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MARYLAND DEPARTMENT OF THE ENVIRONMENT

TECHNICAL MEMORANDUM 91-01

TEST METHODS AND EQUIPMENT SPECIFICATIONS FOR STATIONARY SOURCES

January, 1991

MARYLAND DEPARTMENT OF THE ENVIRONMENT
AIR MANAGEMENT ADMINISTRATION
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USERS GUIDE TO
TM 91-01
"TEST METHODS AND EQUIPMENT SPECIFICATIONS FOR STATIONARY SOURCES"

The document TM 91-01 "Test Methods and Equipment Specifications" (January 1991), which became effective on August 19, 1991, undergoes changes from time to time. These changes are promulgated as supplements. New pages with revised text are identified by a supplement number which appears at the bottom right of the page. Supplement pages must be inserted into the original document.

Procedure for Development and Promulgation of Air Quality Regulations

A description of the process for promulgating Maryland regulations can be found in the Preface to the Code of Maryland Regulations, and will not be repeated here.

Proposed and Emergency Supplements Binder

To maintain an orderly process of informing individuals about proposed supplements, the Secretary of the Environment maintains a Proposed and Emergency Supplements (P.E.S) binder. Proposed supplements have no force until finally adopted by the Secretary of the Environment and promulgated in the Maryland Register. Material which has the status of emergency regulations supersedes specified portions of TM 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January 1991) until the expiration date of emergency status or until the material is finally adopted, whichever occurs earlier.

Maintenance of TM 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January 1991) and P.E.S. Binders

The Secretary of the Environment supplies each holder of a binder with supplements. Supplements are accompanied by instruction sheets regarding replacement, removal, and insertion of material.

Availability of TM 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January 1991)

Authoritative copies of TM 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January 1991) are maintained at the Air Management Administration, 2500 Broening Highway, Baltimore, MD 21224; the Division of State Documents, 11 Bladen Street, Annapolis, MD 21401, and the following state depository libraries.

State Library Resource Center
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Maryland Department
400 Cathedral Street
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Maryland Department of Legislative
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Library
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HISTORY OF ADOPTED AND PENDING SUPPLEMENTS

TECHNICAL MEMORANDUM 91-01
"TEST METHODS AND EQUIPMENT SPECIFICATIONS FOR STATIONARY SOURCES"
(January, 1991)

Note: The original document and all adopted supplements to Technical Memorandum 91-01 can be found in the permanent binder (navy blue) for COMAR 26.11.01.04C. All proposed supplements which have not yet been adopted (indicated by the entry "Future" under the Effective Date column) are found in this Proposed and Emergency Supplements (PES) binder.

<u>Supplement Number</u>	<u>Effective Date</u>
Original Document	August 19, 1991
Supplement 1	February 17, 1992
Supplement 2	February 15, 1993
Supplement 3	November 2, 1998

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Method 1000
General Aspects of Source Emissions Testing

This technical memorandum describes the test methods to be used in the State of Maryland for determining the emissions from stationary sources. Although many of the approved test methods appear in the Code of Federal Regulations (CFR) and are adopted by reference, a listing of the titles of these test methods is included in the Appendix to this document.

Disclaimer

Mention of trade names or commercial products does not constitute Air Management Administration endorsement or recommendation for use.

Standard Conditions

Standard Conditions are 25°C (77°F) temperature and 760 mm of mercury (29.92 inches) pressure. Where applicable the metric units are given first and the English units are then included in parentheses.

Exceptions

Requests for exceptions to these test methods or procedures must be submitted to the Air Management Administration and when applicable, to the U.S. EPA, for approval prior to conducting the tests.

Test Method Notification: EPA 5 Condensibles

Condensibles determined in the impingers of EPA Method 5 for particulate matter, shall be reported, but only the filterable portion of the particulate (front half) shall be used to determine compliance with the regulations except for refuse incinerators with a charging rate less than 50 tons/day.

Stationary Source Applicability

These test methods shall be applicable to all stationary sources unless other test methods are required in a specific regulation. Anyone intending to perform a stack test using any one of these procedures shall first submit, for approval by the Department, a test protocol as outlined in Appendix A of this section.

Test Report

A source test report (Appendix B) will not be considered a demonstration of compliance unless it is performed in accordance with an approved test protocol (Appendix A) and it contains a description of the source operating conditions during the test. This description may be included in the test report under the source operation section when prepared by the test contractor or it may be submitted directly by the source as an addendum to the test report.

GENERAL REQUIREMENTS

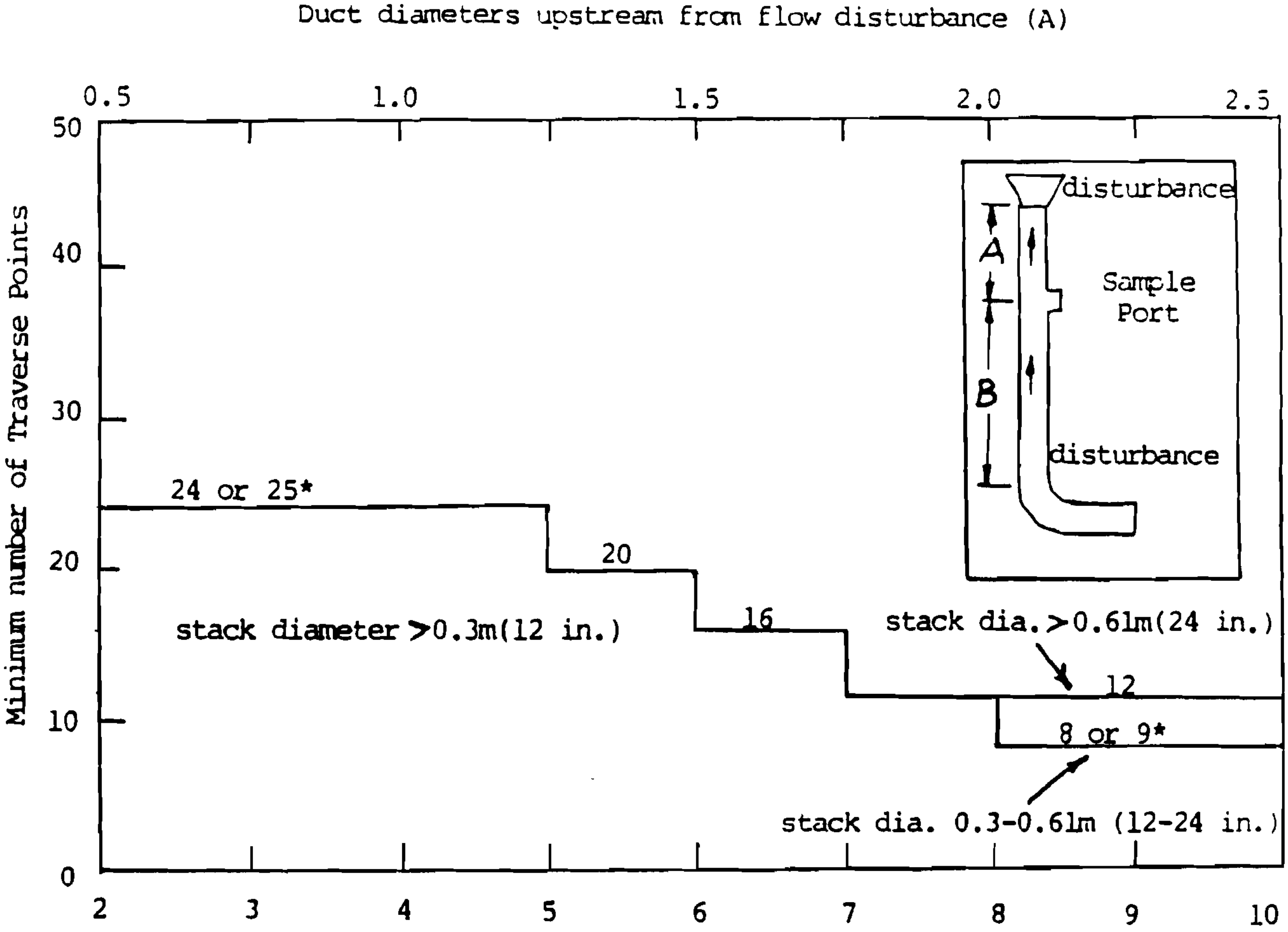
The sampling site designated for an emissions test can affect the quality of the sample extracted. Site selection should be simple at new installations, since most states require installation of an acceptable sampling site as a condition for obtaining a construction permit. Acceptability is generally determined with reference to the distance from the nearest upstream and downstream disturbance to gas flow by an obstruction or change in direction. More sampling points are required for each test the closer it is to a disturbance, as shown in Figure 1.

In addition to flow considerations, accessibility and safety are important. Clearance for the probe and sampling apparatus, availability of electricity, exposure of personnel and equipment to weather or excessive heat, presence of toxic or explosive gases, and other safety factors must be considered. The following guidelines constitute minimum requirements for safe and accessible stack sampling sites.

A. Sampling Ports

1. Port Location - Ideally, ports should be located at least eight stack diameters downstream of any bends, inlets, constrictions, abatement equipment, or other flow distrubances and at least two stack diameters upstream of the stack exit or other flow distrubances. However, the absolute minimum requirements are two stack diameters downstream and one-half stack diameters upstream. Where these criteria are not met, a stack extension may be required unless the plant representative can demonstrate that this remedy is not feasible. In such a case, an alternative port location must be approved by the Air Management Administration.

Figure 1 Recommended No. of Traverse Points



Duct diameters downstream from flow disturbance (B)

* for rectangular stack

Table 1-1

No. of Sample Points	Minimum Stack Diameter for Circular Stacks [a] (In.)
8	14.9
12	22.7
16	30.3
20	40.0
24	47.6

[a] To sample points which represent equal areas of the stack cross section such that no point lies within 1 inch of the stack wall.

2. Port Type - A sampling port should be a standard industrial flanged pipe of 3 inch inside diameter (ID) with a 6-inch bolt circle diameter. An easily-removable blind flange should be provided to close the port when not in use. Ports larger than 3-inches ID are permissible and even desirable on large-diameter, double-walled stacks, which necessitate use of longer probes. These ports should also be equipped with a standard industrial flange of the same ID as the port. Gate valves should be installed only when unusual stack conditions or the presence of hazardous materials require such devices to insure safety. A suitable alternative would be a 4" diameter pipe nipple with a cap.

3. Port Installation - Ports should be installed flush with the interior stack wall. Ports should extend outward from the exterior stack wall no less than 2 inches nor more than 8 inches, unless additional length is required for gate valve installation. Ports should be installed no less than 14 in. nor more than 6 feet above the floor of the platform. Ports should be installed with respect to the limitations of the clearance zone, described later.

4. Number of Ports Required - If the sum of the stack ID plus one port length (stack inside wall to end of port extension) is less than 10 feet, two ports should be installed on diameters 90 degrees apart. If the sum of the stack ID plus one port length (stack inside wall to end of port extension) is equal to or greater than 10 feet, four ports should be installed on diameters 90 degrees apart.

5. Port Loading - The port installations should be capable of supporting the following loads:

a. Vertical shear of 200 pounds

b. Horizontal shear of 50 pounds; and

c. Radial tension of 50 pounds (along stack diameter).

B. Work Platform

1. Size and Extent of Platform - If two ports are required at 90 degrees, the work platform should serve that quarter of the stack circumference between the ports and extend at least 3 feet beyond each port. Minimum platform width is 3 feet. If four ports are required at 90 degrees, the work platform should serve the entire circumference of the stack. Minimum platform width is 4 feet unless the sum of the stack ID plus one port length (stack inside wall to end of port extension) is less than 10 feet, in which case the minimum platform width is 3 feet.

2. Platform Access - Safe and easy access to the work platform should be provided via caged ladder, stairway, or other suitable means.

3. Guardrails, Ladderwells and Stairwells - A safe guardrail should be provided on the platform. Angular rather than round rail members should be used if possible. Ladderwells should be covered at the platform and any stairwell leading directly to the platform should be equipped with a safety bar (or the equivalent) at the opening.

4. Platform Loading - The work platform should be able to support at least three men and 200 pounds of test equipment (at least 800 pounds total). If the stack exits through a building roof, the roof may suffice as the work platform, provided the minimum test site requirements are still met. In such cases, all other requirements are the same as for a remote stack.

5. Clearance Zone - A three-dimensional, obstruction-free clearance zone should be provided around each port. The zone should extend 1 foot above the port, 2 feet below the port, and 2 feet to either side of the port. The zone should extend outward from the

exterior wall of the stack to a distance of at least one stack ID plus one port length (inside wall to end of port extension) plus 3 feet. The clearance zone is illustrated in Figure 2.

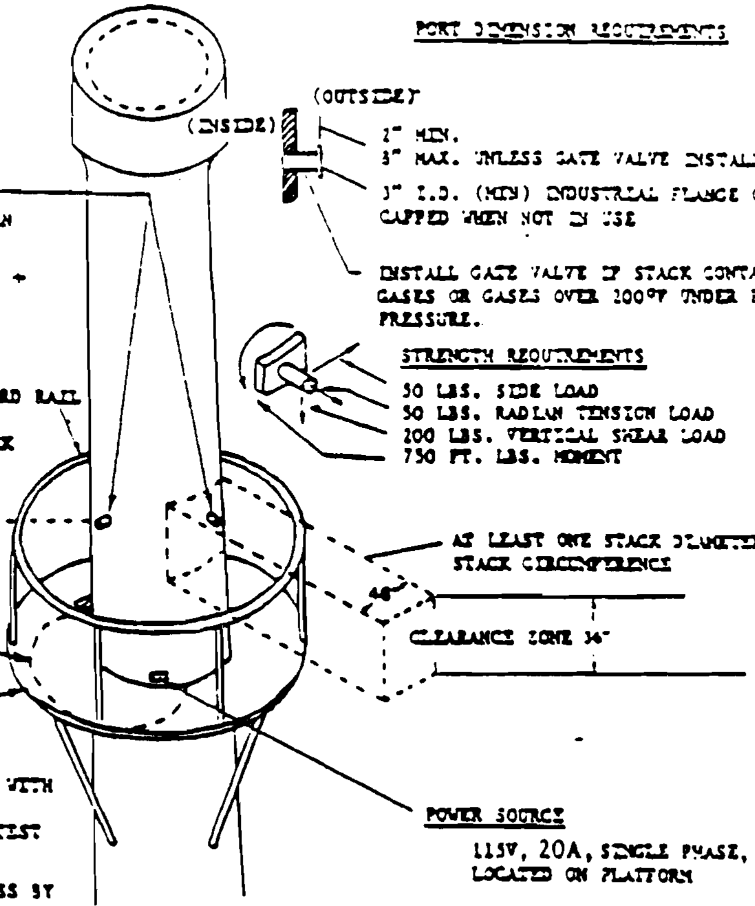
C. Power Supply - Power sources shall be as follows:

1. Platform or Other Sample Site - One 115-volt, 20-amp, single-phase, 60 Hz AC circuit with a ground, two-receptacle weatherproof outlet. Receptacles should accept standard, three-prong, grounded, household-type plugs, or else suitable adapters shall be provided.

D. Vehicle Access and Parking - Except for situations in which sampling operations must be conducted from a rooftop or similar structure, stack sampling is sometimes coordinated and controlled from a cargo van or trailer, which is parked near the base of the stack for the duration of the sampling period. Vehicle access and parking space must be provided, since various umbilical, communications and equipment transport lines will be strung from the van or trailer to the stack platform and will remain in position during the operations.

E. Compliance with Safety Standards - Stack sampling facilities must meet all applicable Occupational Safety and Health Administration (OSHA) requirements and must conform to any other relevant safety guidelines.

F. Optional Permanent Monorail System - Some plants may wish to install a permanent monorail system to facilitate self-monitoring. Persons considering such installations should be aware that commercially available test equipment varies in size and configuration and will not necessarily operate on a permanent monorail system as shown in Figure 3.



PORT DIMENSION REQUIREMENTS

2" MIN.
3" MAX. UNLESS GATE VALVE INSTALLED

3" I.D. (MIN) INDUSTRIAL FLANGE OR NIPPLE CAPPED WHEN NOT IN USE

INSTALL GATE VALVE IF STACK CONTAINS DANGEROUS GASES OR GASES OVER 200°F UNDER POSITIVE PRESSURE.

STRENGTH REQUIREMENTS

50 LBS. SIDE LOAD
50 LBS. RADIAN TENSION LOAD
200 LBS. VERTICAL SHEAR LOAD
750 FT. LBS. MOMENT

SAMPLING PORTS

A. 2 PORTS, 90° APART W/DIAMETER LESS THAN 10' + PORT LENGTH

4 PORTS, 90° APART W/DIAMETER OVER 10' + PORT LENGTH

AT LEAST .5 STACK DIAMETERS BELOW STACK EXIT

AT LEAST TWO STACK DIAMETERS ABOVE LAST OBSTRUCTION

WORK AREA CLEARANCE

AT LEAST ONE STACK DIAMETER PLUS 3' FROM STACK CIRCUMFERENCE

WORK PLATFORM

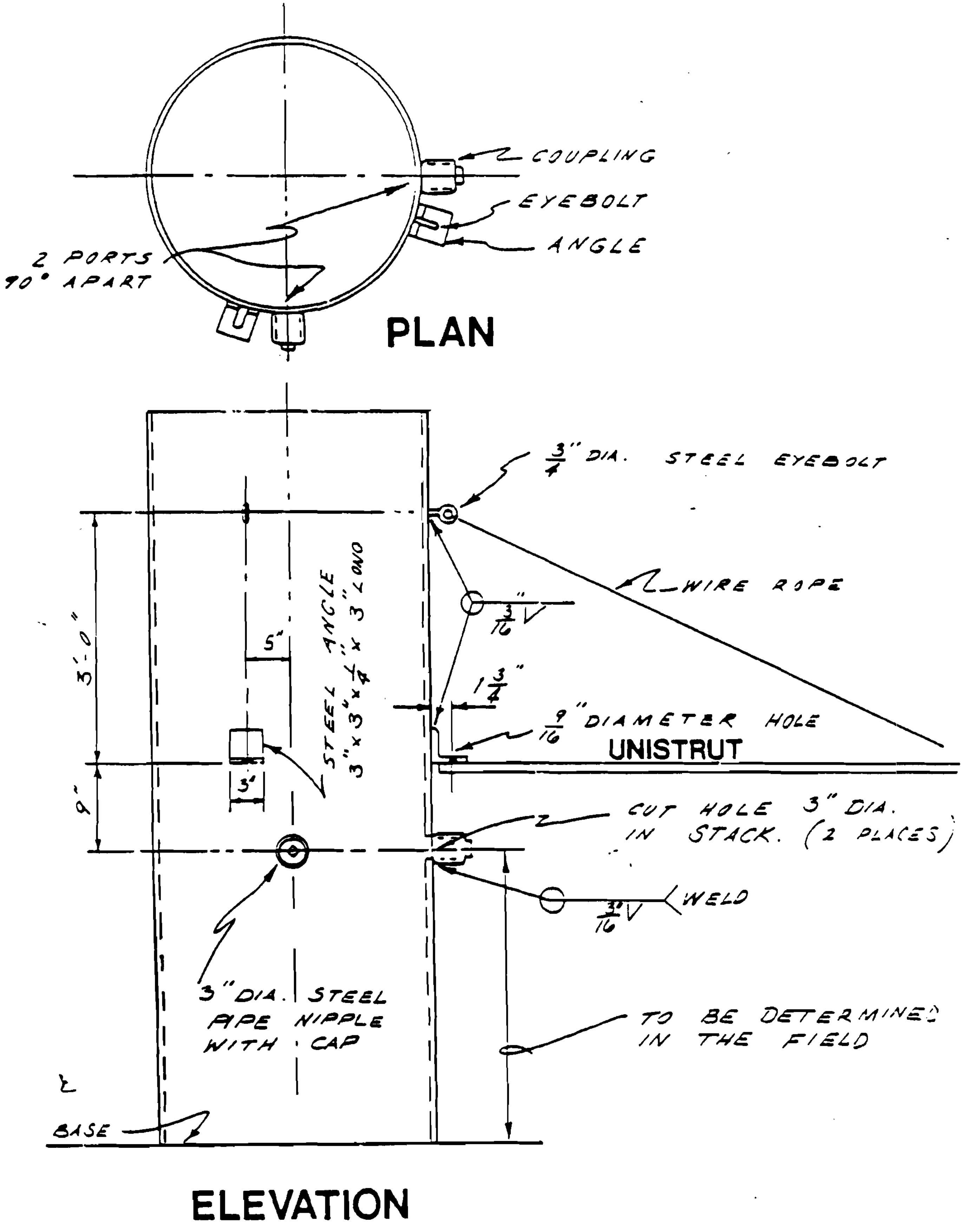
A. AT LEAST 3' WIDE (6' WIDE FOR STACKS WITH 10' OR GREATER I.D.) AND CAPABLE OF SUPPORTING 3 PEOPLE AND 200 LBS. OF TEST EQUIPMENT

B. SAFE GUARDRAIL ON PLATFORM WITH ACCESS BY SAFE LADDER OR OTHER SUITABLE MEANS. IF LADDER IS USED, LADDER WELL MUST BE LOCATED AT LEAST 3' FROM PORTS

C. NO OBSTRUCTIONS TO BE WITHIN 3' HORIZONTAL RADIUS ON PLATFORM BENEATH PORTS

POWER SOURCE

115V, 20A, SINGLE PHASE, 60 HZ AC LOCATED ON PLATFORM


COUPLING
EYEBOLT
ANGLE
2 PORTS 90° APART
PLAN
3/4" DIA. STEEL EYEBOLT
WIRE ROPE
3/16 V
STEEL ANGLE 3"x3"x1/4"x 3" LONG
5"
3'-0"
1 3/4"
9/16" DIAMETER HOLE
UNISTRUT
3'
9"
CUT HOLE 3" DIA. IN STACK. (2 PLACES)
WELD
3/16 V
3" DIA. STEEL PIPE NIPPLE WITH CAP
TO BE DETERMINED IN THE FIELD
BASE
ELEVATION

NOT TO SCALE

G. Excess or Dilution Air - In addition to the above requirements, certain processes (such as sulfur recovery units or unused ducts which vent into the stack being tested) may require provisions for determining the composition and flow rates of feedstock streams. This information, taken at the time of sampling, is needed to determine the amount of excess or dilution air in the stack effluent.

H. Stack Extensions - Many times the maximum straight run of stack is not sufficient to meet the agency requirements or the stack is lined and ports cannot be cut into the stack. A simple solution applicable to some smaller-diameter stacks is the use of a stack extension. Stack extensions need not be permanent and can be made out of sheet metal or plywood for testing purposes only.

I. Test Duration - A test consists of three sample runs on three separate days. Any deviation must be requested in the test protocol and approved by AMA.

J. Report Requirements - If a test report is to be submitted to demonstrate compliance or to meet other requirements of the AMA, it must contain the information contained in Appendix B.

III. RESPONSIBILITIES

A. Plant officials must assume responsibility for:

1. Maintenance of constant process operation for duration of each test.
2. Monitoring of Facility Operations (General Requirements).
    a. Monitoring and recording the process parameters as specified in the test program.
    b. Collect process raw materials or fuel samples for subsequent analysis.
    c. Monitoring and recording the operation of control equipment.
    d. If an equipment malfunction or upset should occur, the

agency observer and test team leader must be notified immediately.

3. Providing unobstructed sample ports for acess to the gas ducts. (See attached diagrams)
4. Providing 110 volt, 20 amp. electrical supply to sample site.
5. Provide access to sampling site and safe working conditions.
6. Prompt clearance of personnel, vehicles and equipment through plant security.

B. The Air Managment Administration representative is responsible for guidance before and during the test, evaluation of sampling procedures, and for taking readings of visible emissions during the tests.

C. The stack testing team (whether the AMA or an outside contractor) sets up the sampling equipment, performs preliminary measurements, conducts sample runs, recovers collected samples, disassembles and removes equipment, and reports results.

IV. AGREEMENT ON FACILITY OPERATION DURING THE TEST PERIOD

Prior to actual sampling, process and control equipment parameters will be agreed upon by the AMA and the company being tested. The following is a typical list of important points.

A. Process

1) Method of process weight or rate determination.
2) Process parameters to be monitored and recorded, and their acceptable limits to document process operation.
3) Raw material feed and/or fuel acceptable analyzed values.
4) Normal operating cycle or procedures.
5) Portions of the operating cycle or procedure that will be represented by each run.

B. **Control Equipment**

6) Control equipment and effluent parameters to be monitored and recorded, and their acceptable limits to document control equipment operations.

7) Normal operating cycle (cleaning, dust removal, etc.).

8) Normal maintenance schedule.

9) Manner in which the control equipment will be operated during test.

C. **Starting the Test**

The plant representative should be present when the test team starts preparations. The test team will unpack the test equipment, check for possible damage, and assemble the sampling train. If an agency-observer is present, he will give close attention to these initial operations. His monitoring of test procedures protects the interests of both the agency and the operating facility. If testing is performed improperly, the agency observer may require performance of another sample run. Any additional sampling necessitated by improper sampling techniques should be performed at the expense of the testing team. As stated earlier, a stack test submitted to demonstrate compliance consists of three sample runs usually run on three separate days.

STATE OF MARYLAND

STACK TEST PROTOCOL

APPENDIX A

1) Source information
    a. Name
    b. Address
    c. Person to contact
    d. Telephone

2) Testing firm information
    a. Name
    b. Address
    c. Person to contact
    d. Telephone

3) Sampling information
    a. Pollutants to be sampled
    b. Number of sample points
    c. Total time per sample
    d. Number of samples - at least 3 under the same process operating parameters
    e. Estimated concentration
    f. Method used to estimate concentration

4) Stack or duct information
    a. Approximate temperature (°F)
    b. Approximate gas flow (acfm)
    c. Approximate percent H_2O

5) Date of last equipment calibration
    a. Velocity measuring equipment
    b. Gas volume metering equipment
    c. Gas flow rate metering equipment
    d. Gas temperature measuring equipment
    e. Sampling train information
        1. A schematic diagram of sample train
        2. Type of capture medium to be used
        3. Sample tube type: glass, teflon, stainless steel, etc.
        4. Probe cleaning method and solvent(s) to be used

6) Lab analysis
    A brief description of lab analysis method to be used

7) Data sheets
    A sample of field data sheets to be used

8) Description of operations
    A description of any operation, process or activity that could vent exhaust gases to the test stack. Include description and feed rates of all materials capable of producing pollutant emissions used in each separate operation.

    All testing shall be performed at maximum rate capacity as specified by the equipment manufacturer or at the maximum rate actually used in the source operation, whichever is greater.

9) A schematic diagram showing entire ducting and stack arrangement. Include all processes or operations venting to the stack or duct to be tested; position of sampling ports relative to nearest upstream and downstream gas flow direction or duct dimensional change, and gas cleaning equipment.

    Include a diagram of stack at sample ports showing position of sample points. With rectangular ducts, show division of duct into equal areas.

STATE OF MARYLAND
SOURCE TESTING REPORT FORMAT
APPENDIX B

Cover

1. Plant name and location
2. Source sampled
3. Testing company or agency, name and address

Certification

1. Certification by team leader
2. Certification by reviewer (e.g., P.E.)

Introduction

1. Test purpose
2. Test location, type of process
3. Test dates
4. Pollutants tested
5. Observer's names (industry and agency)
6. Any other important background information

Summary of Results

1. Emission results
2. Process data, as related to determination of compliance
3. Allowable emissions
4. Description of collected samples
5. Visible emissions summary
6. Discussion of errors, both real and apparent

Source Operation

1. Description of process and control devices
2. Process and control equipment flow diagram
3. Process data and results, with example calculations
4. Representativeness of raw materials and products
5. Any specially required operation demonstrated

Sampling and Analysis Procedures

1. Sampling port location and dimensioned cross section
2. Sampling point description, including labeling system
3. Sampling train description
4. Brief description of sampling procedures, with discussion of deviations from standard methods
5. Brief description of analytical procedures, with discussion of deviations from standard methods

Appendix

1. Complete results with example calculations
2. Raw field data (original, not computer printouts)
3. Laboratory report, with chain of custody
4. Raw production data signed by plant official
5. Test log
6. Calibration procedures and results
7. Project participants and titles
8. Related correspondence
9. Standard procedures

METHOD 1001

[Reserved.]

Designation: D 322 – 85

An American National Standard

Designation: 23/83

Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation[1]

This standard is issued under the fixed designation D 322; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This method was adopted as a joint ASTM-IP standard in 1964.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers determination of the amount of dilution in crankcase oils of engines when gasoline has been used as the fuel.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statement, see 5.4.

2. Referenced Documents

2.1 *ASTM Standard:*

D 235 Specification for Mineral Spirits (Petroleum Spirits) Hydrocarbon Dry Cleaning Solvent[2]

3. Summary of Method

3.1 The sample, mixed with water, is placed in a glass still provided with a reflux condenser discharging into a graduated trap connected to the still. Heat is applied, and the contents of the still are brought to boiling. The diluent in the sample is vaporized with the water and then liquefied in the condenser. The diluent collects at the top of the trap, and the excess water runs back to the still where it is again vaporized, carrying over an additional quantity of diluent. The boiling is continued until all the diluent has been boiled out and recovered in the trap, and the volume is recorded.

4. Significance and Use

4.1 Some fuel dilution of the engine oil may take place during normal operation. However, excessive fuel dilution is of concern in terms of possible performance problems.

5. Apparatus

5.1 *Flask,* round-bottom type as described in the Annex.

5.2 *Condenser,* Liebig straight-tube type, as described in the Annex.

5.3 *Trap,* constructed in accordance with the requirements in Figs. 1 and 2 and in the Annex.

5.4 *Heater*—Any suitable gas burner or electric heater may be used with the glass flask. (**Warning**—Hot exposed surface. See Annex A2.1.)

6. Procedure

6.1 Mix the sample thoroughly, measure 25 mL by means of a 25-mL graduated cylinder, and transfer as much as possible of the contents of the cylinder by pouring it into the flask. Wash the graduated cylinder with successive portions of hot water until only a negligible amount of oil is left in the cylinder. Add additional water to the flask to make a total of approximately 500 mL of water. Fill the trap with cold water and add 1 mL of ethanol to the water in the trap.

6.2 Assemble the apparatus as shown in Fig. 1, so that the tip of the condenser is directly over the indentation in the trap.

6.3 Apply heat (**Warning!** Hot exposed surface. See Annex A2.1) to the flask at such a rate that refluxing starts within 7 to 10 min after heat is applied, with the water and sample being at 21 to 38°C prior to application of heat. After boiling and condensation has commenced, adjust the rate of boiling so that condensed distillate is discharged from the condenser at a rate of 1 to 3 drops per s.

NOTE 1—Bumping with a tendency to froth over is often experienced with dirty oils. The use of "boiling stones," steel wool, or about 5 mL of concentrated hydrochloric acid (HCl) in the flask is often helpful in eliminating this difficulty.

6.4 Obtain readings of the amount of diluent at the following times, taken from the time that refluxing starts: 5, 15, and 30 min, and each 15 min following until the test is complete. Completion of the test shall be determined on the basis of either or both of the following criteria:

6.4.1 The test is complete when the volume of diluent increases by not more than 0.1 mL in any 15-min period during the course of the test.

6.4.2 The test is complete when the volume of diluent obtained in a given time indicates completion, as

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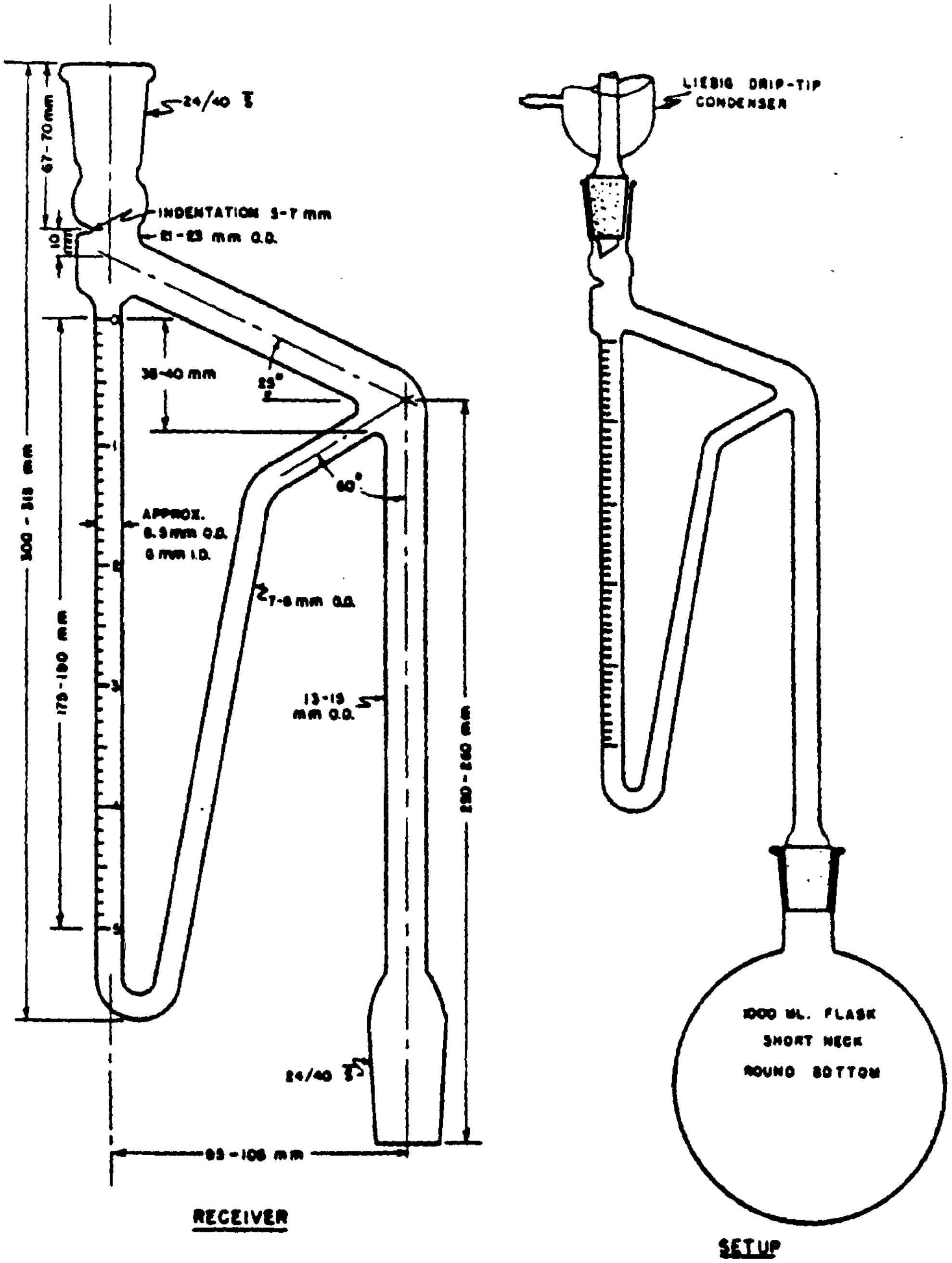
[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

In the IP, this method is under the jurisdiction of the Standardization Committee.

