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40 CFR Part 60, 61, and 63
Amendments for Testing and Monitoring Provisions; Final Rule

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Parts 60, 61, and 63

[FRL–6523–6]

RIN 2060–AG21

Amendments for Testing and Monitoring Provisions

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule: amendments.

SUMMARY: In this rule, we, the Environmental Protection Agency (EPA) are making final minor amendments to our stationary source testing and monitoring rules. These amendments include miscellaneous editorial changes and technical corrections that are needed. We are also promulgating Performance Specification 15, which contains the criteria for certifying continuous emission monitoring systems (CEMS) that use fourier transform infrared spectroscopy (FTIR). In addition, we are changing the outline of the test methods and CEMS performance specifications already listed in Parts 60, 61, and 63 to fit a new format recommended by the Environmental Monitoring Management Council (EMMC). The editorial changes and technical corrections update the rules and help maintain their original intent. Performance Specification 15 will provide the needed acceptance criteria for FTIR CEMS as they emerge as a new technology. We are reformatting the test methods and performance specifications to make them more uniform in content and interchangeable with other Agency methods. The amendments apply to a large number of industries that are already subject to the current provisions of Parts 60, 61, and 63. Therefore, we have not listed specific affected industries or their Standard Industrial Classification codes here.

DATES: *Effective Date.* This regulation is effective October 17, 2000. The incorporation by reference of certain publications listed in the rule is approved by the Director of the Federal Register as of October 17, 2000.

ADDRESSES: *Docket.* Docket No. A–97–12, contains information relevant to this rule. You can read and copy it between 8 a.m. and 5:30 p.m., Monday through Friday, (except for Federal holidays), at our Air and Radiation Docket and Information Center, U.S. Environmental Protection Agency, 401 M Street, SW., Washington, DC 20460; telephone (202) 260–7548. Go to Room M–1500, Waterside Mall (ground floor). The docket office may charge a reasonable fee for copying.

Summary of Comments and Responses Document. You may obtain the Summary of Comments and Responses Document over the Internet at http://www.epa.gov/ttn/emc; choose the "Methods" menu, then choose the "Summary of Comments and Responses" hypertext under Category A.

FOR FURTHER INFORMATION CONTACT: Mr. Foston Curtis, Emission Measurement Center (MD–19), Emissions, Monitoring, and Analysis Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711; telephone (919) 541–1063; facsimile number (919) 541–1039; electronic mail address "curtis.foston@epamail.epa.gov".

SUPPLEMENTARY INFORMATION: *Outline.* The information presented in this preamble is organized as follows:

I. Why were these amendments made?
II. What does the new EMMC Format for methods look like?
III. What were the significant public comments and what resulting changes were made since proposal?
  A. Updates to the ASTM Methods
  B. Performance requirements for continuous instrumental methods of Part 60—Methods 3A, 6C, 7E, 10, and 20
  C. Method 18 (Part 60)
  D. Method 25 (Part 60)
  E. Performance Specification 15 (Part 60)
IV. What revisions were made that were not in the proposed rule?
V. What are the administrative requirements for this rule?
  A. Docket
  B. Office of Management and Budget Review
  C. Regulatory Flexibility Act Compliance
  D. Paperwork Reduction Act
  E. Unfunded Mandates Reform Act
  F. E.O. 13132—Federalism
  G. E.O. 13084—Consultation and Coordination with Indian Tribal Governments
  H. Executive Order 13084—Protection of Children from Environmental Health Risks and Safety Risks
  I. Submission to Congress and the General Accounting Office
  J. National Technology Transfer and Advancement Act
  K. Plain Language in Government Writing

I. Why Were These Amendments Made?

We have compiled miscellaneous errors and editions that are needed for the test methods, performance specifications, and associated regulations in 40 CFR Parts 60, 61, and 63. The corrections and revisions consist primarily of typographical errors, technical errors in equations and diagrams, and narrative that is no longer applicable or is obsolete. Some of the revisions were brought to our attention by the public. The major changes to the rule proposed on August 27, 1997 that resulted from public comments are discussed in Section III. Please note that, although numerous technical corrections were made to Parts 60, 61, and 63 rules, none affected a compliance standard or reporting or recordkeeping requirement. Revisions were only made to sections that pertain to source testing or monitoring of emissions and operations.

II. What Does the New EMMC Format for Methods Look Like?

The new EMMC format we have adopted for analytical methods was developed by consensus and will help integrate make consistent the test methods written by different EPA programs. The test methods and performance specifications being restructured in the new format are shown in Table 1.

TABLE 1.—TEST METHODS AND PERFORMANCE SPECIFICATIONS RESTRUCTURED IN THE EMMC FORMAT

40 CFR 60 App. A	40 CFR 60 App. B	40 CFR 61	40 CFR 63
1, 1A	PS–2	101, 101A	303, 303A
2, 2A, 2B, 2C, 2D, 2E	PS–3	102	304A, 304B
3, 3A, 3B	PS–4, PS–4A	103	305
4	PS–5	104	306, 306A, 306B
5, 5A, 5B, 5D, 5E, 5F, 5G, 5H	PS–6	105	
6, 6A, 6B, 6C		106	
7, 7A 7B, 7C, 7D, 7E		107, 107A	
8		108, 108A, 108B, 108C	
10, 10A, 10B		111	
11			
12			

TABLE 1.—TEST METHODS AND PERFORMANCE SPECIFICATIONS RESTRUCTURED IN THE EMMC FORMAT—Continued

40 CFR 60 App. A	40 CFR 60 App. B	40 CFR 61	40 CFR 63
13A, 13B			
14			
15, 15A			
16, 16A, 16B			
17			
18			
19			
20			
21			
22			
23			
24, 24A			
25, 25A, 25B, 25C, 25D, 25E			
26, 26A			
27			
28, 28A			
29			

The methods and specifications listed in Table 1 were restructured in the format shown in Table 2. Only in a few instances were there deviations from this recommended format.

TABLE 2.—EMMC FORMAT

Section No.	Section heading
1.0	Scope and Application.
2.0	Summary of the Method.
3.0	Definitions.
4.0	Interferences.
5.0	Safety.
6.0	Equipment and Supplies.
7.0	Reagents and Standards.
8.0	Sample Collection, Preservation, Storage and Transport.
9.0	Quality Control.
10.0	Calibration and Standardization.
11.0	Analytical Procedure.
12.0	Calculations and Data Analysis.
13.0	Method Performance.
14.0	Pollution Prevention.
15.0	Waste Management.
16.0	References.
17.0	Tables, Diagrams, Flowcharts, and Validation Data.

III. What Were the Significant Public Comments and What Resulting Changes Were Made Since Proposal?

We asked that public comments on the August 27, 1997 proposal (62 FR 45369) be submitted by October 27, 1997. On November 18, 1997, we reopened (62 FR 61483) the comment period to allow additional time for review and comment. We received comments from facility owners and operators, trade associations, State and Local air pollution control agencies, environmental consultants, and private citizens. Their comments were considered in developing this final action. A detailed discussion of all comments are contained in the Summary of Comments and Responses Document (see **ADDRESSES** section of this preamble). The major public comments and the Agency's responses are summarized below.

A. Update to ASTM Methods

Several commenters supported our updating the references to ASTM Standards to include the dates of the most recent versions. However, some were concerned that updated standards not supplant the versions previously allowed and those promulgated with the original regulation. The ASTM recommended we follow the tradition of other governmental agencies and list only the latest version of each standard. This would present the latest, most improved standard. They felt that previously approved versions would still be acceptable for future use, and this could be noted in the preamble to the final rule.

On January 14, 1998, we published a supplementary **Federal Register** notice to solicit public comments on this idea. We received three comment letters. All commenters objected to the idea of listing only the latest version of the ASTM standard. The commenters noted problems that would be encountered with State Implementation Plans (SIP) wherein only the specific ASTM standards listed in the subparts would be allowed. They feared that listing only the latest version of the standard would change the current allowance to use earlier versions. This could potentially change the intent of the original emission standard. Most commenters didn't think a preamble explanation was sufficient assurance for continued allowance of earlier versions since preambles are not published in the Code of Federal Regulations. There were additional concerns for laboratories using currently acceptable versions who would need to upgrade their practice to reflect the latest version of a standard. The commenters were not amenable to only listing the latest standard unless language were added to the General Provisions of each part stating that previously allowed versions of the standards were still allowed at the discretion of the source. We feel the commenters have valid concerns and have decided to continue the convention of listing all acceptable versions of the ASTM standards including the new updates. The intent of this action is to allow any of the yearly-designated versions of a specific standard to be used in the applications where cited.

B. Performance Requirements for Continuous Instrumental Methods of Part 60—Methods 3A, 6C, 7E, 10, and 20

Several commenters thought the preamble language for this proposal gave inadequate notice of the changes being made. Commenters stated that, in the proposal, we did not provide an adequate basis and purpose statement and misled the readers into thinking that the proposal contained no substantive changes to these test methods. Based on the number of substantive changes in this proposal, and in light of the Section 307(d) requirements, the commenters felt that we must address these issues in a new proposal before the revisions can go final with the rest of the package. We agree with the commenters that the preamble to the proposed rule may not have given adequate public notice for some of the revisions. The revisions to the continuous instrumental methods (Methods 3A, 6C, 7E, 10, and 20) may be considered substantive, but were not enumerated in the preamble nor was a supporting rationale given. Therefore, the revisions to Methods 3A, 6C, 7E, 10, and 20 will be reproposed as a separate rule. The comments already received on the proposal of these methods will be held for consideration with any future comments that result from the reproposal.

C. Method 18 (Part 60, Appendix A)

One commenter thought Method 18 was difficult to follow. The commenter suggested that, to simplify organization of the method, we should divide the method into five categories. Each title would begin with "Measurement of Gaseous Organic Compounds by Gas Chromatography" but have the following subtitles:

18A—Evacuated container sampling procedure.
18B—Bag sampling procedure.
18C—Direct interface procedure.
18D—Dilution interface procedure.
18E—Adsorption tube sampling procedure.

Another commenter suggested dividing the method into two different methods, one for the direct extractive technique, and the other for sample collection into bags, flasks, or adsorbents.

The method is currently divided according to the various sampling procedures; for example, Section 8.2.2 is the Direct Interface Sampling and Analysis Procedures, Section 8.2.3 is Dilution Interface Sampling and Analysis Procedures, and so on. We do not believe that multiple sampling procedures warrant dividing Method 18 into separate methods. We feel a single method allowing different procedures offers the source greater flexibility than citing specific procedures for particular situations. One commenter noted that the proposed method requires triplicate injections for analysis of the calibration standards used for preparing the pre-test calibration curve, triplicate injections of the test samples, and triplicate injections for construction of the post-test calibration curve. The commenter questioned the additional accuracy expected for the extra hours spent in sample analysis and calibration while in the field conducting a source test compared to the current method which requires two consecutive analyses for pre- and post-test calibration and sample analyses meeting the same criteria for acceptance. We are increasing the calibration requirement to triple injections to tighten the method's quality assurance procedures. Triplicate calibration injections is the normal procedure prevalent in the analytical community, as well as in other Agency methodologies. It is difficult to establish precision and accuracy with duplicate injections. However, triplicate injections provide a reasonable measure of analytical precision without being overly burdensome. We do not feel the increase in time and costs associated with the third injection will significantly affect a typical test, considering the added benefits to data quality that are gained.

Several commenters asked us to revise and clarify various aspects of Section 10. We have made these modifications to address their concerns.

Regarding Section 13.1, one commenter noted that Method 18 is not a method in the general sense, but is more of a guideline on how to develop and document a test method. The commenter therefore felt that any prospective method should be written up and submitted to us along with the proper documentation that includes recovery study results. We disagree with this commenter. Method 18, which has been cited and used for many years, is a specific gas chromatography method with specific sampling, analytical, and data quality requirements. The method was written to accomodate many test sites having many possible target compounds and gas matrices. The tester has been given numerous sampling, separation, and analytical system options to make the method adaptable to the needs of various compliance demonstrations.

Several commenters asked us to clarify the 5 to 10 percent relative standard deviation (RSD) requirement for calibration standards in Section 13.1.

We have added clarity to Section 13.1. The 5 to 10 percent RSD is not a precision criterion for calibration standards but a typical precision range for analyzing field samples. Five percent RSD is required for triplicate injections of calibration standards.

D. Method 25 (Part 60, Appendix A)

One commenter noted that Method 25 has limitations due to conditions that may exist in stack gas. If such conditions exist, the commenter recommends interfacing a nonmethane analyzer directly to the source or use Method 25A or 25B to measure the emissions. The commenter recommended modifying Method 25 to allow instruments that are able to determine the methane and nonmethane portions using components different from those described by Method 25 when the analyzer is directly interfaced to the source. The commenter feels that Method 25 would be more practical for determining methane/nonmethane emissions at the field site if the method could be modified to allow these other analyzers. The commenter feels that it will also be necessary that fixed performance specifications be defined in the method, such as those for Method 6C. We believe these comments address method changes that are beyond those covered in the proposal and are, therefore, beyond the scope of this action. The commenter is encouraged to pursue these method changes through other appropriate channels such as submitting a request to use them as an alternative method.

E. Performance Specification 15 (Part 60, Appendix B)

One commenter noted that the statement of applicability for the demonstration is limited to the criteria we gave. The commenter stated that, with performance based measurement systems, the focus is on data quality objectives (DQO) where the performance specifications are coupled with the DQO. We believe the purpose of reference methods and, in this case performance specifications, is to provide standard procedures for sources to follow in order to provide quality emission data. However, we do provide latitude to sources by publishing performance-based methods and PS whenever possible. This performance specification is one such procedure; as long as an FTIR sampling system meets the requirements of the performance specifications, it can be used for any regulated pollutant.

Based on public comments and upon further deliberation, we have removed the system calibration requirement from Section 10.3 of PS–15. Since both a system calibration and the calibration transfer standard measurement basically test instrument function, having both of these requirements in the performance specifications is redundant.

One commenter felt that the number of runs should be given as "guidance" rather than made a requirement. We set the requirement for nine runs (when comparing the FTIR to a reference method) and 10 runs (when comparing the FTIR to a reference monitor) because these are standard prodedures for performance specifications. We note that this performance specification also allows analyte spiking as an option; therefore, a revision on this point is not necessary.

One commenter noted that Section 11.1.1.4.3 states "if the RM is a CEM, synchronize the sampling flow rates of the RM and the FTIR CEM." The commenter noted that instrumental analyzers are currently used for reference methods. EPA Methods 6C, 7E, 3A, and 10 measure SO_2, NO_X, O_2, CO_2, and CO on a continuous basis for a short period of time and are referred to as instrumental analyzers and not CEMs. The commenter felt the statement should read "if the reference method is an instrumental analyzer, synchronize the sampling flow rates of the RM and the FTIR." We agree with the commenter and have made the noted change.

IV. What Revisions Were Made That Were Not in the Proposed Rule?

A revision was made to Section 6.6 of Method 21 of Part 60 to clarify the VOC monitoring instrument specifications. The requirement for the instrument to be intrinsically safe for Classes 1 and 2, Division 1 conditions has been amended to require them to be intrinsically safe for Class 1 and/or Class 2, Division 1 conditions, as appropriate. The performance test provisions of § 60.754(d) for determining control device efficiency when combusting landfill gas were amended to allow the use of Method 25 as an alternative to Methods 18 and 25C. The tester has the option of using either Method 18, 25, or 25C in this case. These amendments were not published in the proposed rule.

V. Administrative Requirements

A. Docket

Docket A–97–12 is an organized and complete file of all information submitted to us or otherwise considered in the development of this final rulemaking. The principal purposes of the docket are: (1) to allow interested parties to identify and locate documents so that they can effectively participate in the rulemaking process, and (2) to serve as the record in case of judicial review (except for interagency review materials) [Clean Air Act Section 307(d)(7)(A), 42 U.S.C. 7607(d)(7)(A)].

B. Office of Management and Budget Review

Under Executive Order 12866 (58 FR 51735 October 4, 1993), we must determine whether the regulatory action is "significant" and therefore subject to Office of Management and Budget (OMB) review and the requirements of this Executive Order. The Order defines "significant regulatory action" as one that is likely to result in a rule that may: (1) Have an annual effect on the economy of $100 million or more or adversely affect in a material way the economy, a sector of the economy, productivity, competition, jobs, the environment, public health or safety, or State, Local, or Tribal governments or communities; (2) Create a serious inconsistency or otherwise interfere with an action taken or planned by another agency; (3) Materially alter the budgetary impact of entitlements, grants, user fees, or loan programs, or the rights and obligations of recipients thereof; or (4) Raise novel legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in the Executive Order.

We have determined that this rule is not a "significant regulatory action" under the terms of Executive Order 12866 and is therefore not subject to OMB review. We have determined that this regulation would result in none of the economic effects set forth in Section 1 of the Order because it does not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard.

C. Regulatory Flexibility Act Compliance

We have determined that it is not necessary to prepare a regulatory flexibility analysis in connection with this final rule. We have also determined that this rule will not have a significant economic impact on a substantial number of small businesses. This rulemaking does not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard.

D. Paperwork Reduction Act

This rule does not impose or change any information collection requirements. The Paperwork Reduction Act of 1980, 44 U.S.C. 3501, *et seq.*, is not required.

E. Unfunded Mandates Reform Act

Title II of the unfunded Mandates Reform Act of 1995 (UMRA), Pub. L. 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory action on State, local, and tribal governments and the private sector. Under section 202 of the UMRA, we generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with "Federal mandates" that may result in expenditures by State, local, and tribal governments, in the aggregate, or by the private sector, of $100 million or more in any one year. Before promulgating an EPA rule for which a written statement is needed, Section 205 of the UMRA generally requires us to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of Section 205 do not apply when they are inconsistent with applicable law. Moreover, Section 205 allows EPA to adopt an alternative other than the least costly, most cost-effective or least burdensome alternative if the Administrator publishes with the final rule an explanation why that alternative was not adopted. Before we establish any regulatory requirement that may significantly or uniquely affect small governments, including tribal governments, we must develop a small government agency plan as required under Section 203 of the UMRA. The plan must provide for notifying potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of our regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

Today's rule contains no Federal mandates (under the regulatory provisions of Title II of the UMRA) for State, local, or tribal governments or the private sector. We have determined that today's rule does not include a Federal mandate because it imposes no enforceable duty on any State, local, and tribal governments, or the private sector. Today's rule simply makes corrections and minor revisions to current testing requirements and promulgates a monitoring specification that can be used to support future monitoring rules. For the same reason we have also determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments.

F. Executive Order 13132 (Federalism)

Executive Order 13132, entitled "Federalism" (64 FR 43255, August 10, 1999), requires EPA to develop an accountable process to ensure "meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications." "Policies that have federalism implications" is defined in the Executive Order to include regulations that have "substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government." Under Executive Order 13132, EPA may not issue a regulation that has federalism implications, that imposes substantial direct compliance costs, and that is not required by statute, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by State and local governments, or EPA consults with State and local officials early in the process of developing the proposed regulation. EPA also may not issue a regulation that has federalism implications and that preempts State law unless the Agency consults with State and local officials early in the process of developing the proposed regulation.

If EPA complies by consulting, Executive Order 13132 requires EPA to provide to the Office of Management and Budget (OMB), in a separately identified section of the preamble to the rule, a federalism summary impact statement (FSIS). The FSIS must include a description of the extent of EPA's prior consultation with State and local officials, a summary of the nature of their concerns and the agency's position supporting the need to issue the regulation, and a statement of the extent to which the concerns of State and local officials have been met. Also, when EPA transmits a draft final rule with federalism implications to OMB for review pursuant to Executive Order 12866, EPA must include a certification from the agency's Federalism Official stating that EPA has met the requirements of Executive Order 13132 in a meaningful and timely manner.

This final rule will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. This final rule simply makes corrections and minor revisions to current testing requirements and promulgates a monitoring specification that can be used to support future monitoring rules. Thus, the requirements of section 6 of the Executive Order do not apply to this rule.

G. Executive Order 13084: Consultation and Coordination With Indian Tribal Governments

Under Executive Order 13084, we may not issue a regulation that is not required by statute, that significantly or uniquely affects the communities of Indian tribal governments, and that imposes substantial direct compliance costs on those communities, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by the tribal governments, or we consult with those governments. If we comply by consulting, Executive Order 13094 requires us to provide to the Office of Management and Budget, in a separately identified section of the preamble to the rule, a description of the extent of our prior consultation with representatives of affected tribal governments, a summary of the nature of their concerns, and a statement supporting the need to issue the regulation. In addition, Executive Order 13084 requires us to develop an effective process permitting elected and other representatives of Indian tribal governments "to provide meaningful and timely input in the development of regulatory policies on matters that significantly or uniquely affect their communities." Today's rule does not significantly or uniquely affect the communities of Indian tribal governments. This rule only amends regulatory requirements that are already in effect and adds no additional requirements. Accordingly, the requirements of Section 3(b) of Executive Order 13084 do not apply to this rule.

H. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks

Executive Order 13045: "Protection of Children from Environmental Health Risks and Safety Risks" (62 FR 19885, April 23, 1997) applies to any rule that: (1) Is determined to be "economically significant" as defined under E.O. 12866, and (2) concerns an environmental health or safety risk that we have reason to believe may have a disproportionate effect on children. If the regulatory action meets both criteria, we must evaluate the environmental health or safety effects of the planned rule on children, and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives we considered.

We interpret E.O. 13045 as applying only to those regulatory actions that are based on health or safety risks, such that the analysis required under section 5-501 of the Order has the potential to influence the regulation. This rule is not subject to E.O. 13045 because it does not establish an environmental standard intended to mitigate health or safety risks.

I. Submission to Congress and the General Accounting Office

The Congressional Review Act, 5 U.S.C. 801, *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. We will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States before it is published in the **Federal Register**. This action is not a "major rule" as defined by 5 U.S.C. 804(2). This rule will be effective October 17, 2000.

J. National Technology Transfer and Advancement Act

Section 12(d) of the National Technology Transfer and Advancement Act of 1995 (NTTAA), P.L. 104–113 (15 U.S.C. 272), directs us to use voluntary consensus standards (VCSs) in our regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (*e.g.*, materials specifications, test methods, sampling procedures, business practices, etc.) that are developed or adopted by VCS bodies.

The NTTAA requires us to provide Congress, through OMB, explanations when we decide not to use available and applicable VCSs.

This rulemaking involves technical standards. Specifically, this rule makes technical corrections to portions of the subparts in Parts 60, 61, and 63 pertaining to source testing or monitoring of emissions and operations. The rule does not, however, change the nature of any of the technical standards currently in use. Moreover, many of the technical standards currently in use are VCSs developed by the American Society for Testing and Materials (ASTM). In fact, we have taken the opportunity presented by this rulemaking to update the references to the ASTM standards to include the dates of the most recent versions of these standards (see Section III.A. of the preamble for a full discussion). A complete list of the ASTM standards updated by this rule can be found in Part 60.17. Thus, today's action is consistent with our obligation to use VCSs in our regulatory activities whenever practicable.

Finally, we are promulgating PS–15, which identifies certification criteria for continuous emission monitoring systems (CEMS) using fourier transform infrared spectroscopy (FTIR). PS–15 is a performance specification that is being issued as an example procedure for use by industry and regulatory agencies as appropriate. While there are no underlying national EPA standards that will require the use of this procedure at this time, we conducted a search for VCS FTIR performance specifications and found none. We plan to periodically conduct rulemaking to make minor updates to test methods and performance specifications. In these rulemakings, we will review updates to VCS incorporated by reference and consider VCSs that may be used in lieu of EPA reference methods. We plan to provide the opportunity for public comment during these update rulemakings in part to allow VCS organizations to suggest where VCSs may be available for our use.

K. Plain Language in Government Writing

This rule is not written in the plain language format. In most cases, the rule corrects errors and makes updates to small portions of existing regulations that are not in plain language. The new plain language format was not used to keep the language of the amended sections consistent with that of the unamended rules. Also, the test methods were reformatted and proposed before the plain language provisions were mandated. Due to their volume, the time and costs associated with the magnitude of effort required to rewrite the final methods in plain language is prohibitive. However, this preamble is written in plain language, and we believe the amendments and reformatted test methods have been written clearly.

List of Subjects

40 CFR Part 60

Environmental protection, Administrative practice and procedure, Air pollution control, Continuous emission monitors, Incorporation by reference.

40 CFR Part 61

Environmental protection, Air pollution control, Incorporation by reference.

40 CFR Part 63

Environmental protection, Administrative practice and procedure, Air pollution control, Hazardous substances, Intergovernmental relations, Incorporation by reference, Reporting and recordkeeping requirements.

Dated: January 10, 2000.

Carol M. Browner,

Administrator.

For the reasons stated in the preamble, The Environmental Protection Agency amends title 40, chapter I of the Code of Federal Regulations as follows:

PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

1. The authority citation for part 60 continues to read as follows:

Authority: 42 U.S.C. 7401, 7411, 7413, 7414, 7416, 7601, and 7602.

§ 60.11 [Amended]

2. Amend § 60.11 by:

a. In paragraphs (b) and (e)(1), by revising the words "Reference Method 9" to read "Method 9" wherever they occur;

b. In paragraph (e)(5), revise the words "to determine opacity compliance" in the last sentence to read "to determine compliance with the opacity standard."

§ 60.13 [Amended]

3. Amend § 60.13 by:

a. Revising the last two sentences in paragraph (d)(1), revising paragraph (g), and revising the first sentence in paragraph (j)(2).

b. Revising the words "ng/J of pollutant" to read "ng of pollutant per J of heat input" in the sixth sentence of paragraph (h).

c. Revising the words "with the effluent gases" to read "in the effluent gases" in paragraph (i)(1).

d. Revising the words "effluent from two or more affected facilities are released" to read "effluent from two or more affected facilities is released" in paragraph (i)(9).

e. Revising the words "relative accuracy test" to read "relative accuracy (RA) test" in the paragraph (j) introductory text.

f. Revising the words "relative accuracy" to read "RA" in paragraphs (j)(1) and (2).

g. Revising the section references "section 7" and "section 10" to read "Section 8.4" and "Section 16.0," respectively, in paragraphs (j)(1) and (2).

The revisions read as follows:

§ 60.13 Monitoring requirements.

* * * * *

(d) * * *

(1) * * * For continuous monitoring systems measuring opacity of emissions not using automatic zero adjustments, the optical surfaces exposed to the effluent gases shall be cleaned prior to performing the zero and span drift adjustments. For systems using automatic zero adjustments, the optical surfaces shall be cleaned when the cumulative automatic zero compensation exceeds 4 percent opacity.

* * * * *

(g)(1) When more than one continuous monitoring system is used to measure the emissions from only one affected facility (*e.g.*, multiple breechings, multiple outlets), the owner or operator shall report the results as required from each continuous monitoring system. When the effluent from one affected facility is released to the atmosphere through more than one point, the owner or operator shall install an applicable continuous monitoring system on each separate effluent unless installation of fewer systems is approved by the Administrator.

(2) When the effluents from two or more affected facilities subject to the same opacity standard are combined before being released to the atmosphere, the owner or operator may either install a continuous opacity monitoring system at a location monitoring the combined effluent or install an opacity combiner system comprised of opacity and flow monitoring systems on each stream, and shall report as per § 60.7(c) on the combined effluent. When the affected facilities are not subject to the same opacity standard, the owner or operator shall report the results as per § 60.7(c) on the combined effluent against the most stringent opacity standard

applicable, except for documented periods of shutdown of the affected facility, subject to the most stringent opacity standard. During such times, the next most stringent opacity standard shall apply.

(3) When the effluents from two or more affected facilities subject to the same emissions standard, other than opacity, are combined before being released to the atmosphere, the owner or operator may install applicable continuous emission monitoring systems on each effluent or on the combined effluent. The owner or operator may report the results as required for each affected facility or for the combined effluent. When the affected facilities are not subject to the same emissions standard, separate continuous emission monitoring systems shall be installed on each effluent and the owner or operator shall report as required for each affected facility.

* * * * *

(j) * * *

(2) The waiver of a CEMS RA test will be reviewed and may be rescinded at such time, following successful completion of the alternative RA procedure, that the CEMS data indicate that the source emissions are approaching the level. * * *

* * * * *

§ 60.14 [Amended]

4. In § 60.14, paragraph (b)(1) is amended by revising the words "utilization of emission factors demonstrate" to read "utilization of emission factors demonstrates."

§ 60.17 [Amended]

5. Amend § 60.17 by:

a. Revising paragraphs (a), (i), and (j).

b. In paragraph (b)(1), revise the words "§§ 60.204(d)(2), 60.214(d)(2), 60.224(d)(2), 60.234(d)(2)" to read "§§ 60.204(b)(3), 60.214(b)(3), 60.224(b)(3), 60.234(b)(3)."

c. In paragraph (d), by revising the words "IBR approved January 27, 1983 for § 60.285(d)(4)" to read "IBR approved January 27, 1983 for § 60.285(d)(3)."

The revisions read as follows:

§ 60.17 Incorporation by reference.

* * * * *

(a) The following materials are available for purchase from at least one of the following addresses: American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, PA 19103; or University Microfilms International, 300 North Zeeb Road, Ann Arbor, MI 48106.

(1) ASTM A99–76, 82 (Reapproved 1987), Standard Specification for Ferromanganese, incorporation by reference (IBR) approved January 27, 1983 for § 60.261.

(2) ASTM A100–69, 74, 93, Standard Specification for Ferrosilicon, IBR approved January 27, 1983 for § 60.261.

(3) ASTM A101–73, 93, Standard Specification for Ferrochromium, IBR approved January 27, 1983 for § 60.261.

