

Issued in Fort Worth, Texas, on August 3, 1983.

C. R. Melugin, Jr.,

Director, Southwest Region.

[FR Doc. 83-28297 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

General Aviation District Office at Fort Worth, Texas; Closing

Notice is hereby given that on or about October 15, 1983, the General Aviation District Office at Fort Worth, Texas, will be closed. All services to the public formerly provided by this office will be provided by the General Aviation District Offices at Dallas, Lubbock, and San Antonio, Texas. This information will be reflected in the FAA Organization Statement the next time it is reissued.

(Sec. 313(a), 72 Stat. 752; 49 U.S.C. 1354)

Issued in Fort Worth, Texas, on August 3, 1983.

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[FR Doc. 83-28298 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

General Aviation District Office at New Orleans, Louisiana, and its Satellite Office at Lafayette, Louisiana; Closing

Notice is hereby given that on or about November 1, 1983, the General Aviation District Office at New Orleans, Louisiana, and the New Orleans satellite office at Lafayette, Louisiana, will be closed. All services to the public formerly provided by these offices will be provided by the General Aviation District Office to be opened at Baton Rouge, Louisiana. This information will be reflected in the FAA Organization Statement the next time it is reissued.

(Sec. 313(a), 72 Stat. 752; 49 U.S.C. 1354.)

Issued in Fort Worth, Texas, on August 3, 1983.

C. R. Melugin, Jr.,

Director, Southwest Region.

[FR Doc. 83-28300 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

General Aviation District Office at Shreveport, Louisiana; Closing

Notice is hereby given that on or about November 1, 1983, the General Aviation District Office at Shreveport, Louisiana, will be closed. All services to the public formerly provided by this office will be provided by the General Aviation District Office to be opened at Baton Rouge, Louisiana, and the General Aviation District Office at Dallas, Texas. This information will be reflected

in the FAA Organization Statement the next time it is reissued.

(Sec. 313(a), 72 Stat. 752; 49 U.S.C. 1354)

Issued in Fort Worth, Texas, on August 3, 1983.

C. R. Melugin, Jr.,

Director, Southwest Region.

[FR Doc. 83-28299 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

Flight Standards District Office at Tulsa, Oklahoma; Closing

Notice is hereby given that on or about September 15, 1983, the Flight Standards District Office at Tulsa, Oklahoma, was closed. All services to the public formerly provided by this office will be provided by the General Aviation District Office at Oklahoma City, Oklahoma, and the Air Carrier District Office at Dallas/Fort Worth Airport, Texas. This information will be reflected in the FAA Organization Statement the next time it is reissued.

(Sec. 313(a), 72 Stat. 752; 49 U.S.C. 1354.)

Issued in Fort Worth, Texas, on August 3, 1983.

C. R. Melugin, Jr.,

Director, Southwest Region.

[FR Doc. 83-28301 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

National Airspace Review; Meeting

AGENCY: Federal Aviation Administration, DOT.

ACTION: Notice of meeting.

SUMMARY: Pursuant to Section 10(a)(2) of the Federal Advisory Committee Act (P.L. 92-463; 5 U.S.C. App. 1) notice is hereby given of a meeting of Task Group 3-1 of the Federal Aviation Administration (FAA) National Airspace Review Advisory Committee. The agenda for this meeting is as follows: A study of requirements and composition of flight plans for commonality and possible combination in a simple, uniform format.

DATE: Beginning November 14, 1983, at 11 a.m., continuing daily, except Saturdays, Sundays, and holidays, not to exceed one week.

ADDRESS: The meeting will be held at the Federal Aviation Administration, conference room 7 A/B, 800 Independence Avenue, S.W., Washington, D.C.

FOR FURTHER INFORMATION CONTACT: National Airspace Review Program Management Staff, Room 1005, Federal Aviation Administration, 800 Independence Avenue, S.W., Washington, D.C. 20591, (202) 426-3560.

Attendance is open to the interested public, but limited to the space available. To insure consideration, persons desiring to make statements at the meeting should submit them in writing to the Executive Director, National Airspace Review Advisory Committee, Air Traffic Service, AAT-1, 800 Independence Avenue, S.W., Washington, D.C. 20591, by November 7, 1983. Time permitting and subject to the approval of the chairman, these individuals may make oral presentations of their previously submitted statements.

Issue in Washington, D.C. on October 12, 1983.

John Watterson,

Acting Manager, Special Projects Staff Air Traffic Service.

[FR Doc. 83-28302 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

Air Carrier District Office at Houston, Tex; Closing

Notice is hereby given that on or about October 1, 1983, the Air Carrier District Office at Houston, Texas, was closed. All services to the public formerly provided by this office will be provided by the Air Carrier District Office at Dallas/Fort Worth Airport, Texas. This information will be reflected in the FAA Organization Statement the next time it is reissued.

(Sec. 313(a), 72 Stat. 752; 49 U.S.C. 1354)

Issued in Fort Worth, Texas, on August 3, 1983.

C. R. Melugin, Jr.,

Director, Southwest Region.

[FR Doc. 83-28303 Filed 10-17-83; 8:45 am]

BILLING CODE 4910-13-M

DEPARTMENT OF THE TREASURY

Office of the Secretary

[Dept. Circular; Public Debt Series No. 31-83]

Treasury Notes of October 31, 1985; Series Z-1985

October 13, 1983.

1. Invitation for Tenders

1.1. The Secretary of the Treasury, under the authority of Chapter 31 of Title 31, United States Code, invites tenders for approximately \$8,000,000,000 of United States securities, designated Treasury Notes of October 31, 1985, Series Z-1985 (CUSIP No. 912827 QB 1). The securities will be sold at auction, with bidding on the basis of yield. Payment will be required at the price

equivalent of the bid yield of each accepted tender. The interest rate on the securities and the price equivalent of each accepted bid will be determined in the manner described below. Additional amounts of these securities may be issued to Government accounts and Federal Reserve Banks for their own account in exchange for maturing Treasury securities. Additional amounts of the new securities may also be issued at the average price to Federal Reserve Banks, as agents for foreign and international monetary authorities.

2. Description of Securities

2.1. The securities will be dated October 31, 1983, and will bear interest from that date, payable on a semiannual basis on April 30, 1984, and each subsequent 6 months on October 31 and April 30 until the principal becomes payable. They will mature October 31, 1985, and will not be subject to call for redemption prior to maturity. In the event an interest payment date or the maturity date is a Saturday, Sunday, or other nonbusiness day, the interest or principal is payable on the next succeeding business day.

2.2. The income derived from the securities is subject to all taxes imposed under the Internal Revenue Code of 1954. The securities are subject to estate, inheritance, gift, or other excise taxes, whether Federal or State, but are exempt from all taxation now or hereafter imposed on the principal or interest thereof by any State, any possession of the United States, or any local taxing authority.

2.3. The securities will be acceptable to secure deposits of public monies. They will not be acceptable in payment of taxes.

2.4. Securities registered as to principal and interest will be issued in denominations of \$5,000, \$10,000, \$100,000, and \$1,000,000. Book-entry securities will be available to eligible bidders in multiples of those amounts. Interchanges of securities of different denominations and of registered and book-entry securities, and the transfer of registered securities will be permitted. Bearer securities will not be available, and the interchange of registered or book-entry securities for bearer securities will not be permitted.

2.5. The Department of the Treasury's general regulations governing United States securities apply to the securities offered in this circular. These general regulations include those currently in effect, as well as those that may be issued at a later date.

3. Sale Procedures

3.1. Tenders will be received at Federal Reserve Banks and Branches and at the Bureau of the Public Debt, Washington, D.C. 20226, prior to 1:30 p.m., Eastern Daylight Saving time, Wednesday, October 19, 1983. Noncompetitive tenders as defined below will be considered timely if postmarked no later than Tuesday, October 18, 1983, and received no later than Monday, October 31, 1983.

3.2. The face amount of securities bid for must be stated on each tender. The minimum bid is \$5,000, and larger bids must be in multiples of that amount. Competitive tenders must also show the yield desired, expressed in terms of an annual yield with two decimals, e.g., 7.10%. Common fractions may not be used. Noncompetitive tenders must show the term "noncompetitive" on the tender form in lieu of a specified yield. No bidder may submit more than one noncompetitive tender, and the amount may not exceed \$1,000,000.

3.3. Commercial banks, which for this purpose are defined as banks accepting demand deposits, and primary dealers, which for this purpose are defined as dealers who make primary markets in Government securities and report daily to the Federal Reserve Bank of New York their positions in and borrowings on such securities, may submit tenders for account of customers if the names of the customers and the amount for each customer are furnished. Others are permitted to submit tenders only for their own account.

3.4. Tenders will be received without deposit for their own account from commercial banks and other banking institutions; primary dealers, as defined above; Federally-insured savings and loan associations; States, and their political subdivisions or instrumentalities; public pension and retirement and other public funds; international organizations in which the United States holds membership; foreign central banks and foreign states; Federal Reserve Banks; and Government accounts. Tenders from others must be accompanied by full payment for the amount of securities applied for (in the form of cash, maturing Treasury securities, or readily collectible checks), or by a payment guarantee of 5 percent of the face amount applied for, from a commercial bank or a primary dealer.

3.5. Immediately after the closing hour, tenders will be opened, followed by a public announcement of the amount and yield range of accepted bids. Subject to the reservations expressed in Section 4, noncompetitive tenders will be accepted in full, and then competitive

tenders will be accepted, starting with those at the lowest yields, through successively higher yields to the extent required to attain the amount offered. Tenders at the highest accepted yield will be prorated if necessary. After the determination is made as to which tenders are accepted, an interest rate will be established, on the basis of a $\frac{1}{8}$ of one percent increment, which results in an equivalent average accepted price close to 100.000 and a lowest accepted price above the original issue discount limit of 99.500. That rate of interest will be paid on all of the securities. Based on such interest rate, the price on each competitive tender allotted will be determined and each successful competitive bidder will be required to pay the price equivalent to the yield bid. Those submitting noncompetitive tenders will pay the price equivalent to the weighted average yield of accepted competitive tenders. Price calculations will be carried to three decimal places on the basis of price per hundred, e.g., 99.923, and the determinations of the Secretary of the Treasury shall be final. If the amount of noncompetitive tenders received would absorb all or most of the offering, competitive tenders will be accepted in an amount sufficient to provide a fair determination of the yield. Tenders received from Government accounts and Federal Reserve Banks will be accepted at the price equivalent to the weighted average yield of accepted competitive tenders.

3.6. Competitive bidders will be advised of the acceptance or rejection of their tenders. Those submitting noncompetitive tenders will be notified only if the tender is not accepted in full, or when the price is over par.

4. Reservations

4.1. The Secretary of the Treasury expressly reserves the right to accept or reject any or all tenders in whole or in part, to allot more or less than the amount of securities specified in Section 1, and to make different percentage allotments to various classes of applicants when the Secretary considers it in the public interest. The Secretary's action under this Section is final.

5. Payment and Delivery

5.1. Settlement for allotted securities must be made at the Federal Reserve Bank or Branch or at the Bureau of the Public Debt, wherever the tender was submitted. Settlement on securities allotted to institutional investors and to others whose tenders are accompanied by a payment guarantee as provided in Section 3.4., must be made or completed on or before Monday, October 31, 1983.

Payment in full must accompany tenders submitted by all other investors.

Payment must be in cash; in other funds immediately available to the Treasury; in Treasury bills, notes, or bonds (with all coupons detached) maturing on or before the settlement date but which are not overdue as defined in the general regulations governing United States securities; or by check drawn to the order of the institution to which the tender was submitted, which must be received from institutional investors no later than Thursday, October 27, 1983. When payment has been submitted with the tender and the purchase price of allotted securities is over par, settlement for the premium must be completed timely, as specified in the preceding sentence. When payment has been submitted with the tender and the purchase price is under par, the discount will be remitted to be the bidder. Payment will not be considered complete where registered securities are requested if the appropriate identifying number as required on tax returns and other documents submitted to the Internal Revenue Service (an individual's social security number or an employer identification number) is not furnished. When payment is made in securities, a cash adjustment will be

made to or required of the bidder for any difference between the face amount of securities presented and the amount payable on the securities allotted.

5.2. In every case where full payment has not been completed on time, an amount of up to 5 percent of the face amount of securities allotted, shall, at the discretion of the Secretary of the Treasury, be forfeited to the United States.

5.3. Registered securities tendered in payment for allotted securities are not required to be assigned if the new securities are to be registered in the same names and forms as appear in the registrations or assignments of the securities surrendered. When the new securities are to be registered in names and forms different from those in the inscriptions or assignments of the securities presented, the assignment should be to "The Secretary of the Treasury for (securities offered by this circular) in the name of (name and taxpayer identifying number)." Specific instructions for the issuance and delivery of the new securities, signed by the owner or authorized representative, must accompany the securities presented. Securities tendered in payment should be surrendered to the Federal Reserve Bank or Branch or to

the Bureau of the Public Debt, Washington, D.C. 20226. The securities must be delivered at the expense and risk of the holder.

5.4. Delivery of securities in registered form will be made after the requested form of registration has been validated, the registered interest account has been established, and the securities have been inscribed.

6. General Provisions

6.1. As fiscal agents of the United States, Federal Reserve Banks are authorized and requested to receive tenders, to make allotments as directed by the Secretary of the Treasury, to issue such notices as may be necessary, and to receive payment for and make delivery of securities on full-paid allotments.

6.2. The Secretary of the Treasury may at any time issue supplemental or amendatory rules and regulations governing the offering. Public announcement of such changes will be promptly provided.

Carole J. Dineen,

Fiscal Assistant Secretary.

[FR Doc. 83-28437 Filed 10-14-83; 12:47 pm]

BILLING CODE 4810-40-M

Sunshine Act Meetings

Federal Register

Vol. 48, No. 202

Tuesday, October 18, 1983

This section of the FEDERAL REGISTER contains notices of meetings published under the "Government in the Sunshine Act" (Pub. L. 94-409) 5 U.S.C. 552b(e)(3).

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FEDERAL HOME LOAN BANK BOARD

FEDERAL REGISTER CITATION OF PREVIOUS ANNOUNCEMENT: Vol. No. 48, Page No. None at this time. Date Published—None at this time.

PLACE: Board Room, 6th Floor, 1700 G St., N.W., Washington, D.C.

STATUS: Open meeting.

CONTACT PERSON FOR MORE INFORMATION: Ms. Gravlee, (202-377-6970).

CHANGES IN THE MEETING: The Bank Board meeting previously scheduled for Friday, October 21, 1983, has been changed to Monday, October 24, 1983, at 10 a.m.

No. 60, October 14, 1983.
[S-1470-83 Filed 10-14-83; 11:21 am]
BILLING CODE 6720-01-M

2

FEDERAL RESERVE SYSTEM, (Board of Governors.)

TIME AND DATE: 10 a.m., Monday, October 24, 1983.

PLACE: 20th Street and Constitution Avenue, N.W., Washington, D.C. 20551.

STATUS: Closed.

MATTERS TO BE CONSIDERED:

1. Personnel actions (appointments, promotions, assignments, reassignments, and salary actions) involving individual Federal Reserve System employees.

2. Any items carried forward from a previously announced meeting.

CONTACT PERSON FOR MORE INFORMATION: Mr. Joseph R. Coyne, Assistant to the Board; (202) 452-3204.

Dated: October 14, 1983.

James McAfee,
Associate Secretary.

[S-1471-83 Filed 10-17-83; 3:01 pm]
BILLING CODE 6210-01-M

Federal Register

Tuesday
October 18, 1983

Part II

Environmental Protection Agency

Standards of Performance for New
Stationary Sources; Synthetic Organic
Chemical Manufacturing Industry;
Equipment Leaks of VOC, Reference
Methods 18 and 22; Final Rule

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[AD-FRL 2297-2]

Standards of Performance for New Stationary Sources; Synthetic Organic Chemical Manufacturing Industry; Equipment Leaks of VOC, Reference Methods 18 and 22

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This action promulgates standards of performance for equipment leaks of Volatile Organic Compounds (VOC) in the Synthetic Organic Chemical Manufacturing Industry (SOCMI). The standards were proposed in the *Federal Register* on January 5, 1981 (46 FR 1136). These standards implement Section 111 of the Clean Air Act and are based on the Administrator's determination that emissions from the synthetic organic chemical manufacturing industry cause, or contribute significantly to, air pollution which may reasonably be anticipated to endanger public health or welfare. The intended effect of these standards is to require all newly constructed, modified, and reconstructed SOCMI process units to use the best demonstrated system of continuous emission reduction for equipment leaks of VOC, considering costs, nonair quality health and environmental impact and energy requirements.

DATES: Effective date: October 18, 1983.

These standards of performance become effective upon promulgation but apply to affected facilities for which construction or modification commenced after January 5, 1981.

Under Section 307(b)(1) of the Clean Air Act, judicial review of these standards of performance is available only by the filing of a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this rule. Under Section 307(b)(2) of the Clean Air Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

The Director of the Federal Register approves the incorporation by reference of certain publications in 40 CFR effective on October 18, 1983.

ADDRESSES: *Background Information Documents.* The background information document (BID) for the

promulgated standards may be obtained from the U.S. EPA Library (MD-35), Research Triangle Park, North Carolina 27711, telephone number (919) 541-2777. Please refer to "Equipment Leaks of VOC in Synthetic Organic Chemicals Manufacturing Industry—Background Information for Promulgated Standards of Performance" (EPA-450/3-80-033b). The BID contains (1) a summary of all the public comments made on the proposed standards and EPA's responses to the comments, (2) a summary of the changes made to the standards since proposal, and (3) the final Environmental Impact Statement which summarizes the impacts of the promulgated standards. The BID for the proposed standards may be obtained from the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22161. Please refer to "VOC Fugitive Emissions in Synthetic Organic Chemicals Manufacturing Industry—Background Information for Proposed Standards," EPA-450/3-80-033b (NTIS PB81-152167). The additional information document (AID) describing emissions, emission reductions, and costs related to equipment leaks of VOC may also be obtained from the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22161. Please refer to "Fugitive Emission Sources of Organic Compounds—Additional Information on Emissions, Emission Reductions, and Costs," EPA-450/3-82-010 (NTIS PB82-217126).

Docket. A docket, number A-79-32, containing information considered by EPA in development of the promulgated standards, is available for public inspection between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section (A-130), West Tower Lobby, Gallery 1, 401 M Street, SW., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Mr. Fred Dimmick, Standards Development Branch, Emission Standards and Engineering Division (MD-13), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone (919) 541-5578.

SUPPLEMENTARY INFORMATION:

Summary of Standards

Standards of performance for new sources established under Section 111 of the Clean Air Act reflect:

... application of the best technological system of continuous emission reduction which (taking into consideration the cost of achieving such emission reduction, and any

nonair quality health and environmental impact and energy requirements) the Administrator determines has been adequately demonstrated [Section 111(a)(1)].

As prescribed by Section 111 of the Clean Air Act, promulgation of these standards was preceded by the Administrator's determination (40 CFR 60.16, 44 FR 49222, dated August 21, 1979) that these sources contribute significantly to air pollution which may reasonably be anticipated to endanger public health or welfare.

Standards of performance for equipment leaks of volatile organic compounds (VOC) in the synthetic organic chemical manufacturing industry (SOCMI) were proposed on January 5, 1981 (46 FR 1136).¹ The promulgated standards apply to specific equipment with the potential to leak VOC within process units operated to produce one or more of the organic chemicals listed in the standards. The equipment covered by the standards and grouped in a process unit are designated as the "affected facility" for determining applicability of the standards. A process unit is a group of equipment that can be used independently, if sufficient raw material and product storage capacity could be supplied, to produce one or more chemicals. Process units used to produce the chemicals covered by the standards may involve chemical synthesis, biological synthesis, other processing, or physical operations, such as separation. A plant may be composed of one or more process units.