Current edition effective Oct. 25, 1985. Published December 1985. Originally published as D 322 – 30. Last previous edition D 322 – 80$^{\epsilon 1}$

[2] *Annual Book of ASTM Standards,* Vol 05.01.



RECEIVER

SETUP

FIG. 1 Apparatus for Determining Diluent in Gasoline Engine Crankcase Oil

follows:

Time from Start of Refluxing	Test is Complete if Apparent Volume of Diluent Collected is Equal to or Less Than:
5 min	no visible diluent[4]
30 min	2.0 mL
60 min	4.0 mL
90 min	5.0 mL

6.5 When the test continues without reaching the limit defined in 6.4.1, to a point at which any of the conditions described in 6.4.2 are encountered, the latter shall define the completion of the test.

6.6 When the test is complete by either of the criteria defined in 6.4.1 and 6.4.2, turn off the heat. Allow the equipment to stand at least 30 min to allow the distillate to settle clear and to cool to approximately room temperature. Read the volume of diluent collected in the trap. If the volume of diluent exceeds the calibrated capacity of the trap, discontinue the test and report the results as 20 % plus.

7. Calculations

7.1 The diluent content of the sample, expressed as volume percent, is equal to the volume of diluent in millilitres multiplied by 4.

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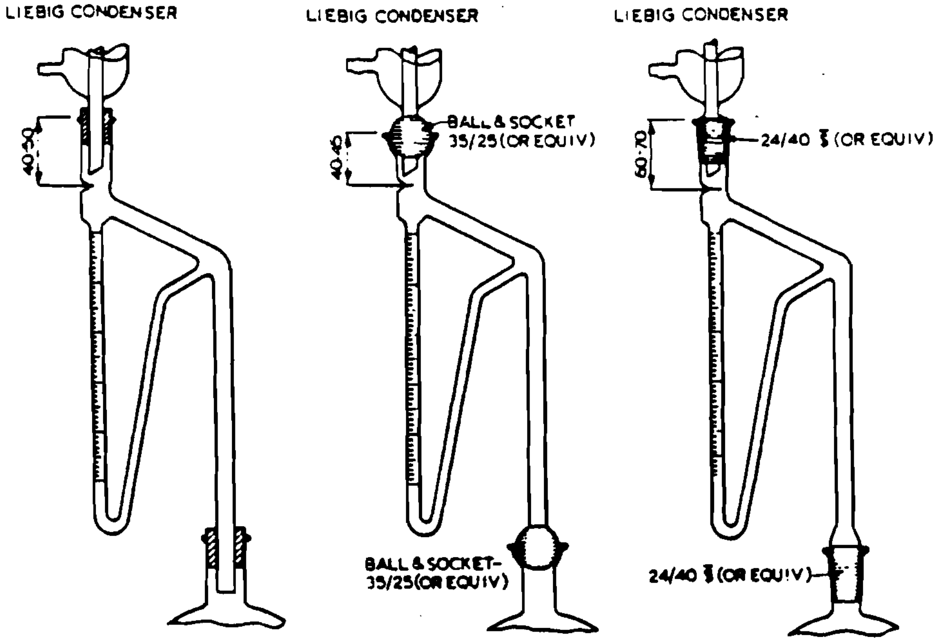
[4] Report as "no dilution"; otherwise the test should be continued at least 30 min.



All dimensions are in millimetres

FIG. 2 Alternate Closures for Diluent Trap

NOTE 2—In some cases with samples containing large amounts of diluent, equipment limitations do not permit collection and measurement of the full 5 mL of diluent even when more is present. This condition exists when the upper limit of the collected diluent is above the zero calibration mark on the trap. When it occurs, finish the test as prescribed in 6.6, read the maximum volume of diluent collected, calculate the corresponding percentage "x", and report the results as "x percent plus."

8. Report

8.1 Report the result as the Diluent Content, ASTM D 322 - IP 23.

9. Precision and Bias

9.1 The precision of the method as obtained by statistical examination of interlaboratory test results is as follows:

9.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions of identical test material, would in the long run, in the normal andcorrect operation of the test method, exceed the following value only in one case in twenty:

0.6 volume %.

9.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

1.4 volume %.

9.2 *Bias*—This test method is empirical and no statement of bias is possible.

ANNEXES

(Mandatory Information)

A1. APPARATUS

A1.1 *Flask*, round-bottomed, short-necked, having a nominal capacity of litre. Figures 1 and 2 show recommended designs and glass connections.

A1.2 *Condenser*, Liebig straight type with a jacket not less than 400 mm long, and with an inner tube having an outside diameter of 10 to 13 mm. Figures 1 and 2 show characteristic details of suitable condensers.

A1.3 *Trap*, in accordance with the details of construction shown in Figs. 1 and 2, and conforming to the following requirements: It shall be graduated from 0 to 5 mL in 0.1-mL divisions. It shall be calibrated at four or more points by first filling it with water and then adding Stoddard solvent or *n*-heptane from a standard buret having a calibrated capacity at least equal to that of the trap. The Stoddard solvent should conform to Specification D 484. The *n*-heptane should conform to the current IP specification. The error of the indicated volume shall not exceed 0.05 mL.

A2. PRECAUTIONARY STATEMENT

A2.1 **Warning:**

There are exposed hot surfaces on the apparatus.

Avoid contact with exposed skin by using protective equipment as required.

Method 1003
Acceptable Capture Efficiency Test Methods for Sources of Volatile Organic Compounds

General.

The following procedures are used to perform a capture efficiency test to determine compliance with prescribed requirements when an alternative control method is used. Protocol G-1 is the preferred method to be used when the VOC inlet and outlet concentrations differ substantially. Otherwise Protocol G-2 is to be used.

When a single VOC component is used in a process, the test requirements established in 40 CFR Part 60 Subpart BBB (New Source Performance Standards for Rubber Tire Manufacturing) as amended September, 1989 may also be used.

For a liquid/liquid system when a carbon adsorber is used as a control device, the test requirements established in 40 CFR Part 60.710 (New Source Performance Standards for Magnetic Tapes) may also be used.

Modification to This Test Method

Method 1003 is a general test method that is used to determine compliance when a control device is used to meet a coating limit or emission standard. Depending upon the configuration of an affected facility, this test method may be modified or replaced with another method in order to more appropriately determine compliance.

A source may request that an alternative test method be used by submitting information to the Department showing that compliance for the installation could be more appropriately demonstrated using an alternative method.

Upon receiving a request, the Department will review the alternative method and will approve the method if it determines that an alternative method is necessary, that the method is technically adequate to determine compliance, and that the method will satisfy the intent of the regulation and will not cause the regulation to be less restrictive.

The Department will submit the alternative method to the United States Environmental Protection Agency for approval. Compliance testing shall not be performed using any alternative test method until it is approved by both the Department and the United States Environmental Protection Agency.

Note: This test method, to be used to determine the capture efficiency for sources of volatile organic compounds, reflects, in full, the version approved by the U.S. EPA April 16, 1990.

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APR 16 1990

VOC CAPTURE EFFICIENCY
Procedure T - Criteria for and Verification of a Permanent or Temporary Total Enclosure

1. INTRODUCTION

1.1 Applicability. This procedure is used to determine whether a permanent or temporary enclosure meets the criteria of a total enclosure.

1.2 Principle. An enclosure is evaluated against a set of criteria. If the criteria are met and if all the exhaust gases are ducted to a control device, then the volatile organic compounds (VOC) capture efficiency (CE) is assumed to be 100 percent and CE need not be measured. However, if part of the exhaust gas stream is not ducted to a control device, CE must be determined.

2. DEFINITIONS

2.1 Natural Draft Opening (NDO) -- Any permanent opening in the enclosure that remains open during operation of the facility and is not connected to a duct in which a fan is installed.

2.2 Permanent Total Enclosure (PTE) -- A permanently installed enclosure that completely surrounds a source of emissions such that all VOC emissions are captured and contained for discharge through a control device.

2.3 Temporary Total Enclosure (TTE) -- A temporarily installed enclosure that completely surrounds a source of emissions such that all VOC emissions are captured and contained for discharge through ducts that allow for the accurate measurement of VOC rates.

3. CRITERIA OF A TEMPORARY TOTAL ENCLOSURE

3.1 Any NDO shall be at least 4 equivalent opening diameters from each VOC emitting point.

3.2 Any exhaust point from the enclosure shall be at least 4 equivalent duct or hood diameters from each NDO.

3.3 The total area of all NDO's shall not exceed 5 percent of the surface area of the enclosure's four walls, floor, and ceiling.

3.4 The average facial velocity (FV) of air through all NDO's shall be at least 3,600 m/hr (200 fpm). The direction of air through all NDO's shall be into the enclosure.

3.5 All access doors and windows whose areas are not included in Section 3.3 and are not included in the calculation in Section 3.4 shall be closed during routine operation of the process.

4. CRITERIA OF A PERMANENT TOTAL ENCLOSURE

4.1 Same as Sections 3.1 and 3.3 - 3.5.

4.2 All VOC emissions must be captured and contained for discharge through a control device.

5. PROCEDURE

5.1 Determine the equivalent diameters of the NDO's and determine the distances from each VOC emitting point to all NDO's. Determine the equivalent diameter of each exhaust duct or hood and its distance to all NDO's. Calculate the distances in terms of equivalent diameters. The number of equivalent diameters shall be at least 4.

5.2 Measure the total area (A_T) of the enclosure and the total area (A_N) of all NDO's of the enclosure. Calculate the NDO to enclosure area ratio (NEAR) as follows:

$$NEAR = A_N/A_T$$

The NEAR must be ≤ 0.05.

5.3 Measure the volumetric flow rate, corrected to standard conditions, of each gas stream exiting the enclosure through an exhaust duct or hood using EPA Method 2. In some cases (e.g., when the building is the enclosure), it may be necessary to measure the volumetric flow rate, corrected to standard conditions, of each gas stream entering the enclosure through a forced makeup air duct using Method 2. Calculate FV using the following equation:

$$FV = [Q_O - Q_I]/ A_N$$

where:

Q_O = the sum of the volumetric flow from all gas streams exiting the enclosure through an exhaust duct or hood.

Q_I = the sum of the volumetric flow from all gas streams into the enclosure through a forced makeup air duct; zero, if there is no forced makeup air into the enclosure.

A_N = total area of all NDO's in enclosure.

The FV shall be at least 3,600 m/hr (200 fpm).

5.4 Verify that the direction of air flow through all NDO's is inward. Use streamers, smoke tubes, tracer gases, etc. Strips of plastic wrapping film have been found to be effective. Monitor the direction of air flow at intervals of at least 10 minutes for at least 1 hour.

6. QUALITY ASSURANCE

6.1 The success of this protocol lies in designing the TTE to simulate the conditions that exist without the TTE, i.e., the effect of the TTE on the normal flow patterns around the affected facility or the amount of fugitive VOC emissions should be minimal. The TTE must enclose the application stations, coating reservoirs, and all areas from the application station to the oven. The oven does not have to be enclosed if it is under negative pressure. The NDO's of the temporary enclosure and a fugitive exhaust fan must be properly sized and placed.

6.2. Estimate the ventilation rate of the TTE that best simulates the conditions that exist without the TTE, i.e., the effect of the TTE on the normal flow patterns around the affected facility or the amount of fugitive VOC emissions should be minimal. Figure 1 may be used as an aid. Measure the concentration (C_G) and flow rate (Q_G) of the captured gas stream, specify a safe concentration (C_F) for the fugitive gas stream, estimate the CE, and then use the plot in Figure 1 to determine the volumetric flowrate of the fugitive gas stream (Q_F). A fugitive VOC emission exhaust fan that has a variable flow control is desirable.

6.2.1 Monitor the concentration of VOC into the capture device without the TTE. To minimize the effect of temporal variation on the captured emissions, the baseline measurement should be made over as long a time period as practical. However, the process conditions must be the same for the measurement in Section 6.2.3 as they are for this baseline measurement. This may require short measuring times for this quality control check before and after the construction of the TTE.

6.2.2 After the TTE is constructed, monitor the VOC concentration inside the TTE. This concentration shall not continue to increase and must not exceed the safe level according to OSHA requirements for permissible exposure limits. An increase in VOC concentration indicates poor TTE design or poor capture efficiency.

6.2.3 Monitor the concentration of VOC into the capture device with the TTE. To limit the effect of the TTE on the process, the VOC concentration with and without the TTE must be within ±10 percent. If the measurements do not agree, adjust the ventilation rate from the TTE until they agree within 10 percent.

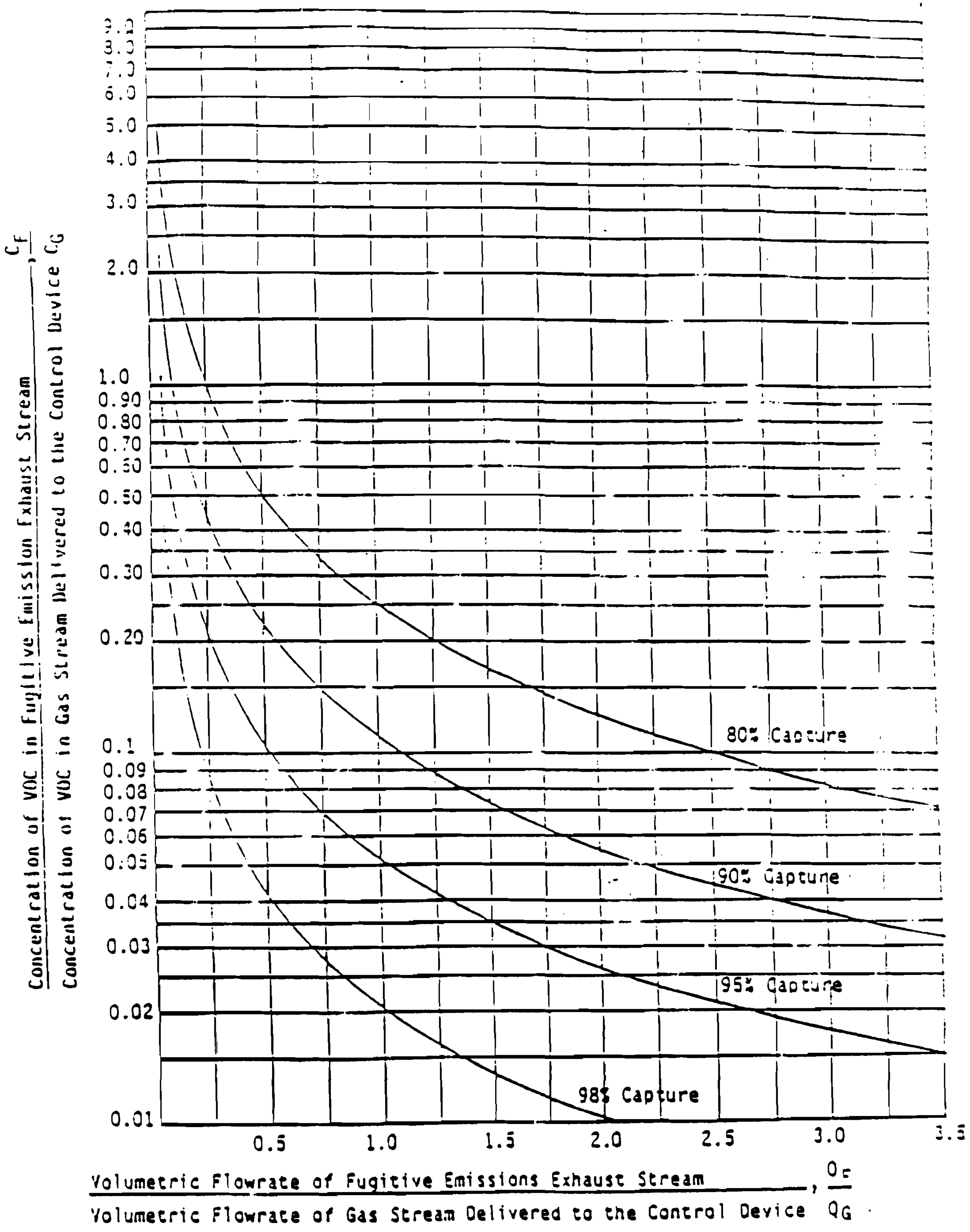


Figure 1. The Crumpler Chart

VOC CAPTURE EFFICIENCY
Procedure F.1 - Fugitive VOC Emissions from Temporary Enclosures

1. INTRODUCTION

1.1 **Applicability.** This procedure is applicable for determining the fugitive volatile organic compounds (VOC) emissions from a temporary total enclosure (TTE). It is intended to be used as a segment in the development of liquid/gas or gas/gas protocols for determining VOC capture efficiency (CE) for surface coating and printing operations.

1.2 Principle. The amount of fugitive VOC emissions (F) from the TTE is calculated as the sum of the products of the VOC content (C_{F_j}), the flow rate (Q_{F_j}), and the sampling time (θ_F) from each fugitive emissions point.

1.3 Estimated Measurement Uncertainty. The measurement uncertainties are estimated for each fugitive emission point as follows: Q_{F_j} = ±5.5 percent and CFj = ±5.0 percent. Based on these numbers, the probable uncertainty for F is estimated at about ±7.4 percent.

1.4 Sampling Requirements. A capture efficiency test shall consist of at least three sampling runs. The sampling time for each run should be at least 8 hours, unless otherwise approved.

1.5 Notes. Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing appropriate equipment and installing and using the equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

2. APPARATUS AND REAGENTS

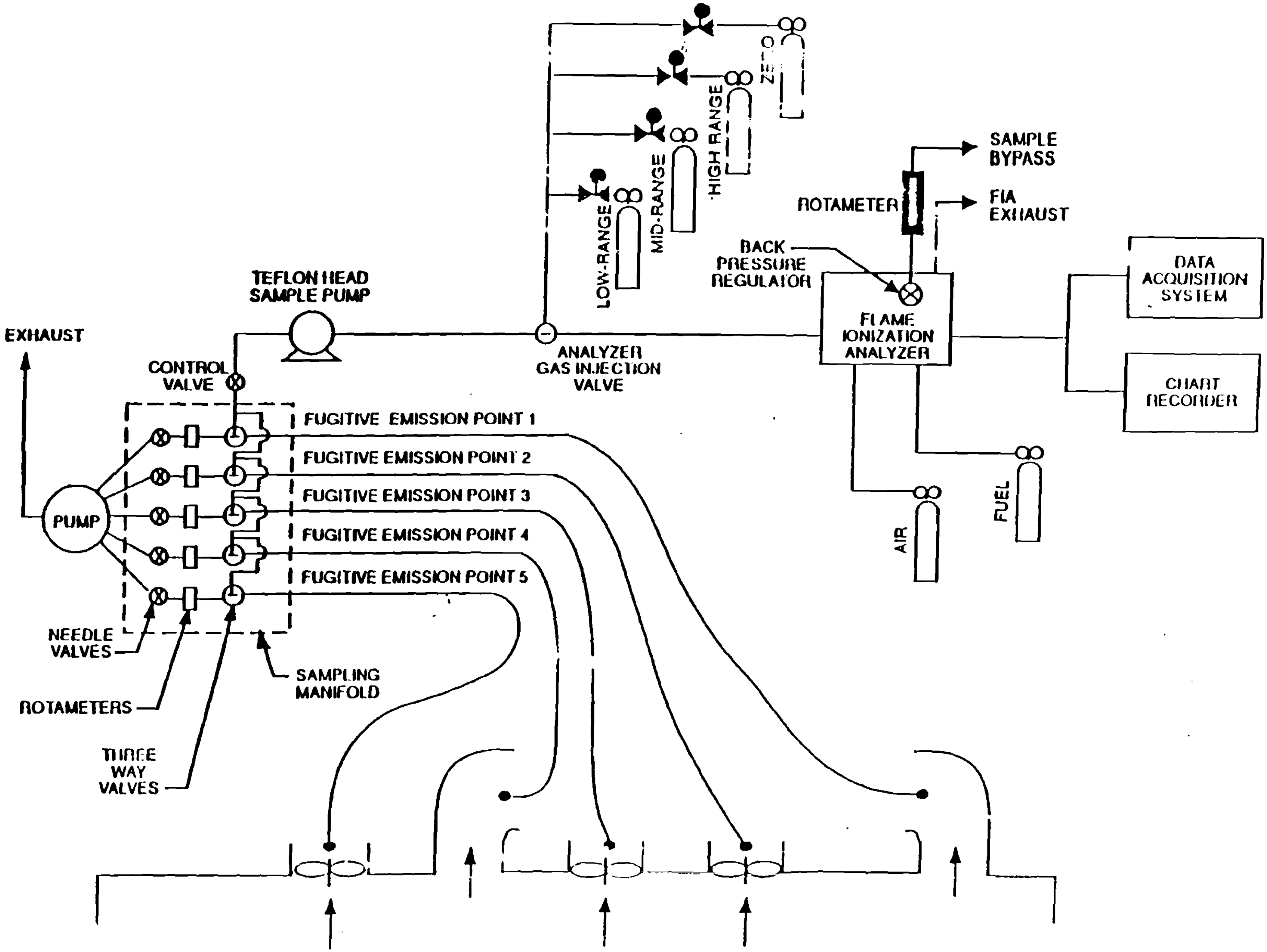
2.1 Gas VOC Concentration. A schematic of the measurement system is shown in Figure 1. The main components are described below:

2.1.1 **Sample Probe. Stainless** steel, or equivalent. The probe shall be heated to **prevent** VOC condensation.

2.1.2 **Calibration Valve Assembly.** Three-way valve assembly at the outlet of sample probe **to direct** the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.

2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream


ZERO
HIGH RANGE
MID-RANGE
LOW-RANGE
SAMPLE BYPASS
FIA EXHAUST
ROTAMETER
BACK PRESSURE REGULATOR
TEFLON HEAD SAMPLE PUMP
FLAME IONIZATION ANALYZER
DATA ACQUISITION SYSTEM
CHART RECORDER
EXHAUST
CONTROL VALVE
ANALYZER GAS INJECTION VALVE
FUGITIVE EMISSION POINT 1
FUGITIVE EMISSION POINT 2
FUGITIVE EMISSION POINT 3
FUGITIVE EMISSION POINT 4
FUGITIVE EMISSION POINT 5
PUMP
AIR
FUEL
NEEDLE VALVES
ROTAMETERS
SAMPLING MANIFOLD
THREE WAY VALVES

shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.

2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.

2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the flame ionization analyzer (FIA), and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.

2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.1.7.1 Zero Drift. Less than ±3.0 percent of the span value.

2.1.7.2 Calibration Drift. Less than ±3.0 percent of the span value.

2.1.7.3 Calibration Error. Less than ±5.0 percent of the calibration gas value.

2.1.7.4 Response Time. Less than 30 seconds.

2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.1.9 **Calibration and Other Gases.** Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to NIST standards and shall be certified by the manufacturer to ±1 percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval.

2.1.9.1 **Fuel.** A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.1.9.2 **Carrier Gas.** High purity air with less than 1 ppm of organic material (as propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

2.1.9.3 **FIA Linearity Calibration Gases.** Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown that more accurate measurements would be achieved.

2.1.10 **Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.

2.2 Fugitive Emissions Volumetric Flow Rate.

2.2.1 **Method 2 or 2A Apparatus.** For determining volumetric flow rate.

2.2.2 **Method 3 Apparatus and Reagents.** For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if it can be justified.

2.2.3 **Method 4 Apparatus and Reagents.** For determining moisture content, if necessary.

2.3 Temporary Total Enclosure.

The criteria for designing a TTE are discussed in Procedure T.

3. DETERMINATION OF VOLUMETRIC FLOW RATE OF FUGITIVE EMISSIONS

3.1 Locate all points where emissions are exhausted from the TTE. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.

3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.

4. DETERMINATION OF VOC CONTENT OF FUGITIVE EMISSIONS

4.1 **Analysis Duration.** Measure the VOC responses at each fugitive emission point during the entire test run or, if applicable, while the process is operating. If there are multiple emission locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.

4.2 Gas VOC Concentration.

4.2.1 Assemble the sample train as shown in Figure 1. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3, respectively.

4.2.2 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

4.2.3 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.

4.2.4 Conduct a system check before and a system drift check after each sampling run according to the procedures in Sections 5.2 and 5.3. If the drift check following a run indicates unacceptable performance, the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.

4.2.5 Verify that the sample lines, filter, and pump temperatures are 120 ± 5°C.

4.2.6 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least 4 separate measurements from each sample point during each hour of testing. Disregard the response measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

4.3 Background Concentration.

4.3.1 Determination of VOC Background Concentration.

4.3.1.1 Locate all NDO's of the TTE. A sampling point shall be centrally located outside of the TTE at 4 equivalent diameters from each NDO, if possible. If there are more than 6 NDO's, choose 6 sampling points evenly spaced among the NDO's.

4.3.1.2 Assemble the sample train as shown in Figure 2. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3.

4.3.1.3 Position the probe at the sampling location.

4.3.1.4 Determine the response time, conduct the system check and sample according to the procedures described in Sections 4.2.3 to 4.2.6.

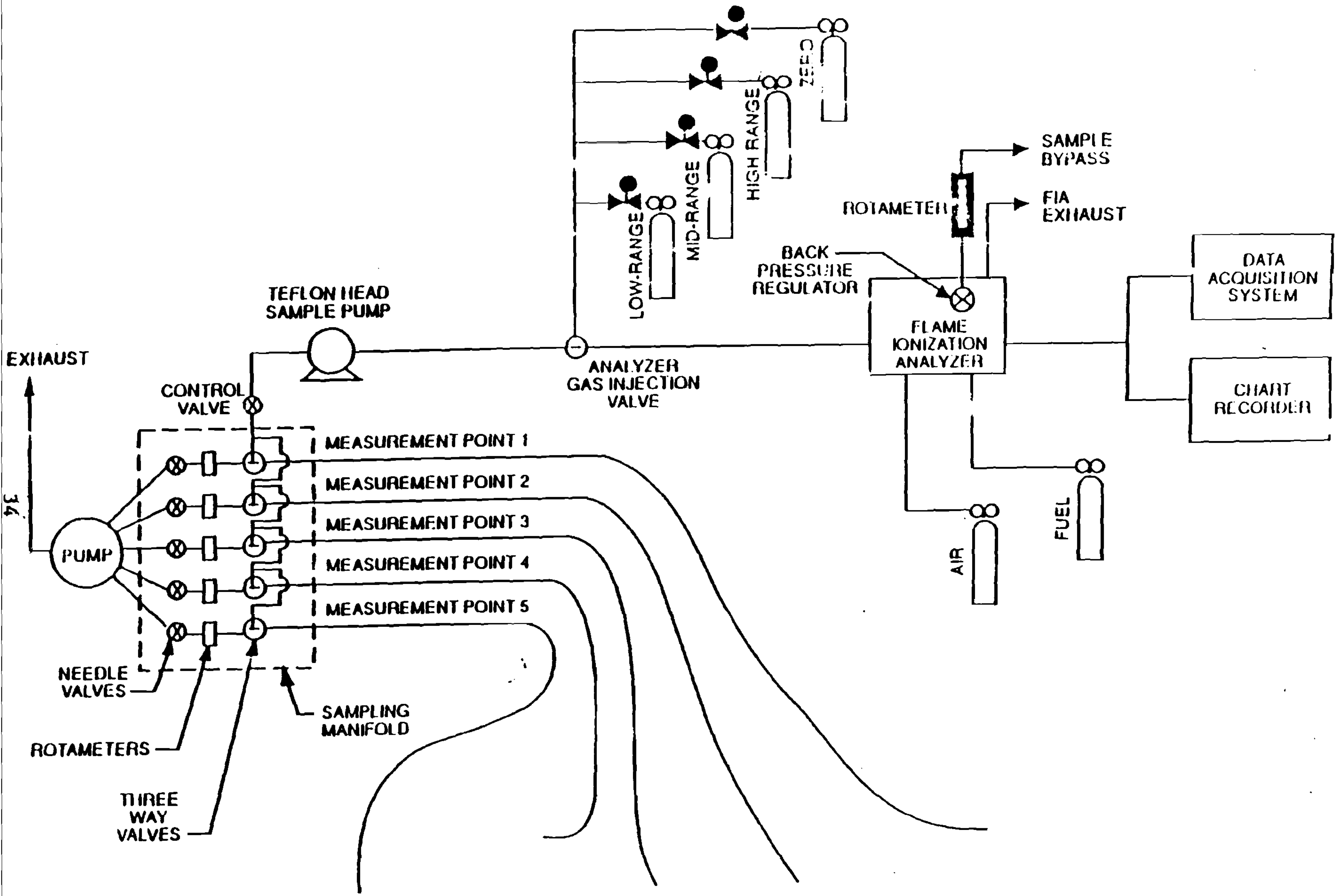


Figure 2 Backgrou   easurement system

4.4 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

5. CALIBRATION AND QUALITY ASSURANCE

5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 Systems Drift Checks. Select the calibration gas concentration that most closely approximates that of the fugitive gas emissions to conduct the drift checks. Introduce the zero and calibration gas at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct a system drift check at the end of each run.

5.3 System Check. Inject the high range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high range calibration gas. Conduct a system check before each test run.

5.4 Analysis Audit. Immediately before each test analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

6. NOMENCLATURE

A_i = area of NDO i, ft^2.

A_N = total area of all NDO's in the enclosure, ft^2.

C_{Bi} = corrected average VOC concentration of background emissions at point i, ppm propane.

C_B = average background concentration, ppm propane.

C_{OH} = average measured concentration for the drift check calibration gas, ppm propane.

C_{OO} = average system drift check concentration for zero concentration gas, ppm propane.

C_{Fj} = corrected average VOC concentration of fugitive emissions at point j, ppm propane.

C_H = actual concentration of the drift check calibration gas, ppm propane.

C_i = uncorrected average background VOC concentration at point i, ppm propane.

C_j = uncorrected average VOC concentration measured at point j, ppm propane.

F = total VOC content of fugitive emissions, kg.

K_1 = 1.830×10^{-6} kg/(m^3-ppm).

n = number of measurement points.

Q_{Fj} = average effluent volumetric flow rate corrected to standard conditions at fugitive emissions point j, m^3/min.

θ_F = total duration of fugitive emissions sampling run, min.

7. CALCULATIONS

7.1 Total VOC Fugitive Emissions.

$$F = \sum_{j=1}^{n} (C_{Fj} - C_B)\, Q_{Fj}\, \theta_F\, K_1 \qquad \text{Eq. 1}$$

7.2 VOC Concentration of the Fugitive Emissions at Point j.

$$C_{Fj} = (C_j - C_{OO}) \frac{C_H}{C_{OH} - C_{OO}} \qquad \text{Eq. 2}$$

7.3 Background VOC Concentration at Point i.

$$C_{Bi} = (C_i - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \qquad \text{Eq. 3}$$

7.4 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^{n} C_{Bi} A_i}{n A_N} \qquad \text{Eq. 4}$$

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, the terms "A_i" and "A_N" may be deleted from Equation 4.

VOC CAPTURE EFFICIENCY
Procedure F.2 - Fugitive VOC Emissions from Building Enclosures

1. INTRODUCTION

1.1 Applicability. This procedure is applicable for determining the fugitive volatile organic compounds (VOC) emissions from a building enclosure (BE). It is intended to be used as a segment in the development of liquid/gas or gas/gas protocols for determining VOC capture efficiency (CE) for surface coating and printing operations.

1.2 Principle. The total amount of fugitive VOC emissions (F_B) from the BE is calculated as the sum of the products of the VOC content (C_{Fj}) of each fugitive emissions point, its flow rate (Q_{Fj}), and time (θ_F).