(4) ASTM A482–76, 93, Standard Specification for Ferrochromesilicon, IBR approved January 27, 1983 for § 60.261.

(5) ASTM A483–64, 74 (Reapproved 1988), Standard Specification for Silicomanganese, IBR approved January 27, 1983 for § 60.261.

(6) ASTM A495–76, 94, Standard Specification for Calcium-Silicon and Calcium Manganese-Silicon, IBR approved January 27, 1983 for § 60.261.

(7) ASTM D86–78, 82, 90, 93, 95, 96, Distillation of Petroleum Products, IBR approved for §§ 60.562–2(d), 60.593(d), and 60.633(h).

(8) ASTM D129–64, 78, 95, Standard Test Method for Sulfur in Petroleum Products (General Bomb Method), IBR approved for Appendix A: Method 19, Section 12.5.2.2.3; and § 60.106(j)(2).

(9) ASTM D240–76, 92, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter, IBR approved January 27, 1983 for §§ 60.46(c), 60.296(b), and Appendix A: Method 19, Section 12.5.2.2.3.

(10) ASTM D270–65, 75, Standard Method of Sampling Petroleum and Petroleum Products, IBR approved January 27, 1983 for Appendix A: Method 19, Section 12.5.2.2.1.

(11) ASTM D323–82, 94, Test Method for Vapor Pressure of Petroleum Products (Reid Method), IBR approved April 8, 1987 for §§ 60.111(l), 60.111a(g), 60.111b(g), and 60.116b(f)(2)(ii).

(12) ASTM D388–77, 90, 91, 95, 98, 98a, Standard Specification for Classification of Coals by Rank, IBR approved for §§ 60.41(f), 60.45(f)(4)(i), 60.45(f)(4)(ii), 60.45(f)(4)(vi), 60.41a, 60.41b, and 60.251(b) and (c).

(13) ASTM D396–78, 89, 90, 92, 95, 96, 97, 98, Standard Specification for Fuel Oils, IBR approved for §§ 60.41b, 60.41c, 60.111(b), and 60.111a(b).

(14) ASTM D975–78, 96, 98, 98a, Standard Specification for Diesel Fuel Oils, IBR approved January 27, 1983 for §§ 60.111(b) and 60.111a(b).

(15) ASTM D1072–80, 90 (Reapproved 1994), Standard Method for Total Sulfur in Fuel Gases, IBR approved July 31, 1984 for § 60.335(d).

(16) ASTM D1137–53, 75, Standard Method for Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer, IBR approved January 27, 1983 for § 60.45(f)(5)(i).

(17) ASTM D1193–77, 91, Standard Specification for Reagent Water, IBR approved for Appendix A: Method 5, Section 7.1.3; Method 5E, Section 7.2.1; Method 5F, Section 7.2.1; Method 6, Section 7.1.1; Method 7, Section 7.1.1; Method 7C, Section 7.1.1; Method 7D, Section 7.1.1; Method 10A, Section 7.1.1; Method 11, Section 7.1.3; Method 12, Section 7.1.3; Method 13A, Section 7.1.2; Method 26, Section 7.1.2; Method 26A, Section 7.1.2; and Method 29, Section 7.2.2.

(18) ASTM D1266–87, 91, 98, Standard Test Method for Sulfur in Petroleum Products (Lamp Method), IBR approved August 17, 1989 for § 60.106(j)(2).

(19) ASTM D1475–60, 80, 90, Standard Test Method for Density of Paint, Varnish Lacquer, and Related Products, IBR approved January 27, 1983 for § 60.435(d)(1), Appendix A: Method 24, Section 6.1; and Method 24A, Sections 6.5 and 7.1.

(20) ASTM D1552–83, 95, Standard Test Method for Sulfur in Petroleum Products (High Temperature Method), IBR approved for Appendix A: Method 19, Section 12.5.2.2.3; and § 60.106(j)(2).

(21) ASTM D1826–77, 94, Standard Test Method for Calorific Value of Gases in Natural Gas Range by Continuous Recording Calorimeter, IBR approved January 27, 1983 for §§ 60.45(f)(5)(ii), 60.46(c)(2), 60.296(b)(3), and Appendix A: Method 19, Section 12.3.2.4.

(22) ASTM D1835–82, 86, 87, 91, 97, Standard Specification for Liquefied Petroleum (LP) Gases, approved for §§ 60.41b and 60.41c.

(23) ASTM D1945–64, 76, 91, 96, Standard Method for Analysis of Natural Gas by Gas Chromatography, IBR approved January 27, 1983 for § 60.45(f)(5)(i).

(24) ASTM D1946–77, 90 (Reapproved 1994), Standard Method for Analysis of Reformed Gas by Gas Chromatography, IBR approved for §§ 60.45(f)(5)(i), 60.18(f)(3), 60.614(e)(2)(ii), 60.614(e)(4), 60.664(e)(2)(ii), 60.664(e)(4), 60.564(f)(1), 60.704(d)(2)(ii), and 60.704(d)(4).

(25) ASTM D2013–72, 86, Standard Method of Preparing Coal Samples for Analysis, IBR approved January 27, 1983, for Appendix A: Method 19, Section 12.5.2.1.3.

(26) ASTM D2015–77 (Reapproved 1978), 96, Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter, IBR

approved January 27, 1983 for § 60.45(f)(5)(ii), 60.46(c)(2), and Appendix A: Method 19, Section 12.5.2.1.3.

(27) ASTM D2016–74, 83, Standard Test Methods for Moisture Content of Wood, IBR approved for Appendix A: Method 28, Section 16.1.1.

(28) ASTM D2234–76, 96, 97a, 97b, 98, Standard Methods for Collection of a Gross Sample of Coal, IBR approved January 27, 1983 for Appendix A: Method 19, Section 12.5.2.1.1.

(29) ASTM D2369–81, 87, 90, 92, 93, 95, Standard Test Method for Volatile Content of Coatings, IBR approved January 27, 1983 for Appendix A: Method 24, Section 6.2.

(30) ASTM D2382–76, 88, Heat of Combustion of Hydrocarbon Fuels by Bomb Calorimeter (High-Precision Method), IBR approved for §§ 60.18(f)(3), 60.485(g)(6), 60.614(e)(4), 60.664(e)(4), 60.564(f)(3), and 60.704(d)(4).

(31) ASTM D2504–67, 77, 88 (Reapproved 1993), Noncondensable Gases in C_3 and Lighter Hydrocarbon Products by Gas Chromatography, IBR approved for § 60.485(g)(5).

(32) ASTM D2584–68 (Reapproved 1985), 94, Standard Test Method for Ignition Loss of Cured Reinforced Resins, IBR approved February 25, 1985 for § 60.685(c)(3)(i).

(33) ASTM D2622–87, 94, 98, Standard Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry, IBR approved August 17, 1989 for § 60.106(j)(2).

(34) ASTM D2879–83, 96, 97, Test Method for Vapor Pressure-Temperature Relationship and Initial Decomposition Temperature of Liquids by Isoteniscope, IBR approved April 8, 1987 for §§ 60.485(e)(1), 60.111b(f)(3), 60.116b(e)(3)(ii), and 60.116b(f)(2)(i).

(35) ASTM D2880–78, 96, Standard Specification for Gas Turbine Fuel Oils, IBR approved January 27, 1983 for §§ 60.111(b), 60.111a(b), and 60.335(d).

(36) ASTM D2908–74, 91, Standard Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography, IBR approved for § 60.564(j).

(37) ASTM D2986–71, 78, 95a, Standard Method for Evaluation of Air, Assay Media by the Monodisperse DOP (Dioctyl Phthalate) Smoke Test, IBR approved January 27, 1983 for Appendix A: Method 5, Section 7.1.1; Method 12, Section 7.1.1; and Method 13A, Section 7.1.1.2.

(38) ASTM D3031–81, Standard Test Method for Total Sulfur in Natural Gas by Hydrogenation, IBR approved July 31, 1984 for § 60.335(d).

(39) ASTM D3173–73, 87, Standard Test Method for Moisture in the Analysis Sample of Coal and Coke, IBR approved January 27, 1983 for Appendix A: Method 19, Section 12.5.2.1.3.

(40) ASTM D3176–74, 89, Standard Method for Ultimate Analysis of Coal and Coke, IBR approved January 27, 1983 for § 60.45(f)(5)(i) and Appendix A: Method 19, Section 12.3.2.3.

(41) ASTM D3177–75, 89, Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke, IBR approved January 27, 1983 for Appendix A: Method 19, Section 12.5.2.1.3.

(42) ASTM D3178–73 (Reapproved 1979), 89, Standard Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke, IBR approved January 27, 1983 for § 60.45(f)(5)(i).

(43) ASTM D3246–81, 92, 96, Standard Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry, IBR approved July 31, 1984 for § 60.335(d).

(44) ASTM D3270–73T, 80, 91, 95, Standard Test Methods for Analysis for Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method), IBR approved for Appendix A: Method 13A, Section 16.1.

(45) ASTM D3286–85, 96, Standard Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter, IBR approved for Appendix A: Method 19, Section 12.5.2.1.3.

(46) ASTM D3370–76, 95a, Standard Practices for Sampling Water, IBR approved for § 60.564(j).

(47) ASTM D3792–79, 91, Standard Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph, IBR approved January 27, 1983 for Appendix A: Method 24, Section 6.3.

(48) ASTM D4017–81, 90, 96a, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method, IBR approved January 27, 1983 for Appendix A: Method 24, Section 6.4.

(49) ASTM D4057–81, 95, Standard Practice for Manual Sampling of Petroleum and Petroleum Products, IBR approved for Appendix A: Method 19, Section 12.5.2.2.3.

(50) ASTM D4084–82, 94, Standard Method for Analysis of Hydrogen Sulfide in Gaseous Fuels (Lead Acetate Reaction Rate Method), IBR approved July 31, 1984 for § 60.335(d).

(51) ASTM D4177–95, Standard Practice for Automatic Sampling of Petroleum and Petroleum Products, IBR approved for Appendix A: Method 19, 12.5.2.2.1.

(52) ASTM D4239–85, 94, 97, Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods, IBR approved for Appendix A: Method 19, Section 12.5.2.1.3.

(53) ASTM D4442–84, 92, Standard Test Methods for Direct Moisture Content Measurement in Wood and Wood-base Materials, IBR approved for Appendix A: Method 28, Section 16.1.1.

(54) ASTM D4444–92, Standard Test Methods for Use and Calibration of Hand-Held Moisture Meters, IBR approved for Appendix A: Method 28, Section 16.1.1.

(55) ASTM D4457–85 (Reapproved 1991), Test Method for Determination of Dichloromethane and 1, 1, 1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph, IBR approved for Appendix A: Method 24, Section 6.5.

(56) ASTM D4809–95, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), IBR approved for §§ 60.18(f)(3), 60.485(g)(6), 60.564(f)(3), 60.614(d)(4), 60.664(e)(4), and 60.704(d)(4).

(57) ASTM D5403–93, Standard Test Methods for Volatile Content of Radiation Curable Materials. IBR approved September 11, 1995 for Appendix A: Method 24, Section 6.6.

(58) ASTM D5865–98, Standard Test Method for Gross Calorific Value of Coal and Coke. IBR approved for § 60.45(f)(5)(ii), 60.46(c)(2), and Appendix A: Method 19, Section 12.5.2.1.3.

(59) ASTM E168–67, 77, 92, General Techniques of Infrared Quantitative Analysis, IBR approved for §§ 60.593(b)(2) and 60.632(f).

(60) ASTM E169–63, 77, 93, General Techniques of Ultraviolet Quantitative Analysis, IBR approved for §§ 60.593(b)(2) and 60.632(f).

(61) ASTM E260–73, 91, 96, General Gas Chromatography Procedures, IBR approved for §§ 60.593(b)(2) and 60.632(f).

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(i) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW–846 Third Edition (November 1986), as amended by Updates I (July 1992), II (September 1994), IIA (August, 1993), IIB (January 1995), and III (December 1996). This document may be obtained from the U.S. EPA, Office of Solid Waste and Emergency Response, Waste Characterization Branch, Washington, DC 20460, and is incorporated by reference for Appendix A to Part 60, Method 29, Sections 7.5.34; 9.2.1; 9.2.3; 10.2; 10.3; 11.1.1; 11.1.3; 13.2.1; 13.2.2; 13.3.1; and Table 29–3.

(j) "Standard Methods for the Examination of Water and Wastewater," 16th edition, 1985. Method 303F: "Determination of Mercury by the Cold Vapor Technique." This document may be obtained from the American Public Health Association, 1015 18th Street, NW., Washington, DC 20036, and is incorporated by reference for Appendix A to Part 60, Method 29, Sections 9.2.3; 10.3; and 11.1.3.

* * * * *

§ 60.18 [Amended]

6. Amend § 60.18 as follows:

a. In paragraph (f)(1), the first sentence is amended by revising "Reference Method 22" to read "Method 22 of Appendix A to this part."

b. In paragraph (f)(3), the definition of "C_i" is amended by revising "ASTM D1946–77" to read "ASTM D1946–77 or 90 (Reapproved 1994)."

c. In paragraph (f)(3), the definition of "H_i" is amended by revising "ASTM D2382–76" to read "ASTM D2382–76 or 88 or D4809–95."

§ 60.41 [Amended]

7. In § 60.41, paragraph (f) is amended by revising the words "the American Society and Testing and Materials, Designation D388–77" to read "ASTM D388–77, 90, 91, 95, or 98a."

§ 60.42 [Amended]

8. In § 60.42, paragraphs (b)(1) and (b)(2), are amended by removing the symbol "%" wherever it appears, and adding "percent" in its place.

§ 60.45 [Amended]

9. Amend § 60.45 as follows:

a. In paragraph (b)(2) by removing the words "under paragraph (d) of this section."

b. In paragraphs (f)(4)(i), (f)(4)(ii), and (f)(4)(vi) by revising the words "ASTM D388–77" to read "ASTM D388–77, 90, 91, 95, or 98a."

c. In paragraph (f)(5)(i) by revising the words "ASTM method D1137–53, (75), D1945–64(76), or D1946–77" to read "ASTM D1137–53 or 75, D1945–64, 76, 91, or 96 or D1946–77 or 90 (Reapproved 1994)."

d. In paragraph (f)(5)(i) by revising the words "ASTM method D3178–74 or D3176" to read "ASTM D3178–73 (Reapproved 1979), 89, or D3176–74 or 89."

e. In paragraph (f)(5)(ii) by revising the words "ASTM D1826–77" to read "ASTM D1826–77 or 94."

f. In paragraph (f)(5)(ii) by revising the words "ASTM D2015–77" to read "ASTM D2015–77 (Reapproved 1978), 96, or D5865–98."

§ 60.46 [Amended]

10. Amend § 60.46 as follows:

a. In paragraph (b)(2)(i), the second sentence is amended by revising the words "in the sampling train may be set to provide a gas temperature no greater than" to read "in the sampling train shall be set to provide an average gas temperature of."

b. In paragraph (b)(2)(ii), the third sentence is amended by revising the words "the arithmetic mean of all the individual O_2 sample concentrations at each traverse point" to read "the arithmetic mean of the sample O_2 concentrations at all traverse points."

c. Paragraph (c)(2) is amended by revising the words "D2015–77" to read "D2015–77 (Reapproved 1978), 96, or D5865–98".

d. Paragraph (c)(2) is further amended by revising the words "D240–76" to read "D240–76 or 92."

e. In paragraph (c)(2) is further amended by revising the words "D1826–77" to read "D1826–77 or 94."

§ 60.41a [Amended]

11. Amend § 60.41a as follows:

a. In the definitions for "subbituminous coal" and "lignite," by revising "D388–77" to read "D388–77, 90, 91, 95, or 98a."

b. In paragraph (a)(2) of the definition of "potential combustion concentration" by revising "75 ng/J" to read "73 ng/J."

§ 60.43a [Amended]

12. In § 60.43a, paragraph (d)(2), revising the words "resource recovery facility" to read "resource recovery unit."

§ 60.47a [Amended]

13. Amend § 60.47a as follows:

a. In paragraph (b)(3) by removing the words "(appendix A)."

b. In the first sentence of paragraph (g) by revising the words "lbs/million Btu" to read "lb/million Btu."

c. In the second sentence of paragraph (h)(3) by revising the words "309 minutes in each hour" to read "30 minutes in each hour."

d. In paragraph (i)(1) by revising the words "6, 7, and 3B, as applicable, shall be used to determine O_2, SO_2, and NO_X concentrations" to read "3B, 6, and 7 shall be used to determine O_2, SO_2, and NO_X concentrations, respectively."

§ 60.48a [Amended]

14. Amend § 60.48a as follows:

a. In paragraph (b)(2)(ii), in the fourth sentence by revising the words "the arithmetic mean of all the individual O_2 concentrations at each traverse point." to read "the arithmetic mean of the sample O_2 concentrations at all traverse points."

b. In paragraph (c)(3), in the first sentence by adding a closing parenthesis after the abbreviation "(%R_g" so that it now reads "(%R_g)".

c. In paragraph (f), in the first and second sentences by removing the words "(appendix A)."

§ 60.40b [Amended]

15. § 60.40b is amended by adding paragraph (j) as follows:

§ 60.40b Applicability and delegation of authority.

* * * * *

(j) Any affected facility meeting the applicability requirements under paragraph (a) of this section and commencing construction, modification, or reconstruction after June 19, 1986 is not subject to Subpart D (Standards of Performance for Fossil-Fuel-Fired Steam Generators, § 60.40).

* * * * *

§ 60.41b [Amended]

16. Amend § 60.41b as follows:

a. In the definition for "coal" by revising "ASTM D388–77" to read "ASTM D388–77, 90, 91, 95, or 98a."

b. In the definition for "distillate oil" by revising "ASTM D396–78" to read "ASTM D396–78, 89, 90, 92, 96, or 98."

c. In the definition for "lignite" by revising "ASTM D388–77" to read "ASTM D388–77, 90, 91, 95, or 98a."

d. In the definition for "natural gas" by revising "ASTM D1835–82" to read "ASTM D1835–82, 86, 87, 91, or 97."

§ 60.42b [Amended]

17. In § 60.42b, paragraph (d), the second sentence is amended by revising the words "facilities under this paragraph" to read "facilities under paragraphs (d)(1), (2), or (3)."

§ 60.43b [Amended]

18. In § 60.43b, paragraph (a)(1) is amended by revising the words "22 ng/J (0.05 lb/million Btu)" to read "22 ng/J (0.051 lb/million Btu)."

§ 60.46b [Amended]

19. Amend § 60.46b as follows:

a. In paragraph (d)(4) by revising the words "160 °C (320 °F)" to read "160±14 °C (320±25 °F)."

b. In paragraph (d)(6)(iii) by removing the words "(appendix A)."

§ 60.41c [Amended]

20. Amend § 60.41c as follows:

a. In the definition for "natural gas" by revising "D1835–86" to read "D1835–86, 87, 91, or 97."

b. In the definitions for "distillate oil" and "residual oil" by revising "D396–78" to read "D396–78, 89, 90, 92, 96, or 98."

§ 60.42c [Amended]

21. Amend § 60.42c as follows:

a. In paragraph (a), in the first sentence by revising the words "the owner the operator" to read "the owner or operator."

b. In paragraph (c), in the second sentence by revising the words "facilities under this paragraph" to read "facilities under paragraphs (c)(1), (2), (3), or (4)."

§ 60.43c [Amended]

22. In § 60.43c, paragraph (a)(1) is amended by revising the words "22 ng/J (0.05 lb/million Btu)" to read "22 ng/J (0.051 lb/million Btu)."

§ 60.44c [Amended]

23. In § 60.44c, paragraph (i), the third sentence is amended by revising the words "24-hour averaged" to read "24-hour average."

§ 60.45c [Amended]

24. Amend § 60.45c as follows:

a. Redesignate paragraphs (a)(5) through (a)(7) as paragraphs (a)(6) through (a)(8), respectively.

b. Revise paragraphs (a)(1) through (a)(4) and add paragraph (a)(5).

The redesignation, revisions and addition read as follows:

§ 60.45c Compliance and performance test methods and procedures for particulate matter.

(a) * * *

(1) Method 1 shall be used to select the sampling site and the number of traverse sampling points.

(2) Method 3 shall be used for gas analysis when applying Method 5, Method 5B, or Method 17.

(3) Method 5, Method 5B, or Method 17 shall be used to measure the concentration of PM as follows:

(i) Method 5 may be used only at affected facilities without wet scrubber systems.

(ii) Method 17 may be used at affected facilities with or without wet scrubber systems provided the stack gas temperature does not exceed a temperature of 160 °C (320 °F). The procedures of Sections 8.1 and 11.1 of Method 5B may be used in Method 17 only if Method 17 is used in conjunction with a wet scrubber system. Method 17 shall not be used in conjunction with a wet scrubber system if the effluent is saturated or laden with water droplets.

(iii) Method 5B may be used in conjunction with a wet scrubber system.

(4) The sampling time for each run shall be at least 120 minutes and the minimum sampling volume shall be 1.7 dry standard cubic meters (dscm) [60 dry standard cubic feet (dscf)] except that smaller sampling times or volumes may be approved by the Administrator when necessitated by process variables or other factors.

(5) For Method 5 or Method 5B, the temperature of the sample gas in the probe and filter holder shall be monitored and maintained at 160±14 °C (320±25 °F).

* * * * *

§ 60.46c [Amended]

25. In § 60.46c, paragraphs (b) and (d) are amended by revising the abbreviation "CEM" to read "CEMS" wherever it appears.

§ 60.47c [Amended]

26. In § 60.47c, paragraphs (a) and (b) are amended by revising the abbreviation "CEMS" to read "COMS" wherever it appears.

§ 60.48c [Amended]

27. In § 60.48c, paragraph (b) is amended by replacing the abbreviation "CEMS" with the words "CEMS and/or COMS."

§ 60.52 [Amended]

28. In § 60.52, paragraph (a) is amended by revising the words "the performance test required to be conducted by § 60.8 is completed" to read "the initial performance test is completed or required to be completed under § 60.8 of this part, whichever date comes first."

§ 60.54 [Amended]

29. Amend § 60.54 as follows:

a. In paragraph (b)(1) by revising the words "The emission rate (c_{12})" to read "The concentration (c_{12})."

b. In paragraph (b)(3)(i), in the third sentence by revising the words "the arithmetic mean of all the individual CO_2 sample concentrations at each traverse point" to read "the arithmetic mean of the sample CO_2 concentrations at all traverse points."

§ 60.51a [Amended]

30. Section 60.51a is amended by adding a new difinition in alphabetical order to read as follows:

§ 60.51a Definitions.

* * * * *

Continuous monitoring system means the total equipment used to sample and condition (if applicable), to analyze, and to provide a permanent record of emissions or process parameters.

* * * * *

§ 60.58a [Amended]

31. Amend § 60.58a as follows:

a. In paragraph (b)(3), in the first sentence by revising the words "particulate matter emission standard" to read "particulate matter emission limit."

b. In paragraph (b)(3), in the third sentence by revising the words "a gas temperature no greater than" to read "a gas temperature of."

c. In paragraph (b)(8) by revising the words "operate a CEMS for measuring opacity" to read "operate a continuous opacity monitoring system (COMS)."

d. In paragraph (e)(10) by revising the word "Section" to read "section."

e. In paragraph (e)(14) by revising the words "outlet to" to read "outlet of."

f. In paragraph (f)(2) by revising the words "Method 26" to read "Method 26 or 26A."

§ 60.58b [Amended]

32–36. Amend § 60.58b as follows:

a. In paragraph (b)(1) by revising the words "(or carbon dioxide)" to read "(or 20 percent carbon dioxide)" each place it appears.

b. In paragraph (f)(1), in the second sentence by removing the words "for Method 26."

c. In paragraph (f)(2) by removing the words "Method 26."

§ 60.56c [Amended]

37. Amend § 60.56c as follows:

a. In paragraph (b)(4), in the first and second sentences by revising the words "Method 3 or 3A" to read "Method 3, 3A, or 3B."

b. In paragraph (b)(10), in the first sentence by revising the words "Method 26" to read "Method 26 or 26A."

§ 60.64 [Amended]

38. Amend § 60.64(b)(1) as follows:

a. In the definition of the term "c_s", "(g/dscf)" is revised to read "(gr/dscf)."

b. In the definition of the term "K", "(453.6 g/lb)" is revised to read "(7000 gr/lb)."

§ 60.84 [Amended]

39. Amend § 60.84 as follows:

a. In paragraph (d), in the third sentence by revising the words "monitoring of" to read "monitoring systems for measuring."

b. In paragraph (d), in the fourth sentence by revising the words "this SO_2" to read "the SO_2."

§ 60.102 [Amended]

40. In § 60.102, paragraph (a)(1) is amended by revising the words "1.0 kg/

1000 kg (1.0 lb/1000 lb)" to read "1.0 kg/Mg (2.0 lb/ton).

§ 60.104 [Amended]

41. In § 60.104, paragraph (b)(2) is amended by revising the words "9.8 kg/1,000 kg" to read "9.8 kg/Mg (20 lb/ton)."

§ 60.105 [Amended]

42. Amend § 60.105 by:

a. In paragraphs (a)(3)(iii) and (a)(5)(ii), the words "Methods 6 and 3" in the second sentence are revised to read "Methods 6 or 6C and 3 or 3A."

b. In paragraph (a)(4)(iii), the words "Method 11 shall be used for conducting the relative accuracy evaluations" are revised to read "Method 11, 15, 15A, or 16 shall be used for conducting the relative accuracy evaluations."

c. In paragraphs (a)(3)(i), (a)(5)(i), (a)(6)(i), and (a)(7)(i), "10" is revised to read "25."

d. In paragraph (a)(6)(ii), the first sentence and paragraphs (a)(8), (a)(9), and (a)(12) are revised.

e. In paragraph (a)(10), the abbreviation "vppm" is revised to read "ppmv".

f. In paragraph (c), "(thousands of kilograms per hour)" is revised to read "(Mg (tons) per hour)."

g. In paragraph (d), the words "(liters/hr or kg/hr)" are removed.

The revisions read as follows:

§ 60.105 Monitoring of emissions and operations.

(a) * * *

(6) * * *

(ii) The performance evaluations for this reduced sulfur (and O_2) monitor under § 60.13(c) shall use Performance Specification 5 of Appendix B of this Part (and Performance Specification 3 of Appendix B of this Part for the O_2 analyzer). * * *

* * * * *

(8) An instrument for continuously monitoring and recording concentrations of SO_2 in the gases at both the inlet and outlet of the SO_2 control device from any fluid catalytic cracking unit catalyst regenerator for which the owner or operator seeks to comply with § 60.104 (b)(1).

(i) The span value of the inlet monitor shall be set 125 percent of the maximum estimated hourly potential SO_2 emission concentration entering the control device, and the span value of the outlet monitor shall be set at 50 percent of the maximum estimated hourly potential sulfur dioxide emission concentration entering the control device.

(ii) The performance evaluations for these SO_2 monitors under § 60.13(c) shall use Performance Specification 2. Methods 6 or 6C and 3 or 3A shall be used for conducting the relative accuracy evaluations.

(9) An instrument for continuously monitoring and recording concentrations of SO_2 in the gases discharged into the atmosphere from any fluid catalytic cracking unit catalyst regenerator for which the owner or operator seeks to comply specifically with the 50 ppmv emission limit under § 60.104 (b)(1).

(i) The span value of the monitor shall be set at 50 percent of the maximum hourly potential SO_2 emission concentration of the control device.

(ii) The performance evaluations for this SO_2 monitor under § 60.13 (c) shall use Performance Specification 2. Methods 6 or 6C and 3 or 3A shall be used for conducting the relative accuracy evaluations.

* * * * *

(12) The owner or operator shall use the following procedures to evaluate the continuous monitoring systems under paragraphs (a)(8), (a)(9), and (a)(10) of this section.

(i) Method 3 or 3A and Method 6 or 6C for the relative accuracy evaluations under the § 60.13(e) performance evaluation.

(ii) Appendix F, Procedure 1, including quarterly accuracy determinations and daily calibration drift tests.

* * * * *

§ 60.106 [Amended]

43. Amend § 60.106 by:

a. In paragraphs (b)(1), (b)(3), (c)(1), (i)(9) by revising the equations and definitions.

b. In paragraph (b)(3)(ii) by revising the words "Method 3" to read "Method 3B."

c. Revising paragraph (e).

d. Revising paragraph (f)(1).

e. In paragraph (f)(3) by revising the words "Method 3" to read "Method 3 or 3A" and by revising "(h)(3)" to read "(h)(6)."

d. In paragraph (g), in the first sentence by revising the words "the applicable test methods and procedures specified in this section" to read "Method 6 or 6C and Method 3 or 3A."

e. In paragraphs (h)(1), (h)(3), and (h)(4) by revising the abbreviation "vppm" to read "ppmv" wherever it occurs.

f. In paragraph (i)(2)(i) by revising the words "for the concentration of sulfur oxides calculated as sulfur dioxide and moisture content" to read "for moisture content and for the concentration of sulfur oxides calculated as sulfur dioxide."

g. Revising paragraph (i)(9) following the introductory text and paragraph (i)(10).

h. In paragraph (i)(11) by revising the words "per 1,000 kg of coke burn-off" to read "per Mg (ton) of coke burn-off."

i. In paragraph (j)(2) by revising the words "ASTM D129–64 (Reapproved 1978)" to read "ASTM D129–64, 78, or 95."

j. In paragraph (j)(2) by revising the words "ASTM D1552–83" to read "ASTM D1552–83 or 95."

k. In paragraph (j)(2) by revising the words "ASTM D2622–87" to read "ASTM D2622–87, 94, or 98."

l. In paragraph (j)(2) by revising the words "ASTM D1266–87" to read "ASTM D1266–87, 91, or 98."

