

VOC Sampling and Analysis Workshop

Volume II Papers and Lecture Notes



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Volume II. Papers and Lecture Notes

Prepared by

PEDCo Environmental, Inc.
11499 Chester Road
Post Office Box 46100
Cincinnati, Ohio 45246-0100

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
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INTENDED PURPOSE

This is not an official policy and standards document. The opinions, findings, and conclusions are those of the authors and not necessarily those of the Environmental Protection Agency. Every attempt has been made to represent the present state of the art as well as subject areas still under evaluation. Any mention of products or organizations does not constitute endorsement by the United States Environmental Protection Agency.

This document is issued by the Stationary Source Compliance Division, Office of Air Quality Planning and Standards, USEPA. It is for use in workshops presented by Agency staff and others receiving contractual or grant support from the USEPA. It is part of a series of instructional manuals addressing VOC compliance testing procedures.

Governmental air pollution control agencies establishing training programs may receive single copies of this document, free of charge, from the Stationary Source Compliance Division Workshop Coordinator, USEPA, MD-7, Research Triangle Park, NC 27711. Since the document is specially designed to be used in conjunction with other training materials and will be updated and revised as needed periodically, it is not issued as an EPA publication nor copies maintained for public distribution.

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SUMMARY OF TEST METHODS

March 3, 1982/RTS

<u>Method</u>	<u>Reference</u>	<u>Description</u>	<u>Key contact</u>	<u>Telephone</u>
1-8	42 FR 41754 08/18/77 43 FR 11984 03/23/78	Velocity, Orsat, PM, SO ₂ , NO _x , etc. Corr. and amend. to M-1 ² thru ^x 8	Roger Shigehara Gary McAlister Pete Westlin	919-541-2237 919-541-2237 919-541-2237
1A	Tentative	Traverse points in small ducts	Roger Shigehara	919-541-2237
1R	Tentative	Revision to reduce number of traverse points	Roger Shigehara	919-541-2237
2A	45 FR 83126 12/17/80 PP	Flow rate in small ducts	Pete Westlin	919-541-2237
2B	45 FR 83126 12/17/80 PP	Flow rate by stoichiometry	Pete Westlin	919-541-2237
2C	Tentative	Flow rate in small ducts	Bill Grimley	919-541-2237
3R	Tentative	Addition of QA/QC	Pete Westlin	919-541-2237
4R,5R	Tentative	Addition of QA/QC	Pete Westlin	919-541-2237
1 5R	45 FR 66752 10/07/80	Filter specification change	Roger Shigehara	919-541-2237
5A	45 FR 76404 11/18/80 PP	PM from asphalt roofing (PP as M-26)	Pete Westlin	919-541-2237
5B	Tentative	Nonsulfuric acid particulate matter	Roger Shigehara Gary McAlister	919-541-2237 919-541-2237
5C	Tentative	PM from small ducts	Pete Westlin	919-541-2237
6A	46 FR 08352 01/26/81 PP	SO ₂ /CO ₂	Pete Westlin	919-541-2237
6B	46 FR 08352 01/26/81 PP	Auto SO ₂ /CO ₂	Pete Westlin	919-541-2237
7A	Tentative	Ion chromatograph NO _x analysis	Foston Curtis	919-541-2237
6R,7R	Tentative	Addition of QA/QC	Foston Curtis	919-541-2237
9	39 FR 39872 11/12/74	Opacity	Pete Westlin	919-541-2237
9A	46 FR 53144 10/28/81	Lidar opacity	Art Dybdal	303-234-4658
10	39 FR 09319 03/08/78	CO	Pete Westlin	919-541-2237
11	43 FR 01494 01/10/78	H ₂ S	Foston Curtis	919-541-2237
12	47 FR 16564 04/16/82	Pb	Bill Grimley	919-541-2237

(continued)

Summary of Test Methods (continued)

<u>Method</u>	<u>Reference</u>	<u>Description</u>	<u>Key contact</u>	<u>Telephone</u>
13A	45 FR 41852 06/20/80	F, colorimetric method	Gary McAlister	919-541-2237
13B	45 FR 41852 06/20/80 45 FR 85016 12/24/80	F, SIE method Corr. to M-13A and 13B	Gary McAlister	919-541-2237
14	45 FR 44202 06/30/80	F from roof monitors	Bill Grimley	919-541-2237
15	43 FR 10866 03/15/78	TRS from petroleum refineries	Gary McAlister Foston Curtis	919-541-2237 919-541-2237
16	43 FR 07568 02/23/78 43 FR 34784 08/07/78 44 FR 02578 01/12/79	TRS from kraft pulp mills Amendments to M-16, H ₂ S loss after filter Amendments to M-16, SO ₂ scrubber added	Gary McAlister Foston Curtis	919-541-2237 919-541-2237
16A	46 FR 31904 06/18/81	PP TRS alternative	Foston Curtis	919-541-2237
17	43 FR 07568 02/23/78	PM, in-stack	Roger Shigehara	919-541-2237
18	Tentative	VOC, general GC method	Bill Grimley	919-541-2237
19	44 FR 33580 06/11/79	F-factor, coal sampling	Pete Westlin	919-541-2237
19A			Pete Westlin	919-541-2237
20	44 FR 52792 09/10/79	NO _x from gas turbines	Pete Westlin	919-541-2237
21	46 FR 01160 01/05/81	PP VOC leaks	Winton Kelley	919-541-5543
22	45 FR 76404 11/18/80	PP Fugitive VE	John Brown	919-541-2237
23	45 FR 39766 06/11/80	PP Halogenated OC	Bill Grimley	919-541-2237
24	45 FR 65956 10/03/80	Solvent in surface coatings	Gary McAlister	919-541-2237
24A	45 FR 71538 10/28/80	PP Solvent in ink (PP as M-29)	Gary McAlister	919-541-2237
25	45 FR 65956 10/03/80	TGNMO	Gary McAlister	919-541-2237
25A	45 FR 83126 12/17/80	PP TOC/FID	Pete Westlin	919-541-2237
25B	45 FR 83126 12/17/80	PP TOC/NDIR	Pete Westlin	919-541-2237
27	45 FR 83126 12/17/80	Tank truck leaks	Winton Kelley	919-541-5543
	Other methods	Ammonium nitrate PM, Urea PM		

(continued)

Summary of Test Methods (continued)

<u>Method</u>	<u>Reference</u>			<u>Description</u>	<u>Key contact</u>	<u>Telephone</u>
101	47 FR	24703	06/08/82	Hg in air streams	Foston Curtis	919-541-2237
101A	47 FR	24703	06/08/82	Hg in sewage sludge incinerators (PP as M-111)	Foston Curtis	919-541-2237
102	47 FR	24703	06/08/82	Hg in H ₂ streams	Foston Curtis	919-541-2237
103	38 FR	08820	04/06/73	Be, screening method	Bill Grimley	919-541-2237
104	38 FR	08820	04/06/73	Be	Bill Grimley	919-541-2237
105	40 FR	48299	10/14/75	Hg in sewage sludge	Foston Curtis	919-541-2237
106R	45 FR	76346	11/18/80	PP VC	Bill Grimley	919-541-2237
107R	45 FR	76346	11/18/80	PP VC in process streams	Bill Grimley	919-541-2237
107A	46 FR	12188	02/12/81	PP VC in process streams	Bill Grimley	919-541-2237
109	Tentative			Coke oven VE	John Brown	919-541-2237
ω	Other methods			As		
PS-1R	44 FR	58602	10/10/79	PP Opacity	Pete Westlin	919-541-2237
PS-2R	46 FR	08352	01/26/81	PP SO ₂ and NO _x	Pete Westlin Roger Shigehara	919-541-2237 919-541-2237
PS-3R	46 FR	08352	01/26/81	PP CO ₂ and O ₂	Pete Westlin Roger Shigehara	919-541-2237 919-541-2237
PS-4	Tentative			CO	Pete Westlin	919-541-2237
PS-5	46 FR	37287	07/20/81	PP TRS	Foston Curtis	919-541-2237

EPA LIST OF 37 POLLUTANTS

Acetaldehyde	Acrolein
Acrylonitrile	Allyl Chloride
Benzyl Chloride	Beryllium
Cadmium	Carbon Tetrachloride
Chlorobenzene	Chloroform
Chloroprene	Coke Oven Emissions
o-,m-,p- Cresol	p-Dichlorobenzene
Dimethyl Nitrosamine	Dioxin
Epichlorohydrin	Ethylene Dichloride
Ethylene Oxide	Formaldehyde
Hexachlorocyclopentadiene	Maleic Anhydride
Methylene Chloride (dichloromethane)	Methyl Chloroform (1,1,1 trichloroethane)
Manganese	Nickel
Nitrobenzene	Nitrosomorpholine
Perchloroethylene	Phenol
Phosgene	Polychlorinated Biphenyls
Propylene Oxide	Toluene
Trichloroethylene	Vinylidene Chloride
o-,m-,p- Xylene	

Dated: August 4, 1983.
William D. Ruckelshaus,
Administrator.

PART 60—[AMENDED]

40 CFR Part 60 is amended as follows:

1. By adding a new subpart as follows:

Subpart XX—Standards of Performance for Bulk Gasoline Terminals

Sec.

60.500 Applicability and designation of affected facility.

60.501 Definitions.

60.502 Standards for Volatile Organic Compound (VOC) emissions from bulk gasoline terminals.

60.503 Test methods and procedures.

60.504 [Reserved.]

60.505 Reporting and recordkeeping.

60.506 Reconstruction.

Authority: Sections 111 and 301(a) of the Clean Air Act, as amended [42 U.S.C. 7411, 7601(a)], and additional authority as noted below.

Subpart XX—Standards of Performance for Bulk Gasoline Terminals

§ 60.500 Applicability and designation of affected facility.

(a) The affected facility to which the provisions of this subpart apply is the total of all the loading racks at a bulk gasoline terminal which deliver liquid product into gasoline tank trucks.

(b) Each facility under paragraph (a) of this section, the construction or modification of which is commenced after December 17, 1980, is subject to the provisions of this subpart.

(c) For purposes of this subpart, any replacement of components of an existing facility, described in paragraph § 60.500(a), commenced before August 18, 1983 in order to comply with any emission standard adopted by a State or political subdivision thereof will not be considered a reconstruction under the provisions of 40 CFR 60.15.

[Note: The intent of these standards is to minimize the emissions of VOC through the application of best demonstrated technologies (BDT). The numerical emission limits in this standard are expressed in terms of total organic compounds. This emission limit reflects the performance of BDT.]

§ 60.501 Definitions.

The terms used in this subpart are defined in the Clean Air Act, in § 60.2 of this part, or in this section as follows:

"Bulk gasoline terminal" means any gasoline facility which receives gasoline by pipeline, ship or barge, and has a gasoline throughput greater than 75,700 liters per day. Gasoline throughput shall be the maximum calculated design throughput as may be limited by compliance with an enforceable

condition under Federal, State or local law and discoverable by the Administrator and any other person.

"Continuous vapor processing system" means a vapor processing system that treats total organic compounds vapors collected from gasoline tank trucks on a demand basis without intermediate accumulation in a vapor holder.

"Existing vapor processing system" means a vapor processing system [capable of achieving emissions to the atmosphere no greater than 80 milligrams of total organic compounds per liter of gasoline loaded], the construction or refurbishment of which was commenced before December 17, 1980, and which was not constructed or refurbished after that date.

"Gasoline" means any petroleum distillate or petroleum distillate/alcohol blend having a Reid vapor pressure of 27.6 kilopascals or greater which is used as a fuel for internal combustion engines.

"Gasoline tank truck" means a delivery tank truck used at bulk gasoline terminals which is loading gasoline or which has loaded gasoline on the immediately previous load.

"Intermittent vapor processing system" means a vapor processing system that employs an intermediate vapor holder to accumulate total organic compounds vapors collected from gasoline tank trucks, and treats the accumulated vapors only during automatically controlled cycles.

"Loading rack" means the loading arms, pumps, meters, shutoff valves, relief valves, and other piping and valves necessary to fill delivery tank trucks.

"Refurbishment" means, with reference to a vapor processing system, replacement of components of, or addition of components to, the system within any 2-year period such that the fixed capital cost of the new components required for such component replacement or addition exceeds 50 percent of the cost of a comparable entirely new system.

"Total organic compounds" means those compounds measured according to the procedures in § 60.503.

"Vapor collection system" means any equipment used for containing total organic compounds vapors displaced during the loading of gasoline tank trucks.

"Vapor processing system" means all equipment used for recovering or oxidizing total organic compounds vapors displaced from the affected facility.

"Vapor-tight gasoline tank truck" means a gasoline tank truck which has

demonstrated within the 12 preceding months that its product delivery tank will sustain a pressure change of not more than 750 pascals (75 mm of water) within 5 minutes after it is pressurized to 4,500 pascals (450 mm of water). This capability is to be demonstrated using the pressure test procedure specified in Reference Method 27.

§ 60.502 Standard for Volatile Organic Compound (VOC) emissions from bulk gasoline terminals.

On and after the date on which § 60.8(b) requires a performance test to be completed, the owner or operator of each bulk gasoline terminal containing an affected-facility shall comply with the requirements of this section.

(a) Each affected facility shall be equipped with a vapor collection system designed to collect the total organic compounds vapors displaced from tank trucks during product loading.

(b) The emissions to the atmosphere from the vapor collection system due to the loading of liquid product into gasoline tank trucks are not to exceed 35 milligrams of total organic compounds per liter of gasoline loaded, except as noted in paragraph (c) of this section.

(c) For each affected facility equipped with an existing vapor processing system, the emissions to the atmosphere from the vapor collection system due to the loading of liquid product into gasoline tank trucks are not to exceed 80 milligrams of total organic compounds per liter of gasoline loaded.

(d) Each vapor collection system shall be designed to prevent any total organic compounds vapors collected at one loading rack from passing to another loading rack.

(e) Loadings of liquid product into gasoline tank trucks shall be limited to vapor-tight gasoline tank trucks using the following procedures:

(1) The owner or operator shall obtain the vapor tightness documentation described in § 60.505(b) for each gasoline tank truck which is to be loaded at the affected facility.

(2) The owner or operator shall require the tank identification number to be recorded as each gasoline tank truck is loaded at the affected facility.

(3) The owner or operator shall cross-check each tank identification number obtained in (e)(2) of this section with the file of tank vapor tightness documentation within 2 weeks after the corresponding tank is loaded.

(4) The terminal owner or operator shall notify the owner or operator of each nonvapor-tight gasoline tank truck loaded at the affected facility within 3 weeks after the loading has occurred.

(5) The terminal owner or operator shall take steps assuring that the nonvapor-tight gasoline tank truck will not be reloaded at the affected facility until vapor tightness documentation for that tank is obtained.

(6) Alternate procedures to those described in (e)(1) through (5) of this section for limiting gasoline tank truck loadings may be used upon application to, and approval by, the Administrator.

(f) The owner or operator shall act to assure that loadings of gasoline tank trucks at the affected facility are made only into tanks equipped with vapor collection equipment that is compatible with the terminal's vapor collection system.

(g) The owner or operator shall act to assure that the terminal's and the tank truck's vapor collection systems are connected during each loading of a gasoline tank truck at the affected facility. Examples of actions to accomplish this include training drivers in the hookup procedures and posting visible reminder signs at the affected loading racks.

(h) The vapor collection and liquid loading equipment shall be designed and operated to prevent gauge pressure in the delivery tank from exceeding 4,500 pascals (450 mm of water) during product loading. This level is not to be exceeded when measured by the procedures specified in § 60.503(b).

(i) No pressure-vacuum vent in the bulk gasoline terminal's vapor collection system shall begin to open at a system pressure less than 4,500 pascals (450 mm of water).

(j) Each calendar month, the vapor collection system, the vapor processing system, and each loading rack handling gasoline shall be inspected during the loading of gasoline tank trucks for total organic compounds liquid or vapor leaks. For purposes of this paragraph, detection methods incorporating sight, sound, or smell are acceptable. Each detection of a leak shall be recorded and the source of the leak repaired within 15 calendar days after it is detected.

(Approved by the Office of Management and Budget under control number 2060-0006)

§ 60.503 Test methods and procedures.

(a) Section 60.8(f) does not apply to the performance test procedures required by this subpart.

(b) For the purpose of determining compliance with § 60.502(h), the following procedures shall be used:

(1) Calibrate and install a pressure measurement device (liquid manometer, magnehelic gauge, or equivalent

instrument), capable of measuring up to 500 mm of water gauge pressure with ± 2.5 mm of water precision.

(2) Connect the pressure measurement device to a pressure tap in the terminal's vapor collection system, located as close as possible to the connection with the gasoline tank truck.

(3) During the performance test, record the pressure every 5 minutes while a gasoline tank truck is being loaded, and record the highest instantaneous pressure that occurs during each loading. Every loading position must be tested at least once during the

(c) For the purpose of determining compliance with the mass emission limitations of § 60.502(b) and (c), the following reference methods shall be used:

(1) For the determination of volume at the exhaust vent:

(i) Method 2B for combustion vapor processing systems.

(ii) Method 2A for all other vapor processing systems.

(2) For the determination of total organic compounds concentration at the exhaust vent, Method 25A or 25B. The calibration gas shall be either propane or butane.

(d) Immediately prior to a performance test required for determination of compliance with § 60.502(b), (c), and (h), all potential sources of vapor leakage in the terminal's vapor collection system equipment shall be monitored for leaks using Method 21. The monitoring shall be conducted only while a gasoline tank truck is being loaded. A reading of 10,000 ppmv or greater as methane shall be considered a leak. All leaks shall be repaired prior to conducting the performance test.

(e) The test procedure for determining compliance with § 60.502(b) and (c) is as follows:

(1) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(2) The time period for a performance test shall be not less than 6 hours, during which at least 300,000 liters of gasoline are loaded. If the throughput criterion is not met during the initial 6 hours, the test may be either continued until the throughput criterion is met, or resumed the next day with another complete 6 hours of testing. As much as possible, testing should be conducted during the 6-hour period in which the highest throughput normally occurs.

(3) For intermittent vapor processing systems:

(i) The vapor holder level shall be recorded at the start of the performance test. The end of the performance test shall coincide with a time when the vapor holder is at its original level.

(ii) At least two startups and shutdowns of the vapor processor shall occur during the performance test. If this does not occur under automatically controlled operation, the system shall be manually controlled.

(4) The volume of gasoline dispensed during the performance test period at all loading racks whose vapor emissions are controlled by the processing system being tested shall be determined. This volume may be determined from terminal records or from gasoline dispensing meters at each loading rack.

(5) An emission testing interval shall consist of each 5-minute period during the performance test. For each interval:

(i) The reading from each measurement instrument shall be recorded, and

(ii) The volume exhausted and the average total organic compounds concentration in the exhaust vent shall be determined, as specified in the appropriate test method. The average total organic compounds concentration shall correspond to the volume measurement by taking into account the sampling system response time.

(6) The mass emitted during each testing interval shall be calculated as follows:

$$M_{e_i} = 10^{-6} K V_{e_i} C_e$$

where:

M_{e_i} = mass of total organic compounds emitted during testing interval i , mg.

V_{e_i} = volume of air-vapor mixture exhausted, m^3 , at standard conditions.

C_e = total organic compounds concentration (as measured) at the exhaust vent, ppmv.

K = density of calibration gas, mg/m^3 , at standard conditions = 1.83×10^6 for propane = 2.41×10^6 for butane.

s = standard conditions, 20°C and 760 mm Hg.

(7) The total organic compounds mass emissions shall be calculated as follows:

$$E = \frac{\sum_{i=1}^n M_{e_i}}{L}$$

where:

E = mass of total organic compounds emitted per volume of gasoline loaded, mg/liter.

M_{e_i} = mass of total organic compounds emitted during testing interval i , mg.

L = total volume of gasoline loaded, liters.

n = number of testing intervals.

(f) The owner or operator may adjust the emission results to exclude the methane and ethane content in the exhaust vent by any method approved by the Administrator.

[Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414)]

(Approved by the Office of Management and Budget under control number 2060-0006.)

§ 60.504 [Reserved].

§ 60.505 Reporting and recordkeeping.

(a) The tank truck vapor tightness documentation required under 160.502(e)(1) shall be kept on file at the terminal in a permanent form available for inspection.

(b) The documentation file for each gasoline tank truck shall be updated at least once per year to reflect current test results as determined by Method 27. This documentation shall include, as a minimum, the following information:

(1) Test Title: Gasoline Delivery Tank Pressure Test—EPA Reference Method 27.

(2) Tank Owner and Address.

(3) Tank Identification Number.

(4) Testing Location.

(5) Date of Test.

(6) Tester Name and Signature.

(7) Witnessing Inspector, if any: Name, Signature, and Affiliation.

(8) Test Results: Actual Pressure Change in 5 minutes, mm of water (average for 2 runs).

(c) A record of each monthly leak inspection required under § 60.502(j) shall be kept on file at the terminal for at least 2 years. Inspection records shall include, as a minimum, the following information:

(1) Date of Inspection.

(2) Findings (may indicate no leaks discovered; or location, nature, and severity of each leak).

(3) Leak determination method.

(4) Corrective Action (date each leak repaired; reasons for any repair interval in excess of 15 days).

(5) Inspector Name and Signature.

(d) The terminal owner or operator shall keep documentation of all notifications required under § 60.502(e)(4) on file at the terminal for at least 2 years.

(e) [Reserved].

(f) The owner or operator of an affected facility shall keep records of all replacements or additions of components performed on an existing vapor processing system for at least 3 years.

[Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414)]

(Approved by the Office of Management and Budget under control number 2060-0006.)

§ 60.506 Reconstruction.

For purposes of this subpart:

(a) The cost of the following frequently replaced components of the affected facility shall not be considered in calculating either the "fixed capital cost of the new components" or the "fixed capital costs that would be required to construct a comparable entirely new facility" under § 60.15: pump seals, loading arm gaskets and swivels, coupler gaskets, overfill sensor couplers and cables, flexible vapor hoses, and grounding cables and connectors.

(b) Under § 60.15, the "fixed capital cost of the new components" includes the fixed capital cost of all depreciable components [except components specified in § 60.506(a)] which are or will be replaced pursuant to all continuous programs of component replacement which are commenced within any 2-year period following December 17, 1980. For purposes of this paragraph, "commenced" means that an owner or operator has undertaken a continuous program of component replacement or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of component replacement.

[Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414)]

2. By adding five new Reference Methods (Method 2A, Method 2B, Method 25A, Method 25B, and Method 27) to Appendix A as follows:

Appendix A—Reference Methods

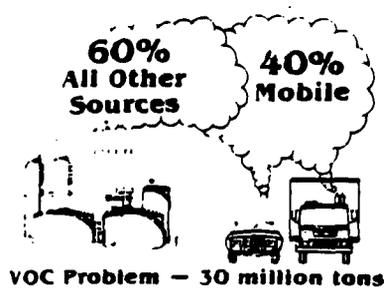
* * * * *

SLIDE 401-1

NOTES

VOC VOLATILE ORGANIC COMPOUNDS

SLIDE 401-2

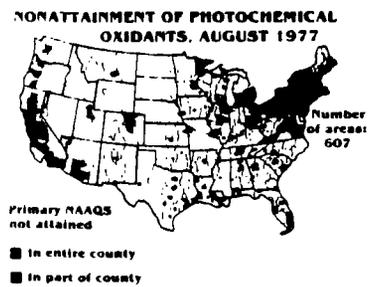


SLIDE 401-3



SLIDE 401-4

NOTES



SLIDE 401-5

**EPA issues CTG's
Control Techniques
Guidelines.**

SLIDE 401-6

CTG

- Working document
- Guidance to states

SLIDE 401-7

**STATE REGULATIONS AND PERMITS
CAN VARY FROM CTG AND MODEL
REGULATION**

**GROUP I
CTG SOURCE CATEGORIES**

SURFACE COATING OF:

- Cans
- Metal coils
- Paper products
- Automobile and light trucks
- Metal furniture
- Magnet wire
- Large appliances

SLIDE 401-9

**GROUP I
CTG SOURCE CATEGORIES
(continued)**

- Fixed roof tanks-storage of liquid petroleum
- Bulk terminals-gasoline loading terminals
- Stage I vapor control system gasoline stations
- Petroleum refineries
- Cutback asphalt
- Degreasers

SLIDE 401-10

**GROUP II
CTG SOURCE CATEGORIES**

- Leaks from petroleum refineries
- Miscellaneous metal parts surface coating
- Surface coating of flat wood paneling
- Pharmaceutical manufacture

GROUP II
CTG SOURCE CATEGORIES
(continued)

- Rubber tire manufacture
- External floating roof petroleum tanks
- Graphic arts
- Perchloroethylene dry cleaning
- Gasoline truck leaks and vapor collection

SLIDE 401-12

GROUP III
CTG SOURCE CATEGORIES

- Large petroleum dry cleaners
- SOCMI-fugitive
- Natural gas/gasoline processing plants
- VOC storage vessels
- SOCMI-air oxidation

SLIDE 401-13

REGULATIONS AFFECTING
EXISTING SOURCES

EXAMPLES:

- SIP — RACT
- Federal, state, and local permit systems

SLIDE 401-14

RACT
REASONABLY AVAILABLE
CONTROL TECHNOLOGY

- Reasonably available technology
- Considers cost

**REGULATIONS
AFFECTING NEW SOURCE
CONSTRUCTION**

- NEW SOURCE REVIEW (NSR)**
- Process for reviewing new sources

SLIDE 401-16

**NSPS
NEW SOURCE
PERFORMANCE STANDARDS**

- Promulgated for various source categories
- Specify emission limitations
- Can be found in CFR

SLIDE 401-17

**BACT
BEST AVAILABLE
CONTROL TECHNOLOGY**

- Best technology available
- Considers cost and energy requirements

SLIDE 401-18

**LAER
LOWEST ACHIEVABLE
EMISSION RATE**

- Control devices to achieve lowest possible emission rate
- Required for sources in nonattainment areas

**PSD
PREVENTION OF
SIGNIFICANT DETERIORATION**

- EPA policy applied to new sources in an attainment area

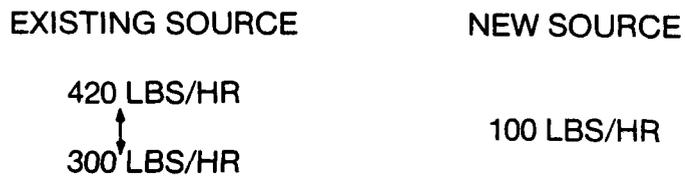
SLIDE 401-20

CONTROLLED TRADING

- Offset policy
- Bubble policy
- Banking emissions

SLIDE 401-21

VOC OFFSET



SLIDE 401-22

**BUBBLE
POLICY**

SUBPART

- Application and designation of affected facility
- Definitions
- Standards of pollutants from each facility
- Test methods and procedures
- Reporting and record keeping

VOLATILE ORGANIC COMPOUND

Defn.: "A volatile organic compound (VOC) is any organic compound that, when released to the atmosphere, can remain long enough to participate in photochemical reactions...almost all organics which can be considered VOC have vapor pressures >0.1 mm Hg at 20°C and 760 mm Hg."

Note: Some VOCs may have vapor pressures <0.1 mm Hg.

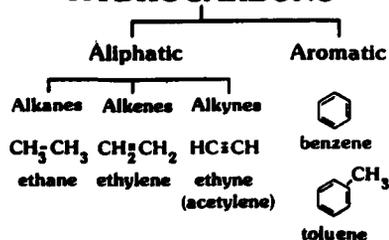
SLIDE 402-2

HC Hydrocarbon

THC Total Hydrocarbon

NMHC NonMethane Hydrocarbon

SLIDE 402-3

HYDROCARBONS

NOMENCLATURE

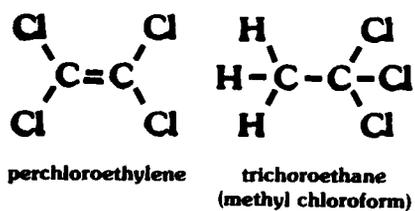
meth - 1 carbon	CH_4	methane
eth - 2 carbons	C_2H_6	ethane
prop - 3 carbons	C_3H_8	propane
but - 4 carbons	C_4H_{10}	butane
pent - 5 carbons	C_5H_{12}	pentane
hex - 6 carbons	C_6H_{14}	hexane
hept - 7 carbons	C_7H_{16}	heptane
oct - 8 carbons	C_8H_{18}	octane

SLIDE 402-5

Alcohols	Aldehydes	Ketones
R-OH	$\text{R}-\overset{\text{O}}{\parallel}{\text{C}}-\text{H}$	$\text{R}_1-\overset{\text{O}}{\parallel}{\text{C}}-\text{R}_2$

Esters	Acids	Ethers
$\text{R}-\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-\text{R}$	$\text{R}-\overset{\text{O}}{\parallel}{\text{C}}-\text{OH}$	R-O-R

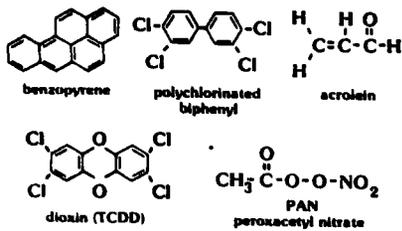
SLIDE 402-6

HALOCARBONS

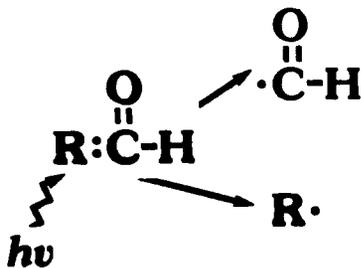
SLIDE 402-7

NITROGEN COMPOUNDS

Nitroparaffins	Amines
$\text{CH}_3-\text{CH}_2-\text{O}-\overset{\text{O}}{\parallel}{\text{N}}=\text{O}$ ethyl nitrate	R-NH_2
$\text{CH}_3-\text{CH}_2-\overset{\text{O}}{\parallel}{\text{N}}=\text{O}$ nitroethane	$\begin{array}{c} \text{H} \\ \\ \text{CH}_3-\text{C}-\text{CH}_3 \\ \\ \text{NH}_2 \end{array}$ 2-propylamine

NASTIES

SLIDE 402-9



SLIDE 402-10

RULE 66

high reactive HC's
replaced with
low reactive HC's

SLIDE 402-11

EPA REVIEW OF RULE 66

- little reduction of oxidant levels
- few low reactivity organics
- many VOC: suspected mutagens, carcinogens, or teratogens
- ozone layer problems
- low reactivity compounds have limited applications

SLIDE 403-1

NOTES

APPLICATION OF EPA VOC REFERENCE METHODS

SLIDE 403-2

CONSIDER LIMITATIONS OF PERFORMANCE EVALUATION TECHNIQUES WHEN REGULATIONS ARE WRITTEN

- Simplicity
- Cost and availability
- Accuracy
- Definition of applicable regulation

SLIDE 403-3

SOURCE PERFORMANCE EVALUATION TECHNIQUES

1. Stack tests
2. Material balance techniques
3. Equipment design parameters

SLIDE 403-4



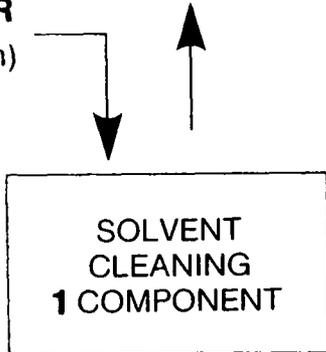
CALIBRATE FOR EACH COMPOUND AND MEASURE TRUE MASS

- Relatively easy for one or two compounds
- Difficulty increases with number

SLIDE 403-5

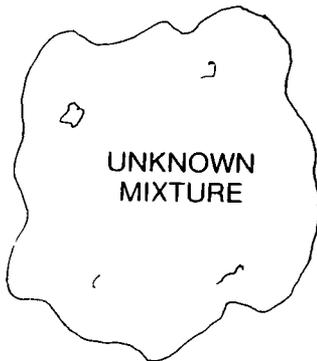
NOTES

LB/HR
(known)



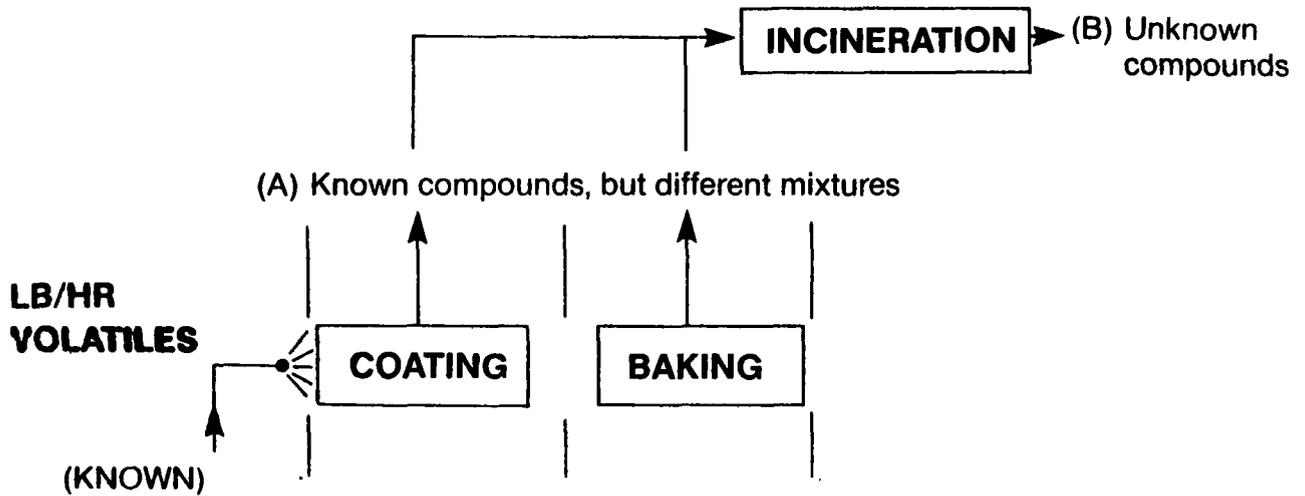
FOR TRUE MASS
FID calibrated with the known
compound

SLIDE 403-6



**GENERALLY, CANNOT REASONABLY
MEASURE TRUE MASS**

- a. For relatively constant mixture — may be able to get somewhat consistent results from source to source
Examples: automotive emission, ambient air
- b. Can more easily make substitute measurement: total carbon
Example: EPA Method 25



(A) FOR TRUE MASS

1. GC/FID with extensive calibration
2. Volatile content of paint (as applicable)

(B) FOR TRUE MASS

1. GC/MS
2. Approximate by Method 25

Note: When open flame is present, the compounds may change. GC/MS is very expensive and is truly a laboratory instrument.

NOTES

VOC ANALYZERS RESPONSE

COMPOUND	MOL. WT.	FID	M 25
CH ₄	16	16	1 carbon
CH ₃ OH	32	~ 7	1 carbon

Note: The complexity of obtaining a true mass increases with the selection of the sampling technique. Adsorption and desorption efficiencies, condensation and reactions must be eliminated or accounted for.

SLIDE 403-9

NO UNIVERSAL VOC EVALUATION TECHNIQUE EXISTS

SLIDE 403-10

SUMMARY

1. Determine best method for compliance determination
 - Material balance (record keeping)
 - Equipment design parameters
 - Pollutant measurement by testing
2. Determine allowable emissions

SLIDE 403-11

(cont)

3. Establish compliance evaluation protocol and give definition of emissions
 - % efficiency, as _____
 - lb/hr, as _____
 - ppm, as _____
 - lb/lb of processed material, as _____

METHOD 106
DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES
(Federal Register, Vol. 47, No. 173, Tuesday, September 7, 1982)

7.3 QUALITY ASSURANCE

7.3.1 Analysis Audit

Immediately after the preparation of the calibration curve and prior to the sample analyses, perform the analysis audit described in Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."

