobenzene	75	1
1,3-Dichlorobenzene	N.D.	1
1,4-Dichlorobenzene	N.D.	1
1,1-Dichloroethane	N.D.	1
1,2-Dichloroethane	13	1
1,1-Dichloroethylene	52	1
1,2-Dichloroethylene (total)	45,600	1
1,2-Dichloropropane	N.D.	1
cis-1,3-Dichloropropene	N.D.	1
trans-1,3-Dichloropropene	N.D.	1
Ethylbenzene	1140	1
2-Hexanone	N.D.	10
Methylene chloride	550	3
4-Methyl-2-pentanone	N.D.	10
Styrene	N.D.	1
1,1,2,2-Tetrachloroethane	N.D.	1
Tetrachloroethene	225,000	1
Toluene	41,500	1
1,1,1-Trichloroethane	1890	1
1,1,2-Trichloroethane	N.D.	1
Trichloroethene	1,340,000	1
Vinyl chloride	N.D.	20
Xylene, Total	6020	1
Freon 113	3740	1
Surrogates:		
Compound	<u>% Recovery</u>	

Toluene d8	92
1,2-Dichloroethane-d4	94
4 BFB	75

Sample: VOC 1B1 and VOC 2B1

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Subject: Volatile Organic Compounds

,		Concentration	Reporting
	Compound	ug/tube	<u>Limit</u>
	Acetone	N.D.	10
	Benzene	8.9	1
L	Bromodichloromethane	N.D.	1
	Bromoform	N.D.	1
	Bromomethane	N.D.	20
	2-Butanone	N.D.	10
	Carbon disulfide	N.D.	10
	Carbon tetrachloride	N.D.	1
	Chlorobenzene	N.D.	1
	Chlorodibromomethane	N.D.	1
	Chloroethane	N.D.	20
	Chloroform	77	ĩ
	Chloromethane	N.D.	20
	1,2-Dibromo-3-chloropropane	N.D.	1
	1,2-Dibromoethane	N.D.	1
	1,2-Dichlorobenzene	N.D.	1
	1,3-Dichlorobenzene	N.D.	1
	1,4-Dichlorobenzene	N.D.	1
	1,1-Dichloroethane	N.D.	1
	1,2-Dichloroethane	13	1
	1,1-Dichloroethylene	968	1
	1,2-Dichloroethylene (total)	11,700	1
	1,2-Dichloropropane	N.D.	1
	cis-1,3-Dichloropropene	N.D.	1
	trans-1,3-Dichloropropene	N.D.	1
	Ethylbenzene	N.D.	1
	2-Hexanone	N.D.	10
	Methylene chloride	445	3
	4-Methy1-2-pentanone	N.D.	10
	Styrene	N.D.	1
	1,1,2,2-Tetrachloroethane	N.D.	1
	Tetrachloroethene	4.3	1
	Toluene	2.6	1
	1,1,1-Trichloroethane	344	1
	1,1,2-Trichloroethane	N.D.	1
	Trichloroethene	4410	1
	Vinyl chloride	N.D.	20
	Xylene, Total	N.D.	1
	' Freon 113	102,000	1
	<u>Surroqates</u> :		
	Compound	% Recovery	

Toluene d8	109
1,2-Dichloroethane-d4	115
4 BFB	74

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Sample: VOC FB

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Subject: Volatile Organic Compounds

		Concentration		Reporting
1	Compound	ug/sample_		Limit
	Acetone	N.D.		10
	Benzene	N.D.		1
i	Bromodichloromethane	N.D.		1
	Bromoform	N.D.		1
	Bromomethane	N.D.		20
	2-Butanone	N.D.		10
	Carbon disulfide	N D.		10
	Carbon tetrachloride	N.D.		1
	Chlorobenzene	N.D.		1
	Chlorodibromomethane	N.D.		1
	Chloroethane	N.D.	· ·	20
	Chloroform	N.D.		1
1	Chloromethane	N.D.		20
	1,2-Dibromo-3-chloropropane	N.D.		1
	1,2-Dibromoethane	N.D.		1
	1,2-Dichlorobenzene	N.D.		1
	1,3-Dichlorobenzene	N.D.		1
	1,4-Dichlorobenzene	N.D.		1
	1,1-Dichloroethane	N.D.		1
,	1,2-Dichloroethane	N.D.		1
	1,1-Dichloroethylene	N.D.		1
	1,2-Dichloroethylene (total)	N.D.		1
i.	1,2-Dichloropropane	N.D.		1
	cis-1,3-Dichloropropene	N.D.		1
	trans-1,3-Dichloropropene	N.D.		1
	Ethylbenzene	N.D.		1
	2-Hexanone	N.D.		10
	Methylene chloride	N.D.		3
	4-Methyl-2-pentanone	N.D.		10
	Styrene	N.D.		l
	1,1,2,2-Tetrachloroethane	N.D.		1
	Tetrachloroethene	N.D.		1
	Toluene	N.D.		1
	1,1,1-Trichloroethane	N.D.		1
	1,1,2-Trichloroethane	N.D.		1
	Trichloroethene	N.D.		1
	Vinyl chloride	N.D.		20
	Xylene, Total	N.D.		1
	Freon 113	N.D.		1
	Surrogates:			