The revisions read as follows:

§ 60.106 Test methods and procedures.

* * * * *

(b) * * *

(1) * * *

$$E = \frac{c_s Q_{sd}}{K R_c}$$

Where:

E = Emission rate of PM, kg/Mg (lb/ton) of coke burn-off.

c_s = Concentration of PM, g/dscm (gr/dscf).

Q_{sd} = Volumetric flow rate of effluent gas, dscm/hr (dscf/hr).

R_c = Coke burn-off rate, Mg/hr (ton/hr) coke.

K=Conversion factor, 1,000 g/kg (7,000 gr/lb).

* * * * *

(3) * * *

$$R_c=K_1Q_r(\%CO_2+\%CO)-(K_2Q_a-K_3Q_r)((\%CO/2)+(\%CO_2+\%O_2))$$

Where:

R_c = Coke burn-off rate, Mg/hr (ton/hr).

Q_r = Volumetric flow rate of exhaust gas from catalyst regenerator before entering the emission control system, dscm/min (dscf/min).

Q_a = Volumetric flow rate of air to FCCU regenerator, as determined from the fluid catalytic cracking unit control room instrumentation, dscm/min (dscf/min).
$\%CO_2$ = Carbon dioxide concentration, percent by volume (dry basis).
%CO = Carbon monoxide concentration, percent by volume (dry basis).
$\%O_2$ = Oxygen concentration, percent by volume (dry basis).
K_1 = Material balance and conversion factor, 2.982×10^{-4} (Mg-min)/(hr-dscm-%) [9.31×10^{-6} (ton-min)/(hr-dscf-%)].
K_2 = Material balance and conversion factor, 2.088×10^{-3} (Mg-min)/(hr-dscm-%) [6.52×10^{-5} (ton-min)/(hr-dscf-%)].
K_3 = Material balance and conversion factor, 9.94×10^{-5} (Mg-min)/(hr-dscm-%) [3.1×10^{-6} (ton-min)/(hr-dscf-%)].

* * * * *

(c) * * *

(1) * * *

$$E_s = F + A\left(H/R_c\right)$$

Where:

E_s = Emission rate of PM allowed, kg/Mg (lb/ton) of coke burn-off in catalyst regenerator.
F=Emission standard, 1.0 kg/Mg (2.0 lb/ton) of coke burn-off in catalyst regenerator.
A = Allowable incremental rate of PM emissions, 7.5×10^{-4} kg/million J (0.10 lb/million Btu).
H = Heat input rate from solid or liquid fossil fuel, million J/hr (million Btu/hr).
R_c = Coke burn-off rate, Mg coke/hr (ton coke/hr).

* * * * *

(e)(1) The owner or operator shall determine compliance with the H_2S standard in § 60.104(a)(1) as follows: Method 11, 15, 15A, or 16 shall be used to determine the H_2S concentration. The gases entering the sampling train should be at about atmospheric pressure. If the pressure in the refinery fuel gas lines is relatively high, a flow control valve may be used to reduce the pressure. If the line pressure is high enough to operate the sampling train without a vacuum pump, the pump may be eliminated from the sampling train. The sample shall be drawn from a point near the centroid of the fuel gas line.

(i) For Method 11, the sampling time and sample volume shall be at least 10 minutes and 0.010 dscm (0.35 dscf). Two samples of equal sampling times shall be taken at about 1-hour intervals. The arithmetic average of these two samples shall constitute a run. For most fuel gases, sampling times exceeding 20 minutes may result in depletion of the collection solution, although fuel gases containing low concentrations of H_2S may necessitate sampling for longer periods of time.

(ii) For Method 15 or 16, at least three injects over a 1-hour period shall constitute a run.

(iii) For Method 15A, a 1-hour sample shall constitute a run.

(2) Where emissions are monitored by § 60.105(a)(3), compliance with § 60.105(a)(1) shall be determined using Method 6 or 6C and Method 3 or 3A. A 1-hour sample shall constitute a run. Method 6 samples shall be taken at a rate of approximately 2 liters/min. The ppm correction factor (Method 6) and the sampling location in paragraph (f)(1) of this section apply. Method 4 shall be used to determine the moisture content of the gases. The sampling point for Method 4 shall be adjacent to the sampling point for Method 6 or 6C.

(f) * * *

(1) Method 6 shall be used to determine the SO_2 concentration. The concentration in mg/dscm obtained by Method 6 or 6C is multiplied by 0.3754 to obtain the concentration in ppm. The sampling point in the duct shall be the centroid of the cross section if the cross-sectional area is less than 5.00 m^2 (53.8 ft^2) or at a point no closer to the walls than 1.00 m (39.4 in.) if the cross-sectional area is 5.00 m^2 or more and the centroid is more than 1 m from the wall. The sampling time and sample volume shall be at least 10 minutes and 0.010 dscm (0.35 dscf) for each sample. Eight samples of equal sampling times shall be taken at about 30-minute intervals. The arithmetic average of these eight samples shall constitute a run. For Method 6C, a run shall consist of the arithmetic average of four 1-hour samples. Method 4 shall be used to determine the moisture content of the gases. The sampling point for Method 4 shall be adjacent to the sampling point for Method 6 or 6C. The sampling time for each sample shall be equal to the time it takes for two Method 6 samples. The moisture content from this sample shall be used to correct the corresponding Method 6 samples for moisture. For documenting the oxidation efficiency of the control device for reduced sulfur compounds, Method 15 shall be used following the procedures of paragraph (f)(2) of this section.

* * * * *

(i) * * *

(9) * * *

$$E_{SO_x} = C_{SO_x} Q_{sd}/K$$

Where:

E_{SO_x} = sulfur oxides emission rate calculated as sulfur dioxide, kg/hr (lb/hr)
C_{SO_x} = sulfur oxides emission concentration calculated as sulfur dioxide, g/dscm (gr/dscf)
Q_{sd} = dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr)
K=1,000 g/kg (7,000 gr/lb)

(10) Sulfur oxides emissions calculated as sulfur dioxide shall be determined for each test run by the following equation:

$$R_{SO_x} = \left(E_{SO_x}/R_c\right)$$

Where:

R_{SO_x} = Sulfur oxides emissions calculated as kg sulfur dioxide per Mg (lb/ton) coke burn-off.
E_{SO_x} = Sulfur oxides emission rate calculated as sulfur dioxide, kg/hr (lb/hr).
R_c = Coke burn-off rate, Mg/hr (ton/hr).

* * * * *

§ 60.107 [Amended]

44. Section 60.107 is amended by revising paragraphs (c)(5) and (c)(6) as follows:

§ 60.107 Reporting and recordkeeping requirements.

* * * * *

(c) * * *

(5) If subject to § 60.104(b)(2), for each day in which a Method 8 sample result required by § 60.106(i) was not obtained, the date for which and brief explanation as to why a Method 8 sample result was not obtained, for approval by the Administrator.

(6) If subject to § 60.104(b)(3), for each 8-hour period in which a feed sulfur measurement required by § 60.106(j) was not obtained, the date for which and brief explanation as to why a feed sulfur measurement was not obtained, for approval by the Administrator.

* * * * *

§ 60.111 [Amended]

45. Section 60.111 is amended as follows:

a. In paragraph (b) by revising "ASTM D396–78" to read "ASTM D396–78, 89, 90, 92, 96, or 98."

b. In paragraph (b) by revising "ASTM D2880–78" to read "ASTM D2880–78 or 96."

c. In paragraph (b) by revising "ASTM D975–78" to read "ASTM D975–78, 96, or 98a."

d. In paragraph (l) by revising "ASTM D323–82" to read "ASTM D323–82 or 94."

§ 60.111a [Amended]

46. Section 60.111a is amended as follows:

a. In paragraph (b) by revising "ASTM D396–78" to read "D396–78, 89, 90, 92, 96, or 98."

b. In paragraph (b) by revising "ASTM D2880–78" to read "ASTM D2880–78 or 96"; and by revising "ASTM D975–78" to read "ASTM D975–78, 96, or 98a."

c. In paragraph (g) by revising "ASTM D323–82" to read "ASTM D323–82 or 94."

§ 60.111b [Amended]

47. Section 60.111b is amended as follows:

a. In paragraph (f)(3) by revising "ASTM Method D2879–83" to read "ASTM D2879–83, 96, or 97."

b. In paragraph (g) by revising "ASTM D323–82" to read "ASTM D323–82 or 94."

§ 60.116b [Amended]

48. Section 60.116b is amended as follows:

a. In paragraph (e)(3)(ii) by revising "ASTM Method D2879–83" to read "ASTM D2879–83, 96, or 97."

b. In paragraph (f)(2)(i) by revising "ASTM Method D2879–83" to read "ASTM D2879–83, 96, or 97."

c. In paragraph (f)(2)(ii) by revising "ASTM Method D323–82" to read "ASTM D323–82 or 94."

§ 60.121 [Amended]

49. In § 60.121, paragraph (d) is added as follows:

§ 60.121 Definitions.

* * * * *

(d) *Blast furnace* means any furnace used to recover metal from slag.

* * * * *

§ 60.133 [Amended]

50. In § 60.133, paragraph (b)(1), the first sentence is amended by revising the words "pouring of the heat" to read "pouring of part of the production cycle."

§ 60.144 [Amended]

51. In § 60.144, paragraph (c) is revised to read as follows:

§ 60.144 Test methods and procedures.

* * * * *

(c) The owner or operator shall use the monitoring devices of § 60.143(b)(1) and (2) for the duration of the particulate matter runs. The arithmetic average of all measurements taken during these runs shall be used to determine compliance with § 60.143(c).

* * * * *

§ 60.143a [Amended]

52. Amend § 60.143a, paragraph (c) as follows:

a. The words "All monitoring devices" in the first sentence are revised to read "All monitoring devices required by paragraph (a) of this section."

b. The words "EPA Reference Method 2" in the first sentence are revised to read "Method 2 of Appendix A of this part."

c. The words "EPA Reference Method 2" in the second sentence are revised to read "Method 2."

§ 60.144a [Amended]

53. In § 60.144a, paragraph (d) is amended by revising it to read as follows:

§ 60.144a Test methods and procedures.

* * * * *

(d) To comply with § 60.143a(d) or (e), the owner or operator shall use the monitoring device of § 60.143a(a) to determine the exhaust ventilation rates or levels during the particulate matter runs. Each owner or operator shall then use these rates or levels to determine the 3-hour averages required by § 60.143a(d) and (e).

* * * * *

§ 60.145a [Amended]

54. In § 60.145a, paragraph (f), in the first sentence by revising the words "Reference Method 5" to read "Method 5."

§ 60.153 [Amended]

55. Amend § 60.153 as follows:

a. In paragraph (b)(3) by revising the word "thermocouple" or "thermocouples" to read "temperature measuring device" or "temperature measuring devices" wherever it occurs.

b. In paragraph (b)(5), in the second sentence by revising the words "with the method specified under § 60.154(c)(2)" to read "with the method specified under § 60.154(b)(5)."

§ 60.154 [Amended]

56. In § 60.154, paragraphs (b)(1) and (b)(3) are revised, and in paragraph (b)(4), the equations and definitions are revised as follows:

§ 60.154 Test methods and procedures.

* * * * *

(b) * * *

(1) The emission rate (E) of particulate matter for each run shall be computed using the following equation:

$$E = \frac{c_s Q_{sd}}{KS}$$

Where:

E = Emission rate of particulate matter, g/kg (lb/ton) of dry sludge input.

c_s = Concentration of particulate matter, g/dscm (gr/dscf).

Q_{sd} = Volumetric flow rate of effluent gas, dscm/hr (dscf/hr).

S = Charging rate of dry sludge during the run, kg/hr (ton/hr).

K = Conversion factor, 1.0 g/g (7,000 gr/lb).

* * * * *

(3) The dry sludge charging rate (S) for each run shall be computed using either of the following equations:

$$S = S_m R_{dm} / \Theta$$

$$S = S_v R_{dv} / K_v \Theta$$

Where:

S = Charging rate of dry sludge, kg/hr (ton/hr).

S_m = Total mass of sludge charge, kg (ton).

R_{dm} = Average mass of dry sludge per unit mass of sludge charged, kg/kg (ton/ton).

θ = Duration of run, hr.

S_v = Total volume of sludge charged, m^3 (gal).

R_{dv} = Average mass of dry sludge per unit volume of sludge charged, kg/m^3 (lb/gal).

K_v = Conversion factor, 1 g/g (2,000 lb/ton).

(4) * * *

$$S_m = \sum_{i=1}^{n} Q/_{mi} \theta_i$$

$$S_v = \sum_{i=1}^{n} \frac{Q_{vi}}{\theta_i}$$

Where:

S_m = Total mass of sludge charged to the incinerator during the test run.

S_v = Total volume of sludge charged to the incinerator during the test run.

Q_{mi} = Average mass flow rate calculated by averaging the flow rates at the beginning and end of each interval "i," kg/hr (ton/hr).

Q_{vi} = Average volume flow rate calculated by averaging the flow rates at the beginning and end of each interval "i," m^3/hr (gal/hr).

θ_i = Duration of interval "i," hr.

* * * * *

57. Paragraph (b)(5)(iii) is amended by revising the words "mg/liter (lb/ft^3) or mg/mg (lb/lb)" to read "kg/m^3 (lb/gal) or kg/kg (ton/ton)."

§ 60.165 [Amended]

58. In § 60.165, paragraph (d)(2) is amended by revising the words

"installed under § 60.163" to read "installed under paragraph (b) of this section."

§ 60.192 [Amended]

59. In § 60.192, paragraph (a) is amended by revising the words "according to § 60.8 above" to read "according to § 60.195."

§ 60.195 [Amended]

60. Amend § 60.195 as follows:

a. In paragraph (b)(1) by revising the words "(mg/dscf)" in the definition of the term "c_s" to read "(gr/dscf)"; and revising the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(2) by revising the words "(mg/dscf)" in the definition of the symbol "c_s" to read "(gr/dscf)"; and revising the words "(453,600 mg/lb)" in the definition of the symbol "K" to read "(7,000 gr/lb)."

§ 60.201 [Amended]

61. In § 60.201 by revising paragraph (c) to read as follows:

§ 60.201 Definitions.

* * * * *

(c) *Equivalent* P_2O_5 *feed* means the quantity of phosphorus, expressed as phosphorus pentoxide, fed to the process.

* * * * *

§ 60.202 [Amended]

62. In § 60.202, paragraph (a) is amended by revising the words "metric ton" to read "Mg."

§ 60.203 [Amended]

63. In § 60.203, paragraph (b) is amended by revising the words "metric ton" to read "Mg."

§ 60.204 [Amended]

64. Amend § 60.204 as follows:

a. In paragraph (b)(1) by revising the words "metric ton" in the definition of the term "E" to read "Mg"; revising the words "(mg/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; revising the words "metric ton" in the definition of the term "P" to read "Mg"; and revising the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(3) by revising the words "metric ton" in the definition of the term "M_p" to read "Mg."

§ 60.211 [Amended]

65. In § 60.211 by revising paragraph (c) to read as follows:

§ 60.211 Definitions.

* * * * *

(c) *Equivalent* P_2O_5 *feed* means the quantity of phosphorus, expressed as phosphorus pentoxide, fed to the process.

* * * * *

§ 60.212 [Amended]

66. In § 60.212, paragraph (a) is amended by revising the words "metric ton" to read "megagram (Mg)."

§ 60.213 [Amended]

67. In § 60.213, paragraph (b) is amended by revising the words "metric ton" to read "Mg."

§ 60.214 [Amended]

68. Amend § 60.214 as follows:

a. In paragraph (b)(1) by revising the words "metric ton" in the definition of the term "E" to read "Mg"; revising the words "(mg/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; revising the words "metric ton" in the definition of the term "P" to read "Mg"; and revising the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(3) by revising the words "metric ton" in the definition of the term "M_p" to read "Mg."

§ 60.222 [Amended]

69. In § 60.222, paragraph (a) is amended by revising the words "metric ton" to read "megagram (Mg)."

§ 60.223 [Amended]

70. Amend § 60.223 as follows:

a. In paragraph (b) by revising the words "metric ton" to read "Mg."

b. In paragraph (c), in the first sentence by revising the word "part" to read "subpart."

§ 60.224 [Amended]

71. Amend § 60.224 as follows:

a. In paragraph (b)(1) by revising the words "metric ton" in the definition of the term "E" to read "Mg"; revising the words "(mg/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; revising the words "metric ton" in the definition of the term "P" to read "Mg"; and revising the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(3) by revising the words "metric ton" in the definition of the term "M_p" to read "Mg."

§ 60.232 [Amended]

72. § 60.232 is amended by removing the paragraph designation and by revising the words "metric ton" to read "megagram (Mg)."

§ 60.233 [Amended]

73. § 60.233 is amended by removing the paragraph designation and by revising the words "metric ton" to read "Mg."

§ 60.234 [Amended]

74. Amend § 60.234 as follows:

a. In paragraph (b)(1) by revising the words "metric ton" in the definition of the term "E" to read "Mg"; revising the words "(mg/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; revising the words "metric ton" in the definition of the term "P" to read "Mg"; and revising the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(3) by revising the words "metric ton" in the definition of the term "M_p" to read "Mg."

§ 60.241 [Amended]

75. In § 60.241, paragraph (c) is amended by italicizing the word "stored."

§ 60.242 [Amended]

76–77. In § 60.242, paragraph (a) is amended by revising the words "metric ton" to read "megagram (Mg)."

§ 60.244 [Amended]

78. Amend § 60.244 as follows:

a. In paragraph (c)(1) by revising the words "metric ton" in the definition of the term "E" to read "Mg"; revising the words "(mg/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; revising the words "metric ton" the words "(453,600 mg/lb)" in the definition of the term "K" to read "(7,000 gr/lb)."

b. In paragraph (b)(3) by revising the words "metric ton" in the definition of the term "M_p"to read "Mg."

§ 60.250 [Amended]

79. In § 60.250, paragraph (a) is amended by revising the words "200 tons" to read "181 Mg (200 tons)."

§ 60.251 [Amended]

80. In § 60.251, paragraphs (b) and (c) are amended by revising "D388–77" to read "D388–77, 90, 91, 95, or 98a."

§ 60.252 [Amended]

81. In § 60.252, paragraph (b)(1) is amended by revising the words "0.040 g/dscm (0.018 gr/dscf)" to read "0.040 g/dscm (0.017 gr/dscf)."

§ 60.253 [Amended]

82. Amend § 60.253 as follows:

a. In paragraph (a)(1), the second sentence is amended by revising the words "±3° Fahrenheit" to read "±1.7 °C (±3 °F)."

b. In paragraph (a)(2)(i), the second sentence is amended by revising the word "gage" to read "gauge."

§ 60.261 [Amended]

83. Amend § 60.261 as follows:

a. Paragraph (n) is amended by revising "ASTM Designation A99–76" to read "ASTM Designation A99–76 or 82 (Reapproved 1987)."

b. Paragraphs (s) and (w) are amended by revising "ASTM Designation A100–69 (Reapproved 1974)" to read "ASTM Designation A100–69, 74, or 93."

c. Paragraph (q) is amended by revising "ASTM Designation A101–73" to read "ASTM Designation A101–73 or 93."

d. Paragraph (t) is amended by revising "ASTM Designation A482–76" to read "ASTM Designation A482–76 or 93."

e. Paragraph (o) is amended by revising "ASTM Designation A483–64 (Reapproved 1974)" to read "ASTM Designation A483–64 or 74 (Reapproved 1988)."

f. Paragraph (v) is amended by revising "ASTM Designation A495–76" to read "ASTM Designation A495–76 or 94."

§ 60.266 [Amended]

84. Amend § 60.266 as follows:

a. Paragraph (c)(1) is amended by revising the words "emissions is quantified" in the definition of the term "n" to read "emissions are quantified"; revising the words "(g/dscf)" in the definition of the term "c_{si}" to read "(gr/dscf)"; and revising the words "(453.6 g/lb)" in the definition of the term "K" to read "(7000 gr/lb)."

b. Paragraph (c)(2)(ii) is amended by revising the words "5.70 dscm (200 dscf)" to read "5.66 dscm (200 dscf)."

§ 60.274 [Amended]

85. Amend § 60.274 as follows:

a–b. Paragraph (a)(4) is amended by revising the words "under paragraph (e) of this section" to read "under paragraph (f) of this section."

c. In § 60.274, paragraph (i), the first sentence is amended by revising the words "required by § 60.275(c)" to read "required by § 60.276(c)."

d. In § 60.274, by revising paragraph (i)(4) to read as follows:

§ 60.274 Monitoring of operations.

* * * * *

(i) * * *

(4) Continuous opacity monitor or Method 9 data.

* * * * *

§ 60.275 [Amended]

86. Amend § 60.275 as follows:

a. Paragraph (e)(2) is amended by revising the words "more then one control" to read "more than one control."

b. Paragraph (e)(4) is amended by revising the words "the test runs shall be conducted concurrently" to read "the Method 9 test runs shall be conducted concurrently with the particulate matter test runs."

c. In paragraph (i), the fifth sentence is amended by revising the words "In the case, Reference Method 9" to read "In this case, Method 9."

§ 60.276 [Amended]

87. Amend § 60.276 by:

a. Paragraphs (a) and (c)(6)(iv) are revised.

b. In paragraph (b), the second sentence is amended by revising the words "postmarked 30 days prior" to read "postmarked at least 30 days prior."

The revisions read as follows:

§ 60.276 Recordkeeping and reporing requirements.

(a) Operation at a furnace static pressure that exceeds the value established under § 60.274(g) and either operation of control system fan motor amperes at values exceeding ±15 percent of the value established under § 60.274(c) or operation at flow rates lower than those established under § 60.274(c) may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility. Operation at such values shall be reported to the Administrator semiannually.

* * * * *

(c) * * *

(6) * * *

(iv) Continuous opacity monitor or Method 9 data.

* * * * *

§ 60.274a [Amended]

88. Amend § 60.274a by:

a. In paragraph (c), the first sentence is revised, and paragraph (h)(4) is revised.

b. Paragraph (f) is amended by adding the following sentence after the first sentence: "The pressure shall be recorded as 15-minute integrated averages."

c. In paragraph (h), the first sentence is amended by revising the words "required by § 60.275a(d)" to read "required by § 60.276a(f)."

The revisions read as follows:

§ 60.274a Monitoring of operations.

* * * * *

(c) When the owner or operator of an EAF is required to demonstrate compliance with the standards under § 60.272a(a)(3), and at any other time that the Administrator may require (under section 114 of the Act, as amended), either the control system fan motor amperes and all damper positions or the volumetric flow rate through each separately ducted hood shall be determined during all periods in which a hood is operated for the purpose of capturing emissions from the affected facility subject to paragraph (b)(1) or (b)(2) of this section. * * *

* * * * *

(h) * * *

(4) Continuous opacity monitor or Method 9 data.

* * * * *

§ 60.275a [Amended]

89. In § 60.275a, paragraph (e)(4) is amended by revising the words "the test runs shall be conducted concurrently" to read "the Method 9 test runs shall be conducted concurrently with the particulate matter test runs."

§ 60.276a [Amended]

90. Amend § 60.276a as follows:

a. In paragraph (e), the second sentence is amended by revising the words "postmarked 30 days prior" to read "postmarked at least 30 days prior."

b. Paragraph (f)(6)(iv) is amended by revising as follows:

§ 60.276a Recordkeeping and reporting requirements.

* * * * *

(f) * * *

(iv) Continuous opacity monitor or Method 9 data.

* * * * *

§ 60.281 [Amended]

91. Amend § 60.281 as follows:

a. In paragraph (c) by revising the words "Reference Method 16" to read "Method 16."

b. In paragraph (d) by revising the words "below tank(s)" to read "blow tank(s)."

c. In paragraph (e) by revising the words "digestion system" to read "digester system."

§ 60.282 [Amended]

92. In § 60.282, paragraph (a)(3)(i) is amended by revising the words "0.15 g/dscm (0.067 gr/dscf)" to read "0.15 g/dscm (0.066 gr/dscf)."

§ 60.283 [Amended]

93. Amend § 60.283 as follows:

a. In paragraph (a)(1)(iii) by revising the words "1200°F." to read "650 °C (1200 °F)."

b. In paragraph (a)(1)(v), in the second sentence by revising the words "5 ppm by volume on a dry basis, corrected to the actual oxygen content of the untreated gas stream" to read "5 ppm by volume on a dry basis, uncorrected for oxygen content."

c. In paragraph (a)(1)(vi) by revising the words "0.005 g/kg ADP" to read "0.005 g/kg air dried pulp (ADP)."

§ 60.284 [Amended]

94. Amend § 60.284 by:

a. In paragraph (a)(2)(ii) by revising the words "20 percent" to read "25 percent"

b. Revising paragraph (c) introductory text.

c. In paragraph (c)(3) by revising the words "Correct all 12-hour average TRS concentrations to 10 volume percent oxygen, except that all 12-hour average TRS concentration from a recovery furnace shall be corrected to 8 volume percent using the following equation:" to read "Using the following equation, correct all 12-hour average TRS concentrations to 10 volume percent oxygen, except that all 12-hour average TRS concentrations from a recovery furnace shall be corrected to 8 volume percent oxygen instead of 10 percent, and all 12-hour average TRS concentrations from a facility to which the provisions of § 60.283(a)(1)(v) apply shall not be corrected for oxygen content:"

d. Paragraph (d)(3)(ii) is amended by revising the words "1200°F" to read "650 °C (1200 °F)."

e. Adding paragraph (f).

The revisions and addition read as follows:

§ 60.284 Monitoring of emissions and operations.

* * * * *

(c) Any owner or operator subject to the provisions of this subpart shall, except where the provisions of § 60.283(a)(1)(iii) or (iv) apply, perform the following:

* * * * *

(f) The procedures under § 60.13 shall be followed for installation, evaluation, and operation of the continuous monitoring systems required under this section.

(1) All continuous monitoring systems shall be operated in accordance with the applicable procedures under Performance Specifications 1, 3, and 5 of appendix B to this part.

(2) Quarterly accuracy determinations and daily calibration drift tests shall be performed in accordance with Procedure 1 of appendix F to this part.

§ 60.285 [Amended]

95. Amend § 60.285 as follows:

a. In paragraph (c)(1) by revising the definition of the term "c_s" to read "c_s = Concentration of particulate matter, g/dscm (lb/dscf)."

b. In paragraph (d)(3) by revising the equation used to calculate "GLS" as follows:

$$GLS = 100\ C_{Na_2S} / \left(C_{Na_2S} C_{NaOH} C_{Na_2CO_3} \right)$$

c. In paragraph (e)(1) by revising the definition of "F" to read "F = conversion factor, 0.001417 g H_2S/m^3-ppm (8.846×10^{-8} lb H_2S/ft^3-ppm)."

d. In paragraph (f)(1) by revising the words "205 °C (400 °F)" to read "204 °C (400 °F)."

e. Revising paragraph (f)(2).

The revisions read as follows:

§ 60.285 Test methods and procedures.

* * * * *

(f) * * *

(2) In place of Method 16, Method 16A or 16B may be used.

* * * * *

§ 60.290 [Amended]

96. In § 60.290, paragraph (c) is amended by revising the words "4,550 kilograms" to read "4.55 Mg (5 tons)."

§ 60.291 [Amended]

97. Amend § 60.291 as follows:

a. The second sentence of the definition of the term "Glass melting furnace" is amended by revising the word "appendaees" to read "appendages."

b. The definition of the term "lead recipe" is amended by revising the chemical formula "Na_2M" to read "Na_2O."

c. The second sentence of the definition of the term "rebricking" is amended by revising the word "replacment" to read "replacement."

§ 60.292 [Amended]

98. In § 60.292, paragraph (a)(2), the definition of the term STD is amended by revising the words "g of particulate/kg" to read "g of particulate/kg (lb of particulate/ton)."

§ 60.293 [Amended]

99. Amend § 60.293 as follows:

a. In paragraph (d)(1) by revising the words "specified in paragraph (b)(1) of this section" to read "specified in paragraph (b) of this section."

b. Paragraph (e) is redesignated as paragraph (f).

c. Paragraph (d)(3) introductory text is redesignated as paragraph (e); paragraphs (d)(3)(i), (ii), and (iii) are redesignated as paragraphs (e)(1), (2), and (3).

d. Newly designated paragraph (f) is amended by revising the words "120±14°C" to read "120±14°C (248±25°F)."

§ 60.296 [Amended]

100. Amend § 60.296 as follows:

In paragraph (b)(3) by revising the words "American Society of Testing and Materials (ASTM) Method D240–76" to read "ASTM Method D240–76 or 92" and by revising "D1826–77" to read "D1826–77 or 94."

§ 60.301 [Amended]

101. In § 60.301, the first paragraph is amended by revising the words "the act" to read "the Act."

§ 60.313 [Amended]

102. Amend § 60.313 as follows:

a. Paragraph (c)(1) is amended by revising the words "Reference Method 24" to read "Method 24" wherever they occur.

b. In paragraph (c)(1)(i)(B), the third sentence is amended by revising the words "other transfer efficiencies other than" to read "transfer efficiencies other than."

c. Paragraph (c)(2)(i) is amended by revising the words "in (c)(2)(i)(A), (B), and (C)" to read "in paragraphs (c)(2)(i)(A), (B), and (C)" wherever they occur.

§ 60.315 [Amended]

103. In § 60.315, paragraph (a)(2) is amended by revising the words "Reference Method 24" to read "Method 24."

§ 60.330 [Amended]

104. In § 60.330, paragraph (a) is amended by revising the words "10.7 gigajoules" to read "10.7 gigajoules (10 million Btu)."