7.3.2 Bag Leak Checks

Checking of bags for leaks is required after bag use and strongly recommended before bag use. After each use, connect a water manometer and pressurize the bag to 5 to 10 cm H₂O (2 to 4 in. H₂O). Allow to stand for 10 min. Any displacement in the water manometer indicates a leak. Also, check the rigid container for leaks in this manner. (Note: An alternative leak check method is to pressurize the bag to 5 to 10 cm H₂O and allow it to stand overnight. A deflated bag indicates a leak.) For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

APPENDIX C - QUALITY ASSURANCE PROCEDURES

Procedure 1 - Determination of Adequate Chromatographic Peak Resolution

In this method of dealing with resolution, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from -2σ to $+2\sigma$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two peaks are separated by a known distance, b , one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compound overlaps the

curve of a compound that is under analysis is found by integrating the contaminant curve over the limits $b-2\sigma_s$ to $b+2\sigma_s$, where σ_s is the standard deviation of the sample curve.

This calculation can be simplified in several ways. Overlap can be determined for curves of unit area; then actual areas can be introduced. Desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calculation programs and tables. An example would be in Program 15 in Texas Instruments Program Manual ST1, 1975, Texas Instruments, Inc., Dallas, Texas 75222.

In judging the suitability of alternate GC columns or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In cases where the sample peak is followed by a contaminant that has a leading edge that rises sharply but the curve then tails off, it may be possible to define an effective width for t_c as "twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line."

Procedure 2 - Procedure for Field Auditing GC Analysis

Responsibilities of audit supervisor and analyst at the source sampling site include the following:

- A. The audit supervisor verifies that audit cylinders are stored in a safe location both before and after the audit to prevent vandalism.
- B. At the beginning and conclusion of the audit, the analyst records each cylinder number and pressure. An audit cylinder is never analyzed when the pressure drops below 200 psi.
- C. During the audit, the analyst performs a minimum of two consecutive analyses of each audit cylinder gas. The audit must be conducted to coincide with the analysis of source test samples, normally immediately after GC calibration and prior to sample analyses.

- D. At the end of audit analyses, the audit supervisor requests the calculated concentrations from the analyst and compares the results with the actual audit concentrations. If each measured concentration agrees with the respective actual concentration within ± 10 percent, he directs the analyst to begin analyzing source samples. Audit supervisor judgment and/or supervisory policy determine action when agreement is not within ± 10 percent. When a consistent bias in excess of 10 percent is found, it may be possible to proceed with the sample analysis, with a corrective factor to be applied to the results at a later time. However, every attempt should be made to locate the cause of the discrepancy, as it may be misleading. The audit supervisor records each cylinder number, cylinder pressure (at the end of the audit), and all calculated concentrations. The individual being audited must not under any circumstance be told actual audit concentrations until calculated concentrations have been submitted to the audit supervisor.

FIELD AUDIT REPORT

Part A

Part A is to be filled out by organization supplying audit cylinders.

1. Organization supplying audit sample(s) and shipping address:

2. Audit supervisor, organization, and phone number:

3. Shipping instructions (name, address, attention):

4. Guaranteed arrival date for cylinders:

5. Planned shipping date for cylinders:

6. Details on audit cylinders from last analysis:

	Low conc.	High conc.
a. Date of last analysis.....
b. Cylinder number.....
c. Cylinder pressure, psi.....
d. Audit gas(es)/balance gas.....
e. Audit gas(es), ppm.....
f. Cylinder construction.....

Part B

Part B is to be filled out by audit supervisor.

1. Process sampled: _____

2. Audit location: _____

3. Name of individual audit: _____
4. Audit date: _____
5. Audit results:

	Low conc. cylinder	High conc. cylinder
a. Cylinder number.....
b. Cylinder pressure before audit, psi.....
c. Cylinder pressure after audit, psi.....
d. Measured concentration, ppm Injection #1* Injection #2* Average.....
e. Actual audit concentration, ppm (Part A, 6e).....
f. Audit accuracy ¹ Low Conc. Cylinder.....
High Conc. Cylinder.....
Percent ¹ accuracy = $\frac{\text{Measured Conc.} - \text{Actual Conc.}}{\text{Actual Conc.}} \times 100$		
g. Problems detected (if any).....

¹Results of two consecutive injections that meet the sample analysis criteria of the test method.

SUPPLEMENT A

DETERMINATION OF ADEQUATE CHROMATOGRAPHIC PEAK RESOLUTION

In this method of dealing with resolution, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from $-2\sigma_s$ to $+2\sigma_s$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two peaks are separated by a known distance, b , one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compounds overlaps the curve of a compound that is under analysis is found by integrating the contaminant curve over the limits $b-2\sigma_s$ to $b+2\sigma_s$, where σ_s is the standard deviation of the sample curve.

There are several ways this calculation can be simplified. Overlap can be determined for curves of unit area and then actual areas can be introduced. The desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calculation programs and tables. An example would be Program 15 in Texas Instruments Program Manual ST1, 1975, Texas Instruments Inc., Dallas, Texas 75222.

$$\frac{1}{\sqrt{2\pi} \sigma_c} \int_{b-2\sigma_s}^{b+2\sigma_s} e^{-\frac{t_c^2}{2\sigma_c^2}} dt = \frac{1}{\sqrt{2\pi}} \int_{\frac{b-2\sigma_s}{\sigma_c}}^{\infty} e^{-\frac{x^2}{2}} dx - \frac{1}{\sqrt{2\pi}} \int_{\frac{b+2\sigma_s}{\sigma_c}}^{\infty} e^{-\frac{x^2}{2}} dx.$$

The following calculation steps are required:*

$$1. \quad 2\sigma_s = t_s / \sqrt{2 \ln 2}$$

$$2. \quad \sigma_c = t_c / 2\sqrt{2 \ln 2}$$

$$3. \quad x_1 = (b - 2\sigma_s) / \sigma_c$$

$$4. \quad x_2 = (b + 2\sigma_s) / \sigma_c$$

$$5. \quad Q(x_1) = \frac{1}{\sqrt{2\pi}} \int_{x_1}^{\infty} e^{-\frac{x^2}{2}} dx$$

$$6. \quad Q(x_2) = \frac{1}{\sqrt{2\pi}} \int_{x_2}^{\infty} e^{-\frac{x^2}{2}} dx$$

$$7. \quad I_o = Q(x_1) - Q(x_2).$$

$$8. \quad A_o = I_o A_c / A_s$$

$$9. \quad \% \text{ overlap} = A_o \times 100$$

* (Note: In most instances, $Q(x_2)$ is very small and may be neglected.)

Where:

- A_s = The area of the sample peak of interest determined by electronic integration, or by the formula $A_s = h_s t_s$.
- A_c = The area of the contaminant peak, determined in the same manner as A_s .
- b = The distance on the chromatographic chart that separates the maxima of the two peaks.
- h_s = The peak height of the sample compound of interest, measured from the average value of the baseline to the maximum of the curve.
- t_s = The width of the sample peak of interest at 1/2 of peak height.
- t_c = The width of the contaminant peak at 1/2 of peak height.
- σ_s = The standard deviation of the sample compound of interest elution curve.
- σ_c = The standard deviation of the contaminant elution curve.
- $Q(x_1)$ = The integral of the normal distribution function from x_1 to infinity.
- $Q(x_2)$ = The integral of the normal distribution function from x_2 to infinity.
- I_o = The overlap integral.
- A_o = The area overlap fraction

In judging the suitability of alternate gas chromatographic columns, or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In those cases where the sample peak is followed by a contaminant that has a leading edge that rises sharply but the curve then tails off, it may be possible to define an effective width for t_c as "twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line."

EPA'S QUALITY ASSURANCE PROGRAM

SLIDE 404-2

PERFORMANCE AUDITS ON SOURCE EMISSION ORGANIC ANALYSIS

BACKGROUND

In 1977, in support of OAQPS, a program was initiated to audit the GC analysis of source test samples. Currently, this audit program has been extended to all EPA, state and local agencies and their contractors.

SLIDE 404-3

REASON FOR AUDITS OF ORGANIC ANALYSIS

OAQPS conducts source tests in setting new regulations. In addition, EPA and states require source owners to test for (1) operating permits and (2) demonstrate compliance to existing regulations. The accuracy and precision of source tests must be known.

SLIDE 404-4

THE ROLE OF QUALITY ASSURANCE IN VOC TESTING

Defn: PERFORMANCE AUDIT

Performance audits refer to independent checks made by supervisor or auditor to evaluate the quality of data and are categorized as:

1. Sampling Audits
2. Analysis Audits
3. Data Processing Audits

SLIDE 404-6

Defn: SYSTEM AUDITS

A system audit is an on-site inspection and review of the quality assurance system used for the total measurement system. Whereas performance audits are a quantitative appraisal, system audits are normally a qualitative appraisal.

SLIDE 404-7

TECHNICAL APPROACH FOR ORGANIC AUDITS

1. Each audit consists of two gas cylinders:
 - a) low concentration 5-20 ppm simulates emission standard
 - b) high concentration 50-700 ppm simulates source emissions
2. Cylinders are analyzed 3 times during the period the source test samples are analyzed.
3. Audit results are returned to the auditor and accuracy and precision are calculated and reported to the auditee and requesting agency.

TABLE 1. AUDIT MATERIALS CURRENTLY HELD IN THE REPOSITORY

Compound	Low Concentration Range			High Concentration Range		
	No. of Cylinders	Concentration Range (ppm)	Cylinder Construction*	No. of Cylinders	Concentration Range (ppm)	Cylinder Construction*
Benzene	14	8 - 13	S	17	60 - 400	Al, S
Ethylene	4	5 - 20	Al	4	300 - 700	Al
				6	3000 - 20,000	Al
Propylene	4	5 - 20	Al	4	300 - 700	Al
Methane/Ethane	-	-----	-	4	1000 - 6000(M), 200 - 700(E)	Al
Propane	4	5 - 20	Al	4	300 - 700	Al
Toluene	2	5 - 20	S	2	300 - 700	S
Hydrogen Sulfide	4	5 - 20	Al	2	300 - 700	Al
Meta-Xylene	2	5 - 20	S	2	300 - 700	LS
Methyl Acetate	2	5 - 20	S	2	300 - 700	S
Chloroform	2	5 - 20	S	2	300 - 700	S

SLIDE 404-9

TABLE 2. SUMMARY OF PERFORMANCE AUDIT RESULTS

Audit No.	Client	Industry	Audit material	RTI audit conc. (ppm)	Client audit % bias (Avg.)	Status of audit
1	A	Ethylene oxide production	Ethylene in N ₂ Ethylene in N ₂	3,239 21,226	-22.5 -20.0	E
2	A	Ethylene oxide production	Methane/ethane in N ₂ Methane/ethane in N ₂	1,710Me/220Et 8,130Me/597Et	+9.00/-20.0 +9.00/-1.00	E
3	A	Ethylene oxide production	Methane/ethane in N ₂ Methane/ethane in N ₂	1,021Me/315Et 6,207Me/773Et	+21.5/-4.50 +23.5/-4.50	E
4	A	Acetone production	Benzene in N ₂ Benzene in N ₂	79.0 374.0	-19.0 -11.0	E
5	A	Maleic anhydride production	Benzene in N ₂ Benzene in N ₂	138 300	-9.40 +4.70	E
6	A	Ethylene oxide production	Ethylene in N ₂ Ethylene in N ₂	5,442 18,918	-27.0 -33.0	E
7	B	Maleic anhydride production	Benzene in N ₂ Benzene in N ₂	80.0 355	+2.30 +27.5	E
8	C	Maleic anhydride production	Benzene in N ₂ Benzene in N ₂	101 387	+12.9 +14.5	E
9	D	Ethyl benzene styrene manufacturer	Benzene in N ₂ Benzene in N ₂	71.0 229	-2.80 -3.90	E

HIGHLIGHTS DURING FY-82

1. Audit repository expanded to 40 compounds (including halocarbons, hydrocarbons and sulfur containing organics).
2. Stability studies underway on all organics to demonstrate utility as audit materials.
3. Complete 20 audits (91 audits completed since 1977).

SLIDE 404-11

EPA PROCEDURE FOR PROVIDING AUDIT SAMPLES

- Request made to EMSL.
- EPA (RTI) verifies cylinder concentrations.
- High-and-low concentration cylinders shipped with standard letter to contractor by express carrier or truck.

SLIDE 404-12

(cont.)

- Contractor analyzes and reports results to project officer.
- Project officer evaluates results and sends report to EPA (RTI).
- If contractor's results are $> \pm 10\%$ of that expected, cylinders are reanalyzed by third laboratory.

SLIDE 404-13

APPENDIX C

PROCEDURE 1 — Determination of adequate chromatographic peak resolution

PROCEDURE 2 — Procedure for field auditing GC analysis

OBSERVATION AND EVALUATION OF STATIONARY SOURCE PERFORMANCE TESTS

SLIDE 405-2

DETERMINE APPLICABLE EMISSION

- Allowable pollutant and definition
- Define facility operation
- Define compliance evaluation method

SLIDE 405-3

ESTABLISH COMPLIANCE PROTOCOL

- Facility operational during testing
- Procedures for compliance evaluation method

SLIDE 405-4

OBTAIN AGREEMENT ON PROTOCOL

- Make presite survey
- Designate a contact person from agency, tester and source
- Clarify protocol procedures and get agreement

ON SITE OBSERVATION

- Observe facility operation
- Observe compliance evaluation method
- Make screening technique values when possible

SLIDE 405-6

BASELINE CONCEPT

Defn:

The documentation of all pertinent operating parameters for both process and control equipment operations to provide a narrow band of operating parameters against which determinations can be made.

SLIDE 405-7

PURPOSES OF DETERMINING REPRESENTATIVE FACILITY OPERATION

1. Evaluate performance test
2. Establish operation and maintenance programs
3. Issue permit to operate
4. Reference point for future evaluations

SLIDE 405-8

AGREEMENTS ON TESTING PROTOCOL

PROCESS:

- parameters to be monitored and recorded
- acceptable values for each parameter
- process samples to be taken and analyzed
- mode of operation
- instruments to be added and/or calibrated

(cont.)

CONTROL EQUIPMENT:

- parameters to be monitored and recorded
- acceptable values for each parameter
- control equipment effluent samples to be taken and analyzed
- mode of operation
- instruments to be added and/or calibrated

SLIDE 405-10

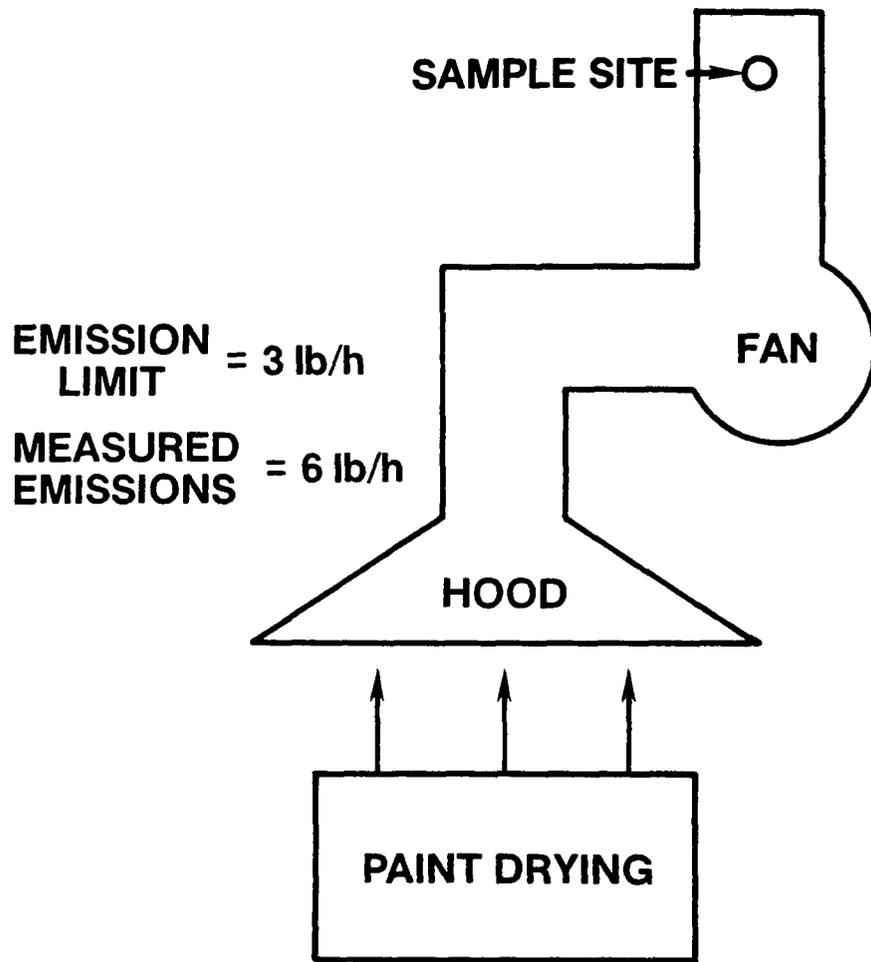
PROCESS OPERATION

- Raw Materials
- Fuel
- Process Rate
- Mode of Operation

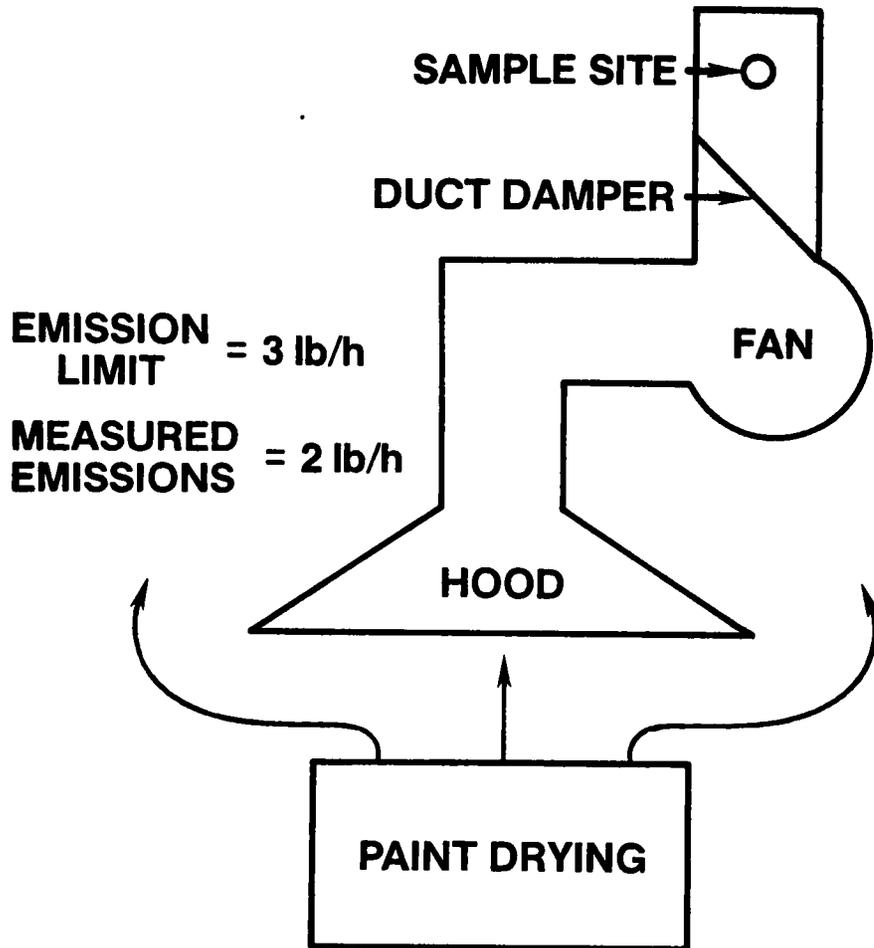
SLIDE 405-11

PROCESS MODE OF OPERATION

- Manual or automatic operation
- Cleaning and auxiliary systems
- Normal period for process cycles
- Diversion or circumvention of pollutants from air pollution control equipment
- Operation personnel



NOTES



SLIDE 405-14

PRINTING OPERATION TRICKS

- Use most efficient solvent
- Reduce line speed
- Reduce ink coverage
- Reduce number of printing lines

CONTROL EQUIPMENT MODE OF OPERATION

- Manual or automatic operation
- Collected pollutant removal cycle
- Cleaning cycle
- Auxillary or gas conditioning systems

SLIDE 405-16

OBSERVE COMPLIANCE EVALUATION TECHNIQUE

- Performance audit
- System audit

SLIDE 405-17

EXIT INTERVIEW

- Conduct exit interview with plant and test team.
- Request additional information if needed.
- Critique test program.

SLIDE 405-18

PERFORMANCE TEST REPORT FORMAT

1. Cover
2. Certification
3. Introduction
4. Summary of Results
5. Source Operations
6. Sampling and Analytical Procedures
7. Appendix

DATA REQUIREMENTS

- Completeness
- Accuracy

SLIDE 405-20

RESULTS OF FACILITY BASELINING

- Performance test can be properly evaluated.
- An operation and maintenance program can be established.
- Permit to operate can become an effective enforcement tool.
- Future inspections by agency can be more effective in determining compliance and reasons for non-compliance.

SLIDE 406-1

NOTES

COMPLIANCE EVALUATION USING MATERIAL BALANCE

SLIDE 406-2

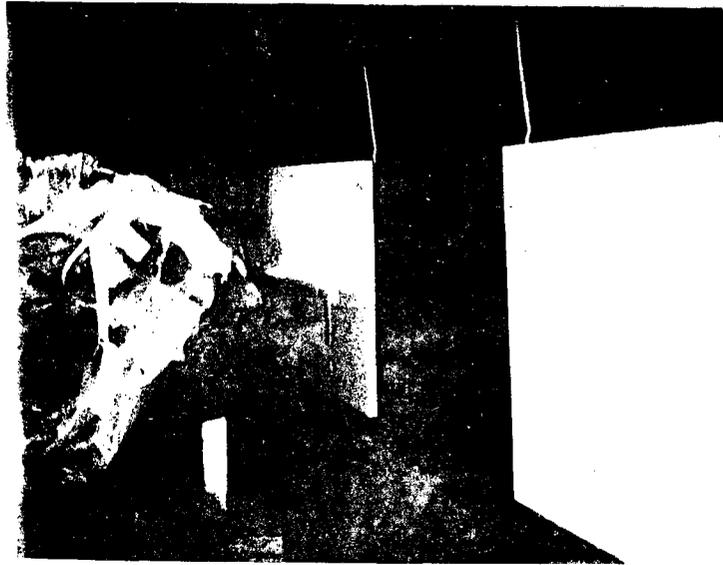
VOC SOURCE CATEGORIES THAT USE MATERIAL BALANCE FOR COMPLIANCE EVALUATION

- Surface Coating
- Graphic Arts
- Degreasing

SLIDE 406-3

SURFACE COATING

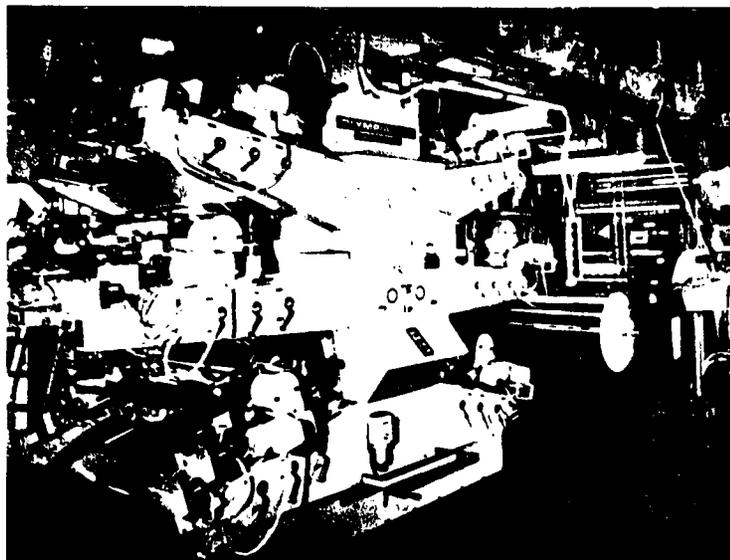
- Cans
- Metal coils
- Paper and fabric products
- Automobiles and light trucks
- Metal furniture
- Magnet wire
- Large appliances
- Flatwood paneling
- Miscellaneous metal parts



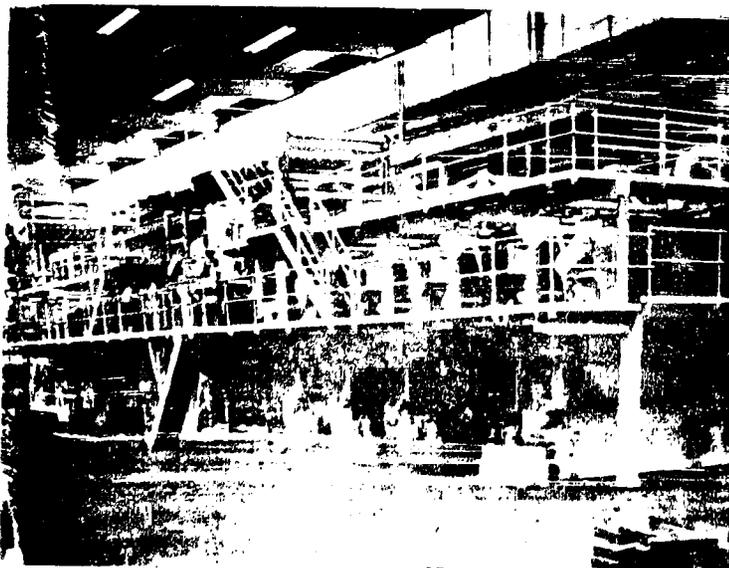
SLIDE 406-5

GRAPHIC ARTS

- Gravure printing presses
- Flexographic printing presses



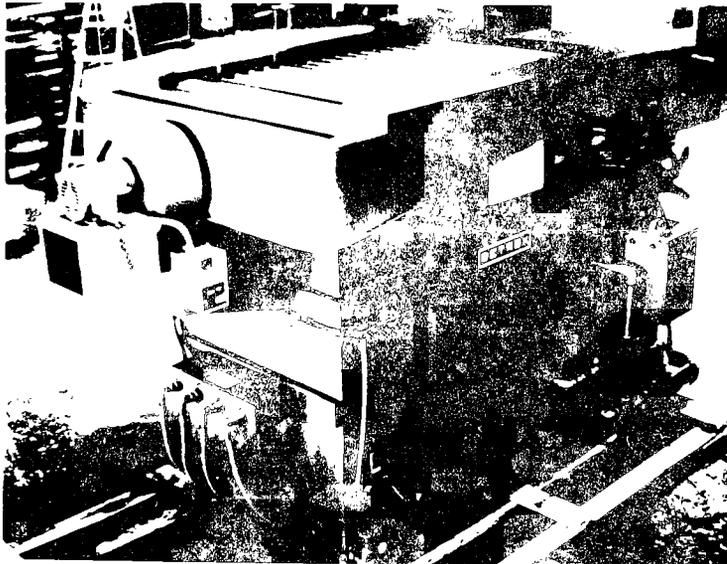
SLIDE 406-7



DEGREASING

- Cold cleaners
- Open-top vapor degreasers
- Conveyorized degreasers

SLIDE 406-9



SLIDE 406-10

SURFACE COATING

VOC INPUTS

- Pretreatment
- Coatings
- Dilution solvents
- Clean-up solvents
- Recycled solvents
 - Internal
 - External

SURFACE COATING

VOC EMISSION POINTS

- Waste solvent
- Carbon adsorber exhaust
- Carbon adsorber decanter (recycle)
- Fugitive emissions
- Flash-off
- Baking
- Product
- Coating application

SLIDE 406-12

% OF TOTAL EMISSIONS

	APPLICATION	PRE-DRY	CURING
Spray	30-50	10-30	20-40
Flow	30-50	20-40	10-30
Dip	5-20	10-30	50-70
Roller	0-5	10-20	60-80

SLIDE 406-13

SURFACE COATING

VOC EMISSION POINTS

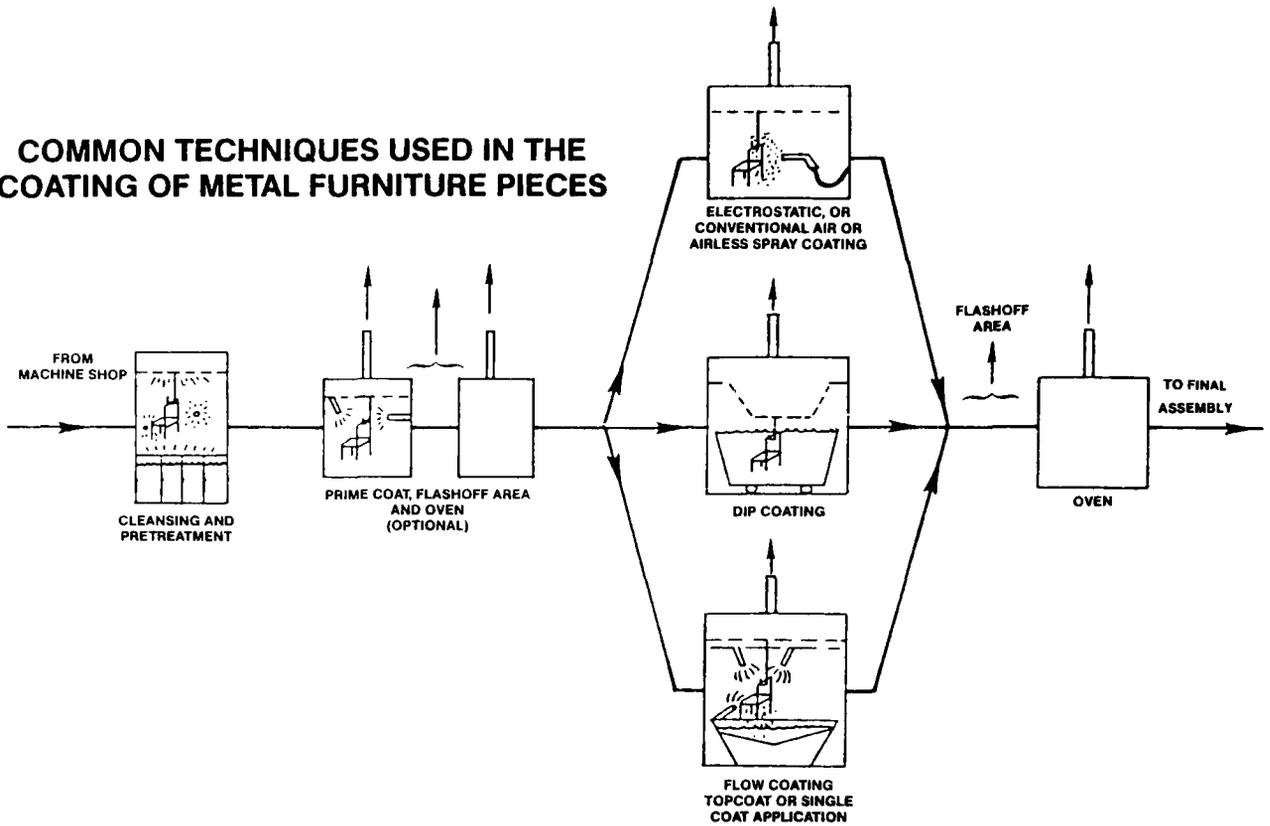
CAN BE QUANTIFIED

- Waste solvent
- Carbon adsorber emissions
- Coating application
- Carbon adsorber decanter
- Baking

CANNOT BE QUANTIFIED

- Fugitive emissions
- Product

COMMON TECHNIQUES USED IN THE COATING OF METAL FURNITURE PIECES



GRAPHIC ARTS

VOC INPUTS

- Inks
- Dilution solvents
- Cleaning solvents
- Recycled solvents
 - Internal
 - External

GRAPHIC ARTS

VOC EMISSION POINTS

- Waste solvent
- Carbon adsorber exhaust
- Carbon adsorber decanter
- Fugitive emissions
- Product

SLIDE 406-17

**GRAPHIC ARTS
VOC EMISSION POINTS**

CAN BE QUANTIFIED

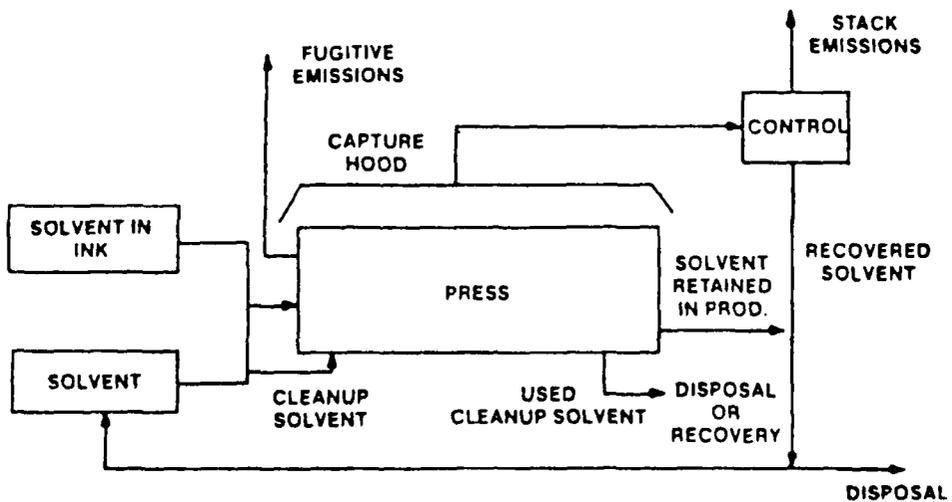
- Waste solvent
- Carbon adsorber exhaust
- Carbon adsorber decanter

CANNOT BE QUANTIFIED

- Fugitive emissions
- Product

SLIDE 406-18

**VOC INPUTS AND EMISSION POINTS
OF A PRINTING OPERATION**



DEGREASING

VOC INPUTS

- Cleaning solvents
- Recycled solvents
 - Internal
 - External

DEGREASING

VOC EMISSION POINTS

- Waste solvent
- Carbon adsorber exhaust
- Carbon adsorber decanter
- Bath evaporation
- Carry-out
- Lip exhaust

DEGREASING VOC EMISSION POINTS

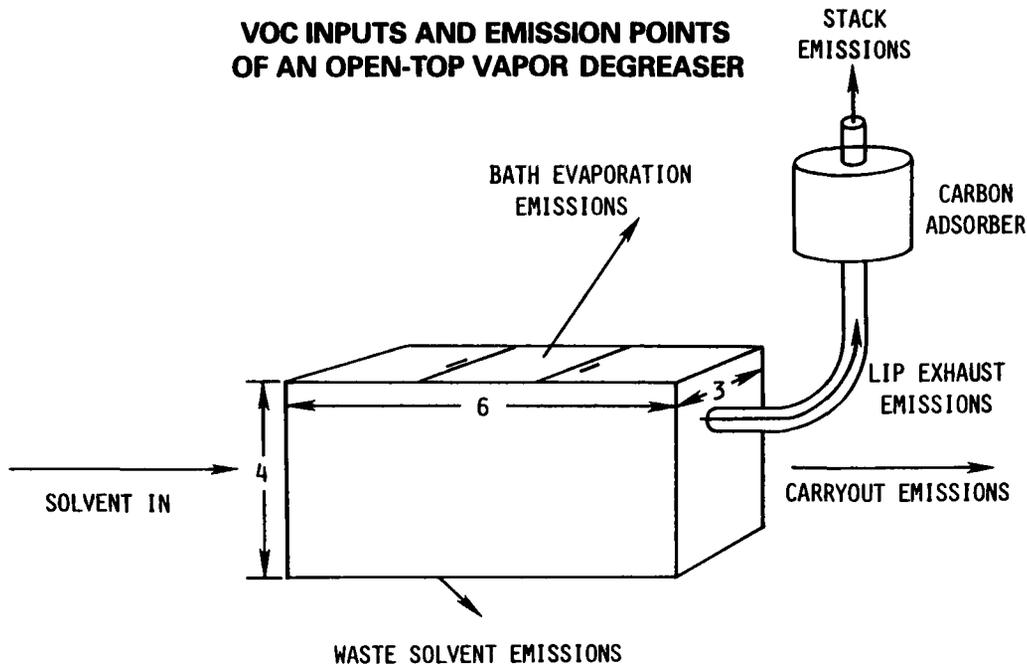
CAN BE QUANTIFIED

- Waste solvent
- Carbon adsorber exhaust
- Carbon adsorber decanter
- Lip exhaust

CANNOT BE QUANTIFIED

- Bath evaporation
- Carry-out

**VOC INPUTS AND EMISSION POINTS
OF AN OPEN-TOP VAPOR DEGREASER**



**INFORMATION NECESSARY FOR
MATERIAL BALANCE**

SURFACE COATING

- Paint inventory at beginning of test period
- Paint added during test period
- Paint inventory at end of test period
- Dilution or clean-up solvent level at beginning of test period
- Quantity of dilution or clean-up solvent added during test period
- Dilution or clean-up solvent level at end of test period
- Hours of operation during test period

SLIDE 406-25

GRAPHIC ARTS

- Ink inventory at beginning of test period
- Ink added during test period
- Ink inventory at end of test period
- Clean-up solvent at beginning of test period
- Clean-up solvent added during test period
- Clean-up solvent at end of test period
- Dilution solvent at beginning of test period
- Dilution solvent added during test period
- Dilution solvent at end of test period
- Hours of operation during test period

SLIDE 406-26

DEGREASING

- Degreaser solvent level at beginning of test period.
- Quantity of solvent added during test period.
- Solvent level at end of test period.
- Hours of operation during test period.