1.3 Measurement Uncertainty. The measurement uncertainties are estimated for each fugitive emissions point as follows: Q_{Fj} = ±5.0 percent and C_{Fj} = ±5.0 percent. Based on these numbers, the probable uncertainty for F_B is estimated at about ±11.2 percent.

1.4 Sampling Requirements. A capture efficiency test shall consist of at least three sampling runs. The sampling time for each run should be at least 3 hours, unless otherwise approved.

1.5 Notes. Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing appropriate equipment and installing and using the equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

2. APPARATUS AND REAGENTS

2.1 Gas VOC Concentration. A schematic of the measurement system is shown in Figure 1. The main components are described below:

2.1.1 Sample Probe. Stainless steel, or equivalent. The probe shall be heated to prevent VOC condensation.

2.1.2 **Calibration Valve Assembly.** Three-way valve assembly at the outlet of sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

2.1.3 Sample Line. Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.

2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.

2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow rate control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.

2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the flame ionization analyzer (FIA), and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold must be heated to prevent condensation.

2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.1.7.1 Zero Drift. Less than ±3.0 percent of the span value.

2.1.7.2 Calibration Drift. Less than ±3.0 percent of the span value.

2.1.7.3 Calibration Error. Less than ±5.0 percent of the calibration gas value.

2.1.7.4 Response Time. Less than 30 seconds.

2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to NIST standards and shall be certified by the manufacturer to ±1 percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval.

2.1.9.1 Fuel. A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.1.9.2 **Carrier Gas.** High purity air with less than 1 ppm of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

2.1.9.3 **FIA Linearity Calibration Gases.** Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown that more accurate measurements would be achieved.

2.1.10 **Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.

2.2 Fugitive Emissions Volumetric Flow Rate.

2.2.1 **Flow Direction Indicators.** Any means of indicating inward or outward flow, such as light plastic film or paper streamers, smoke tubes, filaments, and sensory perception.

2.2.2 **Method 2 or 2A Apparatus.** For determining volumetric flow rate. Anemometers or similar devices calibrated according to the manufacturer's instructions may be used when low velocities are present. Vane anemometers (Young-maximum response propeller), specialized pitots with electronic manometers (e.g., Shortridge Instruments Inc., Airdata Multimeter 860) are commercially available with measurement thresholds of 15 and 8 mpm (50 and 25 fpm), respectively.

2.2.3 **Method 3 Apparatus and Reagents.** For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if it can be justified.

2.2.4 **Method 4 Apparatus and Reagents.** For determining moisture content, if necessary.

3. DETERMINATION OF VOLUMETRIC FLOW RATE OF FUGITIVE EMISSIONS

3.1 **Preliminary Determinations.** The purpose of this exercise is to determine which exhaust points should be measured for volumetric flow rates and VOC concentrations.

3.1.1 **Forced Draft Openings.** Identify all forced draft openings. Determine the volumetric flow rate according to Method 2.

3.1.2 **NDO's Exhaust Points.** The NDO's in the roof of a facility are considered to be exhaust points. Determine volumetric flow rate from these NDO's. Divide the cross-sectional area according to Method 1 using 12 equal areas. Use the appropriate velocity measurement devices, e.g., propeller anemometers.

3.1.3 Other NDO's.

3.1.3.1 This step is optional. Determine the exhaust flow rate, including that of the control device, from the enclosure and the intake air flow rate. If the exhaust flow rate divided by the intake air flow rate is greater than 1.1, then all other NDO's are not considered to be significant exhaust points.

3.1.3.2 If the option above is not taken, identify all other NDO's and other potential points through which fugitive emissions may escape the enclosure. Then use the following criteria to determine whether flow rates and VOC concentrations need to be measured:

3.1.3.2.1 Using the appropriate flow direction indicator, determine the flow direction. An NDO with zero or inward flow is not an exhaust point.

3.1.3.2.2 Measure the outward volumetric flow rate from the remainder of the NDO's. If the collective flow rate is 2 percent, or less, of the flow rate from Sections 3.1.1 and 3.1.2, then these NDO's, except those within two equivalent diameters (based on NDO opening) from VOC sources, may be considered to be non-exhaust points.

3.1.3.2.3 If the percentage calculated in Section 3.1.3.2.2 is greater than 2 percent, those NDO's (except those within two equivalent diameters from VOC sources) whose volumetric flow rate total 2 percent of the flow rate from Sections 3.1.1 and 3.1.2 may be considered as non-exhaust points. All remaining NDO's shall be measured for volumetric flow rate and VOC concentrations during the CE test.

3.1.3.2.4 The tester may choose to measure VOC concentrations at the forced exhaust points and the NDO's. If the total VOC emissions from the NDO's are less than 2 percent of the emissions from the forced draft and roof NDO's, then these NDO's may be eliminated from further consideration.

3.2 Determination of Flow Rates.

3.2.1 Measure the volumetric flow rate at all locations identified as exhaust points in Section 3.1. Divide each exhaust opening into 9 equal areas for rectangular openings and 8 for circular openings.

3.2.2 Measure the velocity at each site at least once every hour during each sampling run using Method 2 or 2A, if applicable, or using the low velocity instruments in Section 2.2.2.

4. DETERMINATION OF VOC CONTENT OF FUGITIVE EMISSIONS

4.1 **Analysis Duration.** Measure the VOC responses at each fugitive emission point during the entire test run or, if applicable, while the process is operating. If there are multiple emissions locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.

4.2 Gas VOC Concentration.

4.2.1 Assemble the sample train as shown in Figure 1. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3, respectively.

4.2.2 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

4.2.3 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.

4.2.4 Conduct a system check before and a system drift check after each sampling run according to the procedures in Sections 5.2 and 5.3. If the drift check following a run indicates unacceptable performance, the run is not valid. The tester may elect to perform drift checks during the run not to exceed one drift check per hour.

4.2.5 Verify that the sample lines, filter, and pump temperatures are 120 ± 5°C.

4.2.6 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least 4 separate measurements from each sample point during each hour of testing. Disregard the response measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

4.3 Alternative Procedure The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

5. CALIBRATION AND QUALITY ASSURANCE

5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 Systems Drift Checks. Select the calibration gas that most closely approximates the concentration of the captured emissions for conducting the drift checks. Introduce the zero and calibration gas at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct a system drift check at the end of each run.

5.3 System Check. Inject the high range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high range calibration gas. Conduct a system check before each test run.

5.4 Analysis Audit. Immediately before each test analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

6. NOMENCLATURE

C_{DH} = average measured concentration for the drift check calibration gas, ppm propane.

C_{D0} = average system drift check concentration for zero concentration gas, ppm propane.

C_{Fj} = corrected average VOC concentration of fugitive emissions at point j, ppm propane.

C_H = actual concentration of the drift check calibration gas, ppm propane.

C_j = uncorrected average VOC concentration measured at point j, ppm propane.

F_B = total VOC content of fugitive emissions from the building, kg.

K_1 = 1.830×10^{-6} kg/(m^3-ppm).

n = number of measurement points.

Q_{Fj} = average effluent volumetric flow rate corrected to standard conditions at fugitive emissions point j, m^3/min.

θ_F = total duration of capture efficiency sampling run, min.

7. CALCULATIONS

7.1 Total VOC Fugitive Emissions From the Building.

$$F_g = \sum_{j=1}^{n} C_{Fj} \, Q_{Fj} \, \theta_F \, K_1 \qquad \text{Eq. 1}$$

7.2 VOC Concentration of the Fugitive Emissions at Point j.

$$C_{Fj} = (C_j - C_{DO}) \frac{C_H}{C_{OH} - C_{DO}} \qquad \text{Eq. 2}$$

VOC CAPTURE EFFICIENCY
Procedure L - VOC Input

1. INTRODUCTION

1.1 Applicability. This procedure is applicable for determining the input of volatile organic compounds (VOC). It is intended to be used as a segment in the development of liquid/gas protocols for determining VOC capture efficiency (CE) for surface coating and printing operations.

1.2 Principle. The amount of VOC introduced to the process (L) is the sum of the products of the weight (W) of each VOC containing liquid (ink, paint, solvent, etc.) used and its VOC content (V). A sample of each VOC containing liquid is analyzed with a flame ionization analyzer (FIA) to determine V.

1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each VOC containing liquid as follows: W = ±2.0 percent and V = ±12.0 percent. Based on these numbers, the probable uncertainty for L is estimated at about ±12.2 percent for each VOC containing liquid.

1.4 **Sampling Requirements.** A capture efficiency test shall consist of at least three sampling runs. The sampling time for each run should be at least 8 hours, unless otherwise approved.

1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing appropriate equipment and installing and using the equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

2. APPARATUS AND REAGENTS

2.1 **Liquid Weight.**

2.1.1 **Balances/Digital Scales.** To weigh drums of VOC containing liquids to within 0.2 lb.

2.1.2 **Volume Measurement Apparatus (Alternative).** Volume meters, flow meters, density measurement equipment, etc., as needed to achieve same accuracy as direct weight measurements.

2.2 **VOC Content (Flame Ionization Analyzer Technique).** The liquid sample analysis system is shown in Figures 1 and 2. The following equipment is required:

2.2.1 **Sample Collection Can.** An appropriately sized metal can to be used to collect VOC containing materials. The can must be constructed in such a way that it can be grounded to the coating container.

2.2.2 **Needle Valves.** To control gas flow.

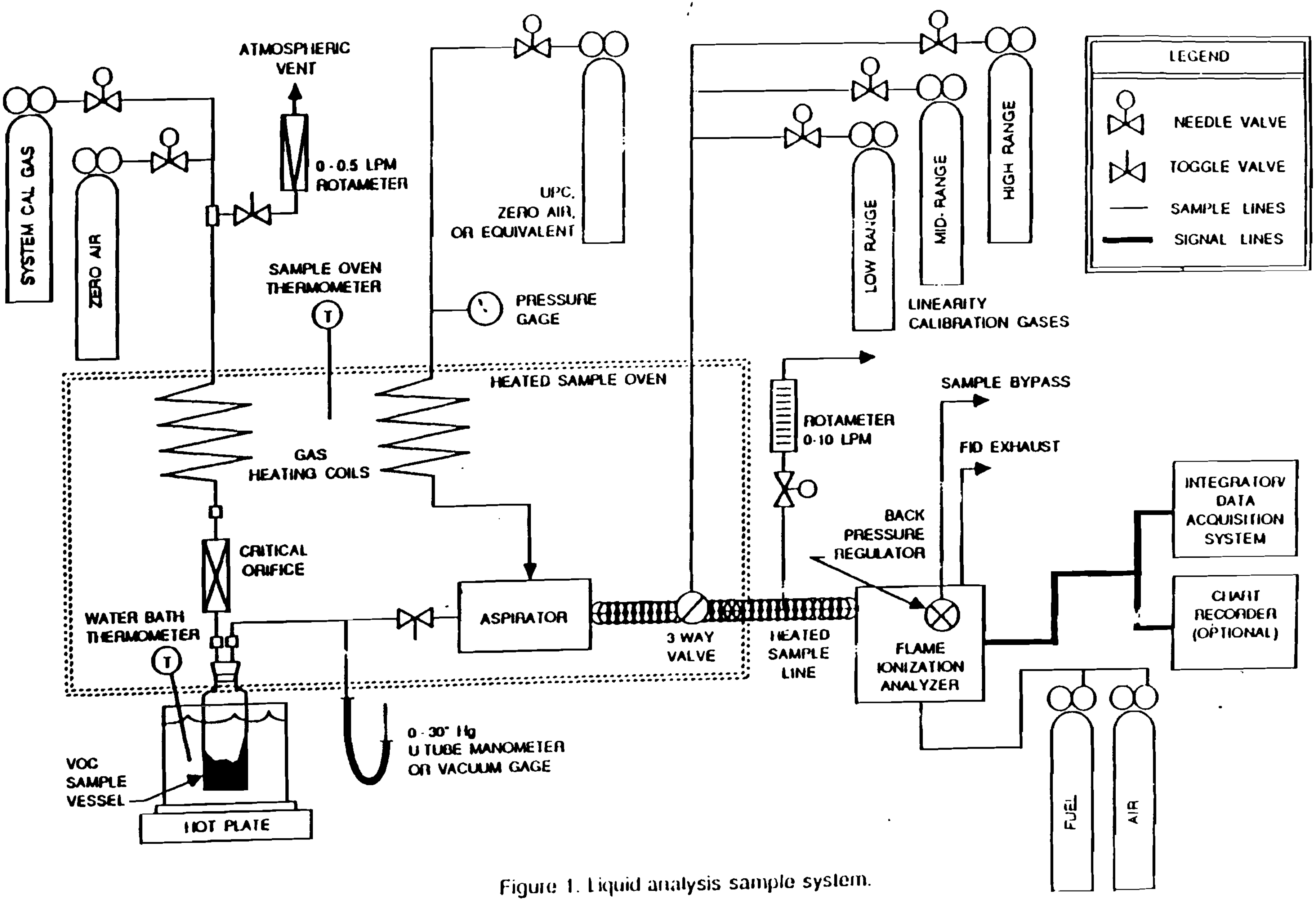


Figure 1. Liquid analysis sample system.

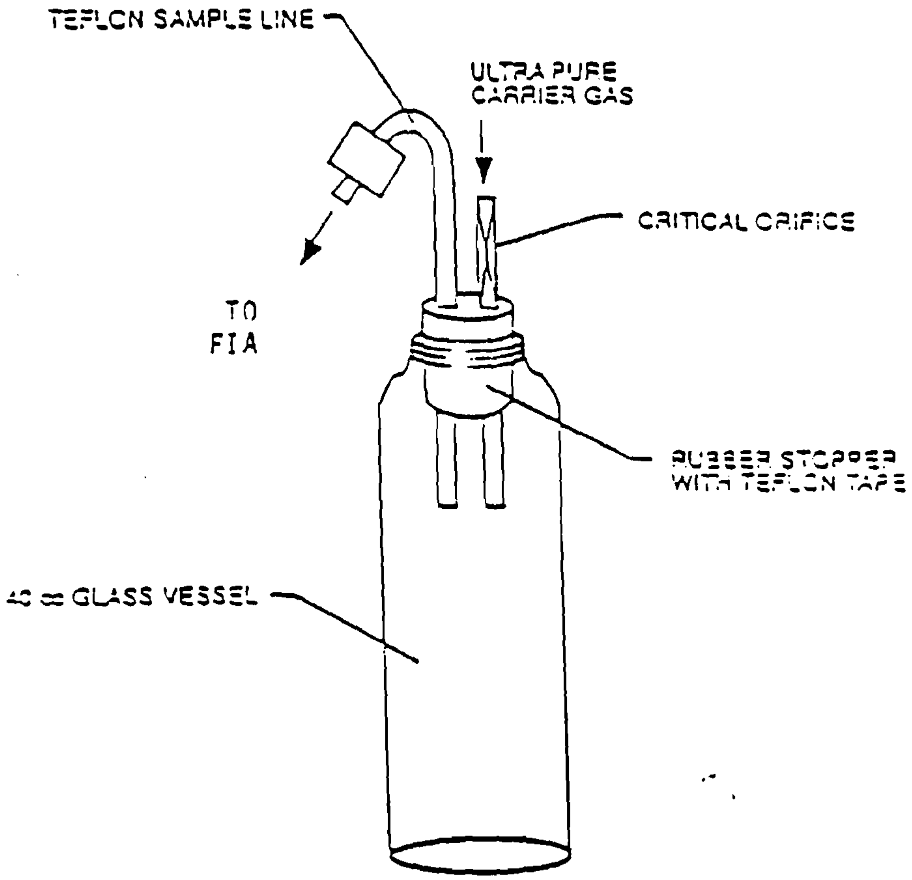


Figure 2. VOC sampling vessel.

2.2.3 **Regulators.** For carrier gas and calibration gas cylinders.

2.2.4 **Tubing.** Teflon or stainless steel tubing with diameters and lengths determined by connection requirements of equipment. The tubing between the sample oven outlet and the FIA shall be heated to maintain a temperature of 120 ± 5°C.

2.2.5 **Atmospheric Vent.** A tee and 0- to 0.5-liter/min rotameter placed in the sampling line between the carrier gas cylinder and the VOC sample vessel to release the excess carrier gas. A toggle valve placed between the tee and the rotameter facilitates leak tests of the analysis system.

2.2.6 **Thermometer.** Capable of measuring the temperature of the hot water bath to within 1°C.

2.2.7 **Sample Oven.** Heated enclosure, containing calibration gas coil heaters, critical orifice, aspirator, and other liquid sample analysis components, capable of maintaining a temperature of 120 ± 5°C.

2.2.8 **Gas Coil Heaters.** Sufficient lengths of stainless steel or Teflon tubing to allow zero and calibration gases to be heated to the sample oven temperature before entering the critical orifice or aspirator.

2.2.9 **Water Bath.** Capable of heating and maintaining a sample vessel temperature of 100 ± 5°C.

2.2.10 **Analytical Balance.** To measure ±0.001 g.

2.2.11 **Disposable Syringes.** 2-cc or 5-cc.

2.2.12 **Sample Vessel.** Glass, 40-ml septum vial. A separate vessel is needed for each sample.

2.2.13 **Rubber Stopper.** Two-hole stopper to accommodate 3.2-mm (1/8-in.) Teflon tubing, appropriately sized to fit the opening of the sample vessel. The rubber stopper should be wrapped in Teflon tape to provide a tighter seal and to prevent any reaction of the sample with the rubber stopper. Alternatively, any leak-free closure fabricated of non-reactive materials and accommodating the necessary tubing fittings may be used.

2.2.14 **Critical Orifices.** Calibrated critical orifices capable of providing constant flow rates from 50 to 250 ml/min at known pressure drops. Sapphire orifice assemblies (available from O'Keefe Controls Company) and glass capillary tubing have been found to be adequate for this application.

2.2.15 **Vacuum Gauge.** 0- to 760-mm (0- to 30-in.) Hg U-Tube manometer or vacuum gauge.

2.2.16 **Pressure Gauge.** Bourdon gauge capable of measuring the maximum air pressure at the aspirator inlet (e.g., 100 psig).

2.2.17 **Aspirator.** A device capable of generating sufficient vacuum at the sample vessel to create critical flow through the calibrated orifice when sufficient air pressure is present at the aspirator inlet. The aspirator must also provide sufficient sample pressure to operate the FIA. The sample is also mixed with the dilution gas within the aspirator.

2.2.18 **Soap Bubble Meter.** Of an appropriate size to calibrate the critical orifices in the system.

2.2.19 **Organic Concentration Analyzer.** An FIA with a span value of 1.5 times the expected concentration as propane; however other span values may be used if it can be demonstrated that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.2.19.1 **Zero Drift.** Less than ±3.0 percent of the span value.

2.2.19.2 **Calibration Drift.** Less than ±3.0 percent of span value.

2.2.19.3 **Calibration Error.** Less than ±5.0 percent of the calibration gas value.

2.2.20 **Integrator/Data Acquisition System.** An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.2.21 **Chart Recorder (Optional).** A chart recorder or similar device is recommended to provide a continuous analog display of the measurement results during the liquid sample analysis.

2.2.22 **Calibration and Other Gases.** For calibration, fuel, and combustion air (if required) contained in compressed gas cylinders. All calibration gases shall be traceable to NIST standards and shall be certified by the manufacturer to ±1 percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval.

2.2.22.1 **Fuel.** A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.2.22.2 **Carrier Gas.** High purity air with less than 1 ppm of organic material (as propane) or less than 0.1 percent of the span value, whichever is greater.

2.2.22.3 FIA Linearity Calibration Gases. Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown that more accurate measurements would be achieved.

2.2.22.4 System Calibration Gas. Gas mixture standard containing propane in air, approximating the undiluted VOC concentration expected for the liquid samples.

3. DETERMINATION OF LIQUID INPUT WEIGHT

3.1 Weight Difference. Determine the amount of material introduced to the process as the weight difference of the feed material before and after each sampling run. In determining the total VOC containing liquid usage, account for: (a) the initial (beginning) VOC containing liquid mixture; (b) any solvent added during the test run; (c) any coating added during the test run; and (d) any residual VOC containing liquid mixture remaining at the end of the sample run.

3.1.1 Identify all points where VOC containing liquids are introduced to the process. To obtain an accurate measurement of VOC containing liquids, start with an empty fountain (if applicable). After completing the run, drain the liquid in the fountain back into the liquid drum (if possible), and weigh the drum again. Weigh the VOC containing liquids to ±0.5 percent of the total weight (full) or ±0.1 percent of the total weight of VOC containing liquid used during the sample run, whichever is less. If the residual liquid cannot be returned to the drum, drain the fountain into a preweighed empty drum to determine the final weight of the liquid.

3.1.2 If it is not possible to measure a single representative mixture, then weigh the various components separately (e.g., if solvent is added during the sampling run, weigh the solvent before it is added to the mixture). If a fresh drum of VOC containing liquid is needed during the run, then weigh both the empty drum and fresh drum.

3.2 Volume Measurement (Alternative). If direct weight measurements are not feasible, the tester may use volume meters and flow rate meters (and density measurements) to determine the weight of liquids used if it can be demonstrated that the technique produces results equivalent to the direct weight measurements. If a single representative mixture cannot be measured, measure the components separately.

4. DETERMINATION OF VOC CONTENT IN INPUT LIQUIDS

4.1 Collection of Liquid Samples.

4.1.1 Collect a 100-ml or larger sample of the VOC containing liquid mixture at each application location at the beginning and end of each test run. A separate sample should be taken of each VOC containing liquid added to the application mixture during the test run. If a fresh drum is needed during the sampling run, then obtain a sample from the fresh drum.

4.1.2 When collecting the sample, ground the sample container to the coating drum. Fill the sample container as close to the rim as possible to minimize the amount of headspace.

4.1.3 After the sample is collected, seal the container so the sample cannot leak out or evaporate.

4.1.4 Label the container to identify clearly the contents.

4.2 Liquid Sample VOC Content.

4.2.1 Assemble the liquid VOC content analysis system as shown in Figure 1.

4.2.2 Permanently identify all of the critical orifices that may be used. Calibrate each critical orifice under the expected operating conditions (i.e., sample vacuum and temperature) against a volume meter as described in Section 5.3.

4.2.3 Label and tare the sample vessels (including the stoppers and caps) and the syringes.

4.2.4 Install an empty sample vessel and perform a leak test of the system. Close the carrier gas valve and atmospheric vent and evacuate the sample vessel to 250 mm (10 in.) Hg absolute or less using the aspirator. Close the toggle valve at the inlet to the aspirator and observe the vacuum for at least one minute. If there is any change in the sample pressure, release the vacuum, adjust or repair the apparatus as necessary and repeat the leak test.

4.2.5 Perform the analyzer calibration and linearity checks according to the procedure in Section 5.1. Record the responses to each of the calibration gases and the back-pressure setting of the FIA.

4.2.6 Establish the appropriate dilution ratio by adjusting the aspirator air supply or substituting critical orifices. Operate the aspirator at a vacuum of at least 25 mm (1 in.) Hg greater than the vacuum necessary to achieve critical flow. Select the dilution ratio so that the maximum response of the FIA to the sample does not exceed the high-range calibration gas.

4.2.7 Perform system calibration checks at two levels by introducing compressed gases at the inlet to the sample vessel while the aspirator and dilution devices are operating. Perform these checks using the carrier gas (zero concentration) and the system calibration gas. If the response to the carrier gas exceeds ±0.5 percent of span, clean or repair the apparatus and repeat the check. Adjust the dilution ratio as necessary to achieve the correct response to the upscale check, but do not adjust the analyzer calibration. Record the identification of the orifice, aspirator air supply pressure, FIA back-pressure, and the responses of the FIA to the carrier and system calibration gases.

4.2.8 After completing the above checks, inject the system calibration gas for approximately 10 minutes. Time the exact duration of the gas injection using a stopwatch. Determine the area under the FIA response curve and calculate the system response factor based on the sample gas flow rate, gas concentration, and the duration of the injection as compared to the integrated response using Equations 2 and 3.

4.2.9 Verify that the sample oven and sample line temperatures are 120 ± 5°C and that the water bath temperature is 100° ± 5°C.

4.2.10 Fill a tared syringe with approximately 1 g of the VOC containing liquid and weigh it. Transfer the liquid to a tared sample vessel. Plug the sample vessel to minimize sample loss. Weigh the sample vessel containing the liquid to determine the amount of sample actually received. Also, as a quality control check, weigh the empty syringe to determine the amount of material delivered. The two coating sample weights should agree within ±0.02 g. If not, repeat the procedure until an acceptable sample is obtained.

4.2.11 Connect the vessel to the analysis system. Adjust the aspirator supply pressure to the correct value. Open the valve on the carrier gas supply to the sample vessel and adjust it to provide a slight excess flow to the atmospheric vent. As soon as the initial response of the FIA begins to decrease, immerse the sample vessel in the water bath. (Applying heat to the sample vessel too soon may cause the FID response to exceed the calibrated range of the instrument, and thus invalidate the analysis.)

4.2.12 Continuously measure and record the response of the FIA until all of the volatile material has been evaporated from the sample and the instrument response has returned to the baseline (i.e., response less than 0.5 percent of the span value). Observe the aspirator supply pressure, FIA back-pressure, atmospheric vent, and other system operating parameters during the run; repeat the analysis procedure if any of these parameters deviate from the values established during the system calibration checks in Section 4.2.7. After each sample perform the drift check described in Section 5.2. If the drift check results are acceptable, calculate the VOC content of the sample using the equations in Section 7. Integrate the area under the FIA response curve, or determine the average concentration response and the duration of sample analysis.

5. CALIBRATION AND QUALITY ASSURANCE

5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 Systems Drift Checks. After each sample, repeat the system calibration checks in Section 4.2.7 before any adjustments to the FIA or measurement system are made. If the zero or calibration drift exceeds ±3 percent of the span value, discard the result and repeat the analysis.

5.3 Critical Orifice Calibration.

5.3.1 Each critical orifice must be calibrated at the specific operating conditions that it will be used. Therefore, assemble all components of the liquid sample analysis system as shown in Figure 3. A stopwatch is also required.

5.3.2 Turn on the sample oven, sample line, and water bath heaters and allow the system to reach the proper operating temperature. Adjust the aspirator to a vacuum of 380 mm (15 in.) Hg vacuum. Measure the time required for one soap bubble to move a known distance and record barometric pressure.

5.3.3 Repeat the calibration procedure at a vacuum of 406 mm (16 in.) Hg and at 25-mm (1-in.) Hg intervals until three consecutive determinations provide the same flow rate. Calculate the critical flow rate for the orifice in ml/min at standard conditions. Record the vacuum necessary to achieve critical flow.

6. NOMENCLATURE

A_L = area under the response curve of the liquid sample, area count.

A_S = area under the response curve of the calibration gas, area count.

C_S = actual concentration of system calibration gas, ppm propane.

$K = 1.830 \times 10^{-9}$ g/(ml-ppm).

L = total VOC content of liquid input, kg.

M_L = mass of liquid sample delivered to the sample vessel, g.

q = flow rate through critical orifice, ml/min.

RF = liquid analysis system response factor, g/area count.

θ_S = total gas injection time for system calibration gas during integrator calibration, min.

V_{Fj} = final VOC fraction of VOC containing liquid j.

V_{Ij} = initial VOC fraction of VOC containing liquid j.

V_{Aj} = VOC fraction of VOC containing liquid j added during the run.

V = VOC fraction of liquid sample.
W_{Fj} = weight of VOC containing liquid j remaining at end of the run, kg.
W_{Ij} = weight of VOC containing liquid j at beginning of the run, kg.
W_{Aj} = weight of VOC containing liquid j added during the run, kg.

7. CALCULATIONS

7.1 Total VOC Content of the Input VOC Containing Liquid.

$$L = \sum_{j=1}^{n} V_{Ij} W_{Ij} - \sum_{j=1}^{n} V_{Fj} W_{Fj} + \sum_{j=1}^{n} V_{Aj} W_{Aj} \qquad \text{Eq. 1}$$

7.2 Liquid Sample Analysis System Response Factor for Systems Using Integrators, Grams/Area Counts.

$$RF = \frac{C_S \, q \, \theta_S \, K}{A_S} \qquad \text{Eq. 2}$$

7.3 VOC Content of the Liquid Sample.

$$V = \frac{A_L \, RF}{M_L} \qquad \text{Eq. 3}$$

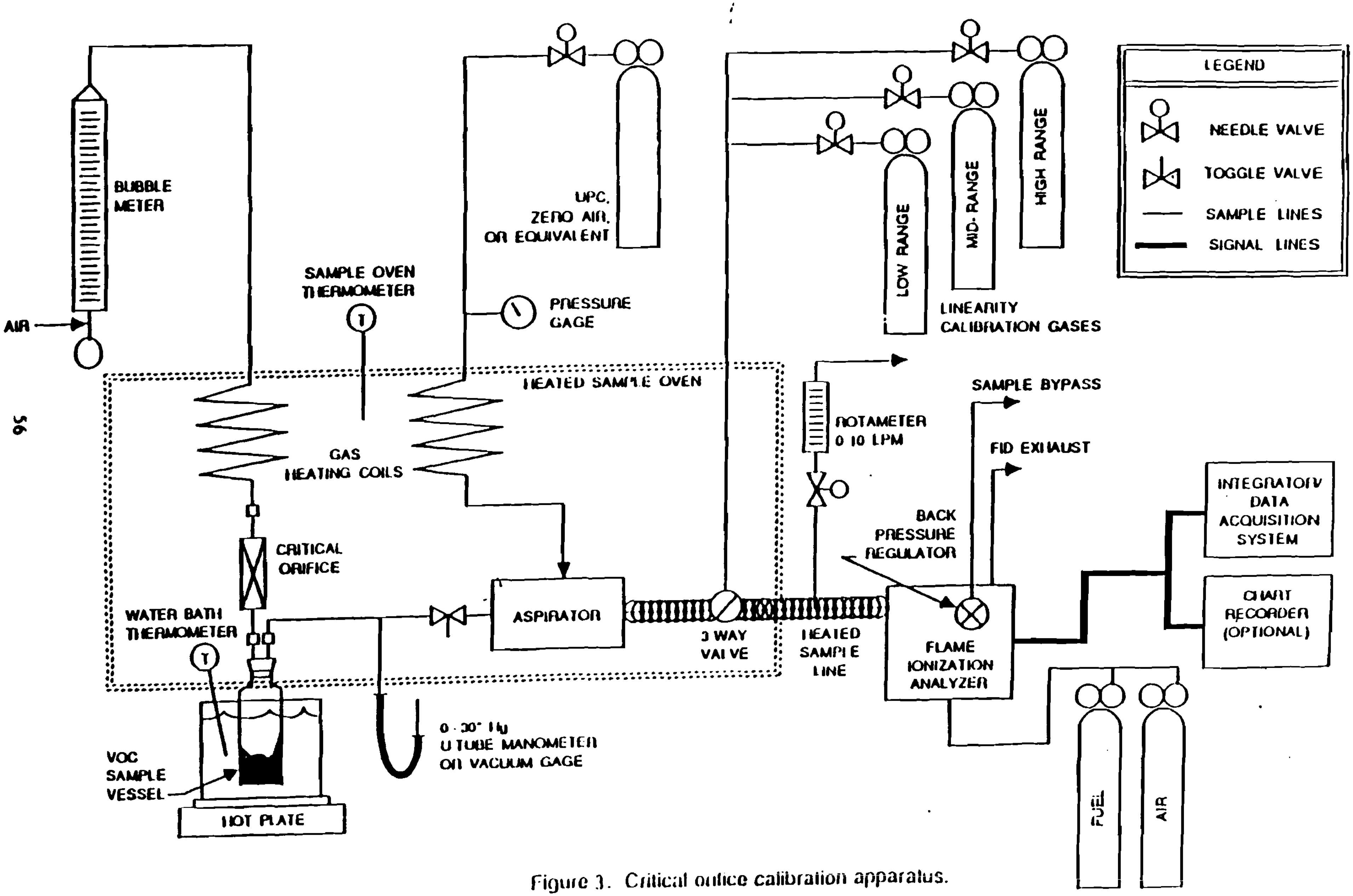


Figure 3. Critical orifice calibration apparatus.

VOC CAPTURE EFFICIENCY
Procedure G.1 - Captured VOC Emissions

1. INTRODUCTION

1.1 **Applicability.** This procedure is applicable for determining the volatile organic compounds (VOC) content of captured gas streams. It is intended to be used as a segment in the development of liquid/gas or gas/gas protocols for determining VOC capture efficiency (CE) for surface coating and printing operations. The procedure may not be acceptable in certain site-specific situations, e.g., when: (1) direct fired heaters or other circumstances affect the quantity of VOC at the control device inlet; and (2) particulate organic aerosols are formed in the process and are present in the captured emissions.

1.2 **Principle.** The amount of VOC captured (G) is calculated as the sum of the products of the VOC content (C_{Gj}), the flow rate (Q_{Gj}), and the sample time (θ_C) from each captured emissions point.

1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each captured or fugitive emissions point as follows: Q_{Gj} = ±5.5 percent and C_{Gj} = ±5.0 percent. Based on these numbers, the probable uncertainty for G is estimated at about ±7.4 percent.

1.4 **Sampling Requirements.** A capture efficiency test shall consist of at least three sampling runs. The sampling time for each run should be at least 8 hours, unless otherwise approved.