§ 60.331 [Amended]

105. In § 60.331, paragraph (s) is removed.

§ 60.332 [Amended]

106. In § 60.332, paragraph (a) is amended by revising the words "the date of the performance test" to read "the date on which the performance test."

§ 60.334 [Amended]

107. In § 60.334, paragraph (c)(3), the first sentence is amended by revising the words "provided in § 60.332(g)" to read "provided in § 60.332(f)."

§ 60.335 [Amended]

108. Amend § 60.335 by:

a. Paragraph (c)(1) is amended by revising the words:

“NO_X = emission rate of NO_X at 15 percent O_2 and ISO standard ambient conditions, volume percent.
NO_X = observed NO_X concentration, ppm by volume.”
“NO_X = emission rate of NO_X at 15 percent O2 and ISO standard ambient conditions, ppm by volume.
NO_X = observed NO_X concentration, ppm by volume at 15 percent O_2.”

b. Paragraph (d) is revised.

c. In paragraph (f)(1), the first sentence is amended by revising the words “in paragraph (b)(1) of this section” to read “in paragraph (c)(1) of this section.”

The revisions read as follows:

§ 60.335 Test methods and procedures.

* * * * *

(d) The owner or operator shall determine compliance with the sulfur content standard in § 60.333(b) as follows: ASTM D 2880–71, 78, or 96 shall be used to determine the sulfur content of liquid fuels and ASTM D 1072–80 or 90 (Reapproved 1994), D 3031–81, D 4084–82 or 94, or D 3246–81, 92, or 96 shall be used for the sulfur content of gaseous fuels (incorporated by reference-see § 60.17). The applicable ranges of some ASTM methods mentioned above are not adequate to measure the levels of sulfur in some fuel gases. Dilution of samples before analysis (with verification of the dilution ratio) may be used, subject to the approval of the Administrator.

* * * * *

§ 60.343 [Amended]

109. In § 60.343, paragraph (e), the first sentence is amended by revising the words “in which the scrubber pressure drop is greater than 30 percent below the rate established during the performance test” to read “in which the scrubber pressure drop or scrubbing liquid supply pressure is greater than 30 percent below that established during the performance test.”

§ 60.344 [Amended]

110. Amend § 60.344 as follows:

a. In paragraph (b)(1), the definition of the term “c_s” is amended by revising the words “(g/dscf)” to read “(gr/dscf).”

b. In paragraph (b)(1), the definition of the term “K” is amended by revising the words “(453.6 g/lb)” to read “(7000 gr/lb).”

c. In paragraph (b)(2), the first sentence is amended by revising the words “Method 5D shall be used as positive-pressure fabric filters” to read “Method 5D shall be used at positive-pressure fabric filters.”

§ 60.372 [Amended]

111. Amend § 60.372 as follows;

a. In paragraph (a)(1) by revising the words “0.40 milligram of lead per dry standard cubic meter of exhaust (0.000176 gr/dscf)” to read “0.40 milligram of lead per dry standard cubic meter of exhaust (0.000175 gr/dscf).”

b. In paragraph (a)(2) by revising the words “1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf)” to read “1.00 milligram of lead per dry standard cubic meter of exhaust (0.000437 gr/dscf).”

c. In paragraph (a)(3) by revising the words “1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf)” to read “1.00 milligram of lead per dry standard cubic meter of exhaust (0.000437 gr/dscf).”

d. In paragraph (a)(5) by revising the words “4.50 milligrams of lead per dry standard cubic meter of exhaust (0.00198 gr/dscf)” to read “4.50 milligrams of lead per dry standard cubic meter of exhaust (0.00197 gr/dscf).”

e. In paragraph (a)(6) by revising the words “1.00 milligram per dry standard cubic meter of exhaust (0.00044 gr/dscf)” to read “1.00 milligram of lead per dry standard cubic meter of exhaust (0.000437 gr/dscf).”

§ 60.374 [Amended]

112. Amend § 60.374 as follows:

a. In paragraph (c)(1), in the definition of the term “c_{Pbi}” by revising the words “mg/dscm” to read “mg/dscm (gr/dscf).”

b. In paragraph (c)(1), in the definition of the term “K” by revising the words “453,600 mg/lb” to read “7000 gr/lb).”

§ 60.381 [Amended]

113. In § 60.381, in the definition of the term “storage bin” by revising the words “or metallic minerals” to read “of metallic minerals.”

§ 60.382 [Amended]

114. In § 60.382, paragraph (a)(1) is amended by revising the words “0.05 grams per dry standard cubic meter” to read “0.05 grams per dry standard cubic meter (0.02 g/dscm).”

§ 60.385 [Amended]

115. In § 60.385, paragraph (c) is amended by revising the words “scrubber pressure loss (or gain) and liquid flow rate” to read “scrubber pressure loss (or gain) or liquid flow rate”.

§ 60.386 [Amended]

116. In § 60.386, paragraph (c) is amended by revising the words “§ 60.3284(a) and (b)” to read “§ 60.384(a) and (b).”

§ 60.391 [Amended]

117. Amend § 60.391 as follows:

a. In paragraph (b), the definition of “E” is amended by revising the words “destruction efficiency” to read “destruction or removal efficiency.”

b. In paragraph (b), the eleventh definition is amended by revising the words

“$L_{ci}{}'l$ = Volume of each coating (i) consumed by each application method (l), as received liters)”

to read

“L_{cil} = Volume of each coating (i) consumed by each application method (l), as received (liters).”

§ 60.393 [Amended]

118. Amend § 60.393 as follows:

a. In paragraph (c)(1)(i) by revising the words “Reference Method 24” to read “Method 24” wherever they occur.

b. Paragraph (c)(2)(ii)(A) is amended by revising the term to read as follows:

$$\sum_{l=1}^{n}$$

to read as follows:

$$\sum_{i=1}^{n}$$

§ 60.395 [Amended]

119. In § 60.395, paragraph (d) is amended by revising the words “Reference Method 25” to read “Method 25.”

§ 60.396 [Amended]

120. In § 60.396, paragraphs (a)(1), (a)(2), (b), and (c) are amended by revising the words “Reference Method” to read “Method.”

§ 60.401 [Amended]

121. In § 60.401, paragraph (b) is amended by revising the words “unit including, moisture” to read “unit, including moisture.”

§ 60.402 [Amended]

122. In § 60.402, paragraph (a)(2)(i) is amended by revising the word “Contains” to read “Contain.”

§ 60.424 [Amended]

123. Amend § 60.424 to read as follows:

a. In the first paragraph (b)(3), in the first sentence by revising the words "scales or computed from material balance shall" to read "scales, or the result of computations using a material balance, shall."

b. The second paragraph (b)(3) is redesignated as (b)(4).

§ 60.431 [Amended]

124. In § 60.431, paragraph (b), the definition of the term "L_{di}" is amended by adding the words "the subject facility (or facilities)" to the end of the definition.

§ 60.433 [Amended]

125. Amend § 60.433 as follows:

a. In paragraph (a)(5), the first sentence is amended by revising the words "material or on at least" to read "material on at least."

b. Paragraph (a)(5)(ii) is amended by revising the punctuation at the end of the paragraph. The words "according to § 60.435." are revised to read "according to § 60.435;"

c. Paragraphs(b)(1), (b)(2), (b)(3), (b)(5), (c)(2)(ii), and (c)(2)(iii) are amended by adding an "=" between the "i" and the "1" under the summation sign.

d. Paragraph (c)(2)(v) is amended by replacing the "e" subscript with "a" wherever it occurs.

e. Paragraph (e)(5)(ii) is amended by replacing the "a" subscript with "e" wherever it occurs.

§ 60.435 [Amended]

126. Amend § 60.435 as follows:

a. Paragraphs (a)(1), (a)(2), and (b) are amended by revising the words "Reference Method" to read "Method" wherever they occur.

b. Paragraph (d)(1) is amended by revising the words "ASTM D1475–60 (Reapproved 1980)" to read "ASTM D1475–60, 80, or 90."

§ 60.440 [Amended]

127. In § 60.440, paragraph (b) is amended by revising the words "45 Mg" to read "45 Mg (50 tons)" wherever they occur.

§ 60.441 [Amended]

128. In § 60.441, paragraphs (a) and (b) are amended by revising the words "Reference Method" to read "Method" wherever they occur.

§ 60.443 [Amended]

129. Amend § 60.443 as follows:

a. In paragraph (b) by revising the words "R_q less" to read "R_q is less."

b. In paragraph (d) by revising the words "in paragraph (b)(1) of this section" to read "in paragraph (b) of this section."

c. In paragraph (e), in the third sentence by revising the words "38°C (50°F)" to read "28°C (50°F)."

d. In paragraph (i) by revising the word "devices" to read "device(s)."

§ 60.446 [Amended]

130. In § 60.446, paragraphs (a) and (b) are amended by revising the words "Reference Method" to read "Method" wherever they occur.

§ 60.453 [Amended]

131. Amend § 60.453 as follows:

a. In paragraph (b) by revising the words "performance text" to read "performance test."

b. In paragraph (b)(1) by revising the words "Reference Method" to read "Method" wherever they occur.

c. In paragraph (b)(1)(i)(B) by revising the word "coatings" to read "coating."

d. In paragraph (b)(1)(i)(C) by revising equation (3).

e. In paragraphs (b)(2)(i)(A) and (b)(2)(i)(B) by revising Equations (6) and (7).

f. In paragraph (b)(2)(i)(B) by removing Equation (7) and its nomenclature, adding them to the end of paragraph (b)(2)(i)(A), and redesignating the equation as Equation (6).

g. In paragraph (b)(3)(i) by revising the word "assumed" to read "consumed."

The revisions reads as follows:

§ 60.453 Test methods and procedures.

* * * * *

(b) * * *

(1) * * *

(i) * * *

(C) * * *

$$T = \frac{\sum_{i=1}^{n}\sum_{k=1}^{m} L_{cik} V_{sik} T_k}{L_s} \qquad (3)$$

* * * * *

(2) * * *

(i) * * *

(A) * * *

$$F = \frac{\sum_{i=1}^{n} Q_{bi} C_{bi}}{\sum_{i=1}^{n} Q_{bi} C_{bi} + \sum_{k=1}^{p} Q_{fk} C_{fk}} \qquad (6)$$

* * * * *

(B) * * *

$$E = \frac{\sum_{i=1}^{n} Q_{bi} C_{bi} - \sum_{j=1}^{m} Q_{aj} C_{aj}}{\sum_{i=1}^{n} Q_{bi}}$$

* * * * *

§ 60.454 [Amended]

132. In § 60.454, paragraph (a)(2) is amended by revising the words "of the greater of 0.75 percent of the temperature being measured expressed in degrees Celsius or ±2.5°C" to read "of 0.75 percent of the temperature being measured, expressed in degrees Celsius, or ±2.5 °C, whichever is greater."

§ 60.455 [Amended]

133. Amend § 60.455 as follows:

a. Paragraphs (c)(1) and (c)(2) are amended by revising the words "28 °C" to read "28 °C" (50 °F)" wherever they occur.

b. In paragraph (d), the first sentence is amended by revising the word "opreator" to read "operator."

§ 60.456 [Amended]

134. Amend § 60.456 as follows:

a. In paragraph (a)(1), the second sentence is amended by revising the words "Reference Method 24" to read "Method 24."

b. In paragraph (a)(1), the third sentence is amended by revising the words "subsection 4.4 of Method 24" to read "Section 12.6 of Method 24."

c. Paragraph (a)(4) is amended by revising the word "volocity" to read "velocity."

d. Paragraph (c) is amended by revising the words "0.003 dscm" to read "0.003 dscm (0.1 dscf)."

§ 60.463 [Amended]

135. Amend § 60.463 as follows:

a. Paragraph (c)(1) is amended by revising the words "Reference Method 24" to read "Method 24" wherever they occur.

b. Paragraph (c)(3)(iii) is amended by revising the word "computation" to read "computations."

c. Paragraph (c)(4)(ii) is amended by revising the defined term "m" to read "n."

§ 60.464 [Amended]

136. In § 60.464, paragraph (c), the second sentence is amended by revising the words "which is greater" to read "whichever is greater."

§ 60.465 [Amended]

137. Amend § 60.465 as follows:

a. In paragraph (c), the first sentence is amended by revising the reference "§ 69.462" to read "§ 60.462."

b. In paragraph (d), the first sentence is amended by revising the reference "§ 69.464" to read "§ 60.464."

§ 60.466 [Amended]

138. Amend § 60.466 as follows:

a. Paragraphs (a)(1) and (a)(2) are amended by revising the words

"Reference Method" to read "Method" wherever they occur.

b. In paragraph (a)(1), the first sentence is amended by revising the words "coating for determining the VOC content" to read "coating, shall be used for determining the VOC content."

c. In paragraph (a)(1), the third sentence is amended by revising the words "section 4.4" to read "Section 12.6."

d. Paragraph (c) is amended by revising the words "0.003 dry standard cubic meter (DSCM)" to read "0.003 dscm (0.11 dscf)."

§ 60.471 [Amended]

139. In § 60.471, the definition of the term "Catalyst" is amended by revising the words "means means" to read "means."

§ 60.472 [Amended]

140. Amend § 60.472 as follows:

a. Paragraph (a)(1)(i) is amended by revising the words "0.04 kilograms of particulate per megagram" to read "0.04 kg/Mg (0.08 lb/ton)."

b. Paragraph (a)(1)(ii) is amended by revising the words "0.04 kilograms per megagram" to read "0.04 kg/Mg (0.08 lb/ton)."

c. Paragraph (b)(1) is amended by revising the words "0.67 kilograms of particulate per megagram" to read "0.67 kg/Mg (1.3 lb/ton)."

d. Paragraph (b)(2) is amended by revising the words "0.71 kilograms of particulate per megagram" to read "0.71 kg/Mg (1.4 lb/ton)."

e. Paragraph (b)(3) is amended by revising the words "0.60 kilograms of particulate per megagram" to read "0.60 kg/Mg (1.2 lb/ton)."

f. Paragraph (b)(4) is amended by revising the words "0.64 kilograms of particulate per megagram" to read "0.64 kg/Mg (1.3 lb/ton)."

g. Paragraph (b)(5) is amended by revising the words "procedures in § 60.474(k)" to read "procedures in § 60.474(g)."

§ 60.473 [Amended]

141. Amend § 60.473 as follows:

a. In paragraph (a), the second sentence is amended by revising the words "±15°C" to read "±15°C (±25°F)."

b. In paragraph (b), the second sentence is amended by revising the words "±10 °C" to read "±10 °C (±18 °F)."

c. In paragraph (c), the first sentence is amended by revising the words "(a) and (b)" to read "(a) or (b)"

§ 60.474 [Amended]

142. Amend § 60.474 as follows:

a. In paragraph (c)(1), the definition of the term "E" is amended by revising the words "kg/Mg" to read "kg/Mg (lb/ton)."

b. In paragraph (c)(1), the definition of the term "c_s" is amended by revising the words "(g/dscf)" to read "(gr/dscf)."

c. In paragraph (c)(1), the definition of the term "K" is amended by revising the words "907.2/(g-Mg)/(kg-ton)" to read "7000 gr/lb)."

d. In paragraph (c)(4), the definition of the term "d" is amended by revising the words "llb/ft³" to read "lb/ft³."

e. Paragraphs (c)(4)(ii) and (f) are revised.

The revisions read as follows:

§ 60.474 Test methods and procedures.

* * * * *

(c) * * *

(4) * * *

(ii) The density (d) of the asphalt shall be computed using the following equation:

$$d = K_1 - K_2 T_i$$

Where:

d = Density of the asphalt, kg/m³ (lb/ft³)

K_1 = 1056.1 kg/m³ (metric units)

= 64.70 lb/ft³ (English Units)

K_2 = 0.6176 kg/(m³ °C) (metric units)

= 0.0694 lb/(ft³ °F) (English Units)

T_i = temperature at the start of the blow, °C (°F)

* * * * *

(f) If at a later date the owner or operator believes that the emission limits in § 60.472(a) and (b) are being met even though one of the conditions listed in this paragraph exist, he may submit a written request to the Administrator to repeat the performance test and procedure outlined in paragraph (c) of this section.

(1) The temperature measured in accordance with § 60.473(a) is exceeding that measured during the performance test.

(2) The temperature measured in accordance with § 60.473(b) is lower than that measured during the performance test.

* * * * *

§ 60.480 [Amended]

143. In § 60.480(d)(2), line 3, revise the words "1,000 Mg/yr" to read "1,000 Mg/yr (1,102 ton/yr)"

§ 60.481 [Amended]

144. Amend § 60.481 as follows:

a. Paragraph (a)(1) under the definition of "Capital expenditure" is amended by revising the words "repair allowance, B, as reflected" to "repair allowance, B, divided by 100 as reflected"

b. The definition for "In vacuum service" is amended by revising the words "5 kilopascals (kPa)" to "5 kilopascals (kPa)(0.7 psia)."

c. The definition of the term "Repaired" is amended by revising the words "instrument reading or 10,000 ppm or greater" to read "instrument reading of 10,000 ppm or greater."

§ 60.482–2 [Amended]

145. Amend § 60.482–2 as follows:

a. Paragraph (e) is amended by revising the words "(a), (c), and (d) if the pump" to read "(a), (c), and (d) of this section if the pump."

b. Paragraph (e)(3) is amended by revising the words "paragraph (e)(2)" to read "paragraph (e)(2) of this section."

c. Paragraph (f) is amended by revising the words "exempt from the paragraphs (a) through (e)" to read "exempt from paragraphs (a) through (e) of this section."

§ 60.482–3 [Amended]

146. In § 60.482–3, paragraph (i)(2) is amended by revising the words "paragraph (i)(1)" to read "paragraph (i)(1) of this section."

§ 60.482–4 [Amended]

147. In § 60.482–4, paragraph (c) is amended by revising the words "paragraphs (a) and (b)" to read "paragraphs (a) and (b) of this section."

§ 60.482–5 [Amended]

148. In § 60.482–5, paragraph (c) is amended by revising the words "paragraphs (a) and (b)." to read "paragraphs (a) and (b) of this section."

§ 60.482–7 [Amended]

149. In § 60.482–7, paragraph (f)(3) is amended by revising the words "paragraph (f)(2)" to read "paragraph (f)(2) of this section."

§ 60.482–10 [Amended]

150. In § 60.482–10, paragraph (c) is amended by revising the words "temperature of 816 °C" to read "temperature of 816 °C (1500 °F)."

§ 60.483–1 [Amended]

151. In § 60.483–1, paragraph (b)(1) is amended by revising the words "specified in § 60.487(b)" to read "specified in § 60.487(d)."

§ 60.483–2 [Amended]

152. In § 60.483–2, paragraph (a)(2) is amended by revising the words "specified in § 60.487(b)" to read "specified in § 60.487(d)."

§ 60.484 [Amended]

153. In § 60.484, paragraph (f)(2) is amended by revising the words "paragraphs (b), (c), (d), and (e)" to read

"paragraphs (b), (c), (d), and (e) of this section."

§ 60.485 [Amended]

154. Amend § 60.485 as follows:

a. In paragraph (c)(2), in the third sentence by revising the word "indicates" is revised to read "indicated."

b. In paragraph (d), in the first sentence by revising the words "in VOC series" to read "in VOC service."

c. In paragraph (d)(1) by revising the words "ASTM E–260, E–168, E–169" to read "ASTM E260–73, 91, or 96, E168–67, 77, or 92, E169–63, 77, or 93."

d. In paragraphs (e)(1) and (e)(2) by revising the words "0.3 kPa at 20°C" to read "0.3 kPa at 20 °C (1.2 in. H_2O at 68 °F)" wherever they occur.

e. In paragraph (e)(1) by revising "ASTM D–2879" to read "ASTM D2879–83, 96, or 97."

f. In paragraph (f) by revising the words "paragraphs (d), (e), and (g)" to read "paragraphs (d), (e), and (g) of this section."

g. Paragraphs (g)(3) and (g)(4) are revised.

h. In paragraph (g)(5) by revising "ASTM D 2504–67" to read "ASTM D2504–67, 77, or 88 (Reapproved 1993)."

i. In paragraph (g)(6) by revising "ASTM D 2382–76" to read "ASTM D2382–76 or 88 or D4809–95."

The revisions read as follows:

§ 60.485 Test methods and procedures.

* * * * *

(g) * * *

(3) The maximum permitted velocity for air assisted flares shall be computed using the following equation:

$$V_{max} = K_1 + K_2 H_T$$

Where:

V_{max} = Maximum permitted velocity, m/sec (ft/sec)
H_T = Net heating value of the gas being combusted, MJ/scm (Btu/scf).
K_1 = 8.706 m/sec (metric units)
= 28.56 ft/sec (English units)
K_2 = 0.7084 m^4/(MJ-sec) (metric units)
= 0.087 ft^4/(Btu-sec) (English units)

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

$$H_T = K \sum_{i=1}^{n} C_i H_i$$

Where:

K = Conversion constant, 1.740 × 10^7 (g-mole)(MJ)/ (ppm-scm-kcal) (metric units)
= 4.674 × 10^8 [(g-mole)(Btu)/(ppm-scf-kcal)] (English units)
C_i = Concentration of sample component "i," ppm
H_i = net heat of combustion of sample component "i" at 25 °C and 760 mm Hg (77 °F and 14.7 psi), kcal/g-mole

* * * * *

§ 60.486 [Amended]

155. In § 60.486, paragraph (c)(8) is amended by revising the word "shutdown" to read "shutdowns."

§ 60.487 [Amended]

156. In § 60.487, paragraph (d) is amended by revising the words "An owner or operator electing to comply with the provisions of §§ 60.483–1 and 60.483–2" to read "An owner or operator electing to comply with the provisions of §§ 60.483–1 or 60.483–2."

§ 60.489 [Amended]

157. Amend the table in § 60.489 as follows:

a. Revise the chemical name "Chlorbenzoyl chloride" to read "Chlorobenzoyl chloride;"

b. Revise the chemical name "Chloronapthalene" to read "Chloronaphthalene;"

c. Revise the CAS No. for diethylene glycol monobutyl ether acetate to read 124–17–4;

d. Revise the chemical name "Ethylne carbonate" to read "Ethylene carbonate;"

e. Revise the chemical name "Ethylene glycol monoethy ether" to read "Ethylene glycol monoethyl ether;"

f. Revise the chemical name "Propional dehyde" to read "Propionaldehyde;" and

g. Revise the chemical name "Tetrahydronapthalene" to read "Tetrahydronaphthalene."

§ 60.491 [Amended]

158. In § 60.491, paragraphs (a)(6) and (b) are amended by revising the word "litre" or "litres" to read "liter" or "liters" wherever it occurs.

§ 60.493 [Amended]

159. Amend § 60.493 as follows:

a. Paragraph (b)(1) is amended by revising the words "Reference Method" to read "Method" wherever they occur.

b. Paragraph (b)(1)(i)(C) is amended by revising the words "volume-weighed average" to read "volume-weighted average."

c. In paragraph (b)(1)(i)(C), equation 3 is revised.

d. Paragraph (b)(1)(iii) is amended by revising the words "weighted average of mass of VOC" to read "weighted average mass of VOC."

The revisions read as follows:

§ 60.493 Performance test and compliance provisions.

* * * * *

(b) * * *

(1) * * *

(i) * * *

(C) * * *

$$G = \frac{M_o + M_d}{L_s} \qquad (3)$$

* * * * *

§ 60.494 [Amended]

160. In § 60.494, paragraph (b), the second sentence is amended by revising the words "accuracy the greater of ±0.75 percent of the temperature being measured expressed in degrees Celsius or ±2.5°C to read "accuracy of 0.75 percent of the temperature being measured, expressed in degrees Celsius, or ±2.5°C, whichever is greater."

§ 60.495 [Amended]

161. In § 60.495, paragraph (a)(1) is amended by revising the words "from data determined using Reference Method 24 or supplies" to read "from data determined using Method 24 or supplied."

§ 60.496 [Amended]

162. Revise § 60.496 as follows:

a. Paragraph (a)(1) is revised.

b. In paragraphs (a)(2), (b), and (c) by revising the words "Reference Method" to read "Method" wherever they occur.

c. In paragraph (a)(2) by revising the words "30 days in advance" to read "at least 30 days in advance."

The revisions read as follows:

§ 60.496 Test methods and procedures.

(a) * * *

(1) Method 24, an equivalent or alternative method approved by the Administrator, or manufacturers' formulation data from which the VOC content of the coatings used for each affected facility can be calculated. In the event of a dispute, Method 24 data shall govern. When VOC content of water-borne coatings, determined from data generated by Method 24, is used to determine compliance of affected facilities, the results of the Method 24 analysis shall be adjusted as described in Section 12.6 of Method 24.

* * * * *

§ 60.501 [Amended]

163. In § 60.501, the definition of "Vapor-tight gasoline tank truck" is amended by revising the words "Reference Method" to read "Method."

§ 60.531 [Amended]

164. Amend § 60.531 as follows:

a. Under the definition of "Coal-only heater", the alphabetical designations of paragraphs (a) through (e) are removed and numerical designations (1) through (5) are added.

b. Under the definition of "Cookstove", the alphabetical designations of paragraphs (a) through (g) are removed and numerical designations (1) through (7) are added.

c. Under the definition of "Wood heater", paragraph (2) is amended by revising the words "20 cubic feet" to read "0.57 cubic meters (20 cubic feet)."

d. Under the definition of "Wood heater", paragraph (3) is amended by revising the words "5 kg/hr" to read "5 kg/hr (11 lb/hr)."

e. Under the definition of "Wood heater", paragraph (4) is amended by revising the words "800 kg" to read "800 kg (1,760 lb)."

§ 60.532 [Amended]

165. Amend § 60.532 as follows:

a. In paragraph (b)(1) by revising the words "4.1 g/hr" to read "4.1 g/hr (0.009 lb/hr)."

b. Paragraphs (b)(1)(i), (b)(1)(ii), and (b)(2) are revised.

The revisions read as follows:

§ 60.532 Standards for particulate matter.

* * * * *

(b) * * *

(1) * * *

(i) At burn rates less than or equal to 2.82 kg/hr (6.2 lb/hr),

$$C = K_1 BR + K_2$$

Where:

BR = Burn rate in kg/hr (lb/hr)

K_1 = 3.55 g/kg (0.00355 lb/lb)

K_2 = 4.98 g/hr (0.0.011 lb/hr)

(ii) At burn rates greater than 2.82 kg/hr (6.2 lb/hr), C = 15 g/hr (0.033 lb/hr).

(2) An affected facility not equipped with a catalytic combustor shall not discharge into the atmosphere any gases which contain particulate matter in excess of a weighted average of 7.5 g/hr (0.017 lb/hr). Particulate emissions shall not exceed 15 g/hr (0.033 lb/hr) during any test run at a burn rate less than or equal to 1.5 kg/hr (3.3 lb/hr) that is required to be used in the weighted average and particulate emissions shall not exceed 18 g/hr (0.040 lb/hr) during any test run at a burn rate greater than 1.5 kg/hr (3.3 lb/hr) that is required to be used in the weighted average.

* * * * *

§ 60.533 [Amended]

166. Amend § 60.533 as follows:

a. In paragraph (k)(1), the third sentence is amended by revising the words "The grant of such a waiver" to read "The granting of such a waiver."

b. Paragraph (k)(2) is amended by revising the words "± ¼ inch" to read "± 0.64 cm (± ¼ inch)."

c. In paragraph (o)(4), the first sentence is amended by revising the word "indicate" to read "indicates."

d. In paragraph (o)(4), the first sentence is amended by revising the words "comply with applicable emission limit" to read "comply with the applicable emission limit."

e. In paragraph (p)(4)(ii)(A), the second sentence is amended by revising the words "± 1 gram per hour" to read "± 1 gram per hour (± 0.0022 lb per hour)."

§ 60.535 [Amended]

167. In § 60.535, paragraph (b)(9) is amended by revising the words "a reporting and recordkeeping requirements" to read "reporting and recordkeeping requirements."

§ 60.536 [Amended]

168. Amend § 60.536 as follows:

a. Paragraph (a)(3)(ii) and the equation in (i)(4)(ii) are revised.

b. Paragraph (j)(2)(v) is amended by revising the words "five inches by seven inches" to read "12.7 centimeters by 17.8 centimeters (5 inches by 7 inches)."

The revisions read as follows:

§ 60.536 Permanent label, temporary label, and owner's manual.

(a) * * *

(3) * * *

(ii) Be at least 8.9 cm long and 5.1 cm wide (3½ inches long and 2 inches wide).

* * * * *

(i) * * *

(4) * * *

(ii) * * *

$HO_E = H_v \times$ (Estimated overall efficiency/100) $\times$ BR

Where:

HO_E = Estimated heat output in Btu/hr

H_v = Heating value of fuel, 19,140 Btu/kg (8,700 Btu/lb)

BR = Burn rate of dry test fuel per hour, kg (lb)

* * * * *

§ 60.541 [Amended]

169. Amend § 60.541 as follows:

a. In paragraph (b), the definitions of the terms "D_c" and "D_r" are amended by revising the words "(grams per liter)" to read "(grams per liter (lb per gallon))."

b. In paragraph (b), the definitions of the terms "G" and "N" are amended by revising the words "(grams per tire)" to read "(grams (lb) per tire)."

c. In paragraph (b), the definitions of the terms "G_b" and "N_b" are amended by revising the words "(grams per bead)" to read "(grams (lb) per bead)."

d. In paragraph (b), the definitions of the terms "L_c" and "L_r" are amended by revising the word "(liters)" to read "(liters (gallons))."

e. In paragraph (b), the definitions of the terms "M", "M_o", and "M_r" are amended by revising the word "(grams)" to read "(grams (lb))."

f. In paragraph (b), the definitions of the terms "Q_a", "Q_b", and "Q_r" are amended by revising the words "(dry standard cubic meters per hour)" to read "(dry standard cubic meters (dry standard cubic feet) per hour)."