ERRORS IN MATERIAL BALANCE

SURFACE COATING

- VOC in coating
- Fugitive emissions

GRAPHIC ARTS

- VOC in paper
- Fugitive emissions

DEGREASING

- Volume of metal chips and other material remaining in sump
- Volume of oil and grease dissolved in solvent.
- Fugitive emissions.

SLIDE 406-28

MATERIAL BALANCE

ADVANTAGES

- Checks total system
- Simple and inexpensive
- Records of solvent use are usually available.

DISADVANTAGES

- Time-consuming

SLIDE 406-29

EQUIPMENT SPECIFICATIONS

Equipment specifications are VOC regulations which involve work practices or equipment design for compliance.

EQUIPMENT SPECIFICATIONS ARE USED IN MANY INDUSTRIES

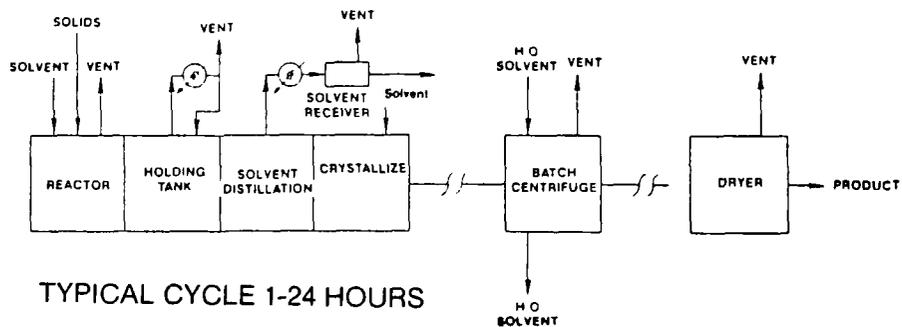
- Pharmaceutical
- Storage tanks
- Vinyl chloride
- Benzene
- Degreasing
- Drycleaning

SLIDE 406-31

PHARMACEUTICAL INDUSTRY

SLIDE 406-32

TYPICAL CHEMICAL SYNTHESIS PHARMACEUTICAL PROCESS



POTENTIAL EMISSION SOURCES

- Dryers
- Reactors
- Distillation units
- Storage and transfer

SLIDE 406-34

**SURFACE CONDENSERS OR EQUIVALENT
CONTROLS ON REACTORS, DISTILLATION
OPERATIONS, CRYSTALLIZERS, CENTRIFUGES,
AND VACUUM DRYERS**

SLIDE 406-35

SURFACE CONDENSER REQUIREMENT

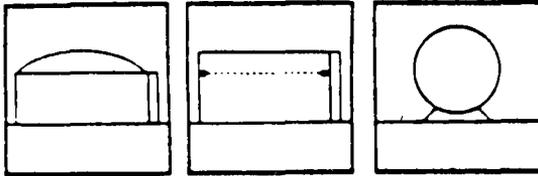
CONDENSER OUTLET GAS TEMPERATURE	VOC VAPOR PRESSURE
77°F	0.5 TO 1.0 PSI
50°F	1.0 TO 2.5 PSI
32°F	1.5 TO 2.9 PSI
5°F	2.9 TO 5.8 PSI
-13°F	> 5.8 PSI

SLIDE 406-36

STORAGE TANK REQUIREMENTS

- Vapor balance or equivalent ($\geq 90\%$ efficiency) on truck or railcar transfers to storage tanks ≥ 2000 gallons that store VOC with vapor pressure > 4.1 PSI.
- Conservation vents set at ± 0.03 PSI on tanks that store VOC with vapor pressure ≥ 1.5 PSI.
- Cover all in-process tanks containing VOC.

STORAGE TANKS



Fixed Roof Floating Roof Pressure

SLIDE 406-38

RACT

All Tanks $\geq 40,000$ gal @ v.p. ≥ 1.5 psia

SLIDE 406-39

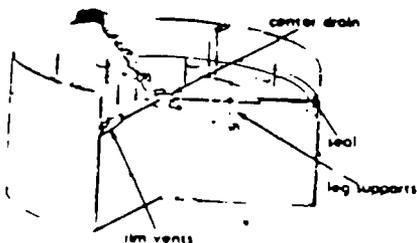
RACT

(for External Floating Roofs)

- secondary rim-mounted seal covering the primary seal

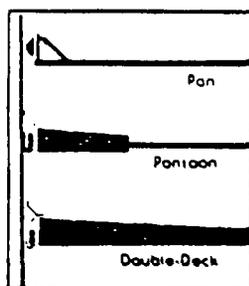
SLIDE 406-40

EXTERNAL FLOATING ROOF TANK



SLIDE 406-41

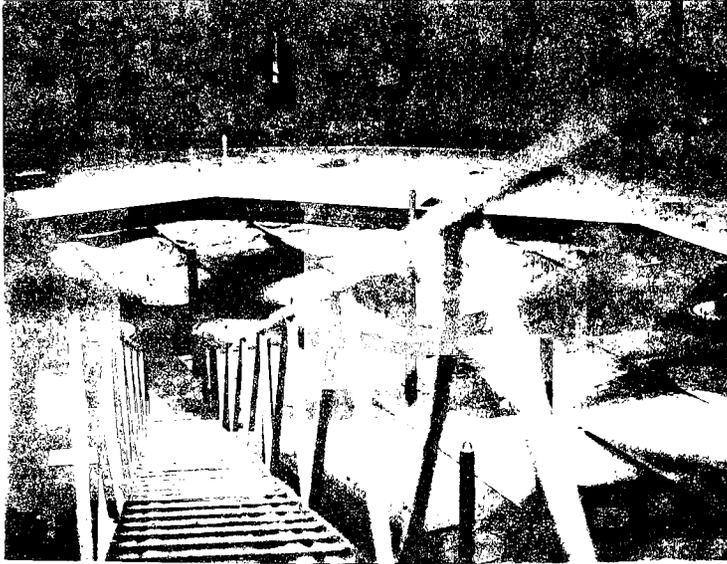
NOTES



TYPES OF
EXTERNAL
FLOATING
ROOFS

SLIDE 406-42





J

SLIDE 406-44



**NATIONAL EMISSION STANDARDS
FOR HAZARDOUS AIR POLLUTANTS
(NESHAPS)**

- Vinyl Chloride
- Benzene

SLIDE 406-46

FUGITIVE EMISSION SOURCES

- Loading and Unloading lines
- Slip Gauges
- Pump Seals
- Compressor Seals
- Agitator Seals
- Relief Valves
- Manual Vents
- Opening of Equipment
- Samples
- Inprocess wastewater

SLIDE 406-47

SLIP GAUGES

- Vent any discharged VC through control device.

ROTATING PUMPS

- Sealless pumps
- Double Mechanical Seals
 1. Maintain pressure so that leak occurs into pump.
 2. Vent emissions through control device.

SLIDE 406-49

RECIPROCATING PUMPS

- Double Outboard Seals

ROTATING COMPRESSORS

- Double Mechanical Seals

RECIPROCATING COMPRESSORS

- Double Outboard Seals

AGITATORS

- Double Mechanical Seals

SLIDE 406-50

RELIEF VALVES

- Install rupture disk between equipment and relief valve.
- Connect relief valve discharge to process line or recovery device.

MANUAL VENTS

- Vent through control system.

SLIDE 406-52

**BENZENE FUGITIVE EMISSIONS
APPLICABILITY**

- Petroleum Refineries
- Organic Chemical Manufacturing Plants

SLIDE 406-53

**COMPONENTS IN BENZENE SERVICE
(Fluid That Is $\geq 10\%$ By Weight Benzene)**

- Pumps
- Compressors
- Pipeline Valves
- Safety/Relief Valves
- Sampling Systems
- Other Sources

SLIDE 406-54

NEW PUMPS AND COMPRESSORS

- A. Dual mechanical seal system with barrier fluid between seals for new pumps and seal system with barrier fluid that prevents leaks to atmosphere for new compressors
(Benzene conc. $< 10\%$ by volume)
OR
- B. Closed vent system to control system
- Sensor on each seal system
 1. Check Daily
OR
 2. Audible Alarm

(cont.) NEW PUMPS AND COMPRESSORS

- Visually Inspect each pump weekly for liquid leak
- If leak is detected, repair within 15 days (first attempt at repair within 5 days)
- For each dual mechanical seal system:
 1. Operate with barrier fluid pressure $>$ stuffing box pressure.
or
 2. Equip with a barrier fluid degassing reservoir connected to control system.
or
 3. Designed and operated with no benzene emission when barrier fluid purged.

SLIDE 406-56

**SAFETY/RELIEF VALVES IN
GAS/VAPOR SERVICE**

- Maintain at $<$ 200 ppm above background.
- After pressure release, return to $<$ 200 ppm within 5 days.

SLIDE 406-57

**PIPELINE VALVES, OPEN-ENDED VALVES,
AND EXISTING PUMPS AND COMPRESSORS**

- Monitor monthly with portable hydrocarbon detector.
- If leak $>$ 10,000 ppm, repair within 15 days — first attempt at repair within 5 days includes.
 1. tightening of bonnet bolts
 2. replacement of bonnet bolts
 3. tightening of packing gland nuts
 4. injection of lubrication into lubricated packing

(cont.) PIPELINE VALVES, ETC.

- Quarterly monitoring if leak not detected for two successive months.
- Equip open-ended valve with cap, blind, plug, or closed second valve.
- Visually inspect each pump weekly for liquid leaks.

SLIDE 406-59

DEGREASING

SLIDE 406-60

CONTROL OF SOLVENT BATH EMISSIONS

- Covers
- Freeboard height
- Carbon adsorption
- Safety switches
- Refrigerated chillers

SLIDE 406-61

CONTROL OF CARRY-OUT EMISSIONS

- Drainage racks
- Drying tunnels
- Rotating baskets

CONTROL OF BOTH SOLVENT BATH AND CARRY-OUT EMISSIONS

- Automated cover-conveyor
- Refrigeration condensation

SLIDE 406-63

RACT GUIDELINES

- Separate guidelines for each type of degreaser
- Each guideline divided into two levels of control

SLIDE 406-64

CONTROL SYSTEMS

CONTROL SYSTEM A

1. Control equipment
2. Operating requirements

CONTROL SYSTEM B

(Control System A plus)

1. Additional control equipment
2. Additional operating requirements

SLIDE 406-65

OPERATING REQUIREMENTS

- Covers
- Conveyor speed
- Freeboard ratio
- Conveyorized degreaser
- Refrigeration system
- Ventilation rate
- Safety switches

DRYCLEANING

SLIDE 406-67

EMISSION SOURCES

- Washer/Extractor
- Dryer/Reclaimer
- Carbon Adsorber Exhaust
- Filter Muck
- Still Residue
- Filter Cartridges
- Leaks (Liquid & Vapor)

SLIDE 406-68

RACT FOR PERC DRYCLEANERS

- Vent dryer exhaust through CA or equivalent
- Exhaust from control device ≤ 100 ppm perc
- Immediately repair liquid leaks
- Filter residue $\leq 25\%$ perc
- Still residue $\leq 60\%$ perc
- Drain filter cartridges ≥ 24 hours or dry before disposal

SLIDE 406-69

INSPECTION ITEMS FOR DRYCLEANING FACILITIES WITH PERC REMOVAL EQUIPMENT

1. Inspect following for vapor leaks
 - A. Ductwork
 - B. Improper gasket seating
 - C. Other
2. Inspect following for liquid perc leaks
 - A. Hose connections, unions, couplings, and valves
 - B. Machine door gaskets and seatings

(cont.)

- C. Filter head gasket and seating
 - D. Pumps
 - E. Base tanks and storage containers
 - F. Water separators
 - G. Filter sludge recovery
 - H. Distillation unit
 - I. Diverter valves
 - J. Saturated lint from lint basket
 - K. Cartridge filters
3. Observe location of control system vent

(cont.)

4. Observe the following equipment to see where vented
- A. Perc removal system
 - B. Still
 - C. Muck cooker
 - D. Separators
 - E. Dry cleaning machine(s)
 - F. Other sources vented through perc removal system

(cont.)

- 5. Inspect perc removal system for lint buildup and corrosion problems
- 6. Inspect floor pickup points for proper operation and for lint accumulation
- 7. Obtain samples of muck cooker and still bottoms if possible
- 8. Observe general housekeeping

INTEGRATED BAG SAMPLING AND AND ANALYSIS

SLIDE 501-2

PRINCIPLE

A gas sample is collected in an evacuated bag by evacuating the rigid air-tight container holding the bag.

SLIDE 501-3

APPLICABILITY

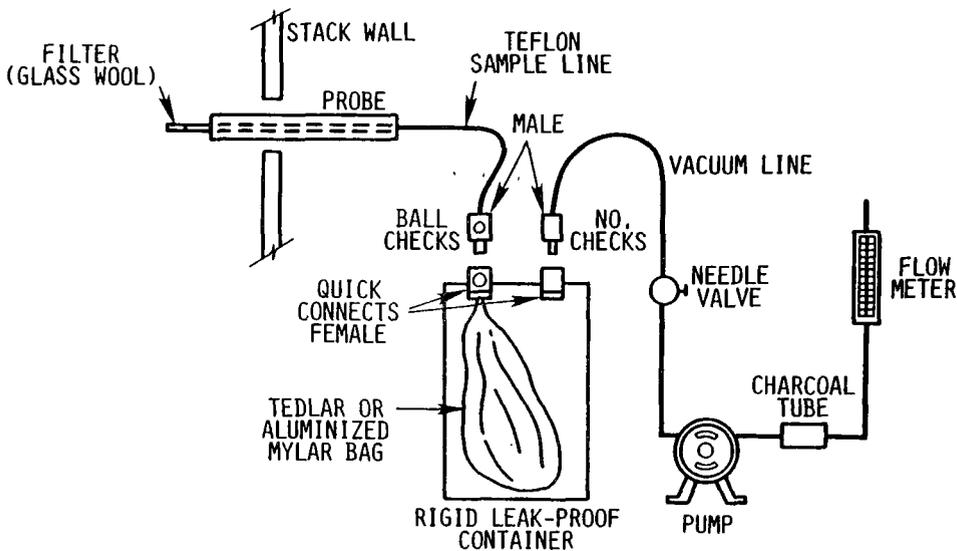
METHOD IS APPLICABLE:

- In situations where a hydrogen flame is a hazard.
- To measure halogenated organics.

METHOD IS NOT APPLICABLE:

- For sampling polar compounds. *such as alcohols*
- For use at sources where organics are contained in particulate matter.
- High moisture.

INTEGRATED BAG SAMPLING TRAIN



proportional sampling is needed for sources with variable flow and concentration. Calibrations are not as important since the sampling is not isokinetic

SLIDE 501-5

LEAK CHECK PROCEDURE

Connect a water manometer and pressurize bag to 5 to 10 cm H₂O; allow to stand for 10 min.

ALTERNATIVE LEAK CHECK PROCEDURE

- Pressurize bag to 5 to 10 cm H₂O; allow to stand over night.
- Place a rotameter in line between the bag and pump inlet and evacuate the bag.

SLIDE 501-6

NOTES



SLIDE 501-7



SAMPLING PROCEDURE

- Assemble and leak check sample train.
- Connect vacuum line to Teflon sample line from the probe and purge.
- Connect vacuum line to bag and evacuate.
- Position sample and vacuum lines for sampling.
- Collect sample keeping rate proportional to stack velocity.

Blank check the bag prior to sampling

SLIDE 501-9

(cont.)

SAMPLING PROCEDURE

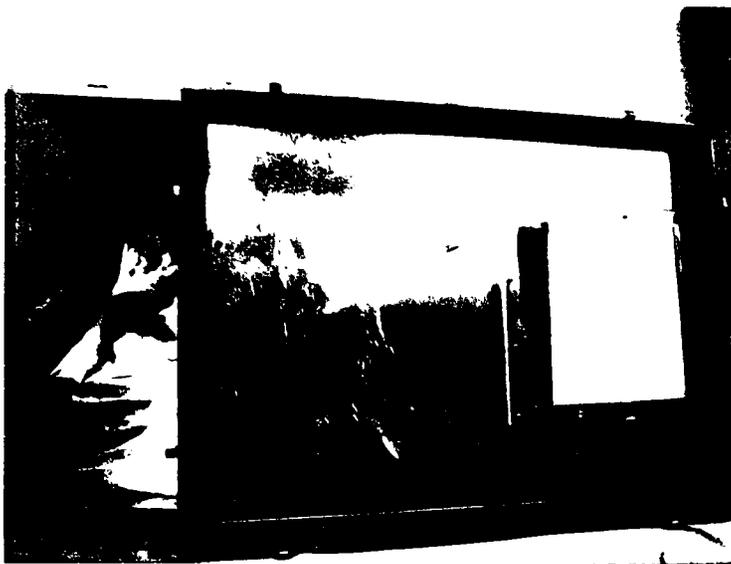
- At the end of sample period, shut off pump, and disconnect sample and vacuum lines.
- Record source temperature, barometric pressure, ambient temperature, sampling flow rate and initial and final sampling times.
- Protect bag and container from sunlight.
- Perform analysis within 2 hours of sample collection.

SLIDE 501-10



SLIDE 501-11

NOTES



SLIDE 501-12



ALTERNATIVE SAMPLING PROCEDURES (Direct Pump Sampling)

- Place pump and needle valve between the probe and the bag.
- Leak check the system and purge with stack gas before connecting to evacuated bag.

SLIDE 501-14

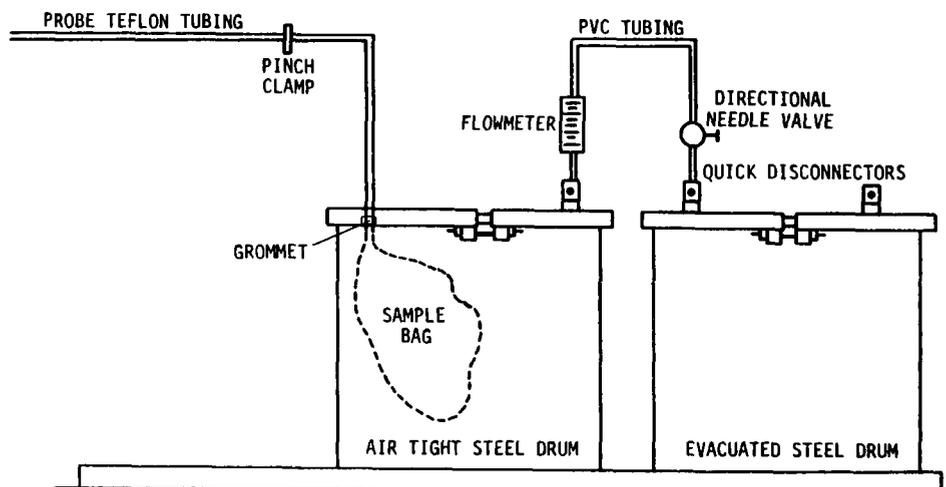
(cont.)

ALTERNATE SAMPLING PROCEDURES (Explosion Risk Area Sampling)

- Sample bag is enclosed in an airtight steel drum.
- Sample pump is replaced with an evacuated steel drum.

SLIDE 501-15

EXPLOSION RISK GAS SAMPLING METHOD



(cont.)

ALTERNATE SAMPLING PROCEDURE

(Modified Procedure When Condensation Is Present)

PROCEDURE I

- Heat box and bag to the source temperature.
- Maintain temperature during transportation and analysis.

PROCEDURE II

- Pre-fill sampling bag with a known quantity of inert gas.
- Meter source gas into partially filled bag through heated sampling lines, flow meter, and pump.

SLIDE 501-17

ANALYTICAL CHECK OF BAG SAMPLE

STABILITY:

- Reanalyze bag sample over extended time period.

should not change over 10%

CONDENSATION OR ABSORPTION:

- Empty bag, refill and analyze.

should be less than 5% of initial reading

ADSORPTION SAMPLING TECHNIQUES

SLIDE 502-2

PRINCIPLE

A gas sample is extracted from the stack and volatile organic vapors are collected on suitable adsorption media.

APPLICABILITY

This method applies to the determination of selected organic compounds.

SLIDE 502-3

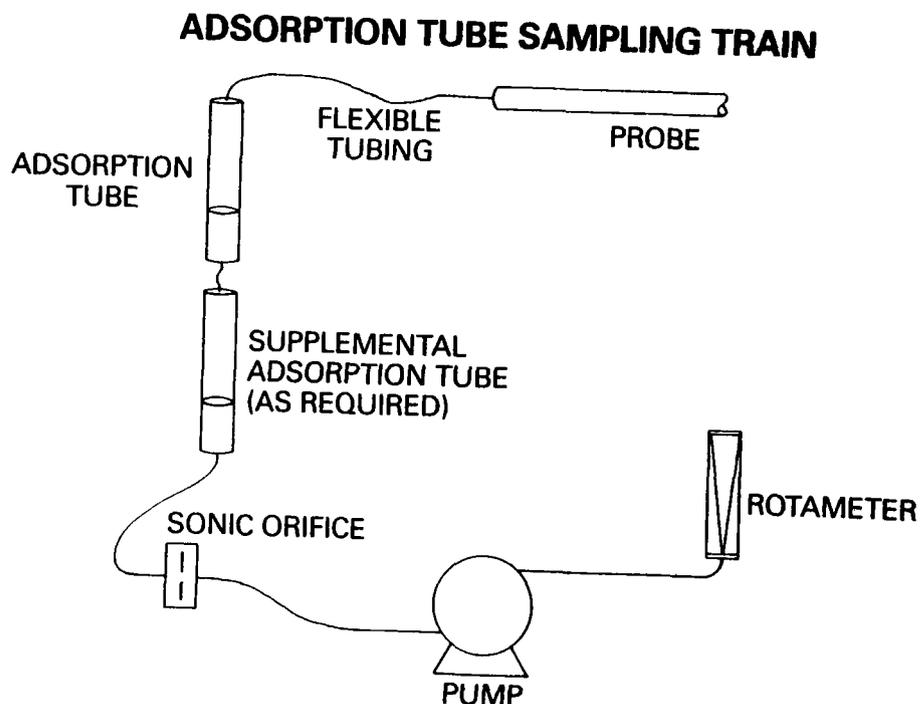
ADVANTAGES

- Simple method and available equipment.
- Collection efficiency is checked.
- Small sample size.
- Increased storage time with freezer.

DISADVANTAGES

- Not real time data.
- Gas temperature must be below 125°F for good adsorption.
- H₂O can diminish collection efficiency.
- Some gases are not collected and/or may be replaced by other gases.
- Backup tube doubles analysis.
- Sample may be lost in the probe.

SLIDE 502-5



Use short length tubing prior to adsorption tube

SAMPLING PROCEDURE

1. Immediately before sampling, break the ends of tube to provide an opening at least one-half internal diameter of tube.
2. The small section of charcoal is used as a back-up and should be positioned nearest the sampling pump.
3. The charcoal tube should be vertical during sampling to reduce channeling through the charcoal.

SLIDE 502-7

(cont.)

SAMPLING PROCEDURE

4. The flow, time, and/or volume must be measured as accurately as possible. The sample should be taken at a flow rate of 1 lpm or less.
5. The temperature and pressure of the atmosphere being sampled should be measured and recorded.
6. The charcoal tube should be capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

SLIDE 502-8

(cont.)

SAMPLING PROCEDURE

7. One tube should be handled in the same manner as the sample tube (break seal and transport), except that no air is sampled through this tube. This tube should be labeled as a blank.
8. Capped tubes should be packed tightly before shipment to minimize tube breakage.
9. Samples of the suspected solvent(s) should be submitted to the laboratory for qualitative characterization.

SAMPLING PARAMETERS ASSOCIATED WITH VARIOUS ORGANIC COMPOUNDS

Organic Solvent	Detection limit (mg/sample)	Sample Volume (liters)		Molecular Weight
		Minimum	Maximum	
Acetone	—	0.5	7.7	58.1
Benzene	0.01	0.5	55	78.1
Carbon tetrachloride	0.20	10	60	154.0
Trichloroethylene	0.05	1	17	131
Toluene	0.01	0.5	22	92.1
Xylene	0.02	0.5	31	106

Don't use NIOSH tubes due to small sites, use EPA approved sample tubes.

SLIDE 502-10

ROUTINE SAMPLE HANDLING

1. Tightly seal and pack separately from any process material.
2. Cushion against breakage.
3. Protect from elevated temperature and low pressure.
4. Store in refrigerator or freezer in the laboratory.

SLIDE 502-11

SPECIAL HANDLING

1. Some sorbents require shipment in cooled (4°C) containers.
2. Samples for thermal desorption require handling with clean, white, nylon gloves at all times, and individually sealed containers.

Refer to NIOSH for handling information

COMMON SORBENTS

- Charcoal
- Tenax®
- Silica Gel
- XAD-2®
- Florisil®

Thermal desorption - can not do additional analysis

SLIDE 502-13

PRELIMINARY DETERMINATIONS

1. Adsorption Efficiency
2. Desorption Method
3. Desorption Efficiency
4. Capacity
5. Breakthrough Volume

EPA is steering away from absorption tubes

SLIDE 502-14

ADSORPTION EFFICIENCY

1. Sorbent Dependent
2. Analyte Dependent
3. Sampling Rate Dependent
4. Sampling Conditions (moisture, temperature, etc.)

Big Killer

SLIDE 502-15

DESORPTION METHOD

1. Solvent Extraction
 - displacement
 - elution
2. Thermal

DESORPTION EFFICIENCY ← *MUST KNOW*

1. Measured in Percent *at least 50% at applicable concentrations*
2. Sorbent Dependent *Best to use stds for check of efficiency*
3. Analyte Dependent
4. Solvent Dependent
5. Moisture, etc., may also have an effect.

SLIDE 502-17

DESORPTION EFFICIENCIES FROM CHARCOAL WITH CARBON DISULFIDE

ANALYTE	MEDIAN (%)	SEMI-QUARTILE
		RANGE (%)
Benzene	98	95-100
Carbon Tetrachloride	99	97-101
Chloroform	99	97-100
1,2-Dichloroethane	99	97-100
p-Dioxane	94	91-98
Methyl Chloroform	100	98-100
Methylene Chloride	99	96-100
Toluene	98	96-100
Trichloroethylene	99	97-101
o-Xylene	97	94-99

SLIDE 502-18

CAPACITY

1. Measure in mass of analyte/unit weight of sorbent.
2. Sorbent and analyte dependent.
3. Dependent on amount of other adsorbed components.

BREAKTHROUGH VOLUME

1. Measured in same units as sample volume.
2. Dependent on analyte, sorbent, temperature.
3. Highly variable with analyte.

SLIDE 502-20

**TENAX GC BREAKTHROUGH VOLUMES
(LITERS) FOR TARGET COMPOUNDS***

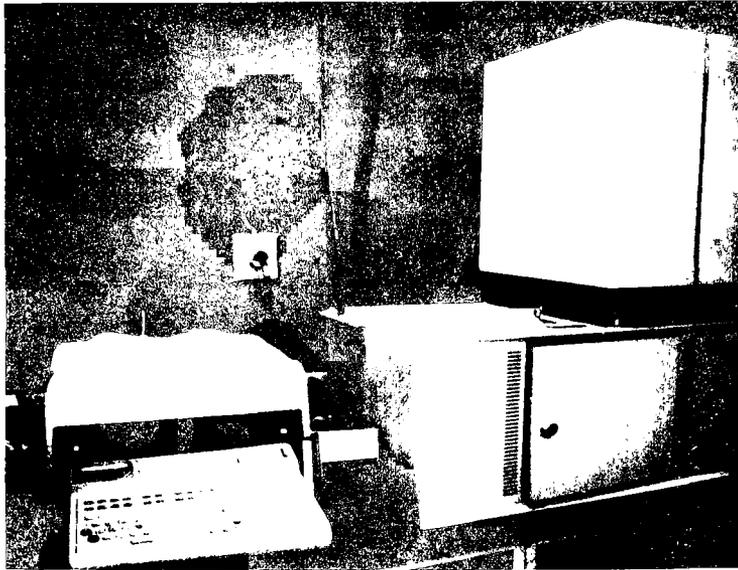
COMPOUND	b.p.	TEMPERATURE (°F)					
	(°C)	50	60	70	80	90	100
Chloroform	61	56	41	32	24	17	13
Carbon Tetrachloride	77	45	36	28	21	17	13
1,2-Dichloroethane	83	71	55	41	31	24	19
Methyl Chloroform	75	31	24	20	16	12	9
Tetrachloroethylene	121	481	356	261	192	141	104
Trichloroethylene	87	120	89	67	51	37	28
Chlorobenzene	132	1989	871	631	459	332	241

*For a Tenax GC bed of 1.5 × 8.0 cm.

SLIDE 502-21

ANALYSIS

1. Any gas chromatograph detector depends on analyte and previous knowledge of constituents.
2. Must be calibrated for each compound to be identified and/or quantified.
3. Field blanks and reagent blanks must be analyzed.



CALIBRATION

1. Liquid standards are used for calibration curve for solvent extraction samples.
2. Gas standards are loaded on tubes for thermally desorbed sample calibration.

ADVANTAGES OF EXTRACTION METHOD

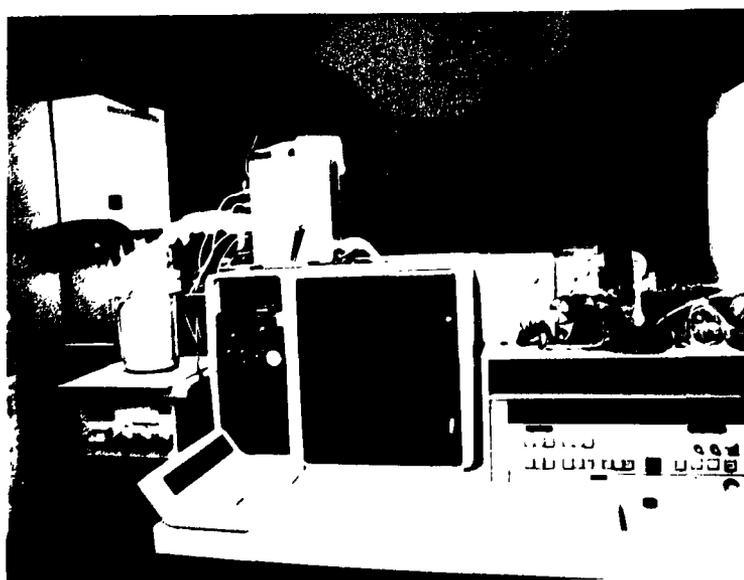
1. Selection of sorbents for different application (XAD-2, Florisil, charcoal, Chromosorb 102, etc.)
2. Relatively simple equipment and handling readily available.
3. Multiple injections are possible from a single sample. Sample dilutions and, sometimes, concentration are possible.

SLIDE 502-25

ADVANTAGES OF THERMAL METHOD

1. Extremely sensitive (nanograms/sample rather than micrograms/sample).
2. No solvent interferences.

SLIDE 502-26

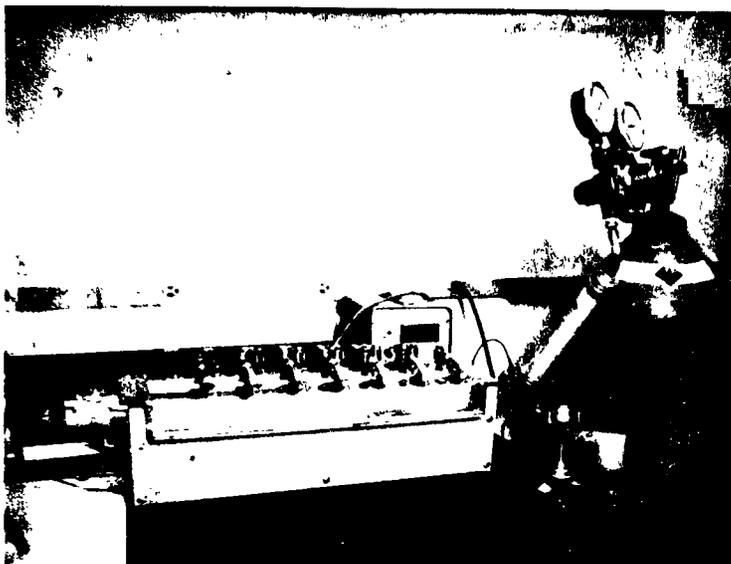


DISADVANTAGES OF EXTRACTION METHOD

1. Requires relatively high concentrations of large sample volumes.
2. Large solvent peak can mask some components completely or interfere with quantification.
3. Blank and/or sorbent preparation (sorbent dependent).

DISADVANTAGES OF THERMAL METHOD

1. Limited validated sorbents (mainly Tenax).
2. Special analytical equipment is required (limited availability).
3. Single shot analysis.
4. Special sorbent preparation and handling procedures required.
5. High blank levels (especially benzene and toluene).
6. Storage problems.



THERMO DESORBER

SLIDE 503-1

NOTES

DIRECT INTERFACE SAMPLING AND ANALYSIS

SLIDE 503-2

PRINCIPLE

A gas sample is extracted from the source using a heated probe and sample line.

The sample is submitted directly to the gas chromatograph for analysis through a heated gas sampling valve.

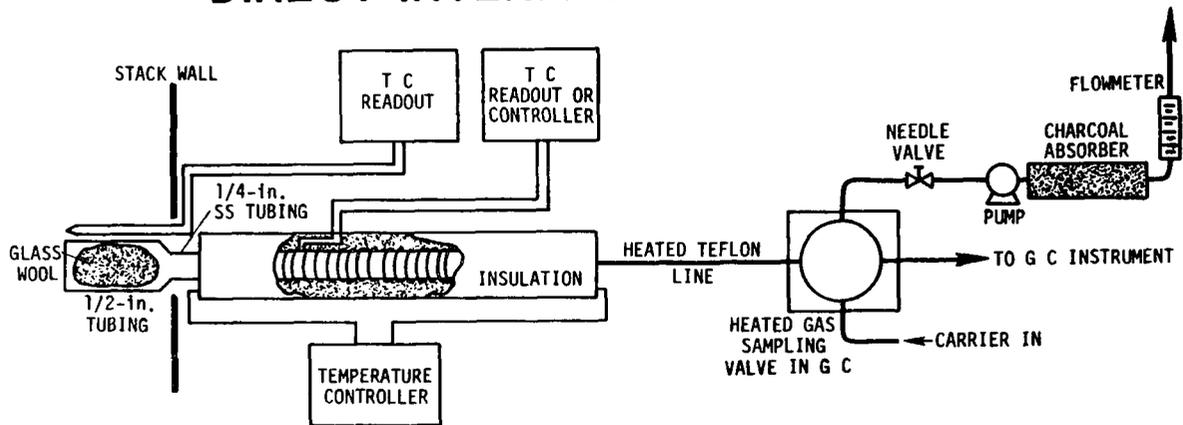
SLIDE 503-3

APPLICABILITY

METHOD IS APPLICABLE AT SOURCES WHERE:

- Moisture content of gas will not interfere with analytical method.
- Physical requirements of equipment can be met at the site.
- Source gas concentration is low enough that detector saturation is not a problem.

DIRECT INTERFACE SAMPLING SYSTEM



SLIDE 503-5

SAMPLING PROCEDURE

- Heat probe and heated sample line to a temperature of 0-3° C above source temperature.
- Inject calibration gas mixture at the gas sampling valve.
- Flush probe, sample line, and sample loop with source gas.
- Analyze sample using the same conditions used for the calibration gas mixture.