Valves, pumps, compressors, pressure relief devices, sampling systems, and open-ended lines in VOC services (that is, containing 10 percent or more VOC by weight) are the equipment covered by the standards. The standards require (1) a leak detection and repair program for valves in gas/vapor and light liquid service and pumps in light liquid service; (2) equipment for compressors, sampling systems, and open-ended lines; (3) no detectable emissions (500 ppm as determined by Reference Method 21) for pressure relief devices in gas/vapor service during normal operation. In response to comments on the proposed standards, EPA is exempting from the

¹The proposed standards referred to fugitive emission sources of VOC as the air pollution emission points covered by the standards. The terminology fugitive emission sources can be confusing. The standards apply to equipment with the potential to leak VOC and, therefore, the promulgated standards refer to equipment leaks of VOC as the air pollution emission points covered by the standards. The Priority List (40 CFR 60.16) specifies fugitive emission sources within the SOCMI source category. Equipment leaks of VOC are included in fugitive emission sources for the purpose of the Priority List.

standards facilities producing less than 1,000 megagrams (Mg) of SO₂MI chemicals per year, facilities processing only heavy liquids or solids, and facilities producing beverage alcohol. In addition, the final standards provide a procedure for determining the equivalency of alternative control measures similar to the proposed procedure.

Standards for Valves. The standards for valves have not substantially changed since proposal. They are based on a leak detection and repair program that requires (1) monthly monitoring for valves in gas/vapor and light liquid service, (2) an initial attempt at repairing these valves within 5 days after detection of a leak, (3) repair of leaking valves within 15 days after detection of the leak unless repair would require a process unit shutdown, and (4) repair of valves during the next process unit shutdown after repair is delayed until a process unit shutdown. Valves found not to leak for 2 successive months can be monitored quarterly until leaks are detected. Monitoring of equipment to detect leaks is conducted in accordance with Reference Method 21 and a leak is defined as a measured organic concentration equal to or greater than 10,000 parts per million by volume (ppmv).

Two alternative standards have been provided for valves in gas/vapor and light liquid service in the final standards. These alternatives are (1) a limit of 2 percent of valves which may be leaking at any one time and (2) a skip-period leak detection and repair program for process units achieving less than 2 percent of their valves leaking. These alternative standards, although slightly different than the alternative standards included in the proposed standards, establish standards for owners and operators who design and operate low-peak process units. For low-leak process units, the costs of the monthly leak detection and repair program is unreasonably high in comparison to the emission reductions.

Standards for Pumps. The final standards include a leak detection and repair program that requires (1) monthly monitoring of pumps in light liquid service, (2) weekly visual inspections of the seals on pumps in light liquid service, (3) an attempt at repairing a pump within 5 days after detection of a leak, and (4) repair of a leaking pump within 15 days after detection of a seal failure or leak unless repair would require a process unit shutdown. Pumps that have repair delayed until a process unit shutdown must be repaired during the next process unit shutdown. If a

pump cannot be repaired without the use of dual seals and barrier fluid systems or vented seal areas, a delay of repair is allowed to install the equipment. In this case, an owner or operator must install the equipment as soon as practicable but may take no longer than 6 months.

The proposed standards required the use of equipment. The use of equipment reduces more emissions than the monthly leak detection and repair program. Even though the standards have changed, pumps in light liquid service alternately could use the equipment specified in the proposed standards and, thereby, not be required to implement the monthly leak detection and repair program.

Standards for Compressors. The standards have not changed since proposal and require compressors to be equipped with seals having a barrier fluid system that prevents leakage of the process fluid to the atmosphere. The system must: (1) Use a barrier fluid that is something other than a light liquid or gaseous VOC; (2) either operate at a pressure greater than the compressor seal area pressure, or be equipped with a barrier fluid degassing reservoir connected by a closed vent system to a control device; and (3) be equipped with a sensor so that seal failures may be detected. When seal failure is detected, repair is required within 15 days unless repair would require a process unit shutdown. An initial attempt at repair is required within 5 days. If a compressor is equipped with a closed vent system to transport leakage from the seal to a control device, it is exempt from the above requirements.

Certain reciprocating compressors covered by virtue of modification and reconstruction may be exempt from the requirements for compressors. Some existing compressors are not designed to use the equipment required by the standards. For these compressors either new compressor or a new distance-piece would be required before compliance with the standards could be achieved. The cost impact of installing the required control equipment on these existing compressors is unreasonable. These compressors will be exempt from the standards until they are replaced by new compressors or the distance pieces are replaced.

Standards for Sampling Connections. The standards require that VOC purged from sampling connections be recycled to the process by a closed sampling loop or that these VOC be collected in a closed collection system for recycle or disposal without VOC emission to atmosphere. In-situ sampling systems

are exempt from these requirements. These standards have not changed since proposal.

Standards for Open-ended Lines. The standards require that: (1) Open-ended lines be sealed with a second valve, cap, blind flange or plug except when the open-ended line is in use; and (2) if a second valve is used, the valve on the process side be closed first to avoid trapping VOC between the valves. These standards have not changed since proposal.

Standards for Pressure Relief Devices. The standards require that: (1) Pressure relief devices have "no detectable emissions" of VOC except in cases of overpressure relief; and (2) after each overpressure relief, pressure relief devices be returned to a state of no detectable emissions within 5 days. These standards have not changed since proposal. As noted in the preamble to the proposed standards, pressure relief devices are one of the few fugitive emission sources for which a performance standard can be established. There are a variety of alternative ways of complying with this standard. "No detectable emissions" of VOC, in this case, means 500 ppm or less above the background level as measured by Reference Method 21.

Standards for Control Devices. The standards include requirements for control devices used in conjunction with control of equipment leaks. The standards require: (1) That vapor recovery systems be designed and operated for at least 95 percent control; (2) that enclosed combustion devices be designed and operated to provide a minimum residence time of 0.75 seconds at a minimum temperature of 816°C or to achieve 95 percent reduction; and (3) that flares (a) be operated with no assist or with air or steam assist, (b) be designed and operated with no visible emissions except for periods of time not to exceed 5 minutes in any 2-hour period, (c) be operated with a flame present, and (d) meet other operational requirements including maximum exit gas velocities and minimum heat content values. Flares have been added to the approaches for achieving the standards since proposal. Otherwise, the standards for control devices have not changed since proposal.

Miscellaneous Provisions. Flanges, pressure relief devices in liquid service, equipment operating at subatmospheric pressures, and all equipment components in "heavy liquid" VOC service are excluded from the routine monitoring requirements of the proposed standards. Heavy liquids are defined as VOC liquids with vapor pressures less

than 0.3 kilopascals at 20°C. Even though the standards do not require monitoring these equipment for leaks, the standards require VOC leaks which are visually or otherwise detected from these equipment to be repaired within 15 days if a leak is confirmed using Reference Method 21. This provision improves current industry housekeeping practices for these pieces of equipment.

Under Section 111(h)(3), any person may request the Administrator to permit the use of an alternative means of emission limitation instead of a design, equipment, work practice or operational standard. The Administrator will permit the use of such alternative means if the Administrator determines, after notice and opportunity for a public hearing, that it will achieve emission reductions at least equivalent to those required by the design, equipment, work practice or operational standards. The permission will take the form of an amendment to the appropriate standards.

Compliance with the leak detection and repair program and equipment requirements will be assessed through semiannual reports, review of records, and by inspection. The semiannual reports provide a summary of the data recorded on leak detection and repair of valves, pumps, and of other equipment types. In response to public comments, requirements for routine reporting were reduced from quarterly to semiannual reporting. Notifications are still required as described in the General Provisions for new source standards (40 CFR 60.7).

The semiannual reports may be waived for affected facilities in States where the regulatory program has been delegated, if EPA, in the course of delegating such authority, approves reporting requirements or an alternative means of source surveillance adopted by the State. In these cases, such sources would be required to comply with the requirements adopted by the State.

Records of leak detection, repair attempts, and maintenance for equipment leaks of VOC are required by the standards. These records, along with semiannual reporting and plant inspections, will be used to enforce the standards. The records remain essentially the same as those in the proposed standards. Some changes have occurred because the requirements for the equipment covered by the standards have changed. In addition, EPA has made it clear that an owner or operator may keep the records for several affected facilities at one location in a plant rather than at each process unit.

Miscellaneous Actions. The General Provisions of Part 60 are being revised to provide technical amendments to the

provisions in 40 CFR 60.7, 60.11, and 60.17. Also, Reference Method 18 is being added to Appendix A of Part 60 and Reference Method 22 is being revised. These methods will be used to determine compliance with the requirements for flares.

Summary of Impacts

Emission Reductions. The standards of performance will reduce equipment leaks of VOC from newly constructed, modified, and reconstructed process units in SOCMI by approximately 56 percent in comparison to those emissions that would result in the absence of the standards. The standards will cover about 830 newly constructed, modified, and reconstructed sources in 1985. The standards will reduce the emissions associated with current industry practices from approximately 83,000 to 37,000 Mg in 1985 (that is, 91,000 to 41,000 tons) for newly constructed, reconstructed, and modified process units.

Cost and Economic Impacts. The cost and economic impacts of the standards are reasonable. The standards will require an industry-wide capital investment over the 5-year period from 1981 to 1986 of approximately \$44 million for those facilities which are newly constructed, reconstructed, or modified. The industry-wide net annualized cost will be about \$14.6 million in 1985. This net annualized cost includes a credit resulting from "recovered" emissions. The costs will be distributed among 830 facilities affected during the 5-year period. Industry-wide price increases are not expected to result from these standards.

Other Impacts. The standards of performance will not increase the energy usage of SOCMI process units. In general, the controls required by the standards do not require energy. Furthermore, the effect of the standards will be to increase efficiency of raw material usage so that a net positive energy impact will result. Implementation of the standards will have no impact on solid waste within SOCMI. In contrast, the standards could also cause a small positive impact on water quality by containment of potential liquid leaks. The recordkeeping and reporting requirements will require an average of 65 industry person years annually for the years of 1983 and 1984.

The environmental, energy, and economic impacts are discussed in greater detail in the background information document for the promulgated standards. [See the ADDRESSES section of this preamble.]

Public Participation

Prior to proposal of the standards, interested parties were advised by public notice in the Federal Register (45 FR 18474, March 21, 1980) of a meeting of the National Air Pollution Control Techniques Advisory Committee to discuss the standards for equipment leaks of VOC in SOCMI recommended for proposal. This meeting was held on April 18, 1980. The meeting was open to the public, and each attendee was given an opportunity to comment on the standards recommended for proposal. The standards were proposed in the Federal Register on January 5, 1981 (46 FR 1136). The preamble to the proposed standards discussed the availability of the background information document for the proposed standards which described in detail the regulatory alternatives considered and the impacts of those alternatives.

Public comments were solicited at the time of proposal and, when requested, copies of the BID were distributed to interested parties. To provide interested persons the opportunity for oral presentation of data, views, or arguments concerning the proposed standards, a public hearing was held on March 3, 1981, at Research Triangle Park, North Carolina. The hearing was open to the public and each attendee was given an opportunity to comment on the proposed standards. The public comment period was from January 5, 1981 to July 31, 1981. The comment period was extended to July 31, 1981, to allow interested parties to comment on several studies performed by EPA's Office of Research and Development which became available at proposal or after proposal. Fifty-six comment letters were received and 9 interested parties testified at the public hearing concerning issues relative to the proposed standards of performance for equipment leaks of VOC from SOCMI units.

EPA published an Additional Information Document (AID) in April of 1982. The AID contains a technical discussion of methodologies and estimates of emissions, emission reductions, and costs associated with control of equipment leaks of VOC. A notice of the availability of the AID and a request for comments on the AID was published in the Federal Register on May 7, 1982 (47 FR 19724). Fourteen letters were received containing comments on the AID.

Comments on the proposed standards and on the AID have been carefully considered and, where determined to be appropriate by the Administrator,

changes have been made in the proposed standards.

Significant Comments and Changes to the Proposed Standards

Comments on the proposed standards were received from industry, State and local air pollution control agencies, trade associations, and environmental groups. A detailed discussion of these comments and responses can be found in the BID for the promulgated standards. [See the ADDRESSES section of this preamble.] The comments and responses in the BID serve as the basis for the revisions which have been made to the standards between proposal and promulgation. Major changes made in the standards since proposal are indicated in the "Summary of Standards" section of the preamble. The major comments and responses are summarized in the next sections of this preamble. The comments and responses in this preamble have been combined into the following areas: Need for Standards, Selection of the Final Standards, Control Technology—Use of Flares, and Test Methods and Monitoring.

Need for Standards

Comment 1: Commenters questioned whether standards of performance are needed for SOCMI because SOCMI equipment leaks emit small quantities of VOC.

Response 1: SOCMI is a significant source of VOC emissions and equipment leaks of VOC are one of the primary contributors to the total VOC emissions from SOCMI. EPA estimates that 540,000 megagrams per year (Mg/yr) of VOC are emitted from all sources in SOCMI (docket item IV-B-24). This estimate of emissions is based on detailed studies of individual process source types including air oxidation processes, distillation operations, storage operations, carrier gas processes, equipment leaks, and secondary sources. Five hundred forty thousand Mg/yr is a significant quantity of VOC to be emitted as air pollution.

The significance of SOCMI VOC emissions is reflected in the Priority List, 40 CFR 60.16. Of the 59 major source categories on this list for which standards of performance were to be promulgated by 1982, the SOCMI source category ranked first. The Priority List consists of categories of air pollution sources that, in EPA's judgment, cause or contribute significantly to air pollution which may reasonably be anticipated to endanger public health or welfare. In developing the Priority List, major source categories were ranked according to three criteria specified in

Section 111(f) of the Act: (1) The quantity of emissions from each source category, (2) the extent to which each pollutant endangers public health or welfare, and (3) the mobility and competitive nature of each stationary source category. Commenters have not presented any new information which would change the decision to list SOCMI on the Priority List.

Based on comments and information received during this rulemaking, EPA has revised the estimates of emissions of VOC from equipment leaks within SOCMI since the standards were proposed. The current estimate of 540,000 Mg/yr includes the revised emission estimates. Even with the revised emission estimates, the Administrator still considers SOCMI a source category that causes or contributes significantly to air pollution which may reasonably be anticipated to endanger public health or welfare. It should be noted that because VOC emissions come from many, diverse source categories, each source category contributes a relatively small percentage to the large overall total emissions. Even though SOCMI may represent a small percentage of total VOC emissions, the magnitude of emissions from SOCMI is significant.

Since SOCMI is on the Priority List as a significant contributor to air pollution under section 111(f), standards of performance are required to be promulgated for those new sources within this source category for which the EPA can identify the best demonstrated technology (considering costs). EPA has identified several alternative systems of control capable of achieving additional emission reduction at reasonable cost at SOCMI sources. It is, therefore, reasonable for EPA to establish standards for these sources.

In addition, standards of performance have other benefits in addition to achieving emissions reductions. Standards of performance establish a degree of national uniformity to air pollution standards, and therefore, preclude situations in which some States may attract new industries as a result of having relaxed standards relative to other States. Further, standards of performance provide documentation that reduces uncertainty in evaluations of available control technology. This documentation includes identification and comprehensive analyses of alternative emission control technologies, development of associated costs, evaluation and verification of applicable emission test methods, and identification of specific emission limits achievable with alternate technologies.

This documentation also provides an economic analysis that reveals the affordability of controls in a study of the economic impact of controls on an industry.

Comment 2: Commenters stated that there is no need for the standards because emissions of VOC in SOCMI are adequately controlled by other regulations, specifically:

(a) National ambient air quality standards (NAAQS) and the State implementation plans (SIP) to implement these standards;

(b) National emission standards for hazardous air pollutants (NESHAP); and

(c) Occupational safety and health standards under the Occupational Safety and Health Act (OSHA).

Response 2: Standards of performance required by Section 111 play a unique role under the Clean Air Act. The main purpose of standards of performance is to require new sources, wherever located, to reduce emissions to the level achievable by the best technological system of continuous emission reduction considering the cost of achieving such emission reduction, any nonair quality health and environmental impact, and energy requirements [(Section 111(a)(1))]. Congress recognized that establishing such standards would minimize increases in air pollution from new sources, thereby improving air quality as the Nation's industrial base is replaced over the long term. The role of standards of performance in achieving the goals set forth in the Clean Air Act is distinct from that of other regulations.

(a) **NAAQS and SIP:** Reasonably available control technology (RACT) requirements apply to existing sources and are based on SIP's developed for the purpose of attaining the NAAQS. Standards of performance supplement the role played by RACT and the two types of control do not conflict. In the few areas where RACT-level control is already in place, the environmental impact of standards of performance will be smaller than calculated. EPA has determined, however, that existing RACT-level facilities that become subject to the standards of performance (e.g., through modification) will be achieving additional emission reductions at reasonable costs. In most areas of the Nation, RACT-level controls are not in place and the standards fulfill their role of preventing new air pollution from developing as the Nation's industrial base is replaced.

(b) **NESHAP:** NESHAP, as mandated under Section 112 of the Clean Air Act, are distinctly separate from NAAQS or new source standards of performance. NESHAP are developed to control

pollutants that are hazardous because they are carcinogens or the cause of other serious diseases. Some of the individual SOCMI chemicals have been identified as hazardous air pollutants and some SOCMI units may be affected by NESHAP regulations. However, SOCMI VOC emissions as a class have not been identified as hazardous pollutants, and therefore, are not subject to NESHAP.

(c) *OSHA*: Many of the chemicals covered by the standards are also listed in Table Z-1, Toxic and Hazardous Substances, in the general provisions for OSHA (29 CFR 1910.1000), and some of these chemicals are also covered by more specific health standards under OSHA. As a consequence, the SOCMI standards and the OSHA standards may affect the same equipment in VOC service. However, this possibility does not negate the need for the standards.

The SOCMI standards of performance serve to limit mass emission rates directly; OSHA standards for toxic chemicals generally do not. Under OSHA, control of emission sources may include substitution with less hazardous materials, process modification, worker rotation, process or worker isolation, ventilation controls, or modification of work practices. These controls reduce occupational exposures, but they do not necessarily reduce the mass rate of VOC emissions to the atmosphere. Furthermore, OSHA regulations would require control to different concentration levels, depending on the toxicity of a specific chemical, while the standards of performance require emission control for all VOC. Relying on indirect controls that may or may not reduce emissions that would degrade air quality would be an unreasonable approach to reducing emissions of VOC.

Comment 3: Commenters claimed that the standards are not needed to protect the public health and welfare. These commenters noted that the ambient air quality standard for ozone which is set to protect the public has recently been raised. The commenters continued by adding that, because most of the country is in compliance with the ozone standard, the public health and welfare is already protected and, therefore, the standards of performance are not needed.

Response 3: Standards of performance are not directly designed to achieve the ambient air quality goals. Their overriding purpose is rather to minimize emissions at all new and modified sources, wherever they are located, to prevent new pollution problems from developing, and to enhance air quality as the Nation's industrial base is replaced. Thus, the standards may not

bear directly on current attainment or nonattainment of NAAQS for ozone. Nonetheless, they do make room for future industrial growth while preventing future air quality problems—complementing the prevention of significant deterioration (PSD) and non-attainment rules as a means of achieving and maintaining the NAAQS. Clearly, residents in both attainment and non-attainment areas will ultimately benefit from such standards.

Basis for the Final Standards

Comment: Several commenters questioned EPA's selection of the proposed standards. The commenters felt that other regulatory options would have been more cost-effective and, therefore, should have been chosen. Referring to the regulatory alternatives presented in EPA-450/3-80-033a, some commenters recommended selection of regulatory alternatives II or III instead of regulatory alternative IV. Some commenters also suggested that EPA select the final standards after considering the cost effectiveness of each control technique for each piece of equipment covered by the standards. Other commenters recommended selection of regulatory alternative IV.

Response: Section 111 of the Clean Air Act, as amended, requires that standards of performance be based on the best system of continuous emission reduction that has been adequately demonstrated, considering costs, nonair quality health and environmental impacts and energy requirements. The control techniques for equipment leaks of VOC have been adequately demonstrated. The nonair quality health and environmental impacts associated with implementation of the standards are positive.

Since proposal, EPA has decided to accept the suggestions of the commenters and focus further on cost effectiveness as the basis for the selection of final standards. In using cost effectiveness of individual control

techniques as the basis for selection, the regulatory alternative framework presented in the BID for the proposed standards is no longer pertinent and is not presented. The control strategies represented by the regulatory alternatives were, however, considered in the selection of the final standards. In choosing among the control techniques for each type of equipment covered by the standards, EPA first considered their relative cost effectiveness. Then, for the control techniques which were selected as the most effective with reasonable costs, EPA considered the economic impact on the industry of these control techniques.