§ 60.542 [Amended]

170. Amend § 60.542 as follows:

a. Paragraphs (a)(1)(ii)(A) through (E), (a)(2)(ii)(A) through (E), (a)(6)(ii)(A) through (E), (a)(8)(ii)(A) through (E), and (a)(9)(ii)(A) through (E) are revised.

b. In paragraph (a)(3) by revising the words "no more than 10 grams of VOC per tire (g/tire)" to read "no more than 10 grams (0.022 lb) of VOC per tire."

c. In paragraph (a)(4) by revising the words "no more than 5 grams of VOC per bead (g/bead)" to read "no more than 5 grams (0.011 lb) of VOC per bead."

d. In paragraph (a)(5)(i) by revising the words "1.2 grams of VOC per tire" to read "1.2 grams (0.0026 lb) of VOC per tire."

e. In paragraph (a)(5)(ii) by revising the words "9.3 grams of VOC per tire" to read "9.3 grams (0.021 lb) of VOC per tire."

f. In paragraph (a)(7)(i) by revising the words "1.2 grams of VOC per tire" to read "1.2 grams (0.0026 lb) of VOC per tire."

g. In paragraph (a)(7)(ii) by revising the words "9.3 grams of VOC per tire" to read "9.3 grams (0.021 lb) of VOC per tire."

The revisions read as follows:

§ 60.542 Standards for volatile organic compounds.

(a) * * *

(1) * * *

(ii) * * *

(A) 3,870 kg (8,531 lb) of VOC per 28 days,

(B) 4,010 kg (8,846 lb) of VOC per 29 days,

(C) 4,150 kg (9,149 lb) of VOC per 30 days,

(D) 4,280 kg (9,436 lb) of VOC per 31 days, or

(E) 4,840 kg (10,670 lb) of VOC per 35 days.

* * * * *

(2) * * *

(ii) * * *

(A) 3,220 kg (7,099 lb) of VOC per 28 days,

(B) 3,340 kg (7,363 lb) of VOC per 29 days,

(C) 3,450 kg (7,606 lb) of VOC per 30 days,

(D) 3,570 kg (7,870 lb) of VOC per 31 days, or

(E) 4,030 kg (8,885 lb) of VOC per 35 days.

* * * * *

(6) * * *

(ii) * * *

(A) 3,220 kg (7,099 lb) of VOC per 28 days,

(B) 3,340 kg (7,363 lb) of VOC per 29 days,

(C) 3,450 kg (7,606 lb) of VOC per 30 days,

(D) 3,570 kg (7,870 lb) of VOC per 31 days, or

(E) 4,030 kg (8,885 lb) of VOC per 35 days.

* * * * *

(8) * * *

(ii) * * *

(A) 1,570 kg (3,461 lb) of VOC per 28 days,

(B) 1,630 kg (3,593 lb) of VOC per 29 days,

(C) 1,690 kg (3,726 lb) of VOC per 30 days,

(D) 1,740 kg (3,836 lb) of VOC per 31 days, or

(E) 1,970 kg (4,343 lb) of VOC per 35 days.

* * * * *

(9) * * *

(ii) * * *

(A) 1,310 kg (2,888 lb) of VOC per 28 days,

(B) 1,360 kg (2,998 lb) of VOC per 29 days,

(C) 1,400 kg (3,086 lb) of VOC per 30 days,

(D) 1,450 kg (3,197 lb) of VOC per 31 days, or

(E) 1,640 kg (3,616 lb) of VOC per 35 days.

* * * * *

§ 60.542a [Amended]

171. In § 60.542a, paragraph (a) is amended by revising the words "25 grams" to read "25 grams (0.055 lb)" wherever they occur.

§ 60.543 [Amended]

172. Amend § 60.543 as follows:

a. In paragraph (c), the first sentence is amended by deleting the abbreviation "(kg/mo)."

b. Paragraph (d) is amended by revising the words "the g/tire limit" to read "the VOC emission per tire limit."

c. Paragraph (e) is amended by revising the words "g/bead limit" to read "VOC emission per bead limit."

d. Paragraph (f) is amended by revising the words "operation that use" to read "operation that uses."

e. Paragraphs (f)(2)(iv)(G) and (f)(2)(iv)(H) are amended by revising the definitions of the terms "W", "V", "Q_i", and "M_i" following the equations as follows:

W = Molecular weight of the single VOC, mg/mg-mole (lb/lb-mole).

V = The volume occupied by one mole of ideal gas at standard conditions [20°C, 760 mm Hg] on a wet basis, 2.405×10^{-5} m^3/mg-mole (385.3 ft^3/lb-mole).

Q_i = Volumetric flow in the capture system during run i, on a wet basis, adjusted to standard conditions, m^3 (ft^3) (see § 60.547(a)(5)).

M_i = Mass of the single VOC used during run i, mg (lb).

f. Paragraphs (g) and (i) are amended by revising the words "operation that use" to read "operation that uses" wherever they occur.

g. Paragraphs (j)(4) and (j)(5)(ii) are amended by revising the words "100 feet per minute" to read "30.5 meters (100 feet) per minute" wherever they occur.

h. Paragraphs (n) and (n)(5) are amended by revising the words "25 g/tire limit" to read "VOC emission per tire limit" wherever they occur.

§ 60.544 [Amended]

173. In § 60.544, paragraph (a)(2) is amended by revising the word "temperatrue" to read "temperature."

§ 60.545 [Amended]

174. Amend § 60.545 as follows:

a. Paragraph (b) is amended by revising the words "28 °C" to read "28 °C (50 °F)."

b. Paragraph (d) is amended by revising the words "specified kg/mo uncontrolled VOC use" to read "specified VOC monthly usage."

c. Paragraph (f) is amended by revising the citation "§ 60.543(B)(4)" to read "§ 60.543(b)(4)."

§ 60.546 [Amended]

175. Amend § 60.546 as follows:

a. Paragraph (a) is amended by revising the words "green tires spraying operation where organic solvent-based spray are used" to read "green tire spraying operation where organic solvent-based sprays are used."

b. Paragraph (c)(1) is amended by revising the words "kg/mo uncontrolled VOC use" to read "VOC monthly usage."

c. Paragraph (c)(1) is amended by revising the words "the number days" to read "the number of days."

d. Paragraphs (c)(2), (c)(3), and (c)(5) are amended by revising the words "g/tire or g/bead limit" to read "VOC emission limit per tire or per bead" wherever they occur.

e. In paragraph (d), the second sentence is amended by revising the words "(kg/hr)" to read "(kg/hr or lb/hr)."

f. Paragraph (f)(1) is amended by revising the words "g/tire or g/bead limit" to read "VOC emission limit per tire or per bead."

g. Paragraph (f)(2) is amended by revising the words "kg/mo VOC use" to read "monthly VOC usage."

h. In paragraph (j), the second sentence is amended by revising the words "shall be reported within 30 days" to read "shall be reported within 30 days of the change."

§ 60.547 [Amended]

176. Amend § 60.547 as follows:

a. Paragraphs (a)(2) and (a)(5) are amended by revising the words "notify the Administrator 30 days in advance" to read "notify the Administrator at least 30 days in advance" wherever they occur.

b. Paragraphs (a)(2) and (a)(5) are amended by revising the words "1 meter" to read "1.0 meter (3.3 feet)" wherever they occur.

c. Paragraphs (a)(2) and (a)(5)(i) are amended by revising the words "0.003 dry standard cubic meter" to read "0.003 dry standard cubic meter (dscm) (0.11 dry standard cubic feet (dscf))" wherever they occur.

§ 60.560 [Amended]

177. Amend § 60.560 as follows:

a. Paragraph (a)(4)(i) is amended by revising the words "1,000 Mg/yr" to read "1,000 Mg/yr (1,102 ton/yr)."

b. In paragraph (b), Table 1 is revised to read as follows:

Polymer	Production process(es)	Process section	Emissions	
			Continuous	Intermittent
Polypropylene	Liquid Phase	Raw Materials Preparation	X	
		Polymerization Reaction	X	

Polymer	Production process(es)	Process section	Emissions	
			Continuous	Intermittent
		Material Recovery	X	X
		Product Finishing	X	
		Product Storage		
Polypropylene	Gas Phase	Raw Materials Preparation		
		Polymerization Reaction		X
		Material Recovery	X	
		Product Finishing		
		Product Storage		
Low Density Polyethylene	High Pressure	Raw Materials Preparation		X
		Polymerization Reaction		X
		Material Recovery		X
		Product Finishing		X
		Product Storage		X
Low Density Polyethylene High Density Polyethylene	Low Pressure Gas Phase	Raw Materials Preparation	X	X
		Polymerization Reaction		X
		Material Recovery		
		Product Finishing	X	
		Product Storage		
High Density Polyethylene	Liquid Phase Slurry	Raw Materials Preparation		X
		Polymerization Reaction		
		Material Recovery	X	
		Product Finishing	X	
		Product Storage		
High Density Polyethylene	Liquid Phase Solution.	Raw Materials Preparation	X	X
		Polymerization Reaction		X
		Material Recovery	X	X
		Product Finishing		
		Product Storage		

c. In paragraph (d), Table 2 is revised.

d. Paragraph (g) is amended by revising the words "1.6 Mg/yr" to read "1.6 Mg/yr (1.76 ton/yr)" wherever they occur.

The revision reads as follows:

§ 60.560 Applicability and designation of affected facilities.

* * * * * * *

(d) * * *

TABLE 2.—MAXIMUM UNCONTROLLED THRESHOLD EMISSION RATES[a]

Production process	Process section	Uncontrolled emission rate, kg TOC/Mg product (See associated footnote)
Polypropylene, liquid phase process	Raw Materials Preparation	0.15[b]
	Polymerization Reaction	0.14[b], 0.24[c]
	Material Recovery	0.19[b]
	Product Finishing	1.57[b]
Polypropylene, gas phase process	Polymerization Reaction	0.12[c]
	Material Recovery	0.02[b]
Low Density Polyethylene, low pressure process	Raw Materials Preparation	0.41[d]
	Polymerization Reaction	[e]
	Material Recovery	[e]
	Product Finishing	[e]
	Product Storage	[e]
Low Density Polythylene, low pressure process	Raw Materials Preparation	0.05[f]
	Polymerization Reaction	0.03[g]
	Product Finishing	0.01[b]
High Density Polyethylene, liquid phase slurry process	Raw Materials Preparation	0.25[c]
	Material Recovery	0.11[b]
	Product Finishing	0.41[b]
High Density Polyethylene, liquid phase solution process	Raw Materials Preparation	0.24[f]
	Polymerization Reaction	0.16[c]
	Material Recovery	1.68[f]
High Density Polyethylene, gas phase process	Raw Materials Preparation	0.05[f]
	Polymerization Reaction	0.03[g]

TABLE 2.—MAXIMUM UNCONTROLLED THRESHOLD EMISSION RATES [a]—Continued

Production process	Process section	Uncontrolled emission rate, kg TOC/Mg product (See associated footnote)
	Product Finishing	0.01 [b]
Polystyrene, continuous process	Material Recovery	0.05 [b, h]
Poly(ethylene terephalate), dimethyl terephthalate process	Material Recovery	0.12 [b h]
	Polymerization Reaction	1.80 [h i j.]
Poly(ethlyene terephthalate), terephthalic acid process	Raw Materials Preparation	(l)
	Polymerization Reaction	1.80 [h j m]
		3.92 [h k m]

[a] "Uncontrolled emission rate" refers to the emission rate of a vent stream that vents directly to the atmosphere and to the emission rate of a vent stream to the atmosphere that would occur in the absence of any add-on control devices but after any material recovery devices that constitute part of the normal material recovery operations in a process line where potential emissions are recovered for recycle or resale.

[b] Emission rate applies to continuous emissions only.

[c] Emission rate applies to intermittent emissions only.

[d] Total emission rate for non-emergency intermittent emissions from raw materials preparation, polymerization reaction, material recovery, product finishing, and product storage process sections.

[e] See footnote d.

[f] Emission rate applies to both continuous and intermittent emissions.

[g] Emission rate applies to non-emergency intermittent emissions only.

[h] Applies to modified or reconstructed affected facilities only.

[i] Includes emissions from the cooling water tower.

[j] Applies to a process line producing low viscosity poly(ethylene terephthlalate).

[k] Applies to a process line producing high viscosity poly(ethylene terephathalate).

[l] See footnote m.

[m] Applies to the sum of emissions to the atmosphere from the polymerization reaction section (including emissions from the cooling tower) and the raw materials preparation section (i.e., the esterifiers).

* * * * *

§ 60.561 [Amended]

178. Amend § 60.561 as follows:

a. The definition of "End finisher" is amended as revising the words "2 torr" in the first sentence to read "2 mm Hg (1 in. H_2O)"; and by revising the words "between 5 and 10 torr" in the second sentence to read "between 5 and 10 mm Hg (3 and 5 in. H_2O)."

b. The definition of "High density polyethylene (HDPE)" is amended by revising the words "0.940 g/cm^3" to read "0.940 gm/cm^{3}3 (58.7 lb/ft^3)."

c. The definition of "High pressure process" is amended by revising the words "15,000 psig" to read "15,000 psig (103,000 kPa gauge)."

d. The definition of "Low density polyethylene (LDPE)" is amended by revising the words "0.940 g/cm^3" to read "0.940 g/cm^3 (58.7 lb/ft^3)."

e. The definition of "Low pressure process" is amended by revising the words "300 psig" to read "300 psig (2,070 kPa gauge)."

§ 60.562–1 [Amended]

179. Amend § 60.562–1 as follows:

a. In paragraph (a)(1)(iii), the second sentence is amended by revising the words "18.2 Mg/yr" to read "18.2 Mg/yr (20.1 ton/yr)."

b. Paragraph (b)(1)(i) is amended by revising the words "0.0036 kg TOC/Mg" to read "0.0036 kg TOC/Mg (0.0072 lb TOC/ton)."

c. Paragraph (c)(1)(i)(A) is amended by revising the words "0.018 kg TOC/Mg" to read "0.018 kg TOC/Mg (0.036 lb TOC/ton)."

d. Paragraph (c)(1)(ii)(A) is amended by revising the words "0.02 kg TOC/Mg" to read "0.02 kg TOC/Mg (0.04 lb TOC/ton)."

e. Paragraph (c)(1)(ii)(C) is amended by inserting a comma after the word "weight".

f. Paragraph (c)(2)(i) is amended by revising the words "0.04 kg TOC/Mg" to read "0.04 kg TOC/Mg (0.08 lb TOC/ton)."

g. Paragraph (c)(2)(ii)(A) is amended by revising the words "0.02 kg TOC/Mg" to read "0.02 kg TOC/Mg (0.04 lb TOC/ton)."

h. Paragraph (c)(2)(ii)(C) is amended by inserting a comma after the word "weight".

§ 60.562–2 [Amended]

180. In § 60.562–2, paragraph (d) is amended by revising the words "150 °C as determined by ASTM Method D86–78" to read "150 °C (302 °F) as determined by ASTM Method D86–78, 82, 90, 95, or 96."

§ 60.564 [Amended]

181. Amend § 60.564 as follows:

a. In paragraph (c)(1), the definitions of the terms "E_{inlet}" and "E_{outlet}" are amended by revising the words "kg TOC/hr" to read "kg TOC/hr (lb TOC/hr)" wherever they occur.

b. In Paragraphs (d)(1), (f) introductory text, and (j)(1)(iv), the equations and definitions are revised; and paragraphs (g)(2) and (g)(3) are revised.

c. Paragraph (f)(1) is amended by revising "ASTM D1946–77" to read "ASTM D1946–77 or 90 (Reapproved 1994)."

d. Paragraph (f)(3) is amended by revising "ASTM D2382–76" to read "ASTM D2382–76 or 88 or D4809–95."

e. In paragraph (h) designate the second paragraph as (h)(1), redesignate existing paragraphs (h)(1) and (h)(2) as paragraphs (h)(2) and (h)(3) and revise the equations and definitions in newly redesignated paragraph (h)(1).

f. Paragraph (h)(3) is amended by revising the words "The rate of polymer produced, P_p (kg/hr), shall be determined by dividing the weight of polymer pulled in kilograms (kg) from the process line during the performance test by the number of hours (hr) taken to perform the performance test. The polymer pulled, in kilograms, shall" to read "The rate of polymer production, P_p, shall be determined by dividing the weight of polymer pulled (in kg (lb)) from the process line during the performance test by the number of hours taken to perform the performance test. The weight of polymer pulled shall."

g. Paragraph (j)(1) introductory text is amended by revising "ASTM D2908–74" to read "ASTM D2908–74 or 91."

h. Paragraph (j)(1)(i) is amended by revising "ASTM D3370–76" to read "ASTM D3370–76 or 96a."

The revisions read as follows:

§ 60.564 Test methods and procedures.

* * * * *

(d) * * *

(1)

$$E_{unc} = K_2 \left(\sum_{j=1}^{n} C_j M_j \right) Q \times 8{,}600$$

Where:

E_{unc} = uncontrolled annual emissions, Mg/yr (ton/yr)
C_j = concentration of sample component j of the gas stream, dry basis, ppmv
M_j = molecular weight of sample component j of the gas stream, g/g-mole (lb/lb-mole)
Q = flow rate of the gas stream, dscm/hr (dscf/hr)
$K_2 = 4.157 \times 10^{-11}$ [(Mg)(g-mole)]/[(g)(ppm)(dscm)] (metric units)
$= 1.298 \times 10^{-12}$ [(ton)(lb-mole)]/[(lb)(ppm)(dscf)] (English units)
8,600 = operating hours per year

* * * * *

(f) * * *

$$H_T = K_3 \left(\sum_{j=1}^{n} C_j J_j \right)$$

Where:

H_T = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of offgas is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F).
$K_3 = 1.74 \times 10^{-7}$ (1/ppm)(g-mole/scm)(MJ/kcal) (metric units), where standard temperature for (g-mole/scm) is 20°C.
$= 4.67 \times 10^{-6}$ (1/ppm)(lb-mole/scf)(Btu/kcal) (English units) where standard temperature for (lb/mole/scf) is 68 °F.
C_j = Concentration on a wet basis of compound j in ppm.
H_j = Net heat of combustion of compound j, kcal/(g-mole) (kcal/(lb-mole)), based on combustion at 25 °C and 760 mm Hg (77 °F and 30 in. Hg).

* * * * *

(g) * * *

(2) If applicable, the maximum permitted velocity (V_{max}) for steam-assisted and nonassisted flares shall be computed using the following equation:

$$\mathrm{Log}_{10}(V_{max}) = (H_T + K_4)/K_5$$

Where:

V_{max} = Maximum permitted velocity, m/sec (ft/sec)
K_4 = 28.8 (metric units), 1212 (English units)
K_5 = 31.7 (metric units), 850.8 (English units)
H_T = The net heating value as determined in paragraph (f) of this section, MJ/scm (Btu/scf).

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

$$V_{max} = K_6 + K_7 H_T$$

Where:

V_{max} = Maximum permitted velocity, m/sec (ft/sec).
K_6 = 8.706 m/sec (metric units)
= 28.56 ft/sec (English units)
K_7 = 0.7084 [(m/sec)/MJ/scm)] (metric units)
= 0.00245 [(ft/sec)/Btu/scf)] (English units)
H_T = The net heating value as determined in paragraph (f) of this section, MJ/scm (Btu/scf).

* * * * *

(h) * * *

(i) * * *

$$ER_{TOC} = K_5 \frac{E_{TOC}}{P_p}$$

Where:

ER_{TOC} = Emission rate of total organic compounds (minus methane and ethane), kg TOC/Mg (lb TOC/ton) product
E_{TOC} = Emission rate of total organic compounds (minus methane and ethane) in the sample, kg/hr (lb/hr)
P_p = The rate of polymer production, kg/hr (lb/hr)
K_5 = 1,000 kg/Mg (metric units)
= 2,000 lb/ton (English units)

* * * * *

(j) * * *

(1) * * *

(iv) * * *

$$CI_{95} = \frac{\sum_{i=1}^{n} X_i}{n} + 2\sqrt{\frac{n\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2}{n(n-1)}}$$

Where:

X_i = daily ethylene glycol concentration for each day used to calculate the 14-day rolling average used in test results to justify implementing the reduced testing program.
n = number of ethylene glycol concentrations.

* * * * *

§ 60.565 [Amended]

182. Amend § 60.565 as follows:

a. In paragraph (a)(1)(ii), the first sentence is amended by revising the words "kilograms TOC (minus methane and ethane) per megagram of product" to read "kg TOC (minus methane and ethane) per Mg (lb TOC/ton) of product."

b. In paragraph (a)(2)(ii) by revising the word "boiler" to read "boilers."

c. In paragraph (f)(1)(i) by removing the words "are exceeded."

§ 60.581 [Amended]

183. Amend § 60.581 as follows:

a. In paragraph (a), the definition of the term "ink solids" is amended by revising the words "Reference Method" to read "Method."

b. In paragraph (b), the definitions of the terms "W_{oi}", "W_{si}", and "W_{oj}" are amended by revising the words "Reference Method" to read "Method" wherever they occur.

§ 60.583 [Amended]

184. Amend § 60.583 as follows:

a. In paragraph (a) introductory text by revising the words "Reference Methods" to read "Methods."

b. In paragraphs (a)(1), (b)(4), (b)(5), (c)(2), (c)(3), and (c)(4) by revising the words "Reference Method" to read "Method" wherever they occur.

§ 60.584 [Amended]

185. Amend § 60.584 as follows:

a. In paragraphs (b)(1) and (c)(1) by revising the words "of ±0.75 percent of the temperature being measured or ±2.5° C" to read "of ±0.75 percent of the temperature being measured, expressed in degrees Celsius, or ±2.5° C."

b. In paragraph (b)(2) by revising the words "more than 28° C" to read "more than 28° C (50° F)."

§ 60.593 [Amended]

186. Amend § 60.593 as follows:

a. In paragraph (b)(2) by revising "ASTM E–260, E–168, or E–169" to read "ASTM E260–73, 91, or 96, E168–67, 77, or 92, or E169–63, 77, or 93."

b. In paragraph (d) by revising "ASTM Method D86" to read "ASTM Method D86–78, 82, 90, 95, or 96."

§ 60.600 [Amended]

187. In § 60.600, paragraph (a) is amended by revising the words "500 megagrams" to read "500 Mg (551 ton)."

§ 60.602 [Amended]

188. Amend § 60.602 as follows:

a. By removing the paragraph designation "(a)".

b. In the first sentence, by revising the words "10 kilograms (kg) VOC per megagram (Mg)" to read "10 kg/Mg (20 lb/ton)."

c. In the second sentence, by revising the words "10 kg VOC per Mg" to read "10 kg/Mg (20 lb/ton)."

d. In the third sentence by revising the words "17 kg VOC per Mg" to read "17 kg/Mg (34 lb/ton)."

§ 60.603 [Amended]

189. Amend § 60.603 as follows:

a. In paragraph (b) introductory text, the first sentence is amended by revising the words "VOC emissions per Mg solvent feed" to read "VOC emissions per unit mass solvent feed."

b. In paragraph (b)(2) by revising the second equation and by revising the definitions following the equations.

c. Paragraph (b)(2)(i) is redesignated as paragraph (b)(3), and newly redesignated paragraph (b)(3) is amended by revising the words "13 kg per Mg solvent feed" to read "13 kg/Mg (26 lb/ton) solvent feed."

The revisions read as follows:

§ 60.603 Performance test and compliance provisions.

* * * * *

(b) * * *

(2) * * *

$$S_W = \frac{S_V S_P D}{K}$$

E = VOC Emissions, in kg/Mg (lb/ton) solvent;

S_V = Measured or calculated volume of solvent feed, in liters (gallons);

S_W = Weight of solvent feed, in Mg (ton);

M_V = Measured volume of makeup solvent, in liters (gallons);

M_W = Weight of makeup, in kg (lb);

N = Allowance for nongaseous losses, 13 kg/Mg (26 lb/ton) solvent feed;

S_P = Fraction of measured volume that is actual solvent (excludes water);

D = Density of the solvent, in kg/liter (lb/gallon);

K = Conversion factor, 1,000 kg/Mg (2,000 lb/ton);

I = Allowance for solvent inventory variation or changes in the amount of solvent contained in the affected facility, in kg/Mg (lb/ton) solvent feed (may be positive or negative);

I_S = Amount of solvent contained in the affected facility at the beginning of the test period, as determined by the owner or operator, in kg (lb);

I_E = Amount of solvent contained in the affected facility at the close of the test period, as determined by the owner or operator, in kg (lb).

* * * * *

§ 60.604 [Amended]

190. In § 60.604, paragraph (b) is amended by revising the words "500 megagrams" to read "500 Mg (551 ton)" wherever they occur.

§ 60.613 [Amended]

191. Amend § 60.613 as follows:

a. In paragraph (c) introductory text by revising the words "in the following equipment" to read "the following equipment."

b. Paragraphs (d) and (e) are redesignated as (e) and (f).

c. Paragraph (c)(3) is redesignated as paragraph (d).

§ 60.614 [Amended]

192. Amend § 60.614 as follows:

a. In paragraph (b)(4)(ii), the definitions of the terms "E_i" and "E_o" are amended by revising the term "kg TOC/hr" to read "kg/hr (lb/hr)."

b. In paragraph (b)(4)(iii), the definition of the terms "Q_i, Q_o" is amended by revising the units "dscf/hr" to read "dscf/min."

c. In paragraph (b)(4)(iii), the definition of the term "K_2" is revised.

d. Paragraphs (b)(5), (c), (d), (e), and (f) are redesignated as paragraphs (c), (d), (e), (f), and (g), respectively.

e. In newly redesignated paragraph (e)(1)(i), the second sentence is amended by revising "§ 60.614(d)(2) and (3)" to read "§ 60.614(e)(2) and (3)" and by revising the section reference "(d)(1)(ii)" to read "(e)(1)(ii)."

f. In newly redesignated paragraph (e)(1)(i), the last sentence is amended by revising the words "4 inches" to read "10 centimeters (4 inches)."

g. In newly redesignated paragraph (e)(1)(ii)(C), the second sentence is amended by revising "§ 60.614(d)(4) and (5)" to read "§ 60.614(e)(4) and (5)."

h. Newly redesignated paragraph (e)(2)(ii) is amended by revising "ASTM D1946–77" to read "D1946–77, or 90 (Reapproved 1994)."

i. In newly redesignated paragraphs (e)(4) and (e)(5), the definitions of the equation terms are revised.

j. Newly redesignated paragraphs (f)(1)(i), including Table 1, and (f)(1)(ii) are revised.

k. In newly redesignated paragraph (f)(2) the definitions of the equation terms and Table 2 are revised.

The revisions read as follows:

§ 60.614 Test methods and procedures.

* * * * *

(b) * * *

(4) * * *

(iii) * * *

K_2 = 2.494×10^{-6} (1/ppm)(g-mole/scm)(kg/g)(min/hr) (metric units), where standard temperature for (g-mole/scm) is 20°C.

= 1.557×10^{-7} (1/ppm)(lb-mole/scf)(min/hr) (English units), where standard temperature for (lb-mole/scf) is 68°F.

* * * * *

(e) * * *

(4) * * *

H_T = Net heating value of the sample, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25°C and 760 mm Hg (77°F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20°C (68°F).

K_1 = 1.74×10^{-7} (1/ppm)(g-mole/scm)(MJ/kcal) (metric units), where standard temperature for (g-mole/scm) is 20°C.

= 1.03×10^{-11} (1/ppm)(lb-mole/scf)(Btu/kcal) (English units) where standard temperature for (lb/mole/scf) is 68°F.

C_j = Concentration on a wet basis of compound j in ppm, as measured for organics by Method 18 and measured for hydrogen and carbon monoxide by ASTM D1946–77, 90, or 94 (incorporation by reference as specified in § 60.17 of this part) as indicated in § 60.614(e)(2).

H_j = Net heat of combustion of compound j, kcal/(g-mole) [kcal/(lb-mole)], based on combustion at 25°C and 760 mm Hg (77 °F and 30 in. Hg).

(5) * * *

E_{TOC} = Measured emission rate of TOC, kg/hr (lb/hr).

K_2 = 2.494×10^{-6} (1/ppm)(g-mole/scm)(kg/g)(min/hr) (metric units), where standard temperature for (g-mole/scm) is 20°C.

= 1.557×10^{-7} (1/ppm)(lb-mole/scf)(min/hr) (English units), where standard temperature for (lb-mole/scf) is 68°F.

C_j = Concentration on a wet basis of compound j in ppm, as measured by Method 18 as indicated in § 60.614(e)(2).

M_j = Molecular weight of sample j, g/g-mole (lb/lb-mole).

Q_s = Vent stream flow rate, scm/hr (scf/hr), at a temperature of 20°C (68°F).

* * * * *

(f) * * *

(1) * * *

(i) Where for a vent stream flow rate that is greater than or equal to 14.2 scm/min (501 scf/min) at a standard temperature of 20 °C (68 °F):

TRE = TRE index value.

Q_s = Vent stream flow rate, scm/min (scf/min), at a temperature of 20°C (68 °F).

H_T = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25°C and 760 mm Hg (68°F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20°C (68°F) as in the definition of Q_s.

$Y_s = Q_s$ for all vent stream categories listed in Table 1 except for Category E vent streams where $Y_s = Q_sH_T/3.6$.