SLIDE 503-6

(cont.)

SAMPLING PROCEDURE

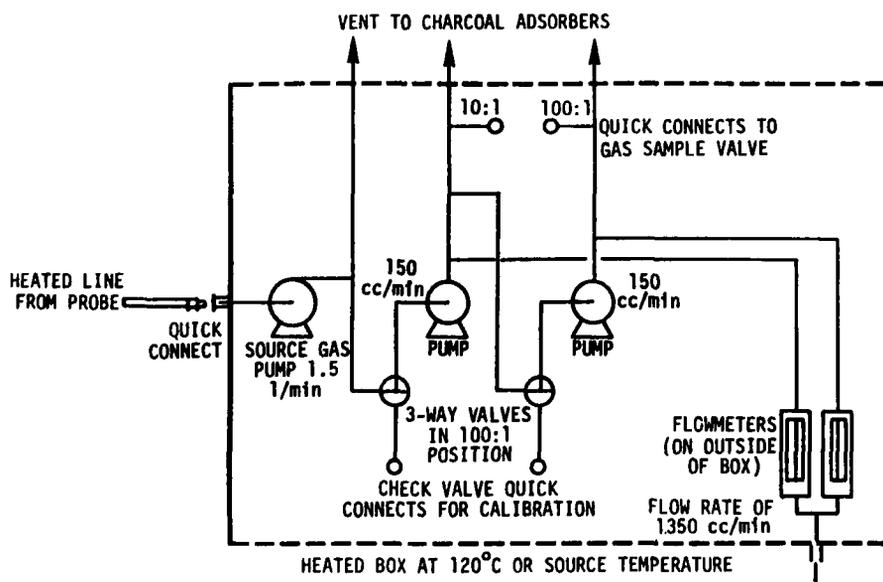
- Analyze two additional samples.
- Measure peak area of three samples. If they do not agree to within 5 percent, analyze additional samples.
- At end of sampling, analyze a second calibration gas mixture.
- Analyze audit samples.
- Record all required data on the data sheet.

DILUTION INTERFACE SAMPLING PROCEDURE

- Heat system to 0-3° C above source temperature or a temperature high enough to prevent condensation of water and/or organic compounds.
- Verify operation of dilution system.
- Analyze source gas samples using the same dilution settings as used for the standards.
- Analyze three separate samples.
- Repeat analysis of calibration gas mixtures.
- Analyze field audit samples.

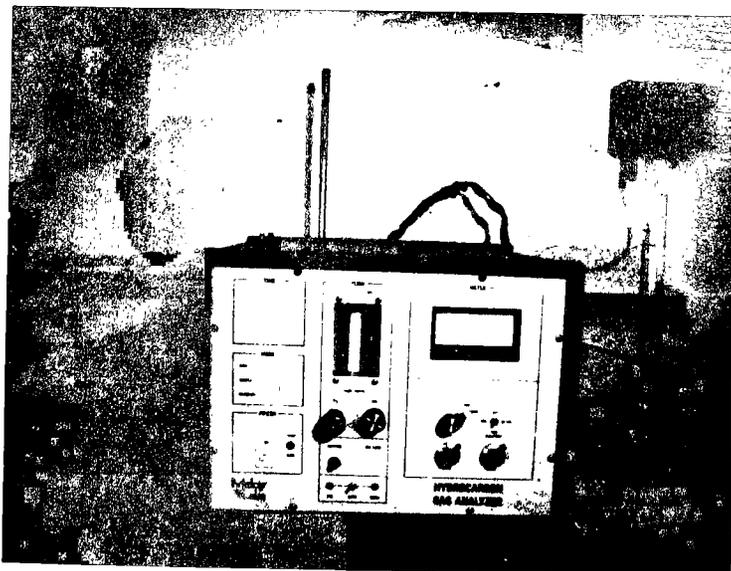
SLIDE 503-8

DIAGRAM OF THE HEATED BOX REQUIRED FOR DILUTION OF SAMPLE GAS





SLIDE 503-10



~~10/1~~

7/27/87 11:05

SLIDE 504-1

NOTES

VOC ANALYSIS BY GAS CHROMATOGRAPHY

SLIDE 504-2

Defn. CHROMATOGRAPHY

Chromatography is a process of separating mixtures by differential migration.

SLIDE 504-3

GENERAL REQUIREMENTS

1. Stationary Phase
2. Mobile Phase
3. Defined Migration Path

SLIDE 504-4

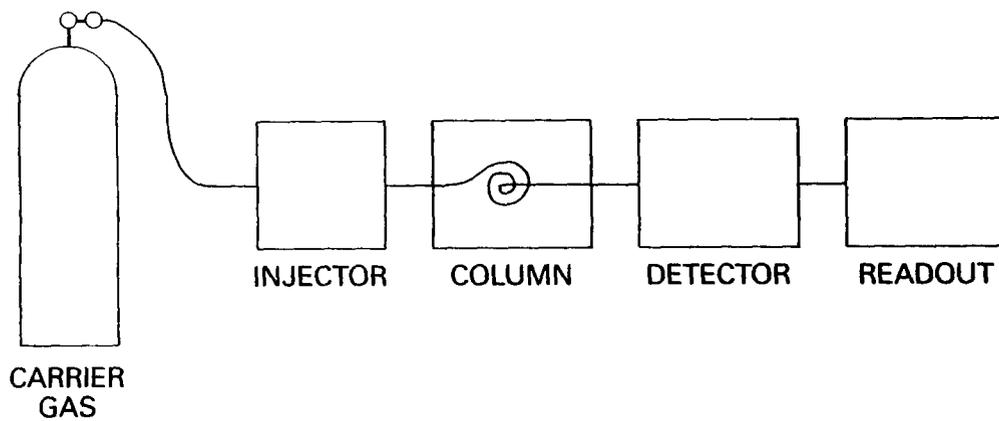
SEPARATION PRINCIPLE

As a mixture travels along the defined path, a component more compatible in or on the stationary phase is slowed in comparison with a component more compatible in the mobile phase.

GAS CHROMATOGRAPHY

In gas chromatography, the mobile phase is a gas; the stationary phase is a liquid or a solid, and the mixture is either gaseous or a liquid which can be vaporized.

COMPONENTS OF A GAS CHROMATOGRAPH



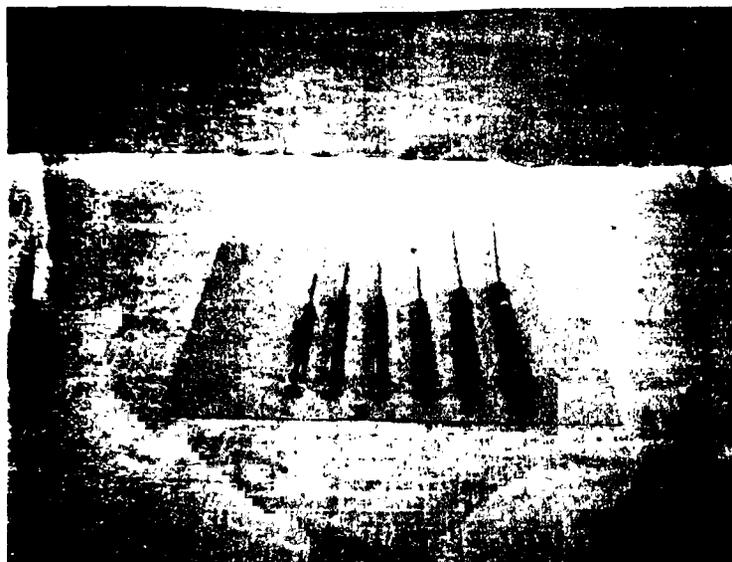
COMMON CARRIER GASES

1. Helium
2. Nitrogen
3. Argon
4. Argon/5% Methane

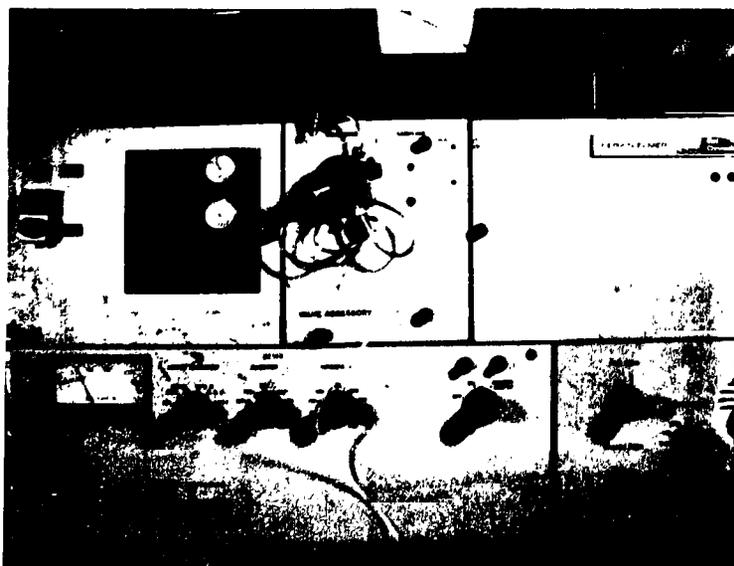
INJECTOR SYSTEMS

- Syringe
 1. For liquid
 2. For gases
- Sample Loops

SLIDE 504-9



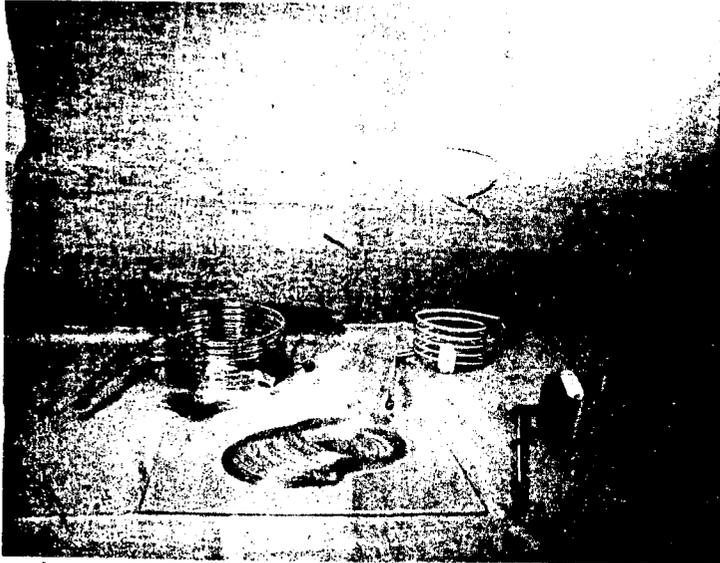
SLIDE 504 10



TYPES OF COLUMNS

- Packed
 1. glass
 2. metal
- Capillary
 1. glass
 2. Fused silica

SLIDE 504-12



SLIDE 504-13





SLIDE 504-15

COLUMN OVEN OPERATION

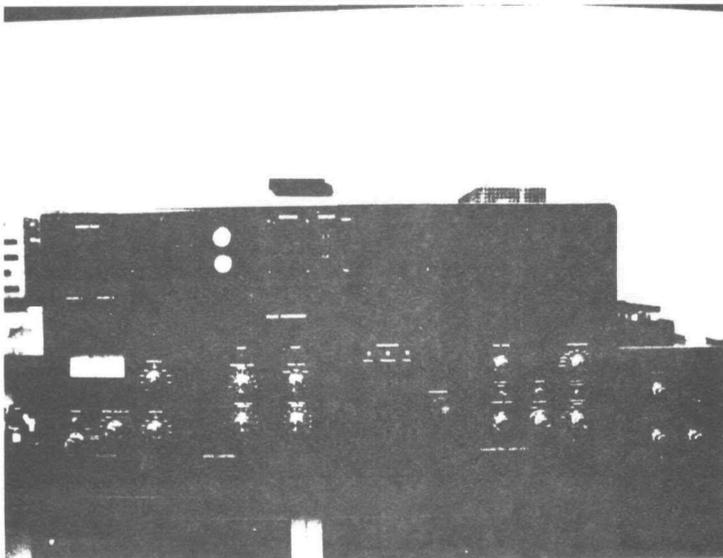
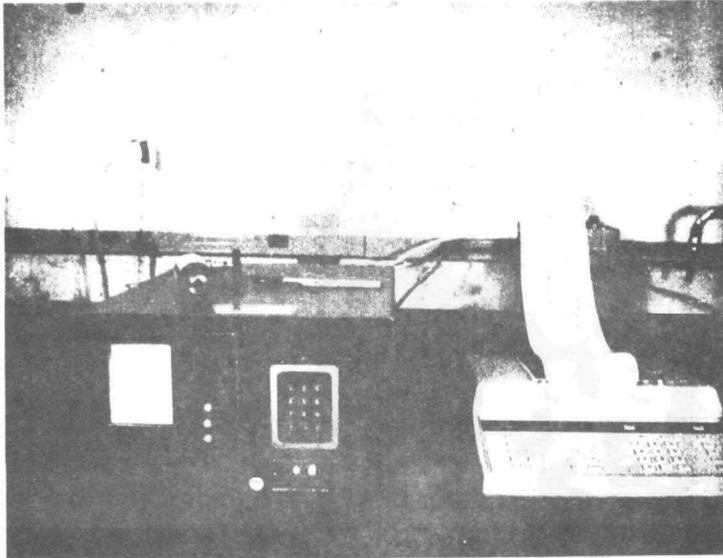
- Isothermal
- Temperature Programmed

SLIDE 504-16

COMMON DETECTORS

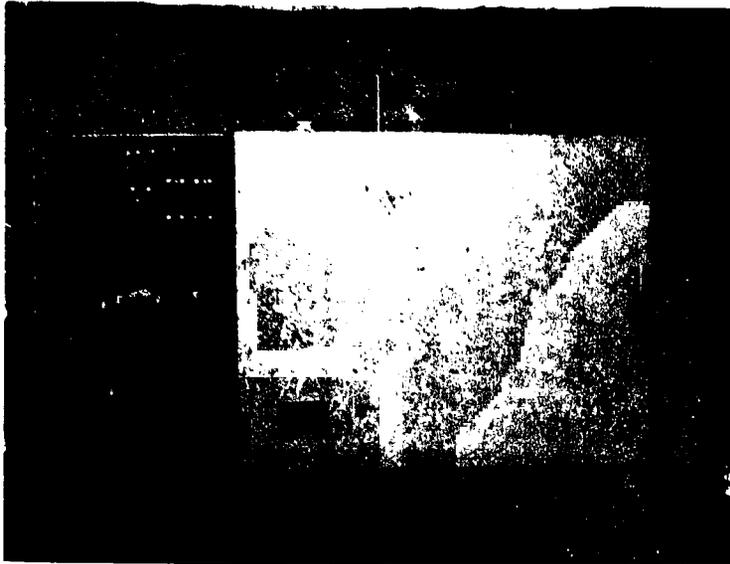
- Thermal Conductivity (TCD)
- Flame Ionization (FID)
- Electron Capture (ECD)
- Flame Photometric (FPD)
- Mass Spectrometer (MS)

Photo Ionization Detector



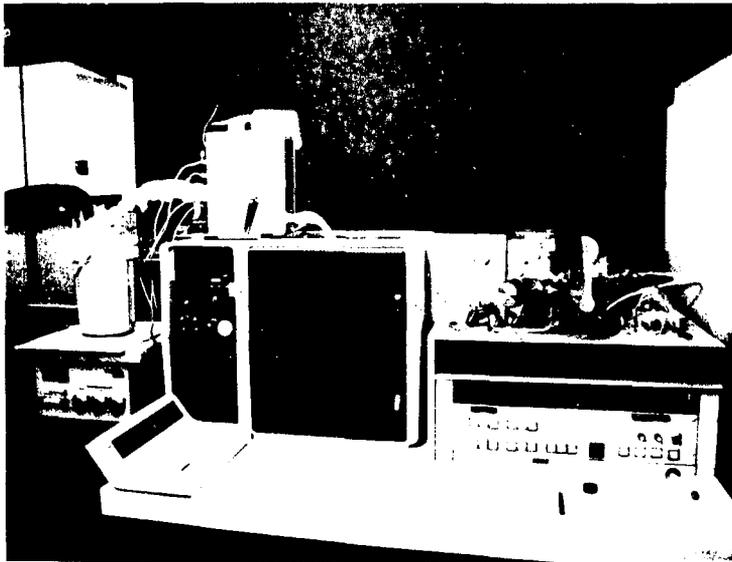
SLIDE 504-19

NOTES



SLIDE 504-20





SLIDE 504-22



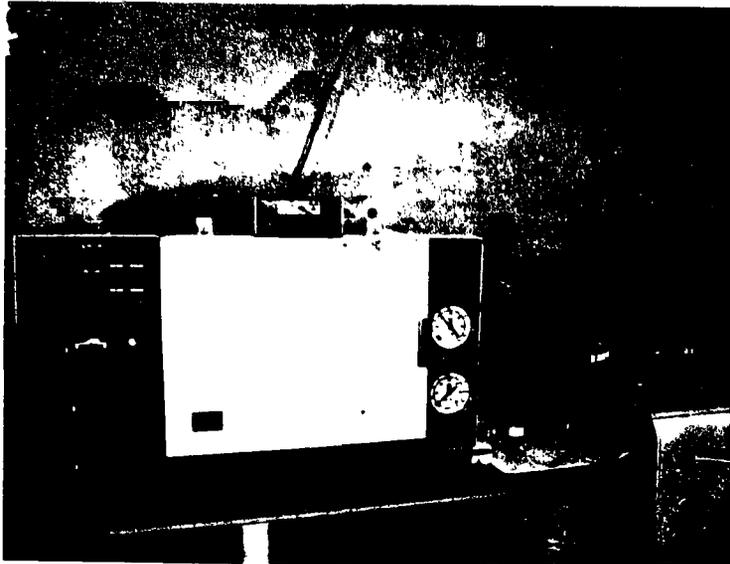
SLIDE 504-23

READOUTS

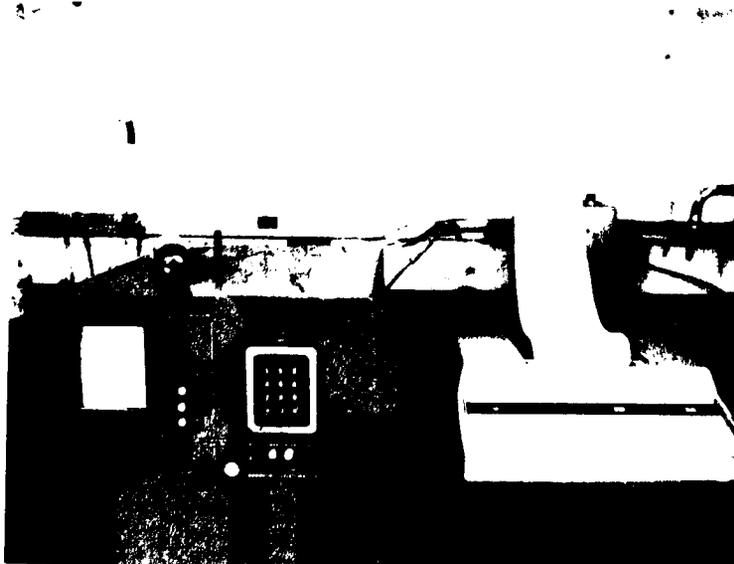
- Strip Charts
- Integration
- Microprocessor
- Computer

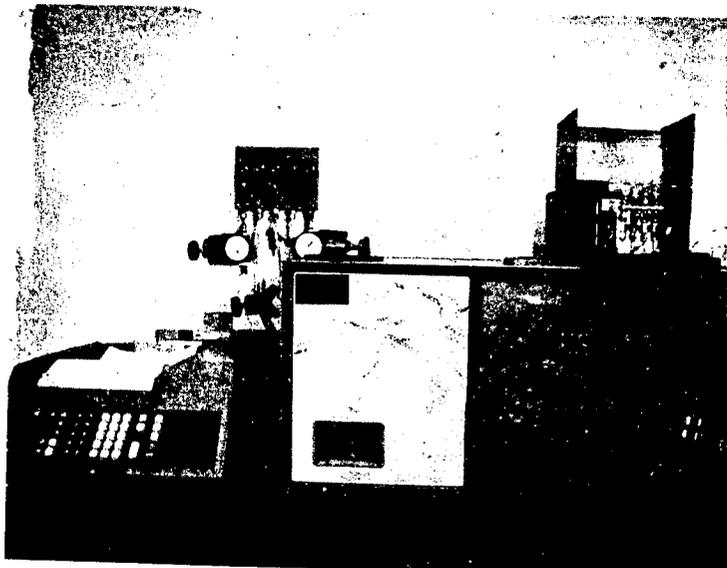
SLIDE 504-24

NOTES

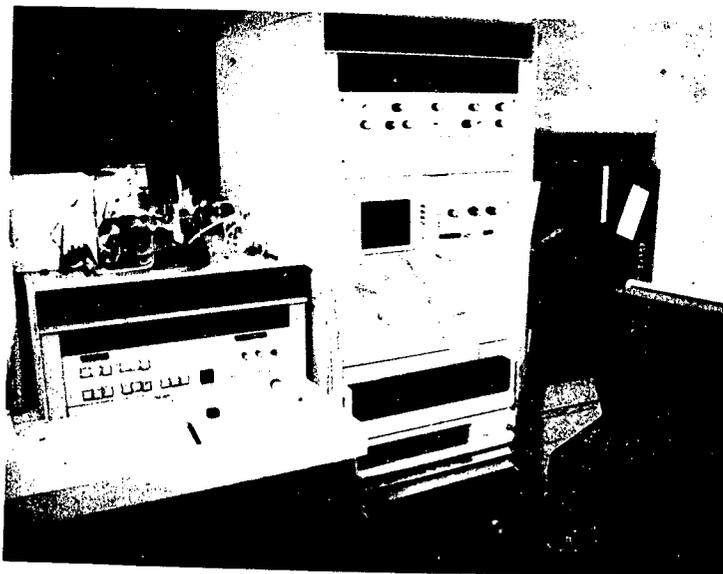


SLIDE 504-25





SLIDE 504-27

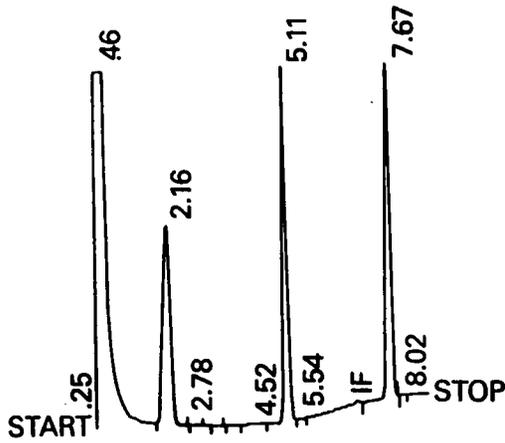


SLIDE 504-28

GAS CHROMATOGRAPHIC DATA

- Qualitative (retention time)
- Quantitative (peak area)

TYPICAL CHROMATOGRAM

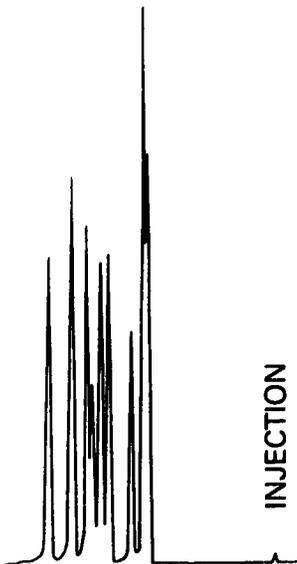


RUN NO. 38

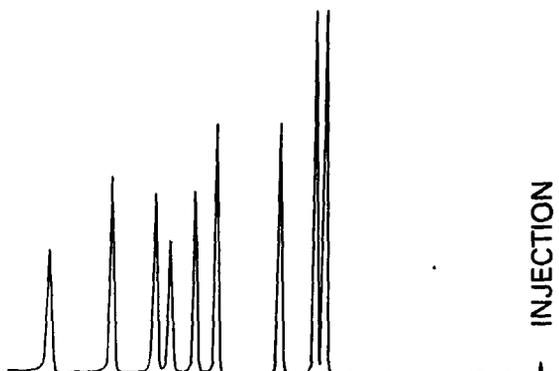
AREA%	RT	AREA	TYPE	AR/HT	AREA%
	0.25	16397	BH	0.193	0.057
	0.46	1.8154E + 07	ISHB	0.209	63.099
	2.16	3720200	TBP	0.220	12.931
	2.78	5931	TVP	0.214	0.021
	4.52	0	TPP	0.000	0.000
	5.11	3422400	TPP	0.106	11.895
	5.54	1296	TPP	0.038	0.005
	7.67	3449400	PB	0.108	11.989
	8.02	871	BP	0.097	0.003

TOTAL AREA = 2.8770E + 07
 MUL FACTOR = 1.0000E + 00

UNRESOLVED PEAKS



RESOLVED PEAKS



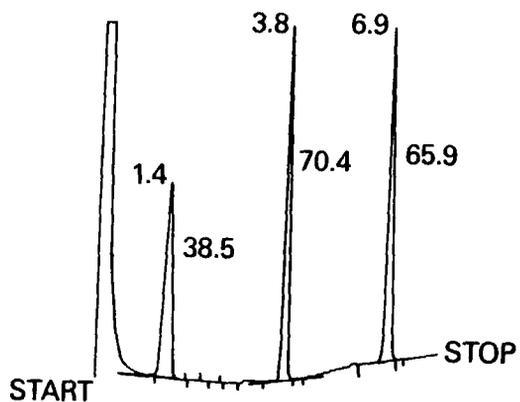
SLIDE 504-32

QUANTITATION METHODS

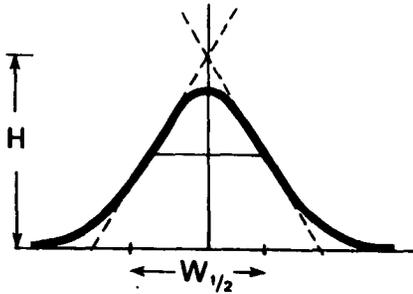
- Peak Height
- Peak Area
- Electronic (or Disc) Integration

SLIDE 504-33

PEAK HEIGHT MEASUREMENTS



PEAK AREA MEASUREMENTS



SLIDE 504-35

AREA CALCULATION

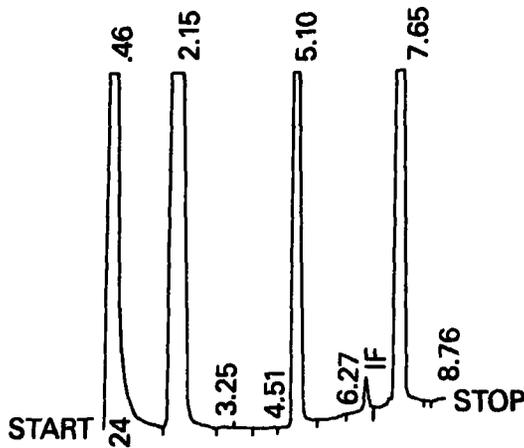
$$A = HW_{1/2}$$

Where:

- A = area
- H = peak height
- W_{1/2} = width at 1/2 the height

SLIDE 504-36

ELECTRONIC INTEGRATION

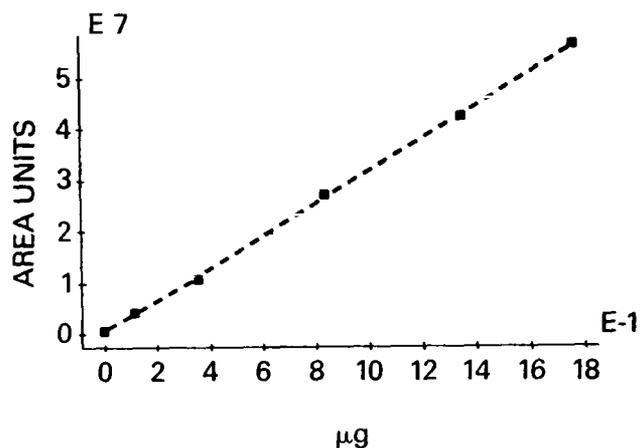


RUN NO. 39

RT	AREA	TYPE	AR/HT	AREA%
0.24	19247	BH	0.207	0.011
0.46	2.2381E + 07	SHH	0.257	12.545
2.15	5.5996E + 07	SHH	0.219	31.387
3.25	13508	TBB	0.140	0.008
4.51	3663	TBB	0.083	0.002
5.10	4.7534E + 07	ISHB	0.102	26.644
6.27	0	TBP	0.000	0.000
7.65	5.2449E + 07	ISBH	0.113	29.399
8.76	6039	ITPB	0.115	0.003

TOTAL AREA = 1.7840E + 08
 MUL FACTOR = 1.0000E + 00

CALIBRATION CURVE



SLIDE 504-38

PROBLEMS IN COMPOUND IDENTIFICATION

There are approximately 2,000,000 organic compounds, of which 15% are volatile. There are approximately 300,000 possible compounds which may be found in a chromatogram.

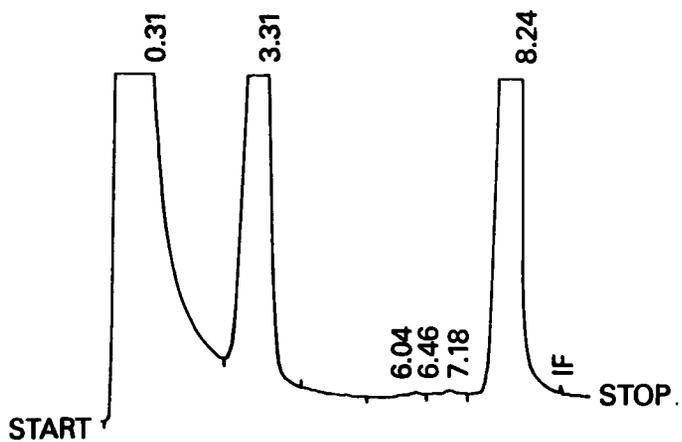
If this number of possibilities is not significantly reduced in the source, identification based on retention time only is impossible.

SLIDE 504-39

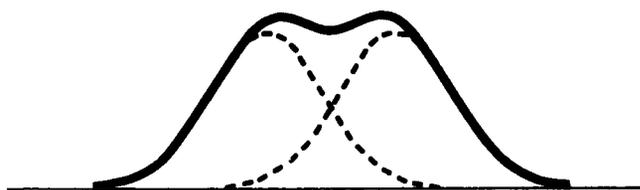
PROBLEMS IN QUANTIFICATION

- Tailing Peaks
- Overlapping Peaks

TAILING PEAK



OVERLAPPING PEAKS



ADEQUATE RESOLUTION

EPA Method 625 criterion for adequate resolution of overlapping compounds with similar mass spectra:

Baseline to valley height between the isomers is $< 25\%$ of the sum of the two peak heights.

SLIDE 504-43

ADEQUATE RESOLUTION METHOD 625



METHOD 1A

**Sample and Velocity Traverses for
Stationary Sources with Small Stacks or
Ducts**

SLIDE 1A-2

APPLICABILITY

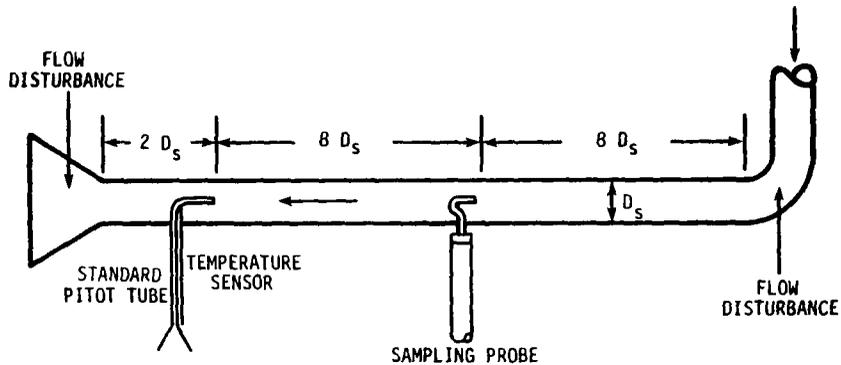
For stacks or ducts less than 0.30 m in diameter, or 0.071 m² in cross-sectional area, but \geq 0.10 m in diameter, or 0.0081 m² in cross-sectional area.

SLIDE 1A-3

PRINCIPLE

- For representative measurement of pollutant emissions and/or total volumetric flow rate.
- Select a measurement site where direction of effluent flow is known.

RECOMMENDED SAMPLING ARRANGEMENT FOR SMALL DUCTS

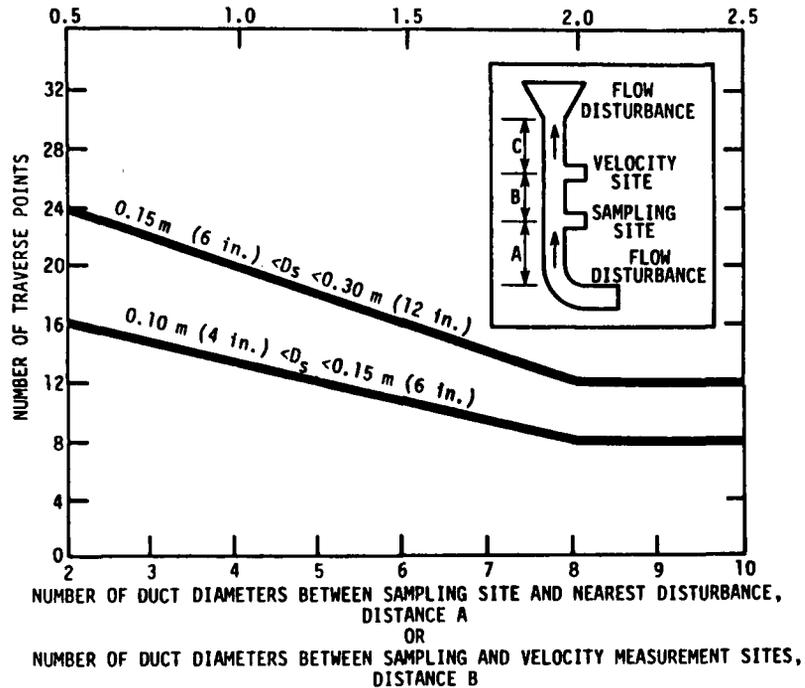


DETERMINING NUMBER OF TRAVERSE POINTS

- Determine distance between velocity and sampling sites and to nearest upstream and downstream disturbances.
- Divide each distance by stack diameter to determine distances in terms of stack diameter.
- Determine number of traverse points corresponding to each of three distances.

MINIMUM NUMBER OF TRAVERSE POINTS FOR SMALL DUCTS

NUMBER OF DUCT DIAMETERS BETWEEN VELOCITY MEASUREMENT AND NEAREST DISTURBANCE, DISTANCE C



SLIDE 1A-7

(cont.)

DETERMINING NUMBER OF TRAVERSE POINTS

- Choose highest of three numbers of traverse points.
- For circular stacks, number of points must be a multiple of 4.
- For rectangular ducts, use a matrix layout from Table 1-1 of Method 1.

SLIDE 1A-8

SELECTION OF MEASUREMENT SITE

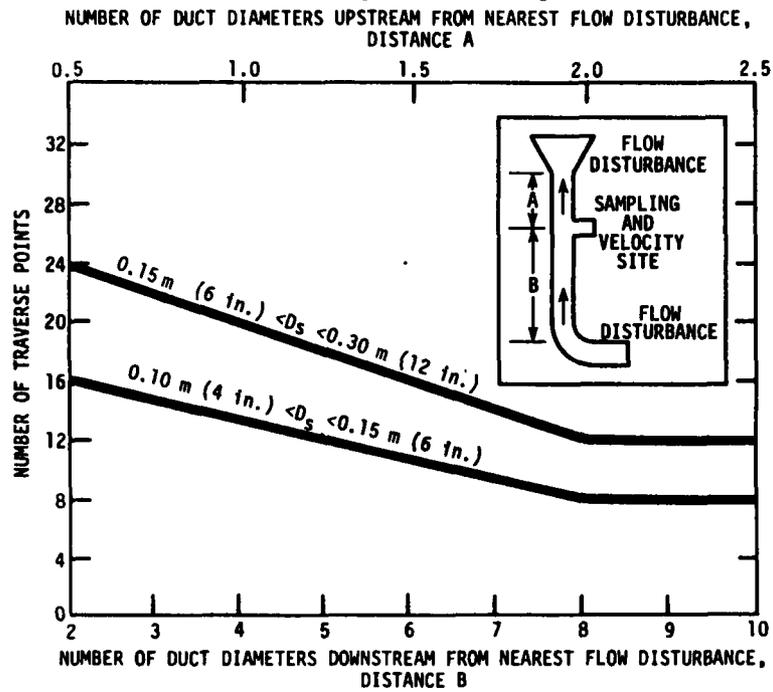
- Select a particulate measurement site located at least 8 stack diameters downstream and 10 diameters upstream from any flow disturbance.
- Locate velocity measurement site 8 equivalent diameters downstream of particulate measurement site.