Cost-Effectiveness Considerations. EPA analyzed the annualized cost of controlling VOC emissions and the resultant VOC reduction for each alternative control technique. The control costs per megagram of VOC reduced for medium-sized process units are presented in Table 1 for each equipment type covered by the standards. These costs do not represent the actual amounts of money spent at any particular plant site. The cost of VOC emission reduction systems will vary according to the chemical product being produced, production equipment, plant layout, geographic location, and company preferences and policies. However, these costs and emission reductions are considered typical of control techniques for leaking equipment within SOCMI units and can be used in selecting the level of control to be required by the standards.

Pressure Relief Devices. The annualized costs and VOC emission reductions achieved for monthly and quarterly leak detection and repair programs and for the use of control equipment (rupture disks) were determined for pressure relief devices in gas service. As Table 1 shows, both the quarterly and monthly leak detection and repair programs are less expensive than installation of rupture disks.

TABLE 1.—CONTROL COSTS PER MEGAGRAM OF VOC REDUCED *

Equipment type and control technique ^a	Emission reduction (Mg/yr)	Average \$/Mg ^b	Incremental \$/Mg ^c
Pressure relief devices:			
Quarterly leak detection and repair	4.4	(*)	
Monthly leak detection and repair	5.1	(*)	500
Rupture disks ^d	10.0	510	1,200
Compressors: Controlled degassing vents^e	4.0	(*)	(*)
Open-ended lines: Caps on open-ended lines ^f	6.2	400	400
Sampling Systems: Closed purge sampling ^g	3.4	590	590
Valves:			
Semi-annual leak detection and repair	17.1	(*)	
Quarterly repair leak detection and repair	26.9	(*)	(*)
Monthly leak detection and repair ^h	33.6	62	480
Pumps:			
Quarterly leak detection and repair	4.1	1,200	
Monthly leak detection and repair ⁱ	7.6	610	(*)

TABLE 1.—CONTROL COSTS PER MEGAGRAM OF VOC REDUCED^a—Continued

Equipment type and control technique ^b	Emission reduction (Mg/yr)	Average \$/Mg ^c	Incremental \$/Mg ^d
Dual mechanical seal systems vented to a flare	12.6	2,300	5,600

^aCosts and emission reductions based on equipment counts in Model Unit B. See Section 3.2 of the BID for the promulgated standards.

^bFurther discussion of control techniques can be found in Section 4 of the BID for the promulgated standards.

^cAverage dollars per megagram (cost effectiveness) = (net annualized cost per component) ÷ (annual VOC emission reduction per component).

^dIncremental dollars per megagram = (net annualized cost of the control technique) - net annualized cost of the next less restrictive control technique = (annual emission reduction of control technique - annual emission reduction of the next less restrictive control technique).

^eValues are savings with the total savings indicated in the text.

^fControl technique selected as the basis for the standard.

Leak detection and repair programs result in average credits of \$240/Mg and \$150/Mg of VOC for quarterly and monthly programs, respectively. A monthly leak detection and repair program achieves an additional 0.7 Mg/yr emission reduction for medium-sized process units at an incremental cost of \$500/Mg compared to a quarterly leak detection and repair program. Rupture disks achieve an additional 4.9 Mg/yr emission reduction at an incremental cost of \$1,200/Mg compared to a monthly leak detection and repair program. However, EPA is establishing a performance standard (as indicated by no detectable emission limit) allowing a variety of alternative ways to complying with the standard. Because EPA used conservative assumptions in making this incremental cost calculation, the \$1,200/Mg incremental cost of achieving this 4.9 Mg/yr of emission reduction is more than what many process units would experience. Thus, a no detectable emission limit was selected as the basis for the pressure relief device standard.

Compressors. Only one control technique can be considered for compressor seals: the installation of control equipment such as barrier fluid systems. If a compressor is found leaking, the repair procedure would be the installation of control equipment. Because compressors are not generally spared, repair would be delayed until the next turnaround, thereby reducing the effectiveness of a leak detection and repair program to essentially zero. The installation of control equipment results in a savings (\$100/Mg of VOC), indicating that the value of product retained by controlling the barrier fluid system exceeds the cost of the control equipment. This cost is reasonable and, therefore, control equipment was selected as the basis for the standard for compressors.

Open-ended Lines and Sampling Systems. EPA considered caps or closures as the control technique for open-ended lines. Caps and closures are in wide-spread use in SOCMIs and are expected to be used even more

frequently in new SOCMIs units. The cost and emission reduction presented in Table 1 are the cost and emission reduction which would be realized for an open-ended line that is not controlled. The \$400/Mg cost for controlling emissions of VOC from open-ended lines is reasonable.

EPA considered closed purge sampling as the control technique for sampling systems. Closed purge systems are becoming increasingly common in the chemical industry. The \$590/Mg cost for controlling emissions of VOC from sampling systems is reasonable.

Valve. Several leak detection and repair programs were considered for valves. The programs differed in the monitoring frequency which would be implemented. As Table 1 shows, the quarterly monitoring program results in savings (\$41/Mg of VOC on the average). This occurs because the value of the recovered VOC is greater than the cost to implement the quarterly monitoring program. However, the largest emission reduction is associated with the monthly program at an average cost of \$62/Mg. Furthermore, the incremental cost per Mg VOC emissions reduced for the monthly program is \$510/Mg with an incremental emission reduction of 6.7 Mg/yr for a medium-sized process unit. EPA considers these costs to be reasonable. Therefore, EPA selected a monthly leak detection and repair program as the basis for the standard for valves.

Pumps. The control costs incurred for each megagram of VOC emissions reduced were determined for two leak detection and repair programs and for the use of dual mechanical seals with controlled degassing vents. Both leak detection and repair programs incur lower costs than the costs which would be incurred with equipment installation. The lowest average and incremental costs per Mg are associated with a monthly leak detection and repair program. The monthly program achieves a higher degree of control than the quarterly program, but it achieves a lower degree of control than installation

of control equipment. However, even though control equipment provides for the greatest amount of VOC reduction, the \$5,600/Mg incremental costs to obtain the additional 5 Mg/yr are judged to be unreasonably high. Because the costs for equipment are unreasonably high, monthly leak detection and repair was selected as the basis for the standard for pumps.

Economic Impact Considerations. An economic analysis was performed which evaluated the economic impacts of the selected standards. The results of that analysis are presented in detail in the BID for the promulgated standards. As summarized in the Summary of the Impacts of the Standards section of this preamble, the industry-wide net annualized cost will be about \$14.6 million in 1985. This cost is not expected to result in industry-wide price increases. These impacts are reasonable.

Control Technology—Use of Flares

Comment: Several commenters expressed the desire to use flares as alternatives to enclosed incinerators or vapor recovery systems. The comments focused on: (1) Data base support of low flare efficiency, (2) high efficiencies reported for flares on refinery gases, (3) alternative flare designs for low flow applications, (4) safety considerations in choosing control systems, and (5) equivalency to other combustion devices.

Response: The control technology for controlling equipment leaks of VOC from SOCMIs is discussed in the background information documents (BID's) for the proposed standards and for the promulgated standards. [See ADDRESSES section of this preamble.] In the BID for the proposed standards, EPA discussed the control technology that can be used in complying with the standards. As discussed in the BID for the promulgated standards, EPA reviewed the comments received on the control technology for controlling equipment leaks and found nothing which would alter the original judgments concerning these control techniques except with regard to the control efficiency associated with flares used in SOCMIs.

The use of flares was reconsidered by EPA. Flares are used widely in SOCMIs for handling emergency releases from process units and for combusting streams from continuous vents. Since the flare efficiency estimate at proposal was made, further actual flare measurement results have become available, most notably from the Chemical Manufacturers Association

(CMA)-EPA study. In the CMA-EPA study, flares were investigated over a wide range of exit velocity, composition, and flare gas heat content conditions. After review of available flare efficiency data, EPA has concluded that smokeless flares designed for and operated under certain conditions are acceptable alternatives to enclosed combustion devices (incinerators, boilers, process heaters) or vapor recovery systems, such as carbon adsorbers and condensation units. For example, steam-assisted flares must have flare gas heat contents greater than 11.2 megajoules per standard cubic meter and exit velocities less than 18 meters per second. Flares achieving these conditions are acceptable because they achieve more than 95 percent emission reduction. The presence of a flame can be ensured by monitoring the flare's pilot light with an appropriate heat sensor, such as a thermocouple. To ensure smokeless operation, visible emissions from a flare would be limited to less than 5 minutes in any 2-hour period as determined by Reference Method 22.

Impact on Small Manufacturers

Comment: Commenters requested an exemption for small manufacturers. These commenters were mostly concerned with the impact on process units with few equipment and process units with low production volumes.

Response: Data from test work on equipment leaks of VOC, however, do not show any definite relationship between emissions and production volume. Equipment leaks of VOC and the cost of their control are proportional to the number of equipment components in a plant rather than the plant size or production volume. However, there are some process units (e.g., research and development facilities) which have production rates so small that their VOC emissions from equipment leaks are likely to be very small and, as a consequence, the cost to control these emissions would be unreasonably high. EPA has excluded from the standards process units producing less than 1,000 Mg/yr. The lower production rate cutoff was developed on the basis of cost and emission reduction considerations and is explained in Section 5.7 of the BID for the promulgated standards.

Test Method and Monitoring

Comment: Commenters requested that hexane be the required calibration gas for the application of Reference Method 21 to SOCMi leak detection and repair programs. Reference Method 21 specifies the monitor and the methodology to be used to detect leaks

for required leak detection and repair programs. The proposed standards required that methane be used as the calibration gas. Two reasons were given for their desire to use hexane. First, commenters believed (on the basis of response factors) that the use of methane would result in the detection of more leaks. Second, there is at least one type of portable VOC detector which responds to hexane but does not respond to methane.

Response: EPA considered the differences between leak frequencies found using hexane and leak frequencies found using methane. The differences are not significant. The variability seen in repeat sampling of the same piece of equipment was 23 percent. The variability is in the same range as the 30 percent difference seen in response between the TLV-hexane system and the OVA-methane systems at the 10,000 ppmv definition of a leak. Because the variability in repeat sampling is similar to the differences in response at 10,000 ppmv, the data can be used interchangeably within 30 percent at the action level. Because an error of 30 percent at the action level is a small error considering all the errors associated with leak detection, there is no need to change the required calibration gas on the basis offered by the commenters. However, since some types of analyzers may be useful in certain SOCMi process units, and these types do not respond to methane, hexane has been added to the regulation as an alternate calibration material.

The use of hexane as an alternate calibration material is not limited to a single type of detector. The owner or operator may choose to use either calibrant with any allowable instrument. However, in any performance test conducted by EPA or a State agency, the VOC detector is likely to be calibrated with methane, even if the calibrant selected by the owner or operator is different, because methane calibrants are easier to obtain and deteriorate less than hexane calibrants.

Docket

The docket is an organized and complete file of all the information considered by EPA in the development of this rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking process. The docketing system is intended to allow members of the public and industries involved to identify and locate documents so that they can participate effectively in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated standards and EPA

responses to significant comments, the contents of the docket will serve as the record in case of judicial review, except for interagency review materials [Section 307(d)(7)(A)].

Miscellaneous

In accordance with Section 117 of the Act, publication of these promulgated standards was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies. This regulation will be reviewed 4 years from the date of promulgation as required by the Clean Air Act. This review will include an assessment of such factors as the need for integration with other programs, the existence of alternative methods, enforceability, improvements in emission control technology, and reporting requirements.

Section 317 of the Clean Air Act requires the Administrator to prepare an economic impact assessment for any new source standard of performance promulgated under Section 111(b) of the Act. An economic impact assessment was prepared for this regulation and for other regulatory alternatives. All aspects of the assessment were considered in the formulation of the standards to insure that cost was carefully considered in determining the best demonstrated technology. The economic impact assessment is included in the background information documents for the proposed standards and the promulgated standards.

Information collection requirements associated with this regulation (those included in 40 CFR Part 60, Subpart A and Subpart VV) have been approved by the Office of Management and Budget (OMB) under the provisions of the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.* and have been assigned OMB control number 2060-0012.

"Major Rule" Determination. Under Executive Order 12291, the Administrator is required to judge whether a regulation is a "major rule" and, therefore, subject to certain requirements of the Order. The Administrator has determined that this regulation would result in none of the adverse economic effects set forth in Section 1 of the Order as grounds for finding a regulation to be a "major rule." Fifth-year annualized costs of the standards would be as much as \$3.3 million for the 830 newly constructed, modified, and reconstructed production facilities projected that could be affected by the standards during the first 5 years. Significant price increases are not expected to result from

implementation of these standards because the annualized cost is a small fraction (about 0.25 percent) of the yearly revenue expected for the new, modified, and reconstructed units affected during the 5-year period. The Administrator has concluded that this rule is not "major" under any of the criteria established in the Executive Order.

As discussed in the "Basis For The Final Standards" section of this preamble, costs per megagram of VOC emission reduction were used in selecting the standards promulgated by this rulemaking. This regulation was submitted to the OMB for review as required by Executive Order 12291. Any comments from OMB to EPA and any EPA responses to those comments are available for public inspection in Docket No. A-79-32, Central Docket Section, at the address given in the ADDRESSES section of this preamble.

Regulatory Flexibility Analysis
Certification. The Regulatory Flexibility Act of 1980 requires that adverse effects of all Federal regulations upon small businesses be identified. According to current Small Business Administration guidelines, a small business in the organic chemical manufacturing industry is one that has 1,000 employees or fewer. Current estimates indicate between 10 and 30 percent of the existing industry employ fewer than 1,000 people. Even if facilities owned by small businesses do become subject to the standards, none will be adversely affected. This conclusion is based on the finding that the annualized cost of the standards would be less than 0.25 percent of the yearly revenue expected for units affected by the standards. This finding reflects the economic impact for facilities owned by small businesses and is not considered significant. Pursuant to the provisions of 5 U.S.C. 605(b), I hereby certify that this rule will not have a significant economic impact on a substantial number of small entities.

List of Subjects in 40 CFR Part 60

Air pollution control, Aluminum, Ammonium sulfate plants, Asphalt, Cement industry, Coal Copper, Electric power plants, Glass and glass products, Grains, Intergovernmental relations, Iron, Lead, Metals, Metallic Minerals, Motor vehicles, Nitric acid plants, Paper and paper products industry, Petroleum, Phosphate, Sewage disposal, Steel Sulfuric acid plants, Waste treatment and disposal, Zinc, Tires, Incorporation by Reference, Can surface coating, Sulfuric acid plants, Industrial organic chemicals.

Dated: September 30, 1983.
 William D. Ruckelshaus,
 Administrator.

PART 60—[AMENDED]

40 CFR Part 60 is amended as follows:

1. By adding paragraph (f) to § 60.7 of Subpart A—General Provisions as follows:

§ 60.7 Notification and recordkeeping.

(f) Individual subparts of this part may include specific provisions which clarify or make inapplicable the provisions set forth in this section.

(Sec. 111, 114, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7411, 7601(a)))

2. By adding paragraph (f) to § 60.11 of Subpart A—General Provisions as follows:

§ 60.11 Compliance with standards and maintenance requirements.

(f) Special provisions set forth under an applicable subpart of this part shall supersede any conflicting provisions of this section.

(Sec. 111, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7411, 7601(a)))

3. By adding paragraphs (a)(34), (a)(35), (a)(36), (a)(37), (a)(38), and (a)(39) to § 60.17 of Subpart A—General Provisions as follows:

§ 60.17 Incorporations by reference.

(a) * * *

(34) ASTM, E169-63 (Reapproved 1977), General Techniques of Ultraviolet Quantitative Analysis, IBR approved for § 60.485(d).

(35) ASTM E168-67 (Reapproved 1977), General Techniques of Infrared Quantitative Analysis, IBR approved for § 60.485(d).

(36) ASTM E260-73, General Gas Chromatography Procedures, IBR approved for § 60.485(d).

(37) ASTM D2879-70, Vapor Pressure—Temperature Relationship and Initial Decomposition Temperature of Liquids by Isotenoscope, IBR approved for § 60.485(e).

(38) ASTM D2382-76, Heat of Combustion of Hydrocarbon Fuels by Bomb Calorimeter (High-Precision Method), IBR approved for § 60.485(g).

(39) ASTM D2504-67 (Reapproved 1977), Noncondensable Gases in C₆ and Lighter Hydrocarbon Products by Gas Chromatography, IBR approved for § 60.485(g).

(Sec. 111, 114, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7411, 7601(a)))

4. By adding a new Subpart VV as follows:

Subpart VV—Standards of Performance for Equipment Leaks of VOC in the Synthetic Organic Chemicals Manufacturing Industry

Sec.

60.480 Applicability and designation of affected facility.

60.481 Definitions.

60.482-1 Standards: General.

60.482-2 Standards: Pumps in light liquid service.

60.482-3 Standards: Compressors.

60.482-4 Standards: Pressure relief devices in gas/vapor service.

60.482-5 Standards: Sampling connection systems.

60.482-6 Standards: Open-ended valves or lines.

60.482-7 Standards: Valves in gas/vapor and in light liquid service.

60.482-8 Standards: Pumps and valves in heavy liquid service, pressure relief devices in light liquid or heavy liquid service, and flanges and other connectors.

60.482-9 Standards: Delay of repair.

60.482-10 Standards: Closed vent systems and control devices.

60.483-1 Alternative standards for valves—allowable percentage of valves leaking.

60.483-2 Alternative standards for valves—skip period leak detection and repair.

60.484 Equivalence of means of emission limitation.

60.485 Test methods and procedures.

60.486 Recordkeeping requirements.

60.487 Reporting requirements.

60.488 [Reserved]

60.489 List of chemicals produced by affected facilities.

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

Subpart VV—Standards of Performance for Equipment Leaks of VOC in the Synthetic Organic Chemicals Manufacturing Industry

§ 60.480 Applicability and designation of affected facility.

(a)(1) The provisions of this subpart apply to affected facilities in the synthetic organic chemicals manufacturing industry.

(2) The group of all equipment (defined in § 60.481) within a process unit is an affected facility.

(b) Any affected facility under paragraph (a) of this section that commences construction or modification after January 5, 1981, shall be subject to the requirements of this subpart.

(c) Addition or replacement of equipment for the purpose of process improvement which is accomplished without a capital expenditure shall not by itself be considered a modification under this subpart.

(d)(1) If an owner or operator applies for one of one of the exemptions in this paragraph, then the owner or operator shall maintain records as required in § 60.486(h).

(2) Any affected facility that has the design capacity to produce less than 1,000 Mg/yr is exempt from § 60.482.

(3) If an affected facility produces heavy liquid chemicals only from heavy liquid feed or raw materials, then it is exempt from § 60.482.

(4) Any affected facility that produces beverage alcohol is exempt from § 60.482.

(5) Any affected facility that has no equipment in VOC service is exempt from § 60.482.

§ 60.481 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act or in Subpart A of Part 60, and the following terms shall have the specific meanings given them.

"Closed vent system" means a system that is not open to the atmosphere and that is composed of piping, connections, and, if necessary, flow inducing devices that transport gas or vapor from a piece or pieces of equipment to a control device.

"Connector" means flanged, screwed, welded, or other joined fittings used to connect two pipe lines or a pipe line and a piece of process equipment.

"Control device" means an enclosed combustion device, vapor recovery system, or flare.

"Distance piece" means an open or enclosed casing through which the piston rod travels, separating the compressor cylinder from the crankcase.

"Equipment" means each pump, compressor, pressure relief device, sampling connection system, open-ended valve or line, valve, and flange or other connector in VOC service and any devices or systems required by this subpart.

"First attempt at repair" means to take rapid action for the purpose of stopping or reducing leakage of organic material to atmosphere using best practices.

"In gas/vapor service" means that the piece of equipment contains process fluid that is in the gaseous state at operating conditions.

"In heavy liquid service" means that the piece of equipment is not in gas/vapor service or in light liquid service.

"In light liquid service" means that the piece of equipment contains a liquid that meets the conditions specified in § 60.485(e).

"Liquids dripping" means any visible leakage from the seal including

spraying, misting, clouding, and ice formation.

"Open-ended valve or line" means any valve, except safety relief valves, having one side of the valve seat in contact with process fluid and one side open to the atmosphere, either directly or through open piping.

"Pressure release" means the emission of materials resulting from system pressure being greater than set pressure of the pressure relief device.