E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr). a, b, c, d, e, and f are coefficients.

The set of coefficients which apply to a vent stream shall be obtained from Table 1.

BILLING CODE 6560–50–P

TABLE 1. AIR OXIDATION NSPS TRE COEFFICIENTS FOR VENT STREAMS CONTROLLED BY AN INCINERATOR

DESIGN CATEGORY A1. FOR HALOGENATED PROCESS VENT STREAMS, IF 0 ≤ NET HEATING VALUE (MJ/scm) ≤ 3.5 OR IF 0 ≤ NET HEATING VALUE (Btu/scf) ≤ 94:

Q_S = Vent Stream Flow rate scm/min (scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 18.8$	19.18370	0.27580	0.75762	-0.13064	0	0.01025
$(501 \le Q_S \le 664)$	(42.29238)	(0.017220)	(0.072549)	(-0.00030361)	(0)	(0.003803)
$18.8 < Q_S \le 699$	20.00563	0.27580	0.30387	-0.13064	0	0.01025
$(664 < Q_S \le 24{,}700)$	(44.10441)	(0.017220)	(0.029098)	(-0.00030361)	(0)	(0.003803)
$699 < Q_S \le 1400$	39.87022	0.29973	0.30387	-0.13064	0	0.01449
$(24{,}700 < Q_S \le 49{,}000)$	(87.89789)	(0.018714)	(0.029098)	(-0.00030361)	(0)	(0.005376)
$1400 < Q_S \le 2100$	59.73481	0.31467	0.30387	-0.13064	0	0.01775
$(49{,}000 < Q_S \le 74{,}000)$	(131.6914)	(0.019647)	(0.029098)	(-0.00030361)	(0)	(0.006585)
$2100 < Q_S \le 2800$	79.59941	0.32572	0.30387	-0.13064	0	0.02049
$(74{,}000 < Q_S \le 99{,}000)$	(175.4849)	(0.020337)	(0.029098)	(-0.00030361)	(0)	(0.007602)
$2800 < Q_S \le 3500$	99.46400	0.33456	0.30387	-0.13064	0	0.02291
$(99{,}000 < Q_S \le 120{,}000)$	(219.2783)	(0.020888)	(0.029098)	(-0.00030361)	(0)	(0.008500)

DESIGN CATEGORY A2. FOR HALOGENATED PROCESS VENT STREAMS, IF NET HEATING VALUE < 3.5 (MJ/scm) OR IF NET HEATING VALUE < 94 (Btu/scf):

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 18.8$	18.84466	0.26742	-0.20044	0	0	0.01025
$(501 \le Q_S \le 664)$	(41.54494)	(0.016696)	(-0.019194)	(0)	(0)	(0.003803)
$18.8 < Q_S \le 699$	19.66658	0.26742	-0.25332	0	0	0.01025
$(664 < Q_S \le 24{,}700)$	(43.35694)	(0.016696)	(-0.024258)	(0)	(0)	(0.003803)
$699 < Q_S \le 1400$	39.19213	0.29062	-0.25332	0	0	0.01449
$(24{,}700 < Q_S \le 49{,}000)$	(86.40297)	(0.018145)	(-0.024258)	(0)	(0)	(0.005376)
$1400 < Q_S \le 2100$	58.71768	0.30511	-0.25332	0	0	0.01775
$(49{,}000 < Q_S \le 74{,}000)$	(129.4490)	(0.019050)	(-0.024258)	(0)	(0)	(0.006585)
$2100 < Q_S \le 2800$	78.24323	0.31582	-0.25332	0	0	0.02049
$(74{,}000 < Q_S \le 99{,}000)$	(172.4950)	(0.019718)	(-0.024258)	(0)	(0)	(0.007602)
$2800 < Q_S \le 3500$	97.76879	0.32439	-0.25332	0	0	0.02291
$(99{,}000 < Q_S \le 120{,}000)$	(215.5411)	(0.020253)	(-0.024258)	(0)	(0)	(0.008500)

DESIGN CATEGORY B. FOR NONHALOGENATED PROCESS VENT STREAMS, IF 0 ≤ NET HEATING VALUE (MJ/scm) ≤ 0.48 OR IF 0 ≤ NET HEATING VALUE (Btu/scf) ≤ 13:

Q_s = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \leq Q_s \leq 1340$	8.54245	0.10555	0.09030	-0.17109	0	0.01025
$(501 \leq Q_s \leq 47{,}300)$	(18.83268)	(0.0065901)	(0.008647)	(-0.00039762)	(0)	(0.003803)
$1340 < Q_s \leq 2690$	16.94386	0.11470	0.09030	-0.17109	0	0.01449
$(47{,}300 < Q_s \leq 95{,}000)$	(37.35443)	(0.0071614)	(0.008647)	(-0.00039762)	(0)	(0.005376)
$2690 < Q_s \leq 4040$	25.34528	0.12042	0.09030	-0.17109	0	0.01775
$(95{,}000 < Q_s \leq 143{,}000)$	(55.87620)	(0.0075185)	(0.008647)	(-0.00039762)	(0)	(0.00658

DESIGN CATEGORY C. FOR NONHALOGENTED PROCESS VENT STREAMS, IF 0.48 < NET HEATING VALUE (MJ/scm) ≤ 1.9 OR IF 13 < NET HEATING VALUE (Btu/scf) ≤ 51:

Q_s = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \leq Q_s \leq 1340$	9.25233	0.06105	0.31937	-0.16181	0	0.01025
$(501 \leq Q_s \leq 47{,}300)$	(20.39769)	(0.003812)	(0.030582)	(-0.00037605)	(0)	(0.003803)
$1340 < Q_s \leq 2690$	18.36363	0.06635	0.31937	-0.16181	0	0.01449
$(47{,}300 < Q_s \leq 95{,}000)$	(40.48446)	(0.004143)	(0.030582)	(-0.00037605)	(0)	(0.005376)
$2690 < Q_s \leq 4040$	27.47492	0.06965	0.31937	-0.16181	0	0.01775
$(95{,}000 < Q_s \leq 143{,}000)$	(60.57121)	(0.004349)	(0.030582)	(-0.00037605)	(0)	(0.006585)

DESIGN CATEGORY D. FOR NONHALOGENATED PROCESS VENT STREAMS, IF 1.9 < NET HEATING VALUE (MJ/scm) ≤ 3.6 OR IF 51 < NET HEATING VALUE (Btu/scf) ≤ 97:

Q_s = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \leq Q_s \leq 1180$	6.67868	0.06943	0.02582	0	0	0.01025
$(501 \leq Q_s \leq 41{,}700)$	(14.72382)	(0.004335)	(0.002472)	(0)	(0)	(0.003803)
$1180 < Q_s \leq 2370$	13.21633	0.07546	0.02582	0	0	0.01449
$(41{,}700 < Q_s \leq 83{,}700)$	(29.13672)	(0.004711)	(0.002472)	(0)	(0)	(0.005376)
$2370 < Q_s \leq 3550$	19.75398	0.07922	0.02582	0	0	0.01775
$(83{,}700 < Q_s \leq 125{,}000)$	(43.54962)	(0.004946)	(0.002472)	(0)	(0)	(0.00658

Q_s = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \leq Y_s \leq 1180$	6.67868	0	0	-0.00707	0.02220	0.01025
$(501 \leq Y_s \leq 41{,}700)$	(14.72382)	(0)	(0)	(-0.0000164)	(0.0001174)	(0.003803)
$1180 < Y_s \leq 2370$	13.21633	0	0	-0.00707	0.02412	0.01449
$(41{,}700 < Y_s \leq 83{,}700)$	(29.13672)	(0)	(0)	(-0.0000164)	(0.0001276)	(0.005376)
$2370 < Y_s \leq 3550$	19.75398	0	0	-0.00707	0.02533	0.01775
$(83{,}700 < Y_s \leq 125{,}000)$	(43.54962)	(0)	(0)	(-0.0000164)	(0.0001340)	(0.006585)

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(ii) Where for a vent stream flow rate that is less than 14.2 scm/min (501 scf/min) at a standard temperature of 20°C (68°F):

TRE = TRE index value.
Q_s = 14.2 scm/min (501 scf/min).
H_T = (FLOW)(HVAL)/Q_s.

Where the following inputs are used:

FLOW = Vent stream flow rate, scm/min (scf/min), at a temperature of 20 °C (68 °F).
HVAL = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F) as in the definition of Q_s.
Y_s = Q_s for all vent stream categories listed in Table 1 except for Category E vent streams where $Y_s = Q_s H_T/3.6$.
E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr).

a, b, c, d, e, and f are coefficients.

The set of coefficients that apply to a vent stream can be obtained from Table 1.

(2) * * *

TRE = TRE index value.
E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr).
Q_s = Vent stream flow rate, scm/min (scf/min), at a standard temperature of 20 °C (68 °F).
H_T = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F) as in the definition of Q_s.

a, b, c, d, and e are coefficients.

* * * * *

TABLE 2.—AIR OXIDATION PROCESSES NSPS TRE COEFFICIENTS FOR VENT STREAMS CONTROLLED BY A FLARE

	a	b	c	d	e
$H_T < 11.2$ MJ/scm	2.25	0.288	−0.193	(−0.0051	2.08
($H_T < 301$ Btu/scf)	(0.140)	(0.0367)	(−0.000448)	(−0.0051)	(4.59)
$H_T \geq 11.2$ MJ/scm	0.309	0.0619	−0.0043	−0.0034	2.08
$H_T \geq 301$ Btu/scf)	(0.0193)	(0.00788)	(−0.000010)	(−0.0034)	(4.59)

* * * * *

§ 60.615 [Amended]

193. Amend § 60.615 as follows:

a. In paragraph (e), the first sentence is amended by revising the words "44 MW" to read "44 MW (150 million Btu/hour)."

b. In paragraph (g), the first sentence is amended by revising "§ 60.613(c)" to read "§ 60.613(e)."

§ 60.620 [Amended]

194. In § 60.620, paragraph (b), the second sentence is amended by revising the words "4,700 gallons" to read "17,791 liters (4,700 gallons)."

§ 60.624 [Amended]

195. In § 60.624, the third sentence is amended by revising the words "is from the outlet" to read "is the outlet."

§ 60.632 [Amended]

196. Amend § 60.632 as follows:

a. In paragraph (f), the second sentence is amended by revising the words "percent VOC content" to read "VOC content."

b. Paragraph (f) is amended by revising "ASTM Methods E169, E168, or E260" to read "ASTM E169–63, 77, or 93, E168–67, 77, or 92, or E260–73, 91, or 96."

§ 60.633 [Amended]

197. Amend § 60.633 as follows:

a. Paragraph (b)(4)(i) is amended by revising "§ 60.482–(b)(1)" to read "§ 60.482–4(b)(1)."

b. Paragraph (d) is amended by revising the words "283,000 standard cubic meters per day (scmd) (10 million standard cubic feet per day (scfd))" to read "283,200 standard cubic meters per day (10 million standard cubic feet per day)."

c. Paragraphs (h)(1) and (2) are amended by revising the words "at 150 °C" to read "at 150 °C (302 °F)."

d. Paragraphs (h)(1) and (2) are amended by revising the words "ASTM Method D86" to read "ASTM Method D86–78, 82, 90, 95, or 96."

§ 60.641 [Amended]

198. Amend § 60.641 as follows:

a. The definition for "Total SO_2" is amended by revising the words "(ppmv or kg/DSCM)" to read "(ppmv or kg/dscm (lb/dscf))."

b. The definitions for "E", "S", and "X" are amended to read as follows:

§ 60.641 Definitions.

* * * * *

E = The sulfur emission rate expressed as elemental sulfur, kilograms per hour (kg/hr) [pounds per hour (lb/hr)], rounded to one decimal place.

* * * * *

S = The sulfur production rate, kilograms per hour (kg/hr) [pounds per hour (lb/hr)], rounded to one decimal place.
X = The sulfur feed rate from the sweetening unit (i.e., the H_2S in the acid gas), expressed as sulfur, Mg/D(LT/D), rounded to one decimal place.

* * * * *

§ 60.644 [Amended]

199. Amend § 60.644 as follows:

a. Paragraphs (b)(1), (c)(3), and (c)(4)(iii) are revised.

b. In paragraph (b)(2), the first sentence is amended by revising the words "dscf/day" to read "dscm/day (dscf/day)."

c. In paragraph (c)(2), the second sentence is amended by revising the words "kg/hr" to read "kg/hr (lb/hr)."

d. In the paragraph (c)(4) introductory text, the first sentence is revised.

e. Paragraph (c)(4)(i) is amended by deleting the words "in mg/dscm" in the third sentence and by revising the last sentence.

f. In paragraph (c)(4)(ii), the last sentence is revised.

g. In paragraph (c)(4)(iv), the fifth sentence is amended by revising the words "(0.35 dscf)" to read "(3.5 dscf)."

h. Paragraph (d) is amended by revising the words "(b) of (c)" to read "(b) or (c)."

The revisions read as follows:

§ 60.644 Test methods and procedures.

* * * * *

(b) * * *

(1) The average sulfur feed rate (X) shall be computed as follows:

$$X = KQ_aY$$

Where:

X = average sulfur feed rate, Mg/D (LT/D).
Q_a = average volumetric flow rate of acid gas from sweetening unit, dscm/day (dscf/day).
Y = average H_2S concentration in acid gas feed from sweetening unit, percent by volume, expressed as a decimal.

K = (32 kg S/kg-mole)/((24.04 dscm/kg-mole)(1000 kg S/ Mg)) = 1.331 × 10^{-3} Mg/dscm, for metric units
= (32 lb S/lb-mole)/((385.36 dscf/lb-mole)(2240 lb S/long ton))
= 3.707 × 10^{-5} long ton/dscf, for English units.

* * * * *

(c) * * *

(3) The emission rate of sulfur shall be computed for each run as follows:

$$E = C_e Q_{sd} / K_1$$

Where:

E = emission rate of sulfur per run, kg/hr.
C_e = concentration of sulfur equivalent (SO_2 + reduced sulfur), g/dscm (lb/dscf).
Q_{sd} = volumetric flow rate of effluent gas, dscm/hr (dscf/hr).
K_1 = conversion factor, 1000 g/kg (7000 gr/lb).

(4) The concentration of sulfur equivalent (C_e) shall be the sum of the SO_2 and reduced sulfur concentrations, after being converted to sulfur equivalents. * * *

(i) * * * The concentration shall be multiplied by 0.5 × 10^{-3} to convert the results to sulfur equivalent.

(ii) * * * The concentration in ppm reduced sulfur as sulfur shall be multiplied by 1.333 × 10^{-3} to convert the results to sulfur equivalent.

(iii) Method 16A or 15 shall be used to determine the reduced sulfur concentration from oxidation-type devices or where the oxygen content of the effluent gas is greater than 1.0 percent by volume. Eight samples of 20 minutes each shall be taken at 30-minute intervals. The arithmetic average shall be the concentration for the run. The concentration in ppm reduced sulfur as sulfur shall be multiplied by 1.333 × 10^{-3} to convert the results to sulfur equivalent.

* * * * *

§ 60.646 [Amended]

200. Amend § 60.646 as follows:

a. In paragraph (b)(1), the second sentence is amended by revising the words "(kg/hr)" to read "(kg/hr (lb/hr))."

b. In paragraph (c), the second sentence is amended by revising the words "(kg/hr)" to read "(kg/hr (lb/hr))."

c. In paragraph (e), the first sentence is amended by revising the words "150 LT/D" to read "152 Mg/D (150 LT/D)."

d. In paragraph (e), the equation and definitions are amended by revising as follows:

§ 60.646 Monitoring of emissions and operations.

* * * * *

(e) * * *

$$R = \frac{K_2 S}{X}$$

Where:

R = The sulfur dioxide removal efficiency achieved during the 24-hour period, percent.
K_2 = Conversion factor, 0.02400 Mg/D per kg/hr (0.01071 LT/D per lb/hr).
S = The sulfur production rate during the 24-hour period, kg/hr (lb/hr).
X = The sulfur feed rate in the acid gas, Mg/D (LT/D).

* * * * *

§ 60.663 [Amended]

201. Amend § 60.663 as follows:

a. In paragraph (c) introductory text by revising the words "in the following equipment" to read "the following equipment."

b. Paragraphs (d) and (e) are redesignated as (e) and (f) and paragraph (c)(3) is redesignated as paragraph (d).

c. In newly redesignated paragraph (f) by revising the words "carbon absorber" to read "carbon adsorber."

§ 60.664 [Amended]

202. Amend § 60.664 as follows:

a. In paragraph (b)(4)(ii), the definitions of the terms "E_i" and "E_o" are amended by revising the term "kg TOC/hr" to read "kg/hr (lb/hr)."

b. In paragraph (b)(4)(iii), the definitions of the terms "Q_i" and "Q_o" are amended by revising the units "dscf/hr" to read "dscf/min."

c. In paragraph (b)(4)(iii), the definition of the term "K_2" is revised.

d. Paragraphs (b)(5), (c), (d), (e), (f), and (g) are redesignated as paragraphs (c), (d), (e), (f), (g), and (h), respectively.

e. In newly redesignated paragraph (e)(1)(i), the second sentence is amended by revising "§ 60.664(d)(2) and (3)" to read "§ 60.664(e)(2) and (3)."

f. In newly redesignated paragraph (e)(1)(i), the second sentence is amended by revising "(d)(1)(ii)" to read "(e)(1)(ii)."

g. In newly redesignated paragraph (e)(1)(i), the third sentence is amended by revising the words "4 inches" to read "10 centimeters (4 inches)."

h. In newly redesignated paragraph (e)(1)(ii)(C), the second sentence is amended by revising "§ 60.664(d)(4) and (5)" to read "§ 60.664(e)(4) and (5)."

i. Newly redesignated paragraph (e)(2)(ii) is amended by revising "ASTM D1946–77" to read "ASTM D1946–77 or 90 (Reapproved 1994)."

j. In newly redesignated paragraphs (e)(4), (e)(5) and (f)(2), the equation definitions are revised; and newly redesignated paragraphs (f)(1)(i), (f)(1)(ii) including Table 1, and Table 2 of (f)(2)are revised.

k. The last sentence in the newly redesignated paragraph (e)(4) is amended by revising "ASTM D2382–76" to read "ASTM D2382–76 or 88 or D4809–95."

The revisions read as follows:

§ 60.664 Test methods and procedures.

* * * * *

(b) * * *
(4) * * *
(iii) * * *

K_2 = 2.494 × 10^{-6} (1/ppm)(g-mole/scm) (kg/g) (min/hr) (metric units), where standard temperature for (g-mole/scm) is 20 °C.
= 1.557 × 10^{-7} (1/ppm) (lb-mole/scf) (min/hr) (English units), where standard temperature for (lb-mole/scf) is 68 °F.

* * * * *

(e) * * *
(4) * * *

H_T = Net heating value of the sample, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (77 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F).
K_1 = 1.74 × 10^{-7} (1/ppm) (g-mole/scm) (MJ/kcal) (metric units), where standard temperature for (g-mole/scm) is 20 °C.
= 1.03 × 10^{-11} (1/ppm) (lb-mole/scf) (Btu/kcal) (English units) where standard temperature for (lb/mole/scf) is 68 °F.
C_j = Concentration on a wet basis of compound j in ppm, as measured for organics by Method 18 and measured for hydrogen and carbon monoxide by ASTM D1946–77 or 90 (Reapproved 1994) (incorporation by reference as specified in § 60.17 of this part) as indicated in § 60.664(e)(2).
H_j = Net heat of combustion of compound j, kcal/(g-mole) [kcal/(lb-mole)], based on combustion at 25 °C and 760 mm Hg (77 °F and 30 in. Hg).

* * * * *

(5) * * *

E_{TOC} = Measured emission rate of TOC, kg/hr (lb/hr).
K_2 = 2.494 x 10^{-6} (1/ppm) (g-mole/scm) (kg/g) (min/hr) (metric units), where standard temperature for (g-mole/scm) is 20 °C.
= 1.557 × 10^{-7} (1/ppm) (lb-mole/scf) (min/hr) (English units), where

standard temperature for (lb-mole/scf) is 68 °F.

C_j = Concentration on a wet basis of compound j in ppm, as measured by Method 18 as indicated in § 60.664(e)(2).

M_j = Molecular weight of sample j, g/g-mole (lb/lb-mole).

Q_s = Vent stream flow rate, scm/min (scf/min), at a temperature of 20 °C (68 °F).

* * * * *

(f) * * *

(1) * * *

(i) Where for a vent stream flow rate that is greater than or equal to 14.2 scm/min (501 scf/min) at a standard temperature of 20 °C (68 °F):

TRE = TRE index value.

Q_s = Vent stream flow rate, scm/min (scf/min), at a temperature of 20 °C (68 °F).

H_T = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F) as in the definition of Q_s.

Y_s = Q_s for all vent stream categories listed in Table 1 except for Category E vent streams where $Y_s = Q_sH_T/3.6$.

E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr).

a, b, c, d, e, and f are coefficients.

The set of coefficients that apply to a vent stream can be obtained from Table 1.

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TABLE 1. DISTILLATION NSPS TRE COEFFICIENTS FOR VENT STREAMS CONTROLLED BY AN INCINERATOR

DESIGN CATEGORY A1. FOR HALOGENATED PROCESS VENT STREAMS, IF 0 ≤ NET HEATING VALUE (MJ/scm) ≤ 3.5 OR IF 0 ≤ NET HEATING VALUE (Btu/scf) ≤ 94:

Q_S = Vent Stream Flow rate scm/min (scf/min)	a	b	c	d	e	f
$14.2 \leq Q_S \leq 18.8$	18.84466	0.26742	-0.20044	0	0	0.01025
$(501 \leq Q_S \leq 664)$	(41.54494)	(0.016696)	(-0.019194)	(0)	(0)	(0.003803)
$18.8 < Q_S \leq 699$	19.66658	0.26742	-0.25332	0	0	0.01025
$(664 < Q_S \leq 24{,}700)$	(43.35694)	(0.016696)	(-0.024258)	(0)	(0)	(0.003803)
$699 < Q_S \leq 1400$	39.19213	0.29062	-0.25332	0	0	0.01449
$(24{,}700 < Q_S \leq 49{,}000)$	(86.40297)	(0.018145)	(-0.024258)	(0)	(0)	(0.005376)
$1400 < Q_S \leq 2100$	58.71768	0.30511	-0.25332	0	0	0.01775
$(49{,}000 < Q_S \leq 74{,}000)$	(129.4490)	(0.019050)	(-0.024258)	(0)	(0)	(0.006585)
$2100 < Q_S \leq 2800$	78.24323	0.31582	-0.25332	0	0	0.02049
$(74{,}000 < Q_S \leq 99{,}000)$	(172.4950)	(0.019718)	(-0.024258)	(0)	(0)	(0.007602)
$2800 < Q_S \leq 3500$	97.76879	0.32439	-0.25332	0	0	0.02291
$(99{,}000 < Q_S \leq 120{,}000)$	(215.5411)	(0.020253)	(-0.024258)	(0)	(0)	(0.008500)

DESIGN CATEGORY A2. FOR HALOGENATED PROCESS VENT STREAMS, IF NET HEATING VALUE < 3.5 (MJ/scm) OR IF NET HEATING VALUE < 94 (Btu/scf):

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 18.8$	18.84466	0.26742	-0.20044	0	0	0.01025
$(501 \le Q_S \le 664)$	(41.54494)	(0.016696)	(-0.019194)	(0)	(0)	(0.003803)
$18.8 < Q_S \le 699$	19.66658	0.26742	-0.25332	0	0	0.01025
$(664 < Q_S \le 24{,}700)$	(43.35694)	(0.016696)	(-0.024258)	(0)	(0)	(0.003803)
$699 < Q_S \le 1400$	39.19213	0.29062	-0.25332	0	0	0.01449
$(24{,}700 < Q_S \le 49{,}000)$	(86.40297)	(0.018145)	(-0.024258)	(0)	(0)	(0.005376)
$1400 < Q_S \le 2100$	58.71768	0.30511	-0.25332	0	0	0.01775
$(49{,}000 < Q_S \le 74{,}000)$	(129.4490)	(0.019050)	(-0.024258)	(0)	(0)	(0.006585)
$2100 < Q_S \le 2800$	78.24323	0.31582	-0.25332	0	0	0.02049
$(74{,}000 < Q_S \le 99{,}000)$	(172.4950)	(0.019718)	(-0.024258)	(0)	(0)	(0.007602)
$2800 < Q_S \le 3500$	97.76879	0.32439	-0.25332	0	0	0.02291
$(99{,}000 < Q_S \le 120{,}000)$	(215.5411)	(0.020253)	(-0.024258)	(0)	(0)	(0.008500)

DESIGN CATEGORY B. FOR NONHALOGENATED PROCESS VENT STREAMS, IF $0 \le$ NET HEATING VALUE (MJ/scm) ≤ 0.48 OR IF $0 \le$ NET HEATING VALUE (Btu/scf) ≤ 13:

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 1340$	8.54245	0.10555	0.09030	-0.17109	0	0.01025
$(501 \le Q_S \le 47{,}300)$	(18.83268)	(0.0065901)	(0.008647)	(-0.00039762)	(0)	(0.003803)
$1340 < Q_S \le 2690$	16.94386	0.11470	0.09030	-0.17109	0	0.01449
$(47{,}300 < Q_S \le 95{,}000)$	(37.35443)	(0.0071614)	(0.008647)	(-0.00039762)	(0)	(0.005376)
$2690 < Q_S \le 4040$	25.34528	0.12042	0.09030	-0.17109	0	0.01775
$(95{,}000 < Q_S \le 143{,}000)$	(55.87620)	(0.0075185)	(0.008647)	(-0.00039762)	(0)	(0.006585)

DESIGN CATEGORY C. FOR NONHALOGENTED PROCESS VENT STREAMS, IF 0.48 < NET HEATING VALUE (MJ/scm) ≤ 1.9 OR IF 13 < NET HEATING VALUE (Btu/scf) ≤ 51:

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 1340$	9.25233	0.06105	0.31937	-0.16181	0	0.01025
$(501 \le Q_S \le 47{,}300)$	(20.39769)	(0.003812)	(0.030582)	(-0.00037605)	(0)	(0.003803)
$1340 < Q_S \le 2690$	18.36363	0.06635	0.31937	-0.16181	0	0.01449
$(47{,}300 < Q_S \le 95{,}000)$	(40.48446)	(0.004143)	(0.030582)	(-0.00037605)	(0)	(0.005376)
$2690 < Q_S \le 4040$	27.47492	0.06965	0.31937	-0.16181	0	0.01775
$(95{,}000 < Q_S \le 143{,}000)$	(60.57121)	(0.004349)	(0.030582)	(-0.00037605)	(0)	(0.006585)

DESIGN CATEGORY D. FOR NONHALOGENATED PROCESS VENT STREAMS, IF 1.9 < NET HEATING VALUE (MJ/scm) ≤ 3.6 OR IF 51 < NET HEATING VALUE (Btu/scf) ≤ 97:

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Q_S \le 1180$	6.67868	0.06943	0.02582	0	0	0.01025
$(501 \le Q_S \le 41{,}700)$	(14.72382)	(0.004335)	(0.002472)	(0)	(0)	(0.003803)
$1180 < Q_S \le 2370$	13.21633	0.07546	0.02582	0	0	0.01449
$(41{,}700 < Q_S \le 83{,}700)$	(29.13672)	(0.004711)	(0.002472)	(0)	(0)	(0.005376)
$2370 < Q_S \le 3550$	19.75398	0.07922	0.02582	0	0	0.01775
$(83{,}700 < Q_S \le 125{,}000)$	(43.54962)	(0.004946)	(0.002472)	(0)	(0)	(0.006585)

DESIGN CATEGORY E. FOR NONHALOGENATED PROCESS VENT STREAMS, IF NET HEATING VALUE > 3.6 MJ/scm OR IF NET HEATING VALUE > 97 (Btu/scf):

Q_S = Vent Stream Flow rate scm/min(scf/min)	a	b	c	d	e	f
$14.2 \le Y_S \le 1180$	6.67868	0	0	-0.00707	0.02220	0.01025
$(501 \le Y_S \le 41{,}700)$	(14.72382)	(0)	(0)	(-0.0000164)	(0.0001174)	(0.003803)
$1180 < Y_S \le 2370$	13.21633	0	0	-0.00707	0.02412	0.01449
$(41{,}700 < Y_S \le 83{,}700)$	(29.13672)	(0)	(0)	(-0.0000164)	(0.0001276)	(0.005376)
$2370 < Y_S \le 3550$	19.75398	0	0	-0.00707	0.02533	0.01775
$(83{,}700 < Y_S \le 125{,}000)$	(43.54962)	(0)	(0)	(-0.0000164)	(0.0001340)	(0.006585)

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(ii) Where for a vent stream flow rate that is less than 14.2 scm/min (501 scf/min) at a standard temperature of 20 °C (68 °F):

TRE = TRE index value.

Q_s = 14.2 scm/min (501 scf/min).

H_T = (FLOW) (HVAL)/Q_s.

Where the following inputs are used:

FLOW = Vent stream flow rate, scm/min (scf/min), at a temperature of 20 °C (68 °F).

HVAL = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F) as in the definition of Q_s.

Y_s = Q_s for all vent stream categories listed in Table 1 except for Category E vent streams where $Y_s = Q_sH_T/3.6$.

E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr).

a, b, c, d, e, and f are coefficients

The set of coefficients that apply to a vent stream can be obtained from Table 1.

(2) * * *

TRE = TRE index value.

E_{TOC} = Hourly emissions of TOC, kg/hr (lb/hr).