1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing appropriate equipment and installing and using the equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

2. APPARATUS AND REAGENTS

2.1 **Gas VOC Concentration.** A schematic of the measurement system is shown in Figure 1. The main components are described below:

2.1.1 **Sample Probe.** Stainless steel, or equivalent. The probe shall be heated to prevent VOC condensation.

2.1.2 **Calibration Valve Assembly.** Three-way valve assembly at the outlet of sample probe to direct the zero and calibration gases to the analyzer. Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

2.1.3 **Sample Line.** Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.

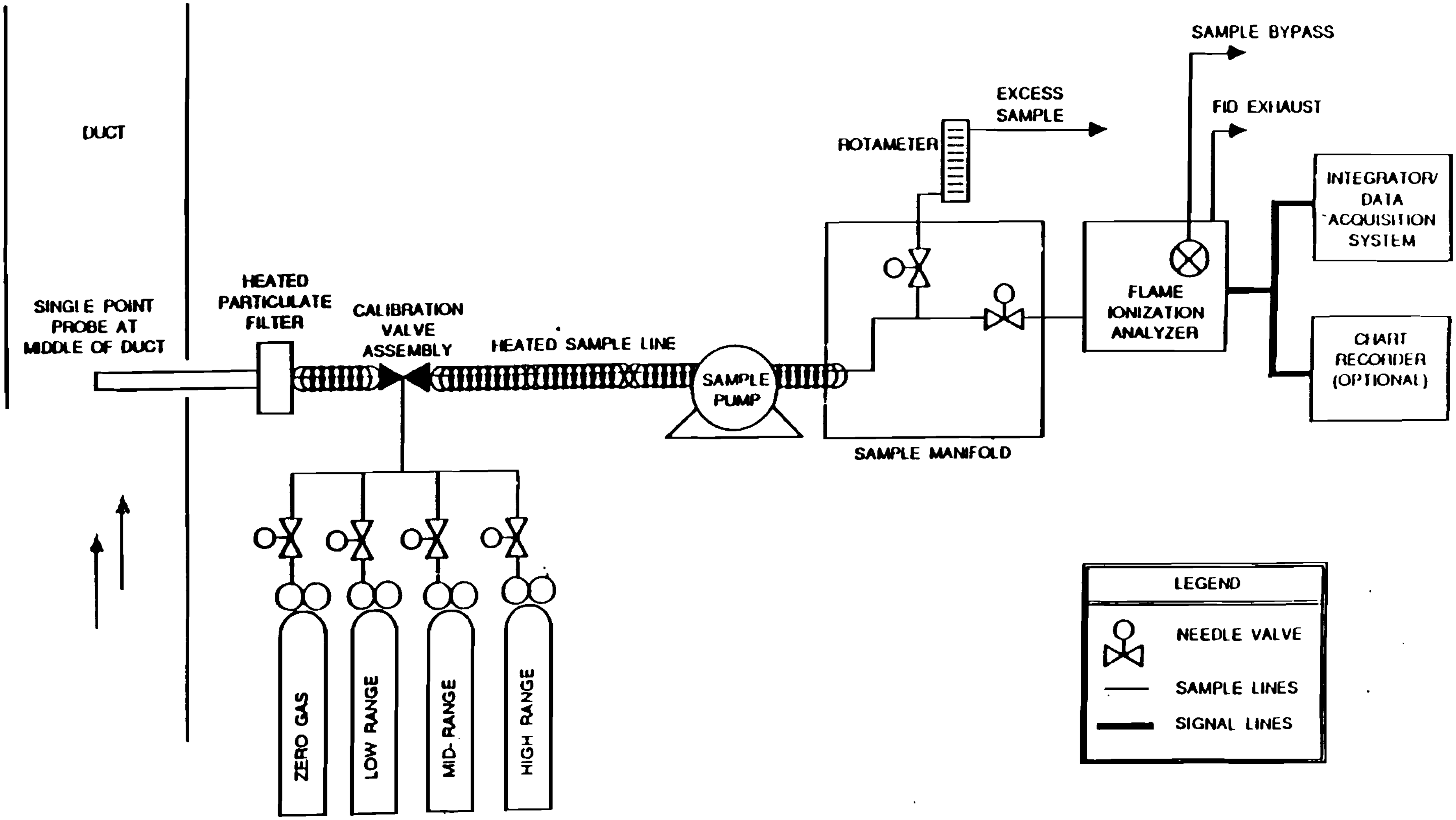


Figure 1. Gas VOC concentration measurement system.

2.1.4 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.

2.1.5 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow rate control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.

2.1.6 Sample Gas Manifold. Capable of diverting a portion of the sample gas stream to the flame ionization analyzer (FIA), and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If captured or fugitive emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.

2.1.7 Organic Concentration Analyzer. An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.1.7.1 Zero Drift. Less than ±3.0 percent of the span value.

2.1.7.2 Calibration Drift. Less than ±3.0 percent of the span value.

2.1.7.3 Calibration Error. Less than ±5.0 percent of the calibration gas value.

2.1.7.4 Response Time. Less than 30 seconds.

2.1.8 Integrator/Data Acquisition System. An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.1.9 Calibration and Other Gases. Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to NIST standards and shall be certified by the manufacturer to ±1 percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval.

2.1.9.1 **Fuel.** A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.1.9.2 **Carrier Gas.** High purity air with less than 1 ppm of organic material (as propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

2.1.9.3 **FIA Linearity Calibration Gases.** Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown that more accurate measurements would be achieved.

2.1.10 **Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.

2.2 **Captured Emissions Volumetric Flow Rate.**

2.2.1 **Method 2 or 2A Apparatus.** For determining volumetric flow rate.

2.2.2 **Method 3 Apparatus and Reagents.** For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if it can be justified.

2.2.3 **Method 4 Apparatus and Reagents.** For determining moisture content, if necessary.

3. DETERMINATION OF VOLUMETRIC FLOW RATE OF CAPTURED EMISSIONS

3.1 Locate all points where emissions are captured from the affected facility. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.

3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.

4. DETERMINATION OF VOC CONTENT OF CAPTURED EMISSIONS

4.1 **Analysis Duration.** Measure the VOC responses at each captured emissions point during the entire test run or, if applicable, while the process is operating. If there are multiple captured emission locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.

4.2 Gas VOC Concentration.

4.2.1 Assemble the sample train as shown in Figure 1. Calibrate the FIA according to the procedure in Section 5.1.

4.2.2 Conduct a system check according to the procedure in Section 5.3.

4.2.3 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

4.2.4 Inject zero gas at the calibration valve assembly. Allow the measurement system response to reach zero. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.

4.2.5 Conduct a system check before and a system drift check after each sampling run according to the procedures in Sections 5.2 and 5.3. If the. drift check following a run indicates unacceptable performance, the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.

4.2.6 Verify that the sample lines, filter, and pump temperatures are 120 ± 5°C.

4.2.7 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple captured emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least 4 separate measurements from each sample point during each hour of testing. Disregard the measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

4.3 Background Concentration.

4.3.1 Locate all NDO's of the TTE. A sampling point shall be centrally located outside of the TTE at 4 equivalent diameters from each NDO, if possible. If there are more than 6 NDO's, choose 6 sampling points evenly spaced among the NDO's.

4.3.2 Assemble the sample train as shown in Figure 2. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.3. NOTE: This sample train shall be a separate sampling train from the one to measure the captured emissions.

4.3.3 Position the probe at the sampling location.

4.3.4 Determine the response time, conduct the system check and sample according to the procedures described in Sections 4.2.4 to 4.2.7.

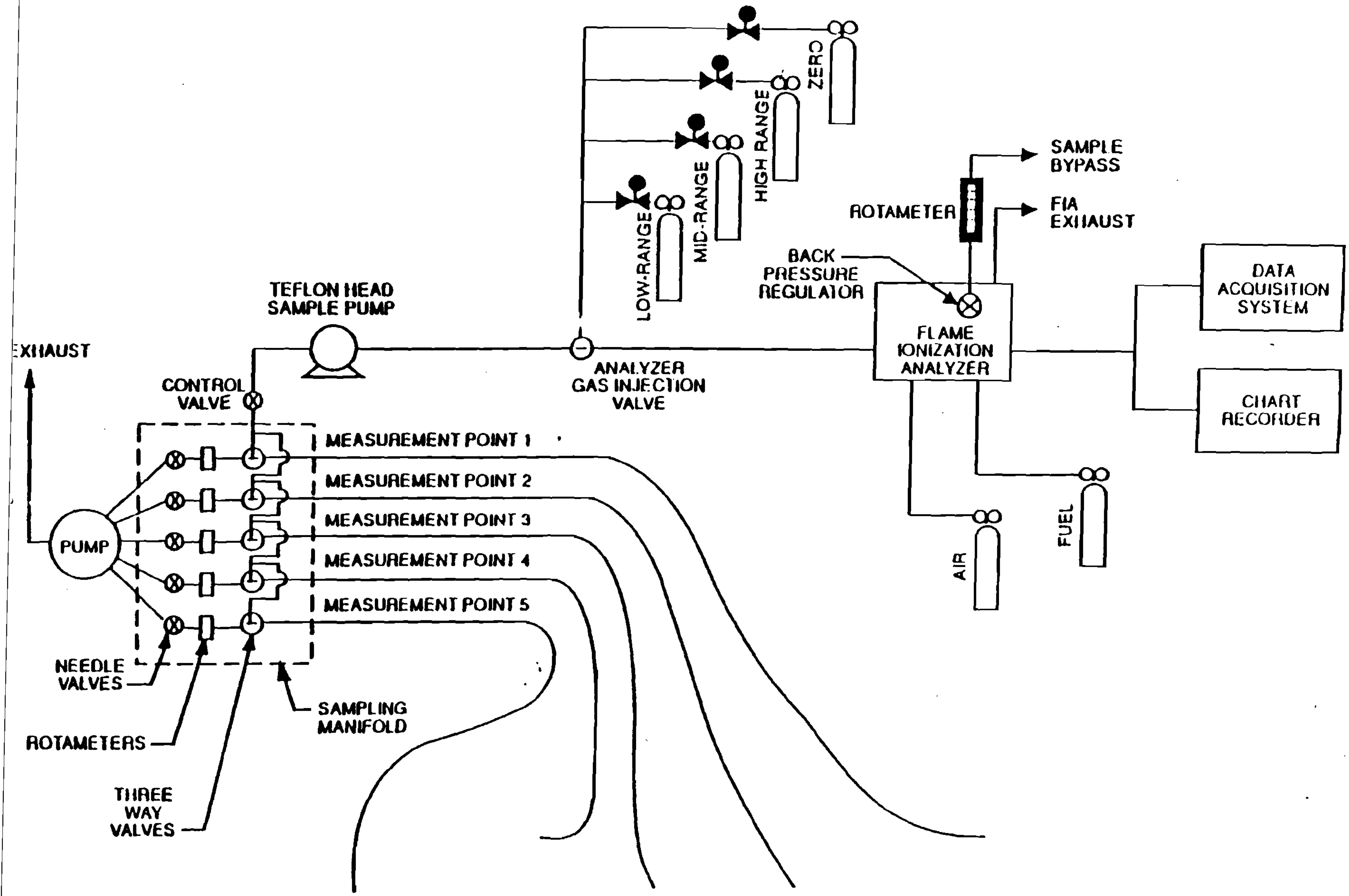


Figure 2. Background [illegible] urement system.

4.4 Alternative Procedure. The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

5. CALIBRATION AND QUALITY ASSURANCE

5.1 FIA Calibration and Linearity Check. Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 Systems Drift Checks. Select the calibration gas that most closely approximates the concentration of the captured emissions for conducting the drift checks. Introduce the zero and calibration gas at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct the system drift checks at the end of each run.

5.3 System Check. Inject the high range calibration gas at the inlet of the sampling probe and record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high range calibration gas. Conduct a system check before and after each test run.

5.4 Analysis Audit. Immediately before each test analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

6. NOMENCLATURE

A_i = area of NDO i, ft^2.

A_N = total area of all NDO's in the enclosure, ft^2.

C_{Bi} = corrected average VOC concentration of background emissions at point i, ppm propane.

C_B = average background concentration, ppm propane.

C_{Gj} = corrected average VOC concentration of captured emissions at point j, ppm propane.

C_{DH} = average measured concentration for the drift check calibration gas, ppm propane.

C_{DO} = average system drift check concentration for zero concentration gas, ppm propane.

C_H = actual concentration of the drift check calibration gas, ppm propane.

C_i = uncorrected average background VOC concentration measured at point i, ppm propane.

C_j = uncorrected average VOC concentration measured at point j, ppm propane.

G = total VOC content of captured emissions, kg.

K_1 = 1.830×10^{-6} kg/(m^3-ppm).

n = number of measurement points.

Q_{Gj} = average effluent volumetric flow rate corrected to standard conditions at captured emissions point j, m^3/min.

θ_C = total duration of captured emissions sampling run, min.

7. CALCULATIONS

7.1 Total VOC Captured Emissions.

$$G = \sum_{j=1}^{n} (C_{Gj} - C_B)\, Q_{Gj}\, \theta_C\, K_1 \qquad \text{Eq. 1}$$

7.2 VOC Concentration of the Captured Emissions at Point j.

$$C_{Gj} = (C_j - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \qquad \text{Eq. 2}$$

7.3 Background VOC Concentration at Point i.

$$C_{Bi} = (C_i - C_{DO}) \frac{C_H}{C_{DH} - C_{DO}} \qquad \text{Eq. 3}$$

7.4 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^{n} C_{Bi} A_i}{n A_N} \qquad \text{Eq. 4}$$

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, the terms "A_i" and "A_N" may be deleted from Equation 4.

VOC CAPTURE EFFICIENCY
Procedure G.2 - Captured VOC Emissions (Dilution Technique)

1. INTRODUCTION

1.1 **Applicability.** This procedure is applicable for determining the volatile organic compounds (VOC) content of captured gas streams. It is intended to be used as a segment in the development of a gas/gas protocol in which fugitive emissions are measured for determining VOC capture efficiency (CE) for surface coating and printing operations. A dilution system is used to reduce the VOC concentration of the captured emission to about the same concentration as the fugitive emissions. The procedure may not be acceptable in certain site-specific situations, e.g., when: (1) direct fired heaters or other circumstances affect the quantity of VOC at the control device inlet; and (2) particulate organic aerosols are formed in the process and are present in the captured emissions.

1.2 **Principle.** The amount of VOC captured (G) is calculated as the sum of the products of the VOC content (C_{Gj}), the flow rate (Q_{Gj}), and the sampling time (θ_C) from each captured emissions point.

1.3 **Estimated Measurement Uncertainty.** The measurement uncertainties are estimated for each captured or fugitive emissions point as follows: Q_{Gj} = ±5.5 percent and C_{Gj} = ±5 percent. Based on these numbers, the probable uncertainty for G is estimated at about ±7.4 percent.

1.4 **Sampling Requirements.** A capture efficiency test shall consist of at least three sampling runs. The sampling time for each run should be at least 8 hours, unless otherwise approved.

1.5 **Notes.** Because this procedure is often applied in highly explosive areas, caution and care should be exercised in choosing appropriate equipment and installing and using the equipment. Mention of trade names or company products does not constitute endorsement. All gas concentrations (percent, ppm) are by volume, unless otherwise noted.

2. APPARATUS AND REAGENTS

2.1 **Gas VOC Concentration.** A schematic of the measurement system is shown in Figure 1. **The main** components are described below:

2.1.1 **Dilution System.** A Kipp in-stack dilution probe and controller or similar device may be used. The dilution rate may be changed by substituting different critical orifices or adjustments of the aspirator supply pressure. The dilution system shall be heated to prevent VOC condensation. Note: An out-of-stack dilution device may be used.

2.1.2 **Calibration Valve Assembly.** Three-way valve assembly at the outlet of sample probe to direct the zero and calibration gases to the analyzer.

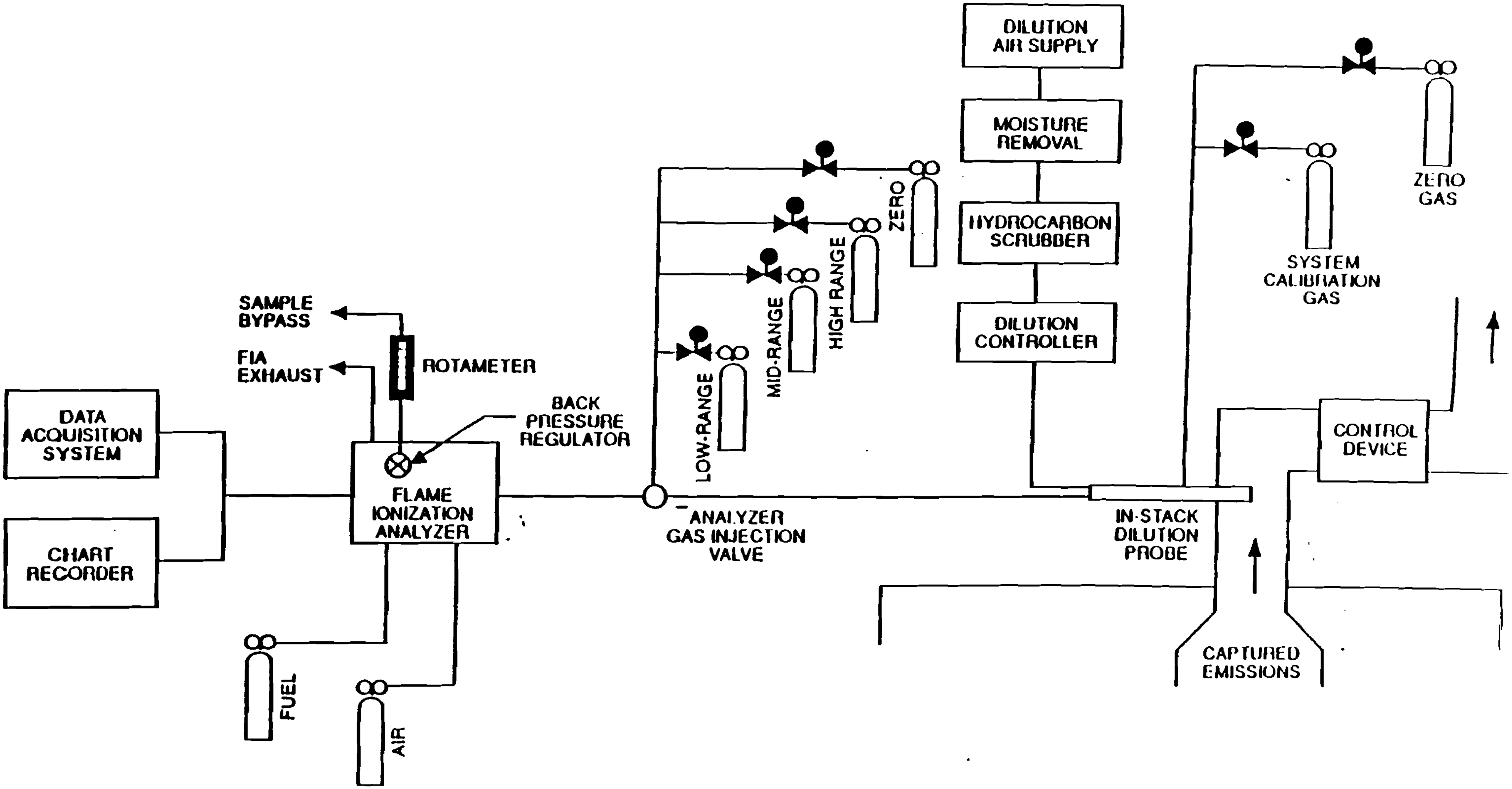


Figure 1. Captured emissions measurement system.

Other methods, such as quick-connect lines, to route calibration gases to the outlet of the sample probe are acceptable.

2.1.3 **Sample Line.** Stainless steel or Teflon tubing to transport the sample gas to the analyzer. The sample line must be heated to prevent condensation.

2.1.4 **Sample Pump.** A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The components of the pump that contact the gas stream shall be constructed of stainless steel or Teflon. The sample pump must be heated to prevent condensation.

2.1.5 **Sample Flow Rate Control.** A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent. The flow control valve and rotameter must be heated to prevent condensation. A control valve may also be located on the sample pump bypass loop to assist in controlling the sample pressure and flow rate.

2.1.6 **Sample Gas Manifold.** Capable of diverting a portion of the sample gas stream to the flame ionization analyzer (FIA), and the remainder to the bypass discharge vent. The manifold components shall be constructed of stainless steel or Teflon. If captured or fugitive emissions are to be measured at multiple locations, the measurement system shall be designed to use separate sampling probes, lines, and pumps for each measurement location and a common sample gas manifold and FIA. The sample gas manifold and connecting lines to the FIA must be heated to prevent condensation.

2.1.7 **Organic Concentration Analyzer.** An FIA with a span value of 1.5 times the expected concentration as propane; however, other span values may be used if it can be demonstrated that they would provide more accurate measurements. The system shall be capable of meeting or exceeding the following specifications:

2.1.7.1 **Zero Drift.** Less than ±3.0 percent of the span value.

2.1.7.2 **Calibration Drift.** Less than ±3.0 percent of the span value.

2.1.7.3 **Calibration Error.** Less than ±5.0 percent of the calibration gas value.

2.1.7.4 **Response Time.** Less than 30 seconds.

2.1.8 **Integrator/Data Acquisition System.** An analog or digital device or computerized data acquisition system used to integrate the FIA response or compute the average response and record measurement data. The minimum data sampling frequency for computing average or integrated values is one measurement value every 5 seconds. The device shall be capable of recording average values at least once per minute.

2.1.9 **Calibration and Other Gases.** Gases used for calibration, fuel, and combustion air (if required) are contained in compressed gas cylinders. All calibration gases shall be traceable to NIST standards and shall be certified by the manufacturer to ±1 percent of the tag value. Additionally, the manufacturer of the cylinder should provide a recommended shelf-life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available, alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval.

2.1.9.1 Fuel. A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

2.1.9.2 **Carrier Gas and Dilution Air Supply.** High purity air with less than 1 ppm of organic material (as propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

2.1.9.3 **FIA Linearity Calibration Gases.** Low-, mid-, and high-range gas mixture standards with nominal propane concentrations of 20-30, 45-55, and 70-80 percent of the span value in air, respectively. Other calibration values and other span values may be used if it can be shown that more accurate measurements would be achieved.

2.1.9.4 **Dilution Check Gas.** Gas mixture standard containing propane in air, approximately half the span value after dilution.

2.1.10 **Particulate Filter.** An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter must be heated to prevent any condensation unless it can be demonstrated that no condensation occurs.

2.2 **Captured Emissions Volumetric Flow Rate.**

2.2.1 **Method 2 or 2A Apparatus.** For determining volumetric flow rate.

2.2.2 **Method 3 Apparatus and Reagents.** For determining molecular weight of the gas stream. An estimate of the molecular weight of the gas stream may be used if it can be justified.

2.2.3 **Method 4 Apparatus and Reagents.** For determining moisture content, if necessary.

3. DETERMINATION OF VOLUMETRIC FLOW RATE OF CAPTURED EMISSIONS

3.1 Locate all points where emissions are captured from the affected facility. Using Method 1, determine the sampling points. Be sure to check each site for cyclonic or swirling flow.

3.2 Measure the velocity at each sampling site at least once every hour during each sampling run using Method 2 or 2A.

4. DETERMINATION OF VOC CONTENT OF CAPTURED EMISSIONS

4.1 **Analysis Duration.** Measure the VOC responses at each captured emissions point during the entire test run or, if applicable, while the process is operating. If there are a multiple captured emissions locations, design a sampling system to allow a single FIA to be used to determine the VOC responses at all sampling locations.

4.2 Gas VOC Concentration.

4.2.1 Assemble the sample train as shown in Figure 1. Calibrate the FIA according to the procedure in Section 5.1.

4.2.2 Set the dilution ratio and determine the dilution factor according to the procedure in Section 5.3.

4.2.3 Conduct a system check according to the procedure in Section 5.4.

4.2.4 Install the sample probe so that the probe is centrally located in the stack, pipe, or duct, and is sealed tightly at the stack port connection.

4.2.5 Inject zero gas at the calibration valve assembly. Measure the system response time as the time required for the system to reach the effluent concentration after the calibration valve has been returned to the effluent sampling position.

4.2.6 Conduct a system check before and a system drift check after each sampling run according to the procedures in Sections 5.2 and 5.4. If the drift check following a run indicates unacceptable performance, the run is not valid. The tester may elect to perform system drift checks during the run not to exceed one drift check per hour.

4.2.7 Verify that the sample lines, filter, and pump temperatures are 120 ± 5°C.

4.2.8 Begin sampling at the start of the test period and continue to sample during the entire run. Record the starting and ending times and any required process information as appropriate. If multiple captured emission locations are sampled using a single FIA, sample at each location for the same amount of time (e.g., 2 minutes) and continue to switch from one location to another for the entire test run. Be sure that total sampling time at each location is the same at the end of the test run. Collect at least 4 separate measurements from each sample point during each hour of testing. Disregard the measurements at each sampling location until two times the response time of the measurement system has elapsed. Continue sampling for at least 1 minute and record the concentration measurements.

4.3 Background Concentration.

4.3.1 Locate all NDO's of the TTE. A sampling point shall be centrally located outside of the TTE at 4 equivalent diameters from each NDO, if possible. If there are more than 6 NDO's, choose 6 sampling points evenly spaced among the NDO's.

4.3.2 Assemble the sample train as shown in Figure 2. Calibrate the FIA and conduct a system check according to the procedures in Sections 5.1 and 5.4.

4.3.3 Position the probe at the sampling location.

4.3.4 Determine the response time, conduct the system check and sample according to the procedures described in Sections 4.2.4 to 4.2.8.

4.4 **Alternative Procedure.** The direct interface sampling and analysis procedure described in Section 7.2 of Method 18 may be used to determine the gas VOC concentration. The system must be designed to collect and analyze at least one sample every 10 minutes.

5. CALIBRATION AND QUALITY ASSURANCE

5.1 **FIA Calibration and Linearity Check.** Make necessary adjustments to the air and fuel supplies for the FIA and ignite the burner. Allow the FIA to warm up for the period recommended by the manufacturer. Inject a calibration gas into the measurement system after the dilution system and adjust the back-pressure regulator to the value required to achieve the flow rates specified by the manufacturer. Inject the zero- and the high-range calibration gases and adjust the analyzer calibration to provide the proper responses. Inject the low- and mid-range gases and record the responses of the measurement system. The calibration and linearity of the system are acceptable if the responses for all four gases are within 5 percent of the respective gas values. If the performance of the system is not acceptable, repair or adjust the system and repeat the linearity check. Conduct a calibration and linearity check after assembling the analysis system and after a major change is made to the system.

5.2 **Systems Drift Checks.** Select the calibration gas that most closely approximates the concentration of the diluted captured emissions for conducting the drift checks. Introduce the zero and calibration gas at the calibration valve assembly and verify that the appropriate gas flow rate and pressure are present at the FIA. Record the measurement system responses to the zero and calibration gases. The performance of the system is acceptable if the difference between the drift check measurement and the value obtained in Section 5.1 is less than 3 percent of the span value. Conduct the system drift check at the end of each run.

5.3 **Determination of Dilution Factor.** Inject the dilution check gas into the measurement system before the dilution system and record the response. Calculate the dilution factor using Equation 3.

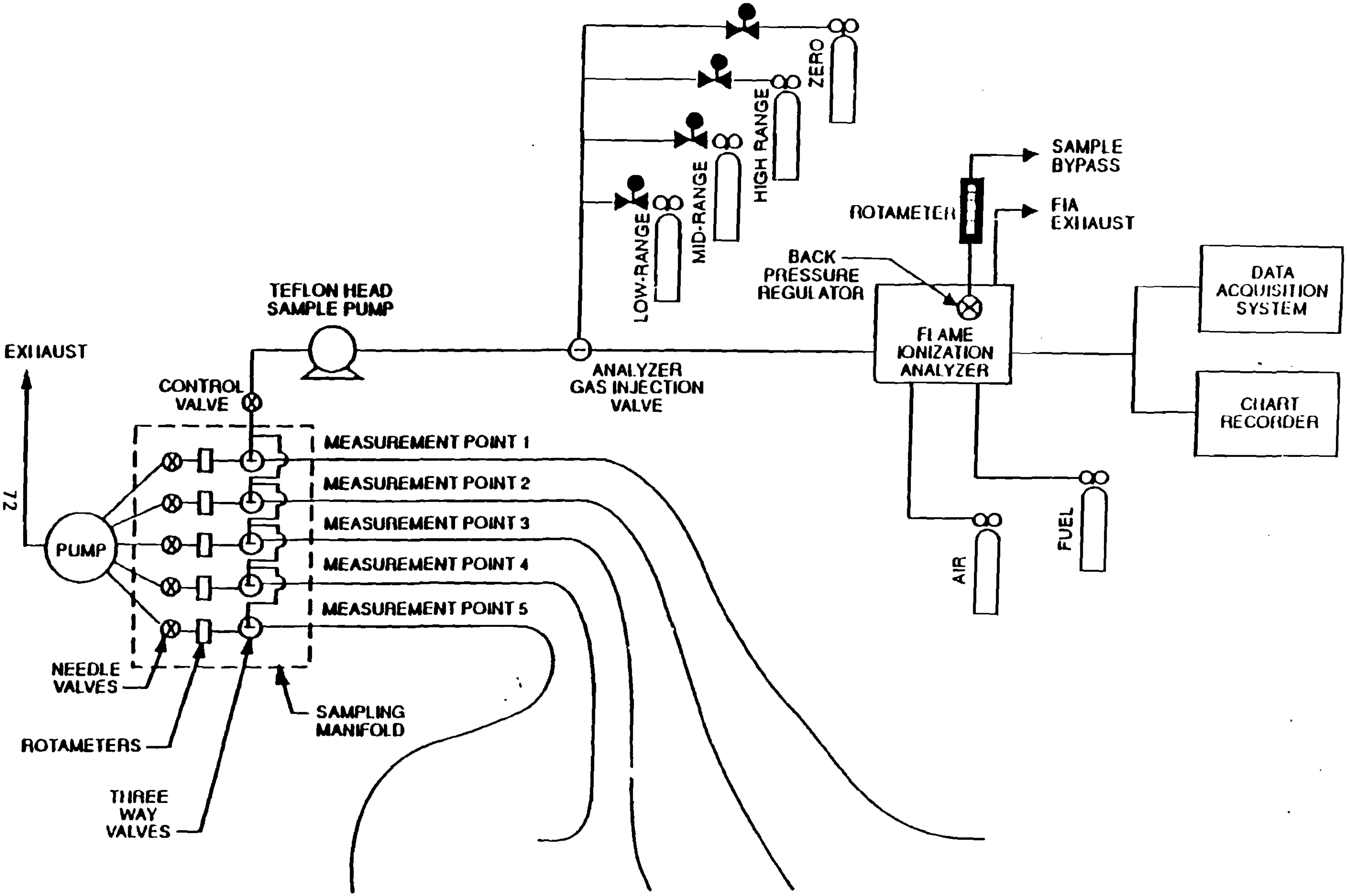


Figure 2. Backgrou[illegible]easurement system

5.4 System Check. Inject the high range calibration gas at the inlet to the sampling probe while the dilution air is turned off. Record the response. The performance of the system is acceptable if the measurement system response is within 5 percent of the value obtained in Section 5.1 for the high range calibration gas. Conduct a system check before and after each test run.

5.5 Analysis Audit. Immediately before each test analyze an audit cylinder as described in Section 5.2. The analysis audit must agree with the audit cylinder concentration within 10 percent.

6. NOMENCLATURE

A_i = area of NDO i, ft^2.

A_N = total area of all NDO's in the enclosure, ft^2.

C_A = actual concentration of the dilution check gas, ppm propane.

C_{Bi} = corrected average VOC concentration of background emissions at point i, ppm propane.

C_B = average background concentration, ppm propane.

C_{DH} = average measured concentration for the drift check calibration gas, ppm propane.

C_{D0} = average system drift check concentration for zero concentration gas, ppm propane.

C_H = actual concentration of the drift check calibration gas, ppm propane.

C_i = uncorrected average background VOC concentration measured at point i, ppm propane.

C_j = uncorrected average VOC concentration measured at point j, ppm propane.

C_M = measured concentration of the dilution check gas, ppm propane.

DF = dilution factor.

G = total VOC content of captured emissions, kg.

K_1 = 1.830×10^{-6} kg/(m^3-ppm).

n = number of measurement points.

Q_{Gj} = average effluent volumetric flow rate corrected to standard conditions at captured emissions point j, m^3/min.

θ_C = total duration of capture efficiency sampling run, min.

7. CALCULATIONS

7.1 Total VOC Captured Emissions.

$$G = \sum_{j=1}^{n} C_{Gj}\, Q_{Gj}\, \theta_C\, K_1 \qquad \text{Eq. 1}$$

7.2 VOC Concentration of the Captured Emissions at Point j.

$$C_{Gj} = DF\ (C_j - C_{DO})\ \frac{C_H}{C_{OH} - C_{DO}} \qquad \text{Eq. 2}$$

7.3 Dilution Factor.

$$DF = \frac{C_A}{C_M} \qquad \text{Eq. 3}$$

7.4 Background VOC Concentration at Point i.

$$C_{Bi} = (C_i - C_{DO})\ \frac{C_H}{C_{OH} - C_{DO}} \qquad \text{Eq. 4}$$

7.5 Average Background Concentration.

$$C_B = \frac{\sum_{i=1}^{n} C_{Bi}\, A_i}{n\, A_N} \qquad \text{Eq. 5}$$

NOTE: If the concentration at each point is within 20 percent of the average concentration of all points, the terms "A_i" and "A_N" may be deleted from Equation 4.