"Process improvement" means routine changes made for safety and occupational health requirements, for energy savings, for better utility, for ease of maintenance and operation, for correction of design deficiencies, for bottleneck removal, for changing product requirements, or for environmental control.

"Process unit" means components assembled to produce, as intermediate or final products, one or more of the chemicals listed in § 60.489 of this part. A process unit can operate independently if supplied with sufficient feed or raw materials and sufficient storage facilities for the product.

"Process unit shutdown" means a work practice or operational procedure that stops production from a process unit or part of a process unit. An unscheduled work practice or operational procedure that stops production from a process unit or part of a process unit for less than 24 hours is not a process unit shutdown. The use of spare equipment and technically feasible bypassing of equipment without stopping production are not process unit shutdowns.

"Quarter" means a 3-month period; the first quarter concludes on the last day of the last full month during the 180 days following initial startup.

"Repaired" means that equipment is adjusted, or otherwise altered, in order to eliminate a leak as indicated by one of the following: an instrument reading or 10,000 ppm or greater, indication of liquids dripping, or indication by a sensor that a seal or barrier fluid system has failed.

"Sensor" means a device that measures a physical quantity or the change in a physical quantity such as temperature, pressure, flow rate, pH, or liquid level.

"In-situ sampling systems" means nonextractive samplers or in-line samplers.

"Synthetic organic chemicals manufacturing industry" means the industry that produces, as intermediates or final products, one or more of the chemicals listed in § 60.489.

"In vacuum service" means that equipment is operating at an internal

pressure which is at least 5 kilopascals (kPa) below ambient pressure.

"Volatile organic compounds" or VOC means, for the purposes of this subpart, any reactive organic compounds as defined in § 60.2 Definitions.

"In VOC Service" means that the piece of equipment contains or contacts a process fluid that is at least 10 percent VOC by weight. (The provisions of § 60.485(d) specify how to determine that a piece of equipment is not in VOC service.)

§ 60.482-1 Standards: General.

(a) Each owner or operator subject to the provisions of this subpart shall demonstrate compliance with the requirements of § 60.482-1 to § 60.482-10 for all equipment within 180 days of initial startup.

(b) Compliance with § 60.482-1 to § 60.482-10 will be determined by review of records and reports, review of performance test results, and inspection using the methods and procedures specified in § 60.485.

(c)(1) An owner or operator may request a determination of equivalence of a means of emission limitation to the requirements of § 60.482-2, -3, -5, -6, -7, -8, and -10 as provided in § 60.484.

(2) If the Administrator makes a determination that a means of emission limitation is at least equivalent to the requirements of § 60.482-2, -3, -5, -6, -7, -8, or -10, an owner or operator shall comply with the requirements of that determination.

(d) Equipment that is in vacuum service is excluded from the requirements of § 60.482-2 to § 60.482-10 if it is identified as required in § 60.486(e)(4).

§ 60.482-2 Standards: Pumps in light liquid service.

(a)(1) Each pump in light liquid service shall be monitored monthly to detect leaks by the methods specified in § 60.485(b), except as provided in § 60.482-1(c) and paragraphs (d), (e), and (f) of this section.

(2) Each pump in light liquid service shall be checked by visual inspection each calendar week for indications of liquids dripping from the pump seal.

(b)(1) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(2) If there are indications of liquids dripping from the pump seal, a leak is detected.

(c)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 60.482-9.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(d) Each pump equipped with a dual mechanical seal system that includes a barrier fluid system is exempt from the requirements of paragraph (a), provided the following requirements are met:

(1) Each dual mechanical seal system is:

(i) Operated with the barrier fluid at a pressure that is at all times greater than the pump stuffing box pressure; or

(ii) Equipment with a barrier fluid degassing reservoir that is connected by a closed vent system to a control device that complies with the requirements of § 60.482-10; or

(iii) Equipped with a system that purges the barrier fluid into a process stream with zero VOC emissions to the atmosphere.

(2) The barrier fluid system is in heavy liquid service or is not in VOC service.

(3) Each barrier fluid system is equipped with a sensor that will detect failure of the seal system, the barrier fluid system, or both.

(4) Each pump is checked by visual inspection, each calendar week, for indications of liquids dripping from the pump seals.

(5)(i) Each sensor as described in paragraph (d)(3) is checked daily or is equipped with an audible alarm, and

(ii) The owner or operator determines, based on design considerations and operating experience, a criterion that indicates failure of the seal system, the barrier fluid system, or both.

(6)(i) If there are indications of liquids dripping from the pump seal or the sensor indicates failure of the seal system, the barrier fluid system, or both based on the criterion determined in paragraph (d)(5)(ii), a leak is detected.

(ii) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 60.482-9.

(iii) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(e) Any pump that is designated, as described in § 60.486(e) (1) and (2), for no detectable emission, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraphs (a), (c), and (d) if the pump:

(1) Has no externally actuated shaft penetrating the pump housing.

(2) Is demonstrated to be operating with no detectable emissions as indicated by an instrument reading of less than 500 ppm above background as

measured by the methods specified in § 60.485(c), and

(3) Is tested for compliance with paragraph (e)(2) initially upon designation, annually, and at other times requested by the Administrator.

(f) If any pump is equipped with a closed vent system capable of capturing and transporting any leakage from the seal or seals to a control device that complies with the requirements of § 60.482-10, it is exempt from the paragraphs (a)-(e).

§ 60.482-3 Compressors.

(a) Each compressor shall be equipped with a seal system that includes a barrier fluid system and that prevents leakage of VOC to the atmosphere, except as provided in § 60.482-1(c) and paragraph (h) and (i) of this section.

(b) Each compressor seal system as required in paragraph (a) shall be:

(1) Operated with the barrier fluid at a pressure that is greater than the compressor stuffing box pressure; or

(2) Equipped with a barrier fluid system that is connected by a closed vent system to a control device that complies with the requirements of § 60.482-10; or

(3) Equipped with a system that purges the barrier fluid into a process stream with zero VOC emissions to the atmosphere.

(c) The barrier fluid system shall be in heavy liquid service or shall not be in VOC service.

(d) Each barrier fluid system as described in paragraph (a) shall be equipped with a sensor that will detect failure of the seal system, barrier fluid system, or both.

(e)(1) Each sensor as required in paragraph (d) shall be checked daily or shall be equipped with an audible alarm.

(2) The owner or operator shall determine, based on design considerations and operating experience, a criterion that indicates failure of the seal system, the barrier fluid system, or both.

(f) If the sensor indicates failure of the seal system, the barrier system, or both based on the criterion determined under paragraph (e)(2), a leak is detected.

(g)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 60.482-9.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(h) A compressor is exempt from the requirements of paragraphs (a) and (b), if it is equipped with a closed vent system capable of capturing and transporting any leakage from the seal

to a control device that complies with the requirements of § 60.482-10, except as provided in § 60.482-3(i).

(i) Any compressor that is designated, as described in § 60.486(e) (1) and (2), for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraphs (a)-(h) if the compressor:

(1) Is demonstrated to be operating with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the methods specified in § 60.485(c); and

(2) Is tested for compliance with paragraph (i)(1) initially upon designation, annually, and at other times requested by the Administrator.

(j) Any existing reciprocating compressor in a process unit which becomes an affected facility under provisions of § 60.14 or 60.15 is exempt from § 60.482 (a), (b), (c), (d), (e), and (h), provided the owner or operator demonstrates that recasting the distance piece or replacing the compressor are the only options available to bring the compressor into compliance with the provisions of § 60.4823 (a), (b), (c), (d), (e), and (h).

§ 60.482-4 Standards: Pressure relief devices in gas/vapor service.

(a) Except during pressure releases, each pressure relief device in gas/vapor service shall be operated with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as determined by the methods specified in § 60.485(c).

(b)(1) After each pressure release, the pressure relief device shall be returned to a condition of no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as soon as practicable, but not later than 5 calendar days after the pressure release, except as provided in § 60.482-9.

(2) No later than 5 calendar days after the pressure release, the pressure relief device shall be monitored to confirm the conditions of no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, by the methods specified in § 60.485(c).

(c) Any pressure relief device that is equipped with a closed vent system capable of capturing and transporting leakage through the pressure relief device to a control device as described in § 60.482-10 is exempted from the requirements of paragraphs (a) and (b).

§ 60.482-5 Standards: Sampling connection systems.

(a) Each sampling connection system shall be equipped with a closed purge system or closed vent system, except as provided in § 60.482-1(c).

(b) Each closed purge system or closed vent system as required in paragraph (a) shall:

(1) Return the purged process fluid directly to the process line with zero VOC emissions to the atmosphere; or

(2) Collect and recycle the purged process fluid with zero VOC emissions to the atmosphere; or

(3) Be designed and operated to capture and transport all the purged process fluid to a control device that complies with the requirements of § 60.482-10.

(c) In-situ sampling systems are exempt from paragraphs (a) and (b).

§ 60.482-6 Standards: Open-ended valves or lines.

(a)(1) Each open-ended valve or line shall be equipped with a cap, blind flange, plug, or a second valve, except as provided in § 60.482-1(c).

(2) The cap, blind flange, plug, or second valve shall seal the open end at all times except during operations requiring process fluid flow through the open-ended valve or line.

(b) Each open-ended valve or line equipped with a second valve shall be operated in a manner such that the valve on the process fluid end is closed before the second valve is closed.

§ 60.482-7 Standards: Valves in gas/vapor service in light liquid service.

(a) Each valve shall be monitored monthly to detect leaks by the methods specified in § 60.485(b) and shall comply with paragraphs (b)-(e), except as provided in paragraphs (f), (g), and (h), § 60.483-1, 2, and § 60.482-1(c).

(b) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(c)(1) Any valve for which a leak is not detected for 2 successive months may be monitored the first month of every quarter, beginning with the next quarter, until a leak is detected.

(2) If a leak is detected, the valve shall be monitored monthly until a leak is not detected for 2 successive months.

(d)(1) When a leak is detected, it shall be repaired as soon as practicable, but no later than 15 calendar days after the leak is detected, except as provided in § 60.482-9.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(e) First attempts at repair include, but are not limited to, the following best practices where practicable:

(1) Tightening of bonnet bolts;

(2) Replacement of bonnet bolts;

(3) Tightening of packing gland nuts;

(4) Injection of lubricant into lubricated packing.

(f) Any valve that is designated, as described in § 60.486(e)(2), for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraph (a) if the valve:

(1) Has no external actuating mechanism in contact with the process fluid,

(2) Is operated with emissions less than 500 ppm above background as determined by the method specified in § 60.485(c), and

(3) Is tested for compliance with paragraph (f)(2) initially upon designation, annually, and at other times requested by the Administrator.

(g) Any valve that is designated, as described in § 60.486(f)(1), as an unsafe-to-monitor valve is exempt from the requirements of paragraph (a) if:

(1) The owner or operator of the valve demonstrates that the valve is unsafe to monitor because monitoring personnel would be exposed to an immediate danger as a consequence of complying with paragraph (a), and

(2) The owner or operator of the valve adheres to a written plan that requires monitoring of the valve as frequently as practicable during safe-to-monitor times.

(h) Any valve that is designated, as described in § 60.486(f)(2), as a difficult-to-monitor valve is exempt from the requirements of paragraph (a) if:

(1) The owner or operator of the valve demonstrates that the valve cannot be monitored without elevating the monitoring personnel more than 2 meters above a support surface.

(2) The process unit within which the valve is located becomes an affected facility through § 60.14 or § 60.15, and

(3) The owner or operator of the valve follows a written plan that requires monitoring of the valve at least once per calendar year.

§ 60.482-8 Standards: Pumps and valves in heavy liquid service, pressure relief devices in light liquid or heavy liquid service, and flanges and other connectors.

(a) Pumps and valves in heavy liquid service, pressure relief devices in light liquid or heavy liquid service, and flanges and other connectors shall be monitored within 5 days by the method specified in § 60.485(b) if evidence of a potential leak is found by visual,

audible, olfactory, or any other detection method.

(b) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(c)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 60.482-9.

(2) The first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(d) First attempts at repair include, but are not limited to, the best practices described under § 60.482-7(e).

§ 60.482-9 Standards: Delay of repair.

(a) Delay of repair of equipment for which leaks have been detected will be allowed if the repair is technically infeasible without a process unit shutdown. Repair of this equipment shall occur before the end of the next process unit shutdown.

(b) Delay of repair of equipment will be allowed for equipment which is isolated from the process and which does not remain in VOC service.

(c) Delay of repair for valves will be allowed if:

(1) The owner or operator demonstrates that emissions of purged material resulting from immediate repair are greater than the fugitive emissions likely to result from delay of repair, and

(2) When repair procedures are effected, the purged material is collected and destroyed or recovered in a control device complying with § 60.482-10.

(d) Delay of repair for pumps will be allowed if:

(1) Repair requires the use of a dual mechanical seal system that includes a barrier fluid system, and

(2) Repair is completed as soon as practicable, but not later than 6 months after the leak was detected.

(e) Delay of repair beyond a process unit shutdown will be allowed for a valve, if valve assembly replacement is necessary during the process unit shutdown, valve assembly supplies have been depleted, and valve assembly supplies had been sufficiently stocked before the supplies were depleted. Delay of repair beyond the next process unit shutdown will not be allowed unless the next process unit shutdown occurs sooner than 6 months after the first process unit shutdown.

§ 60.482-10 Standards: Closed vent systems and control devices.

(a) Owners or operators of closed vent systems and control devices used to comply with provisions of this subpart

shall comply with the provisions of this section.

(b) Vapor recovery systems (for example, condensers and adsorbers) shall be designed and operated to recover the VOC emissions vented to them with an efficiency of 95 percent or greater.

(c) Enclosed combustion devices shall be designed and operated to reduce the VOC emissions vented to them with an efficiency of 95 percent or greater, or to provide a minimum residence time of 0.75 seconds at a minimum temperature of 816°C.

(d)(1) Flares shall be designed for and operated with no visible emissions as determined by the methods specified in § 60.485(g), except for periods not to exceed a total of 5 minutes during any 2 consecutive hours.

(2) Flares shall be operated with a flame present at all times, as determined by the methods specified in § 60.485(g).

(3) Flares shall be used only with the net heating value of the gas being combusted being 11.2 MJ/scm (300 Btu/scf) or greater if the flare is steam-assisted or air-assisted; or with the net heating value of the gas being combusted being 7.45 MJ/scm or greater if the flare is nonassisted. The net heating value of the gas being combusted shall be determined by the methods specified in § 60.485(g).

(4) Steam-assisted and nonassisted flares shall be designed for and operated with an exit velocity, as determined by the methods specified in § 60.485(g)(4), less than 18 m/sec (60 ft/sec).

(5) Flares used to comply with this subpart shall be steam-assisted, air-assisted, or nonassisted.

(6) Air-assisted flares shall be designed and operated with an exit velocity less than the velocity, V_{max} , as determined by the methods specified in § 60.485(g)(5).

(e) Owners or operators of control devices used to comply with the provisions of this subpart shall monitor these control devices to ensure that they are operated and maintained in conformance with their designs.

(f)(1) Closed vent systems shall be designed and operated with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background and visual inspections, as determined by the methods specified in § 60.485(c).

(2) Closed vent systems shall be monitored to determine compliance with this section initially in accordance with § 60.8, annually and at other times requested by the Administrator.

(g) Closed vent systems and control devices used to comply with provisions

of this subpart shall be operated at all times when emissions may be vented to them.

§ 60.483-1 Alternative standards for valves—allowable percentage of valves leaking.

(a) An owner or operator may elect to comply with an allowable percentage of valves leaking of equal to or less than 2.0 percent.

(b) The following requirements shall be met if an owner or operator wishes to comply with an allowable percentage of valves leaking:

(1) An owner or operator must notify the Administrator that the owner or operator has elected to comply with the allowable percentage of valves leaking before implementing this alternative standard, as specified in § 60.487(b).

(2) A performance test as specified in paragraph (c) of this section shall be conducted initially upon designation, annually, and at other times requested by the Administrator.

(3) If a valve leak is detected, it shall be repaired in accordance with § 60.482-7(d) and (e).

(c) Performance tests shall be conducted in the following manner:

(1) All valves in gas/vapor and light liquid service within the affected facility shall be monitored within 1 week by the methods specified in § 60.485(b).

(2) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(3) The leak percentage shall be determined by dividing the number of valves for which leaks are detected by the number of valves in gas/vapor and light liquid service within the affected facility.

(d) Owners and operators who elect to comply with this alternative standard shall not have an affected facility with a leak percentage greater than 2.0 percent.

§ 60.483-2 Alternative standards for valves—skip period leak detection and repair.

(a)(1) An owner or operator may elect to comply with one of the alternative work practices specified in paragraphs (b) (2) and (3) of this section.

(2) An owner or operator must notify the Administrator before implementing one of the alternative work practices, as specified in § 60.487(b).

(b)(1) An owner or operator shall comply initially with the requirements for valves in gas/vapor service and valves in light liquid service, as described in § 60.482-7.

(2) After 2 consecutive quarterly leak detection periods with the percent of valves leaking equal to or less than 2.0, an owner or operator may begin to skip

1 of the quarterly leak detection periods for the valves in gas/vapor and light liquid service.

(3) After 5 consecutive quarterly leak detection periods with the percent of valves leaking equal to or less than 2.0, an owner or operator may begin to skip 3 of the quarterly leak detection periods for the valves in gas/vapor and light liquid service.

(4) If the percent of valves leaking is greater than 2.0, the owner or operator shall comply with the requirements as described in § 60.482-7 but can again elect to use this section.

(5) The percent of valves leaking shall be determined by dividing the sum of valves found leaking during current monitoring and valves for which repair has been delayed by the total number of valves subject to the requirements of § 60.483-2.

(6) An owner or operator must keep a record of the percent of valves found leaking during each leak detection period.

§ 60.484 Equivalence of means of emission limitation.

(a) Each owner or operator subject to the provisions of this subpart may apply to the Administrator for determination of equivalence for any means of emission limitation that achieves a reduction in emissions of VOC at least equivalent to the reduction in emissions of VOC achieved by the controls required in this subpart.

(b) Determination of equivalence to the equipment, design, and operational requirements of this subpart will be evaluated by the following guidelines:

(1) Each owner or operator applying for an equivalence determination shall be responsible for collecting and verifying test data to demonstrate equivalence of means of emission limitation.

(2) The Administrator will compare test data for the means of emission limitation to test data for the equipment, design, and operational requirements.

(3) The Administrator may condition the approval of equivalence on requirements that may be necessary to assure operation and maintenance to achieve the same emission reduction as the equipment, design, and operational requirements.

(c) Determination of equivalence to the required work practices in this subpart will be evaluated by the following guidelines:

(1) Each owner or operator applying for a determination of equivalence shall be responsible for collecting and verifying test data to demonstrate

equivalence of an equivalent means of emission limitation.

(2) For each affected facility for which a determination of equivalence is requested, the emission reduction achieved by the required work practice shall be demonstrated.

(3) For each affected facility, for which a determination of equivalence is requested, the emission reduction achieved by the equivalent means of emission limitation shall be demonstrated.

(4) Each owner or operator applying for a determination of equivalence shall commit in writing to work practice(s) that provide for emission reductions equal to or greater than the emission reductions achieved by the required work practice.

(5) The Administrator will compare the demonstrated emission reduction for the equivalent means of emission limitation to the demonstrated emission reduction for the required work practices and will consider the commitment in paragraph (c)(4).

(6) The Administrator may condition the approval of equivalence on requirements that may be necessary to assure operation and maintenance to achieve the same emission reduction as the required work practice.

(d) An owner or operator may offer a unique approach to demonstrate the equivalence of any equivalent means of emission limitation.

(e)(1) After a request for determination of equivalence is received, the Administrator will publish a notice in the *Federal Register* and provide the opportunity for public hearing if the Administrator judges that the request may be approved.

(2) After notice and opportunity for public hearing, the Administrator will determine the equivalence of a means of emission limitation and will publish the determination in the *Federal Register*.

(3) Any equivalent means of emission limitations approved under this section shall constitute a required work practice, equipment, design, or operational standard within the meaning of Section 111(h)(1) of the Clean Air Act.

(f)(1) Manufacturers of equipment used to control equipment leaks of VOC may apply to the Administrator for determination of equivalence for any equivalent means of emission limitation that achieves a reduction in emissions of VOC achieved by the equipment, design, and operational requirements of this subpart.