SLIDE 1A-9

(cont.)**SELECTION OF MEASUREMENT SITE**

- Alternatively, locate particulate measurement site at least 2 diameters downstream and 2½ diameters upstream from any flow disturbance.
- Locate velocity measurement site 2 diameters downstream from particulate measurement site.

MINIMUM NUMBER OF TRAVERSE POINTS FOR SMALL DUCTS (Steady Flow Only)



METHOD 2A

**Direct Measurement of Gas Volume Through
Pipes and Small Ducts**

SLIDE 2A-2

APPLICABILITY

For measurement of gas flow rates in small ducts, either in-line or at exhaust positions, within temperature range of 0° to 50° C.

SLIDE 2A-3

PRINCIPLE

- **Gas volume meter is used to directly measure gas volume.**
- **Temperature and pressure measurement are made to correct volume to standard conditions.**

EQUIPMENT

GAS VOLUME METER

A positive displacement meter, turbine meter, or other direct volume measuring device capable of 2% accuracy.

BAROMETER

A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg.

SLIDE 2A-5

(cont.)

STOPWATCH

A stopwatch capable of measurement to within 1 second.

SLIDE 2A-6

PROCEDURE

INSTALLATION

Install volume meter in such a manner to assure leak-tight connections and in a location to avoid severe vibrations and other factors that may affect meter calibration.

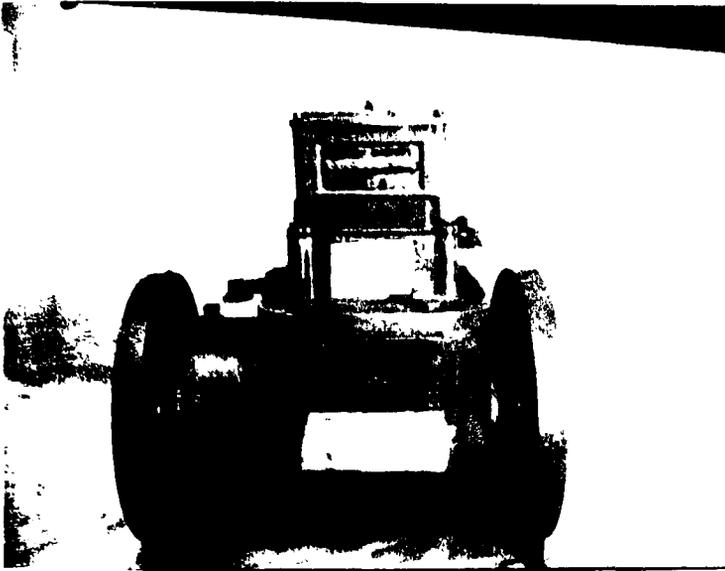
LEAK TEST

Positive Pressure

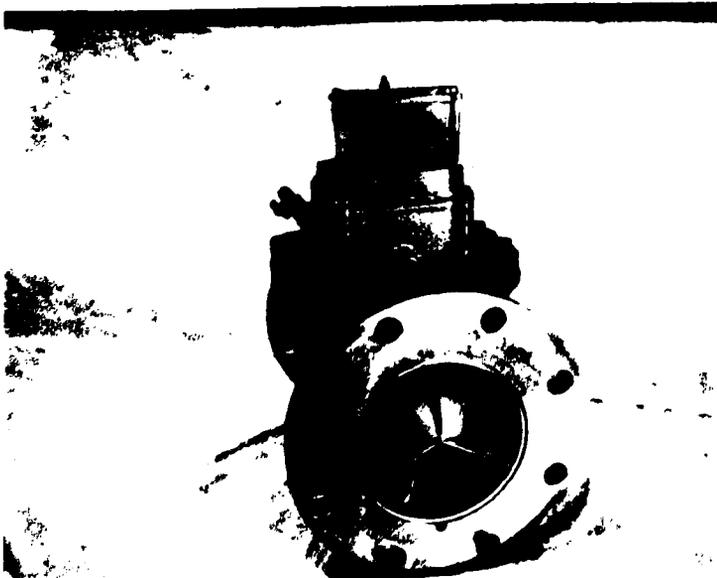
Leak-check meter connections using a liquid leak detector solution containing a surfactant.

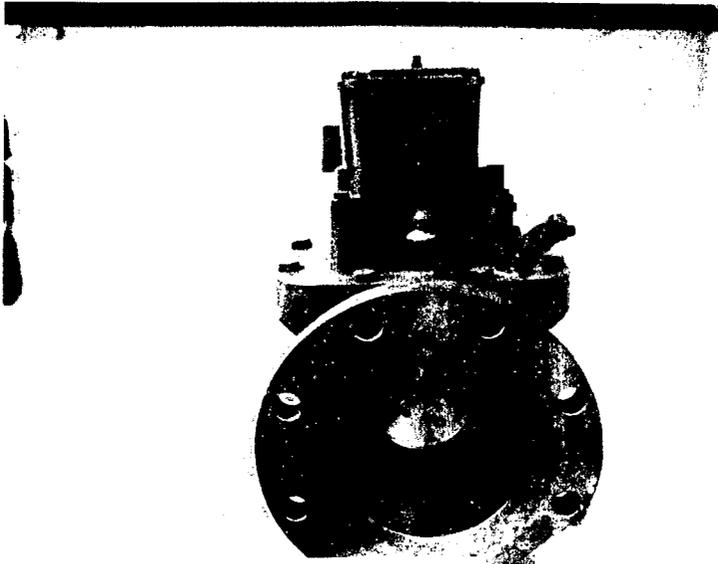
SLIDE 2A-7

NOTES

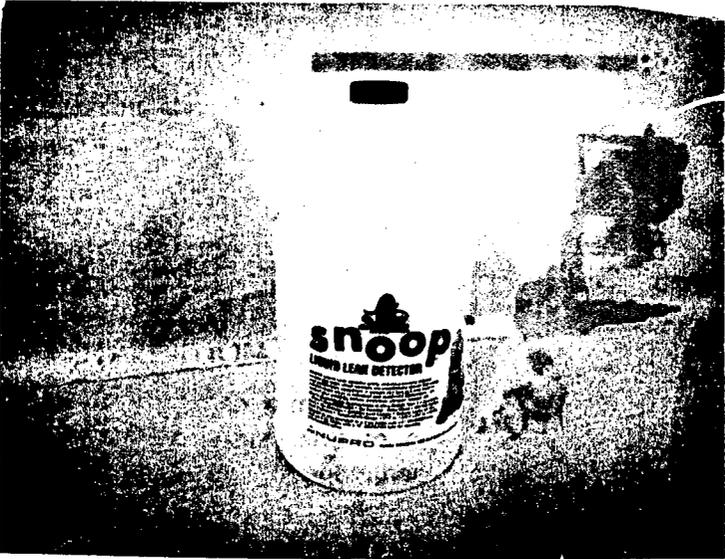


SLIDE 2A-8





SLIDE 2A-10



(cont.)

LEAK TEST

Negative Pressure

Block flow at inlet of line and observe meter.
Alternatively, visually check all connections
and assure tight seals.

SLIDE 2A-12

VOLUME MEASUREMENT

CONTINUOUS STEADY FLOW

1. Record initial meter volume reading, temperature, and pressure and start stopwatch.
2. Record meter temperature and pressure throughout test period.
3. At end of test, stop timer and record elapsed time, final volume reading, meter temperature and pressure.

SLIDE 2A-13

(cont.)

CONTINUOUS STEADY FLOW

4. Record barometric pressure at beginning and end of each test run.

NONCONTINUOUS FLOW

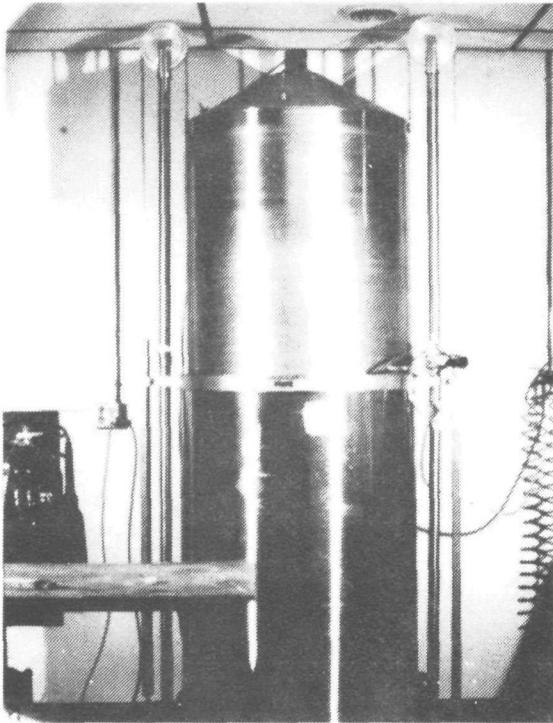
1. Record all meter parameters and start and stop times corresponding to each process cyclical or noncontinuous event.

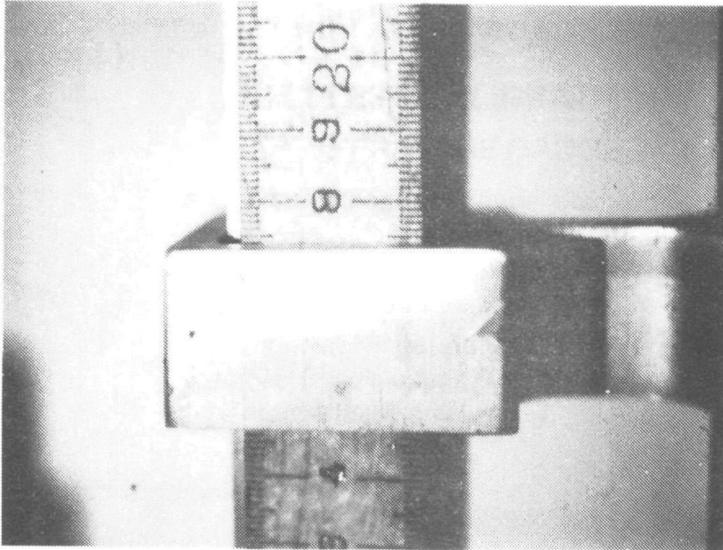
INITIAL CALIBRATION

VOLUME METER

- Calibrate volume meter against standard reference meter prior to initial use in field.
- Run calibration over at least 3 different flow rates.
- Difference between maximum and minimum values at each flow rate should be no greater than 0.030 and meter coefficient should be between 0.95 and 1.05.

SLIDE 2A-15





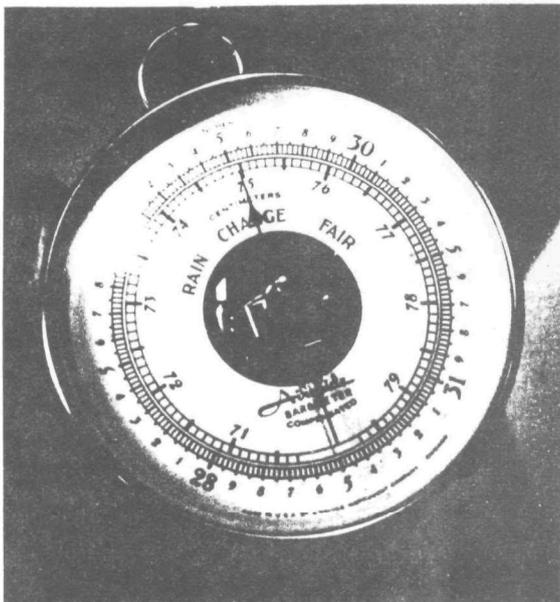
SLIDE 2A-17

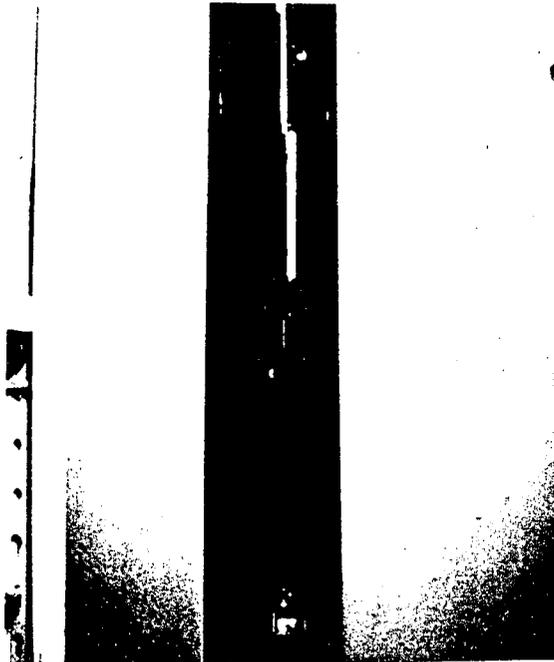
(cont.)

INITIAL CALIBRATION BAROMETER

- Calibrate barometer against mercury barometer prior to field test.

SLIDE 2A-18





POSTTEST CALIBRATION

VOLUME METER

- Check volume meter calibration by performing 3 calibration runs at a single intermediate flow rate with meter pressure set at average value encountered during field tests.
- Calibration is acceptable if posttest value is within 5% of pretest value.

(cont.)

POSTTEST CALIBRATION

TEMPERATURE GAUGE

- Check temperature gauge after each test series against ASTM mercury-in-glass reference thermometer, at ambient temperature.
- Temperature gauge should agree within 2% absolute temperature of reference thermometer.

SLIDE 2A-22



VOLUME METER

$$Y_m = \frac{(V_{rf} - V_{ri}) (t_r + 273)}{(V_{mf} - V_{mi}) (t_m + 273)} \frac{P_b}{(P_b + P_g)}$$

Where:

Y_m = test volume meter calibration coefficient, dimensionless.

V_r = reference meter volume reading, m³.

V_m = test meter volume reading, m³.

t_r = 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.

SLIDE 2A-24

VOLUME

$$V_{ms} = 0.3853 \bar{Y}_m (V_{mf} - V_{mi}) \left(\frac{P_b + P_g}{T_m} \right)$$

Where:

V_{ms} = volume meter reading, m³ at standard conditions, 20°C and 760 mm Hg.

\bar{Y}_m = meter calibration coefficient, dimensionless.

V_{mf} = meter volume reading, m³ at final reading for run.

V_{mi} = meter volume reading, m³ at initial reading for run.

P_b = barometric pressure, mm Hg.

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

T_m = average absolute meter temperature, °K.

GAS FLOW RATE

$$Q_s = \frac{V_{ms}}{\Theta}$$

Where:

Q_s = gas flow rate, m³/min, standard conditions.

V_{ms} = volume meter reading, m³ at standard conditions, 20^oC and 760 mm Hg.

Θ = elapsed run time, min.

METHOD 2B

Determination of Exhaust Gas Volume Flow
Rate from Gasoline Vapor Incinerators

SLIDE 2B-2

APPLICABILITY

For measurement of exhaust volume flow rate from incinerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons), assuming amount of auxiliary fuel is negligible.

SLIDE 2B-3

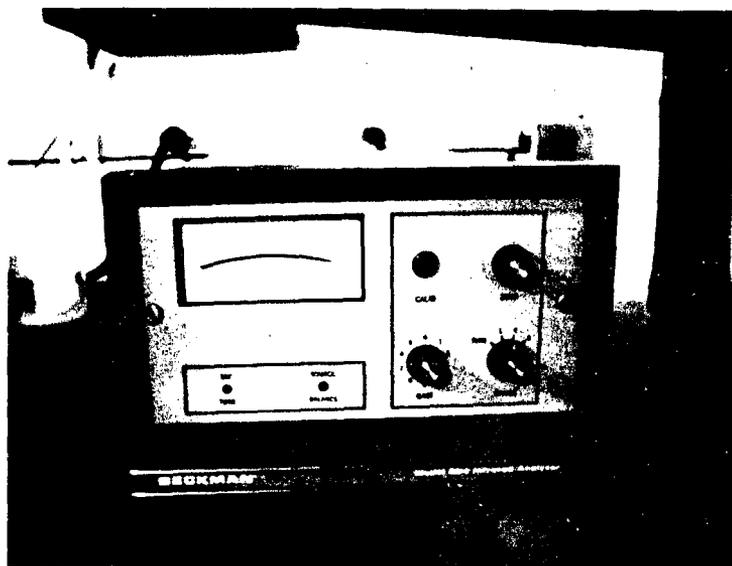
PROCEDURE

INLET INSTALLATION

- Install volume meter in vapor line to incinerator inlet.
- Install sample probe, sample line and organic analyzer system at volume meter inlet.

EXHAUST INSTALLATION

- Install sample probe, heated sample line and sample manifold to incinerator exhaust.
- Install CO₂, CO and organic analyzers to manifold system.



SLIDE 2B-5

EXHAUST GAS VOLUME

$$V_{es} = V_{is} \frac{K(HC_i)}{K(HC_e) + CO_{2e} + CO_e - 300}$$

where:

V_{es} = exhaust gas volume, m^3 .

V_{is} = inlet gas volume, m^3 .

K = calibration gas factor - 2 for ethane calibration gas.
 3 for propane calibration gas.
 4 for butane calibration gas.

HC_e = mean organic concentration in system exhaust as defined by the calibration gas, ppmv.

HC_i = mean organic concentration in system inlet as defined by the calibration gas, ppmv.

CO_{2e} = mean carbon dioxide concentration in system exhaust, ppmv.

CO_e = mean carbon monoxide concentration in system exhaust, ppmv.

(cont.)

PROCEDURE RECORDING

- Permanently record output of each analyzer on an analog strip chart, digital recorder or other recording device.
- Chart speed or number of readings per time unit must be similar for all analyzers.
- Minimum data recording requirement for each analyzer is one measurement value per minute.

SLIDE 2B-7

CALIBRATION INITIAL

- Prepare and calibrate all equipment and analyzers according to procedures in respective methods.
- Introduce all calibration gases at the connection between probe and sample line.
- If manifold system is used for exhaust analyzers, all analyzers and sample pumps must be operating when calibrations are complete.

SLIDE 2B-8

CALIBRATION INITIAL

- Methane should not be used as an organic calibration gas.

POSTTEST

- Introduce calibration gases as specified in respective methods.
- If analyzer output does not meet specifications of method, invalidate test data for that period.

(cont.)**POSTTEST**

- Alternatively, calculate volume results using initial and final calibration data and report both volumes.
- Calculate results using volume which results in greatest emission rate or concentration.

SLIDE 2B-10

SAMPLING PROCEDURE

- Record initial parameters for inlet volume meter.
- Make all recorder strip charts to indicate start of test.
- Continue recording inlet and exhaust concentrations throughout test.
- Note periods of process interruption on strip charts.
- At end of test, record final parameters for inlet volume meter and mark end on all strip charts.

SLIDE 2B-11

EXHAUST GAS VOLUME

$$V_{es} = V_{is} \frac{K(HC_i)}{K(HC_e) + CO_{2e} + CO_e - 300}$$

(See Slide 2B-5 for nomenclature.)

EXHAUST GAS VOLUME FLOW RATE

$$Q_{es} = \frac{V_{es}}{\Theta}$$

Where:

Q_{es} = exhaust gas volume flow rate, m³/min.

V_{es} = exhaust gas volume, m³.

Θ = sample run time, min.

METHOD 2C

Determination of Stack Gas Velocity and Volumetric Flow Rate from Small Stacks or Ducts (Standard Pitot Tube)

SLIDE 2C-2

APPLICABILITY

Method applicability is identical to Method 2 except it is limited to stationary source stacks less than 0.30 m in diameter, or 0.071 m² in cross-sectional area, but ≥ 0.10 m in diameter or 0.0081 m² in cross-sectional area.

SLIDE 2C-3

PRINCIPLE

Average gas velocity in a stack or duct is determined from gas density and from measurement of velocity heads with a standard pitot tube.

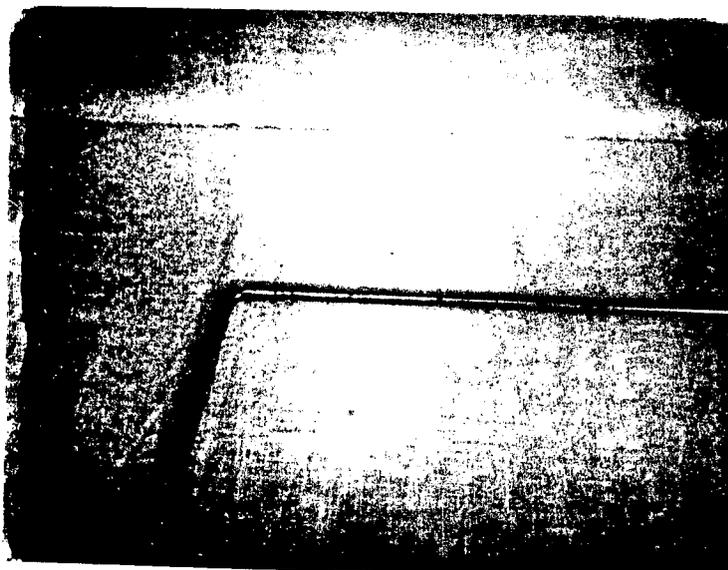
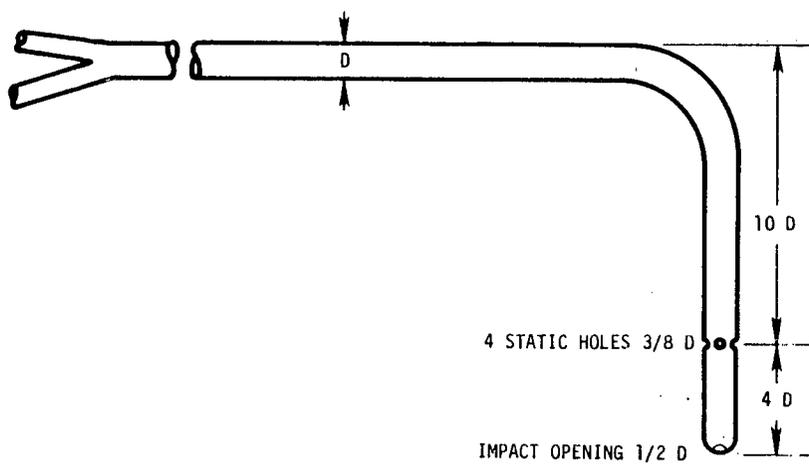
SLIDE 2C-4

APPARATUS

STANDARD PITOT TUBE

- One which meets specifications of Method 2.
- Assign coefficient of 0.99 unless it is calibrated against standard pitot tube with NBS-traceable coefficient.

MODIFIED HEMISPHERICAL-NOSED PITOT TUBE



(cont.)**ALTERNATIVE PITOT TUBE**

- Modified hemispherical-nosed pitot tube with shortened stem and enlarged impact and static pressure holes.
- Assign coefficient of 0.99 unless it is calibrated.

SLIDE 2C-8

VELOCITY PRESSURE VALIDITY TEST

- Measure velocity pressure at final traverse point.
- Clean out impact and static holes of standard pitot tube by back-purging with pressurized air.
- Remeasure velocity pressure at final traverse point.
- If velocity pressure readings made before and after air purge are the same ($\pm 5\%$), traverse is acceptable.

SLIDE 18-1

NOTES

METHOD 18

Measurement of Gaseous Organic Compound Emissions by Gas Chromatography

SLIDE 18-2

APPLICABILITY

Provides concentration data on approximately 90% of total gaseous organic mass emitted from an industrial source.

Note: Does not include techniques to identify and measure trace amounts of organic compounds, such as those found in building air and fugitive air emission sources.

SLIDE 18-3

PRINCIPLE

Based on separating components of a gas mixture in a gas chromatographic column and measuring separated components with suitable detector.

SLIDE 18-4

METHOD CRITERIA

- Range — 1 ppm to upper limit of GC
- Sensitivity — minimum detection limit or signal-to-noise ratio 3:1
- Precision — $\pm 5\%$ of mean value
- Accuracy — $\pm 10\%$ audit sample value

PRESURVEY SAMPLES

A presurvey shall be performed on each source to be tested to obtain all information necessary to design emission test.

SLIDE 18-6

PRESURVEY DATA

OBTAIN:

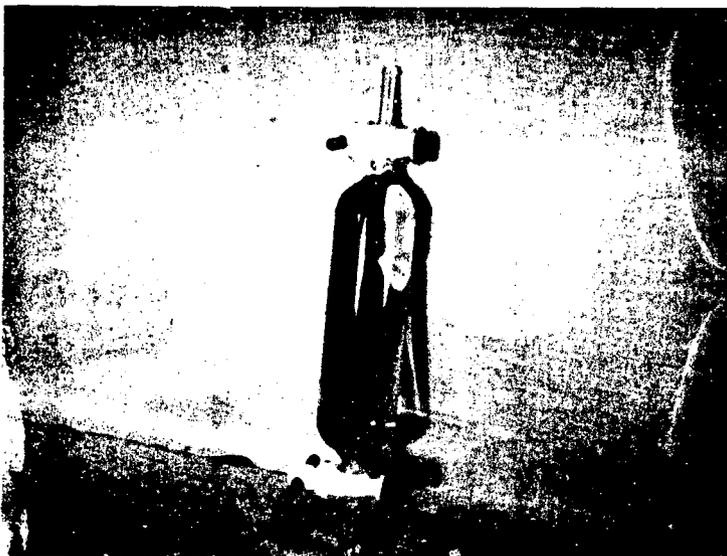
- stack temperature and temperature range
- approximate particulate concentration
- static pressure
- water vapor content

SLIDE 18-7

PRESURVEY SAMPLE TRAIN

- 250 ml double-ended glass sampling flask
- Method 7 evacuated flask
- Tedlar or aluminized Mylar flexible bag
- Adsorption tubes

SLIDE 18-8



PRESURVEY SAMPLE ANALYSIS

- Select GC column
- Select GC conditions for good resolution
- Prepare presurvey samples
- Analyze presurvey samples

SLIDE 18-10

CRITERIA FOR SAMPLE ANALYSIS

1. Prepare calibration standards by proper technique
2. Determine optimum GC setting
3. Obtain retention times with repeatability of ± 0.5 s
4. Use smaller sample loop or dilution if necessary
5. Identify all peaks $>5\%$ of total

SLIDE 18-11

CALIBRATION STANDARDS PREPARATION

- Liquid standard in desorbing solution
- Direct analysis of NBS reference gases or commercial certified gas mixtures
- Rotameter dilution of high concentration cylinder gases
- Direct syringe-bag dilution for known quantity volatile liquid material
- Indirect syringe-bag dilution for known quantity of less volatile liquid materials

CALIBRATION OF DILUTION TECHNIQUE

1. Use rotameter and micrometer values on high concentration and diluent gas
2. A positive displacement pump or other meter device may be used to provide a fixed flow of high concentration gas.

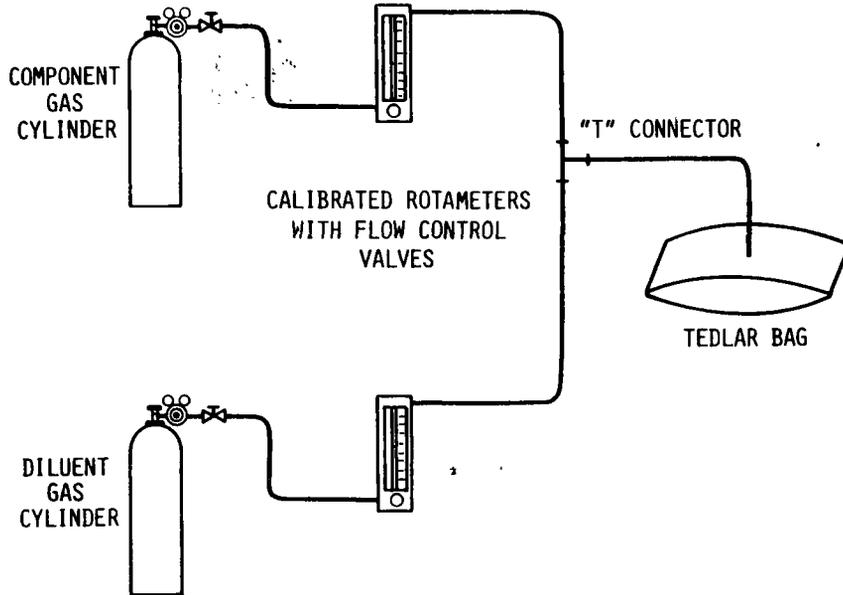
SLIDE 18-13

(cont.) CALIBRATION OF DILUTION TECHNIQUE

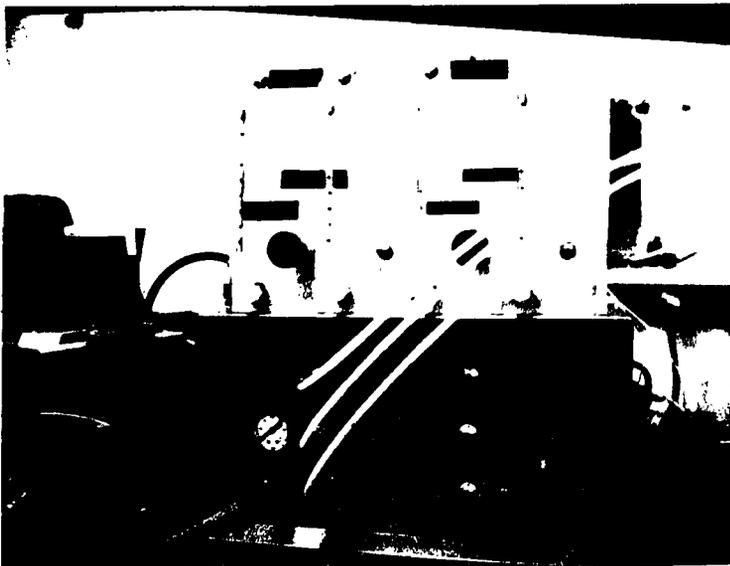
3. Calibrate rotameter or other metering devices with a suitably sized bubble meter, spirometer or wet test meter.
4. Check dilution system by comparing calculated concentration of diluted high concentration gas to direct analysis of low concentration gas.

Note: Use single-stage dilutions to prepare calibration mixtures up to about 1:20 dilution factor. For greater dilutions, use a double dilution system and check in similar manner.

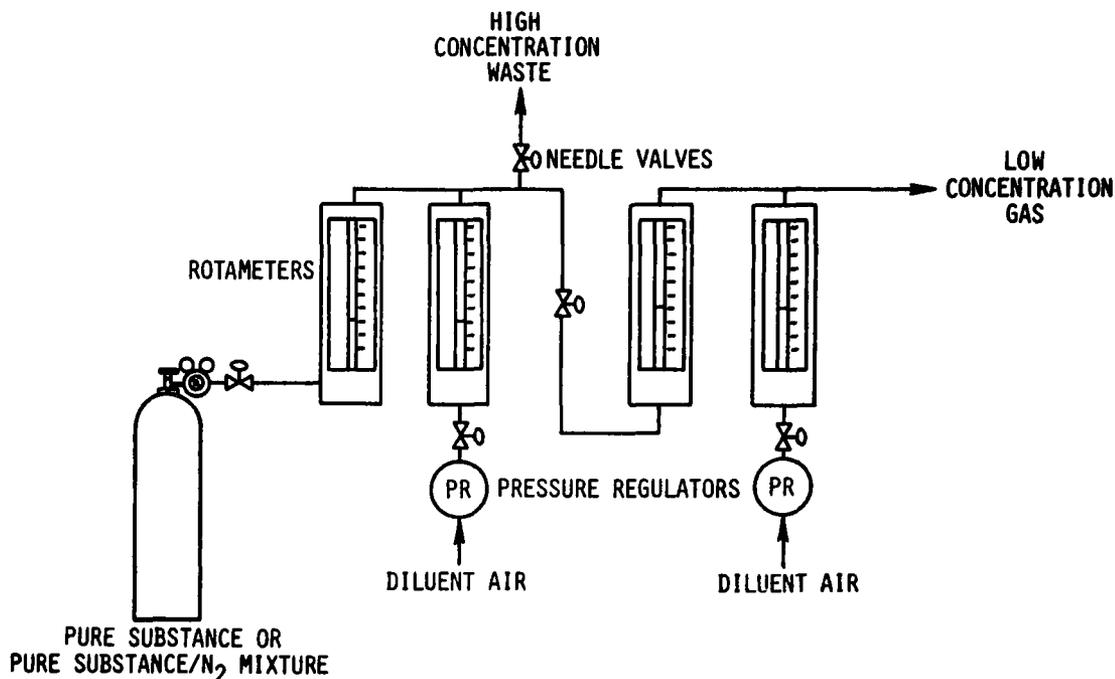
SINGLE-STAGE CALIBRATION GAS DILUTION SYSTEM



SLIDE 18-15



TWO-STAGE DILUTION APPARATUS



SLIDE 18-17

DILUTED GAS CONCENTRATION

$$C_a = \frac{10^6(\bar{X}_a q_a)}{q_a + q_d}$$

Where:

C_a = concentration of component "a" in ppm.

\bar{X}_a = mole fraction of component "a" in the calibration gas to be diluted.

q_a = flow rate of component "a" at measured temperature and pressure.

q_d = diluent gas flow at measured temperature and pressure.

**COMPONENT IN FINAL GAS MIXTURE
CONCENTRATION**

$$C_a = 10^6 \bar{X} \left(\frac{q_{a1}}{q_{a1} + q_{d1}} \right) \left(\frac{q_{a2}}{q_{a2} + q_{d2}} \right)$$

Where:

C_a = concentration of component "a" in ppm.

\bar{X}_a = mole fraction of component "a" in original gas.

q_{a1} = flow rate of component "a" in stage 1.

q_{a2} = flow rate of component "a" in stage 2.

q_{d1} = flow rate of diluent gas in stage 1.

q_{d2} = flow rate of diluent gas in stage 2.

DILUTION BY DIRECT SYRINGE BAG TECHNIQUE

1. Use a 10-liter Tedlar bag that has passed a leak check.
2. Meter a known volume of about 5 liters with a 0.5 liter per revolution dry gas meter.
3. While filling the bag inlet inject a known quantity of material through the wall of bag or septum.
4. Withdraw syringe and cover hole with tape.

SLIDE 18-20



DILUTION BY INDIRECT SYRINGE/ BAG TECHNIQUE

1. Use a 50-liter Tedlar bag that has passed a leak check.
2. Adjust the flow from the nitrogen tank so it will pass through the dry gas meter and impinger on a hot plate with boiling water to fill bag in approximately 15 mins.

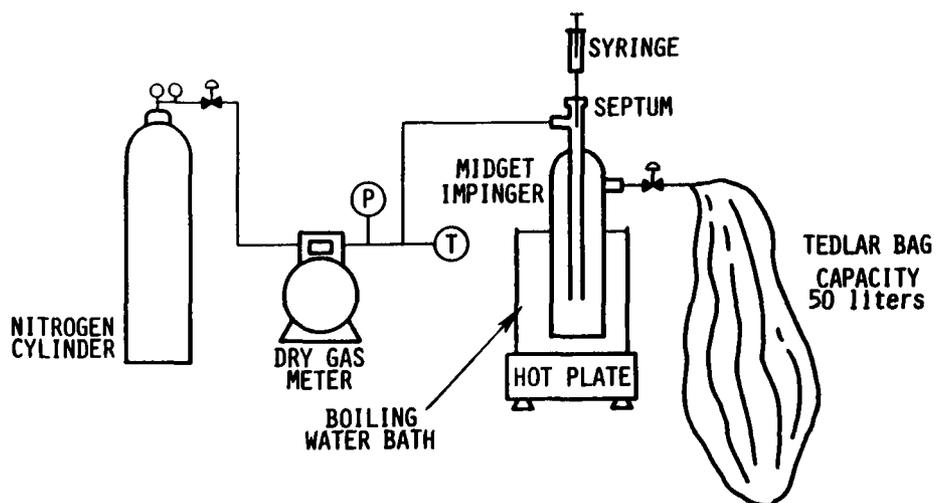
SLIDE 18-22

(cont.) DILUTION BY INDIRECT SYRINGE/ BAG TECHNIQUE

3. Fill the liquid syringe with desired liquid volume.
4. Inject liquid to inlet of heated impinger while bag is filling.
5. Fill bag and record all necessary data.
6. Let sample equilibrate for one hour and analyze.