METHOD 1004
DETERMINATION OF VISIBLE EMISSIONS FROM STEEL MAKING OPERATIONS

The following test methods within Method 1004 are used to determine compliance with visible emission requirements at specified iron and steel installations. The applicable visible emission standards for which the test methods are used to determine compliance are found in the steel making regulation COMAR 26.11.10 Control of Iron and Steel Production Installations.

The procedures used to conduct visual observations of stack emissions is the EPA Method 9 which has been adopted by reference and is listed in the Appendix of this document. Method 9 is used when no other visible emission observation test method is specified. Under normal conditions, Method 9 is used to determine visible emissions from confined emission sources. The Department has established alternative visible emission standards for unconfined or fugitive emission sources for which the observation procedures under Method 9 cannot appropriately be used. Examples of these unconfined sources are leak conditions when emissions are discharged from several locations simultaneously, or building emissions. The emission requirements are expressed as allowable visible emissions per unit of time.

For each existing method or methods to be developed in the future for unconfined source emissions, the visible emission observation method always relates to a specific standard established in COMAR 26.11.10.

METHOD 1004F
DETERMINATION OF THE OPACITY OF VISIBLE FUGITIVE EMISSIONS FROM THE 'G', 'H', 'J', & 'K' BLAST FURNACE CASTHOUSES

1. Principle and Applicability.

1.1 Principle. The opacity of fugitive emissions from the "G', "H", "J", & "K" Blast Furnace Casthouse Buildings is determined visually by a qualified observer. The observer shall be qualified in accordance with the requirements of Method 9.

1.2 Applicability. This method is only applicable for the determination of the opacity of visible fugitive emissions from the "G", "H", "J" & "K" Blast Furnace Casthouse Buildings located at the Bethlehem Steel Corporation plant at Sparrows Point, Maryland.

2. Procedures. The qualified observer shall use the following procedures for visually determining the opacity of fugitive emissions from the "G", "H", "J", & "K" Blast Furnace Casthouse Buildings:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the fugitive emissions such that his line of vision is, as much as possible, approximately perpendicular to the long axis of the "G", "H", "J", & "K" Blast Furnace Casthouses. The observer's position shall be such that the sun is oriented in the 140° sector to his back.

2.2 Field Records. The observer shall record all of the data required by form AQ-19, Report of Observation of Visible Emissions. Weather conditions (wind direction, wind speed, ambient temperature, sky conditions), observer location, and sun location shall be recorded at the start of the observation. Any variations during the observation shall be noted in remarks. The observer shall indicate on the form the time duration of any furnace taps during the observation, if known.

2.3 Observations. Only fugitive emissions from the "G", "H", "J", & "K" Blast Furnace Casthouses are to be observed. Opacities shall be observed using any contrasting background, including the furnace structure and any appurtenances, and at the point of the visible fugitive emissions. Opacity observations shall not be made at a point where there is interference from visible emissions originating from other sources.

2.4 Recording Observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on the observational record sheet. A minimum of 240 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period. When two persons are used for observation, one person shall be located in the "G", "H", "J", & "K" Blast Furnace Casthouse area to monitor the furnace tapping operations. The time and duration of all furnace taps shall be transmitted to the outside observer is via walkie-talkie or recorded for later data reduction by using synchronized watches.

2.5 Data Reduction. The opacity of fugitive emissions from the "G", "H", "J", & "K" Blast Furnace Casthouse Building shall be determined as an average of 240 consecutive observations recorded at 15-second intervals.

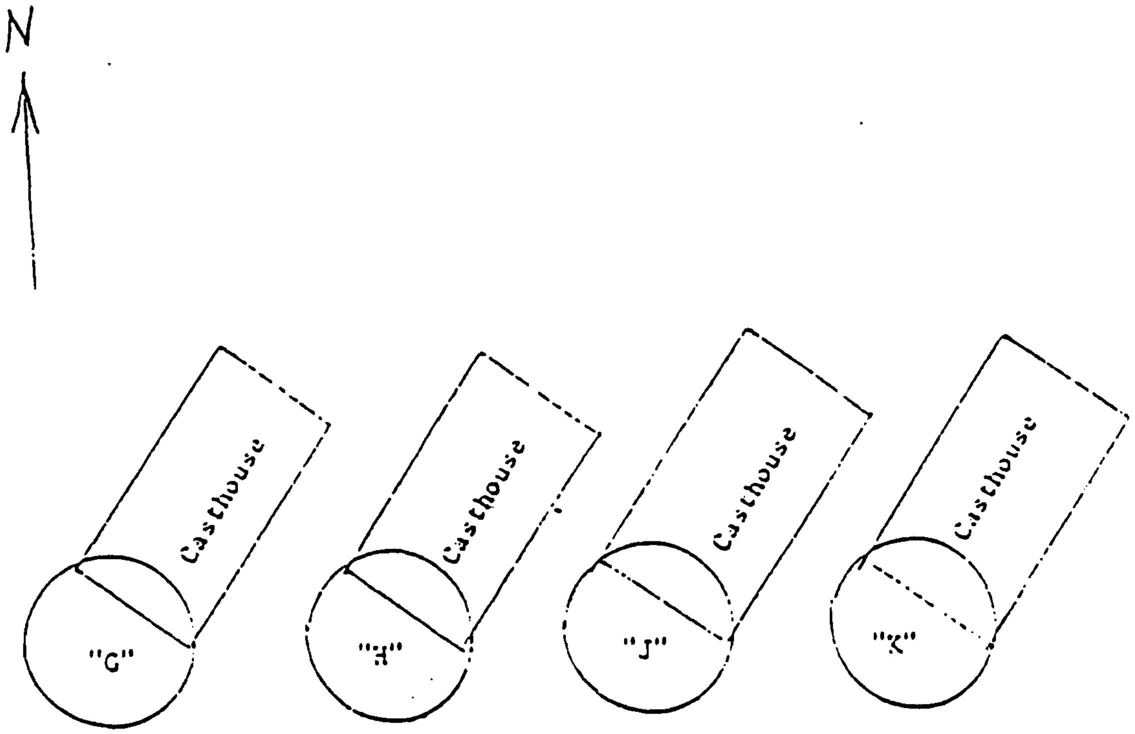


Figure 1: "G", "H", "J", & "K" Blast Furnace Casthouses Directional Orientation

METHOD 1004G
DETERMINATION OF THE OPACITY OF VISIBLE FUGITIVE EMISSIONS FROM THE 'L' BLAST FURNACE CASTHOUSES

1. Principle and Applicability.

1.1 Principle. The opacity of fugitive emissions from the "L" Blast Furnace Casthouse Buildings is determined visually by a qualified observer. The observer shall be qualified in accordance with the requirements of Method 9.

1.2 Applicability. This method is only applicable for the determination of the opacity of visible fugitive emissions from the "L" Blast Furnace Casthouse Buildings located at the Bethlehem Steel Corporation plant at Sparrows Point, Maryland.

2. Procedures. The qualified observer shall use the following procedures for visually determining the opacity of fugitive emissions from the "L" Blast Furnace Casthouse Buildings:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the fugitive emissions such that his line of vision is, as much as possible, approximately perpendicular to the long axis of the "L" Blast Furnace Casthouses. Figure 1 shows the directional orientation and typical observer positions for the "L" Blast Furnace Casthouses. The observer's position shall be such that the sun is oriented in the 140° sector to his back.

2.2 Field Records. The observer shall record all of the data required by form AQ-19, Report of Observation of Visible Emissions, Weather conditions (wind direction, wind speed, ambient temperature, sky conditions), observer location, and sun location shall be recorded at the start of the observation. Any variations during the observation shall be noted in remarks. The observer shall indicate on the form the time duration of any furnace taps and tap hole mudding during the observation, if known.

2.3 Observations. Only fugitive emissions from the "L" Blast Furnace Casthouse Buildings are to be observed. Opacities shall be observed using any contrasting background, including the furnace structure and any appurtenances, and at the point of maximum opacity of the visible fugitive emissions. Opacity observations shall not be made at a point where there is interference from visible emissions originating from other sources.

2.4 Recording Observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on the observational record sheet. A minimum of 240 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period. When two persons are used for an observation, one person shall be located in the "L" Blast Furnace Casthouse area to monitor the furnace tapping and tap hole mudding operations. The time and duration of all furnace taps and tap hole mudding shall be transmitted to the outside observer via walkie-talkie or recorded for later data reduction by using synchronized watches.

2.5 Data Reduction. The opacity of fugitive emissions from the "L" Blast Furnace Casthouse Buildings shall be determined as an average of 24 consecutive observations recorded at 15-second intervals.

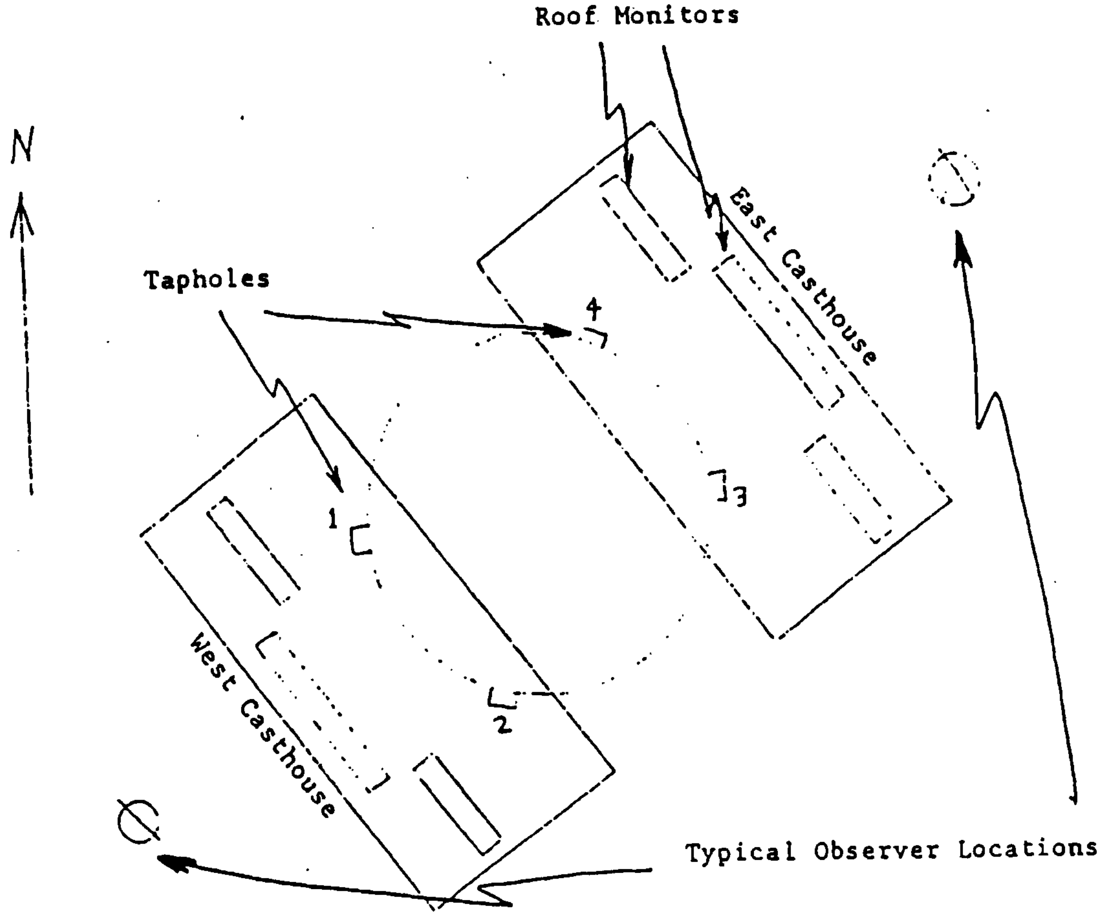


Figure 1: "L" Blast Furnace Casthouses
Directional Orientation and Typical Observer Locations

METHOD 1004H
DETERMINATION OF THE OPACITY OF VISIBLE FUGITIVE EMISSIONS FROM THE NO. 7 SINTER PLANT

1. Principle and Applicability.

1.1 Principle. The opacity of fugitive emissions from the No. 7 Sinter Plant Building is determined visually by a qualified observer. The observer shall be qualified in accordance with the requirements of Method 9.

1.2 Applicability. This method is only applicable for the determination of the opacity of visible fugitive emissions from the No. 7 Sinter Plant Building located at the Bethlehem Steel Corporation plant at Sparrows Point, Maryland.

2. Procedures. The qualified observer shall use the following procedures for visually determining the opacity of fugitive emissions from the No. 7 Sinter Plant Building:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the fugitive emissions such that his line of vision is approximately perpendicular to the long axis of the No. 7 Sinter Plant Building. The observer's position shall be such that the sun is oriented in the 140° sector to his back.

2.2 Field Records. The observer shall record all of the data required by form AQ-19, Report of Observation of Visible Emissions. Weather conditions (wind direction, wind speed, ambient temperature, sky conditions), observer location, and sun location shall be recorded at the start of the observation. Any variations during the observation shall be noted in remarks.

2.3 Observations. Only fugitive emissions from the No. 7 Sinter Plant Building are to be observed. Opacities shall be observed using only the sky as the contrasting background and at the point of maximum opacity of the visible fugitive emissions. Opacity observations shall not be made at a point where there is interference from visible emissions originating from other sources.

2.4 Recording Observations. Opacity observations shall be recorded at the nearest 5 percent at 15-second intervals on the observational record sheet. A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. The opacity of fugitive emissions from the No. 7 Sinter Plant Building shall be determined as an average of 24 consecutive observations recorded at 15-second intervals.

METHOD 1004I

DETERMINATION OF THE OPACITY OF VISIBLE FUGITIVE EMISSIONS FROM THE BASIC OXYGEN FURNACE SHOP ROOF MONITORS

1. Principle and Applicability

1.1 Principle. The opacity of visible emissions, other than water in an uncombined form, from the Basic Oxygen Furnace (BOF) Shop roof monitor is determined visually by a qualified observer. The observer shall be qualified in accordance with the requirements of EPA Reference Method 9.

1.2 Applicability. This method is only applicable for the determination of the opacity of visible emissions, other than water in an uncombined form, from the roof monitor of the BOF Shop Building located at the Bethlehem Steel Corporation plant at Sparrows Point, Maryland.

2. Procedures. The qualified observer shall use the following procedures for visually determining the opacity of visible emissions from the BOF Shop Building roof monitor:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the fugitive emissions such that his line of vision is approximately perpendicular to the long axis of the BOF Shop Building. The observer's position shall be such that the sun is oriented in the 140° sector to his back.

2.2 Field Records. The observer shall record all of the data required by Form AMA-19 (or equivalent form), Report of Observation of Visible Emissions. Weather conditions (wind direction, wind speed, ambient temperature, sky conditions), observer location, and sun location shall be recorded at the start of the observation. Any variations during the observation shall be noted in remarks.

2.3 Observations. Only visible emissions, other than water in an uncombined form, from the BOF Shop roof monitor are to be observed. Opacities shall be observed using only the sky as the contrasting background and at the point of maximum opacity of the visible emissions. Opacity observations shall not be made at a point where there is interference from visible emissions originating from other sources.

Each observation shall be performed for one hour utilizing EPA Reference Method 9 (Visible Determination of the Opacity of Emissions from Stationary Sources - 40 CFR Ch. 1, Part 60, App. A), except for the provision in Paragraph 2.4 of Method 9 setting the minimum number of 24 observations to be recorded and the data reduction provision in Paragraph 2.5 of Method 9. The "opacity record" of each observation is determined as the highest average of any 6 consecutive minutes of readings. Prior to making a "opacity record" calculation, the three highest minutes from the 60-minute observation may be removed in 1, 2, or 3-minute groups. After removing these minutes, the remaining minutes of readings are treated as if they were consecutive when calculating an "opacity record".

Observations may be performed at any time by any qualified EPA, MDE, or BSC observer (including qualified observers who are contractors for EPA, MDE, or BSC) whenever the conditions set forth in EPA Reference Method 9 are satisfied. In the event of any observations performed by EPA or MDE, EPA or MDE shall make the written records available to BSC for copying and BSC shall obtain copies of the written records of such observations to utilize such observations in its compliance and reporting obligations. For each calendar day on which more than one valid observation is performed, and therefore more than one "opacity record" is established, the highest "opacity record" of that calendar day shall be the only one utilized in a determination of compliance.

For each calendar day when a "opacity record" is established, compliance is determined by calculating the arithmetic average of the "opacity record" for that day and the two immediately preceding "opacity records".

2.4 Recording Observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on the observational record sheet. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period. When two persons are used for an observation, one person shall be located at the BOF Shop to monitor the Shop operation. The data collected shall include:

(1) Number of vessels in operation and each heat identification.

(2) Time of starting and completing each charge, tap and oxygen blow

(3) Number of scrubbers operating and operating parameters for each.

(4) Information on the operating procedures used by the shop workers during charging and tapping of each vessel.

Sampling Protocol for Fluoride Standards to Ensure Attainment of Ambient Air Quality Standards

Test Method 1005

MARYLAND DEPARTMENT OF THE ENVIRONMENT
AIR MANAGEMENT ADMINISTRATION
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SAMPLING PROTOCOL FOR FLUORIDE STANDARDS

In order to determine if the fluoride ambient air quality standards set forth in COMAR 26.11.04 are being met, the following minimum sampling and analysis protocol are hereby established.

Samples shall be collected from areas designed to circle the source of fluoride emissions as much as possible given the influence of topography and human activities and shall extend out radially from the source far enough so that the area of impact of the emissions is determined. The sampling network design must be approved by the Department.

Methods for measuring the fluoride content of any plant tissue shall be by suitable modification of the Willard and Winter method (See Willard, H.H. and Winter, O.B., "Volumetric Method for Determination of Fluoride", 5 Ind. Eng. Chem. Anal. Ed. 7-10, 1933) such as:

> Method 204 -- "Tentative Method of Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)", METHODS OF AIR SAMPLING AND ANALYSIS, Second Edition, pp. 403-417, by the Intersociety Committee, Published by American Public Health Association, 1015 Eighteenth Street, N.W., Washington, D.C., 1977, Library of Congress Catalog Number: 77-6826.

or other methods demonstrated to be equivalent and if approved by the Department.

Results are expressed on a dry weight basis in washed and unwashed samples as noted. Micrograms F per gram dry tissue means micrograms of fluorides as the ion, per gram of dry material.

Section I.(1)a. Field Crops.

> Field crops shall be considered to be those crops other than fruit crops referred to in Section I.(1)c. commercially grown and intended for human consumption. Representative samples of the material shall be collected at least once a month when the crop is growing.
>
> Samples shall consist of whole leaf portions taken from the areas considered to have the heaviest exposure to airborne fluoride. Samples shall not be collected from plants that are senescent. Exact guidelines for selection of these areas cannot be given due to the effects of meteorology, topography, etc., but will often tend to be the outermost rows of crops or crops grown on hills as compared to valleys, unshielded by trees and buildings.

Section I.(1)b. Forage

a. Forage.

Forage is defined as growing grasses, green chop and other growing vegetation that is consumed in the fresh state by livestock. Growing forage samples shall be secured from an area currently being grazed and cut two inches above ground. Forage samples shall not be collected in areas where it is impractical to obtain a valid sample and the livestock do not obtain a significant portion of their daily ration (such as loafing areas). Eight different locations in the prescribed area shall be composited and placed in a Kraft paper bag to make up the sample (at least 50 grams). If samples are not refrigerated or placed in a drying oven within 48 hrs. after collection, the grass sample bags are to be left open permitting the free flow of air to prevent spoilage. All locations selected shall be at least thirty (30) feet from the nearest stand of trees or other objects which might cause shielding or air disturbance from the source of fluoride emission. If green chop is being fed, instead of growing grasses or other growing vegetation, a representative sample (at least 50 grams) should be procured from the service wagon or the ground area where the green shop has been distributed using care to prevent foreign material from being entrained in the sample. If green chop is being fed, in addition to growing grasses or other growing vegetation, a representative sample (at least 50 grams) composed of both the green chop and growing grasses or other growing vegetation should be procured.

b. Hay and Silage.

When hay and/or silage is being fed, samples of these materials shall be taken for fluoride determination. Specimens collected from bale, pile, silo, or other locations will consist of random portions collected from a recently exposed location only and composited and analyzed by the referenced method. An auger shall be used to collect samples from bales. At least one core from at least ten bales from a recently exposed section of storage area is to be used for composite analysis. Any silage material which is obtained and analyzed for comparison to the standards must be free of such naturally occurring foreign material as dirt or mud, or added fluoride containing mineral supplements which would add relatively insoluble forms of fluoride to the sample. If it is not possible to determine if foreign material is entrained in the sample, this should be noted along with the sample.

c. Averages.

A minimum of three composite samples of forage and/or hay and/or silage collected at least six days apart during the month are required for a valid monthly average. During the growing season, the average of the growing forage and/or green chop (if being fed) shall be used as the monthly average, in the two-month average and in the running twelve-month average. Hay and/or silage shall be collected during this period for information on the animals total intake. If forage and/or green chop is not being fed, the average of the hay and/or silage shall be used during the growing season for all three averaging times. During the period of the year when growing forage or green chop is not available and cattle are on hay and/or silage, samples of these materials shall be taken for fluoride determination. The average of the hay and silage shall be used during this period to obtain the appropriate one-, two- and twelve-month average. The number and types of samples should approximately represent the animal diet at the farm location sampled.

Section I.(1)c. Fruit Trees, Berries, and Other Commercial Crops, and
Section I.(1)d. Deciduous Trees and Shrubs.

Samples of whole leaves shall be taken at least twice a year, early and late in the growing season. Senescent leaves shall not be sampled. Leaves shall be collected from the portion of the trees suspected to have the heaviest fluoride exposure. Exact guidelines for this selection cannot be made, but leaves should be collected from the upper portions of the trees (facing the source of fluoride emissions), using along pruner or other appropriate means. Samples are to be analyzed by the referenced procedures.

Section I.(1)e. Conifers and Evergreen Trees and Shrubs.

Whole leaves or needles of the current year and the preceding years shall be collected at least twice a year. Care should be taken to keep the needles representing the years separate. Leaves or needles shall be collected from the portion of the trees suspected of having the heaviest fluoride exposure and should include several samples of leaves or needles exhibiting possible leaf tip damage.

Section I.(1)f. Grasses and Herbs.

When requested by the Department, grasses not subject to browsing shall be collected once per year for analysis. The procedure outlined in Section I.(1)b. shall be followed for sampling and analysis of these grasses and herbs.

Section I(1)g. Ornamental Plantings.

When requested by the Department, whole leaves of ornamental plants shall be collected once per year for fluoride analysis. Leaves shall be taken from the portion of the plant suspected of having the heaviest fluoride exposure and any leaves exhibiting visible evidence of possible fluoride damage. Because a suitable sample may be relatively large when compared to an individual plant, sampling or ornamental plants shall only be done with the permission of the owner.

Section I.(1)h. Other Values.

Monthly static limed filter stations should be located at least five (5), but not more than twenty (20), feet above the ground facing in the direction of the nearest fluoride emission source unshielded by buildings, trees, or other obstructions. The method of making and reporting static limed filter paper measurements shall be in accordance with Method III--The Determination of Monthly Fluoride Measurement--as specified in the "Standard Methods for the Determination of Air Pollution Measurements Made at Ambient Air Sampling Stations in the State of Maryland", December, 1972. Other methods (approved by the Department) may be used if they have been demonstrated to be equally or more specific, accurate, sensitive, and reproducible.

Gaseous air samples shall be measured by the "Carbonate Tube" method or the "Dual Tape" method by Weinstein et. a., or by equivalent methods if approved by the Department. (See Weinstein, L.H. and Mandl, R.H., "The Separation and Collection of Gaseous and Particulate Fluoride", VDI Berichte,164, 1970.) The carbonate tube method shall be modified to require the simultaneous collection of two tubes, one of which has the particulate filter after the carbonate tube and one of which has the filter before the tube. For comparison to this secondary standard, the arithmetic means of the two carbonate tubes shall be computed and used.

EQUIPMENT SPECIFICATIONS FOR
SOLVENT METAL CLEANING
TEST METHOD 1006

MARYLAND DEPARTMENT OF THE ENVIRONMENT
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TM 91-01

A. <u>General</u>

This technical memorandum describes the types of equipment and the operating procedures that owners or operators of solvent metal cleaning equipment shall follow in the State of Maryland. Three types of cleaners are addressed: cold cleaners, conveyorized degreasers and open top vapor degreasers.

B. <u>Definitions</u>

(1) "Air to vapor interface area" means the total area of the solvent surface that is directly exposed to the atmosphere.

(2) "Cold cleaning" means the batch process of cleaning or removing foreign matter from metal surfaces by spraying, brushing,flushing or immersing articles in a solvent that is at a temperature lower than its boiling point.

(3) "Conveyorized degreasing" means the process of cleaning or removing foreign matter from metal surfaces by conveying articles into contact with the solvent on a continuous carrier.

(4) "Freeboard height" means the distance from the solvent surface or top of the vapor zone to the top edge of the solvent tank.

(5) "Freeboard ratio" means the freeboard height divided by the width of the degreaser.

(6) "Open top vapor degreasing" means the batch process of cleaning or removing foreign matter from a metal surface by condensing hot solvent vapor on the article to be cleaned.

(7) "Solvent" means any volatile organic compound (VOC) that is used to clean metal surfaces in cold cleaners, conveyorized degreasers or open top vapor degreasers.

C. Control Requirements for all Solvent Metal Cleaners

(1) A person who owns a solvent metal cleaner or degreaser shall limit exhaust ventilation to 65 cfm per square foot of cleaner air to vapor interface and shall not use fans near the degreaser opening. Higher ventilation rates will be approved by the Department if they are required to meet occupational safety standards.

(2) A person who owns a solvent metal cleaner or degreaser shall post a permanent label on or close to the equipment that describes the proper operating procedures for the equipment and is easily visible to any person who operates the cleaner.

(3) A person who owns or operates a solvent metal cleaner may not store waste solvent in open containers or dispose of waste solvent such that more than 20 pounds (9.07 kilograms) per day are allowed to evaporate.

D. Control Requirements for Cold Cleaners

(1) A person who owns a cold cleaner shall equip the cleaner tank with a cover and drainage facilities.

(2) A person who owns a cold cleaner that contains a solvent with a vapor pressure of 0.3 psi at 100°F (15.5 mm Hg @ 38°C) or greater, or that agitates or heats the solvent, shall equip the cleaner tank with a cover that can be easily operated with one hand. In larger degreasers, this may require mechanical assistance such as spring loads, counterweights or powered systems. The owner shall also equip the tank with internal drainage facilities such that the cleaned parts are enclosed under the cover while draining.

(3) A person who owns a cold cleaner that contains a solvent with a

vapor pressure of 0.6 psi at 100°F (31 mm Hg @ 38°C) or greater, or that heats the solvent to more than 120°F (49°C), shall equip the cleaner tank with one of the following control methods:

(a) A freeboard height that provides a freeboard ratio of at least 0.7;

(b) A water cover for solvents that are insoluble in and heavier than water;

(c) A refrigerated freeboard device;

(d) A carbon adsorption system with a ventilation rate greater than 50 cfm per square foot of air to vapor interface area (15.2 m^3/min. per m^2) when the cover is open, and exhausting less than 25 ppmv of solvent averaged over a complete adsorption cycle; or

(e) Any other control method approved by the Department.

E. Control Requirements for Conveyorized Degreasers

(1) A person who owns a conveyorized degreaser with an air to vapor interface area greater than 20 square feet (1.8m^2) shall equip the degreaser with one of the following vapor control methods:

(a) A refrigerated freeboard device;

(b) A carbon adsorption system with a ventilation rate greater than 50 cfm per square foot of air to vapor interface area (15.2 m^3/min per m^2) when the cover is open, and exhausting less than 25 ppmv of solvent averaged over a complete adsorption cycle; or

(c) Any other control method approved by the Department.

(2) The owner of a degreaser subject to §E(1) above shall also equip the degreaser with the following features:

(a) A drying tunnel, tumbling baskets or other method approved by the Department to remove excess solvent from cleaned parts:

(b) Degreaser entrance and exit openings that silhouette work loads such that the average clearance between parts and the edge of the opening is either no more than 4 inches (10 cm) or 10 percent of the width of the opening; and

(c) Covers that close the degreaser during periods when it is not in use.

(3) The owner of a degreaser subject to §E(1) above shall also equip the degreaser with the following safety switches:

(a) A condenser flow switch and thermostat to shut off the pump heater if the condenser coolant is either not circulating or too hot;

(b) A spray safety switch that shuts off the spray pump and the conveyor if the vapor level drops more than 4 inches (10 cm); and

(c) A vapor level control thermostat that shuts off the pump heater when the vapor level rises significantly above design limits.

F. <u>Control Requirements for Open Top Vapor Degreasers</u>

(1) A person who owns an open top vapor degreaser shall equip the cleaner tank with a cover that can be opened and closed without disturbing the vapor zone.

(2) The owner of a degreaser subject to §F(1) above shall equip the degreaser with the following safety switches:

(a) A condenser flow switch and thermostat to shut off the pump heater if the condenser coolant is either not circulating or too hot; and

(b) A spray safety switch that shuts off the spray pump if the vapor level drops more than 4 inches (10 cm).

(3) A person who owns an open top vapor degreaser with an open area larger than 10 square feet (1 m^2) shall equip the degreaser with one of the following vapor control methods:

(a) A refrigerated freeboard device

(b) A freeboard height that provides a freeboard ratio of at least 0.75 and a self-powered cover;

(c) A cover that opens only when dry parts are entering or leaving the degreaser;

(d) A carbon adsorption system with a ventilation rate greater than 50 cfm per square foot of air to vapor interface area (15.2 m^3/min. per m^2) when the cover is open, and exhausting less than 25 ppmv of solvent averaged over a complete adsorption cycle; or

(e) Any other control method approved by the Department.

G. Operating Procedures for Cold Cleaners

The operator of a cold cleaner may not operate the unit except under the following circumstances:

(1) For degreasing operations that require the spraying of solvent, sprays shall be a solid stream of fluid rather than an atomized or droplet stream, and at a pressure that will not cause excessive splashing;

(2) Covers shall be closed when parts are not being handled in the cleaner; and

(3) Solvent carryout shall be minimized by tipping of parts to drain solvent pools and by keeping cleaned parts over the solvent until dripping ceases.

H. Operating Procedures for Conveyorized Degreasers

The operator of a conveyorized degreaser may not operate the unit except under the following circumstances:

(1) Covers shall be closed during all times that parts are not being processed through the unit;

(2) Parts shall be placed on racks within the unit to allow for complete drainage;

(3) Conveyor speeds shall be slower than 11 feet per minute (3.3 m/min.);

(4) Parts that are kept within the vapor zone for at least 30 seconds or until condensation ceases;

(5) Solvent carryout shall be minimized by tipping of parts to drain solvent pools and by keeping parts over the solvent for 15 seconds or until they are dry;

(6) Workloads shall not occupy more than half of the degreaser's open top area;

(7) Solvent shall not be sprayed above the vapor zone;

(8) Solvent leaks shall be repaired immediately or cause the unit to be shut down until they are repaired.

(9) Solvent exiting the water separator shall contain no visible water; and

(10) Loading that shall be limited in quantity such that the vapor level does not drop more than 4 inches (10 cm) when the workload is removed from the vapor zone.

METHOD 1007

PRESSURE-VACUUM TEST PROCEDURES FOR LEAK TIGHTNESS OF TRUCK TANKS

1. PRINCIPLE

Pressure and vacuum are applied to the compartments of gasoline truck tanks and the change in pressure/vacuum is recorded after a specified period of time.

2. APPLICABILITY

This method is applicable to determining the leak tightness of gasoline truck tanks in use and equipped with vapor collection equipment.

3. DEFINITIONS

3.1 Truck tank. Any container, including associated pipes and fittings, that is used for the transport of gasoline.

3.2 Compartment. A liquid-tight division of a truck tank.

3.3 Truck tank vapor collection equipment. Any piping, hoses, and devices on the truck tank used to collect and route the gasoline vapors in the tank to the bulk terminal, bulk plant, or service station vapor control system.

4. APPARATUS

4.1 Pressure source. Pump or compressed gas cylinder of air or inert gas sufficient to pressurize the truck tank to 6250 pascals (25 inches H_2O) above atmospheric pressure.

4.2 Regulator. Low pressure regulator for controlling pressurization of the truck tank.

4.3 Vacuum source. Vacuum pump capable of evacuating the truck tank to 2500 pascals (10 inches H_2O) below atmospheric pressure.

4.4 Manometer. Liquid manometer, or equivalent, capable of measuring up to 6250 pascals (25 inches H_2O) gauge pressure with ± 25 pascals (± 0.1 inch H_2O) precision.

4.5 Test cap for vapor recovery hose fittings. This cap should have a tap for manometer connection and a fitting with shut-off valve for connection to the pressure/vacuum supply hose.