(2) The Administrator will make an equivalence determination according to the provisions of paragraphs (b), (c), (d), and (e).

§ 60.485 Test methods and procedures.

(a) Each owner or operator subject to the provisions of this subpart shall comply with the test method and procedure requirements provided in this section.

(b) Monitoring, as required in §§ 60.482, 60.483, and 60.484, shall comply with the following requirements:

(1) Monitoring shall comply with Reference Method 21.

(2) The detection instrument shall meet the performance criteria of Reference Method 21.

(3) The instrument shall be calibrated before use on each day of its use by the methods specified in Method 21.

(4) Calibration gases shall be:

(i) Zero air (less than 10 ppm of hydrocarbon in air); and

(ii) A mixture of methane or n-hexane and air at a concentration of approximately, but less than, 10,000 ppm methane or n-hexane.

(5) The instrument probe shall be traversed around all potential leak interfaces as close to the interface as possible as described in Reference Method 21.

(c) When equipment is tested for compliance with no detectable emissions as required in § 60.482-2(e), -3(i), -4, -7(f), and -10(e), the test shall comply with the following requirements:

(1) The requirements of paragraphs (b)(1)-(4) shall apply.

(2) The background level shall be determined, as set forth in Reference Method 21.

(3) The instrument probe shall be traversed around all potential leak interfaces as close to the interface as possible as described in Reference Method 21.

(4) The arithmetic difference between the maximum concentration indicated by the instrument and the background level is compared with 500 ppm for determining compliance.

(d)(1) Each piece of equipment within a process unit is presumed to be in VOC service unless an owner or operator demonstrates that the piece of equipment is not in VOC service. For a piece of equipment to be considered not in VOC service, it must be determined that the percent VOC content can be reasonably expected never to exceed 10 percent by weight. For purposes of determining the percent VOC content in the process fluid that is contained in or contacts equipment, procedures that conform to the general methods described in ASTM E-260, E-168, E-169 (incorporated by reference as specified in § 60.17) shall be used.

(2) If an owner or operator decides to exclude non-reactive organic compounds from the total quantity of

organic compounds in determining the percent VOC content of the process fluid, the exclusion will be allowed if:

(i) Those substances excluded are those considered as having negligible photochemical reactivity by the Administrator; and

(ii) The owner or operator demonstrates that the percent organic content, excluding non-reactive organic compounds, can be reasonably expected never to exceed 10 percent by weight.

(3)(i) An owner or operator may use engineering judgment rather than the procedures in paragraphs (d) (1) and (2) of this section to demonstrate that the percent VOC content does not exceed 10 percent by weight, provided that the engineering judgment demonstrates that the VOC content clearly does not exceed 10 percent by weight. When an owner or operator and the Administrator do not agree on whether a piece of equipment is not in VOC service, however, the procedures in paragraphs (d) (1) and (2) shall be used to resolve the disagreement.

(ii) If an owner or operator determines that a piece of equipment is in VOC service, the determination can be revised only after following the procedures in paragraphs (d) (1) and (2).

(e) Equipment is in light liquid service if the following conditions apply:

(1) The vapor pressure of one or more of the components is greater than 0.3 kPa at 20° C. Vapor pressures may be obtained from standard reference texts or may be determined by ASTM D-2879 (incorporated by reference as specified in § 60.17).

(2) The total concentration of the pure components having a vapor pressure greater than 0.3 kPa at 20° C is equal to or greater than 20 percent by weight; and

(3) The fluid is a liquid at operating conditions.

(f) Samples used in conjunction with paragraphs (d), (e), and (g) shall be representative of the process fluid that is contained in or contacts the equipment or the gas being combusted in the flare.

(g)(1) Reference Method 22 shall be used to determine the compliance of flares with the visible emission provisions of this subpart.

(2) The presence of a flare pilot flame shall be monitored using a thermocouple or any other equivalent device to detect the presence of a flame.

(3) The net heating value of the gas being combusted in a flare shall be calculated using the following equation:

$$H_T = K \left(\frac{n}{\sum_{i=1}^n C_i H_i} \right)$$

Where:

H_T = Net heating value of the sample, MJ/scm; where the net enthalpy per mole of offgas is based on combustion at 25°C and 760 mm Hg, but the standard temperature for determining the volume corresponding to one mole is 20°.

$$K = \text{Constant, } \frac{1}{1.740 \times 10^7} \left(\frac{\text{g mole}}{\text{ppm}} \right) \left(\frac{\text{MJ}}{\text{scm}} \right) \left(\frac{\text{Mj}}{\text{kcal}} \right)$$

where

$$\text{standard temperature for } \frac{\text{g mole}}{\text{scm}} \text{ is } 20^\circ\text{C.}$$

C_i = Concentration of sample component i in ppm, as measured by Reference Method 18 and ASTM D2504-67 (reapproved 1977) (incorporated by reference as specified in § 60.17).

H_i = Net heat of combustion of sample component i , kcal/g mole. The heats of combustion may be determined using ASTM D2382-76 (incorporated by reference as specified in § 60.17) if published values are not available or cannot be calculated.

(4) The actual exit velocity of a flare shall be determined by dividing the volumetric flowrate (in units of standard temperature and pressure), as determined by Reference Method 2, 2A, 2C, or 2D as appropriate; by the unobstructed (free) cross sectional area of the flare tip.

(5) The maximum permitted velocity, V_{max} , for air-assisted flares shall be determined by the following equation:

$$V_{max} = 8.706 + 0.7084(H_T)$$

V_{max} = Maximum permitted velocity, m/sec.
8.706 = Constant.

0.7084 = Constant.

H_T = The net heating value as determined in paragraph (g)(4).

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

§ 60.486 Recordkeeping requirements.

(a)(1) Each owner or operator subject to the provisions of this subpart shall comply with the recordkeeping requirements of this section.

(2) An owner or operator of more than one affected facility subject to the provisions of this subpart may comply with the recordkeeping requirements for

these facilities in one recordkeeping system if the system identifies each record by each facility.

(b) When each leak is detected as specified in § 60.482-2, -3, -7, -8, and § 60.483-2, the following requirements apply:

(1) A weatherproof and readily visible identification, marked with the equipment identification number, shall be attached to the leaking equipment.

(2) The identification on a valve may be removed after it has been monitored for 2 successive months as specified in § 60.482-7(c) and no leak has been detected during those 2 months.

(3) The identification on equipment except on a valve, may be removed after it has been repaired.

(c) When each leak is detected as specified in § 60.482-2, -3, -7, -8, and § 60.483-2, the following information shall be recorded in a log and shall be kept for 2 years in a readily accessible location:

(1) The instrument and operator identification numbers and the equipment identification number.

(2) The date the leak was detected and the dates of each attempt to repair the leak.

(3) Repair methods applied in each attempt to repair the leak.

(4) "Above 10,000" if the maximum instrument reading measured by the methods specified in § 60.485(a) after each repair attempt is equal to or greater than 10,000 ppm.

(5) "Repair delayed" and the reason for the delay if a leak is not repaired within 15 calendar days after discovery of the leak.

(6) The signature of the owner or operator (or designate) whose decision it was that repair could not be effected without a process shutdown.

(7) The expected date of successful repair of the leak if a leak is not repaired within 15 days.

(8) Dates of process unit shutdown that occur while the equipment is unrepaired.

(9) The date of successful repair of the leak.

(d) The following information pertaining to the design requirements for closed vent systems and control devices described in § 60.482-10 shall be recorded and kept in a readily accessible location:

(1) Detailed schematics, design specifications, and piping and instrumentation diagrams.

(2) The dates and descriptions of any changes in the design specifications.

(3) A description of the parameter or parameters monitored, as required in § 60.482-10(e), to ensure that control devices are operated and maintained in

conformance with their design and an explanation of why that parameter (or parameters) was selected for the monitoring.

(4) Periods when the closed vent systems and control devices required in § 60.482-2, -3, -4, and -5 are not operated as designed, including periods when a flare pilot light does not have a flame.

(5) Dates of startups and shutdowns of the closed vent systems and control devices required in § 60.482-2, -3, -4, and -5.

(e) The following information pertaining to all equipment subject to the requirements in § 60.482-1 to -10 shall be recorded in a log that is kept in a readily accessible location:

(1) A list of identification numbers for equipment subject to the requirements of this subpart.

(2)(i) A list of identification numbers for equipment that are designated for no detectable emissions under the provisions of § 60.482-2(e), -3(i) and -7(f).

(ii) The designation of equipment as subject to the requirements of § 60.482-2(e), -3(i), or -7(f) shall be signed by the owner or operator.

(3) A list of equipment identification numbers for pressure relief devices required to comply with § 60.482-4.

(4)(i) The dates of each compliance test as required in § 60.482-2(e), -3(i), -4, and -7(f).

(ii) The background level measured during each compliance test.

(iii) The maximum instrument reading measured at the equipment during each compliance test.

(5) A list of identification numbers for equipment in vacuum service.

(f) The following information pertaining to all valves subject to the requirements of § 60.482-7 (g) and (h) shall be recorded in a log that is kept in a readily accessible location:

(1) A list of identification numbers for valves that are designated as unsafe-to-monitor, an explanation for each valve stating why the valve is unsafe-to-monitor, and the plan for monitoring each valve.

(2) A list of identification numbers for valves that are designated as difficult-to-monitor, an explanation for each valve stating why the valve is difficult-to-monitor, and the schedule for monitoring each valve.

(g) The following information shall be recorded for valves complying with § 60.483-2:

(1) A schedule of monitoring

(2) The percent of valves found leaking during each monitoring period.

(h) The following information shall be recorded in a log that is kept in a readily accessible location:

(1) Design criterion required in § 60.482-2(d)(5) and § 60.482-3(e)(2) and explanation of the design criterion; and

(2) Any changes to this criterion and the reasons for the changes.

(i) The following information shall be recorded in a log that is kept in a readily accessible location for use in determining exemptions as provided in § 60.480(d):

(1) An analysis demonstrating the design capacity of the affected facility.

(2) A statement listing the feed or raw materials and products from the affected facilities and an analysis demonstrating whether these chemicals are heavy liquids or beverage alcohol, and

(3) An analysis demonstrating that equipment is not in VOC service.

(j) Information and data used to demonstrate that a piece of equipment is not in VOC service shall be recorded in a log that is kept in a readily accessible location.

(k) The provisions of §§ 60.7 (b) and (d) do not apply to affected facilities subject to this subpart.

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

(Approved by the Office of Management and Budget under the control number 2060-0012)

§ 60.487 Reporting Requirements.

(a) Each owner or operator subject to the provisions of this subpart shall submit semiannual reports to the Administrator beginning six months after the initial start up date.

(b) The initial semiannual report to the Administrator shall include the following information:

(1) Process unit identification.

(2) Number of valves subject to the requirements of § 60.482-7, excluding those valves designated for no detectable emissions under the provisions of § 60.482-7(f).

(3) Number of pumps subject to the requirements of § 60.482-2, excluding those pumps designated for no detectable emissions under the provisions of § 60.482-2(e) and those pumps complying with § 60.482-2(f).

(4) Number of compressors subject to the requirements of § 60.482-3, excluding those compressors designated for no detectable emissions under the provisions of § 60.482-3(i) and those compressors complying with § 60.482-3(h).

(c) All semiannual reports to the Administrator shall include the following information, summarized from the information in § 60.486:

(1) Process unit identification.

(2) For each month during the semiannual reporting period,

(i) Number of valves for which leaks were detected as described in § 60.482-7(b) or § 60.483-2,

(ii) Number of valves for which leaks were not reported as required in § 60.482-7(d)(1),

(iii) Number of pumps for which leaks were detected as described in §§ 60.482-2(b) and (d)(6)(i),

(iv) Number of pumps for which leaks were not repaired as required in §§ 60.482-2(c)(1) and (d)(6)(ii),

(v) Number of compressors for which leaks were detected as described in § 60.482-3(f),

(vi) Number of compressors for which leaks were not reported as required in § 60.482-3(g)(1), and

(vii) The facts that explain each delay of repair and, where appropriate, why a process unit shutdown was technically infeasible.

(3) Dates of process unit shutdowns which occurred within the semiannual reporting period.

(4) Revisions to items reported according to paragraph (b) if changes have occurred since the initial report or subsequent revisions to the initial report.

(d) An owner or operator electing to comply with the provisions of §§ 60.483-1 and -2 shall notify the Administrator of the alternative standard selected 90 days before implementing either of the provisions.

(e) An owner or operator shall report the results of all performance tests in accordance with § 60.8 of the General Provisions. The provisions of § 60.8(d) do not apply to affected facilities subject to the provisions of this subpart except that an owner or operator must notify the Administrator of the schedule for the initial performance tests at least 30 days before the initial performance tests.

(f) The requirements of paragraphs (a) through (c) of this subsection remain in force until and unless EPA, in delegating enforcement authority to a State under Section 111(c) of the Act, approves reporting requirements or an alternative means of compliance surveillance adopted by such State. In that event, affected sources within the State will be relieved of the obligation to comply with the requirements of paragraphs (a) through (c) of this subsection, provided that they comply with the requirements established by the State.

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

Approved by the Office of Management and Budget under the control number 2060-0012)

§ 60.488 [Reserved]

§ 60.489 List of chemicals produced by affected facilities.

(a) The following chemicals are produced, as intermediates or final products, by process units covered under this subpart. The applicability date for process units producing one or more of these chemicals is January 5, 1981.

CAS No. *	Chemical
105-57-7	Acetal.
75-07-0	Acetaldehyde.
107-89-1	Acetalsol.
60-35-5	Acetamide.
103-84-4	Acetanilide.
64-19-7	Acetic acid.
108-24-7	Acetic anhydride.
67-64-1	Acetone.
75-85-5	Acetone cyanohydrin.
75-05-8	Acetonitrile.
98-96-2	Acetophenone.
75-36-5	Acetyl chloride.
74-86-2	Acetylene.
107-02-8	Acrolein.
79-06-1	Acrylamide.
79-10-7	Acrylic acid.
107-13-1	Acrylonitrile.
124-04-9	Adipic acid.
111-89-3	Adiponitrile.
(*)	Alkyl naphthalenes.
107-18-6	Allyl alcohol.
107-05-1	Allyl chloride.
1321-11-5	Aminobenzoic acid.
111-41-1	Aminoethylethanolamine.
123-30-6	p-Aminophenol.
628-63-7, 123-92-2	Amyl acetates.
71-41-0*	Amyl alcohols.
110-58-7	Amyl amine.
543-59-9	Amyl chloride.
110-66-7*	Amyl mercaptans.
1322-06-1	Amyl phenol.
62-53-3	Aniline.
142-04-1	Aniline hydrochloride.
29191-52-4	Anisidine.
100-66-3	Anisole.
118-92-3	Anthranilic acid.
84-65-1	Anthraquinone.
100-52-7	Benzaldehyde.
55-21-0	Benzamide.
71-43-2	Benzene.
98-48-6	Benzenedisulfonic acid.
98-11-3	Benzenesulfonic acid.
134-81-6	Benzil.
76-93-7	Benzilic acid.
65-85-0	Benzic acid.
119-53-9	Benzoin.
100-47-0	Benzonitrile.
119-61-9	Borazophenone.
98-07-7	Benzotrichloride.
98-88-4	Benzoyl chloride.
100-51-6	Benzyl alcohol.
100-46-9	Benzylamine.
120-51-4	Benzyl benzoate.
100-44-7	Benzyl chloride.
98-87-3	Benzyl dichloride.
92-52-4	Biphenyl.
80-05-7	Bisphenol A.
10-88-1	Bromobenzene.
27497-51-4	Bromonaphthalene.
106-99-0	Butadiene.
106-95-9	1-butene.
123-86-4	n-butyl acetate.
141-32-2	n-butyl acrylate.
71-36-3	n-butyl alcohol.
78-92-2	s-butyl alcohol.
75-65-0	t-butyl alcohol.
109-73-9	n-butylamine.
13952-84-6	s-butylamine.
75-64-9	t-butylamine.
98-73-7	p-tert-butyl benzoic acid.
107-88-0	1,3-butylene glycol.
123-72-8	n-butylaldehyde.
107-92-6	Butyric acid.
106-31-0	Butyric anhydride.
109-74-0	Butyronitrile.
105-60-2	Caprolactam.
75-1-50	Carbon disulfide.