SLIDE 18-23

APPARATUS FOR PREPARING STANDARD GAS MIXTURES



STANDARD CONCENTRATION

$$C_{\text{std sol}} = \frac{760 (L_v (\rho) (273 + T_m))}{293(M_f - M_i) (P_{\text{bar}} + P_m)}$$

Where:

$C_{\text{std sol}}$ = standard solvent concentration, mg/std liter.

L_v = liquid volume injected, ml.

ρ = liquid density at room temperature, g/ml.

T_m = meter temperature, °C.

M_f, M_i = final and initial meter reading, liters.

P_{bar} = local barometric pressure (absolute), mm Hg.

P_m = meter pressure (gauge), mm Hg.

SLIDE 18-25

CALIBRATION CURVES PREPARATION

1. Obtain calibration standards such that three concentrations per attenuator range are available. *written to strip chart recording electronic integrator can calc areas accurately*
2. Establish proper GC conditioning.
3. Flush sampling loop for 30 s at 100 ml/min.
4. Repeat all standard injections until two consecutive injections agree within $\pm 5\%$ of average.

SLIDE 18-26

(cont.)**CALIBRATION CURVES PREPARATION**

5. Plot concentrations along abscissa and calibration area values along ordinate.
6. Perform a regression analysis and draw a least squares line.
7. When dilution technique is used, calculated value should be within $\pm 10\%$ of expected concentration.

ADSORPTION AND DESORPTION EFFICIENCY

ADSORPTION—The backup portion of the sample must be < 10% of the total sample.

DESORPTION—A minimum desorption efficiency of 50% must be obtained.

SLIDE 18-28

EVALUATION OF CALIBRATION AND ANALYSIS PROCEDURES

1. Perform audit analysis as described in Part 61 Appendix C, Procedure 2.
2. Audit analysis shall agree within $\pm 10\%$ of audit concentration

SLIDE 18-29

FINAL SAMPLING AND ANALYSIS PROCEDURES

CONSIDER:

- Safety
- Source conditions
- Compounds to be measured

SAFETY

In situations where a hydrogen flame is a hazard and no intrinsically safe GC is suitable, use flexible bag collection technique or an absorption technique.

SLIDE 18-31

SOURCE CONDITIONS

- If source temperature is below 100°C and organic concentrations are suitable for detector, use direct interface method.
- If source emissions require dilution, use a dilution interface and either bag sample or adsorption tubes.

SLIDE 18-32

COMPOUNDS TO BE MEASURED

If compounds have a stability problem or polar compounds are to be sampled, use direct interfacing or dilution direct interfacing.

METHOD 21

Determination of Volatile Organic Compound Leaks

SLIDE 21-2

APPLICABILITY

Applies to determination of volatile organic compound leaks from the following process equipment:

- flanges and other connections
- pumps and compressors
- pressure relief devices
- process drains
- open-ended valves
- accumulator vessel vents
- pump and compressor seal system
degassing vents
- agitator seals
- access door seals

SLIDE 21-3

PRINCIPLE

- Suitable portable instrument is used to detect VOC leaks from individual sources.
- Procedure is intended to locate and classify leaks only, and is not to be used as a direct measure of mass emission rates from individual sources.

METHOD 21 PERFORMANCE REQUIREMENTS AND SPECIFICATIONS

1. The device must respond to those organic compounds processed at the facility.
2. The analyzer shall be capable of detecting the leak definition concentration (often 10,000 ppmv) specified in the applicable regulation.
3. The scale of the instrument meter shall be readable to ± 5 percent of the specified leak definition concentration.

SLIDE 21-5

(cont.)

4. The instrument shall be equipped with a pump so that a continuous sample is provided to the detector. The nominal sample flow rate shall be 0.5 to 3 liters per minute.
5. The instrument shall be intrinsically safe for operation in explosive atmospheres as defined by the National Electrical Code by the National Fire Prevention Association.

SLIDE 21-6

(cont.)

6. The instrument response factors for the individual compounds to be measured must be less than 10 (compared to the reference compound specified in the applicable regulation).
7. The instrument response time must be equal to or less than 30 seconds, using the instrument configuration used during testing.
8. The calibration precision must be equal to or less than 10 percent of the calibration gas valve (relative standard deviation of ≤ 10 percent).

TYPES OF METHOD 21 ANALYZERS

- Flame ionization
- Photoionization
- Infrared adsorption
- Catalytic combustion

SLIDE 21-8

FLAME IONIZATION DETECTOR (FID)

PRINCIPLE OF OPERATION

The sample stream is introduced into a hydrogen flame. Upon combustion in the flame, organic compounds produce ions which are collected at an electrode. The resultant current flow is measured with an electrometer.

SLIDE 21-9

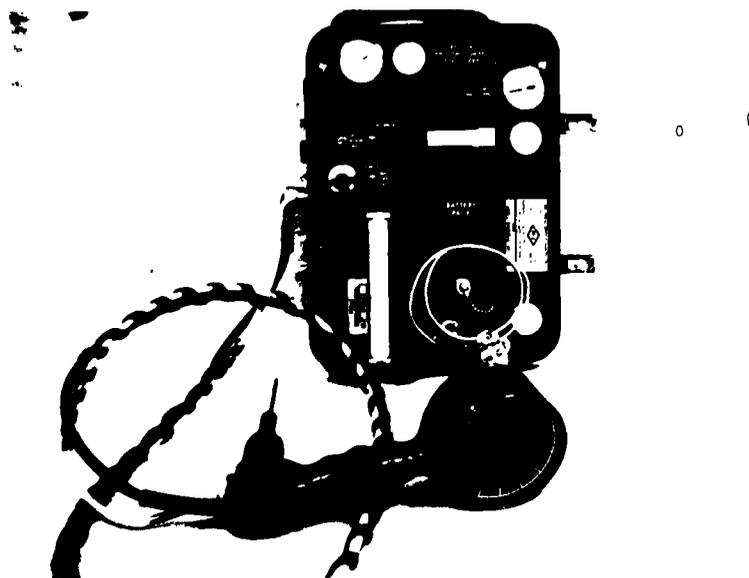
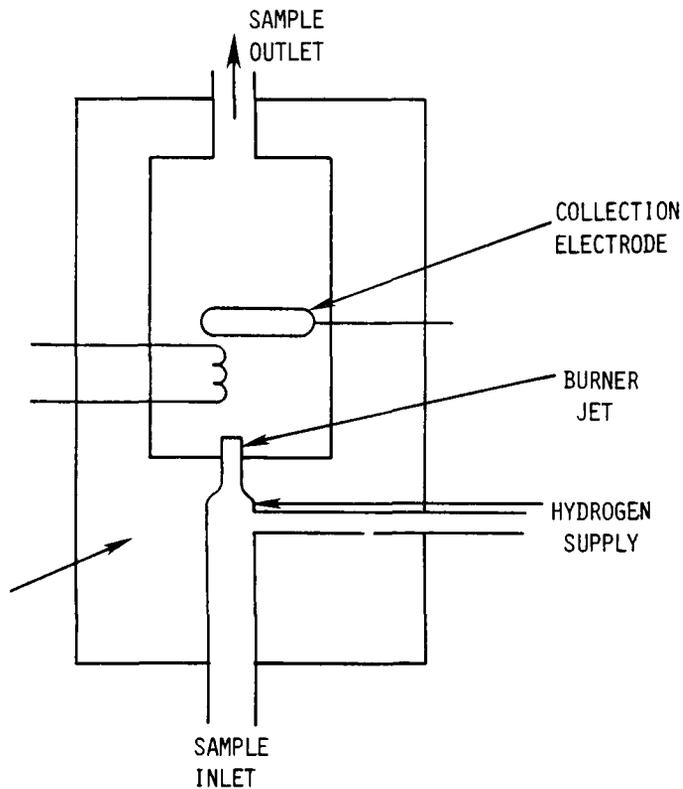
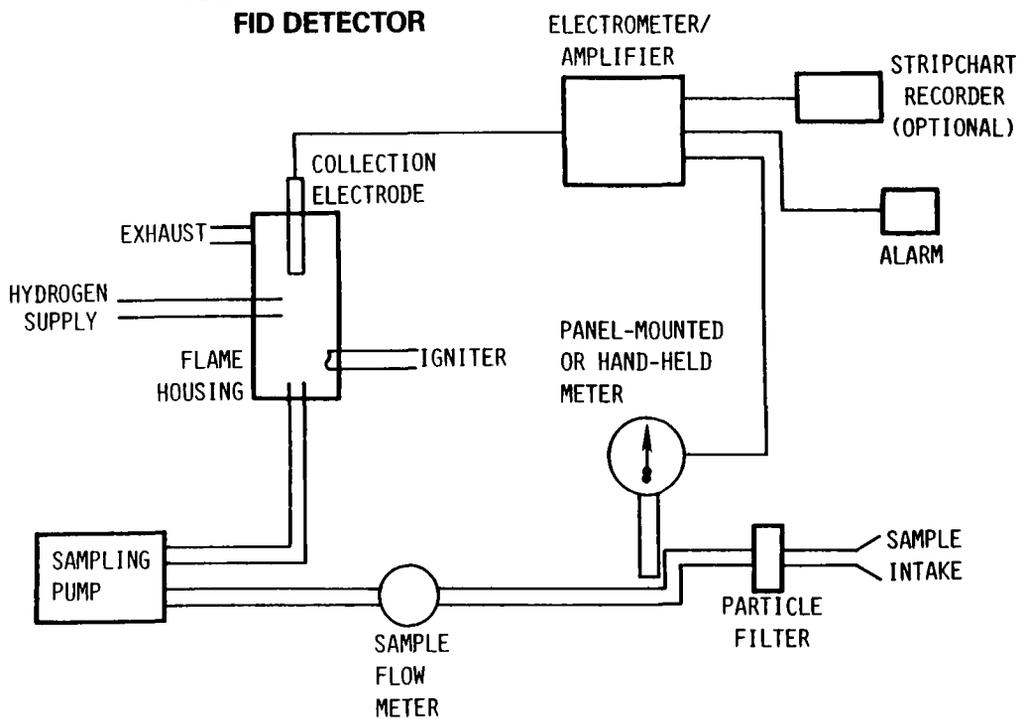


DIAGRAM OF A FLAME IONIZATION DETECTOR (FID)



VOC ANALYZER USING FID DETECTOR



GENERAL GUIDELINES FOR SYSTEM OPERATION USING A FID ANALYZER

SLIDE 21-13

FID OPERATION

1. Place sample probe at source.
2. Periodically recheck zero setting.
3. Avoid monitoring obvious leaks.
4. Periodically check flow rate and fuel supply and perform maintenance as required.

SLIDE 21-14

FID CALIBRATION

1. Set instrument zero with flame off.
 2. Ignite flame and allow to stabilize.
 3. Set zero.
 4. Introduce known concentration of calibration compound near leak definition limit and observe reading.
- Note:** If necessary, adjust calibration knob to read known concentration and repeat zero and calibration determinations until further adjustment is not required.

FID QUALITY CONTROL/PERFORMANCE VERIFICATION PROCEDURES

1. Check background (zero) air reading several times per day and after each high level measurement.
2. Check calibration daily.
3. Check air flow daily.
4. Leak check air flow system and fuel line daily.

SLIDE 21-16

(cont.)

5. Check instrument response factor.
6. Check calibration precision for three replicate determinations every three months or after each use, whichever is longer.
7. Check response time before performing leak detection measurements and whenever changes are made in the air flow, detector or electronic portions of the instruments.

SLIDE 21-17

FID SAFETY CONSIDERATIONS

- User should consult manufacturer's literature.
- Primary concern is possibility for explosion.
- Operator should make sure that flame arrestors are properly installed.

(cont.)

- Refueling, battery charging, or replacement and similar servicing and maintenance procedures should not be performed in an area where high VOC levels might be present.
- Only VOC units which are certified as intrinsically safe for the particular facility being monitored are permitted to be used for Method 21.

SLIDE 21-19

FID PROBLEMS

- Chlorinated solvents
- Moisture

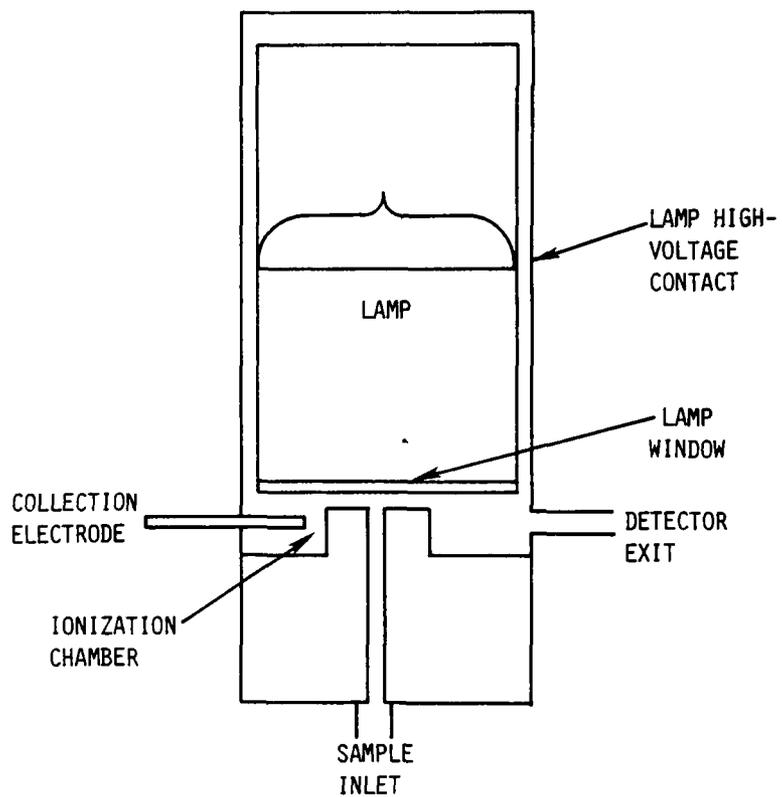
SLIDE 21-20

PHOTOIONIZATION DETECTOR (PID)

PRINCIPLE OF OPERATION

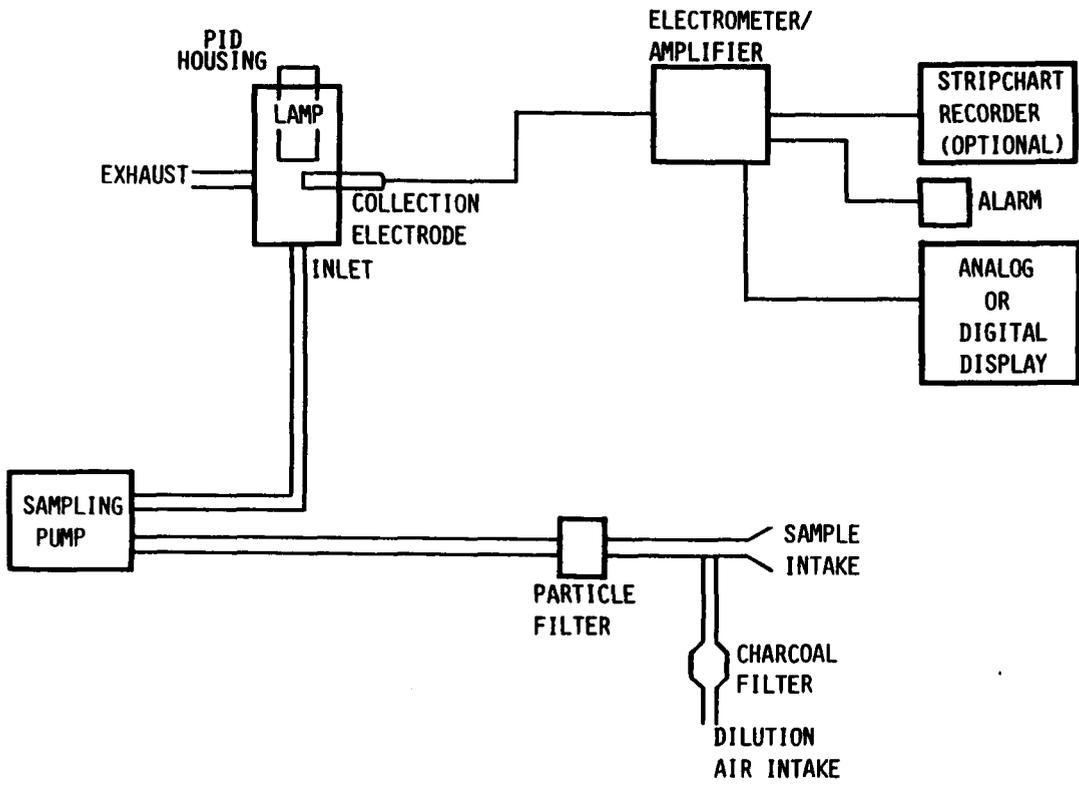
Ions are produced by an ultraviolet radiation source rather than a flame. The ions are collected at an electrode and the resultant current flow is measured with an electrometer.

**DIAGRAM OF PHOTOIONIZATION
DETECTOR (PID)**



NOTES

VOC ANALYZER USING PID DETECTOR



NOTES

PID PROBLEMS

- 2,000 ppm maximum leak detection.
- No detection of methane and ethane.
- Short-circuits.

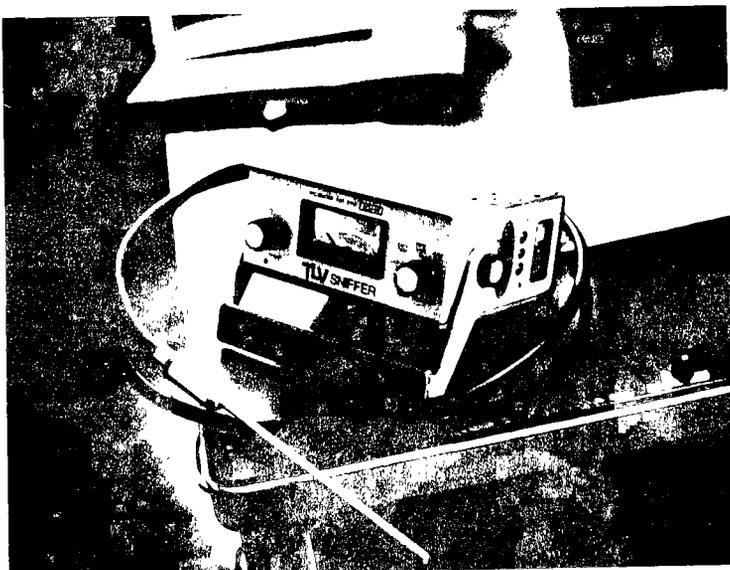
SLIDE 21-24

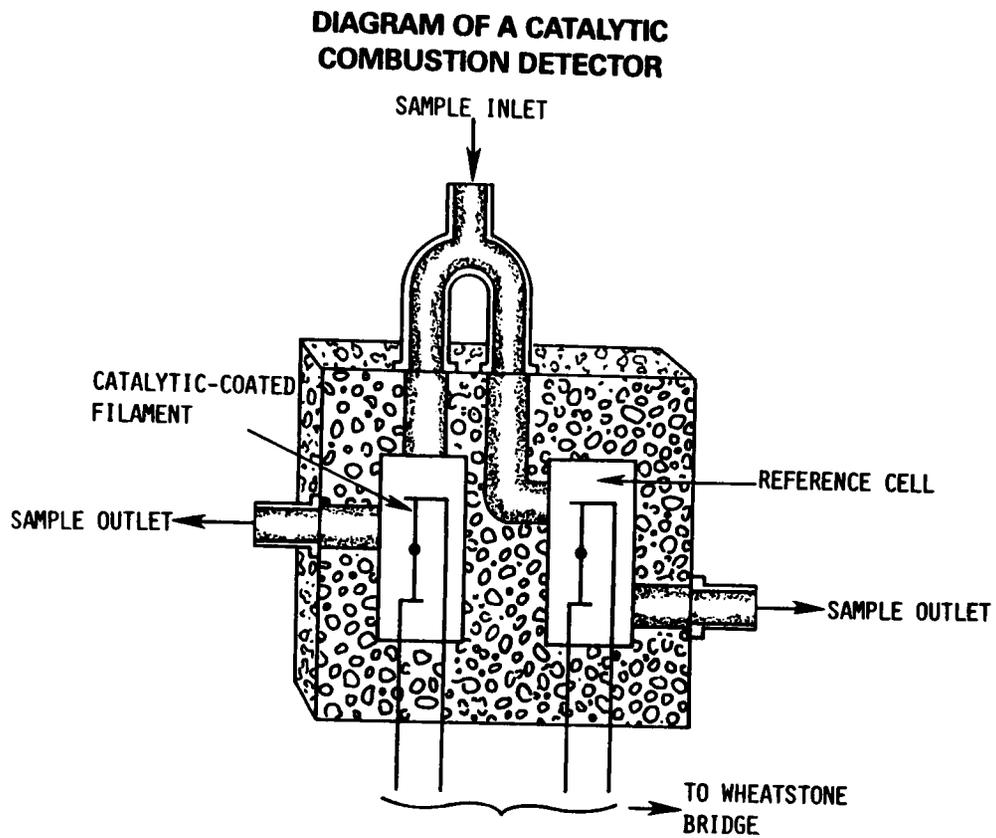
CATALYTIC COMBUSTION DETECTOR

PRINCIPLE OF OPERATION

This system uses a dual "hot-wire" cell in which one of the wires is coated with a catalyst to promote oxidation (combustion) of organic compounds present in the sample stream. The heat released during the combustion process results in a resistance change in the coated wire when compared to the reference wire. This imbalance produces a signal which is related to the VOC concentration.

SLIDE 21-25





SLIDE 21-27

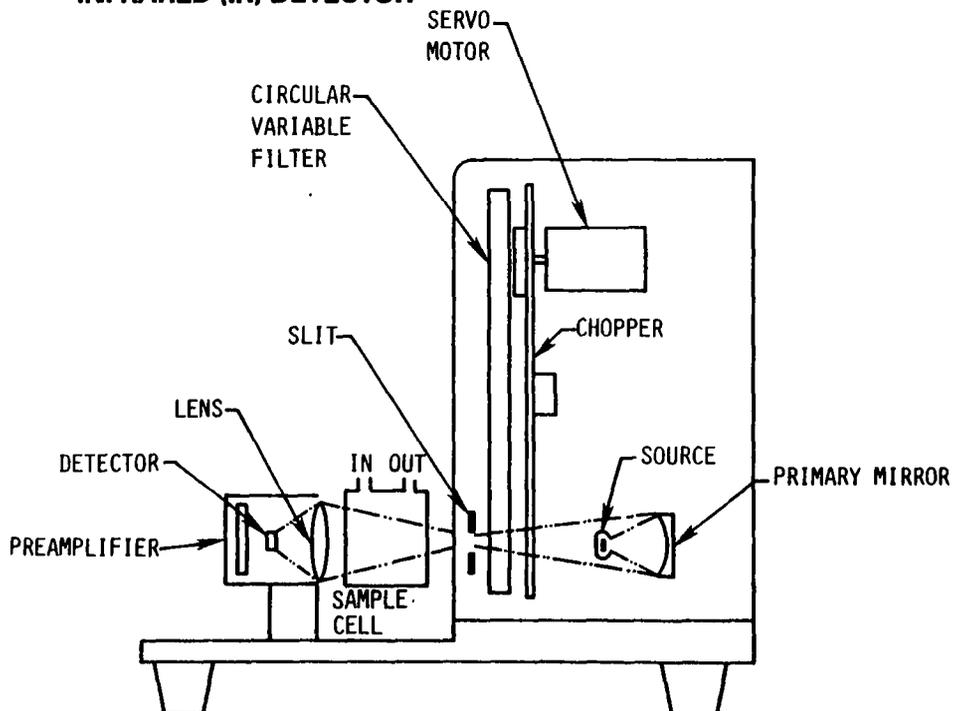
CATALYTIC COMBUSTION DEVICE PROBLEMS

- Slow response time
- Noncombustible
- Chlorinated solvents

DISPERSIVE INFRARED ADSORPTION (IR)**PRINCIPLE OF OPERATION**

A dispersive IR detector involves a direct adsorbance measurement at a particular wavelength selected by a filter or grating monochromator.

SLIDE 21-29

DIAGRAM OF DISPERSIVE INFRARED (IR) DETECTOR

IR DEVICE PROBLEMS

- Specific response
- Calibration gases

SLIDE 21-31

OTHER DETECTION SCHEMES

- Thermal conductivity
- Electron capture
- Enhancement of radiation from halogens by a spark source

PERFORMANCE COMPARISON OF FOUR MAJOR VOC ANALYZERS

	FID	PID	Catalytic Combustion	IR
1. Well detected VOC classes	Aliphatic olefinic, and aromatic hydrocarbons	Aromatic and olefinic hydrocarbons, chlorinated compounds	Similar to FID	Hydrocarbons
2. Poorly detected VOC Classes	Highly oxygenated or halogenated compounds. Sulfur, nitrogen, phosphorus containing compounds also reduced response	Aliphatic hydrocarbons	Similar to FID	Highly dependent on IR absorption spectrum. Water vapor will interfere with certain compounds
3. Typical calibration compound	Methane, propane	Benzene, butadiene	Methane, propane	Compound of interest or very similar compound
4. Typical detection range, ppmv	1-10,000	1-2000 or 10-20,000 (with dilution)	Several ranges available from 10 ppm up to lower explosion limit	1-10,000 (highly dependent on specific compound)
5. Typical calibration accuracy, %	2-10	5-10	2-10	2-10
6. Typical calibration precision, %	2-5	2-5	2-5	2-5
7. Typical response time, seconds	5-10	5-10	5-20	5-100
8. Approximate cost	\$2500-5000	\$4000-5000	\$300-2000	\$4000-7000
9. Major advantages	a) uniform response for most hydrocarbons b) some response obtained for almost all organic compounds	a) detects oxygenated and chlorinated compounds not detected by FID b) no external gas supply required	a) low cost b) relatively uniform response for most hydrocarbons c) no external gas supply required	a) qualitative information may be obtained b) no external gas supply required
10. Major limitations	a) poor response for highly oxygenated or chlorinated compounds b) external gas supply (hydrogen) required	a) does not respond to aliphatic hydrocarbons b) detection of high levels may require dilution of the sample stream	a) poor response for highly oxygenated or chlorinated compounds b) not as sensitive as the other technique	a) water vapor and other atmospheric constituents may interfere b) sensitivity highly dependent on the specific compound of interest c) expensive

COMMERCIALLY AVAILABLE PORTABLE VOC ANALYZERS ^a

Manufacturer	Model number	Weight, lbs	Cost, \$	Detection range, ppmv	Sensitivity ppmv	Calibration precision, %	Calibration accuracy, %	Response time, seconds	Nominal sampling rate, F/min	Comments
FID										
Analytical Inst. Development, Inc., Avondale, PA	712	14	\$4,300	1-2000 or 10-20,000	0.1 (as methane)	--	--	5	1.5	Optional GC available
Foxboro Analytical, S. Norwalk, CT	OVA-108	12	\$4,200	0-10,000	0.5 (as methane)	+2	2	2	2.0	Optional GC available
Health Consultants, Inc., Stroughton, MA	Detecto-Pak II	8	\$2,950	0-1,000	5 (as methane)	--	4	15	--	
Survey and Analysis, Inc., Northboro, MA	A-500	17	\$2,295	0-10,000	2 (as methane)	--	20	4	--	
PID										
Analytical Inst. Development, Avondale, PA	585	8.2	\$4,200	0-1,000	1 (as benzene)	--	--	2	0.5	10.0 and 118 eV lamps available
HNU Systems, Newton, MA	PI-101	12	\$4,000	0-2,000	1 (as benzene)	--	--	5	0.5	9.5, 10.2, 10.9 and 11.7 eV lamps available

COMMERCIALLY AVAILABLE PORTABLE VOC ANALYZERS (Continued)

Manufacturer	Model number	Weight, lbs	Cost, \$	Detection range, ppmv	Sensitivity ppmv	Calibration precision, %	Calibration accuracy, %	Response time, seconds	Nominal sampling rate, </min	Comments
CATALYTIC COMBUSTION										
Bacharach Inst. Co., Santa Clara, CA	TLV Sniffer	5	\$900	0-10,000	2 (as methane)	+3	3	--	--	
Biomarine Industries, Inc., Malvern, PA	922	1.5	\$500	0-100%LEL ^b	--	--	--	5	--	
Gas Tech, Inc., Mountain View, CA	1177	6	\$500	0-100%LEL or 0-500 ppm	--	+5	2	4	--	
Mine Safety Appliances Co., Pittsburgh, PA	260	6	\$800	0-100%LEL	--	--	--	5	--	
Survey and Analysis, Inc., Northboro, MA	Onmark Model 5	5	\$300	0-5%LEL	--	+3	3	10	--	
INFRARED DEVICES (DISPERSIVE)										
Foxboro Analytical, S. Norwalk, CT	Miran-1A	32	\$7,000	ppm-% level	1 (highly dependent on compound of interest)	+2	3-4	Selectable 1-40	--	

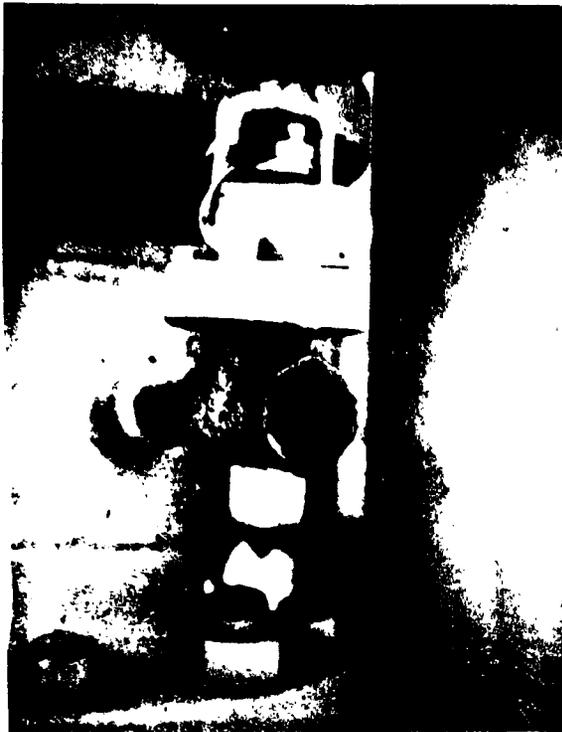
^aThis list is not comprehensive. Available information may no longer be accurate in certain cases.

^bLEL lower explosion limit. Usually about 10,000 ppm.

PORTABLE VOC DETECTOR INSTRUMENT CERTIFICATION

Manufacturer	Model No.	Certification
Analytical Instrument Development, Co., Avondale, Pennsylvania	712	Intrinsically safe, Class 1, Division 1, Groups A, B, C, and D
Bacharach Instrument Co., Santa Barbara, CA	L	Intrinsically safe, Class 1, Division 1, Groups C and D
	TLV Sniffer	Intrinsically safe, Class 1, Division 1, Groups A, B, C, and D, and Class 1, Division 2, Groups A and B
Century Systems, Arkansas City, Kansas	OVA-128	Intrinsically safe, Class 1, Division 1, Groups A, B, C, and D
	OVA-108	Intrinsically safe, Class 1, Division 1, Groups A, B, C, and D
HNU Systems, Inc., Newton Upper Falls, Massachusetts	PI-101	Intrinsically safe, Class 1, Division 2, Groups A, B, C, and D
Mine Safety Appliances Co., Pittsburgh, Pennsylvania	40	Intrinsically safe, Class 1, Division 1, Group D, and Class 1, Division 2, Groups A, B, and C
Survey and Analysis, Inc., Northboro, Massachusetts	OnMark Model 5	Intrinsically safe, Class 1, Division 1, Groups A, B, C, and D

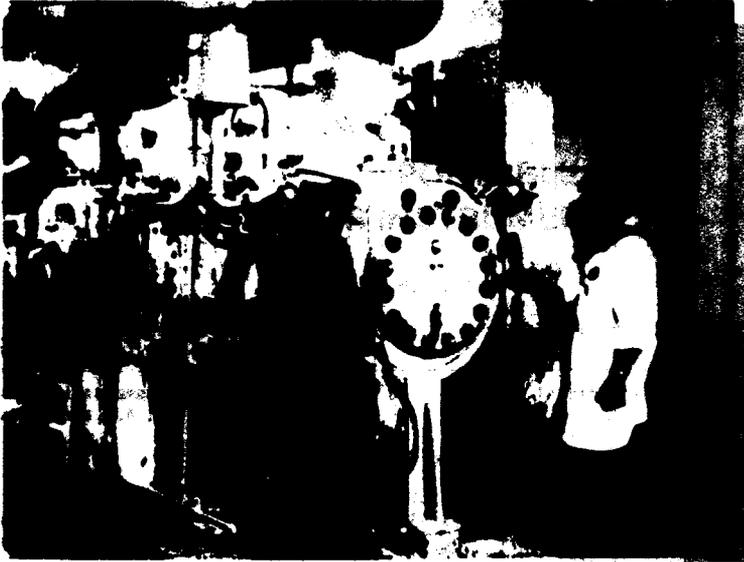
NOTES



SLIDE 21-37



SLIDE 21-38



SLIDE 21-39



P-18



SLIDE 21-41

INSTRUMENT SUITABILITY

REFINERIES

- FID
- Catalytic combustion

SOCMI

- FID
- PID
- IR
- Catalytic combustion

SLIDE 21-42

NOTES

FIELD PROBLEMS

- Moisture
- Calibration gases
- Durability

SLIDES FOR EXTENDED LECTURE

SLIDE 21-43

NOTES

GENERAL GUIDELINES FOR SYSTEM OPERATION USING A FID ANALYZER

SLIDE 21-44

FID ASSEMBLY/START-UP

1. Install probe.
2. Check fuel supply.
3. Test battery.
4. Turn on electronics and set instrument zero.
5. Check installation of flame arrestors.

(cont.)

6. Start sampling pump and check probe leakage.
7. Start hydrogen flow, leak check, and ignite flame.
8. Null meter output and set alarm levels.
9. Calibrate daily.

SLIDE 21-46

FID OPERATION

1. Place sample probe at source.
2. Periodically recheck zero setting.
3. Avoid monitoring obvious leaks.
4. Periodically check flow rate and fuel supply and perform maintenance as required.

SLIDE 21-47

FID SHUTDOWN

1. Close fuel supply valve and tank valve.
2. Shut down electronics except for sample pump.
3. Wait 10 to 20 seconds and shutdown sampling pump.

FID CALIBRATION

1. Set instrument zero with flame off.
2. Ignite flame and allow to stabilize.
3. Set zero.
4. Introduce known concentration of calibration compound near leak definition limit and observe reading.

Note: If necessary, adjust calibration knob to read known concentration and repeat zero and calibration determinations until further adjustment is not required.

SLIDE 21-49

FID ROUTINE MAINTENANCE

1. Replenish fuel supply.
2. Check/recharge batteries.
3. Replace/clean particle filter.
4. Replace charcoal filter.
5. Clean/replace flame arrestors.
6. Leak check flow system/gas supply lines.

SLIDE 21-50

FID QUALITY CONTROL/PERFORMANCE VERIFICATION PROCEDURES

1. Check background (zero) air reading several times per day and after each high level measurement.
2. Check calibration daily.
3. Check air flow daily.
4. Leak check air flow system and fuel line daily.

(cont.)