4.6 Pressure/vacuum relief valves. The test apparatus shall be equipped with an in-line pressure/vacuum relief valve set to activate at 7000 pascals (28 inches H_2O) above atmospheric pressure or 3000 pascals (12 inches H_2O) below atmospheric pressure, with a capacity equal to the pressurizing or evacuating pumps.

4.7 Caps for liquid delivery line.

4.8 Pressure/vacuum supply hose.

5. PRETEST CONDITION

5.1 Purging of vapor. The truck tank shall be purged of gasoline vapors and tested empty. The tank may be purged by any safe method such as flushing with diesel fuel or heating fuel.

5.2 Location. The truck tank shall be tested where it will be protected from direct sunlight.

6. TEST PROCEDURE

6.1 The dome covers are to be opened and closed.

6.2 Connect static electrical ground connections to tank. Attach the delivery and vapor hoses, remove the delivery elbows, and plug the liquid delivery fittings.

6.3 Attach the test cap to the vapor recovery line of the truck tank.

6.4 Connect compartments of the tank internally to each other if possible. (If not possible, each compartment must be tested separately.)

6.5 Connect the pressure/vacuum supply hose and the pressure/vacuum relief valve to the shut-off valve. Attach the pressure source to the hose. Attach a manometer to the pressure tap.

6.6 Open the shut-off valve in the vapor recovery hose cap. Applying air pressure slowly, pressurize the tank, or alternatively the first compartment, to 4500 pascals (18 inches H_2O).

6.7 Close the shut-off valve and allow the pressure in the truck tank to stabilize, adjusting the pressure if necessary to maintain 4500 pascals (18 inches H_2O). When the pressure stabilizes, record the time and initial pressure.

6.8 At the end of 5 minutes, record the time and final pressure.

6.9 Disconnect the pressure source from the pressure/vacuum supply hose, and slowly open the shut-off valve to bring the tank to atmospheric pressure.

6.10 Connect the vacuum source to the pressure/vacuum supply hose.

6.11 Slowly evacuate the tank, or alternatively the first compartment, to 1500 pascals (6 inches H_2O).

6.12 Close the shut-off valve and allow the pressure in the truck tank to stabilize, adjusting the pressure if necessary to maintain 1500 pascals (6 inches H_2O) vacuum. When the pressure stabilizes, record the time and initial pressure.

6.13 At the end of 5 minutes, record the time and final pressure.

6.14 Repeat steps 6.5 through 6.13 for each compartment if they were not interconnected.

7. ALTERNATIVE TEST METHODS

Techniques, other than specified above, may be used for purging and pressurizing the truck tanks, if prior approval is obtained from the air pollution control officer. Such approval will be based upon demonstrated equivalency with the above method.

METHOD 1008

GASOLINE VAPOR LEAK DETECTION PROCEDURE BY COMBUSTIBLE GAS DETECTOR

1. PRINCIPLE

A combustible gas detector is used to indicate any incidence of leakage from gasoline truck tanks and vapor control systems. This qualitative monitoring procedure is an enforcement tool to confirm the continuing existence of leak-tight conditions.

2. APPLICABILITY

This method is applicable to determining the leak-tightness of gasoline truck tanks during loading without taking the truck tank out of service. The method is applicable only if the vapor control system does not create back-pressure in excess of the pressure limits of the truck tank compliance leak test. For vapor control systems, this method is applicable to determining leak-tightness at any time.

3. DEFINITIONS

3.1 Truck tank. Any container, including associated pipes and fittings, that is used for the transport of gasoline.

3.2 Truck tank vapor collection equipment. Any piping, hoses, and devices on the truck tank used to collect and route the gasoline vapors in the tank to the bulk terminal, bulk plant, or service station vapor control system.

3.3 Vapor control system. Any piping, hoses, equipment, and devices at the bulk terminal, bulk plant, or service station, which is used to collect, store, and/or process gasoline vapors.

4. APPARATUS AND SPECIFICATIONS

4.1 Manometer. Liquid manometer, or equivalent, capable of measuring up to 6250 pascals (25 inches H_2O) gauge pressure with ±25 pascals (0.1 inch H_2O) precision.

4.2 Combustible gas detector. A portable hydrocarbon gas analyzer with associated sampling line and probe.

4.2.1 Safety. Certified as safe for operation in explosive atmospheres.

4.2.2 Range. Minimum range of 0-100 percent of the lower explosive limit (LEL) as propane.

4.2.3 Probe diameter. Sampling probe internal diameter of 0.625 cm (1/4 inch).

4.2.4 Probe length. Probe sampling line of sufficient length for easy maneuverability during testing.

4.2.5 Response time. Response time for full-scale deflection of less than 8 seconds for detector with sampling line and probe attached.

5. TEST PROCEDURE

5.1 Pressure. Place a pressure tap in the terminal, plant, or service station vapor control system, as close as possible to the connection with the truck tank. Record the pressure periodically during testing.

5.2 Calibration. Calibrate the combustible gas detector with 2.2 percent propane by volume in air for 100 percent LEL response.

5.3 Monitoring procedure. During loading or unloading, check the periphery of all potential sources of leakage of the truck tank and of the terminal, plant, or service station vapor collection system with a combustible gas detector.

5.3.1 Probe distance. The probe inlet shall be 2.5 cm from the potential leak source.

5.3.2 Probe movement. Move the probe slowly (2.0 cm/second). If there is any meter deflection at a potential leak source, move the probe to locate the point of highest meter response.

5.3.3 Probe position. As much as possible, the probe inlet shall be positioned in the path of (parallel to) the vapor flow from a leak.

5.3.4 Wind. Attempt as much as possible to block the wind from the area being monitored.

5.4 Recording. Record the highest detector reading and location for each incidence of leakage.

Method 1009

APPENDIX A

A.1 EMISSION TEST PROCEDURE FOR TANK TRUCK GASOLINE LOADING TERMINALS

Hydrocarbon mass emissions are determined directly using flow meters and hydrocarbon analyzers. The volume of liquid gasoline dispensed is determined by calculation based on the metered quantity of gasoline at the loading rack. Test results are expressed in milligrams of hydrocarbons emitted per liter of gasoline transferred.

A.2 APPLICABILITY

This method is applicable to determining hydrocarbon emission rates at tank truck loading terminals employing vapor balance collection systems and either continuous or intermittent vapor processing devices. This method is applicable to motor tank truck and trailer loading only.

A.3 DEFINITIONS

3.1 Tank Truck Gasoline Terminal

A primary distribution point for delivering gasoline to bulk plants, service stations, and other distribution points, where the total gasoline throughput is greater than 76,000 liters/day.

3.2 Loading Rack

An aggregation or combination of gasoline loading equipment arranged so that all loading outlets in the combination can be connected to a tank truck or trailer parked in a specified loading space.

3.3 Vapor Balance Collection System

A vapor transport system which uses direct displacement by the liquid loaded to force vapors from the tank truck or trailer into the recovery system.

3.4 Continuous Vapor Processing Device

A hydrocarbon vapor control system that treats vapors from tank trucks or trailers on a demand basis without intermediate accumulation.

3.5 Intermittent Vapor Processing Device

A hydrocarbon vapor control system that employs an intermediate vapor holder to accumulate recovered vapors from tank trucks or trailers. The processing unit treats the accumulated vapors only during automatically controlled cycles.

A.4 SUMMARY OF THE METHOD

This method describes the test conditions and test procedures to be followed in determining the emissions from systems installed to control hydrocarbon vapors resulting from tank truck and trailer loading operations at bulk terminals. Under this procedure, direct measurements are made to calculate the hydrocarbon mass exhausted from the vapor processing equipment. All possible sources of leaks are qualitatively checked to insure that no unprocessed vapors are emitted to the atmosphere. The results are expressed in terms of mass hydrocarbons emitted per unit volume of gasoline transferred. Emissions are determined on a total hydrocarbon basis. If methane is present in the vapors returned from the tank trucks or trailers, provisions are included for conversion to a total non-methane hydrocarbon basis.

A.5 TEST SCOPE AND CONDITIONS APPLICABLE TO TEST

5.1 Test Period

The elapsed time during which the test is performed shall not be less than three 8-hour test repetitions.

5.2 Terminal Status During Test Period

The test procedure is designed to measure control system performance under conditions of normal operation. Normal operation will vary from terminal-to-terminal and from day-to-day. Therefore, no specific criteria can be set forth to define normal operation. The following guidelines are provided to assist in determining normal operation.

5.2.1 Closing of Loading Racks

During the test period, all loading racks shall be open for each product line which is controlled by the system under test. Simultaneous use of more than one loading rack shall occur to the extent that such use would normally occur.

5.2.2 Simultaneous use of more than one dispenser on each loading rack shall occur to the extent that such use would normally occur.

5.2.3 Dispensing rates shall be set at the maximum rate at which the equipment is designed to be operated. Automatic product dispensers are to be used according to normal operating practices.

5.3 Vapor Control System Status During Tests

Applicable operating parameters shall be monitored to demonstrate that the processing unit is operating at design levels. For intermittent vapor processing units employing a vapor holder, each test repetition shall include at least one fully automatic cycle of the vapor holder and processing device. Tank trucks shall be essentially leak free as determined by EPA Mobile Source Enforcement Division.

A.6 BASIC MEASUREMENTS AND EQUIPMENT REQUIRED

6.1 Basic measurements required for evaluation of emissions from gasoline bulk loading terminals are described below. The various sampling points are numbered in Figure 1.

Sample Point	Measurements Necessary
1. Gasoline dispensers	- Amount dispensed
2. Vapor Return Line	- Leak check all fittings
3. Processing unit exhaust	- Temperature of vapors exhausted
	- Pressure of vapors exhausted
	- Volume of vapors exhausted
	- HC concentration of vapors
	- Gas chromatograph analysis of HC*
	- Leak check all fittings and vents

6.2 The equipment required for the basic measurements are listed below:

Sample Point	Equipment and Specifications
2	1 portable combustible gas detector, (0-100% LEL).
3	1 flexible thermocouple with recorder
	1 gas volume meter, appropriately sized for exhaust flow rate and range
	1 total hydrocarbon analyzer with recorder; (FID or NDIR type, equipped to read out 0-10% by volume hydrocarbons as propane for vapor recovery processing device; or, 0-10,000 ppmv HC as propane for incineration processing devices)
Miscellaneous	1 barometer
	1 GC/FID w/column to separate C_1 - C_7 alkanes**

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*Required if methane is present in recovered vapors

**Required if methane is present in recovered vapors or if incineration is the vapor processing technique.

A.7 TEST PROCEDURES

7.1 Preparation for testing includes:

7.1.1 Install an appropriately sized gas meter on the exhaust vent of the vapor processing device. A gas volume meter can be used at the exhaust of most vapor recovery processing devices. For those where size restrictions preclude the use of a volume meter; or when incineration is used for vapor processing, a gas flow rate meter (orifice, pitot tube annubar, etc.) is necessary. At the meter inlet, install a thermocouple with recorder. Install a tap at the volume meter outlet. Attach a sample line for a total hydrocarbon analyzer (0-10% as propane) to this tap. If the meter pressure is different than barometric pressure, install a second tap at the meter outlet and attach an appropriate manometer for pressure measurement. If methane analysis is required, install a third tap for connection to a constant volume sample pump/evacuated bag assembly.*

7.1.2 Calibrate and span all instruments as outlined in Section 9.

7.2 Measurements and data required for evaluating the system emissions include:

7.2.1 At the beginning and end of each test repetition, record the volume readings on each product dispenser on each loading rack served by the system under test.

7.2.2 At the beginning of each test repetition and each two hours thereafter, record the ambient temperature and the barometric pressure.

7.2.3 For intermittent processing units employing a vapor holder, the unit shall be manually started and allowed to process vapors in the holder until the lower automatic cut-off is reached. This cycle should be performed immediately prior to the beginning of the test repetition before readings in 7.2.1 are taken. No loading shall be in progress during this manual cycle.

7.2.4 For each cycle of the processing unit during each test repetition, record the processor start and stop time, the initial and final gas meter

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*Described in Method 3, <u>Federal Register</u>, V36, n247, December 23, 1971.

readings, and the average vapor temperature, pressure and hydrocarbon concentration. If a flow rate meter is used, record flow meter readouts readings, and the average vapor temperature, pressure and hydrocarbon continuously during the cycle. If required, extract a sample continuously during each cycle for chromatographic analysis for specific hydrocarbons.

7.2.5 For each tank truck or trailer loading during the test period, check all fittings and seals on the tanker compartments with the combustible gas detector. Record the maximum combustible gas reading for any incidents of leakage of hydrocarbon vapors. Explore the entire periphery of the potential leak source with the sample hose inlet 1 cm away from the interface.

7.2.6 During each test period, monitor all possible sources of leaks in the vapor collection and processing system with the combustible gas indicator. Record the location and the combustible gas reading for any incidents of leakage.

7.2.7 For intermittent systems, the processing unit shall be manually started and allowed to process vapors in the holder until the lower automatic shut-off is reached at the end of each test repetition. Record the data in 7.2.4 for this manual cycle. No loading shall be in progress during this manual cycle.

A.8 CALCULATIONS

8.1 Terminology

T_a = Ambient temperature (°C)

P_b = Barometric pressure (mm Hg)

L_t = Total volume of liquid dispensed from all controlled racks during the test period (liters)

V_e = Volume of air-hydrocarbon mixture exhausted from the processing unit (M^3)

V_{es} = Normalized volume of air-hydrocarbon mixture exhausted, NM^3 @ 20°C, 760 mm Hg

C_e = Volume fraction of hydrocarbons in exhausted mixture (volume % as $C_3H_{10}/100$, corrected for methane content if required)

T_e = Temperature at processing unit exhaust (°C)

P_e = Pressure at processing unit exhaust (mm Hg abs)

$(M/L)_e$ = Mass of hydrocarbons exhausted from the processing unit per volume of liquid loaded, (mg/l)

8.2 Processing Unit Emissions

Calculate the following results for each period of processing unit operation:

8.2.1 Volume of air-hydrocarbon mixture exhausted from the processing unit:

$$V_e = V_{ef} - V_{ei}, \text{ or } \qquad (m^3)$$

$$V_e = \text{totalized volume from flow rate and time records.}$$

8.2.2 Normalized volume of exhausted mixture:

$$V_{es} = \frac{(0.3858\ °K/mm\ Hg)\ V_e P_e}{Tet\ 273.2} \qquad NM^3\ @\ 20°C,\ 760\ mm\ Hg$$

8.2.3 Mass of hydrocarbons exhausted from the processing unit:

$$M_e = \left(1.833 \times 10^6\ \frac{mg\ C_3H_8}{NM^3 C_3H_8}\right) \times V_{es} C_e \qquad (mg)$$

8.3 Average Processing Unit Emissions

8.3.1 Average mass of hydrocarbons emitted per volume of gasoline loaded:

$$(M/L)_e = \frac{\Sigma M_e}{L_t} \qquad (mg/liter)$$

A.9 CALIBRATIONS

9.1 Flow Meters

Use standard methods and equipment which have been approved by the Administrator to calibrate the gas meters.

9.2 Temperature Recording Instruments

Calibrate prior to the test period and following the test period using an ice bath (0°C) and a known reference temperature source of about 35°C. Daily during the test period, use an accurate reference to measure the ambient temperature and compare the ambient temperature reading of all other instruments to this value.

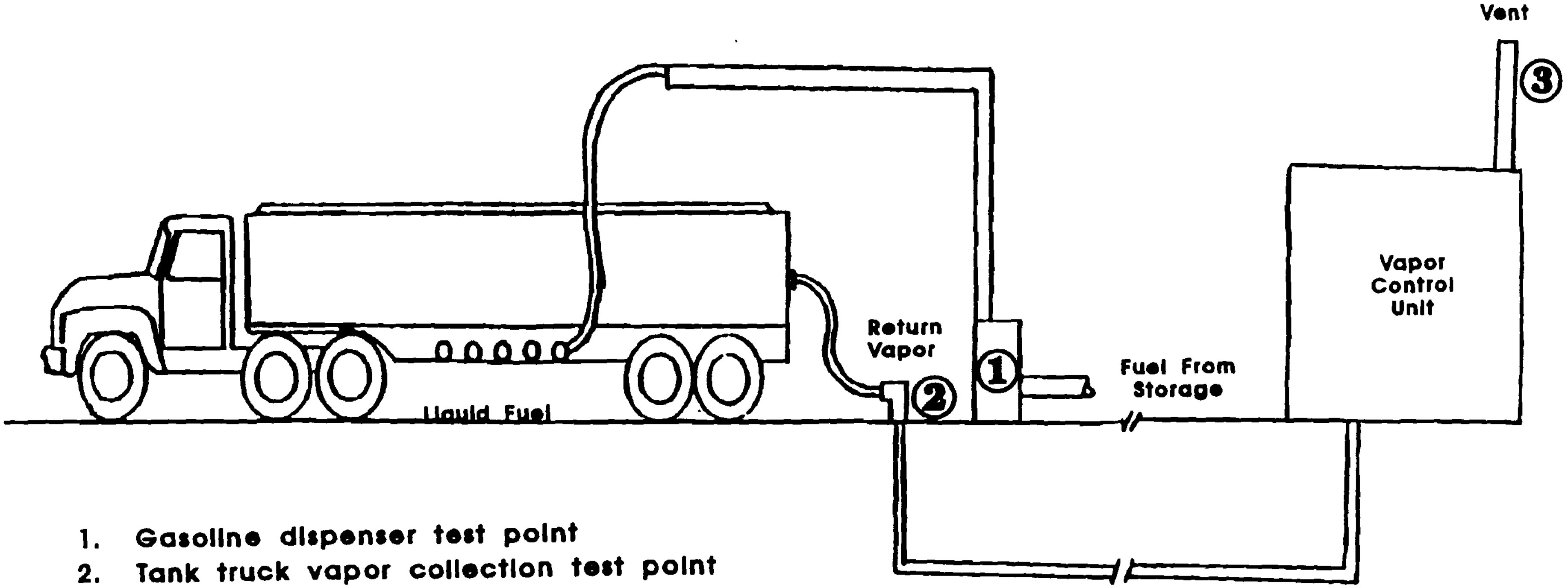
9.3 Total hydrocarbon analyzer

Follow the manufacturer's instructions concerning warm-up and adjustments. Prior to and immediately after the emission test, perform a comprehensive laboratory calibration on each analyzer used. Calibration gases should be propane in nitrogen prepared gravimetrically with mass quantities of approximately 100 percent propane. A calibration curve shall be provided using a minimum of five prepared standards in the range of concentrations expected during testing.

For each repetition, zero with zero gas (3 ppm C) and span with 70% propane for instruments used in the vapor return lines and with 10% propane for instruments used at the control device exhaust.

The zero and span procedure shall be performed at least once prior to the first test measurement, once during the middle of the run, and once following the final test measurement for each run.

Conditions in calibration gas cylinders must be kept such that condensation of propane does not occur. A safety factor of 2 for pressure and temperature is recommended.



1. Gasoline dispenser test point
2. Tank truck vapor collection test point
3. Vapor control unit test point

Figure A-1. TANK TRUCK GASOLINE LOADING VAPOR CONTROL SCHEMATIC

Table A-1 GASOLINE BULK TRANSFER TERMINAL DATA SHEET No. 1

Terminal Name: ______________________________ Date: ________________

Location: ____________________________________

Daily Ambient Data: (record every 2 hours)

Schematic Diagram of Rack Layout

	Time	T_a	P_b
Start:			
End:			

Dispenser Meter Readings

Time			Time		
Pump No.	Initial	Final	Pump No.	Initial	Final

GASOLINE BULK TRANSFER TERMINAL CONTROL SYSTEM DATA SHEET No. 2

Terminal Name: ______________________ Date: __________

Location: ______________________

Control Device Outlet

Gas meter readings Initial ________ Final ________

Time Test Start __________ Test End __________

Record the following for each processing unit operating cycle or emission period.

TIME		VOLUME READING		AVERAGE		
Start	Stop	Initial	Final	Temperature	Pressure	HC Concentration % as ____

Method 1010 - Determination of Total Fluoride Emissions from Primary Aluminum Production Plants

1. Principle and Applicability

1.1 Principle. Gaseous and particulate fluoride is withdrawn isokinetically from the gas stream and collected on a glass fiber filter and in the water of the impinger train and the fluoride content determined by a technicon auto analyzer.

1.2 Applicability. This method is applicable for the determination of gaseous and particulate fluoride from primary aluminum reduction plants, method 1001 is to be used to select the sampling location and method 1002 is to be used to determine gas velocity when testing the anode bake plant and the dry scrubber (section 4.B).

2. Air Velocity Measurement

2.1 The average gas velocity in the potroom roof monitor stack is determined by direct measurement with a vane anemometer (see 4.C.8).

2.2 Apparatus.

2.2.1 Vane anemometer, Taylor Model 3132.

2.2.2 Stopwatch.

2.3. Procedures.

2.3.1 .Position the anemometer in the air way so that it faces squarely into the airstream and the dial faces downstream. Support the anemometer in a manner such that the air flow through and around the instrument is as free and undisturbed as possible.

2.3.2 Simultaneously turn the engaging lever to the "on" position and start the stopwatch. NOTE: DO NOT turn the engaging lever to the "on" position unless the fan blades are in motion. DO NOT turn the fan blades with the fingers. This will alter the angular position of the blades and affect the accuracy of instrument. The instrument may be tested by blowing on the blades.

2.3.3 After a period of 1-3 minutes, simultaneously turn the engaging lever to the "off" postion and stop the stopwatch. Record the anemometer and stopwatch reading. The measurement period should be short enough so that the total dial registration does not exceed 10,000 feet.

2.3.4 DO NOT reset the anemometer to zero unless the engaging lever is in the "off" position.

2.3.5 Measure the stack area.

2.4 Calculations.

2.4.1 Air Velocity, ft/min.

$$V = \frac{F}{T} + C \qquad \text{eqn. 1}$$

Where:

V = air velocity, ft/min.
F = anemometer dial reading, ft.
T = measurement time, minutes
C = calibration curve correction factor

2.4.2 Volumetric flow, ft^3/min.

$$Q = VA \quad \text{eqn. 2}$$

Where:

Q = volumetric flow, ft^3/min.
V = air velocity, ft/min.
A = stack area, ft^2

2.5 Notes.

2.5.1 The Taylor anemometer is not guaranteed correct when used in temperatures over 300°F.

2.5.2 Do not use the anemometer in dusty airstreams or airstreams with entrained water droplets.

2.5.3 Meter should be calibrated regularly and must be calibrated if it does not zero properly or if it has been subject to rough handling. Calibration should be done in accordance with procedures outlined in the 16th edition "Industrial Ventilation", page 9-14, published by ACGIH or equivalent.

2.6. References.

2.6.1 Taylor Model 3132 Jeweled Anemometer Instruction Manual. Taylor Instrument Consumer Products Division, Arden, North Carolina 28704.

3. Determination of Particulate and Gaseous Fluoride Emissions - Separation and Collection Using an Impinged Air Sampling Train

3.1 Outline of Method. Impingement is a process for the collection of particulate matter in which a gas is directed forcibly against a surface which serves to retain particulate matter. The gas stream then passes through a liquid medium which selectively extracts the gaseous emissions; in this case fluoride. Subsequent semi-automated fluoride determinations are analyzed by the lab. Total particulate is gravimetrically determined prior to fluoride analysis. An alkaline fusion is required to put the particulate fluorides into solution whereas it is not required for gaseous fluoride is already soluble.

3.2 Apparatus.

3.2.1 Low fluoride muffle furnace.

3.2.2 Low fluoride charring oven.

3.3.3 75 ml inconel crucibles.

3.3.4 Assorted volumetric glassware.

3.3.5 Polypropylene graduated centrifuge tubes, 50 ml.

3.3.6 Filtering assembly.

3.3.7 Whatman #42 filter paper - 12.5 cm.

3.3.8 Analytical balance accurate to 0.1 mg.

3.3.9 Technicon AutoAnalyzer I.

3.4 Reagents.

3.4.1 Reagent grade acetone.

3.4.2 Sodium hydroxide pellets.

3.4.3 Saturated low fluoride calcium oxide suspension.

3.4.4 Stack sample dilution mixture, 60 g NaOH and 200 ml conc. $HClO_4$ per liter.

3.4.5 Phenolphthalein indicator.

3.5. Procedure.

3.5.1 Gravimetrically determine particulate on preweighed filter papers by removing from petri dishes and weighing to nearest 0.1 mg.

3.5.2 Preweigh (tare) Whatman #42 filter papers to be used for filtering acetone rinse samples.

3.5.3 Filter the acetone rinse samples carefully through the tared filters to avoid splashing. (A glass rod should be used.)

3.5.4 Rinse the polyethylene bottles with acetone making sure no particulate matter remains. Wash down the sides of the filter papers with acetone so the particulate matter will accumulate in the base of the fold. This prevents accidental loss during subsequent handling.

3.5.5 Air dry the filter papers to a constant weight and record to the nearest 0.1 mg.

3.5.6 Transfer all of the filter papers (filter samples and acetone rinse samples) to inconel crucibles and record data.

3.5.7 Saturate the papers by dropwise addition of a well-mixed, low fluoride calcium oxide suspension. Add a drop of phenolphthalein indicator making sure sample is basic until completely dry.

3.5.8 Prepare a blank by treating an unexposed filter paper in the same manner as the samples.

3.5.9 Evaporate to dryness and char in the brown Thelco oven (set at highest temperature setting) until partially ashed, or overnight.

3.5.10 Place samples (3 at a time) in the front of the low fluoride muffle furnace with the door open to permit further charring without flaming. When completely charred, place the samples in the rear of the furnace and ash for 1-2 hours at 600° C until the ash is white or gray indicating the removal of all organic matter. (This is essential since samples will be treated with perchloric acid.)

3.5.11 When samples are completely ashed, remove them from the furnace and add approximately 3 grams of sodium hydroxide pellets. Return the crucibles to the furnace for approximately five minutes, watching for "creep." (No more than 8 at a ime.)

3.5.12 When the sodium hydroxide has melted, remove the crucibles from the furnace, one at a time, and swirl to suspend the melt uniformly until it solidifies.

3.5.13 Allow the crucibles to cool until they do not "spatter" when distilled water is added. Dissolve the contents of the crucibles with successive rinsings of distilled water. Scrape the sides of the crucibles with a polyethylene policeman to help dissolve the sample. (If necessary, the water may be heated.) Rinse * into a 250 ml volumetric flask and dilute to the mark with distilled water. (100 ml volumetric flask for dry system tests.)

3.5.14 Impinger volumes should be measured prior to analysis. Pipet an aliquot (1-20 ml) of each sample (particulate and gaseous) and place in a 50 ml polypropylene centrifuge tubes. Dilute to 50 ml with stack sample dilution mixture and mix thoroughly by inversion. Analyze on the Technicon AutoAnalyzer I.

3.5.15 Prepare an impinger blank pipetting 20 ml deionized H_2O into a 50 ml centrifuge tube and dilute to 50 ml with stack sample dilution mixture.

3.5.16 All results are to be blank corrected.

3.6 Calculations.

$$\text{mg F-} = \frac{(\text{ppm from tech}) \times (A) \times (50 \text{ ml})}{(\text{aliquot vol}) \times (1000)}$$

A = 250 ml for filter and rinses but equals impinter volume for impingers. (100 ml for filters and rinses from dry system tests.)

3.7 Precision & Accuracy. The precision and accuracy of this method is limited by the precision and accuracy of the Technicon AutoAnalyzer procedure (10% error). Other precision and accuracy data on this particular method have not be compiled or are not available at the present time.

3.7 Notes.

3.7.1 Sample aliquots should be chosen so the Technicon results fall within the good operating range of the standard curve. If samples don't fall in this range, redilute them using another more appropriate aliquot size.

3.7.2 For further dilutions of the final samples a dilution mixture containing 30 g NaOH and 100 ml perchloric acid per 500 ml is to be used.

3.7.3 Crucibles are to be cleaned by soaking in 10% NaOH over night. Rince crucibles with tap water and deionized water. Immerse crucibles in 4N HCl for 1 hour. Wash with hot water, scour, and rinse thoroughly with distilled water. Dry crucibles in the charring oven.

3.7.4 Calcium oxide residues left in the glassware should be removed by rinsing with diluted hydrochloric acid.

3.7.5 Low fluoride calcium oxide may be prepared by hearing low fluoride calcium carbonate at 900°C for 2 hours. Test for CO_3 present when cool.

3.8 References.

3.8.1 ASTM Method D3267-78 & D3270-78, 1975 ASTM Annual Standards, Part 26, pp. 691-697, pp. 724-737.

3.8.2 Method of Air Sampling and Analysis, 2nd Edition, pp. 384-386, pp. 403-417.

4. Source Sampling for Fluoride Emissions

4.1 Outline of Method. Gaseous and particulate fluorides are withdrawn isokinetically from the source using a sampling train. The fluorides are collected in the impinger water and on the filter of the sampling train. The weight of total fluorides in the train is determined using the Technicon AutoAnalyzer (Analytical Method F-3). Fluorocarbons, such as Freon, are not quantitatively collected or measured by this method.

4.2 Apparatus.

4.2.1 Sampling train (figure 1).

4.2.2 Nozzle - Stainless steel (316) with sharp, tapered leading edge.

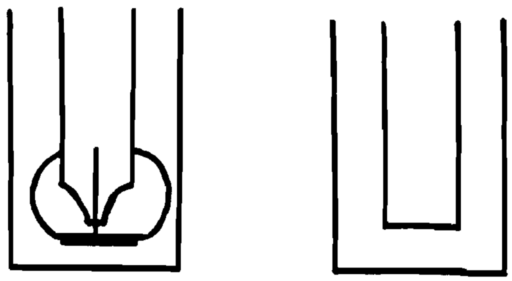
4.2.3 Probe - Stainless steel (316) with a heating system capable of maintaining a gas temperature of about 250°F at the probe when required to prevent condensation in the probe.

4.2.4 Pitote tube - Type S attached to probe

4.2.5 Filter holder - Borosilicate glass with a stainless steel 20 mesh screen filter support and a silicone rubber gasket.

4.2.6 Filter heating system - capable of maintaining the filter holder enclosure at about 250°F. The heating system is required only when the filter is located downstream of the impingers, or for sampling saturated gases when the filter is upstream of the impingers.

4.2.7 Impingers - Greenburg-Smith (G-S) (1) and modified Greenburg-Smith (3) impingers with ground glass, vacuum tight 28/12 or 28/15 ball-and-socket fittings.



Greenburg-Smith Modified Greenburg-Smith

tip should extend to within ½" from bottom of the flask.

4.2.8 U-adapters and modular elbow - 28/12 or 28/15 ball-and-socket joints.

4.2.9 Metering system - RAC Model 2343 Meter Control Console, Eastalco Samping Console.

4.2.10 Nomograph or Stak Samplr slide rule.

4.2.11 Sample storage bottles, 1- wide mouth polypropylene (p.p.) bottles. Polyethylene (p.e.) bottles are not acceptable.

4.2.12 Davis vane anemometer, 0-3000 fpm.

4.3 Reagents.

4.3.1 Filters - Whatman No. 1 to fit filter holder.

4.3.2 Silica gel - indicating type, 6-16 mesh, dried at 350°F for 2 hours. (Blue when dry - pinkish when wet.)

4.3.3 Water - distilled.

4.3.4 Acetone - ACS reagent grade.

4.3.5 Silicone stopcock grease.

4.4 Procedure.

Sampler Preparation.

4.4.1 Place a thin ring of silicone grease around the ground glass joints of all glassware used in the sample case. Do not use excessive amounts of grease. Use only enough to ensure a vacuum-tight seal around the joint.

4.4.2 Place a tared filter in the filter holder being certain that the filter is on the side of the holder that attaches to the sampling probe. The filter must have been in a desiccator at least 24 hours before use.

4.4.3 If the source to be tested has a high particulate loading (such as an inlet), attach a cyclone assembly to the filter assembly; otherwise attach a cyclone eliminator to the filter assembly.

4.4.4 Place the assembled filter holder in a plastic bag and seal it for transport to the field.

4.4.5 Attach a calibrated nozzle to the probe making sure that the nozzle will face directly into the airstream and when the probe is inserted into the stack. Place a plastic bag (Whirlpack) over each end of the probe for transport to the field.

4.4.5 Note: usually a nominal ¼" diameter nozzle for potline baghouse testing and a nominal ½" diameter nozzle for potline wet scrubber testing will be used.

4.4.6 Place 100 ml of distilled water in a modified G-S impinger and in a G-S impinger. Put the modified G-S impinger in the 1st position in the sampler the G-S impinger in the 2nd position.

4.4.7 Place an empty modified G-S impinger in the third position.

4.4.8 Weigh approximately 200-300 g of silica gel to the nearest gram and place it in a modified G-S impinger. Place the G-S impinger in the 4th position in the sample case.

4.4.9 Attach the modular elbow to the inlet (center tube) of the first impinger.

4.4.10 Connect a U-adapter from the outlet (side-arm) of the 1st impinger to the inlet of the second impinger. Connect the remaining impingers in similar fashion; outlet to inlet.

4.4.11 Place a piece of Parafilm over the inlet of the modular elbow and over the outlet of the 4th impinger to seal the system for transport to the field.

4.4.12 Attach the umbilical sample head hood-up to the sample box but do not connect it to the final impinger at this time.