CAS No. *	Chemical	CAS No. *	Chemical	CAS No. *	Chemical
558-13-4	Carbon tetrabromide.	51-28-5	Dinitrophenol.	67-56-1	Methanol.
56-23-5	Carbon tetrachloride.	25321-14-8	Dinitrotoluene.	79-20-9	Methyl acetate.
9004-35-7	Cellulose acetate.	123-91-1	Dioxane.	105-45-3	Methyl acetoacetate.
79-11-8	Chloroacetic acid.	646-06-0	Dioxetane.	74-89-5	Methylamine.
106-42-9	m-chloroaniline.	122-39-4	Diphenylamine.	100-61-8	n-methylaniline.
95-51-2	o-chloroaniline.	101-84-8	Diphenyl oxide.	74-83-9	Methyl bromide.
106-47-8	p-chloroaniline.	102-08-9	Diphenyl thiourea.	37365-71-2	Methyl butynol.
35913-09-8	Chlorobenzaldehyde.	25265-71-8	Dipropylene glycol.	74-87-3	Methyl chloride.
106-90-7	Chlorobenzene.	25378-22-7	Dodecene.	106-87-2	Methylcyclohexane.
118-91-2, 535-80-8, 74-11-3*	Chlorobenzoic acid.	28675-17-4	Dodecylamine.	1331-22-2	Methylcyclohexanone.
2136-81-4,	Chlorobenzotrifluoride.	27193-88-8	Dodecylphenol.	75-09-2	Methylene chloride.
2136-89-2,		106-89-8	Epichlorohydrin.	101-77-9	Methylene dianiline.
5216-25-1*		64-17-5	Ethanol.	101-68-8	Methylene diphenyl diisocyanate.
1321-03-5	Chlorobenzoyl chloride.	141-43-5*	Ethanolamines.	78-93-3	Methyl ethyl ketone.
25497-29-4	Chlorodifluoromethane.	141-78-6	Ethyl acetate.	107-31-3	Methyl formate.
75-45-6	Chlorodifluoroethane.	141-87-9	Ethyl acetoacetate.	108-11-2	Methyl isobutyl carbinol.
67-66-3	Chloroform.	140-88-5	Ethyl acrylate.	108-10-1	Methyl isobutyl ketone.
25586-43-0	Chloronaphthalene.	75-04-7	Ethylamine.	60-62-6	Methyl methacrylate.
68-73-3	o-chloronitrobenzene.	100-41-4	Ethylbenzene.	77-75-6	Methylphenylol.
100-00-5	p-chloronitrobenzene.	74-96-4	Ethyl bromide.	98-83-9	a-methylstyrene.
25167-80-0	Chlorophenols.	9004-57-3	Ethylcellulose.	110-91-8	Morpholine.
126-99-8	Chloroprene.	75-00-3	Ethyl chloride.	85-47-2	a-naphthalene sulfonic acid.
7790-94-5	Chlorosulfonic acid.	105-39-5	Ethyl chloroacetate.	120-18-3	b-naphthalene sulfonic acid.
108-41-8	m-chlorotoluene.	105-56-6	Ethylcyanoacetate.	90-15-3	a-naphthol.
95-49-8	o-chlorotoluene.	74-85-1	Ethylene.	135-19-3	b-naphthol.
106-43-4	p-chlorotoluene.	98-49-1	Ethylene carbonate.	75-98-9	Neopentanoic acid.
75-72-9	Chlorotrifluoromethane.	107-07-3	Ethylene chlorohydrin.	88-74-4	o-nitroaniline.
108-39-4	m-cresol.	107-15-3	Ethylenediamine.	100-01-6	p-nitroaniline.
95-48-7	o-cresol.	105-93-4	Ethylene dibromide.	91-23-6	o-nitroanisole.
106-44-5	p-cresol.	107-21-1	Ethylene glycol.	100-17-4	p-nitroanisole.
1319-77-3	Mixed cresols.	111-55-7	Ethylene glycol diacetate.	96-95-3	Nitrobenzene.
1319-77-3	Cresylic acid.	110-71-4	Ethylene glycol dimethyl ether.	27178-83-2*	Nitrobenzoic acid (o,m, and p).
4170-30-0	Crotonaldehyde.	111-76-2	Ethylene glycol monobutyl ether.	79-24-3	Nitroethane.
3724-65-0	Crotonic acid.	112-07-2	Ethylene glycol monobutyl ether acetate.	75-52-5	Nitromethane.
98-82-8	Cumene.	110-80-5	Ethylene glycol monoethyl ether.	88-75-5	2-Nitrophenol.
80-15-9	Cumene hydroperoxide.	111-15-9	Ethylene glycol monethyl ether acetate.	25322-01-4	Nitropropane.
372-09-8	Cyanoacetic acid.	109-86-4	Ethylene glycol monomethyl ether.	1321-12-6	Nitrotoluene.
506-77-4	Cyanogen chloride.	110-49-6	Ethylene glycol monomethyl ether acetate.	27215-95-8	Nonene.
106-80-5	Cyanuric acid.	122-99-6	Ethylene glycol monophenyl ether.	25154-52-3	Nonylphenol.
106-77-0	Cyanuric chloride.	2607-30-9	Ethylene glycol monopropyl ether.	27493-28-8	Octylphenol.
110-82-7	Cyclohexane.	75-21-8	Ethylene oxide.	123-63-7	Paraldehyde.
108-93-0	Cyclohexanol.	60-29-7	Ethyl ether.	115-77-5	Pentaerythritol.
108-94-1	Cyclohexanone.	104-76-7	2-ethylhexanol.	109-66-0	n-pentane.
110-83-8	Cyclohexene.	122-51-0	Ethyl orthoformate.	109-67-1	1-pentene.
108-91-8	Cyclohexylamine.	95-92-1	Ethyl oxalate.	127-18-4	Perchloroethylene.
111-78-4	Cyclooctadiene.	41892-71-1	Ethyl sodium oxalacetate.	594-42-3	Perchloromethyl mercaptan.
112-30-1	Decanol.	50-00-0	Formaldehyde.	94-70-2	o-phenothione.
123-42-2	Decetone alcohol.	75-12-7	Formamide.	156-43-4	p-phenothione.
27576-04-1	Diaminobenzoic acid.	64-18-6	Formic acid.	106-95-2	Phenol.
95-78-1, 95-82-9, 554-00-7,	Dichloroaniline.	110-17-6	Fumaric acid.	98-67-9, 585-38-6, 609-46-1, 1333-39-7*	Phenolsulfonic acids.
808-27-5,		98-01-1	Furfural.	91-40-7	Phenyl anthranilic acid.
809-31-1,		56-81-5	Glycerol.	(*)	Phenylendiamine.
626-43-7,		26545-73-7	Glycerol dichlorohydrin.	75-44-5	Phosgene.
27134-27-6,		25791-98-2	Glycerol triether.	85-44-9	Phthalic anhydride.
57311-92-9*		56-40-6	Glycine.	85-41-6	Phthalimide.
541-73-1	m-dichlorobenzene.	107-22-2	Glyoxal.	108-99-6	p-picoline.
95-50-1	o-dichlorobenzene.	118-74-1	Hexachlorobenzene.	110-85-0	Piperazine.
106-46-7	p-dichlorobenzene.	67-72-1	Hexachloroethane.	9003-29-6	Polybutenes.
75-71-8	Dichlorodifluoromethane.	36853-82-4	Hexadecyl alcohol.	25036-29-7*	
111-44-4	Dichloroethyl ether.	124-09-4	Hexamethylenediamine.	25322-68-3	Polyethylene glycol.
107-06-2	1,2-dichloroethane (EDC).	629-11-8	Hexamethylene glycol.	25322-69-4	Polypropylene glycol.
98-23-1	Dichlorohydrin.	100-97-0	Hexamethylenetetramine.	123-38-6	Propionaldehyde.
28952-23-8	Dichloropropene.	74-90-6	Hydrogen cyanide.	79-09-4	Propionic acid.
101-83-7	Dicyclohexylamine.	123-31-9	Hydroquinone.	71-23-6	n-propyl alcohol.
109-89-7	Diethylamine.	99-96-7	p-hydroxybenzoic acid.	107-10-8	Propylamine.
111-46-6	Diethylene glycol.	26760-84-5	Isamylene.	540-54-5	Propyl chloride.
112-36-7	Diethylene glycol diethyl ether.	78-83-1	Isobutanol.	115-07-1	Propylene.
111-96-6	Diethylene glycol dimethyl ether.	110-19-0	Isobutyl acetate.	127-00-4	Propylene chlorohydrin.
112-34-5	Diethylene glycol monobutyl ether.	115-11-7	Isobutylene.	78-87-5	Propylene dichloride.
124-17-7	Diethylene glycol monobutyl ether acetate.	78-84-2	Isobutyraldehyde.	57-55-6	Propylene glycol.
111-90-0	Diethylene glycol monomethyl ether.	79-31-2	Isobutyric acid.	75-58-9	Propylene oxide.
112-15-2	Diethylene glycol monoethyl ether acetate.	25339-17-7	Isodecanol.	110-86-1	Pyridine.
111-77-3	Diethylene glycol monomethyl ether.	28952-21-8	Isocetyl alcohol.	106-51-4	Quinone.
64-67-5	Diethyl sulfate.	78-78-4	Isopentane.	106-46-3	Resorcinol.
75-37-6	Difluoroethane.	78-59-1	Isophorone.	27138-57-4	Resorcylic acid.
25167-70-8	Disobutylene.	121-81-5	Isophthalic acid.	69-72-7	Salicylic acid.
26761-40-0	Dodecyl phthalate.	78-79-5	Isoprene.	127-09-3	Sodium acetate.
27554-26-3	Disooctyl phthalate.	67-83-0	Isopropanol.	532-32-1	Sodium benzoate.
674-82-8	Diketene.	108-21-4	Isopropyl acetate.	9004-32-4	Sodium carboxymethyl cellulose.
124-40-3	Dimethylamine.	75-31-0	Isopropylamine.	3926-62-3	Sodium chloroacetate.
121-69-7	N,N-dimethylaniline.	75-29-6	Isopropyl chloride.	141-53-7	Sodium formate.
115-10-6	N,N-dimethyl ether.	25168-06-3	Isopropylphenol.	139-02-6	Sodium phenate.
68-12-2	N,N-dimethylformamide.	463-51-4	Ketene.	110-44-1	Sorbic acid.
57-14-7	Dimethylhydrazine.	(*)	Linear alkyl sulfonate.	100-42-5	Styrene.
77-78-1	Dimethyl sulfate.	123-01-3	Linear alkylbenzene (linear dodecylbenzene).	110-15-6	Succinic acid.
75-16-3	Dimethyl sulfide.	110-16-7	Maleic acid.	110-61-2	Succinonitrile.
67-68-5	Dimethyl sulfoxide.	108-31-6	Maleic anhydride.	121-57-3	Sulfamic acid.
120-61-6	Dimethyl terphenylate.	6915-15-7	Malic acid.	128-33-0	Sulfone.
98-34-3	3,5-dinitrobenzoic acid.	141-79-7	Mesityl oxide.	1401-55-4	Tannic acid.
		121-47-1	Metanilic acid.	100-21-0	Terephthalic acid.
		79-41-4	Methacrylic acid.	79-34-5*	Tetrachloroethanes.
		563-47-3	Methyl chloride.	117-06-8	Tetrachlorophthalic anhydride.

CAS No. *	Chemical
78-00-2	Tetraethyl lead.
119-64-2	Tetrahydronaphthalene.
85-43-8	Tetrahydrophthalic anhydride.
75-74-1	Tetramethyl lead.
110-60-1	Tetramethylenediamine.
110-18-9	Tetramethylethylenediamine.
108-88-3	Toluene.
95-80-7	Toluene-2,4-diamine.
584-84-9	Toluene-2,4-disocyanate.
26471-62-5	Toluene diisocyanates (mixture).
1333-07-9	Toluenesulfonamide.
104-15-4*	Toluenesulfonic acids.
98-59-9	Toluenesulfonyl chloride.
26915-12-8	Toluidines.
87-61-6, 108-70-3, 120-82-1*	Trichlorobenzenes.
71-55-6	1,1,1-trichloroethane.
79-00-5	1,1,2-trichloroethane.
79-01-6	Trichloroethylene.
75-69-4	Trichlorofluoromethane.
96-18-4	1,2,3-trichloropropane.
76-13-1	1,1,2-trichloro-1,2,2-trifluoroethane.
121-44-8	Triethylamine.
112-27-6	Triethylene glycol.
112-49-2	Triethylene glycol dimethyl ether.
7756-94-7	Trisobutylene.
75-50-3	Trimethylamine.
57-13-6	Urea.
108-05-4	Vinyl acetate.
75-01-4	Vinyl chloride.
75-35-4	Vinylidene chloride.
25013-15-4	Vinyl toluene.
1330-20-7	Xylenes (mixed).
95-47-6	o-xylene.
106-42-3	p-xylene.
1300-71-6	Xylenol.
1300-73-8	Xylidine.

*CAS numbers refer to the Chemical Abstracts Registry numbers assigned to specific chemicals, isomers, or mixtures of chemicals. Some isomers or mixtures that are covered by the standards do not have CAS numbers assigned to them. The standards apply to all of the chemicals listed, whether CAS numbers have been assigned or not.

*No CAS number(s) have been assigned to this chemical, its isomers, or mixtures containing these chemicals.

*CAS numbers for some of the isomers are listed; the standards apply to all of the isomers and mixtures, even if CAS numbers have not been assigned.

5. In Appendix A of Part 60, Method 18 is added as follows:

Method 18—Measurement of Gaseous Organic Compound Emissions by Gas Chromatography

Introduction

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

1. Applicability and Principle

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

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

1.2 Principle. This method is based on separating the major components of a gas mixture with a gas chromatograph (GC) and measuring the separated components with a suitable detector.

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

2. Range and Sensitivity

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

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

3. Precision and Accuracy

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

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

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

4. Interferences

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

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

Sample cross-contamination that occurs when high-level and low-level samples or standards are analyzed alternately, is best dealt with by thorough purging of the GC sample loop between samples.

To assure consistent detector response, calibration gases are contained in dry air. To eliminate errors in concentration calculations due to the volume of water vapor in the samples, moisture concentrations are determined for each sample, and a correction factor is applied to any sample with greater than 2 percent water vapor.

5. Presurvey and Presurvey Sampling

A presurvey shall be performed on each source to be tested. The purpose of the presurvey is to obtain all information necessary to design the emission test. The most important presurvey data are the

average stack temperature and temperature range, approximate particulate concentration, static pressure, water vapor content, and identity and expected concentration of each organic compound to be analyzed. Some of this information can be obtained from literature surveys, direct knowledge, or plant personnel. However, presurvey samples of the gas shall be obtained for analysis to confirm the identity and approximate concentrations of the specific compounds prior to the final testing.

5.2 Apparatus.

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

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

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

5.2.4 Flow Meter. To accurately monitor sample loop flow rate of 100 ml/min.

5.2.5 Regulators. Used on gas cylinders for GC and for cylinder standards.

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

5.2.7 Syringes. 1.0- and 10-microliter size, calibrated, maximum accuracy (gas tight) for preparing standards and for injecting head space vapor from liquid standards in retention time studies.

5.2.8 Tubing Fittings. To plumb GC and gas cylinders.

5.2.9 Septums. For syringe injections.

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

5.2.11 Soap Film Flow Meter. To determine flow rates.

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

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

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

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

5.2.16 Adsorption Tubes. If necessary, blank tubes filled with necessary adsorbent (charcoal, Tenax, XAD-2, etc.) for presurvey samples.

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

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

5.2 Reagents.

5.2.1 Deionized Distilled Water.

5.2.2 Methylene Dichloride.

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

5.2.4 Calibration Solutions. Samples of all the compounds of interest in a liquid form, for retention time studies.

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

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

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

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

5.3 Sampling.

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

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

5.3.1.1 Evacuated Flask Procedure. Use a high-vacuum pump to evacuate the flask to the capacity of the pump; then close off the stopcock leading to the pump. Attach a 6-mm outside diameter (OD) glass tee to the flask inlet with a short piece of Teflon tubing. Select a 6-mm OD borosilicate sampling probe, enlarged at one end to a 12-mm OD end of sufficient length to reach the centroid of the duct to be sampled. Insert a glass wool plug in the enlarged end of the probe to remove particulate matter. Attach the other end of the probe to the tee with a short piece of Teflon tubing. Connect a rubber suction bulb to the third leg of the tee. Place the filter end of the probe at the centroid of the duct, and purge the probe with the rubber suction bulb. After the probe is completely purged and filled with duct gases, open the stopcock to the grab flask until the pressure in the flask reaches duct pressure. Close off the stopcock, and remove the probe from the duct. Remove the tee from the flask and tape the stopcocks to prevent leaks during shipment. Measure and record the duct temperature and pressure.

5.3.1.2 Purged Flask Procedure. Attach one end of the sampling flask to a rubber suction bulb. Attach the other end to a 6-mm OD glass probe as described in Section 5.3.1.1. Place the filter end of the probe at the centroid of the duct, and apply suction with the bulb to completely purge the probe and

flask. After the flask has been purged, close off the stopcock near the suction bulb, and then close the stopcock near the probe. Remove the probe from the duct, and disconnect both the probe and suction bulb. Tape the stopcocks to prevent leakage during shipment. Measure and record the duct temperature and pressure.

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

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

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

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

6. Analysis Development

Presurvey samples shall be used to develop and confirm the best sampling and analysis scheme.

6.1 Selection of GC Parameters.

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

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

Plants with analytical laboratories may be able to provide information on their analytical procedures, including extractions, detector type, column types, compounds emitted, and approximate concentrations.

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

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

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

Use the GC conditions determined by the procedures of Section 6.1.2 for the first injection. Vary the GC parameters during subsequent injections to determine the optimum settings. Once the optimum settings have been determined, perform repeat injections of the sample to determine the retention time of each compound. To inject a sample, draw sample through the loop at a constant rate (100 ml/min for 30 seconds). Be careful not to pressurize the gas in the loop. Turn off the pump and allow the gas in the sample loop to come to ambient pressure. Activate the sample valve, and record injection time, loop temperature, column temperature, carrier flow rate, chart speed, and attenuator setting. Calculate the retention time of each peak using the distance from injection to the peak maximum divided by the chart speed. Retention times should be repeatable within 0.5 seconds.

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

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

6.2 Calibration Standards. If the presurvey samples are collected in an adsorbent tube (charcoal, XAD-2, Tenax, etc.), prepare the standards in the same solvent used for the extraction procedure for the adsorbent. Prepare several standards for each compound throughout the range of the sample.

6.2.1 Cylinder Calibration Gases. If available, use NBS reference gases or commercial gas mixtures certified through direct analysis for the calibration curves.

6.2.1.1 Optional Cylinder Approach. As an alternative procedure, maintain high and low calibration standards. Use the high concentration (50 to 100 ppm) standard to prepare a three-point calibration curve with an appropriate dilution technique. Use this

same approach also to verify the dilution techniques for high-concentration source gases.

To prepare the diluted calibration samples, use calibrated rotameters to meter both the high concentration calibration gas and the diluent gas. Adjust the flow rates through the rotameters with micrometer valves to obtain the desired dilutions. A positive displacement pump or other metering techniques may be used in place of the rotameter to provide a fixed flow of high concentration gas.

To calibrate the rotameters, connect each rotameter between the diluent gas supply and a suitably sized bubble meter, spirometer, or wet test meter. While it is desirable to calibrate the calibration gas flowmeter with calibration gas, generally the available amount of this gas will preclude it. The error introduced by using the diluent gas is insignificant for gas mixtures of up to 1,000 to 2,000 ppm of each organic component. Record the temperature and atmospheric pressures as follows:

$$Q_2 = Q_1 \left(\frac{P_2 T_1}{P_1 T_2} \right)^{1/2} \text{ Eq. 18-1}$$

Where:

Q_2 = Flow rate at new absolute temperature

(T_2) and new absolute pressure (P_2).

Q_1 = Flow rate at calibration absolute temperature (T_1) and absolute pressure (P_1).

Connect the rotameters to the calibration and diluent gas supplies using 6-mm Teflon tubing. Connect the outlet side of the rotameters through a connector to a leak-free Tedlar bag as shown in Figure 18-5. (See Section 7.1 for leak check procedures.) Adjust the gas flows to provide the desired dilution, and fill the bag with sufficient gas for calibration. Be careful not to fill to the point where it applies additional pressure on the gas. Record the flow rates of both rotameters, the ambient temperature, and atmospheric pressure. Calculate the concentration of diluted gas as follows:

$$C_a = \frac{10^6 \bar{X}_a q_a}{q_a + q_d} \text{ Eq. 18-2}$$

Where:

C_a = Concentration of component "a" in ppm.
 $\bar{X}_a$ = Mole fraction of component "a" in the calibration gas to be diluted.

q_a = Flow rate of the calibration gas contains mg component "a" at measured temperature and pressure.

q_d = Diluent gas flow at measured temperature and pressure.

Use single-stage dilutions to prepare calibration mixtures up to about 1:20 dilution factor. For greater dilutions, use a double dilution system. Assemble the apparatus, as shown in Figure 18-6, using calibrated flowmeters of suitable range. Adjust the control valves so that about 90 percent of the diluted gas from the first stage is exhausted,

and 10 percent goes to the second stage flowmeter. Fill the Tedlar bag with the dilute gas from the second stage. Record the temperature, ambient pressure, and water manometer pressure readings. Correct the flow reading in the first stage as indicated by the water manometer reading. Calculate the concentration of the component in the final gas mixture as follows:

$$C_a = 10^6 \bar{X}_a \left(\frac{q_{a1}}{q_{a1} + q_{d1}} \right) \left(\frac{q_{a2}}{q_{a2} + q_{d2}} \right) \text{ Eq. 18-3}$$

Where:

C_a = Concentration of component "a" in ppm.

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

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

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

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

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

Further details of the calibration methods for rotameters and the dilution system can be found in Citation 21 in Section 8.

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

6.2.2.1 Bag Technique. Evacuate a 10-liter Tedlar bag that has passed a leak check (see Section 7.1), and meter in 5.0 liters of nitrogen through a 0.5 liter per revolution dry test meter. While the bag is filling, use a 0.5-ml syringe to inject a known quantity of the material of interest through the wall of the bag or through a septum—capped tee at the bag inlet. Withdraw the syringe needle, and immediately cover the resulting hole with a piece of masking tape. In a like manner, prepare dilutions having other concentrations. Prepare a minimum of three concentrations. Place each bag on a smooth surface, and alternately depress opposite sides of the bag 50 times to mix the gases. Record the average meter temperature, gas volume, liquid volume, barometric pressure, and meter pressure.

Set the electrometer attenuator to the X1 Position. Flush the sampling loop with zero helium or nitrogen, and activate the sample valve. Record the injection time, sample loop temperature, column temperature, carrier gas flow rate, chart speed, and attenuator setting. Record peaks and detector responses that occur in the absence of any sample. Maintain conditions. Flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the calibration mixtures, and open the sample valve. Record the injection time. Select the peak that corresponds to the compound of interest. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. Divide this quantity by the chart speed, and record the resulting value as the retention time.

6.2.2.2 Preparation of Standards from less Volatile Liquid Materials. Use the equipment shown in Figure 18-8. Calibrate the dry gas meter with a wet test meter or a spirometer. Use a water manometer for the pressure gauge and glass, Teflon, brass, or stainless steel for all connections. Connect a valve to the inlet of the 50-liter Tedlar bag.

To prepare the standards, assemble the equipment as shown in Figure 18-8, and leak check the system. Completely evacuate the

bag. Fill the bag with hydrocarbon-free air, and evacuate the bag again. Close the inlet valve.

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

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

Complete filling of the bag; note and record the meter pressure and temperature at regular intervals, preferably 1 minute.

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

Disconnect the bag from the impinger outlet, and set it aside for at least 1 hour to equilibrate. Analyze the sample within the proven life period of its preparation.

6.2.2.3 Concentration Calculations. Average the meter temperature (T_m) and pressure (P_m) readings over the bag filling process.

Measure the solvent liquid density at room temperature by accurately weighing a known volume of the material on an analytical balance to the nearest 1.0 milligram. Take care during the weighing to minimize evaporation of the material. A ground-glass stoppered 25-ml volumetric flask or a glass-stoppered specific gravity bottle is suitable for weighing. Calculate the result in terms of g/ml. As an alternative, literature values of the density of the liquid at 20°C may be used.