5. Check instrument response factor.
6. Check calibration precision for three replicate determinations every three months or after each use, whichever is longer.
7. Check response time before performing leak detection measurements and whenever changes are made in the air flow, detector or electronic portions of the instruments.

SLIDE 21-52

FID SAFETY CONSIDERATIONS

- User should consult manufacturer's literature.
- Primary concern is possibility for explosion.
- Operator should make sure that flame arrestors are properly installed.

SLIDE 21-53

(cont.)

- Refueling, battery charging, or replacement and similar servicing and maintenance procedures should not be performed in an area where high VOC levels might be present.
- Only VOC units which are certified as intrinsically safe for the particular facility being monitored are permitted to be used for Method 21.

SLIDE 21-54

FID PROBLEMS

- Chlorinated solvents
- Moisture

GENERAL GUIDELINES FOR SYSTEM OPERATION USING A PID ANALYZER

SLIDE 21-56

PID ASSEMBLY/START-UP

1. Install probe.
2. Test battery.

SLIDE 21-57

(cont.)

3. Turn on electronics and visually observe lamp.
4. Leak check sample line.
5. Set electronic zero and alarm levels.
6. Set zero.
7. Calibrate.

PID ROUTINE MAINTENANCE

1. Charge/replace batteries.
2. Replace/clean particle filter.
3. Replace charcoal filter.
4. Replace/clean lamp cell window.
5. Replace lamp.
6. Leak check sample flow system.

SLIDE 21-59

PID QUALITY CONTROL/PERFORMANCE VERIFICATION PROCEDURES

1. Check background (zero) air reading several times each day and after each high level measurement.
2. Check calibration daily.
3. Check air flow periodically.
4. Check instrument response factor.

SLIDE 21-60

(cont.)

5. Check calibration precision for three replicate determinations every three months or after each use, whichever is longer.
6. Check response time before performing leak detection measurements and whenever changes are made in the air flow, detection, or electronic portions of the instrument.

PID OPERATION

1. Place sample probe at source.
2. Periodically recheck zero setting.
3. Avoid direct measurement of obvious leaks to avoid gross contamination of the instrument.
4. Periodically check battery level and visually confirm lamp operation.

SLIDE 21-62

PID SHUTDOWN

1. Place instrument in standby position.
2. Shut off electronics.

SLIDE 21-63

PID CALIBRATION

1. Set electronic zero.
2. Set zero using background air.
3. Introduce known concentration of calibration compound representing approximately 80% of the leak definition.

Note: If necessary, adjust calibration control to read known concentration. Repeat zero and calibration measurements until further adjustment is not necessary.

PID SAFETY CONSIDERATIONS

- Electrical system has potential to create sparks which could initiate an explosion.
- Any maintenance or repair work must be performed away from potential sources of VOC contamination.

PID PROBLEMS

- 2,000 ppm maximum leak detection.
- No detection of methane and ethane.
- Short-circuits.

SLIDE 21-66

**GENERAL GUIDELINES FOR SYSTEM
OPERATION USING A CATALYTIC
COMBUSTION ANALYZER**

SLIDE 21-67

CCA ASSEMBLY/START-UP

1. Install probe assembly.
2. Test battery.
3. Check installation of flame arrestors.
4. Turn on electronics and allow a 5 to 10 minute warm-up period.
5. Null meter output and set alarm levels.
6. Calibrate daily.

SLIDE 21-68

CCA OPERATION

1. Place sample probe at source.
2. Periodically recheck zero setting.
3. Avoid monitoring obvious leaks.
4. Periodically check flow rate and perform maintenance as required.

CCA SHUTDOWN

1. Shut down electronics except for sample pump.
2. Wait 10 to 20 seconds and shutdown sample pump.

SLIDE 21-70

CCA CALIBRATION

1. Set instrument zero.
2. Set zero using background (charcoal filtered) or clean air supply.
3. Introduce known concentrations of calibration compound near (~80% of) leak definition limit and observe reading.

NOTE: If necessary, adjust calibration knob to read known concentration and repeat zero and calibration determinations until further adjustment is not required.

SLIDE 21-71

CCA ROUTINE MAINTENANCE

1. Replace filaments.
2. Check/recharge batteries.
3. Replace/clean particle filter.
4. Replace charcoal filter.
5. Clean/replace flame arrestor.
6. Leak check sample flow system.

CCA QUALITY CONTROL/PERFORMANCE VERIFICATION PROCEDURES

1. Check background (zero) air reading several times each day and after each high level measurement.
2. Check calibration daily.
3. Check air flow daily.
4. Leak check air flow system daily.

SLIDE 21-73

(cont.)

5. Check instrument response factor.
6. Check calibration precision for three replicate determinations every three months or after each use, whichever is longer.
7. Check response time before performing leak detection measurements and whenever changes are made in the air flow, detector or electronic portions of the instrument.

SLIDE 21-74

CCA SAFETY CONSIDERATIONS

- Possibility for explosion.
- Servicing and maintenance procedures should not be performed in an area where high VOC levels might be present.

SLIDE 21-75

CATALYTIC COMBUSTION DEVICE PROBLEMS

- Slow response time
- Noncombustible
- Chlorinated solvents

GENERAL GUIDELINES FOR SYSTEM OPERATION USING AN IR DETECTOR

SLIDE 21-77

IR ASSEMBLY/START-UP

1. Install probe.
2. Check battery.
3. Turn on electronics and allow instrument to warm-up for 10 to 15 minutes. Purge optical cell with clean, dry air or nitrogen if moisture condensation is a problem.

SLIDE 21-78

(cont. (cont.(cont.)

4. Select analytical and reference wavelengths for VOC of interest.
5. Leak check sample flow assembly.
6. Check optical system alignment.
7. Determine detector output for background (zero) air supply.
8. Calibrate daily.

SLIDE 21-79

IR OPERATION

1. Place sample probe at source to be measured.
2. Periodically recheck zero setting.
3. Avoid monitoring obvious leaks to avoid gross contamination of the instrument.

(cont.)

4. Periodically check sample flow rate and perform maintenance as required.
5. Perform measurements at alternate wavelengths as appropriate to confirm identity of the VOC being monitored.

SLIDE 21-81

IR SHUTDOWN

1. Shut down electronics.

SLIDE 21-82

IR CALIBRATION

1. Set zero using background (charcoal filtered) or zero air supply.
2. Introduce known concentration of calibration compound near leak definition limit and observe reading.
3. Repeat process for each analytical wavelength of interest.

NOTE: If necessary, adjust calibration output to read known concentration and repeat zero and calibration determinations until further adjustment is not required.

IR ROUTINE MAINTENANCE

1. Flush/clean optical cell.
2. Check/recharge batteries.
3. Replace/clean particle filter.
4. Replace charcoal filter.
5. Leak check sample flow system.

SLIDE 21-84

IR QUALITY CONTROL/PERFORMANCE VERIFICATION PROCEDURES

1. Check background (zero) air reading several times each day and after each high level measurement.
2. Check calibration daily.
3. Check air flow daily.
4. Leak check air flow system daily.
5. Check instrument response factor for regulated compound (relative to reference compound) before performing measurements.
6. Check calibration precision for three replicate determinations every three months or after each use, whichever is longer.
7. Check response time before performing leak detection measurements and whenever changes are made in the air flow, detector, or electronic portions of the instrument.

SLIDE 21-85

IR DEVICE PROBLEMS

- Specific response
- Calibration gases

METHOD 23

Determination of Halogenated Organics from Stationary Sources

SLIDE 23-2

APPLICABILITY

Applies to measurement of halogenated organics such as:

- carbon tetrachloride
- ethylene dichloride
- perchloroethylene
- trichloroethylene
- methylene chloride
- 1, 1, 1-trichloroethane
- trichlorotrifluoroethane

Note: Does not measure halogenated organics contained in particulate matter.

SLIDE 23-3

PRINCIPLE

An integrated bag sample of stack gas containing one or more halogenated organics is subjected to gas chromatographic analysis, using a flame ionization detector.

INTERFERENCES

A suitable gas chromatograph column must be used to provide adequate resolution of the halogenated compounds.

SLIDE 23-5

APPARATUS

SAMPLING—Integrated Bag Sampling Train

ANALYSIS—Gas Chromatograph with FID, potentiometric strip chart recorder, 1.0 to 2.0 ml sampling loop, and a stainless steel 3.05 m by 3.2 mm column containing 20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport.

Note: Adequate peak resolution is defined as an overlap of not more than 10% of the halogenated organic compound.

SLIDE 23-6

SAMPLING

Use standard integrated bag sampling technique. The sample must be analyzed within 1 day for:

- Methylene Chloride
- Ethylene Dichloride
- Trichlorotrifluoroethane

within 2 days for:

- Perchloroethylene
- Trichloroethylene
- 1,1,1-Trichloroethane
- Carbon Tetrachloride

ANALYSIS

SAMPLE ANALYSIS—Directly from bag to sample loop.
CALIBRATION STANDARDS—Three certified gas cylinders or
 prepare standards from 99 Mol.
 % halogenated organic
 compound.

SLIDE 23-8

INJECTION VALUES FOR PREPARATION OF STANDARDS

Compound	Molecular weight, g/g-mole	Density at 293 K, g/ml	$\mu\text{l}/50$ liter of N_2 required for approximate concentration of:		
			200 ppm	100 ppm	50 ppm
Perchloroethylene, C_2Cl_4	165.85	1.6230	42.5	21.2	10.6
Trichloroethylene, C_2HCl_3	131.40	1.4649	37.3	18.6	9.3
1,1,1-Trichloroethane, $\text{C}_2\text{H}_3\text{Cl}_3$	133.42	1.4384	38.6	19.3	9.6
Methylene Chloride, CH_2Cl_2	84.94	1.3255	26.6	13.3	6.7
Trichlorotrifluoroethane, $\text{C}_2\text{Cl}_3\text{F}_3$	187.38	1.5790	49.3	24.7	12.3
Carbon Tetrachloride, CCl_4	153.84	1.5940	40.1	20.1	10.0
Ethylene Dichloride, $\text{C}_2\text{H}_4\text{Cl}_2$	98.96	1.2569	32.7	16.4	8.2

SLIDE 23-9

QUALITY ASSURANCE

- Sample bags must pass leak check.
- Analysis audit according to Appendix E,
Supplement B.

**HALOGENATED ORGANIC
STANDARD CONCENTRATION**

$$C_c = \frac{BD}{M} \frac{(24.055 \times 10^3)}{V_m Y \frac{293}{T_m} \frac{P_m}{760}}$$

$$= 6.240 \times 10^4 \frac{BD T_m}{M V_m Y P_m}$$

Where:

B = volume of halogenated organic injected, μl .

D = density of compound at 293°K , g/ml .

M = molecular weight of compound, g/g-mole .

V_m = gas volume measured by dry gas meter, liters.

Y = dry gas meter calibration factor, dimensionless.

P_m = absolute pressure of dry gas meter, mm Hg.

T_m = absolute temperature of dry gas meter, $^\circ\text{K}$.

24.055 = ideal gas molal volume at 293°K and 760 mm Hg, liters/g-mole.

10^3 = conversion factor. $[(\text{ppm})(\text{ml})]/\mu\text{l}$.

SAMPLE CONCENTRATIONS

$$C_s = \frac{C_c P_r T_i}{P_i T_r (1 - S_{wb})}$$

Where:

C_c = concentration of the halogenated organic indicated by the gas chromatograph, ppm.

P_r = reference pressure, the laboratory pressure recorded during calibration, mm Hg.

T_i = sample loop temperature at the time of analysis, °K.

P_i = laboratory pressure at time of analysis, mm Hg.

T_r = reference temperature, the sample loop temperature recorded during calibration, °K.

S_{wb} = water vapor content of the bag sample, volume fraction.

METHOD 24

**Determination of Volatile Matter Content,
Water Content, Density, Volume Solids,
and Weight Solids of Surface Coatings**

SLIDE 24-2

APPLICABILITY

For volatile organic content of paints used in auto, appliance, metal furniture, metal coil coating, and other industries. Can be used for both water-borne and solvent-borne coatings.

SLIDE 24-3

ADVANTAGE

- Less costly.

DISADVANTAGE

- Considerable error may be introduced in measurement of organic content of water-borne coatings since this is an indirect measurement technique.

NOT APPLICABLE

1. For all kinds of coatings or printing materials. Method 24A should be used for printing inks. No method has been specified for glues and adhesives.
2. For two package (component) coatings, particularly if the coatings react during curing and form volatile reaction products.
3. For coatings that require energy other than heat to initiate curing. May not work on coatings that require high temperature catalysis for curing.

SLIDE 24-5

SUMMARY OF METHODS

WATER CONTENT

Standard Method of Test for Water in Water-Reducible Paint by Direct Injection Into a Gas Chromatograph. ASTM D 3792-79.

OR

ASTM Provisional Method of Test for Water in Paint or Related Coatings by the Karl Fischer Titration Method.

SLIDE 24-6

SUMMARY OF METHODS (continued)

VOLATILE MATTER

Provisional Method of Test for Volatile Content of Paints. ASTM D 2369-81.

DENSITY

Standard Method of Test for Density of Paint, Lacquer, and Related Products. ASTM D 1475-60.

DATA VALIDATION PROCEDURE

Run duplicate analyses on each sample tested and compare results with the within-laboratory precision statements for each parameter.

SLIDE 24-8

ANALYTICAL PRECISION STATEMENTS

PARAMETER	WITHIN- LABORATORY	BETWEEN- LABORATORY
Volatile Matter Content, W_v	1.5% \bar{W}_v	4.7% \bar{W}_v
Water Content, W_w	2.9% \bar{W}_w	7.5% \bar{W}_w
Density, D_c	0.001 kg/liter	0.002 kg/liter

CALCULATIONS**NONAQUEOUS VOLATILE MATTER**

Solvent-borne Coatings

$$W_o = W_v$$

Waterborne Coatings

$$W_o = W_v - W_w$$

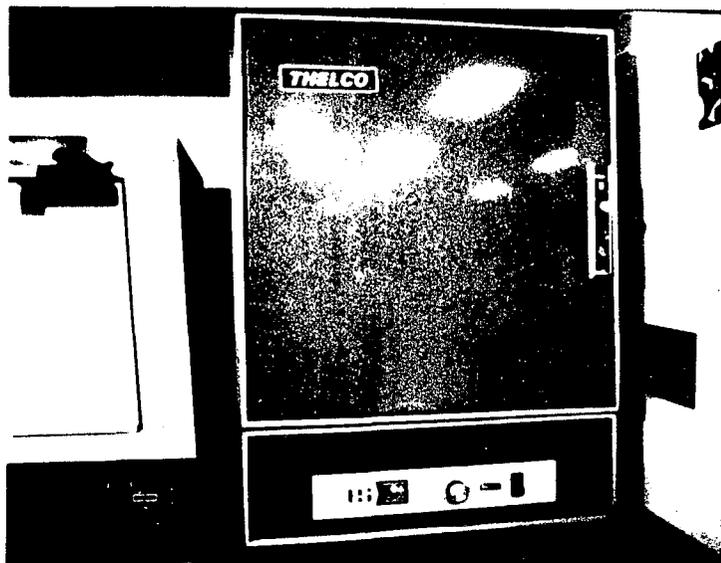
WEIGHT FRACTION SOLIDS

$$W_s = 1 - W_v$$

Where:

 W_o = weight fraction nonaqueous volatile matter, g/g. W_v = volatile matter content. W_w = water content. W_s = weight solids, g/g.

SLIDE 24-10



METHOD 24A

Determination of Volatile Matter
Content and Density of Printing Inks
and Related Coatings

SLIDE 24A-2

APPLICABILITY AND PRINCIPLE

Standard methods are used to determine components of solvent-borne printing inks or related coatings.

The VOC weight fraction is determined by measuring the weight loss of a known quantity which has been heated for a specified length of time at a specified temperature.

SLIDE 24A-3

SAMPLING

Obtain a representative sample of the ink or coating material.

SLIDE 24A-4

ANALYSIS

1. Tare three aluminum foil dishes to the nearest 0.1 mg.
2. Using a 5 ml syringe without needle remove a sample and weigh to the nearest 0.1 mg.
3. Transfer 1 to 3 g of sample to a tared weighing dish.
4. Reweigh the syringe to the nearest 0.1 mg.

(cont.)

ANALYSIS

5. Heat the weighing dish and sample in a vacuum oven at an absolute pressure of 510 ± 51 mm Hg and a temperature of $120 \pm 2^\circ\text{C}$.
6. Alternatively, heat the dish and sample in a forced draft oven at $120 \pm 2^\circ\text{C}$ for 24 hours.
7. After dish has cooled, reweigh to the nearest 0.1 mg.
8. Repeat this procedure for a total of three determinations of each sample.

SLIDE 24A-6

(cont.)

ANALYSIS

9. Determine the density of the ink or coating according to ASTM D 1475-60.
10. Determine the density of the solvent according to ASTM D 1475-60.
11. Calculate the weight fraction volatile organic content (W_o).

**WEIGHT FRACTION
VOLATILE ORGANIC CONTENT**

$$W_o = \frac{M_{x1} + M_{CY1} - M_{CY2} - M_{x2}}{M_{CY1} - M_{CY2}}$$

Where:

- W_o = weight fraction of VOC, g/g.
- M_{x1} = tare weight of aluminum dish, g.
- M_{CY1} = weight of full syringe, g.
- M_{CY2} = weight of syringe after dispensing sample, g.
- M_{x2} = final weight of dish after heating, g.

SLIDE 24A-8

**WEIGHT FRACTION
VOLATILE ORGANIC CONTENT**

$$W_o = \frac{M_{CY1} - M_{CY2} - (M_{x2} - M_{x1})}{M_{CY1} - M_{CY2}}$$

(see nomenclature above)

**VOLUME FRACTION
VOLATILE ORGANIC CONTENT**

$$V_o = \frac{W_o D_c}{D_o}$$

Where:

V_o = volume fraction, ml/ml.

W_o = weight fraction of VOC, g/g.

D_c = density of coating.

D_o = density of solvent.

METHOD 25

Determination of Total Gaseous
Nonmethane Organic Emissions
as Carbon

SLIDE 25-2

APPLICABILITY

For the measurement of volatile organic compounds (VOC) as total gaseous nonmethane organics (TGNMO) as carbon in source emissions.

SLIDE 25-3

APPLICABILITY

For measuring control efficiency from coating operations including auto, appliance, metal furniture, and metal coil coating.

NOT APPLICABLE

1. For measuring concentrations of VOC or mass emissions of VOC from sources whose concentrations are < 50 ppm (as C_1).
2. For measuring emissions from sources whose principle solvents are chlorinated hydrocarbons.
3. Generally, for any situation where a simpler procedure is more accurate.

PRINCIPLE

A gas sample is withdrawn from the stack at a constant rate through a chilled condensate trap by means of an evacuated sample tank.

TGNMO are determined by combining the analytical results obtained from independent analyses of the condensate trap and sample tank fractions.

INTERFERENCE

Organic particulate matter will interfere with the analysis; therefore, an in-stack particulate filter may be required.

ADVANTAGES

1. Gives consistent results from source to source whether sample composition is known or not.
2. Sample train requires no electricity at sample site (minimizes explosion hazard).

DISADVANTAGES

1. Will not yield true mass emission rate nor instantaneous results.
2. No real time data (sample must be returned to lab).
3. High moisture and CO₂ together can cause interferences.

RM 25-SUMMARY

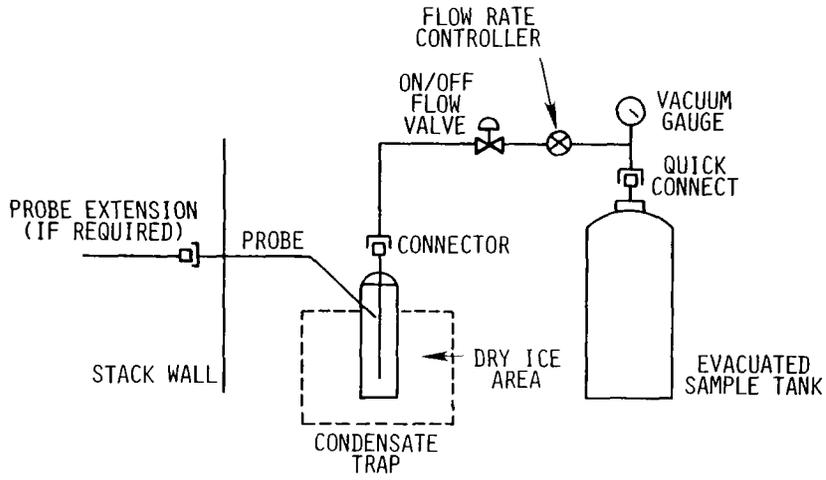
- Withdraw emission sample from stack through chilled condensate trap into evacuated cylinder.
- Analyze contents of trap and cylinder separately.
- Oxidize organic content of trap to CO₂, reduce to methane, measure with FID.
- Inject portion of cylinder sample into GC to separate nonmethane organics, oxidize NMO to CO₂, reduce to methane, measure with FID.
- Combine results and report as total gaseous nonmethane organics.

SLIDE 25-8

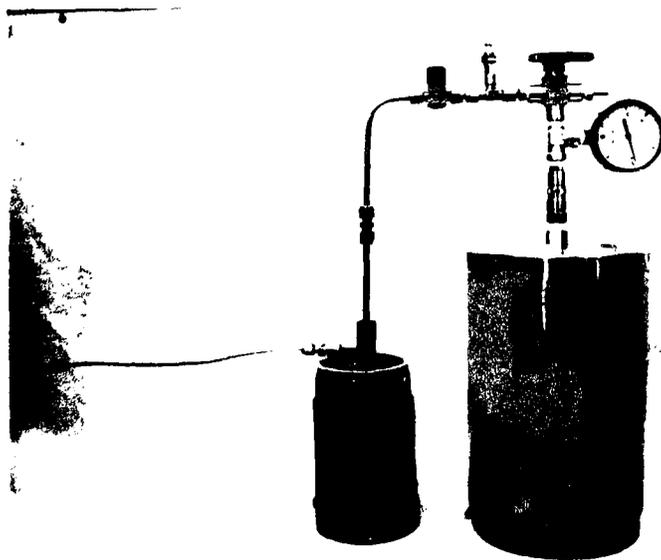
APPARATUS**SAMPLING SYSTEM:**

- Probe
- Condensate Trap
- Flow Control System
- Sample Tank

SAMPLING SYSTEM



SLIDE 25-10

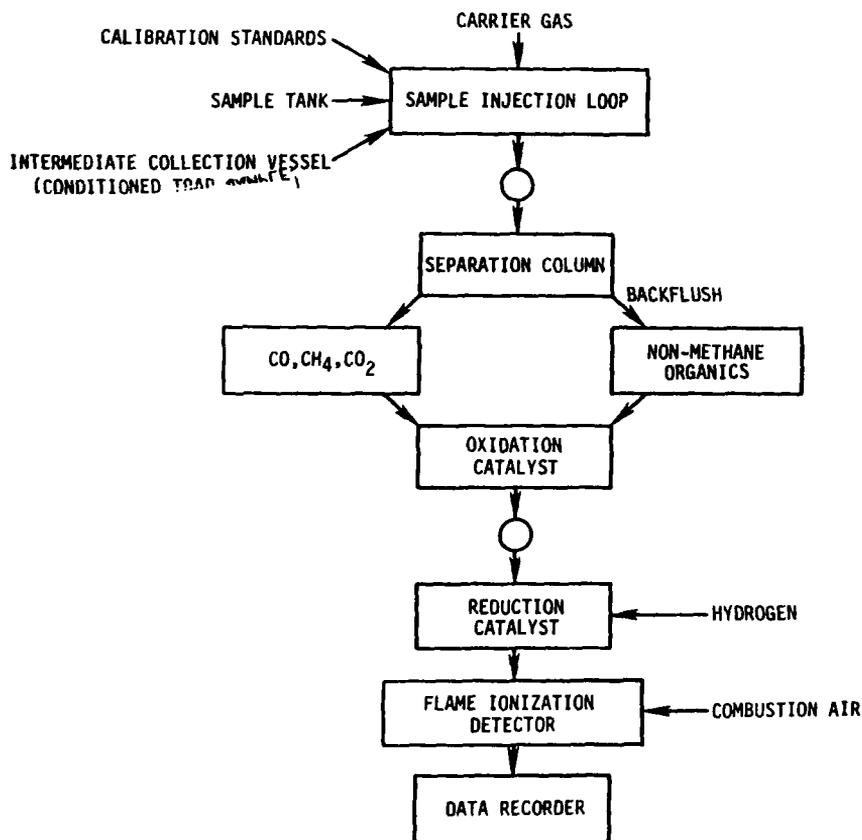


ANALYTICAL SYSTEM

- Oxidation system for recovery and conditioning of condensate trap contents
 1. Heat source
 2. Oxidation catalyst
 3. Nondispersive infrared analyzer
 4. Intermediate collection vessel
- NMO Analyzer
 1. GC with backflush capabilities
 2. Oxidation catalyst
 3. Reduction catalyst
 4. FID

SLIDE 25-12

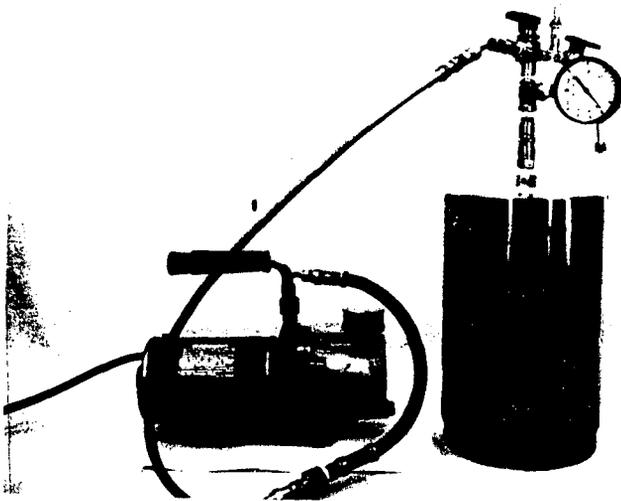
SCHEMATIC OF NONMETHANE ORGANIC (NMO) ANALYZER



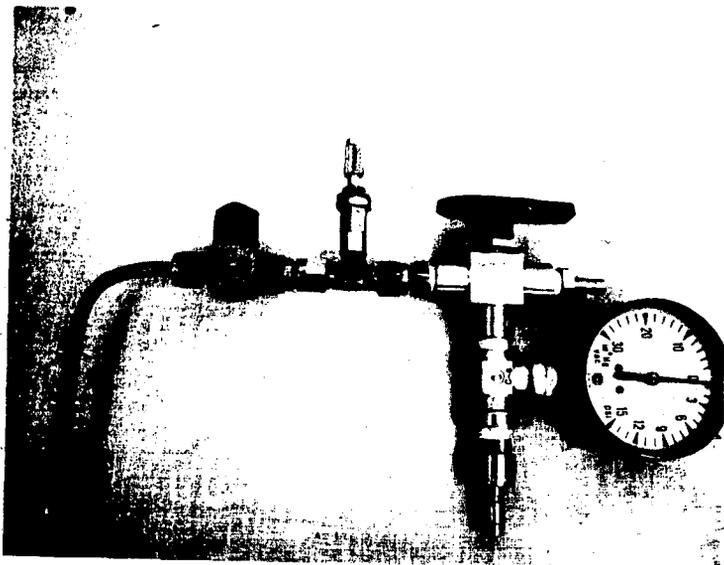
SAMPLING

- Sample Tank Evacuation and Leak Check
- Sample Train Assembly
- Pretest Leak Check
- Sample Train Operation
- Post Test Leak Check

SLIDE 25-14

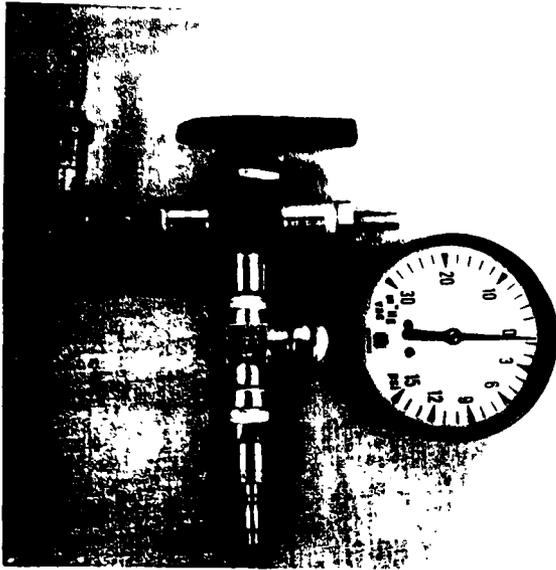


SLIDE 25-15

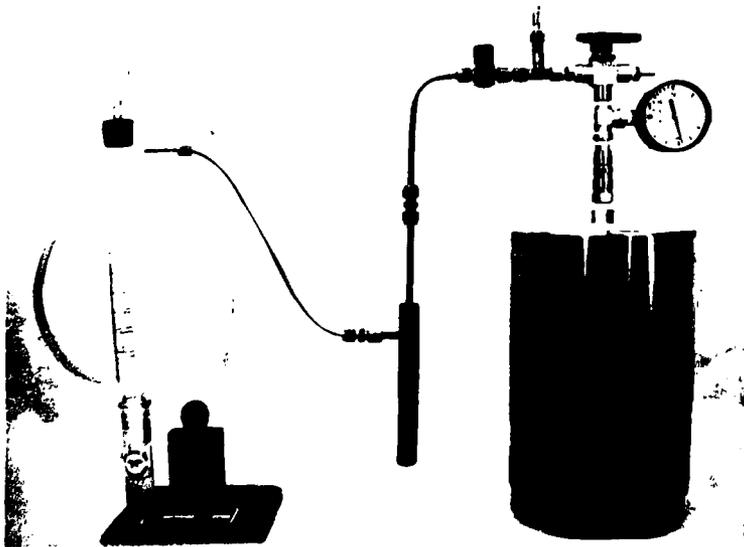


SLIDE 25-16

NOTES

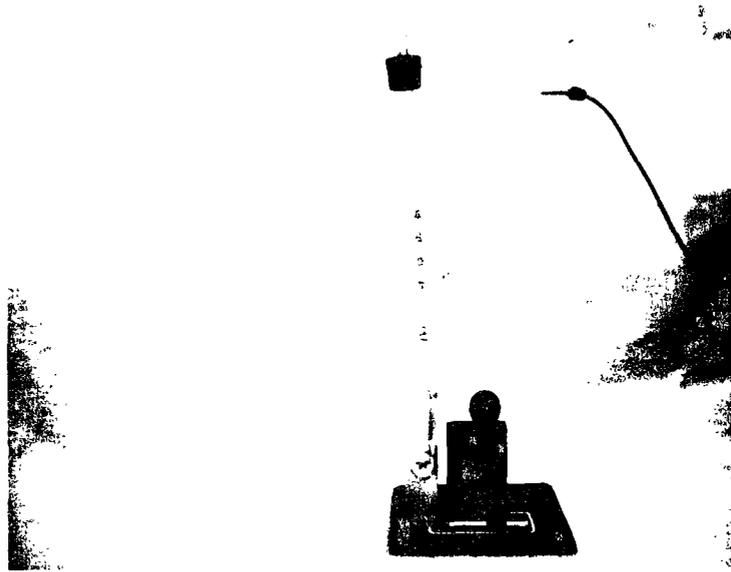


SLIDE 25-17



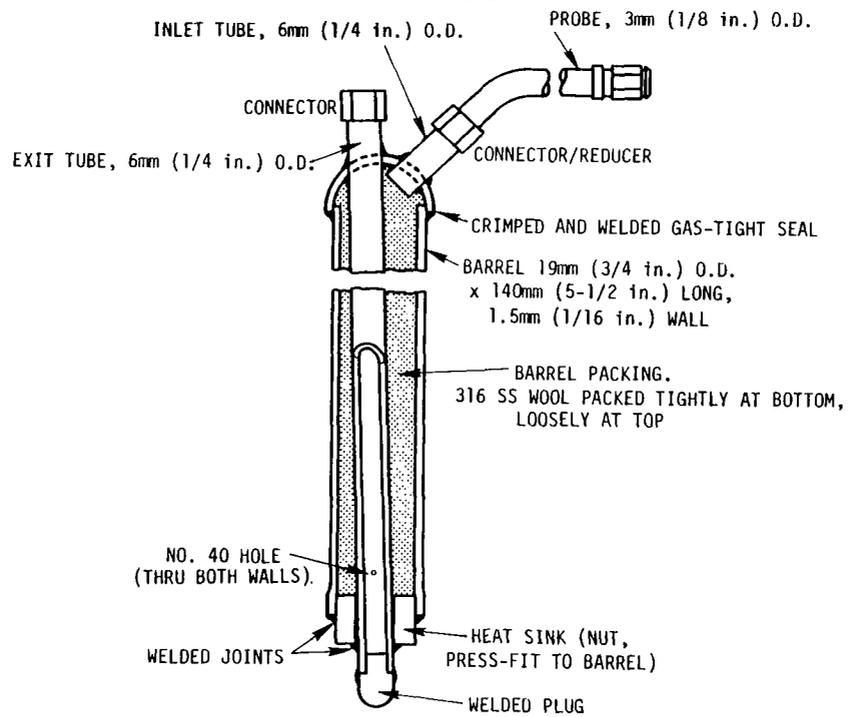
T-7

SLIDE 25-18



SLIDE 25-19

CONDENSATE TRAP



MATERIAL: TYPE 316 STAINLESS STEEL

SAMPLE RECOVERY

1. Disconnect condensate trap and seal both ends.
2. Keep trap in dry ice until returned to lab for analysis.
3. Remove flow metering system from sample tank.
4. Attach manometer and record final tank vacuum.
5. Record tank temperature and barometric pressure.
6. Disconnect manometer.
7. Properly identify condensate trap and sample tank(s).