Q_s = Vent stream flow rate, scm/min (scf/min), at a standard temperature of 20 °C (68 °F).

H_T = Vent stream net heating value, MJ/scm (Btu/scf), where the net enthalpy per mole of vent stream is based on combustion at 25 °C and 760 mm Hg (68 °F and 30 in. Hg), but the standard temperature for determining the volume corresponding to one mole is 20 °C (68 °F) as in the definition of Q_s.

a, b, c, d, and e are coefficients.

* * * * *

TABLE 2.—DISTILLATION NSPS TRE COEFFICIENTS FOR VENT STREAMS CONTROLLED BY A FLARE

	a	b	c	d	e
H_T < 11.2 MJ/scm	2.25	0.288	−0.193	−0.0051	2.08
(H_T < 301 Btu/scf)	(0.140)	(0.0367)	(−0.000448)	(−0.0051)	(4.59)
H_T ≥ 11.2 MJ/scm	0.309	0.0619	−0.0043	−0.0034	2.08
(H_T ≥ 301 Btu/scf)	(0.0193)	(0.00788)	(−0.0000010)	(−0.0034)	(4.59)

* * * * *

§ 60.665 [Amended]

203. Amend § 60.665 as follows:

a. Paragraph (b)(4)(i) is amended by revising the word "adsorbing" to read "absorbing."

b. In paragraph (e), the first sentence is amended by revising the words "44 MW" to read "44 MW (150 million Btu/hour)."

c. In paragraph (g), the first sentence is amended by revising the section reference "§ 60.663(d)" to read "§ 60.663(e)."

d. Paragraph (i) is amended by revising the words "0.008 m^3/min" to read "0.008 scm/min (0.3 scf/min)."

e. In paragraph (l)(6), the fourth sentence is amended by revising the words "vent stream flow rate, heating value, E_{TOC}" to read "vent stream flow rate, heating value, and E_{TOC}."

f. Paragraph (n) is amended by revising the word "capcity" to read "capacity."

§ 60.672 [Amended]

204. In § 60.672, paragraph (a)(1) is amended by revising the words "0.05 g/dscm" to read "0.05 g/dscm (0.022 gr/dscf)."

§ 60.676 [Amended]

205. In § 60.676, paragraphs (a)(1)(i), (a)(4)(i), and (a)(4)(ii) are amended by revising the word "tons" to read "megagrams or tons" wherever it occurs.

§ 60.685 [Amended]

206. Amend § 60.685 as follows:

a. In paragraph (c)(1), the equation definitions are revised.

b. In paragraph (c)(2) by revising the words "2.55 dscm (90 dscf)" to read "2.55 dscm (90.1 dscf)."

c. In paragraph (c)(3)(i) by revising the words "ASTM Standard Test Method D2584–68 (Reapproved 1979)" to read "ASTM D2584–68 (Reapproved 1985) or 94."

The revisions read as follows:

§ 60.685 Test methods and procedures.

* * * * *

(c) * * *

(1) * * *

E = emission rate of particulate matter, kg/Mg (lb/ton).

C_t = concentration of particulate matter, g/dscm (gr/dscf).

Q_{sd} = volumetric flow rate of effluent gas, dscm/hr (dscf/hr).

P_{avg} = average glass pull rate, Mg/hr (ton/hr).

K = 1,000 g/kg (7,000 gr/lb).

* * * * *

§ 60.692–3 [Amended]

207. In § 60.692–3, paragraph (b) is amended by revising the words "16 liters per second (250 gpm)" to read "16 liters per second (250 gallons per minute (gpm))."

§ 60.695 [Amended]

208. In § 60.695, paragraphs (a)(1) and (2) are amended by revising the words "an accuracy of 1 percent of the temperature being measured in °C or ±0.5 °C (±1.0 °F), whichever is greater" to read "an accuracy of ±1 percent of the temperature being measured, expressed in °C, or ±0.5 °C (0.9 °F), whichever is greater."

§ 60.697 [Amended]

209. Amend § 60.697 by adding paragraph (k) as follows:

§ 60.697 Recordkeeping requirements.

* * * * *

(k) For oil-water separators subject to § 60.693–2, the location, date, and corrective action shall be recorded for inspections required by §§ 60.693–2(a)(1)(iii)(A) and (B), and shall be maintained for the time period specified in paragraphs (k)(1) and (2) of this section.

(1) For inspections required by § 60.693-2(a)(1)(iii)(A), ten years after the information is recorded.

(2) For inspections required by § 60.693–2(a)(1)(iii)(B), two years after the information is recorded.

§ 60.704 [Amended]

210. Amend § 60.704 as follows:

a. Paragraph (d)(2)(ii) is amended by revising "ASTM D1946–77" to read "ASTM D1946–77 or 90 (Reapproved 1994)."

b. The definition of "C_j" in paragraph (d)(4) is amended by revising "ASTM D1946–77" to read "ASTM D1946–77 or 90 (Reapproved 1994)."

c. The definition of "H_j" in paragraph (d)(4) is amended by revising "ASTM D2382–76" to read "ASTM D2382–76 or 88 or D4809–95."

§ 60.723 [Amended]

211. In § 60.723, paragraph (b)(1) is amended by revising the words "Reference Method" to read "Method" wherever they occur.

§ 60.724 [Amended]

212. In § 60.724, paragraph (a)(2) is amended by revising the words "Reference Method" to read "Method."

§ 60.732 [Amended]

213. In § 60.732, paragraph (a) is amended by revising the words "0.057 g/dscm for dryers" to read "0.057 g/dscm (0.025 gr/dscf) for dryers."

§ 60.753 [Amended]

214. In § 60.753, paragraph (c)(2) introductory text is amended by revising the words "Method 3A" to read "Method 3A or 3C."

§ 60.754 [Amended]

215. Amend § 60.754 as follows;

a. In paragraphs (a)(1)(i) and (a)(1)(ii), the equations are amended by revising "$C_{NM}OC$" to read "C_{NMOC}."

b. In paragraph (a)(3), the introductory text is revised; and in paragraph (d), the first sentence is removed and three sentences are added in its place to read as follows:

§ 60.754 Test methods and procedures.

(a) * * *

(3) *Tier 2.* The landfill owner or operator shall determine the NMOC concentration using the following sampling procedure. The landfill owner or operator shall install at least two sample probes per hectare of landfill surface that has retained waste for at least 2 years. If the landfill is larger than 25 hectares in area, only 50 samples are required. The sample probes should be located to avoid known areas of nondegradable solid waste. The owner or operator shall collect and analyze one sample of landfill gas from each probe to determine the NMOC concentration using Method 25 or 25C of Appendix A of this part. Method 18 of Appendix A of this part may be used to analyze the samples collected by the Method 25 or 25C sampling procedure. Taking composite samples from different probes into a single cylinder is allowed; however, equal sample volumes must be taken from each probe. For each composite, the sampling rate, collection times, beginning and ending cylinder vacuums, or alternative volume measurements must be recorded to verify that composite volumes are equal. Composite sample volumes should not be less than one liter unless evidence can be provided to substantiate the accuracy of smaller volumes. Terminate compositing before the cylinder approaches ambient pressure where measurement accuracy diminishes. If using Method 18, the owner or operator must identify all compounds in the sample and, as a minimum, test for those compounds published in the most recent Compilation of Air Pollutant Emission Factors (AP–42), minus carbon monoxide, hydrogen sulfide, and mercury. As a minimum, the instrument must be calibrated for each of the compounds on the list. Convert the concentration of each Method 18 compound to C_{NMOC} as hexane by multiplying by the ratio of its carbon atoms divided by six. If more than the required number of samples are taken, all samples must be used in the analysis. The landfill owner or operator must divide the NMOC concentration from Method 25 or 25C of Appendix A of this part by six to convert from C_{NMOC} as carbon to C_{NMOC} as hexane. If the landfill has an active or passive gas removal system in place, Method 25 or 25C samples may be collected from these systems instead of surface probes provided the removal system can be shown to provide sampling as representative as the two sampling probe per hectare requirement. For active collection systems, samples may be collected from the common header pipe before the gas moving or condensate removal equipment. For these systems, a minimum of three samples must be collected from the header pipe.

* * * * *

(d) For the performance test required in § 60.752(b)(2)(iii)(B), Method 25, 25C, or Method 18 of Appendix A of this part must be used to determine compliance with the 98 weight-percent efficiency or the 20 ppmv outlet concentration level, unless another method to demonstrate compliance has been approved by the Administrator as provided by § 60.752(b)(2)(i)(B). Method 3 or 3A shall be used to determine oxygen for correcting the NMOC concentration as hexane to 3 percent. In cases where the outlet concentration is less than 50 ppm NMOC as carbon (8 ppm NMOC as hexane), Method 25A should be used in place of Method 25. * * *

* * * * *

216. In Part 60, Appendix A is amended by revising Methods 1, 1A, 2, 2A, 2B, 2C, 2D, 2E, 3, 3B, 4, 5, 5A, 5B, 5D, 5E, 5F, 5G, 5H, 6, 6A, 6B, 7, 7A, 7B, 7C, 7D, 8, 10A, 10B, 11, 12, 13A, 13B, 14, 15, 15A, 16, 16A, 16B, 17, 18, 19, 21, 22, 24, 24A, 25, 25A, 25B, 25C, 25D, 25E, 26, 26A, 27, 28, 28A, and 29 to read as follows:

METHOD 1—Sample and Velocity Traverses for Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test method: Method 2.

1.0 *Scope and Application*

1.1 Measured Parameters. The purpose of the method is to provide guidance for the selection of sampling ports and traverse points at which sampling for air pollutants will be performed pursuant to regulations set forth in this part. Two procedures are presented: a simplified procedure, and an alternative procedure (see Section 11.5). The magnitude of cyclonic flow of effluent gas in a stack or duct is the only parameter quantitatively measured in the simplified procedure.

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

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

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

2.0 *Summary of Method*

2.1 This method is designed to aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source. A measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. Traverse points are then located within each of these equal areas.

3.0 *Definitions [Reserved]*

4.0 *Interferences [Reserved]*

5.0 *Safety*

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 *Equipment and Supplies.*

6.1 Apparatus. The apparatus described below is required only when utilizing the alternative site selection procedure described in Section 11.5 of this method.

6.1.1 Directional Probe. Any directional probe, such as United Sensor Type DA Three-Dimensional Directional Probe, capable of measuring both the pitch and yaw angles of gas flows is acceptable. Before using the probe, assign an identification number to the directional probe, and permanently mark or engrave the number on the body of the probe. The pressure holes of directional probes are susceptible to plugging when used in particulate-laden gas streams. Therefore, a procedure for cleaning the pressure holes by "back-purging" with pressurized air is required.

6.1.2 Differential Pressure Gauges. Inclined manometers, U-tube manometers, or other differential pressure gauges (*e.g.*, magnehelic gauges) that meet the specifications described in Method 2, Section 6.2.

Note: If the differential pressure gauge produces both negative and positive readings, then both negative and positive pressure readings shall be calibrated at a minimum of three points as specified in Method 2, Section 6.2.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Storage, and Transport [Reserved]

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization [Reserved]

11.0 Procedure

11.1 Selection of Measurement Site.

11.1.1 Sampling and/or velocity measurements are performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance.

11.1.2 An alternative procedure is available for determining the acceptability of a measurement location not meeting the criteria above. This procedure described in Section 11.5 allows for the determination of gas flow angles at the sampling points and comparison of the measured results with acceptability criteria.

11.2 Determining the Number of Traverse Points.

11.2.1 Particulate Traverses.

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

11.2.1.2 When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1–1. Before referring to the figure, however, determine the distances from the measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1–1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1–1.

11.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that used for particulate traverses (Section 11.2.1) is followed, except that Figure 1–2 may be used instead of Figure 1–1.

11.3 Cross-Sectional Layout and Location of Traverse Points.

11.3.1 Circular Stacks.

11.3.1.1 Locate the traverse points on two perpendicular diameters according to Table 1–2 and the example shown in Figure 1–3. Any equation (see examples in References 2 and 3 in Section 16.0) that gives the same values as those in Table 1–2 may be used in lieu of Table 1–2.

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

11.3.1.3 In addition, for elliptical stacks having unequal perpendicular diameters, separate traverse points shall be calculated and located along each diameter. To determine the cross-sectional area of the elliptical stack, use the following equation:

Square Area = $D_1 \times D_2 \times 0.7854$

Where: D_1 = Stack diameter 1

D_2 = Stack diameter 2

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

11.3.2 Stacks With Diameters Greater Than 0.61 m (24 in.).

11.3.2.1 When any of the traverse points as located in Section 11.3.1 fall within 2.5 cm (1.0 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.0 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

11.3.2.2 Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling and/or velocity measurement procedure, and in recording of the data.

11.3.3 Stacks With Diameters Equal To or Less Than 0.61 m (24 in.). Follow the procedure in Section 11.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

11.3.4 Rectangular Stacks.

11.3.4.1 Determine the number of traverse points as explained in Sections 11.1 and 11.2 of this method. From Table 1–1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1–4.

11.3.4.2 To use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1–1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4 × 3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9 × 4 or 12 × 3, and would not necessarily have to be 6 × 6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area.

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

11.4 Verification of Absence of Cyclonic Flow.

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

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

11.5 The alternative site selection procedure may be used to determine the rotation angles in lieu of the procedure outlined in Section 11.4.

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

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

11.5.2 Traverse Points. Use a minimum of 40 traverse points for circular ducts and 42 points for rectangular ducts for the gas flow angle determinations. Follow the procedure outlined in Section 11.3 and Table 1–1 or 1–2 for the location and layout of the traverse points. If the measurement location is determined to be acceptable according to the criteria in this alternative procedure, use the same traverse point number and locations for sampling and velocity measurements.

11.5.3 Measurement Procedure.

11.5.3.1 Prepare the directional probe and differential pressure gauges as recommended by the manufacturer. Capillary tubing or surge tanks may be used to dampen pressure fluctuations. It is recommended, but not required, that a pretest leak check be conducted. To perform a leak check, pressurize or use suction on the impact opening until a reading of at least 7.6 cm (3 in.) H_2O registers on the differential pressure gauge, then plug the impact opening. The pressure of a leak-free system will remain stable for at least 15 seconds.

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

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

11.5.3.4 A post-test check as described in Section 11.5.3.1 is required. If the criteria for a leak-free system are not met, repair the equipment, and repeat the flow angle measurements.

11.5.4 Calibration. Use a flow system as described in Sections 10.1.2.1 and 10.1.2.2 of Method 2. In addition, the flow system shall have the capacity to generate two test-section velocities: one between 365 and 730 m/min (1,200 and 2,400 ft/min) and one between 730 and 1,100 m/min (2,400 and 3,600 ft/min).

11.5.4.1 Cut two entry ports in the test section. The axes through the entry ports shall be perpendicular to each other and intersect in the centroid of the test section. The ports should be elongated slots parallel to the axis of the test section and of sufficient length to allow measurement of pitch angles while maintaining the pitot head position at the test-section centroid. To facilitate alignment of the directional probe during calibration, the test section should be constructed of plexiglass or some other transparent material. All calibration measurements should be made at the same point in the test section, preferably at the centroid of the test section.

11.5.4.2 To ensure that the gas flow is parallel to the central axis of the test section, follow the procedure outlined in Section 11.4 for cyclonic flow determination to measure the gas flow angles at the centroid of the test section from two test ports located 90° apart. The gas flow angle measured in each port must be ±2° of 0°. Straightening vanes should be installed, if necessary, to meet this criterion.

11.5.4.3 Pitch Angle Calibration. Perform a calibration traverse according to the manufacturer's recommended protocol in 5° increments for angles from −60° to +60° at one velocity in each of the two ranges specified above. Average the pressure ratio values obtained for each angle in the two flow ranges, and plot a calibration curve with the average values of the pressure ratio (or other suitable measurement factor as recommended by the manufacturer) versus the pitch angle. Draw a smooth line through the data points. Plot also the data values for each traverse point. Determine the differences between the measured data values and the angle from the calibration curve at the same pressure ratio. The difference at each comparison must be within 2° for angles between 0° and 40° and within 3° for angles between 40° and 60°.

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

12.0 Data Analysis and Calculations

12.1 Nomenclature.

L = length.
n = total number of traverse points.
P_i = pitch angle at traverse point i, degree.
R_{avg} = average resultant angle, degree.
R_i = resultant angle at traverse point i, degree.
S_d = standard deviation, degree.
W = width.
Y_i = yaw angle at traverse point i, degree.

12.2 For a rectangular cross section, an equivalent diameter (D_e) shall be calculated using the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2(L)(W)}{L+W} \qquad \text{Eq. 1-1}$$

12.3 If use of the alternative site selection procedure (Section 11.5 of this method) is required, perform the following calculations using the equations below: the resultant angle at each traverse point, the average resultant angle, and the standard deviation. Complete the calculations retaining at least one extra significant figure beyond that of the acquired data. Round the values after the final calculations.

12.3.1 Calculate the resultant angle at each traverse point:

$$R_i = \text{arc cosine}\left[(\text{cosine } Y_i)(\text{cosine } P_i)\right] \qquad \text{Eq. 1-2}$$

12.3.2 Calculate the average resultant for the measurements:

$$R_{avg} = \sum R_i/n \qquad \text{Eq. 1-3}$$

12.3.3 Calculate the standard deviations:

$$S_d = \sqrt{\frac{\sum_{i=1}^{n}\left(R_i - R_{avg}\right)^2}{(n-1)}} \qquad \text{Eq. 1-4}$$

12.3.4 Acceptability Criteria. The measurement location is acceptable if $R_{avg} \leq 20°$ and $S_d \leq 10°$.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

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17.0 Tables, Diagrams, Flowcharts, and Validation Data

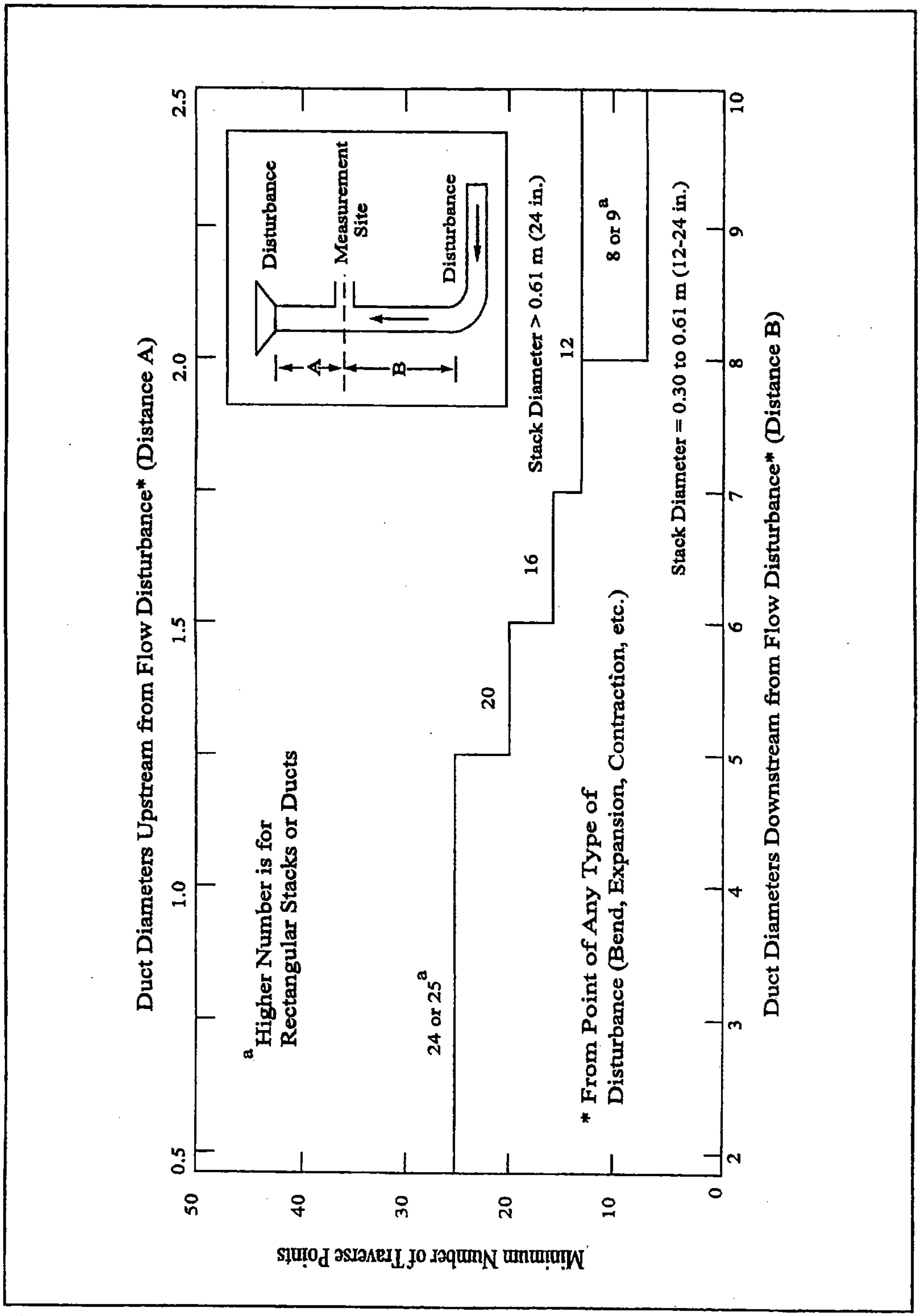

Figure 1-1. Minimum number of traverse points for particulate traverses.

TABLE 1–1 CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

Number of tranverse points layout	Matrix
9	3×3
12	4×3
16	4×4
20	5×4
25	5×5
30	6×5
36	6×6
42	7×6
49	7×7

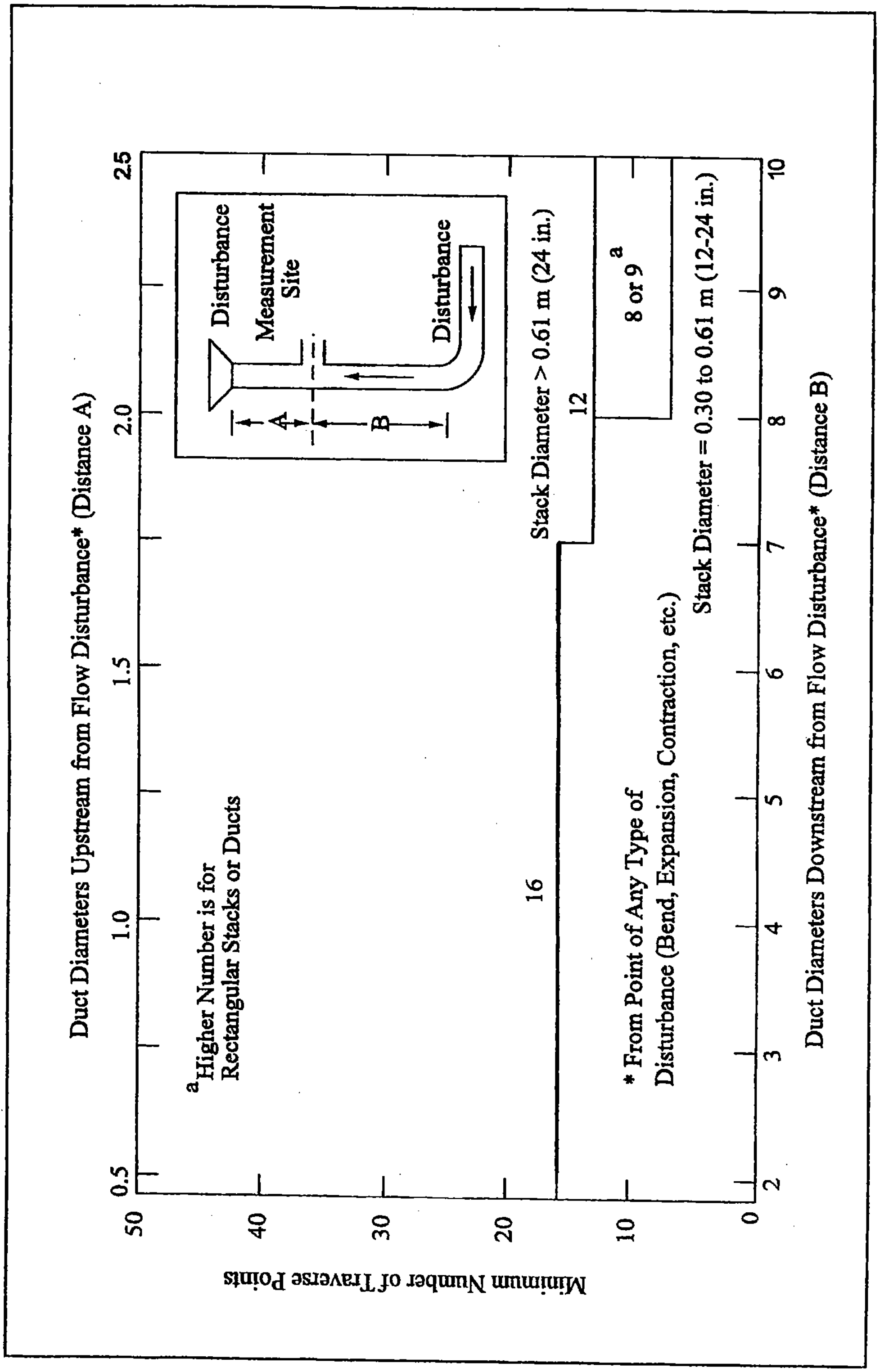

Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

TABLE 1–2.—LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

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

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												99.9

Traverse Point	% of diameter Distance
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

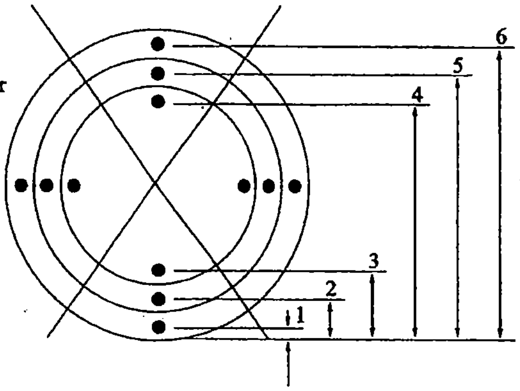

Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points.

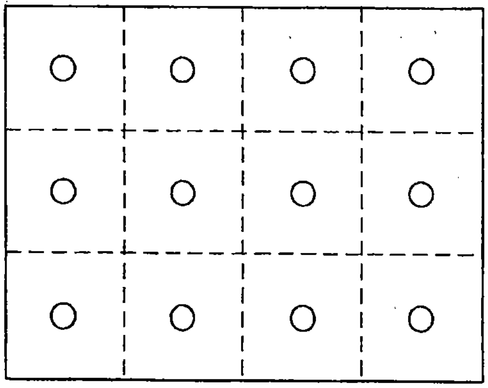

Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with traverse points at centroid of each area.

Method 1A—Sample and Velocity Traverses for Stationary Sources With Small Stacks or Ducts

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test method: Method 1.

1.0 Scope and Application

1.1 Measured Parameters. The purpose of the method is to provide guidance for the selection of sampling ports and traverse points at which sampling for air pollutants will be performed pursuant to regulations set forth in this part.

1.2 Applicability. The applicability and principle of this method are identical to Method 1, except its applicability is limited to stacks or ducts. This method is applicable to flowing gas streams in ducts, stacks, and flues of less than about 0.30 meter (12 in.) in diameter, or 0.071 m^2 (113 $in.^2$) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter, or 0.0081 m^2 (12.57 $in.^2$) in cross-sectional area. This method cannot be used when the flow is cyclonic or swirling.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 The method is designed to aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source. A measurement site or a pair of measurement sites where the effluent stream is flowing in a known direction is (are) selected. The cross-section of the stack is divided into a number of equal areas. Traverse points are then located within each of these equal areas.

2.2 In these small diameter stacks or ducts, the conventional Method 5 stack assembly (consisting of a Type S pitot tube attached to a sampling probe, equipped with a nozzle and thermocouple) blocks a significant portion of the cross-section of the duct and causes inaccurate measurements. Therefore, for particulate matter (PM) sampling in small stacks or ducts, the gas velocity is measured using a standard pitot tube downstream of the actual emission sampling site. The straight run of duct between the PM sampling and velocity measurement sites allows the flow profile, temporarily disturbed by the presence of the sampling probe, to redevelop and stabilize.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies [Reserved]

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Storage, and Transport [Reserved]

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization [Reserved]

11.0 Procedure

11.1 Selection of Measurement Site.

11.1.1 Particulate Measurements—Steady or Unsteady Flow. Select a particulate measurement site located preferably at least eight equivalent stack or duct diameters downstream and 10 equivalent diameters upstream from any flow disturbances such as bends, expansions, or contractions in the stack, or from a visible flame. Next, locate the velocity measurement site eight equivalent diameters downstream of the particulate measurement site (see Figure 1A–1). If such locations are not available, select an alternative particulate measurement location at least two equivalent stack or duct diameters downstream and two and one-half diameters upstream from any flow disturbance. Then, locate the velocity measurement site two equivalent diameters downstream from the particulate measurement site. (See Section 12.2 of Method 1 for calculating equivalent diameters for a rectangular cross-section.)

11.1.2 PM Sampling (Steady Flow) or Velocity (Steady or Unsteady Flow) Measurements. For PM sampling when the volumetric flow rate in a duct is constant with respect to time, Section 11.1.1 of Method 1 may be followed, with the PM sampling and velocity measurement performed at one location. To demonstrate that the flow rate is constant (within 10 percent) when PM measurements are made, perform complete velocity traverses before and after the PM sampling run, and calculate the deviation of the flow rate derived after the PM sampling run from the one derived before the PM sampling run. The PM sampling run is acceptable if the deviation does not exceed 10 percent.