Compound

<u>% Recovery</u>

Toluene d8	96
l,2-Dichloroethane-d4	105
4 BFB	110

Sample: VOC 1W

.

Subject: Volatile Organic Compounds

	Concentration	Reporting
Compound	ug/sample	Limit
Acetone	N.D.	50
Benzene	N.D.	5
Bromodichloromethane	N.D.	5
Bromoform	N.D.	5
Bromomethane	N.D.	100
2-Butanone	N.D.	50
Carbon disulfide	N.D.	50
Carbon tetrachloride	N.D.	5
Chlorobenzene	N.D.	5
Chlorodibromomethane	N.D.	5
Chloroethane	N.D.	100
Chloroform '	N.D.	5
Chloromethane	N.D.	100
1,2-Dibromo-3-chloropropane	N.D.	5
1,2-Dibromoethane	N.D.	5
1,2-Dichlorobenzene	N.D.	5
, 3-Dichlorobenzene	N.D.	5
1,4-Dichlorobenzene	N.D.	5
L,1-Dichloroethane	N.D.	5
1,2-Dichloroethane	N.D.	5
1,1-Dichloroethylene	N.D.	5
1,2-Dichloroethylene (total)	775	5
1,2-Dichloropropane	N.D.	5
cis-1,3-Dichloropropene	N.D.	5
trans-1,3-Dichloropropene	N.D.	5
Ethylbenzene	7.5	5
2-Hexanone	N.D.	50
Methylene chloride	N.D.	200
4-Methyl-2-pentanone	N.D.	50
Styrene	N.D.	5
1.1.2.2-Tetrachloroethane	N.D.	5
Tetrachloroethene	221	5
Тојџеле	115	5
1.1.1-Trichloroethane	N.D.	5
1,1,2-Trichloroethane	N.D.	5
Trichloroethene	228	5
Vinyl chloride	N.D.	100
Xylene, Total	43	5
Freon 113	N.D.	0.05

<u>% Recovery</u>

<u>Surroqates</u>: <u>Compound</u>

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Toluene d8	96
1,2-Dichloroethane-d4	94
4 BFB	88

Sample: VOC 1WFB

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Subject: Volatile Organic Compounds

		Concentration	Reporting			
-	Compound	ug/sample	Limit			
	Acetone	N.D.	0.5			
	Benzene	N.D.	0.05			
	Bromodichloromethane	N.D.	0.05			
	Bromoform	N.D.	0.05			
	Bromomethane	N.D.	1			
-	2-Butanone	N.D.	0.5			
	Carbon disulfide	N.D.	0.5			
	Carbon tetrachloride	N.D.	0.05			
	Chlorobenzene	N.D.	0.05			
-	Chlorodibromomethane	N.D.	0.05			
	Chloroethane	N.D.	1			
	Chloroform	N.D.	0.05			
	Chloromethane	N.D.	1			
	1,2-Dibromo-3-chloropropane	N.D.	0.05			
	1,2-Dibromoethane	N.D.	0.05			
-	1,2-Dichlorobenzene	N.D.	0.05			
_	1,3-Dichlorobenzene	N.D.	0.05			
	1,4-Dichlorobenzene	N.D.	0.05			
	1,1-Dichloroethane	N.D.	0.05			
-	1,2-Dichloroethane	N.D.	0.05			
	1,1-Dichloroethylene	N.D.	0.05			
	1,2-Dichloroethylene (total)	N.D.	0.05			
-	1,2-Dichloropropane	N.D.	0.05			
	cis-1,3-Dichloropropene	N.D.	0.05			
	trans-1,3-Dichloropropene	N.D.	0.05			
	Ethylbenzene	N.D.	0.05			
	2-Hexanone	N.D.	0.5			
	Methylene chloride	N.D.	2			
	4-Methyl-2-pentanone	N.D.	0.5			
-	Styrene	N.D.	0.05			
	1,1,2,2-Tetrachloroethane	N.D.	0.05			
	Tetrachloroethene	N.D.	0.05			
-	Toluene	2.8	0.05			
	1,1,1-Trichloroethane	N.D.	0.05			
	1,1,2-Trichloroethane	N.D.	0.05			
	Trichloroethene	1.2	0.05			
	Vinyl chloride	N.D.	1			
	Xylene, Total	N.D.	0.05			
	Freon 113	N.D.	0.05			