4.4.13 Assemble the two halves of the sample case and insert the bolt and wing nuts in the interface flange.

4.4.14 Fill the impinger section of the sample case with crushed ice. Ice should be added during the test to maintain an impinger exit temperature of 70°F.

Train Operation - Dry Scrubbers

4.4.15 Determine the barometric pressure and record the value on the field data sheet.

4.4.16 Conduct a velocity traverse on the stack and calculate the average p value. Be sure the meter box is level and the manometer fluid levels are at zero. The nozzle should be plugged during the velocity traverse.

4.4.17 Measure the stack temperature.

4.4.18 Set the nomograph or Stak Samplr slide rule for isokinetic flow conditions following the instructions printed on the nomograph and slide rule.

4.4.19 Perform a leak check on the meter box. Insert a quick-disconnect plug in the umbilical cord connector. Turn on the vacuum pump and adjust the vacuum to 15" Hg using the coarse and fine vacuum control knobs. The dry gas meter should register less than 0.002 ft^3 over a 1 minute time period. If the leak rate is greater than 0.002 ft^3/min., the meter box should be repaired before use.

4.4.20 Field check the orifice meter calibration coefficient, $H_@$. Close the coarse adjust valve and open the fine adjust valve. Check that the H manometer fluid is zeroed, and the valve switch is off (down). Start the pump and open the coarse vacuum control knob ¼ turn. Switch the valve control to the up position and slowly close the fine vacuum control valve. Open the vacuum controls until the orifice meter differential (H) is set to the valve of the orifice meter calibration coefficient ($H_@$). Time the flow through the dry gas meter for 1 minute. The flow rate should be 0.75 ft^3/min. If the flow rate does not agree with 0.75 ft^3/min., the meter box should be recalibrated before use.

4.4.21 Complete assembly of the sampling box. Attach the filter holder to the inlet of the modular elbow. The umbilical sample head hook-up should be connected to the outlet of the 4th impinger. Check that all joints are greased, firmly seated, and held with ball joint clamps.

4.4.22 Attach the umbilical to the meter box and sample box.

4.4.23 Leak check the sample train. Plug the inlet to the filter holder. Close the coarse vacuum adjust valve and open the fine vacuum adjust valve. Start the pump. Partially open the coarse adjust valve and slowly close the fine adjust valve until a vacuum of 15" Hg is obtained. <u>DO NOT</u> reverse the direction of the fine adjust valve; this will cause water to back-up into the filter holder. When the leak check is completed, <u>FIRST</u> slowly remove the plug from the filter inlet and then immediately stop the vacuum. This prevents water from backing up into the filter holder and silical gel from being entrained backwards into the third impinger. If this happens, the sample train must be cleaned and reassembled before the test resumes.

The leakage rate should not exceed 0.02 ft^3/min. If this limit is exceeded, carefully examine all glass joints and umbilical connections until the leak is identified and eliminated.

4.4.24 Attach the probe to the filter inlet. Turn on the filter compartment heater and the probe heater. Set the probe heater rheostat control using the graphs in figure 3. The probe and filter compartment should be at 250°F before starting the sampling.

4.4.25 Be sure the bolt and wing nuts are in the interface flange of the sample case to prevent the two halves from separating during the test. Check that the sample case support bar is securely attached to the case. Attach the support trolley to the sample case and suspend the sample case from the monorail attached to the stack.

4.4.26 Carefully insert the probe into the stack. Be careful not to bang the nozzle tip or pitot tips on the stack nipple or the inside stack wall. Position the nozzle at a point in the stack where the velocity pressure most nearly approximates the average velocity pressure determined during the initial velocity traverse. Secure the sample case at this position. (If the stack is under a large negative static pressure, start the pump before inserting the probe.)

4.4.27 Record the dry gas meter reading and start the sampling pump. Adjust the sampling flow rate to obtain the desired H setting as determined from the nomograph. Record all the data requested on the sampling field data form.

4.4.28 The sampling train should be run 4.5-5.0 hours unattended. Periodically check the sampling train and baghouse for operational status.

4.4.29 Prior to stopping the sampling pump, conduct a velocity traverse in the stack. Record all the data requested on the sampling field data form.

4.4.30 Stop the sampling pump and carefully withdraw the probe from the stack. If the stack is under a large negative static pressure, the pump should not be stopped until after the probe has been withdrawn. Record the dry gas meter reading.

4.4.31 Cover the end of the probe and disassemble the sample case. Seal the inlet/outlet of the first and fourth impingers, cover the end of the probe, and replace the filter assembly in the plastic bag.

4.4.32 On the meter control console, turn off all switches, close the coarse vacuum adjust valve, open the fine vacuum adjust valve, and disconnect the tygon tubing from the inclined manometer taps and close the taps. Coil the umbilical and snap all the quick disconnects and amphenol connectors together.

4.4.33 Return the sample case to the laboratory for sample recovery.

Train Operation - Roof Monitor Point Source

4.4.34 Determine the barometric pressure and record the value on the field data sheet.

4.4.35 Using a stopwatch, adjust the sampling rate to approximately 0.60 ± 0.05 ft^3/min.

4.4.36 Assemble the sampling train. Check all ground glass joints to see that they are securely seated. All clamps should be tight.

4.4.37 Connect the sample case to the sampling pump using the neoprene sample line.

4.4.58 Plug the probe heater into the rheostat control. Set the rheostat so a probe temperature of 250°F is obtained. Use the graphs in figure 3 to determine the proper rheostat setting.

4.4.39 Position the probe over the point source so that any water droplets being blown through the mist eliminators will not be sampled.

4.4.40 Record the dry gas meter reading and start time while starting the sampling pump. Adjust the sampling rate to 0.60 ± 0.05 ft^3/min. if necessary. Record all the information requested on the field sampling data form.

4.4.41 Determine the average stack velocity using the vane anemometer. Measure the velocity on a 4 X 4 grid in the same horizontal plane as the probe nozzle.

4.4.42 Allow the sampler to run 4.5-5.0 hours unattended. Periodically check the sampler and the potline operational status.

4.4.43 Before stopping the sampling pump, check the sampling rate using a stopwatch. Record all the data requested on the field sampling data form. Determine the average stack velocity as done at the start of the testing period.

4.4.44 Stop the sampling pump while recording the stopping time and the dry gas meter reading.

4.4.45 Disassemble the sample case. Cover the ends of the sample probe, reseal the inlet/outlet of the first and fourth impingers, and replace the filter assembly in the plastic bag.

4.4.46 Transport the sample case to the laboratory for sample recovery.

Sample Recovery

4.4.47 Carefully disassemble the filter holder assembly and place the filter in a plastic petri dish. Brush any particulate matter or filter fibers adhering to the filter holder gasket onto the filter.

4.4.48 ACETONE RINSE: While the probe nozzle is plugged, carefully brush off any particulate adhering to the external surface of the nozzle and probe. Discard this particulate. Rinse all sample exposed surfaces with acetone, i.e., nozzle, probe, cyclone eliminator or cyclone flask, and front half of the filter holder assembly. Using a nylon brush and acetone, thoroughly clean all the surfaces until no visible material remains. The acetone rinse should be placed in a glass container. Use the minimum amount of acetone necessary to clean the surfaces well.

4.4.49 WATER RINSE: Volumetrically measure (to the nearest 1 ml) the water in the first three impingers and then place it in a polypropylene container. With distilled water, rinse the impingers, U-tubes, and back half of the filter holder three times. Place the rinsings in the polypropylene bottle. Do not use more than 500 ml of rinse water.

4.4.50 Reweigh (to the nearest 1 gram) the silica gel in the fourth impinger. Place the silica gel in a drying oven set at 350°F for 2 hours. The regenerated silica gel should be a bright blue.

4.4.51 Record the appropriate data on the lab analysis forms and submit the filter, acetone rinse, and water rinse to the laboratory. Submit an acetone blank for each new batch of acetone that is used.

4.4.52 Remove the silicone grease from the ground glass joints using 50% NaOH solution and paper towels (Caution: wear plastic gloves). Clean all glassware with an Alconox solution and store it in the cabinet until next usage occurs.

4.6 Calculations

4.6.1 Dry gas volume, ft^3, corrected to standard conditions (68°F & 29.92" Hg).

$$V_{m(std)} = V_m \frac{T_{std}}{T_m} \frac{P_{bar} + (\quad H \div 13.6)}{P_{std}} \qquad \text{eqn. 1}$$

Where:

$V_{m(std)}$ = Volume of gas through dry gas meter (standard conditions), ft^3

V_m = Volume of gas through dry gas meter (meter conditions), ft^3

T_{std} = Absolute temperature at standard conditions, 528°R

T_m = Average dry gas meter temperature, °R

P_{bar} = Barometric pressure at the orifice meter, in. Hg

H = Average pressure drop across orifice meter, in. Hg

13.6 = Specific gravity of mercury

P_{std} = Absolute pressure at standard conditions, 29.92" Hg

4.6.2 Volume of water vapor.

$$V_{w(std)} = V \; \frac{P_{H_2O} \; R \; T_{std}}{M_{H_2O} \quad P_{std}}$$

eqn. 2

Where:

$V_{w(std)}$ = Volume of water vapor in the gas sample (std. conditions), ft^3

V = Total volume of liquid collected in impingers and silica gel, ml

P_{H_2O} = Density of water, 0.0022 lb/ml

M_{H_2O} = Molecular weight of water, 18 lb/lb-mole

R = Ideal gas constant, 21.83 in. Hg - ft^3/lb-mole-°R

T_{std} = Absolute temperature at standard conditions, 528°R

P_{std} = Absolute pressure at standard conditions, 29.92" Hg

4.6.3 Moisture content.

$$B_{wo} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

eqn. 3

Where:

B_{wo} = Proportion by volume of water vapor, dimensionless

$V_{w(std)}$ = Volume of water in the gas sample (std. conditions), ft^3

$V_{m(std)}$ = Volume of gas sample through the dry gas meter (std. conditions), ft^3

4.6.4 Stack volumetric flow rate, dry basis (std. conditions), ft^3/min.

$$V_{sd} = V_s \; (1 - B_{wo})$$

eqn. 4

Where:

V_{sd} = Stack volumetric flow rate, dry basis (std. conditions), ft^3/min.

V_s = Stack volumetric flow rate (std. conditions), ft^3/min., as determined by Method S-2 "Average Velocity in a Duct (Pitot Method)" or Method S-3 "Average Velocity Measurement (Vane Anemometer)."

B_{wo} = Proportion by volume of water vapor, dimensionless

4.6.5 Concentration of fluoride (or particulate) in stack gas (std. conditions), dry basis, mg/m^3.

$$C_s = \frac{F_t}{V_{m(std)}} \quad \frac{35.31\ ft^3}{m^3} \qquad \text{eqn. 5}$$

Where:

C_s = Concentration of fluoride (or particulate) in stack gas (std. conditions), dry basis, mg/m^3

F_t = Total weight of fluoride (or particulate) in sample, mg

$V_{m(std)}$ = Volume of gas sample through the dry gas meter (std. conditions), ft^3

4.6.6 Mass emission rate of fluoride (or particulate), lbs/day

$$Q = C_s \quad V_{sd} \quad K \quad N$$

Where:

Q = Mass emission rate of fluoride (or particulate), lbs/day

C_s = Concentrations of fluoride (or particulate) in stack gas (std. conditions), dry basis, mg/m^3

V_{sd} = Stack volumetric flow rate, dry basis, (std. conditions, ft^3/min.

K = Dimensional proportionality constant,

8.99×10^{-5} - lb - min/ft^3 - mg - day

N = Number of fans in operation on the dry scrubber at the time of the test.

4.6.7 The results from Part 6 above represents the mass emission rate in lbs/day from a single emission point within a dry or wet scrubber system. It has been determined that the emission rate from each emission point within a scrubber system that was not specifically tested is reflected by the geometric mean of the test results. Therefore, the geometric mean of the test results for each scrubber system on a potline must be determined, and this average is applied to the formula in COMAR 26.11.06 07C(2).

4.7 References.

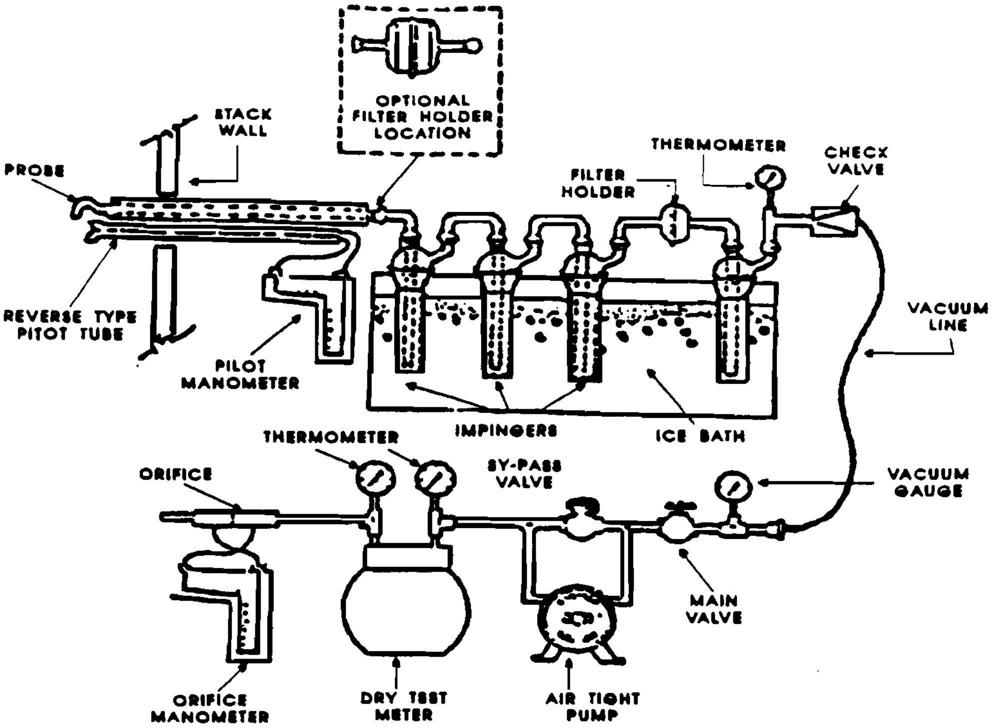
4.7.1 Federal Register, Vol. 3, No. 206, October 23, 1974, pp. 37730-37736.

4.7.2 Rom, Jerome, J., "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment," Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0576.

4.7.3 EPA-650.4-74-005d "Guidelines for Development of a Quality Assurance Program: Volume IV - Determination of Particulate Emissions from Stationary Sources."

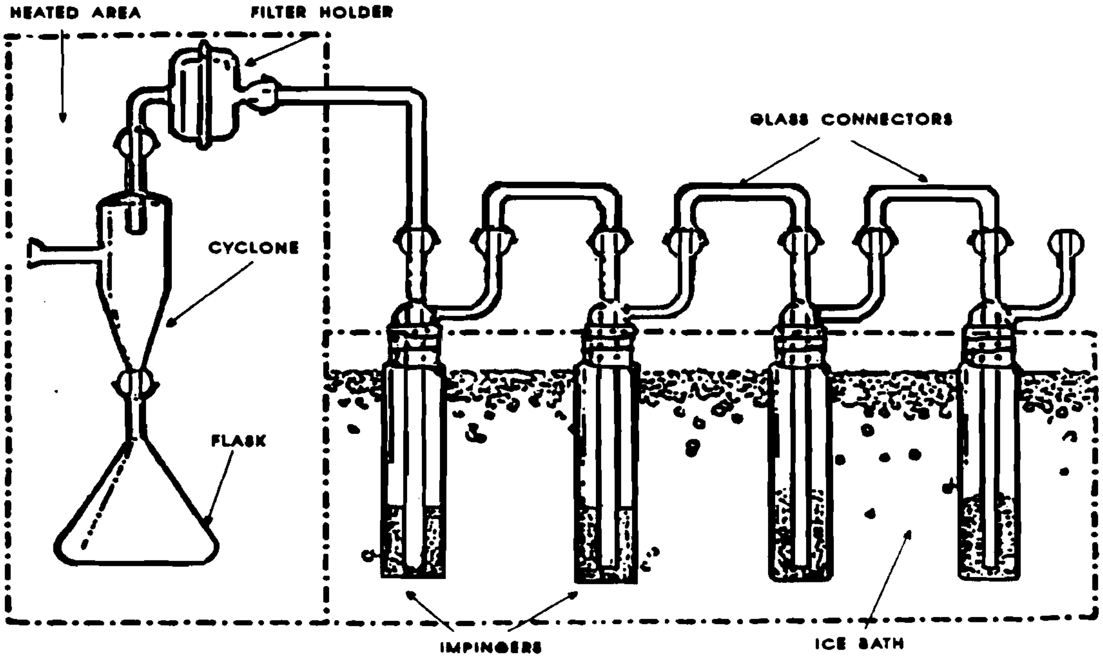
4.7.4 Brenchley, Turley, and Yarmac, Industrial Source Sampling, Ann Arbor Science: Ann Arbor, Michigan, 1974.

Figure 1

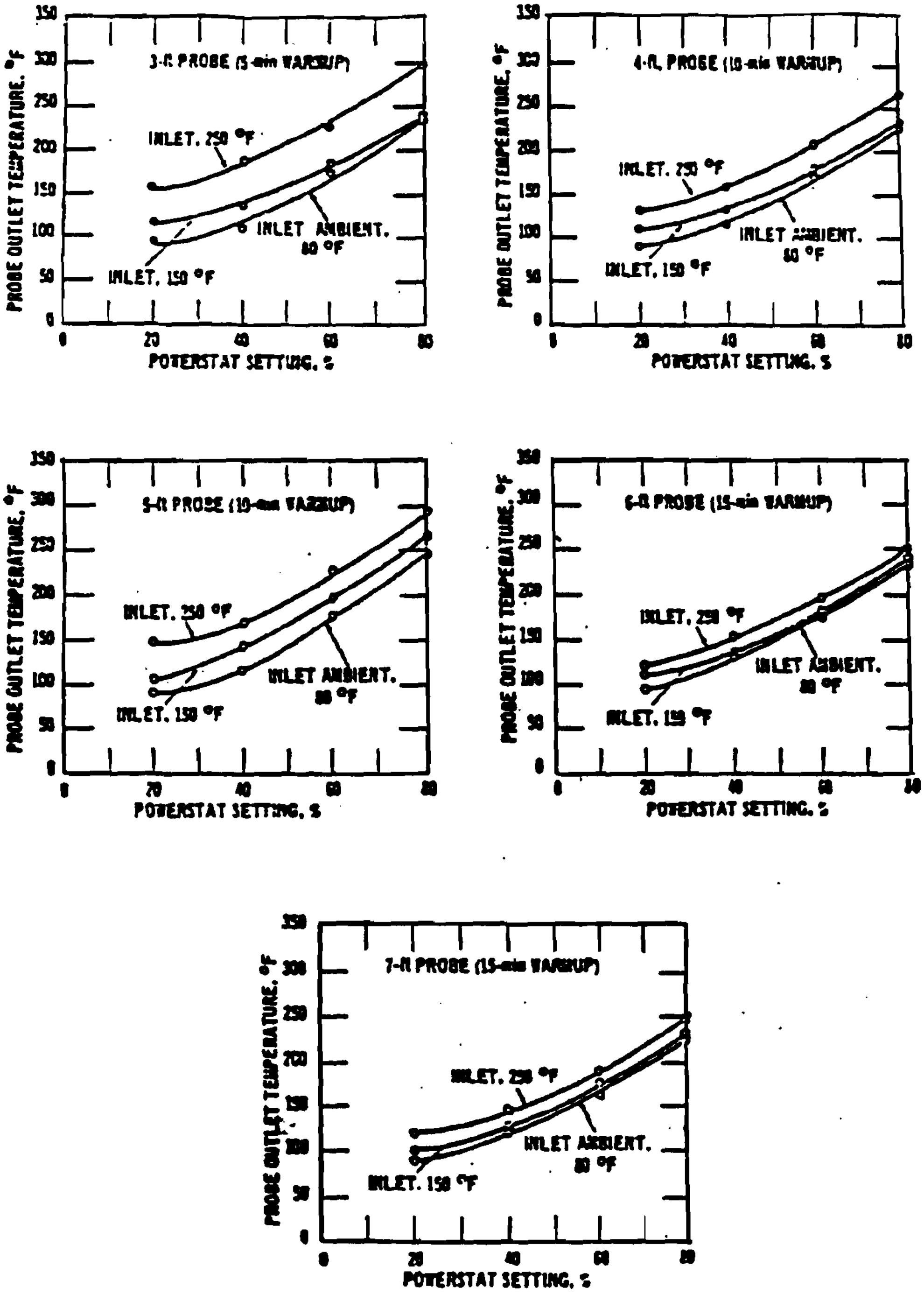


FLUORIDE SAMPLING TRAIN

Figure 2



SAMPLE BOX GLASSWARE ASSEMBLY



NOTE: Flow rate held constant at 0.75; 50% change in flow rate has little effect on probe temperature.

Probe Temperatures

EMISSION SAMPLING ANALYTICAL DATA

Test I.D._____S_____ Date Received_____A_____ Results to:

Test Date_____S_____ Date Reported_____A_____ _____S_____

<u>Moisture</u> Technician_____S_____

Impinger Volume		Silica Gel		Total Water Volume
finish	__S__	f.__S__	__S__	
start	__S__ml	s.__S__	__S__	
net	__S__ml	n.__S__g	__S__g	__S__ml

<u>Filter</u> Technician_____A_____

Identification_____S_____ Sample to be saved:S ☐ yes ☐ no

Fluoride_____A_____mg Particulate_____A_____mg

<u>Probe</u> Technician_____A_____

Identification_____S_____ Sample to be saved:S ☐ yes ☐ no

Fluoride_____A_____mg Particulate_____A_____mg

<u>Impingers</u> Technician_____A_____

Identification_____S_____ Sample to be saved:S ☐ yes ☐ no

Fluoride_____A_____mg

<u>Totals</u>

Total particulate _____A_____mg

Particulate fluoride _____A_____mg

Gaseous fluoride _____A_____mg

Total fluoride _____A_____mg

S = filled in by samplers

A = filled in by analysts

DRY SCRUBBER EMISSIONS TEST FIELD DATA

Location______________________________

Run #_________________________________

Date:__________/__________/___________

Technicians___________________________

Sample Box #__________________________

Meter Box #___________________________

Meter box ΔH@_________________________

Nomograph #___________________________

Reference ΔP__________________________

Assumed %H_2O_______________________

Leak check:

Filter #______________________________

Imp. Vol.:S_______________/ E_____________

Sil. Gel.:S_______________/ E_____________

Bar. Press:S______________/ E_____________

Amb. Temp.:S______________/ E_____________

Probe #_______________length_______________ft.

Cp:___________________________________

Nozzle #______________diam._______________in.

Stat. Press.__________________________"H_2O

Fans Oper. S________________/ E_____________

Calibration check:

________________CFM @_______________"Hg

____________________________CFM @ ΔH@

Time	DGM (ft³)	ΔP ("H_2O)	ΔH ("H_2O)		DGM ( )		Stack ( )	Imp. ( )	Vac. ("Hg)
			DES.	ACT.	IN	OUT			

DRY SCRUBBER EMISSIONS TEST OPERATING DATA

Test #__________________ Date____________________ Observer_________________

Fan Amps		**Cells Isolated During Test**				**Baghouse Feed (%)**
1		1		7		Fresh
2		2		8		Recycle
3		3		9		
4		4		10		
5		5		11		
6		6		12		

Potline Activity			
Section	tapping	anode change	sampling
Section	tapping	anode change	sampling
Section	tapping	anode change	sampling
Section	tapping	anode change	sampling

Comments: (baghouse maintenance being performed, feeder problems, etc.)

EMISSIONS CALCULATIONS - DRY SCRUBBER

Test I.D.__________ Test Date__________ Results By__________

Technicians__________ Report Date__________ Checked By__________

I. INPUT DATA

1. Barometric pressure __________"Hg
2. Average ΔH __________"Hg
3. Stack static pressure __________"Hg
4. Meter pressure
   a. for HP-65 program (1&2) __________"Hg
   b. for TI-59 program (2) __________"Hg
5. Stack pressure (1&3) __________"Hg
6. Dry gas meter calibration factor (Y) __________
7. Gas Volume sampled, total __________ft^3
8. Water volume collected, total __________ml
9. Meter temperature __________°F
   __________°R
10. Stack temperature __________°F
11. Stack area __________ft^3
12. Pitote coefficient - C_p __________
13. Number of fans running __________

II. OUTPUT DATA

1. Sample volume, STP __________ft^3
2. % H_2O __________
3. ACFM __________
4. SCFM __________

	lbs/Day	mg/ft^3	mg/m^3
Particulate			
Particulate fluoride			
Gaseous fluoride			
Total fluoride			

ROOF MONITOR POINT SOURCE EMISSIONS TEST DATA

Run # ______ Date ______ Operators ______

Probe # ______ Length ______ Nozzle # ______ Dia. ______

Filter # ______ Sample Box # ______ Pump Unit # ______

Amb. Temp. ______ °C of Baro. Press. ______ "Hg inlet/outlet test

		Start	Finish	Dif/Avg/Total
Silica gel wt. (grams)	a)			
	b)			
Impinger vol., total (ml)	a)			
	b)			
Dry gas meter reading (ft^3)				
Start - Stop Time				
Flow rate (cfm)				
Stack temp. (°C °F)				
Meter inlet temp (°C °F)				
Meter outlet temp (°C °F)				
Vacuum ("Hg)				

Number of sources running in test section ______ in potline ______

Number of pots on line in test section ______ in potline ______

Potline activity in test section # ______ tapping anode change sampling

Potline activity in test section # ______ tapping anode change sampling

Comments:

EMISSIONS CALCULATIONS - ROOF MONITOR POINT SOURCE

Test I.D.______ Test Date______ Results By______

Technicians______ Report Date______ Checked By______

I. INPUT DATA

1. Barometric pressure ______"Hg
2. Dry gas meter pressure ______"Hg
3. Meter pressure
   a. for HP-65 program (1&2) ______"Hg
   b. for TI-59 program (2) ______"Hg
4. Stack pressure (1) ______"Hg
5. Gas volume sampled, actual ______ft^3
6. Water volume collected, total ______ml
7. ACFM ______
8. Meter temperature ______°F
   ______°R
9. Stack temperature ______°F
   ______°R
10. Start time ______
11. Stop time ______

II. OUTPUT DATA

1. Sample volume, STP ______ft^3
2. % H_2O ______
3. SCFM ______
4. Test length ______minutes

	lbs/Day	mg/ft^3	mg/m^3
Particulate			
Particulate fluoride			
Gaseous fluoride			
Total fluoride			

Method 1011
GASOLINE DISPENSING FACILITY
LEAK TEST PROCEDURE

1. Applicability

1.1 This test procedure is used to quantify the vapor tightness of any vapor recovery system installed at a gasoline dispensing facility (GDF). Leaks in a balance system may cause excessive vapor emissions. Leaks in a vacuum assist system may decrease the efficiency of the vapor collection or processing system.

2. Principle

2.1 The entire vapor recovery system is pressurized to ten (10) inches of water column and then allowed to decay for five (5) minutes. The acceptability of the final pressure is based upon the vapor system volume or ullage space.

3. Range

3.1 The minimum and maximum full-scale ranges of the pressure gauge are 0-10 and 0-20 inches of water column, respectively. Maximum incremental graduations of the pressure gauge shall be one-tenth of an inch water column.

4. Interferences

4.1 On vacuum assist systems the processor must be isolated and the vapor system capped. On a balance system the vent pipes must be capped or plugged. Any leakage at these points will show up as a system component leak.

5. Apparatus

5.1 Nitrogen. Use commercial grade nitrogen in a high pressure cylinder, equipped with a two-stage pressure regulator and a one psig pressure relief valve.

5.2 Pressure Gauge or Water Manometer. Use a 0-10 inch water column pressure gauge, or water manometer, to measure the pressure decay in the vapor recovery system. The pressure gauge shall be readable to the nearest tenth of an inch (0.1) water column.

5.3 Vent Cap Assembly. See Figure 30-1 for example.

5.4 "T" Connector Assembly. See Figure 30-2 for example.

5.5 Stopwatch. Use a stopwatch accurate to within 0.2 seconds.

6. Pre-Test Procedures

6.1 Dispensing shall not take place during the test. There shall have been no bulk drops into the storage tanks within the three hours prior to the test.

6.2 Measure the gasoline gallonage in each underground storage tank. Determine the actual capacity of each storage tank. Calculate the ullage space for each tank by subtracting the gasoline gallonage present from the actual tank capacity. The minimum ullage during the test shall be 30 percent of the tank capacity or 500 gallons, whichever is greater. The vent pipes may be manifolded during the test to achieve the required ullage.

6.3 Insure that all Phase II couplers are equipped with a locking dust cap. Replace the manhole covers as a safety precaution.

6.4 Disconnect the dispenser end of one vapor recovery hose and install the "T" connector assembly (see Figure 30-2). Connect the nitrogen gas supply (do not use air), and the pressure gauge to "T" connector.

6.4.1 For those Phase II systems utilizing a remote vapor check valve, the "T" connector assembly shall be installed on the vapor riser side of the check valve unless the remote check valve is disabled by removing the poppet on the fuel side.

6.5 Install the vent cap assembly(s) (see Figure 30-1). For manifolded systems all storage tanks vent pipes shall be capped during the test.

6.6 If the storage tank vent pipe is open, and easily accessible, a modified version of the "T" connector may be installed at the vent pipe (see Figure 30-3). This will allow the test to be conducted without any dispenser modifications. This is advantageous at certain facilities using coaxial Phase II systems.

7. Testing

7.1 Open the nitrogen gas supply valve, regulate the delivery pressure to 5 psig, and pressurize the vapor system (or subsystem for individual vapor return line systems) to or slightly above 10 inches H_2O initial pressure. It is critical to maintain the nitrogen flow until both flow and pressure stabilize, indicating temperature and vapor pressure stabilization in the tanks. Check the vent cap assembly(s) and "T" connector assembly using leak detecting solution to verify that the test equipment is leak tight.

7.2 Close the nitrogen supply valve and start the stopwatch when the pressure decreases to the initial starting pressure of 10 inches of water column.

7.3 After each minute record the system pressure. After five minutes, record the final system pressure. See Table 30-1 to determine the acceptability of the final system pressure results.

7.4 If the system failed to meet the criteria set forth in Table I repressurize the system and check all accessible vapor connections using leak detector solution or a combustible gas detector. If vapor leaks in the system are encountered, repair or replace the defective component and repeat the test.

7.5 Carefully remove the vent cap assembly(s). Allow any remaining pressure to be relieved through vent pipes(s). Keep all potential ignition sources away from the vent pipe(s).

7.6 After the pressure is relieved, remove the "T" connector assembly and reconnect the vapor recovery hose. If the fuel poppet was removed from a remote vapor check valve to conduct the test, carefully replace the poppet and reconnect the vapor hose.

7.7 If the vapor recovery system utilizes individual vapor lines, repeat the leak test for each of the other gasoline grades. Avoid leaving any vapor return line open longer than is necessary to install or remove the "T" connector assembly.

8. Reporting

8.1 The calculated ullage and system pressures for each five minute vapor recovery system test shall be reported as shown in Figure 30-4.

TABLE 30-1

GASOLINE DISPENSING FACILITY
LEAK RATE CRITERIA
INITIAL PRESSURE - 10 INCHES WATER COLUMN

ULLAGE SPACE (GALLONS)	MINIMUM PRESSURE AFTER FIVE MINUTES (Inches of Water)
500	3.7
600	4.5
700	5.2
800	5.8
900	6.2
1,000	6.5
1,500	7.6
2,000	8.2
2,500	8.5
3,000	8.7
3,500	8.9
4,000	9.1
4,500	9.2
5,000	9.3
7,500	9.5
10,000	9.6
15,000	9.7
30,000	9.8

Use linear interpolation for intermediate values of ullage space.