SLIDE 25-21

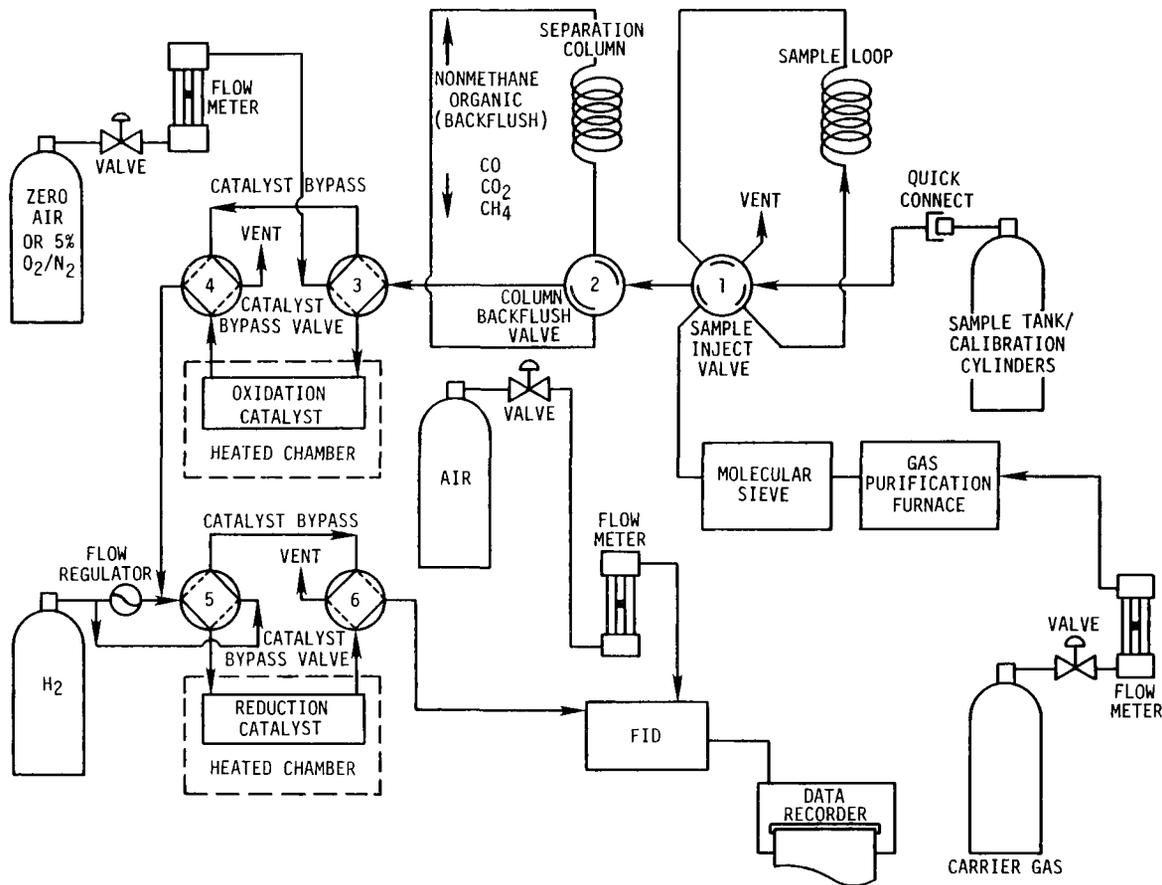


SLIDE 25-22

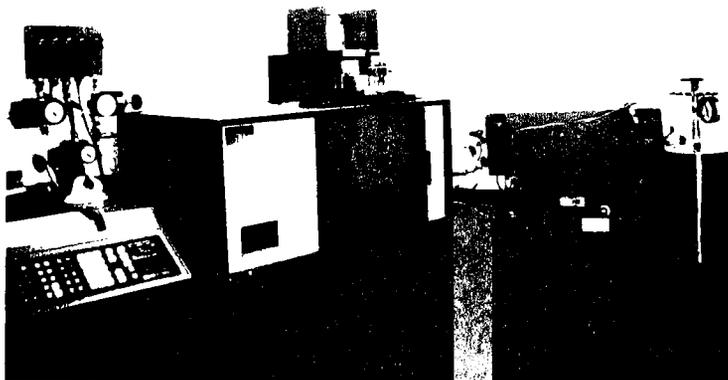
ANALYSIS

- Initial Performance Test
- Daily Operations and Calibration Checks
- Analysis of Recovered Condensate Sample
- Analysis of Sample Tank

NONMETHANE ORGANIC (NMO) ANALYZER



SLIDE 25-24



INITIAL PERFORMANCE CHECK OF CONDENSATE RECOVERY AND CONDITIONING APPARATUS

- Carrier Gas and Auxiliary Oxygen Blank
- Catalyst Efficiency Check
- System Performance Check

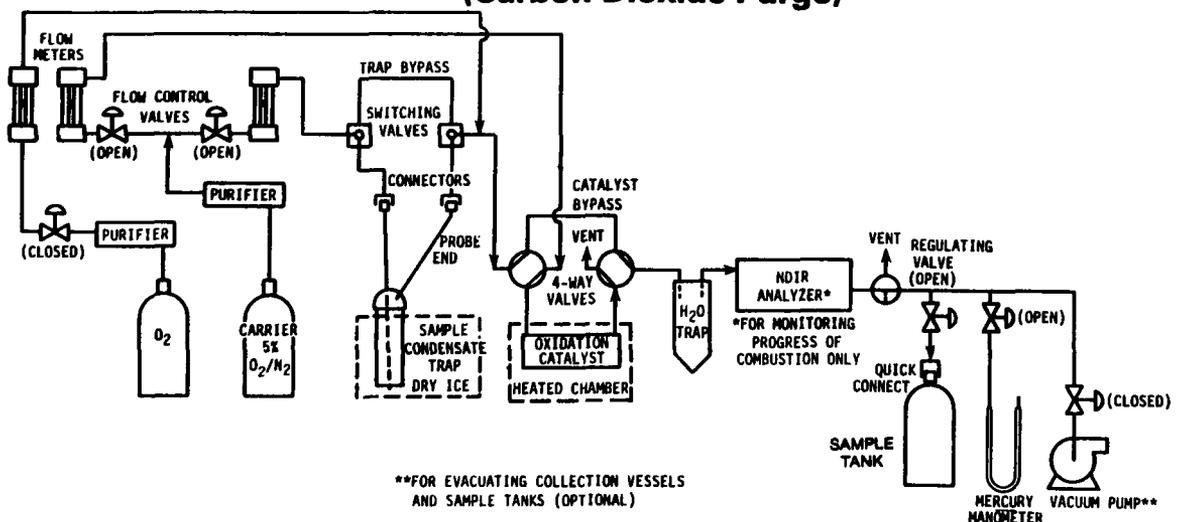
SLIDE 25-26

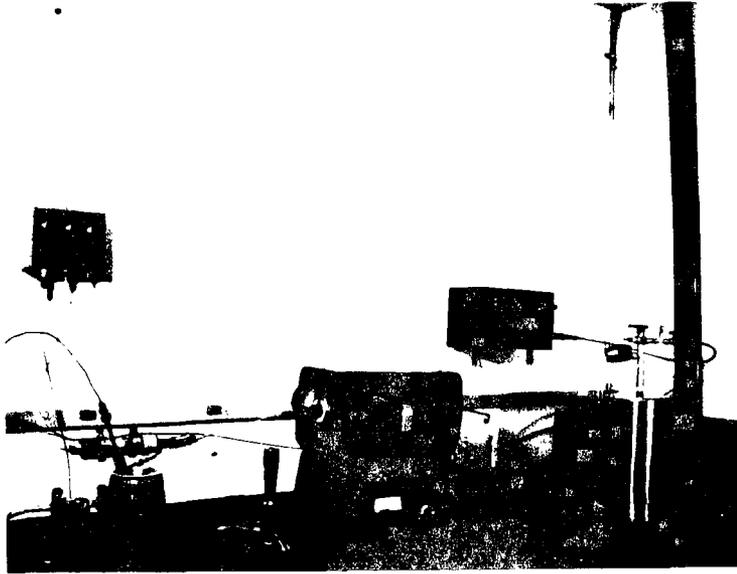
CONDENSATE RECOVERY AND CONDITIONING

- System Blank and Catalyst Efficiency Check
- Condensate Trap Carbon Dioxide Purge and Sample Tank Pressurization
- Recovery of Condensate Trap Sample

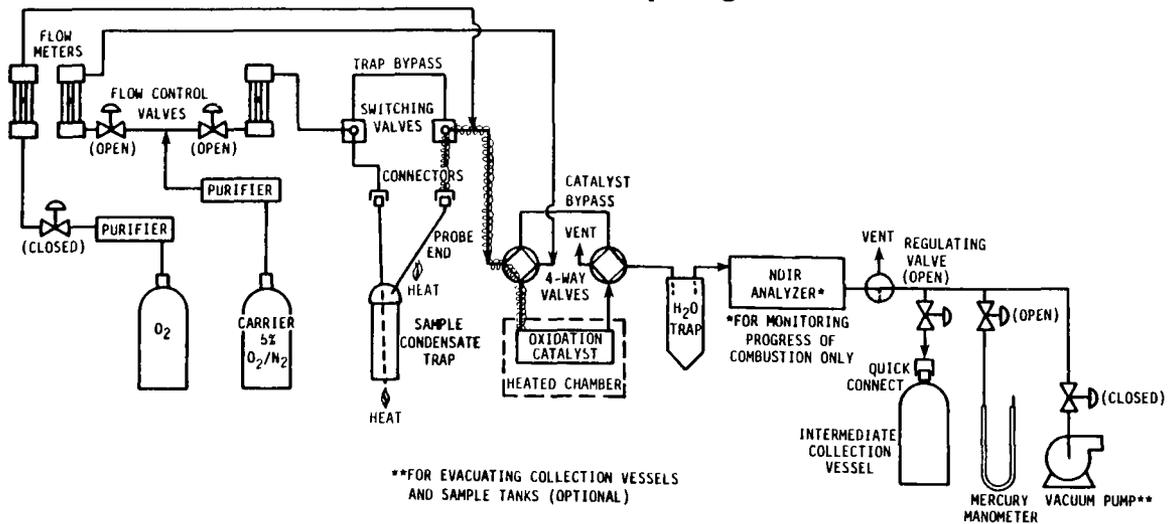
SLIDE 25-27

CONDENSATE RECOVERY AND CONDITIONING APPARATUS (Carbon Dioxide Purge)



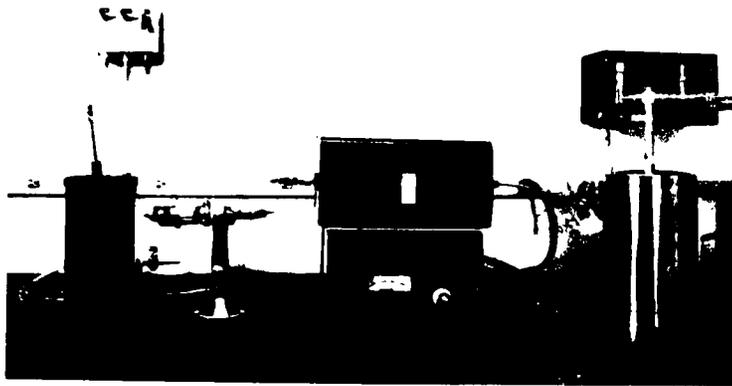


CONDENSATE RECOVERY AND CONDITIONING APPARATUS (Collection of Trap Organics)

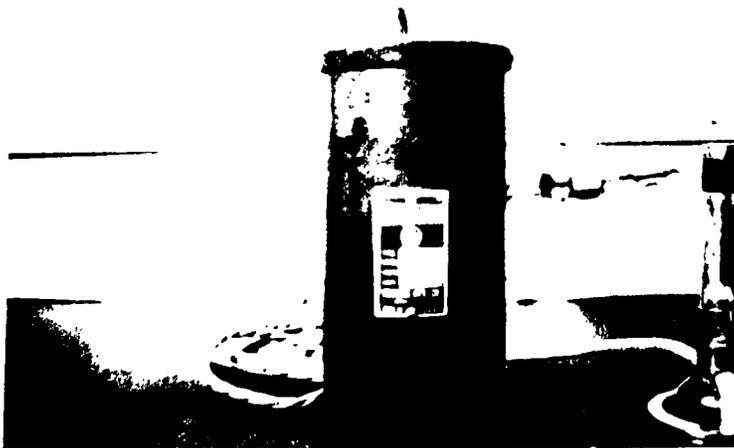


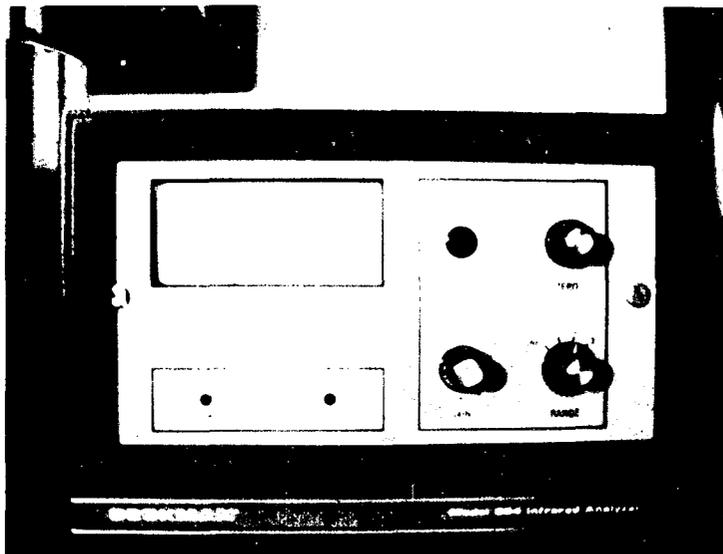
SLIDE 25-30

NOTES



SLIDE 25-31





SLIDE 25-33

INITIAL NMO ANALYZER PERFORMANCE TEST

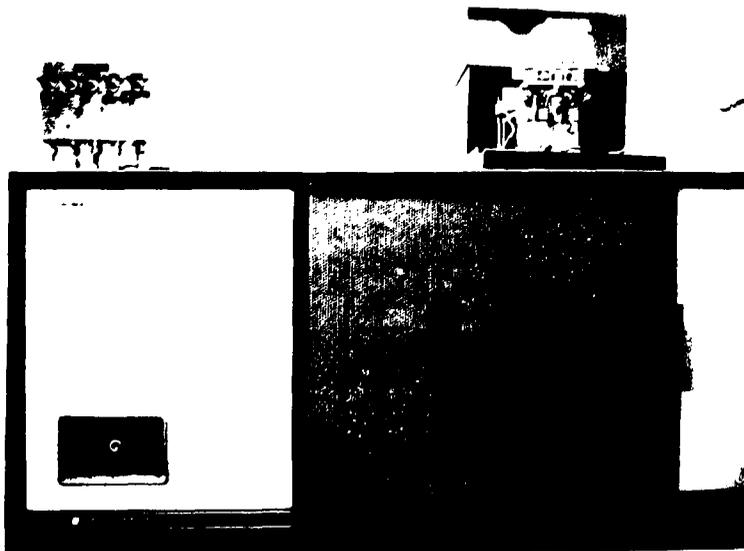
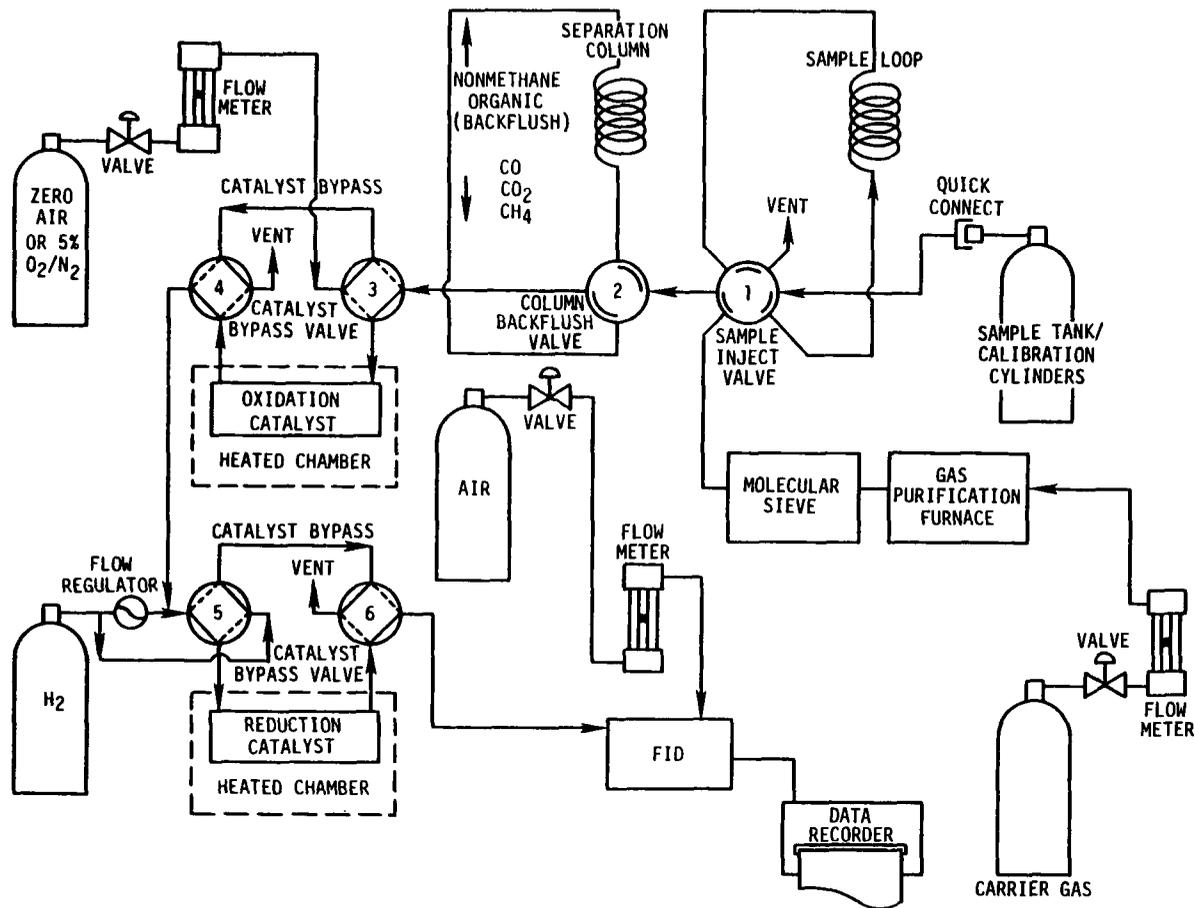
- Oxidation Catalyst Efficiency Check
- Analyzer Linearity Check and NMO Calibration
- Reduction Catalyst Efficiency Check and CO₂ Calibration.
- NMO System Blank
- System Performance Check

SLIDE 25-34

NMO ANALYZER DAILY CALIBRATION

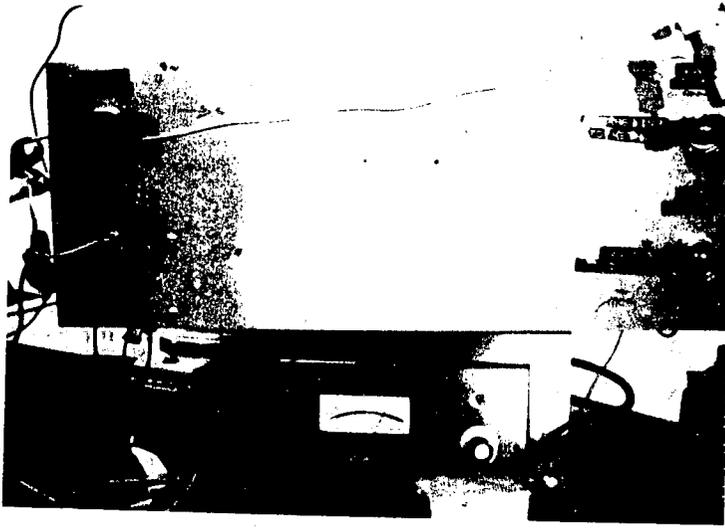
- NMO Blank and CO₂
- NMO Calibration

NONMETHANE ORGANIC (NMO) ANALYZER

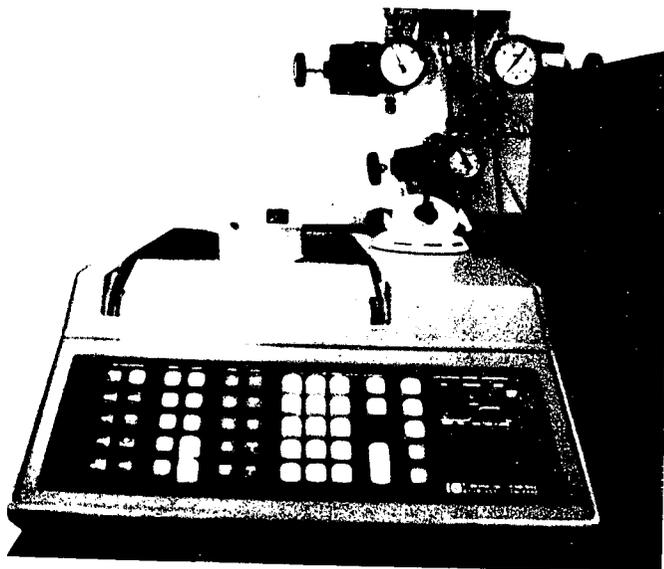


SLIDE 25-37

NOTES



SLIDE 25-38



ANALYSIS**RECOVERED CONDENSATE SAMPLE:**

Inject triplicate samples from the intermediate collection vessel and record values obtained for condensible organics as CO₂.

SLIDE 25-40

ANALYSIS (continued)**SAMPLE TANK:**

Inject triplicate samples from the sample tank and record the values obtained for nonmethane organics.

SLIDE 25-41

**SAMPLE TANK AND INTERMEDIATE COLLECTION
VESSEL VOLUME DETERMINATION-
PRIOR TO SERVICE**

- Determine volume by weighing empty, then fill with deionized water, weigh to nearest 5 gm.

OR

- Measure volume of water used to fill tank to nearest 5 ml.

CALCULATIONS

- Sample Volume
- Noncondensable Organics
- Condensible Organics
- Total Gaseous Nonmethane Organics
- Percent Recovery
- Relative Standard Deviation

SLIDE 25-43

GAS VOLUME SAMPLED

$$V_s = 0.386 \frac{^{\circ}\text{K}}{\text{mm Hg}} V \left(\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}} \right)$$

Where:

V_s = sample volume, ml.

$$0.386 = \frac{293^{\circ}\text{K}}{760 \text{ mm Hg}}$$

V = sample tank volume, ml.

P_t = gas sample tank pressure after sampling, but prior to pressurizing, mm Hg absolute.

T_t = gas sample tank temperature after sampling, but prior to pressurizing, $^{\circ}\text{K}$.

P_{ti} = sample tank pressure prior to sampling, mm Hg absolute.

T_{ti} = sample tank temperature prior to sampling, $^{\circ}\text{K}$.

SOURCE CONCENTRATION
NONCONDENSIBLE ORGANICS

$$C_t = \frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}}} \times C_{tm}$$

Where:

C_t = calculated noncondensable organic concentration (sample tank) of effluent, ppm C equivalent.

P_{tf} = final gas sample tank pressure after pressurizing, mm Hg absolute.

T_{tf} = final gas sample tank temperature after pressurizing, $^{\circ}\text{K}$.

C_{tm} = measured concentration (NMO analyzer) for the sample tank, ppm NMO.

SLIDE 25-45

SOURCE CONCENTRATIONS
CONDENSIBLE ORGANICS

$$C_c = 0.386 \frac{V_v \times P_f}{V_s \times T_t} \times C_{cm}$$

Where:

C_c = calculated condensible organic (condensate trap).

V_v = intermediate collection vessel volume, cm.

P_f = final pressure of the intermediate collection vessel, mm Hg absolute.

V_s = gas volume sampled, dscm.

T_t = sample tank temperature at completion of sampling, $^{\circ}\text{K}$.

**TOTAL GASEOUS NONMETHANE
ORGANIC CONCENTRATION**

$$C = C_t + C_c$$

Where:

C = total gaseous nonmethane organic concentration of the effluent, ppm C equivalent.

C_t = calculated noncondensable organic concentration (sample tank) of the effluent, ppm C equivalent.

C_c = calculated condensable organic (condensate trap).

SLIDE 25-47

**TOTAL GASEOUS NONMETHANE ORGANIC
MASS CONCENTRATION**

$$M_c = 0.498 C$$

Where:

M_c = total gaseous nonmethane organic (TGNMO) mass concentration of the effluent.

$$0.498 = \frac{12}{24.15}$$

C = total gaseous nonmethane organic concentration of the effluent, ppm C equivalent.

PERFORMANCE OF CONDENSATE RECOVERY AND CONDITIONING SYSTEM

PERCENT RECOVERY

$$\% = 1.6 \frac{M V_v P_f C_{cm}}{L \rho T_f N}$$

RELATIVE STANDARD DEVIATION

$$RSD = \frac{100}{\bar{X}} \sqrt{\frac{\sum (X_i - \bar{X})^2}{n - 1}}$$

Where:

M = molecular weight of the liquid injected, g/g-mole.

L = volume of liquid injected, microliters.

V_v = intermediate collection vessel volume, cm.

ρ = density of liquid injected, g/cc.

P_f = final pressure of the intermediate collection vessel, mm Hg absolute.

T_f = final temperature of intermediate collection vessel, $^{\circ}K$.

C_{cm} = measured concentration (NMO analyzer) for the condensate trap (intermediate collection vessel), ppm CO_2 .

N = carbon number of the liquid compound injected (N = 7 for toluene, N = 6 for hexane).

X_i = individual measurements.

\bar{X} = mean value.

METHOD 25A

**Determination of Total Gaseous
Organic Concentration Using
Flame Ionization Analyzer**

SLIDE 25A-2

APPLICABILITY

For the measurement of total gaseous organic concentration of vapors consisting of non-methane alkanes, alkenes and/or arenes (aromatic hydrocarbons).

SLIDE 25A-3

PRINCIPLE

A gas sample is extracted from the source through a heated sample line and glass fiber filter to a flame ionization analyzer (FIA).

ADVANTAGES

1. Yields continuous measurement and real time results.
2. FIA measurement devices are commercially available.

DISADVANTAGES

1. Cannot measure true mass and is not consistent in response from point to point.
2. Requires electricity and gas cylinders at sampling site.
3. Condensable matter in sample can cause sample loss in sample lines and instrument unless adequate precautions are taken.

SLIDE 25A-5

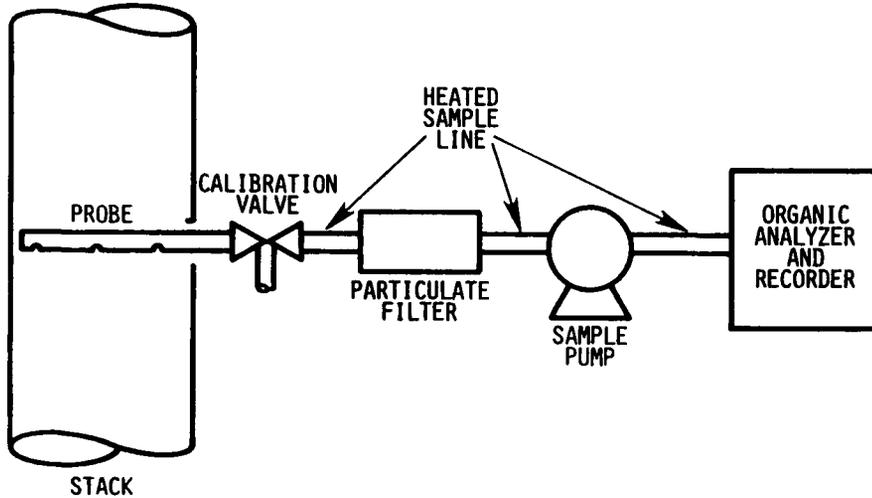
RESULTS

Results are reported as concentration equivalents of the calibration gas organic constituent, carbon, or other organic compound.

SLIDE 25A-6

METHOD 25A—SUMMARY

- Extract sample from source through heated sample line and glass fiber filter.
- Route to flame ionization analyzer.

ORGANIC CONCENTRATION MEASUREMENT SYSTEM

SLIDE 25A-8

ESSENTIAL COMPONENTS OF MEASUREMENT SYSTEM

- Organic Concentration Analyzer
- Sample Probe (three-hole rake type)
- Sample Line
- Calibration Valve Assembly
- Particulate Filter
- Recorder

GASES

CALIBRATION:

Usually consist of propane in air or nitrogen and are determined in terms of the span value.

Organic compounds other than propane can be used by making the appropriate correction for response factor.

- Zero gas — <0.1 ppmv of organic material or <0.1 percent of span value, whichever is greater
- Low-level calibration gas — concentration equivalent to 25 to 35 percent of span value

SLIDE 25A-10

GASES (continued)

CALIBRATION GASES (cont.):

- Mid-level calibration gas — concentration equivalent to 45 to 55 percent of span value.
- High-level calibration gas — concentration equivalent to 80 to 90 percent of span value.

FUEL GASES:

40 percent H₂/60 percent He or 40 percent H₂/60 percent N₂ gas mixture is recommended.

MEASUREMENT SYSTEM PERFORMANCE SPECIFICATIONS

- ZERO DRIFT — $< \pm 3\%$ of span value/hr
CALIBRATION DRIFT — $< \pm 3\%$ of span value/hr
CALIBRATION ERROR — $< \pm 5\%$ of calibration value
(for low and medium standard)

SLIDE 25A-12

PRETEST PREPARATIONS

- Select sampling site
- Install sample probe
- Prepare measurement system
- Conduct calibration error and response time tests

SLIDE 25A-13

TEST PROCEDURE

- Organic Measurement
 1. Begin sampling at start of test.
 2. Record time and required process information.
 3. Note process interruption or cyclic operation.
- Drift Determination
 1. At end of test period, reintroduce zero and mid-level calibration gases.
 2. Record analyzer response.

ORGANIC CONCENTRATION CALCULATION

- Determine average organic concentration (ppmv) as propane or other calibration gas
- If concentration required in terms of organic carbon adjust by:

$$C_o = K \times C_{\text{meas}}$$

K = 2 for ethane

K = 3 for propane

K = 4 for butane

C_o = Carbon observed

C_{meas} = Carbon measured

METHOD 25B

Determination of Total Gaseous Organic Concentration Using a Nondispersive Infrared Analyzer

SLIDE 25B-2

APPLICABILITY

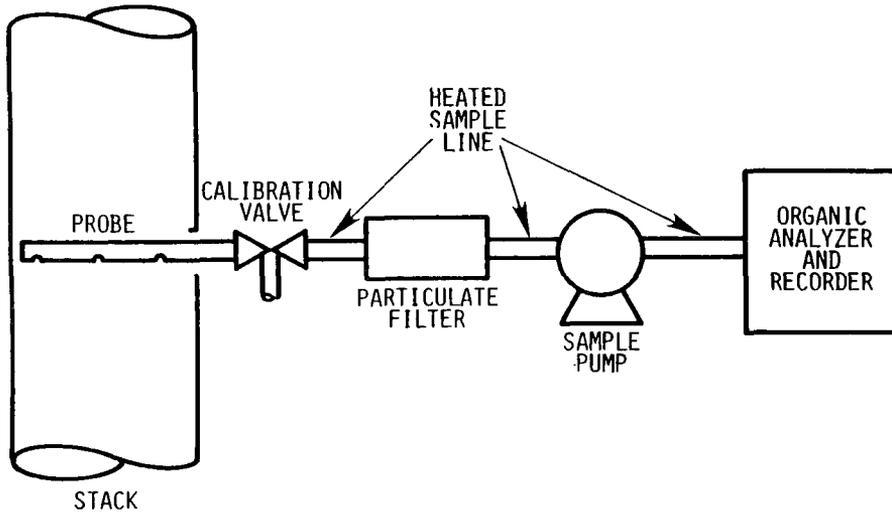
For measurement of total gaseous organic concentration of vapors consisting primarily of alkanes. Concentration is expressed in terms of appropriate organic calibration gas or in terms of carbon.

SLIDE 25B-3

PRINCIPLE

- Gas sample is extracted from source through a heated sample line, if necessary, and glass fiber filter to a nondispersive infrared analyzer.
- Results are reported as volume concentration equivalents of calibration gas or as a carbon equivalent.

ORGANIC CONCENTRATION MEASUREMENT SYSTEM



SLIDE 25B-5

GASES Calibration

1. Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value.
2. Organic compounds other than propane can be used by making the appropriate correction for response factor.
 - Zero gas— < 0.1 ppmv of organic material or $< 0.1\%$ of span value whichever is greater.
 - Low-level calibration gas—concentration equivalent to 25 to 35% of span value.
 - Mid-level calibration gas—concentration equivalent to 45 to 55% of span value.
 - High-level calibration gas—concentration equivalent to 80 to 90% of span value.

**MEASUREMENT SYSTEM
PERFORMANCE SPECIFICATIONS**

ZERO DRIFT —<±3% of span value/hr
CALIBRATION DRIFT —<±3% of span value/hr
CALIBRATION ERROR —<±5% of calibration value
(for low and medium standard)

SLIDE 25B-7

PRETEST PREPARATIONS

- Select Sampling Site
- Install Sample Probe
- Prepare Measurement System
- Conduct Calibration Error and Response Time Tests

SLIDE 25B-8

TEST PROCEDURE

During sampling, record process data and note process interruption or cyclic operation.

DRIFT DETERMINATION

Determine measurement system drift hourly during the test and immediately following the completion of the test period.

SLIDE 27-1

NOTES

METHOD 27

**Determination of Vapor Tightness
of Gasoline Delivery Tank Using
Pressure Vacuum Test**

SLIDE 27-2

APPLICABILITY

For determination of vapor tightness of a gasoline delivery tank equipped with vapor collection equipment.

SLIDE 27-3

PRINCIPLE

Pressure and vacuum are applied alternately to compartments of gasoline delivery tank and change in pressure or vacuum is recorded after specified time period.

SLIDE 27-4

N01

PRESSURE CHECK

With the use of a pressure source, or by filling the tank, obtain a stable pressure of 450 mm H₂O; shut off valve and observe pressure for 5 min.

For the arithmetic average of two consecutive runs which agree within ± 12.5 mm H₂O, the change in pressure must be < 75 mm H₂O.

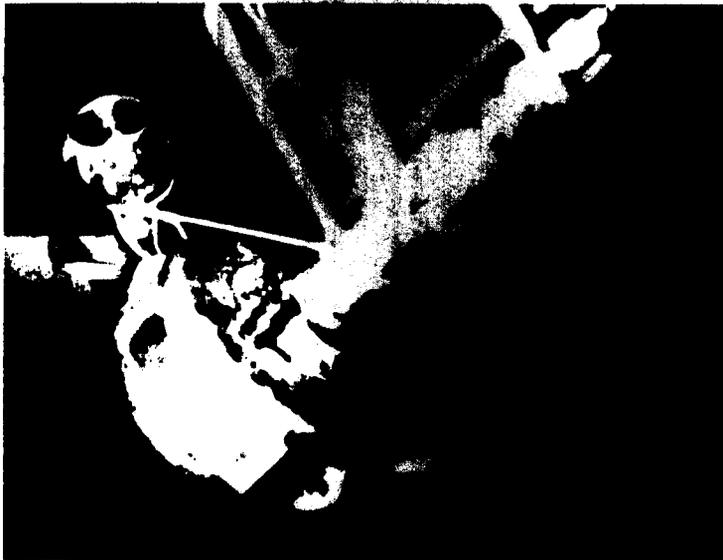
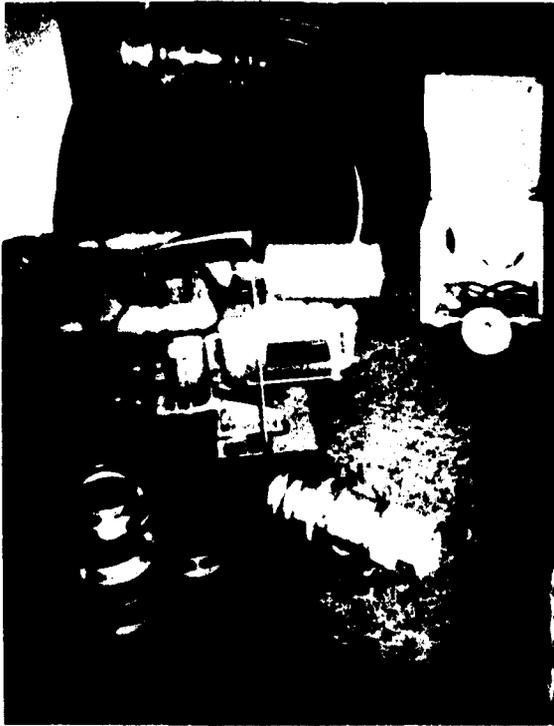
SLIDE 27-5

VACUUM CHECK

The same procedure should be used with a vacuum source or alternatively by draining the tank.

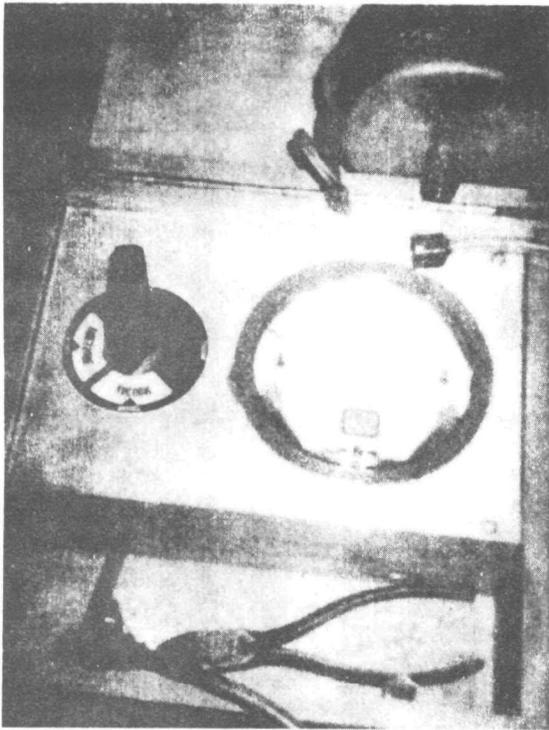
SLIDE 27-6





SLIDE 27-9

NOTES



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Environmental Protection
Agency

Office of Air and Radiation
Office of Air Quality Planning and Standards
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