Calculate the concentration of material in the sample in mg/liter at standard conditions as follows:

$$C_{std} = \frac{760(L_v)(p)(273 + T_m)}{273(M_f - M_i)(P_{bar} + P_m)} \text{ Eq. 18-4}$$

Where:

C_{std} = Standard solvent concentration, mg/std liter.

L_v = Liquid volume injected, ml.

p = Liquid density at room temperature, g/ml.

T_m = Meter temperature, °C.

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

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

P_m = Meter pressure (gauge), mm Hg.

6.3 Preparation of Calibration Curves. Obtain gas standards as described in Section 6.2 such that three concentrations per attenuator range are available. Establish proper GC conditioning, then flush the sampling loop for 30 seconds at a rate of 100 ml/min. Allow the sample loop pressure to equilibrate with atmospheric pressure, and activate the injection valve. Record the standard concentration, attenuator setting, injection time, chart speed, retention time,

peak area, sample loop, temperature, column temperature, and carrier gas flow rate. Repeat the standard injection until two consecutive injections give area counts within 5 percent of their average. The average multiplied by the attenuator setting is then the calibration area value for that concentration.

Repeat this procedure for each standard. Plot concentrations along the abscissa and the calibration area values along the ordinate. Perform a regression analysis, and draw the least squares line.

6.4 Optional Use of Prepared Cylinders for Dilution Calibration Checks, and Response Factor Determinations. A set of three standards of the major component in the emissions is required. This set of standards can be taken into the field and thereby replace the need to prepare standards as described in Section 6.2.2.

The high concentration standard can be run through the dilution system to assess the accuracy of the system. First, prepare a calibration curve using the three standards following the procedure described in Section 6.3. Then, prepare a dilute sample using the high concentration standard so that the dilute sample will fall within the lower limits of the calibration curve.

Next, analyze the dilute sample, and calculate the measured concentration from the calibration curve as described in Section 6.3. The dilute concentration calculated from the analysis shall be within 10 percent of the concentration expected from the dilution system; otherwise determine the source of error in the dilution system, and correct it.

The calibration curve from the cylinder standards for a single organic can also be related to the GC response curves of all the compounds in the source by response factors developed in the laboratory. In the field, the single calibration curve from the cylinder standards and the calculated response factors measured in the laboratory can then be used to replace the need to prepare and analyze calibration standards for each organic compound (see Section 6.5 on daily quality control procedure).

Recheck the relative peak area of one of the calibration standards daily to guard against degradation. If the relative peak areas on successive days differ by more than 5 percent, remake all of the standards before proceeding to the final sample analyses.

6.5 Evaluation of Calibration and Analysis Procedure. Immediately after the preparation of the calibration curve and prior to the final sample analyses, perform the analysis audit described in Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis" (47 FR 39179, September 7, 1982). The information required to document the analysis of the audit samples has been included on the example data sheets shown in Figures 18-3 and 18-7. The audit analyses shall agree with the audit concentrations within 10 percent. When available, the tester may obtain audit cylinders by contacting: Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711. Audit cylinders obtained from a commercial gas manufacturer may be

used provided: (a) the gas manufacturer certifies the audit cylinder as described in Section 5.2.3.1 of Method 23 and (b) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined as an analysis performed by an individual other than the individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis concentration is within 5 percent of the gas manufacturer's concentration.

7. Final Sampling and Analysis Procedure

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

7.1 Integrated Bag Sampling and Analysis

7.1.1 Evacuated Container Sampling Procedure. In this procedure, the bags are filled by evacuating the rigid air-tight container holding the bags. Therefore, check both the bags and the container for leaks before and after use as follows: Connect a water manometer using a tee connector between the bag or rigid container and a pressure source. Pressurize the bag or container to 5 to 10 cm H₂O (2 to 4 in. H₂O), and allow it to stand overnight. A deflated bag indicates a leak.

7.1.1.1 Apparatus.

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

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

7.1.1.1.3 Needle Valve. To control gas flow.

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

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

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

7.1.1.2 Sampling Procedure. To obtain a sample, assemble the sample train as shown in Figure 18-9. Leak check both the bag and the container. Connect the vacuum line from the needle valve to the Teflon sample line

from the probe. Place the end of the probe at the centroid of the stack, and start the pump with the needle valve adjusted to yield a flow of 0.5 liter/minute. After allowing sufficient time to purge the line several times, connect the vacuum line to the bag, and evacuate until the rotameter indicates no flow. Then position the sample and vacuum lines for sampling, and begin the actual sampling, keeping the rate proportional to the stack velocity. As a precaution, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Record the source temperature, barometric pressure, ambient temperature, sampling flow rate, and initial and final sampling time on the data sheet shown in Figure 18-10. Protect the Tedlar bag and its container from sunlight. When possible, perform the analysis within 2 hours of sample collection.

7.1.2 Direct Pump Sampling Procedure.

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

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

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

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

To use the second procedure, prefill the Tedlar bag with a known quantity of inert gas. Meter the inert gas into the bag according to the procedure for the preparation of gas concentration standards of volatile liquid materials (Section 6.2.2.2), but eliminate the midjet impinger section. Take the partly filled bag to the source, and meter the source gas into the bag through heated sampling lines and a heated flowmeter, or

Teflon positive displacement pump. Verify the dilution factors periodically through dilution and analysis of gases of known concentration.

7.1.5 Analysis of Bag Samples. Connect the needle valve, pump, charcoal tube, and flowmeter to draw gas samples through the gas sampling valve. Flush the sample loop with gas from one of the three Tedlar bags containing a calibration mixture, and analyze the sample. Obtain at least two chromatograms for the sample. The results are acceptable when the peak areas from two consecutive injections agree to within 5 percent of their average. If they do not agree, run additional samples until consistent area data are obtained. If this agreement is not obtained, correct the instrument technique problems before proceeding. If the results are acceptable, analyze the other two calibration gas mixtures in the same manner. Prepare the calibration curve by using the least squares method.

Analyze the two field audit samples as described in Section 6.5 by connecting each Tedlar bag containing an audit gas mixture to the sampling valve. Calculate the results; record and report the data to the audit supervisor. If the results are acceptable, proceed with the analysis of the source samples.

Analyze the source gas samples by connecting each bag to the sampling valve with a piece of Teflon tubing identified with that bag. Follow the restrictions on replicate samples specified for the calibration gases. Record the data. Analyze the other two bag samples of source gas in the same manner. After all three bag samples have been analyzed, repeat the analysis of the calibration gas mixtures. Use the average of the two calibration curves to determine the respective sample concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then report the final results by both calibration curves.

7.1.6 Determination of Bag Water Vapor Content. Measure the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.)

Use the field analytical data sheet as shown in Figure 18-11. The sheet has been designed to tabulate information from the bag collection, direct interface, and dilution interface systems; as a result, not all of the requested information will apply to any single method. Note the data that do not apply with the notation "N.A." Summarize the analysis.

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

7.2.1 Apparatus.

7.2.1.1 Probe. Constructed of stainless steel, Pyrex glass, or Teflon tubing as

required by duct temperature, 6.4-mm OD, enlarged at duct end to contain glass wool plug. If necessary, heat the probe with heating tape or a special heating unit capable of maintaining duct temperature.

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

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

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

7.2.1.5 Heated Gas Sampling Valve. Of two-position, six-port design, to allow sample loop to be purged with source gas or to direct source gas into the GC instrument.

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

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

7.2.1.8 Flowmeter. Of suitable range to measure sampling rate.

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

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

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

7.2.1.12 Recorder/Integrator. To record results.

7.2.2 Procedure. To obtain a sample, assemble the sampling system as shown in Figure 18-12. Make sure all connections are tight. Turn on the probe and sample line heaters. As the temperature of the probe and heated line approaches the source temperature as indicated on the thermocouple readout device, control the heating to maintain a temperature of 0 to 3°C above the source temperature. While the probe and heated line are being heated, disconnect the sample line from the gas sampling valve, and attach the line from the calibration gas mixture. Flush the sample loop with calibration gas and analyze a portion of that gas. Record the results. After the calibration gas sample has been flushed into the GC instrument, turn the gas sampling valve to flush position, then reconnect the probe sample line to the valve. Move the probe to the sampling position, and draw source gas into the probe, heated line, and sample loop. After thorough flushing, analyze the sample using the same conditions as for the calibration gas mixture. Repeat the analysis on an additional sample. Measure the peak areas for the two samples, and if they do not agree to within 5 percent of their mean value, analyze additional samples until two consecutive analyses meet this criteria. Record the data. After consistent results are

obtained, remove the probe from the source and analyze a second calibration gas mixture. Record this calibration data and the other required data on the data sheet shown in Figure 18-11, deleting the dilution gas information.

(Note.—Take care to draw all samples, calibration mixtures, and audits through the sample loop at the same pressure.)

In addition, analyze the field audit samples by connecting the audit sample cylinders to the gas sampling valve. Use the same instrument conditions as were used for the source samples. Record the data, and report the results of these analyses to the audit supervisor.

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

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

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

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

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

7.3.1.4 Flowmeters. Two, for measurement of diluent gas, expected delivery flow rate to be 1,350 cc/min.

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

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

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

The heated box shown in Figure 18-13 is designed to receive a heated line from the probe. An optional design is to build a probe unit that attaches directly to the heated box.

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

7.3.2 Procedure. Assemble the apparatus by connecting the heated box, shown in Figure 18-13, between the heated sample line from the probe and the gas sampling valve on the chromatograph. Vent the source gas from the gas sampling valve directly to the charcoal filter, eliminating the pump and rotameter. Heat the sample probe, sample line, and heated box. Insert the probe and source thermocouple at the centroid of the duct. Measure the source temperature, and adjust all heating units to a temperature 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds. Verify the operation of the dilution system by analyzing a high concentration gas of known composition through either the 10:1 or 100:1 dilution stages, as appropriate. (If necessary, vary the flow of the diluent gas to obtain other dilution ratios.) Determine the concentration of the diluted calibration gas using the dilution factor and the calibration curves prepared in the laboratory. Record the pertinent data on the data sheet shown in Figure 18-11. If the data on the diluted calibration gas are not within 10 percent of the expected values, determine whether the chromatograph or the dilution system is in error, and correct it. Verify the GC operation using a low concentration standard by diverting the gas into the sample loop, bypassing the dilution system. If these analyses are not within acceptable limits, correct the dilution system to provide the desired dilution factors. Make this correction by diluting a high-concentration standard gas mixture to adjust the dilution ratio as required.

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

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

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

7.4.1 Additional Apparatus. In addition to the equipment listed in the NIOSH method

for the particular organic(s) to be sampled, the following items (or equivalent) are suggested.

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

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

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

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

7.4.1.5 Stopwatch. To time sampling and pump rate calibration.

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

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

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

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

Use a sample probe, if required. Minimize the length of flexible tubing between the probe and adsorption tubes. Several adsorption tubes can be connected in series, if the extra adsorptive capacity is needed. Provide the gas sample to the sample system at a pressure sufficient for the limiting orifice to function as a sonic orifice. Record the total time and sample flow rate (or the number of pump strokes), the barometric pressure, and ambient temperature. Obtain a total sample volume commensurate with the expected concentration(s) of the volatile organic(s) present, and recommended sample loading factors (weight sample per weight adsorption media). Laboratory tests prior to actual sampling may be necessary to predetermine this volume. When more than one organic is present in the emissions, then develop relative adsorptive capacity information. If water vapor is present in the sample at concentrations above 2 to 3 percent, the adsorptive capacity may be severely reduced. Operate the gas chromatograph according to the manufacturer's instructions. After establishing optimum conditions, verify and document these conditions during all operations. Analyze the audit samples (see Section 7.4.4.3), then the emission samples.

Repeat the analysis of each sample until the relative deviation of two consecutive injections does not exceed 5 percent.

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

7.4.4 Quality Assurance.

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

7.4.4.2 Determination of Sample Collection Efficiency. For the source samples, analyze the primary and backup portions of the adsorption tubes separately. If the backup portion exceeds 10 percent of the total amount (primary and backup), repeat the sampling with a larger sampling portion.

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

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

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

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

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BILLING CODE 6560-50-M

I. Name of company _____ Date _____
 Address _____
 Contacts _____ Phone _____
 Process to be sampled _____
 Duct or vent to be sampled _____

II. Process description _____

III. Sampling site
 A. Description
 Site description _____
 Duct shape and size _____
 Material _____
 Wall thickness _____ inches
 Upstream distance _____ inches diameter
 Downstream distance _____ inches diameter
 Size of port _____
 Size of access area _____
 Hazards _____ Ambient temp. _____ °F

B. Properties of gas stream
 Temperature _____ °C _____ °F, Data source _____
 Velocity _____, Data source _____
 Static pressure _____ inches H₂O, Data source _____
 Moisture content _____ %, Data source _____
 Particulate content _____, Data source _____
 Gaseous components
 N₂ _____ % Hydrocarbons _____ ppm
 O₂ _____ %
 CO _____ %
 CO₂ _____ %
 SO₂ _____ %
 Hydrocarbon components
 _____ ppm
 _____ ppm
 _____ ppm
 _____ ppm
 _____ ppm
 _____ ppm

Operating cycle
 Check: Batch _____ Continuous _____ Cyclic _____
 Timing of batch or cycle _____
 Best time to test _____

Figure 18-1. Preliminary survey data sheet.

Figure 18-1 (continued). Preliminary survey data sheet.

C. Sampling considerations
Location to set up GC _____

Special hazards to be considered _____

Power available at duct _____

Power available for GC _____

Plant safety requirements _____

Vehicle traffic rules _____

Plant entry requirements _____

Security agreements _____

Potential problems _____

D. Site diagrams. (Attach additional sheets if required).

Figure 18-1 (continued). Preliminary survey data sheet.

Components to be analyzed _____

Expected concentration _____

Suggested chromatographic column _____

Column flow rate _____ ml/min Head pressure _____ mm Hg

Column temperature: _____

Isothermal _____ °C

Programmed from _____ °C to _____ °C at _____ °C/min

Injection port/sample loop temperature _____ °C

Detector temperature _____ °C

Detector flow rates: Hydrogen _____ ml/min.

head pressure _____ mm Hg

Air/Oxygen _____ ml/min,

head pressure _____ mm Hg

Chart speed _____ inches/minute

Compound data:

Compound _____

Retention time _____

Attenuation _____

Calibration Curve Data - Volatile and
Liquid Samples Collected in a Tedlar Bag

	Blank	Mixture 1	Mixture 2	Mixture 3
Size of Tedlar bag (liters)				
Dilution gas (name)				
Vol. of dilution gas (liters)				
Component (name)				
Volume of component (ml)				
Average meter temp. (°C)				
Average meter pressure (mm)				
Atmospheric pressure (mm)				
Density of liquid component (g/ml)				
Sample loop volume (ml)				
Sample loop temp. (°C)				
Carrier gas flow rate (ml/min)				
Column temperature initial (°C)				
Program rate (°C/min) final (°C)				
Injection time (24 hr. basis)				
Distance to peak (cm)				
Chart speed (cm/min)				
Retention time (min)				
Calculated concentration (ppm)				
Attenuator setting				
Peak height (mm)				
Peak area (mm ²)				
Area x attenuation				
Plot peak area x attenuation against concentration to obtain calibration curve.				

Figure 18-3. Calibration curve data sheet - injection of volatile sample into Tedlar bag.

125

Rotameter number

Gas used

Method: Bubble meter _____ Spirometer _____ Wet test meter _____

Rotameter construction _____

Float type _____

Laboratory temperature (T obs.) _____ °C _____ °F

Laboratory pressure (P obs.) _____ in Hg _____ mm Hg

1. Flowmeter reading Time (min) Gas volume^a (lab conditions)^b Flow rate

a Vol. of gas may be measured in milliliters, liters or cubic feet.

b Convert to standard conditions (20° C and 760 mm Hg).

1/2

$$Q_{STD} = Q_{obs} \frac{P_{obs.}}{P_{std.}} \times \frac{T_{std.}}{T_{obs.}}$$

Flowmeter reading

Flow rate (STD conditions)

Plot meter reading against flow rate (std) and draw smooth curve.

Figure 18-4. Rotameter calibration data sheet.

126

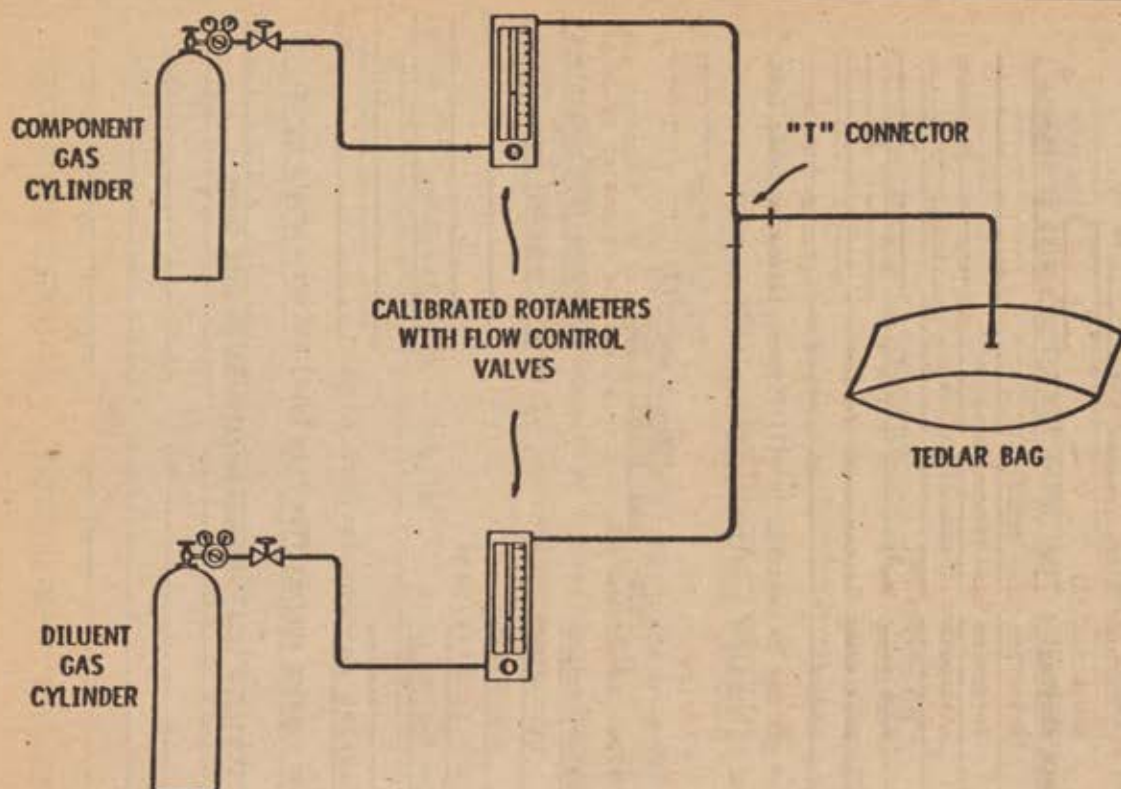


Figure 18-5. Single-stage calibration gas dilution system.