FIGURE 30 - 1

VENT CAP ASSEMBLY

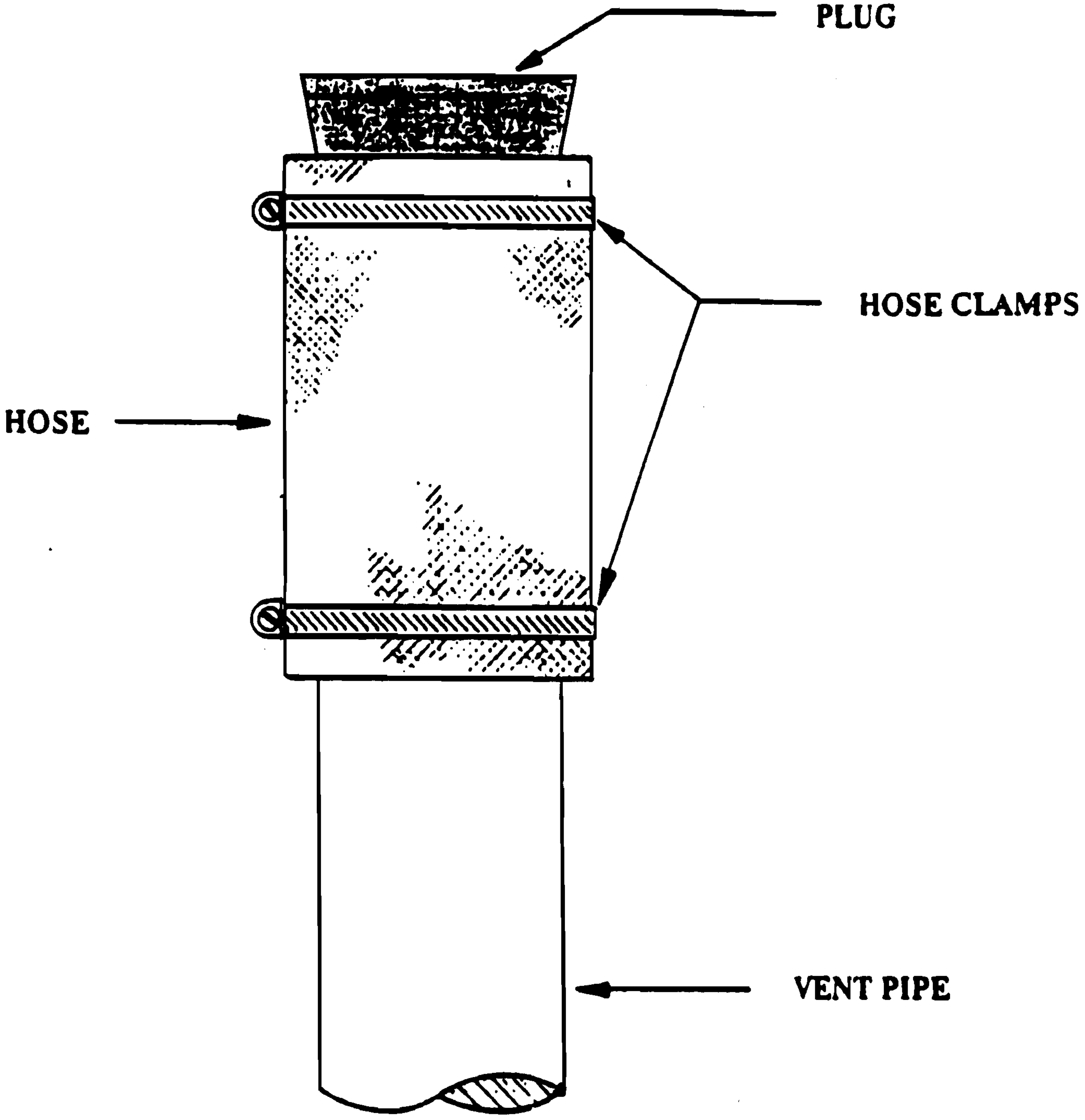


FIGURE 30 - 2

"T" CONNECTOR ASSEMBLY

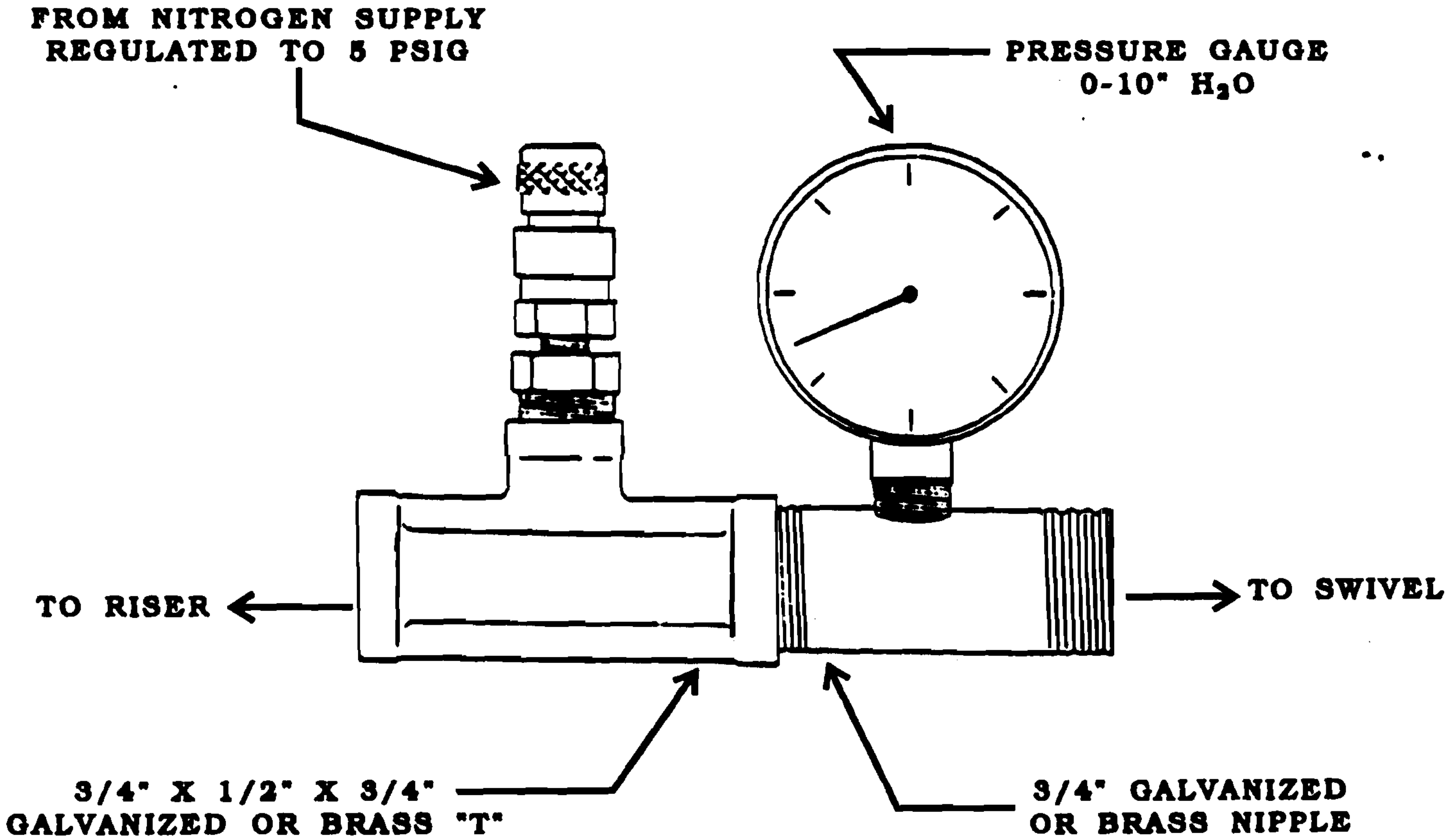
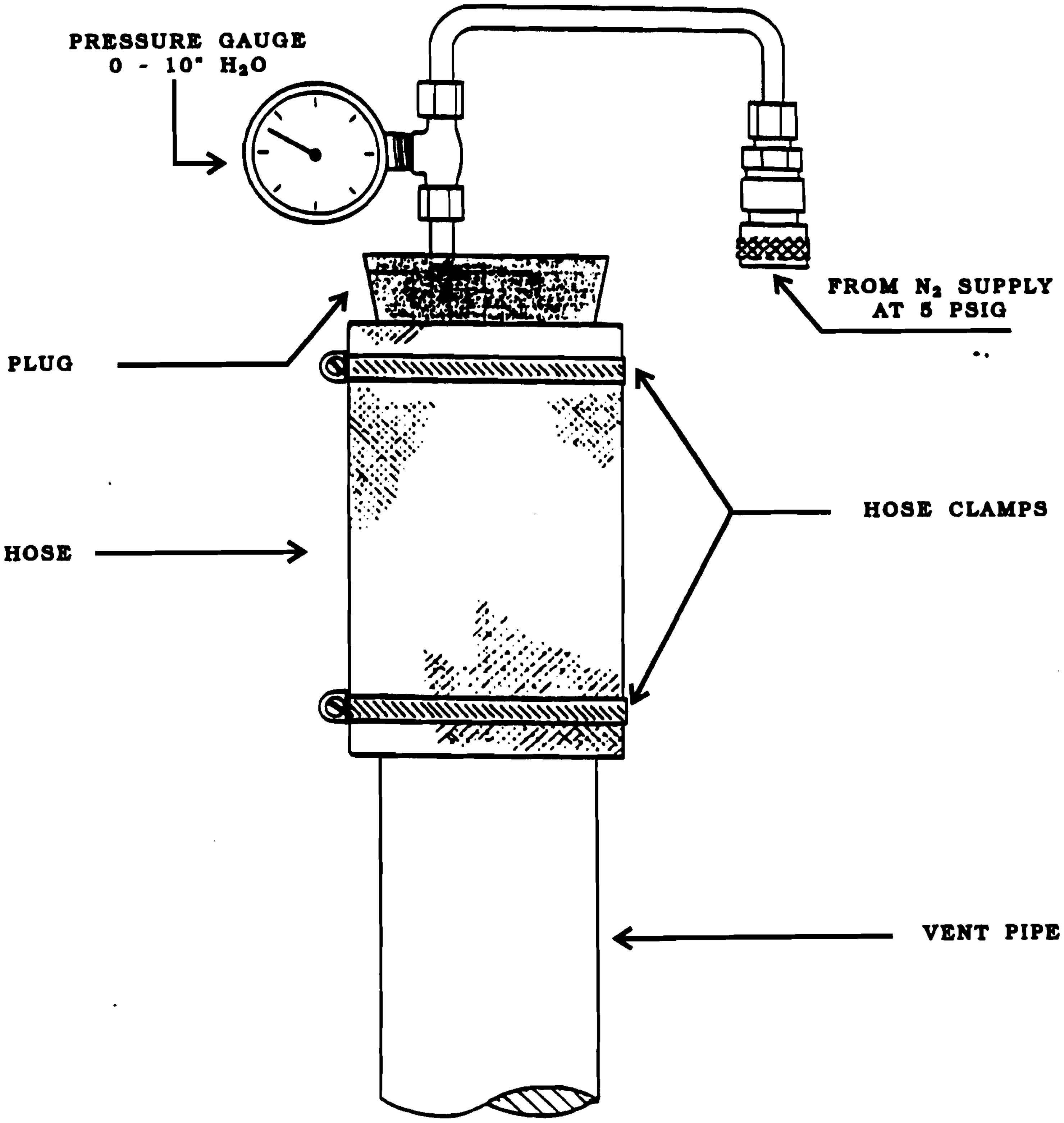


FIGURE 30 - 3

ALTERNATIVE VENT CAP ASSEMBLY



Test Date: ______
Test Times:
Run A: ______
Run B: ______
Run C: ______

FIGURE 30-4
SUMMARY OF SOURCE TEST RESULTS

SOURCE INFORMATION		FACILITY PARAMETERS	
Name and Address	Firm Representative and Title	PHASE II SYSTEM TYPE (Check One)	
		Balance	___
	Phone No.	Hirt	___
Permit Conditions	Source:	Red Jacket	___
	Vapor Recovery System	Hasstech	___
	Plant No. Permit No.	Healy	___
	Operates hr/day & days/yr	Manifolded? (Y or N)	___

Operating Parameters

Tank #	Capacity	Gallons Present
1	______	______
2	______	______
3	______	______

Applicable Regulations: **VN Recommended:**

Source Test Results and Comments:

Tank #:	1	2	3
Product Grade:	___	___	___
Actual Tank Capacity, gallons	___	___	___
Gasoline Volume, gallons	___	___	___
Ullage, gallons	___	___	___
Initial Pressure, inches H_2O	___	___	___
Pressure After 1 Minute, inches H_2O	___	___	___
Pressure After 2 Minutes, inches H_2O	___	___	___
Pressure After 3 Minutes, inches H_2O	___	___	___
Pressure After 4 Minutes, inches H_2O	___	___	___
Final Pressure After 5 Minutes, inches H_2O	___	___	___

NO COMMERCIAL USE OF THESE RESULTS IS AUTHORIZED

Test Conducted by	Test Company	Date of Test

Method 1012
GASOLINE DISPENSING FACILITY
DYNAMIC BACK PRESSURE

1. Applicability

1.1 This procedure is used to quantify the dynamic back pressure in the vapor path leading from the dispensing nozzle to the underground tank, inclusively. It is applicable in all cases where vapor balance or Hirt vacuum assist Phase II systems are utilized.

2. Principle

2.1 The dynamic back pressure during refueling is simulated by passing nitrogen through the Phase II recovery system at a constant rate. The resultant dynamic back pressure is measured using a pressure gauge. Alternate Methods 1 and 2 are also included for those Phase II systems which utilize a remote vapor check valve.

3. Range

3.1 The minimum and maximum dynamic back pressures that can be measured are dependent upon available pressure gauges. Recommended gauge ranges are 0-.5 inches H_2O and 0-2 inches H_2O for Alternate Methods 1 and 3. Recommended ranges for Alternate Method 2 are 0-.50 inches H_2O and 0-1 inch H_2O.

4. Interferences

4.1 Any leaks in the nozzle vapor path, vapor hose, or underground vapor return piping will result in erroneously low dynamic back pressure measurements.

5. Apparatus

5.1 Nitrogen High Pressure Cylinder with Pressure Regulator. Use a high pressure nitrogen cylinder capable of maintaining a pressure of 2000 psig and equipped with a compatible two-stage pressure regulator.

5.2 Rotameter. Use a calibrated rotameter capable of accurately measuring nitrogen flowrates of 20, 60, and 100 CFH and equipped with a control valve.

5.3 Pressure gauges. Use two Magnehelic differential pressure gauges, or equivalent, with appropriate ranges, and equipped with toggle valves connected to the high pressure inlets.

5.4 Automobile fill pipe. Use a fill neck known to be compatible with all vapor recovery nozzles and equipped with a pressure tap.

5.5 Nitrogen. Use a commercial grade nitrogen.

5.6 Hand Pump. Use a gasoline compatible hand pump to drain condensate pots.

6. Pre-Test Procedures

6.1 For those Phase II systems which do not utilize a remote vapor check valve, assemble the apparatus as shown in Figure 27-1, ensuring that the riser shut-off valve on the test equipment is closed. If a Hirt Phase II system is used, the vacuum producing device should be turned off during this test.

6.2 The test equipment must be leak-checked prior to use. Plug the nozzle end of the auto fill pipe, open the nitrogen cylinder and the toggle valves on the magnehelic gauges. Adjust the flow meter control valve until a pressure of 50 percent of full scale is indicated on the high range pressure gauge. Close the nitrogen cylinder valve and toggle valves. A pressure decay of 0.2 inches H_2O, in five minutes, is considered acceptable.

6.3 Perform an initial visual examination for vapor leaks at the nozzle and hose of the Phase II system to be tested.

6.4 Disconnect and drain the vapor hose for all dispensers to be tested. Pour two (2) gallons of gasoline into each vapor return riser. Reconnect vapor hose. Allow fifteen (15) minutes for liquid in the vapor return piping to drain. For Phase II systems which do not employ a remote vapor check valve, the 2 gallons of gasoline may be introduced through the vapor passage in the nozzle.

6.5 Completely drain all gasoline from the spout and bellows.

6.6 For those vapor piping configurations which utilize a condensate pot, drain the pot prior to testing.

6.7 For Alternate Methods 1, 2, and 3 the Phase I vapor poppet shall be propped open in such a manner that the valve is not damaged.

7. Testing

7.1 **Alternate Method 1.** Phase II systems which do not utilize a remote vapor check valve may be tested using the following methodology. Insert the nozzle into the fill pipe of the pressure drop test unit, ensuring that a tight seal at the fillpipe/nozzle interface is achieved. Ensure that the riser shut-off valve on the test equipment is closed.

7.2 Close both toggle valves and connect the nitrogen supply.

7.3 Open the nitrogen supply, set the delivery pressure to 10 psig, and use the flowmeter control valve to adjust the flowrate to 20 CFH.

7.4 Open the toggle valve on the 0-.5 inches H_2O gauge. If the pressure is greater than 0.5 inches H_2O, close this valve and use the 0-2 inches H_2O gauge.

7.5 A pulsating gauge needle indicates nitrogen passing through a liquid obstruction in the vapor return system. If this occurs, close the flowmeter control valve, disengage the nozzle and redrain the nozzle and hose assembly. Re-engage the nozzle, open the flowmeter control valve and repeat the test.

7.6 Repeat Sections 7.3 through 7.5 for nitrogen flowrates of 60 and 100 CFH.

7.7 The following information should be recorded on the field data sheet shown in Figure 27-2:

Pump Number and Product Grade
Nozzle make and model
Nitrogen flowrate, CFH
Dynamic back pressure, inches H_2O

7.8 Close and replace the dust cover on the phase I poppet.

7.9 **Alternate Method 2.** Phase II systems which utilize a remote vapor check valve may be tested using the following methodology.

7.9.1 Disconnect the vapor recovery hose from the remote vapor valve. Test the nozzle/hose assembly pursuant to Sections 7.1 through 7.8 and record the results.

7.9.2 Disconnect the vapor check valve from the riser and connect a compatible pipe fitting to the riser as shown in Figure 27-1.

7.9.3 Plug the nozzle end of the fill pipe on the pressure drop test unit and open the riser shut-off valve on the test equipment.

7.9.4 Repeat Sections 7.2 through 7.8. In addition to the information required in Section 7.7, record the make and model of the remote vapor check valve.

7.9.5 Record on the field data sheet the pressure drop across the remote vapor check valve. This data is available from the manufacturer.

7.9.6 Add the dynamic back pressures, for each nitrogen flowrate, obtained from Sections 7.9.1, 7.9.4, and 7.9.5.

7.10 Alternate Method 3. Phase II balance and Hirt systems which use those models of remote vapor check valves which can be disabled by removing the poppet on the fuel side may be tested using the following methodology. The Emco-Wheaton A-228 remote vapor check valve cannot be tested using this method.

7.10.1 Carefully open the fuel side of the remote vapor check valve and remove the fuel poppet. Carefully replace the threaded plug on the fuel side of the valve.

7.10.2 Test the Phase II system pursuant to Sections 7.1 through 7.8, recording the data on the field data sheet shown in Figure 27-2.

7.10.3 Carefully reassemble the remote vapor check valve by removing the plug on the fuel side and reinserting the fuel poppet. Replace the threaded fuel plug.

8. Reporting

8.1 Results from Alternate Methods 1 or 3 shall be reported as shown in Figure 27-2. Results from Alternate Method 2 shall be reported as shown in Figure 27-3. The maximum allowable system dynamic back pressures, with the dry brakes open, are as follows:

NITROGEN FLOWRATE, CFH	**DYNAMIC BACK PRESSURE, INCHES H_2O**
20	0.15
60	0.45
100	0.95

Figure 27 - 1
PRESSURE DROP TEST UNIT

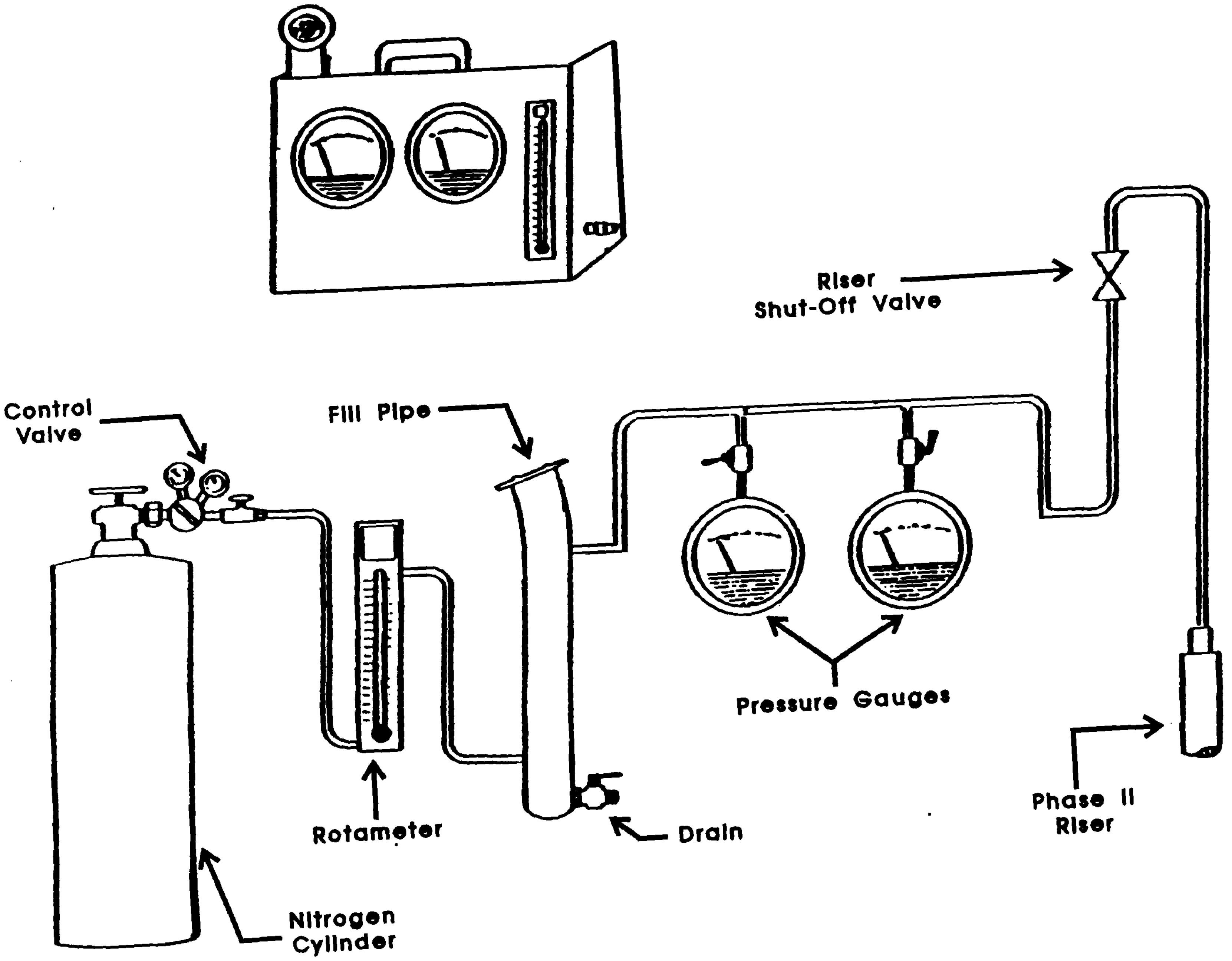


FIGURE 27-2
SUMMARY OF SOURCE TEST RESULTS

Company Name and Address

Testing Company Name and Address

Pump #	Gasoline Grade	Nozzle Mfg. & Model Number	Dynamic Back Pressure, INS. W. C.			Comments
			20 CFH	60 CFH	100 CFH	

Test Conducted By

Date

Company Name ____________ **Address** ____________ **City** ____________ **Phone** ________

Test Date ____________ **Test Company** ____________ **Address** ____________ **City** ________

FIGURE 27-3
SUMMARY OF SOURCE TEST RESULTS

PUMP #	GAS GRADE	NOZZLE MODEL	NITROGEN FLOW, CFH	VAPOR VALVE MODEL	DYNAMIC BACK PRESSURE, INCHES OF WATER COLUMN			
					NOZZLE/HOSE ASSEMBLE	RISER TO U.G. TANK	VAPOR VALVE	TOTAL Δ P, IN H_2O

Supplement 2

Appendix A

List of EPA Approved Test Methods
Found in 40 CFR

The following listed test methods are all found in the Code of Federal Regulations, 40 CFR Part 60, Appendix A. They are listed here for the convenience of those sources and testing contractors who are required to demonstrate compliance through an appropriate test. Although the Department may provide guidance on the type of test to be performed in any demonstration of compliance, any significant deviation from the procedures in the CFR must be approved by the Department and by EPA before the test is performed.

Method	Title
1	Sample and velocity traverses for stationary sources
1A	Sample and velocity traverses for stationary sources with small stacks or ducts
2	Determination of stack gas velocity and volumetric flow rate (Type S pitot tube)
2A	Direct measurement of gas volume through pipes and small ducts
2B	Determination of exhaust gas volume flow rate from gasoline vapor incinerators
2C	Determination of stack gas velocity and volumetric flow rate in small stacks or ducts (standard pitot tube)
2D	Measurement of gas volumetric flow rates in small pipes and ducts
3	Gas analysis for carbon dioxide, oxygen, excess air, and dry molecular weight
3A	Determination of oxygen and carbon dioxide concentrations in emissions from stationary sources (instrumental analyzer procedure)
4	Determination of moisture contentin stack gases
5	Determination of particulate emissions from stationary sources

Appendix A (continued)

Method	Title
5A	Determination of particulate emissions from the asphalt processing and asphalt roofing industry
5B	Determination of nonsulfuric acid particulate matter from stationary sources
5C	[Reserved]
5D	Determination of particulate emissions from positive pressure fabric filters
5E	Determination of particulate emissions from the wool fiberglass insulation manufacturing industry
5F	Determination of nonsulfate particulate matter from stationary sources
5G	Determination of particulate emissions from wood heaters from a dilution tunnel sampling location
5H	Determination of particulate emissions from wood heaters from a stack location
6	Determination of sulfur dioxide emissions from stationary sources
6A	Determination of sulfur dioxide, moisture, and carbon dioxide emissions from fossil fuel combustion sources
6B	Determination of sulfur dioxide and carbon dioxide daily average emissions from fossil fuel combustion sources
6C	Determination of sulfur dioxide emissions from stationary sources (Instrumental Analyzer Procedure)
7	Determination of nitrogen oxide emissions from stationary sources
7A	Determination of nitrogen oxide emissions from stationary sources-ion chromatographic method
7B	Determination of nitrogen oxide emissions from stationary sources (ultraviolet spectrophotometry)
7C	Determination of nitrogen oxide emissions from stationary sources-alkaline-permanganate/ colorimetric method

Appendix A (continued)

Method	Title
7D	Determination of nitrogen oxide emissions from stationary sources-alkaline-permanganate/ion chromatographic method
7E	Determination of nitrogen oxides emissions from Stationary Sources (instrumental analyzer procedure)
8	Determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources
9	Visual determination of the opacity of emissions from stationary sources
Alt 1	Determination of the opacity of emissions from stationary sources remotely by lidar
10	Determination of carbon monoxide emissions from stationary sources
10A	Determination of carbon monoxide emissions in certifying continuous emission monitoring systems at petroleum refineries
10B	Determination of carbon monoxide emissions from stationary sources
11	Determination of hydrogen sulfide content of fuel gas streams in petroleum refineries
12	Determination of inorganic lead emissions from stationary sources
13A	Determination of total fluoride emissions from stationary sources-SPADNS zirconium lake method
13B	Determination of total fluoride emissions from stationary sources-specific ion electrode method
14	Determination of fluoride emissions from potroom roof monitors for primary aluminum plants
15	Determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide emissions from stationary sources
15A	Determination of total reduced sulfur emissions from sulfur recovery plants in petroleum refineries
16	Semicontinuous determination of sulfur emissions from stationary sources
16A	Determination of total reduced sulfur emissions from stationary sources (impinger technique)
16B	Determination of total reduced sulfur emissions from stationary sources

Appendix A (continued)

Method	Title
17	Determination of particulate emissions from stationary sources (instack filtration method)
18	Measurement of gaseous organic compound emissions by gas chromatography
19	Determination of sulfur dioxide removal efficiency and particulate, sulfur dioxide and nitrogen oxides emission rates
20	Determination of nitrogen oxides, sulfur dioxide, and diluent emissions from stationary gas turbines
21	Determination of volatile organic compound leaks
22	Visual determination of fugitive emissions from material sources and smoke emissions from flares
23	[Reserved]
24	Determination of volatile matter content, water content, density, volume solids, and weight solids of surface coatings
24A	Determination of volatile matter content and density of printing inks and related coatings
25	Determination of total gaseous nonmethane organic emissions as carbon
25A	Determination of total gaseous organic concentration using a flame ionization analyzer
25B	Determination of total gaseous organic concentration using a nondispersive infrared analyzer
27	Determination of vapor tightness of gasoline delivery tank using pressure-vacuum test
28	Certification and auditing of wood heaters
28A	Measurement of air to fuel ratio and minimum achievable burn rates for wood fired appliances

APPENDIX B
COMAR CITATIONS TO THIS TECHNICAL MEMORANDUM

The following Air Quality regulations require the use of test methods or equipment specifications contained in this document:

COMAR 26.11.01 General Administrative Provisions
Regulation .01 Definitions
Regulation .04 Testing and Monitoring

COMAR 26.11.04 State Ambient Air Quality Standards
Regulation .01 Fluorides

COMAR 26.11.06 General Emission Standards, Prohibitions, and Restrictions
Regulation .07 Control of Sources of Fluorides Emissions

COMAR 26.11.08 Control of Incinerators
Regulation .07 Requirements for Municipal Waste Combustors with a Burning Capacity Greater Than 50 Tons Per Day

COMAR 26.11.10 Control of Iron and Steel Production Installations
Regulation .07 Test and Observation Procedures

COMAR 26.11.13 Control of Gasoline and Volatile Organic Compound Storage and Handling
Regulation .04 Loading Operations
Regulation .05 Gasoline Leaks from Tank Trucks

COMAR 26.11.14 Control of Kraft Pulp Mill TRS Emissions
Regulation .05 Monitoring and Reporting Requirements

COMAR 26.11.19 Volatile Organic Compounds from Specific Processes
Regulation .02 Applicability, Determining of Compliance, Reporting, and General Requirements
Regulation .09 Volatile Organic Compound Metal Cleaning
Regulations .12 Dry Cleaning Installations

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COMAR 26.11.01.04C

Title Technical Memorandum 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January, 1991)

Adopted Supplement No. 1

TO: STATE DEPOSITORIES

The agency which incorporated by reference the document titled above has determined that it will be subject to frequent supplementation. To facilitate supplementation, the permanent document will be transferred from its present black Accopress binder to a navy blue three-ring binder.

In addition, Supplement 1 to this document, which has previously been sent to you in a black Accopress binder, has been adopted and is now ready for insertion in the permanent document binder. Please follow these instructions carefully.

Instructions:

1. Locate the permanent black Accopress binder having the COMAR number and document title appearing above.
2. Find enclosed a navy blue three-ring binder with the same COMAR number and document title.
3. Remove the entire contents of the black Accopress binder and insert the document pages in precisely the same order into the navy blue three-ring binder. Discard the empty black Accopress binder.
4. Locate a separate black Accopress binder bearing the same COMAR number and document name, and bearing the phrase "Proposed and Emergency Supplements" directly above the document name. Now follow carefully the "Remove/Insert" instructions appearing below.
5. Remove from the permanent navy blue three-ring binder the obsolete pages listed under the "Remove Pages" column, and insert into the permanent navy blue three-ring binder the new or replacement pages listed under the "Insert Pages" column. Each of these pages, except new pages UG1 and UG2, has "Supplement 1" at the lower right hand corner. Obsolete pages removed from the permanent navy blue three-ring binder may be retained in a separate place for legal research, or discarded.

(Continued on next page)

ALL PAGES ARE INCLUSIVE

Remove Pages	Insert Pages
Title page-January 1991	Title page w/ Sup. 1
------	UG1--UG2
Table of Contents	i--ii
3--4	3--4
93--98	93--114
------	A1--A4
------	B1--B2

6. File this instruction sheet in the permanent navy blue three-ring binder behind the tab labeled "Instruction Sheet."

7. You will be sent further instructions about upcoming supplements when later action affects this document.

INQUIRIES TO: Dorothy M. Guy
AMA Regulations Coordinator
Air Management Administration
2500 Broening Highway
Baltimore, MD 21224
(410) 631-3240

COMAR 26.11.01.04C

Title Technical Memorandum 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January, 1991)

Adopted Supplement No. 2

TO: STATE DEPOSITORIES

Supplement 2 to the document Technical Memorandum 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January, 1991) has previously been sent to you in the Proposed and Emergency Supplements (PES) binder. Supplement 2 has been adopted and is now ready for insertion in the permanent document binder. Please follow these instructions carefully.

Instructions:

1. Locate the permanent and PES binders having the COMAR number and document title appearing above. Locate Supplement 2 in the PES binder.

2. Carefully follow the "Remove/Insert" instructions appearing below. Remove the obsolete pages listed under the column "Remove Pages" from the permanent binder. From the PES binder, insert the new or replacement pages listed under the column "Insert Pages" in the permanent binder. Each of these pages has "Supplement 2" at the lower right hand corner. The obsolete pages removed from the permanent binder may be retained in a separate place for legal research.

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Table of Contents (i--ii)	iii--iv
23--24	23--24
93--98	93--98
-----	115--162
B1--B2	B1--B2

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4. You will be sent further instructions about upcoming supplements when later action affects this chapter.

INQUIRIES TO: Dorothy M. Guy, AMA Regulations Coordinator
Air Management Administration
2500 Broening Highway
Baltimore, MD 21224
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COMAR 26.11.01.04C

Title: Technical Memorandum 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January, 1991)

Proposed Supplement No. 3

TO: STATE DEPOSITORIES

Instructions: Supplement 3 to the document Memorandum 91-01 "Test Methods and Equipment Specifications for Stationary Sources" (January, 1991) is being proposed for adoption. Supplement 3 includes the following pages (all pages are inclusive):

Supplement History (pages i--ii)
Table of Contents (pages iii -- iv)
15 -- 18
75 -- 86
91 -- 92

There is no page removal or insertion to do in the permanent document binder at this time. You will be sent further instructions when later action affects this proposed supplement.

INQUIRIES TO: Deborah Rabin
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