<u>% Recovery</u>

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<u>Surrogates</u>: <u>Compound</u>

Toluene d8	88
1,2-Dichloroethane-d4	77
4 BFB	81

Sample: SVOC 1F and SVOC 1B

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Case No. D0625-08

Compound	Concentration, ug/tube	Reporting Limit
Isophorone	N.D.	5
2-Methylnaphthalene	N.D.	1
Naphthalene	16	1
2-Nitroaniline	N.D.	2
3-Nitroaniline	N.D.	2
4-Nitroaniline	N.D.	2
Nitrobenzene	N.D.	2
N-Nitrosodiphenylamine	N.D.	10
N-Nitroso-di-n-propylamine	N.D.	5
Phenanthrene	N.D.	1
Pyrene	N.D.	1
1,2,4-Trichlorobenzene	N.D.	.1

Semivolatile Acid Extractable Compounds

Compound	Concentration, uq/tube	Reporting Limit				
4-Chloro-3-methylphenol	N.D.	5				
2-Chlorophenol	N.D.	1				
2,4-Dichlorophenol	N.D.	5				
2,4-Dimethylphenol	N.D.	1				
4,6-Dinitro-2-methylphenol	N.D.	20				
2,4-Dinitrophenol	N.D.	20				
2-Methylphenol	N.D.	1				
4-Methylphenol	N.D.	1				
2-Nitrophenol	N.D.	10				
4-Nitrophenol	N.D.	10				
Pentachlorophenol	N.D.	10				
Phenol	N.D.	1				
2,4,5-Trichlorophenol	N.D.	5				
2,4,6-Trichlorophenol	N .D.	5				
Aroclor 1016	N.D.	0.2				
Aroclor 1221	N.D.	0.2				
Aroclor 1232	N.D.	0.2				
Aroclor 1242	N.D.	0.2				
Aroclor 1248	N.D.	1.0				
Aroclor 1254	N.D.	5.0				
Aroclor 1260	N.D.	10.0				
Surrogates:						
Compound	<u>% Recovery</u>					

Compound	<u>% Recovery</u>
Nitrobenzene d5	43
2-Fluorobiphenyl	76
p-Terphenyl dl4	85
Phenol d6	24
2,4,6-Tribromophenol	21
2-Fluorophenol	63

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proj. no. Samplers <i>Rich</i> sample no.	5: (Signat Rom DATE		VISY.	s I	VY Krci SAMPLE L	DCATION	NO. OF CON- TAINERS	4	To. SVO.	LUL VOAS	,			/		RE	MARKS	,
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UM alinguish elinguish	ed by: (S	Signature) Signature)		C/Z.4/43 Date	3 pm	Received by: (Signalu Received for Labora (Signalure)	atory by:	Rellr	nquish	ed by: (ate / 7	'Ime			Date	/ Time	Receive DIATIO	d by: (Signatu	
EMARKS:	5v 1.5	36 T [82] N	F	(LCS	NOC							REM	E D I A	TION		Damonmill Square 9 Pond Lane Concord, MA 01742		

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ROJ. NO.		PROJECT	NAME							-7	-7-	7-	T T		0 000				
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lingulshe	d by: (Si	gnalure)		Da	te / Tin	ne	Received by: (Signat	ure)		Relir	nquish	ed by: (S)	gnaturi	e)		Date) / Time	Received by: (Sig	gnature)
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