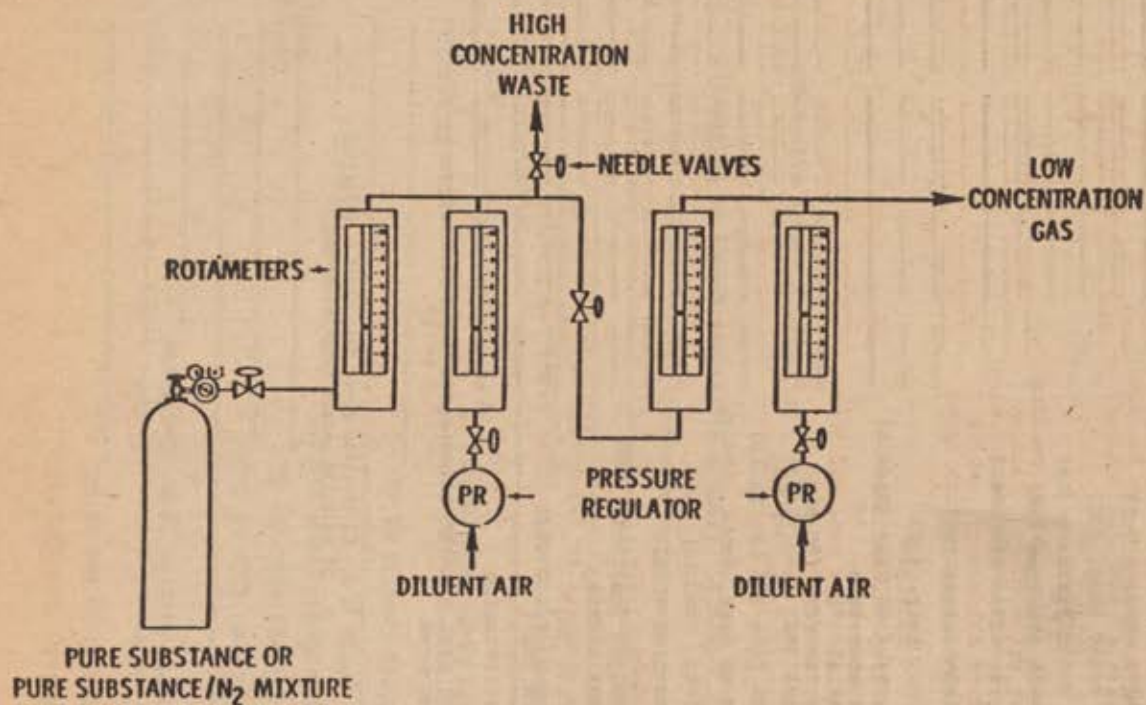


Figure 18-6. Two-stage dilution apparatus.

1. High concentration as mixture		Concentration _____ Ppm	
Component _____		Date _____	
Diluent gas _____		Mixture 1 Mixture 2 Mixture 3	
2. Dilution and analysis are			
Stage 1			
Component gas-rotameter reading			
Diluent gas-rotameter reading			
Ambient temp. (°C)			
Manometer reading, inches H ₂ O			
Flow rate component gas (ml/min)			
Flow rate diluent gas (ml/min)			
Stage 2			
Component gas-rotameter reading			
Diluent gas-rotameter reading			
Flow rate component gas (ml/min)			
Flow rate diluent gas (ml/min)			
Calculated concentration (ppm)			
Analysis			
Sample loop volume (ml)			
Sample loop temp. (°C)			
Carrier gas flow rate (ml/min)			
Column temperature			
initial (°C)			
program rate (°C/min)			
final (°C)			
Injection time (24-hr. basis)			
Distance to peak (inches)			
Chart speed (inch/min)			
Retention time (min)			
Attenuator factor			
Peak height (mm)			
Peak area (mm ²)			
Area x Attenuation factor (mm ²)			
Plot peak area x attenuator factor against concentration to obtain calibration curve.			

Figure 18-7. Calibration curve data sheet - dilution method.

1.9

3. Low concentration standard		Sample 1 Sample 2	
Known concentration (ppm)			
Retention time (min)			
Injection time (24-hour basis)			
Attenuation factor			
Peak height (mm)			
Peak area (mm ²)			
Peak area x attenuation (mm ²)			
Calculated concentration (ppm)			
Deviation (%)			
4. Audit samples			
Retention time (min)			
Injection time 24-hour basis)			
Attenuation factor			
Peak height (mm)			
Peak area (mm ²)			
Peak area x attenuation factor			
Measured concentration			
Data reported on (date)			
Data reported by (initial)			
Certified concentration (ppm)			
Deviation (%)			

Note: If a pump is used instead of a rotameter for component gas flow, substitute pump delivery rate for rotameter readings).

Figure 18-7 (continued). Calibration curve data sheet - dilution method.

1.5

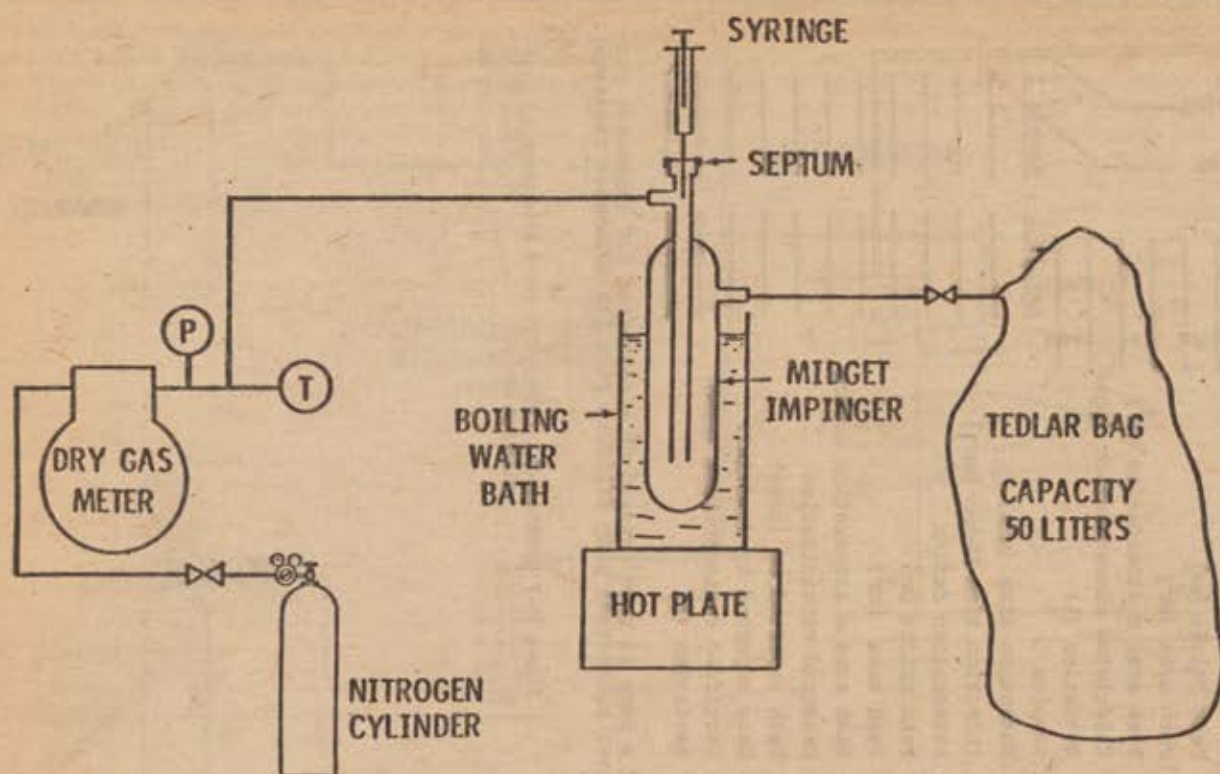


Figure 18-8. Apparatus for preparation of liquid materials.

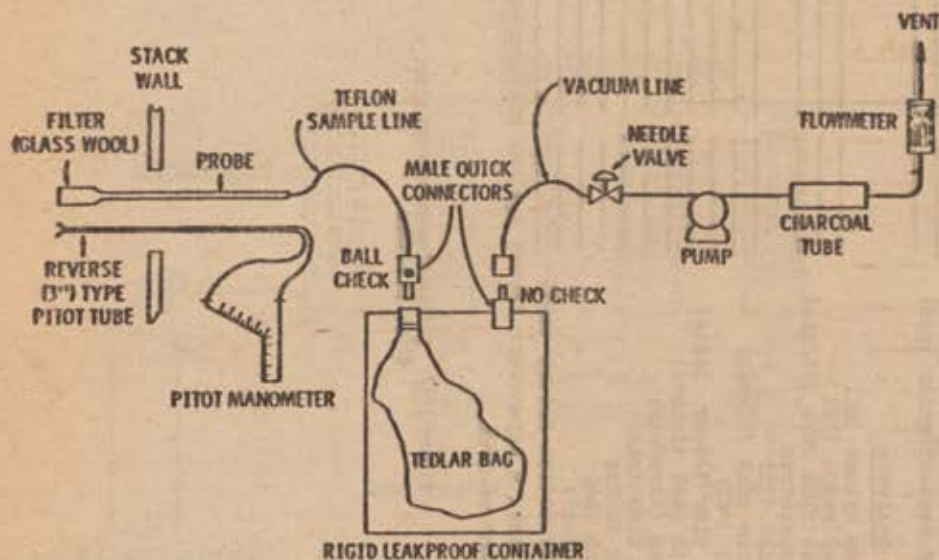


Figure 18-9. Integrated bag sampling train.

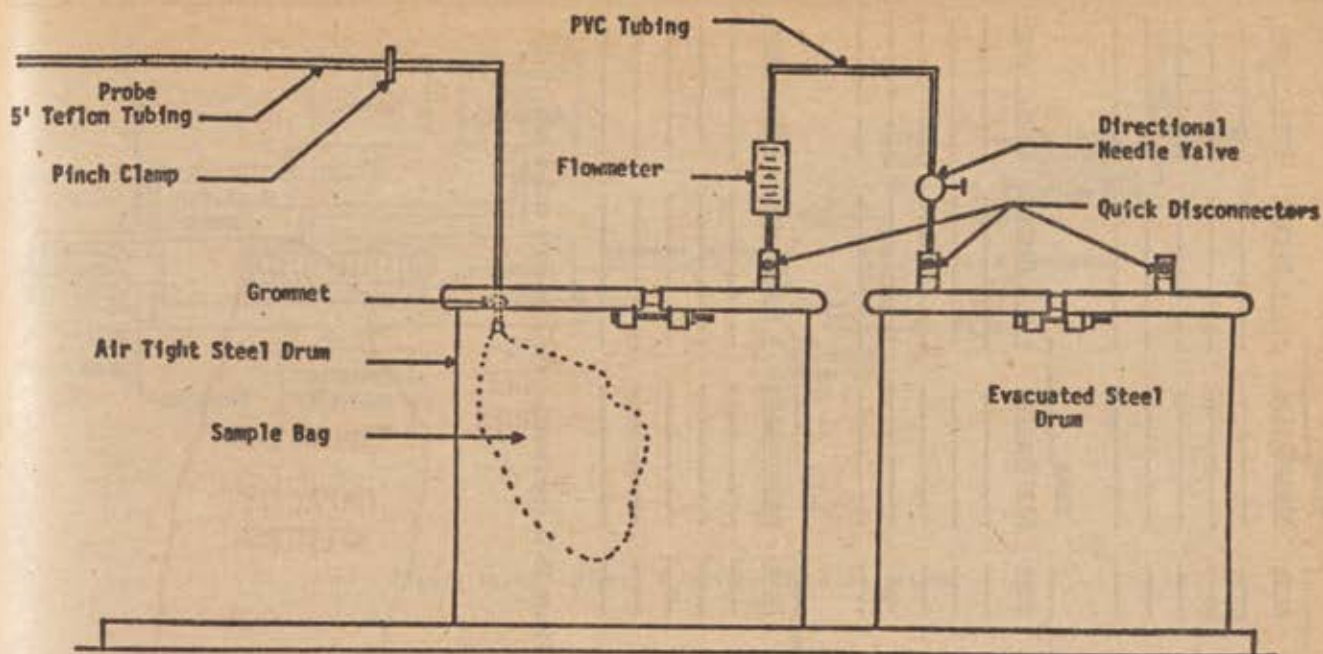


Figure 18-9a. Explosion risk gas sampling method.

Plant _____ Date _____

Site _____

	<u>Sample 1</u>	<u>Sample 2</u>	<u>Sample 3</u>
Source temperature (°C)	_____	_____	_____
Barometric pressure (mm Hg)	_____	_____	_____
Ambient temperature (°C)	_____	_____	_____
Sample flow rate (appr.)	_____	_____	_____
Bag number	_____	_____	_____
Start time	_____	_____	_____
Finish time	_____	_____	_____

Figure 18-10. Field sample data sheet - Tedlar bag collection method.

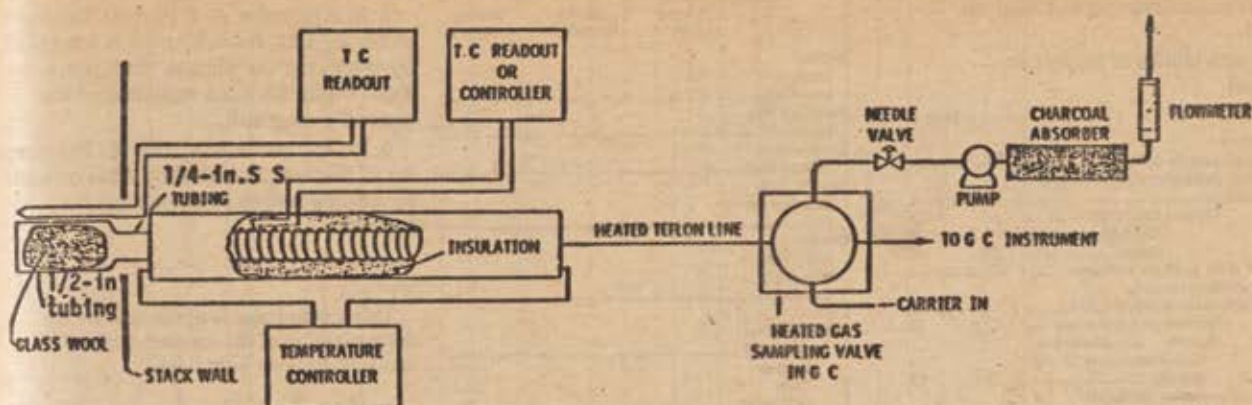


Figure 18-12. Direct interface sampling system.

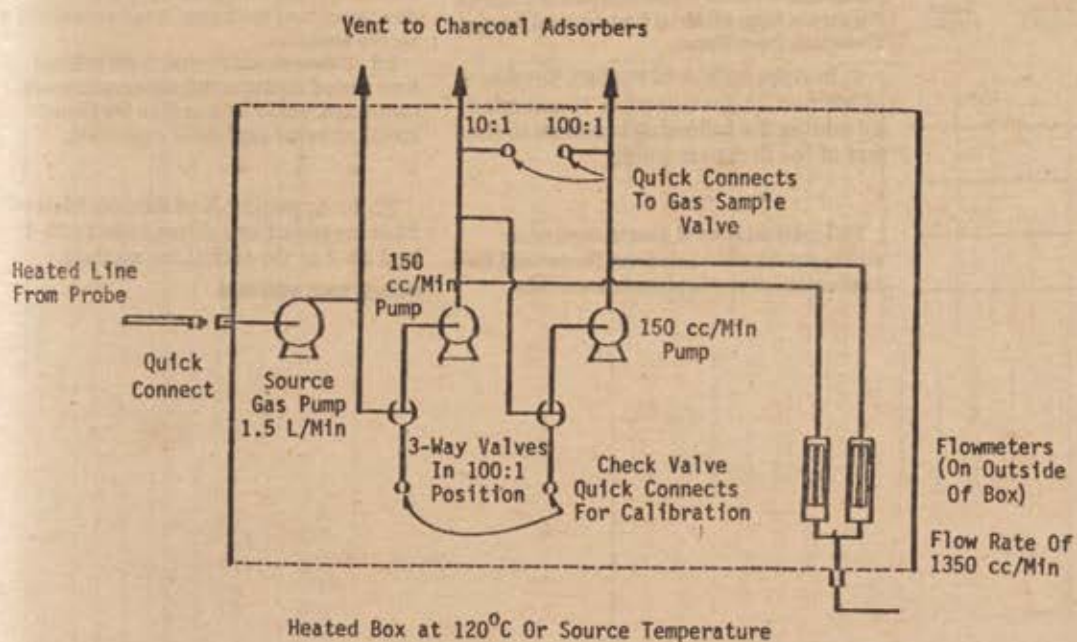


Figure 18-13. Schematic diagram of the heated box required for dilution of sample gas.

Gaseous Organic Sampling and Analysis
Check List

(Respond with initials or number as appropriate)

	Date
1. Presurvey data:	
A. Grab sample collected.....	☐
B. Grab sample analyzed for composition.....	☐
Method GC.....	☐
GC/MS.....	☐
Other.....	☐
C. GC-FID analysis performed.....	☐
2. Laboratory calibration data:	
A. Calibration curves prepared.....	☐
Number of components.....	☐
Number of concentrations/component (3 required).....	☐
B. Audit samples (optional):	
Analysis completed.....	☐
Verified for concentration.....	☐
OK obtained for field work.....	☐
3. Sampling procedures:	
A. Method:	
Bag sample.....	☐
Direct interface.....	☐
Dilution interface.....	☐
B. Number of samples collected.....	☐
4. Field analysis:	
A. Total hydrocarbon analysis performed.....	☐
B. Calibration curve prepared.....	☐
Number of components.....	☐
Number of concentrations per component (3 required).....	☐

Figure 18-14. Sampling and analysis check.

Gaseous Organic Sampling and Analysis
DataPlant—
Date—
Location—

	Source sample 1	Source sample 2	Source sample 3
1. General information:			
Source temperature (°C).....			
Probe temperature (°C).....			
Ambient temperature (°C).....			
Atmospheric pressure (mm Hg).....			

	Source sample 1	Source sample 2	Source sample 3
Source pressure (mm Hg).....			
Sampling rate (ml/min).....			
Sample loop volume (ml).....			
Sample loop temperature (°C).....			
Sample collection time (24-hr basis).....			
Columns:			
temperature:			
Initial (°C).....			
Program rate (°C/min).....			
Final (°C).....			
Carrier gas flow rate (ml/min).....			
Detector temperature (°C).....			
Chart speed (cm/min).....			
Dilution gas flow rate (ml/min).....			
Diluent gas used (symbol).....			
Dilution ratio.....			
Performed by (signature):.....			
Date.....			

Figure 18-14. Sampling and analysis sheet.

6. In Appendix A of Part 60, the title of Method 22 is revised to read as follows:

Method 22—Visual Determination of Fugitive Emissions from Material Sources and Smoke Emissions from Flares.7. In Appendix A of Part 60, Section 1 of Method 22, *Introduction*, is amended by adding the following sentence to the end of the first paragraph:

This method is used also to determine visible smoke emissions from flares used for combustion of waste process materials.

8. In Appendix A of Part 60, Section 1 of Method 22, *Introduction*, is amended by removing the phrase "Reference Test" from the third sentence of the second paragraph.

9. In Appendix A of Part 60, Paragraph 2.1 of Section 2 of Method 22 is amended by adding a second paragraph as follows:

2.1 * * *

This method also is applicable for the determination of the frequency of visible smoke emissions from flares.

10. In Appendix A of Part 60, Paragraph 2.2 of Section 2 of Method 22 is revised as follows:

2.2 *Principle.* Fugitive emissions produced during material processing, handling, and transfer operations or smoke emissions from flares are visually determined by an observer without the aid of instruments.11. In Appendix A of Part 60, Method 22 Section 3, *Definitions*, Paragraphs 3.4 and 3.5 are revised to read as follows:3.4 *Smoke Emissions.* Pollutant generated by combustion in a flare and occurring immediately downstream of the flame. Smoke occurring within the flame, but not downstream of the flame, is not considered a smoke emission.3.5 *Observation Period.* Accumulated time period during which observations are conducted, not to be less than the period specified in the applicable regulation.

12. In Appendix A of Part 60, Method 22 is amended by adding Figures 22-1 and 22-2 at the end of the method.

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Fugitive Emission Inspection Indoor Location Table

FUGITIVE EMISSION INSPECTION INDOOR LOCATION			
Company	Observer		
Location	Affiliation		
Company Representative	Date		
Industry	Process unit		
Light type (fluorescent, incandescent, natural)			
Light location (overhead, behind observer, etc.)			
Illuminance (lux or footcandles)			
Sketch process unit; indicate observer position relative to source; indicate potential emission points and/or actual emission points.			
OBSERVATIONS	Clock time	Observation period duration, min:sec	Accumulated emission time, min:sec
Beginning observation			
End observation			

Figure 22-2

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FUGITIVE OR SMOKE EMISSION INSPECTION OUTDOOR LOCATION			
Company	Observer		
Location	Affiliation		
Company representative	Date		
Sky Conditions	Wind direction		
Precipitation	Wind speed		
Industry	Process unit		
Sketch process unit; indicate observer position relative to source and sun; indicate potential emission points and/or actual emission points.			
OBSERVATIONS	Clock time	Observation period duration, min:sec	Accumulated emission time, min:sec
Begin Observation			
End observation			

Figure 22-3

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