11.2 Determining the Number of Traverse Points.

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

11.2.2 PM Sampling (Steady Flow) or only Velocity (Non-Particulate) Measurements. Use Figure 1–2 of Method 1 to determine number of traverse points, following the same procedure used for PM sampling as described in Section 11.2.1 of Method 1. When the optimum duct diameter location criteria can be satisfied, the minimum number of traverse points required is eight for circular ducts and nine for rectangular ducts.

11.3 Cross-sectional Layout, Location of Traverse Points, and Verification of the Absence of Cyclonic Flow. Same as Method 1, Sections 11.3 and 11.4, respectively.

12.0 Data Analysis and Calculations [Reserved]

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 1, Section 16.0, References 1 through 6, with the addition of the following:

1. Vollaro, Robert F. Recommended Procedure for Sample Traverses in Ducts Smaller Than 12 Inches in

Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, North Carolina. January 1977.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

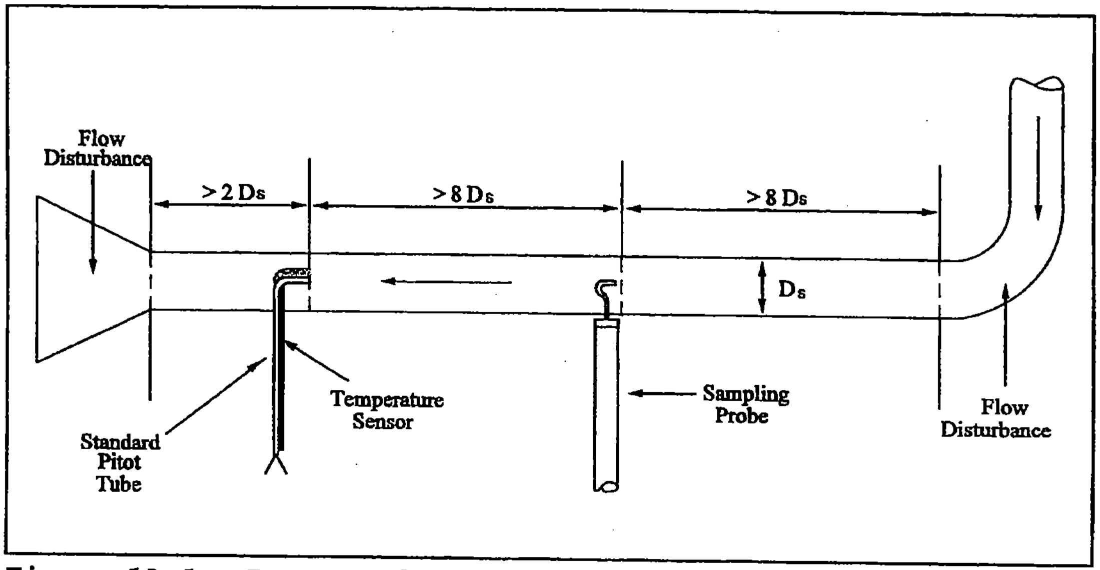

Figure 1A-1. Recommended sampling arrangement for small ducts

Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test method: Method 1.

1.0 Scope and Application.

1.1 This method is applicable for the determination of the average velocity and the volumetric flow rate of a gas stream.

1.2 This method is not applicable at measurement sites that fail to meet the criteria of Method 1, Section 11.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 11.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, must be employed to produce accurate flow rate determinations. Examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method.

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

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

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

6.1 Type S Pitot Tube.

6.1.1 Pitot tube made of metal tubing (*e.g.*, stainless steel) as shown in Figure 2–1. It is recommended that the external tubing diameter (dimension D_t, Figure 2–2b) be between 0.48 and 0.95 cm ($^3/_{16}$ and $^3/_8$ inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_A and P_B, Figure 2–2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2–2; however, slight misalignments of the openings are permissible (see Figure 2–3).

6.1.2 The Type S pitot tube shall have a known coefficient, determined as outlined in Section 10.0. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube. A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 6.7 and 10.2. Note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period. This can be accomplished by comparing the velocity head (Δp) measurement recorded at a selected traverse point (readable Δp value) with a second Δp measurement recorded after "back purging" with pressurized air to clean the impact and static holes of the standard pitot tube. If the before and after Δp measurements are within 5 percent, then the traverse data are acceptable. Otherwise, the data should be rejected and the traverse measurements redone. Note that the selected traverse point should be one that demonstrates a readable Δp value. If "back purging" at regular intervals is part of a routine procedure, then comparative Δp measurements shall be conducted as above for the last two traverse points that exhibit suitable Δp measurements.

6.2 Differential Pressure Gauge. An inclined manometer or equivalent device. Most sampling trains are equipped with a 10 in. (water column) inclined-vertical manometer, having 0.01 in. H_20 divisions on the 0 to 1 in. inclined scale, and 0.1 in. H_20 divisions on the 1 to 10 in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.27 mm (0.05 in.) H_20. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.27 mm (0.05 in.) H_20; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.27 mm (0.05 in.) H_20; or (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.27 mm (0.05 in.) H_20. Reference 18 (see Section 17.0) describes commercially available instrumentation for the measurement of low-range gas velocities.

6.2.1 As an alternative to criteria (1) through (3) above, Equation 2–1 (Section 12.2) may be used to determine the necessity of using a more sensitive differential pressure gauge. If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

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

6.3 Temperature Sensor. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperatures to within 1.5 percent of the minimum absolute stack temperature. The temperature sensor shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2–1 and Figure 2–4). Alternative positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 10.0. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube. This alternative is subject to the approval of the Administrator.

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

6.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.54 mm (0.1 in.) Hg.

Note: The barometric pressure reading may be obtained from a nearby National Weather Service station. In this case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be made at a rate of minus 2.5 mm (0.1 in.) Hg per 30 m (100 ft) elevation increase or plus 2.5 mm (0.1 in.) Hg per 30 m (100 ft.) for elevation decrease.

6.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 8.6), to determine the stack gas dry molecular weight, and Method 4 (reference method) or Method 5 equipment for moisture content determination. Other methods may be used subject to approval of the Administrator.

6.7 Calibration Pitot Tube. When calibration of the Type S pitot tube is necessary (see Section 10.1), a standard pitot tube shall be used for a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Institute of Standards and Technology (NIST), Gaithersburg MD 20899, (301) 975–2002, or (2) by calibration against another standard pitot tube with an NIST-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in Sections 6.7.1 through 6.7.5 below and illustrated in Figure 2–5 (see also References 7, 8, and 17 in Section 17.0) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of 0.99 ± 0.01.

6.7.1 Standard Pitot Design.

6.7.1.1 Hemispherical (shown in Figure 2–5), ellipsoidal, or conical tip.

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

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

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

6.7.1.5 90° bend, with curved or mitered junction.

6.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent. If the single-velocity calibration technique is employed (see Section 10.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.127 mm (0.005 in.) H_20. For multivelocity calibrations, the gauge shall be readable to the nearest 0.127 mm (0.005 in.) H_20 for Δp values between 1.27 and 25.4 mm (0.05 and 1.00 in.) H_20, and to the nearest 1.27 mm (0.05 in.) H_20 for Δp values above 25.4 mm (1.00 in.) H_20. A special, more sensitive gauge will be required to read Δp values below 1.27 mm (0.05 in.) H_20 (see Reference 18 in Section 16.0).

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection and Analysis

8.1 Set up the apparatus as shown in Figure 2–1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3.0 in.) H_20 velocity head registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3.0 in.) H_20. Other leak-check procedures, subject to the approval of the Administrator, may be used.

8.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse (at least once per hour). Record all necessary data on a form similar to that shown in Figure 2–6.

8.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 6.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 8.1 above, to validate the traverse run.

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

8.5 Determine the atmospheric pressure.

8.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO_2, O_2, CO, and N_2, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

8.7 Obtain the moisture content from Method 4 (reference method, or equivalent) or from Method 5.

8.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints. Do not assume that stack diameters are equal. Measure each diameter distance to verify its dimensions.

9.0 Quality Control

Section	Quality control measure	Effect
10.1–10.4	Sampling equipment calibration	Ensure accurate measurement of stack gas flow rate, sample volume.

10.0 Calibration and Standardization

10.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figures 2–2 and 2–3. The pitot tube shall not be used if it fails to meet these alignment specifications. After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_t, Figure 2–2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2–2b). If D_t is between 0.48 and 0.95 cm 3/16 and 3/8 in.), and if P_A and P_B are equal and between 1.05 and 1.50 D_t, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 10.1.1). If D_t, P_A, and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in Sections 10.1.2 through 10.1.5.

10.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (e.g., thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Reference 9 in Section 17.0); therefore, an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2–4, 2–7, and 2–8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2–4, 2–7, and 2-8 shall be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

Note: Do not use a Type S pitot tube assembly that is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2–6B).

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

10.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross sections, the minimum duct diameter shall be 30.48 cm (12 in.); for rectangular cross sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

10.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross section, use an equivalent diameter, calculated according to Equation 2–2 (see Section 12.3), to determine the number of duct diameters. To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

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

10.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 910 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 910 m/min (3,000 ft/min) will generally be valid to ±3 percent for the measurement of velocities above 300 m/min (1,000 ft/min) and to ±6 percent for the measurement of velocities between 180 and 300 m/min (600 and 1,000 ft/min). If a more precise correlation between the pitot tube coefficient, (C_p), and velocity is desired, the flow system should have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,500 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see References 9 and 14 in Section 17.0 for details).

10.1.2.4 Two entry ports, one for each of the standard and Type S pitot tubes, shall be cut in the test section. The standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of Plexiglas™ or some other transparent material.

10.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 10.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

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

10.1.3.2 Level and zero the manometer. Switch on the fan, and allow the flow to stabilize. Seal the Type S pitot tube entry port.

10.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 10.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

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

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

10.1.3.6 Read Δp_s, and enter its value in the data table. Remove the Type S pitot tube from the duct, and disconnect it from the manometer.

10.1.3.7 Repeat Steps 10.1.3.3 through 10.1.3.6 until three pairs of Δp readings have been obtained for the A side of the Type S pitot tube.

10.1.3.8 Repeat Steps 10.1.3.3 through 10.1.3.7 for the B side of the Type S pitot tube.

10.1.3.9 Perform calculations as described in Section 12.4. Use the Type S pitot tube only if the values of σ_A and σ_B are less than or equal to 0.01 and if the absolute value of the difference between $\bar{C}_{p(A)}$ and $\bar{C}_{p(B)}$ is 0.01 or less.

10.1.4 Special Considerations.

10.1.4.1 Selection of Calibration Point.

10.1.4.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Section 10.1.3. The Type S pitot coefficients measured or calculated, (*i.e.* $\bar{C}_{p(A)}$ and $\bar{C}_{p(B)}$) will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2–4, 2–7, and 2–8).

10.1.4.1.2 For Type S pitot tube-thermocouple combinations (without probe assembly), select a calibration point at or near the center of the duct, and follow the procedures outlined in Section 10.1.3. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2–4, 2–7, and 2–8).

10.1.4.1.3 For Type S pitot tube combinations with complete probe assemblies, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area interference and blockage and yield incorrect coefficient values (Reference 9 in Section 17.0). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2–10a), and 3 percent or less for assemblies with external sheaths (Figure 2–10b).

10.1.4.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specifications illustrated in Figure 2–7A), the value of $C_{p(s)}$ depends upon the amount of free space between the tube and nozzle and, therefore, is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 910 m/min (3,000 ft/min), which is the calibration velocity. Note also that it is not necessary to draw an isokinetic sample during calibration (see Reference 19 in Section 17.0).

10.1.4.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2–2 or 2–3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 10.1.4.4).

10.1.5 Field Use and Recalibration.

10.1.5.1 Field Use.

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

10.1.5.1.2 When a probe assembly is used to sample a small duct, 30.5 to 91.4 cm (12 to 36 in.) in diameter, the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $C_{p(s)}$. Consult Reference 9 (see Section 17.0) for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 30.5 cm (12 in.) (see Reference 16 in Section 17.0).

10.1.5.2 Recalibration.

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

10.1.5.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 10.1.5.2.1. Also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figure 2–2 and Figure 2–3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings, or recalibrate the assembly.

10.2 Standard Pitot Tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 6.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

10.3 Temperature Sensors.

10.3.1 After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other sensors at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405 °C (761 °F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference. Alternatively, either a reference thermocouple and potentiometer (calibrated against NIST standards) or thermometric fixed points (*e.g.*, ice bath and boiling water, corrected for barometric pressure) may be used. For temperatures above 405°C (761 °F), use a reference thermocouple-potentiometer system calibrated against NIST standards or an alternative reference, subject to the approval of the Administrator.

10.3.2 The temperature data recorded in the field shall be considered valid. If, during calibration, the absolute temperature measured with the sensor being calibrated and the reference sensor agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

10.4 Barometer. Calibrate the barometer used against a mercury barometer.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

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

12.1 Nomenclature.

A = Cross-sectional area of stack, m^2 (ft^2).
B_{ws} = Water vapor in the gas stream (from Method 4 (reference method) or Method 5), proportion by volume.
C_p = Pitot tube coefficient, dimensionless.
$C_{p(s)}$ = Type S pitot tube coefficient, dimensionless.
$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 6.7.1 to 6.7.5 of this method.
D_e = Equivalent diameter.
K = 0.127 mm H_2O (metric units). 0.005 in. H_2O (English units).
K_p = Velocity equation constant.
L = Length.
M_d = Molecular weight of stack gas, dry basis (see Section 8.6), g/g-mole (lb/lb-mole).
M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).
n = Total number of traverse points.
P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).
P_g = Stack static pressure, mm Hg (in. Hg).
P_s = Absolute stack pressure ($P_{bar} + P_g$), mm Hg (in. Hg),
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).
T = Sensitivity factor for differential pressure gauges.
T_s = Stack temperature, °C (°F).
$T_{s(abs)}$ = Absolute stack temperature, °K (°R).
= 273 + T_s for metric units,
= 460 + T_s for English units.
T_{std} = Standard absolute temperature, 293 °K (528 °R).
V_s = Average stack gas velocity, m/sec (ft/sec).
W = Width.
Δp = Velocity head of stack gas, mm H_2O (in. H_{2o}).
Δp_i = Individual velocity head reading at traverse point "i", mm (in.) H_2O.
Δp_{std} = Velocity head measured by the standard pitot tube, cm (in.) H_2O.
Δp_s = Velocity head measured by the Type S pitot tube, cm (in.) H_2O.
3600 = Conversion Factor, sec/hr.
18.0 = Molecular weight of water, g/g-mole (lb/lb-mole).

12.2 Calculate T as follows:

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}} \qquad \text{Eq. 2-1}$$

12.3 Calculate D_e as follows:

$$D_e = \frac{2LW}{L+W} \qquad \text{Eq. 2-2}$$

12.4 Calibration of Type S Pitot Tube.

12.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 10.1.3, calculate the value of the Type S pitot tube coefficient according to Equation 2–3:

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p}} \qquad \text{Eq. 2-3}$$

12.4.2 Calculate $\bar{C}_{p(A)}$, the mean A-side coefficient, and $\bar{C}_{p(B)}$, the mean B-side coefficient. Calculate the difference between these two average values.

12.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\bar{C}_{p(A)}$, and the deviation of each of the three B-side values of $C_{p(s)}$ from $\bar{C}_{p(B)}$, using Equation 2–4:

$$\text{Deviation} = C_{p(s)} - \bar{C}_{p(A \text{ or } B)} \qquad \text{Eq. 2-4}$$

12.4.4 Calculate σ the average deviation from the mean, for both the A and B sides of the pitot tube. Use Equation 2–5:

$$\sigma_{A \text{ or } B} = \frac{\sum_{i=1}^{3} \left| C_{p(s)} - \bar{C}_{p(A \text{ or } B)} \right|}{3} \qquad \text{Eq. 2-5}$$

12.5 Molecular Weight of Stack Gas.

$$M_s = M_d(1 - B_{ws}) + 18.0\, B_{ws} \qquad \text{Eq. 2-6}$$

12.6 Average Stack Gas Velocity.

$$V_s = K_p C_p \sqrt{\Delta p_{avg}} \sqrt{\frac{T_{s(abs)}}{P_s M_s}} \qquad \text{Eq. 2-7}$$

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(\text{g/g} \cdot \text{mole})(\text{mmHg})}{(^\circ\text{K})(\text{mmH}_2\text{O})} \right]^{\frac{1}{2}} \qquad \text{Metric}$$

$$85.49 \frac{\text{m}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(^\circ\text{R})(\text{in. H}_2\text{O})} \right]^{\frac{1}{2}} \qquad \text{English}$$

12.7 Average Stack Gas Dry Volumetric Flow Rate.

$$Q = 3600(1 - B_{ws}) v_s A \left[\frac{T_{std} P_s}{T_{s(abs)} P_{std}} \right] \qquad \text{Eq. 2-8}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

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14. Vollaro, R.F. The Use of Type S Pitot Tubes for the Measurement of Low Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, NC. November 1976.

15. Smith, Marvin L. Velocity Calibration of EPA Type Source Sampling Probe. United Technologies Corporation, Pratt and Whitney Aircraft Division, East Hartford, CT. 1975.

16. Vollaro, R.F. Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, NC. November 1976.

17. Ower, E. and R.C. Pankhurst. The Measurement of Air Flow, 4th Ed. London, Pergamon Press. 1966.

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19. Gnyp, A.W., et al. An Experimental Investigation of the Effect of Pitot Tube-Sampling Probe Configurations on the Magnitude of the S Type Pitot Tube Coefficient for Commercially Available Source Sampling Probes. Prepared by the University of Windsor for the Ministry of the Environment, Toronto, Canada. February 1975.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

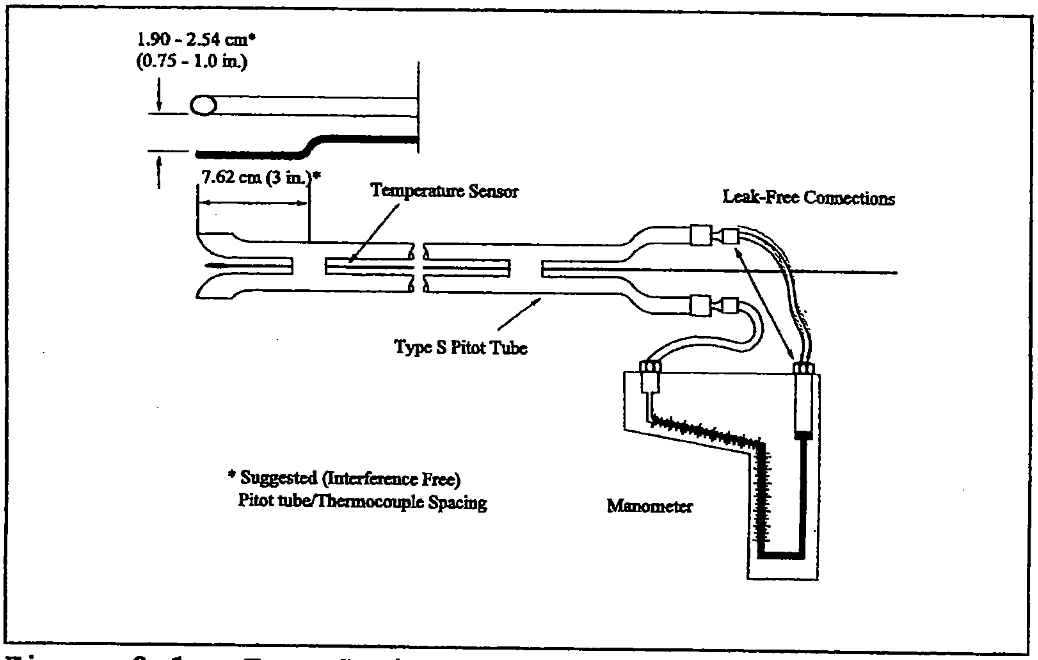
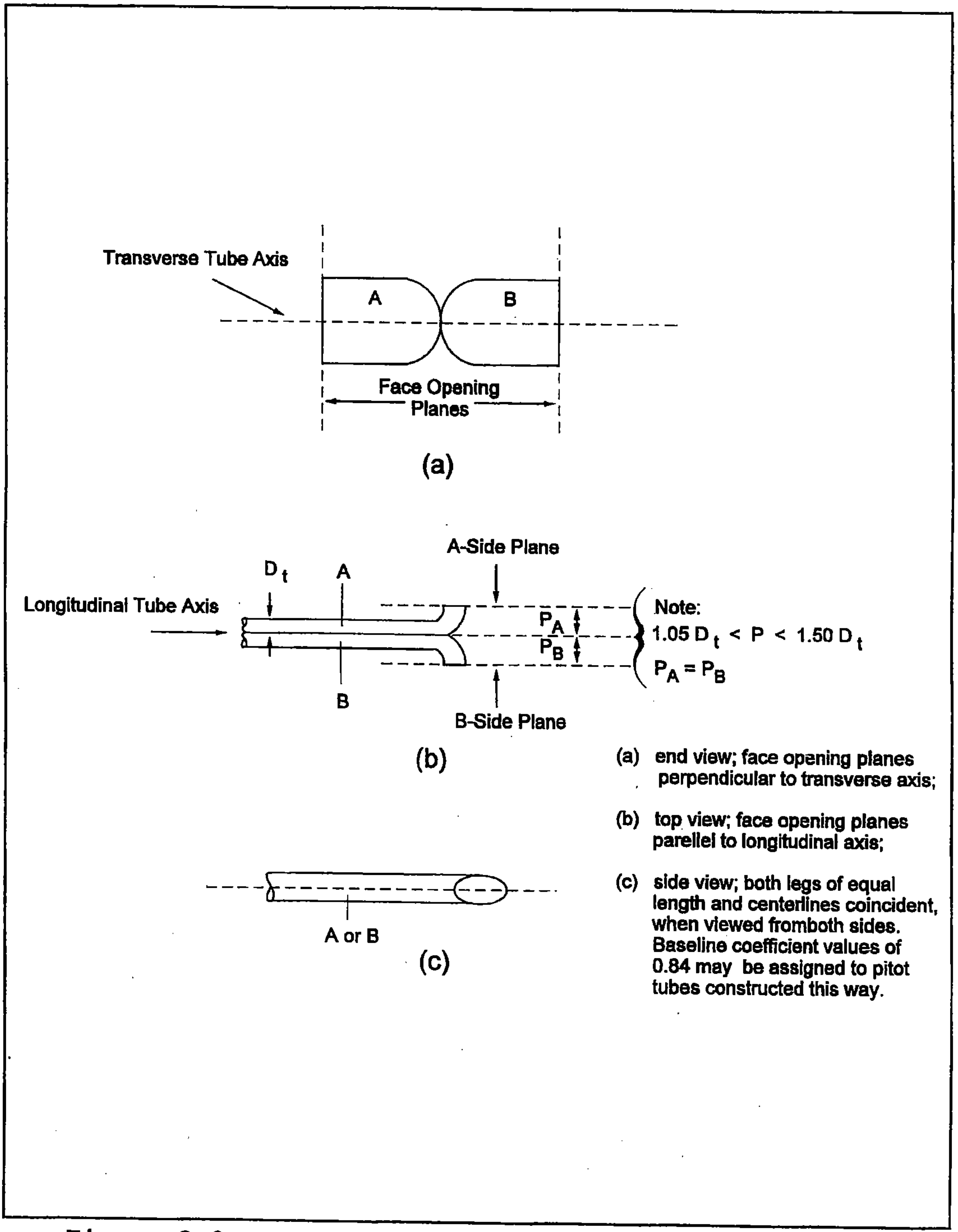


Figure 2-1. Type S Pitot Tube Manometer Assembly.

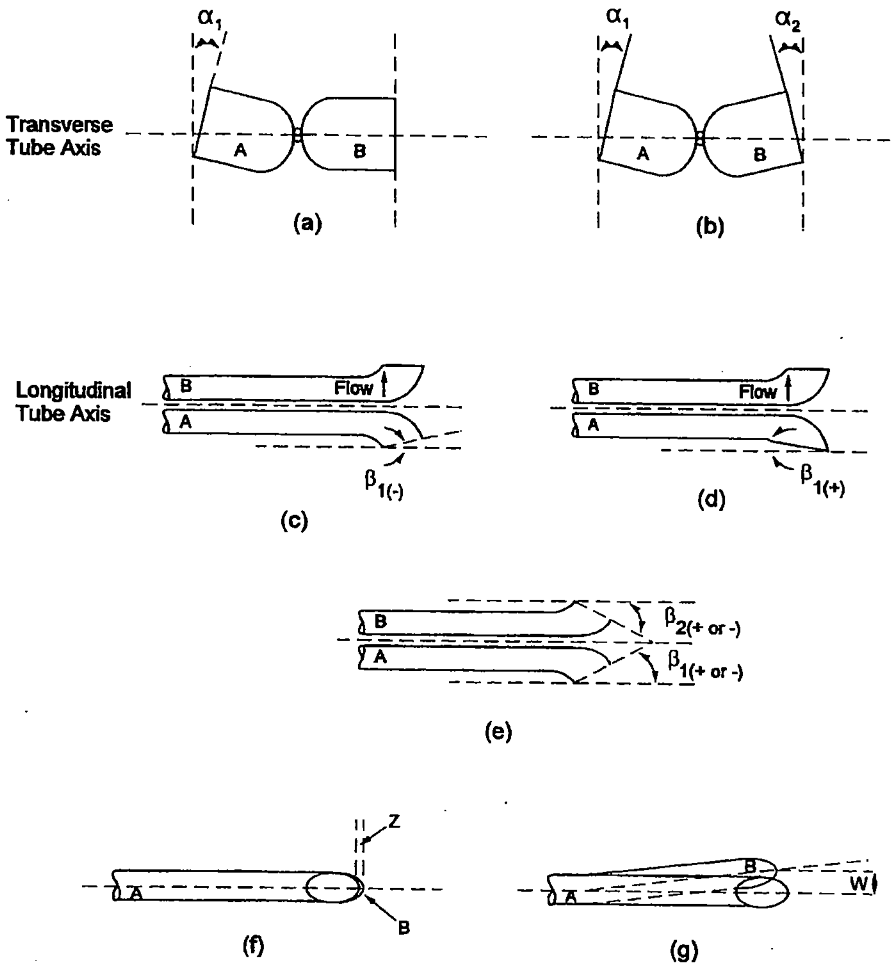


(a) end view; face opening planes perpendicular to transverse axis;

(b) top view; face opening planes parellel to longitudinal axis;

(c) side view; both legs of equal length and centerlines coincident, when viewed fromboth sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

Figure 2-2. Properly Constructed Type S Pitot Tube.



The types of face-opening misalignment shown above will not affect the baseline value of $C_{p(s)}$ so long as α_1 and $\alpha_2 \leq 10°$, β_1 and $\beta_2 \leq 5°$, $z \leq 0.32$ cm (1/8 in.), and $w \leq 0.08$ cm (1/32 in.) (Reference 11.0 in Section 16.0)

Figure 2-3. Types of face-opening misalignments that can result from field use or improper construction of type S pitot tubes.

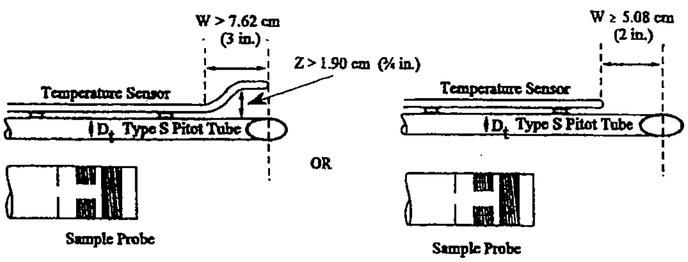


Figure 2-4. Proper temperature sensor placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in).

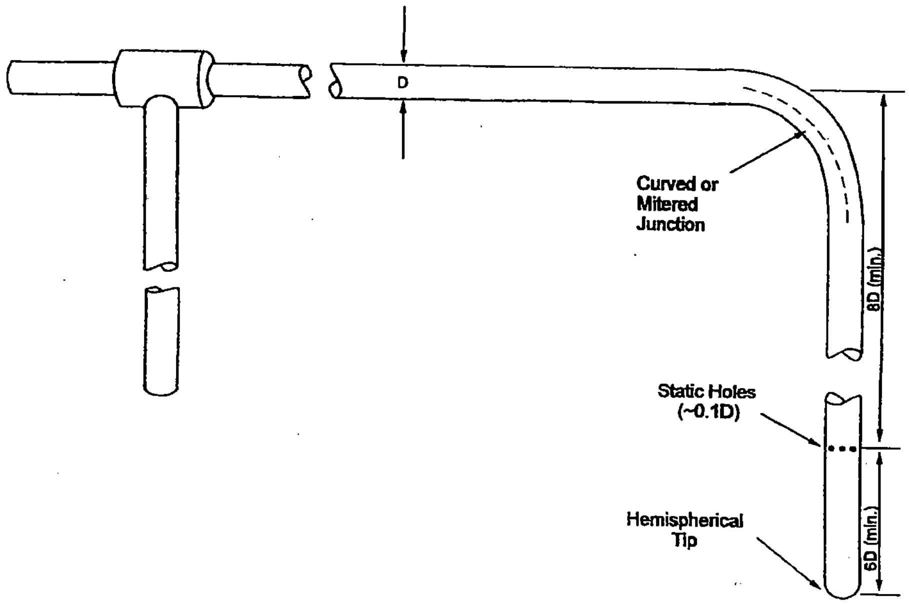


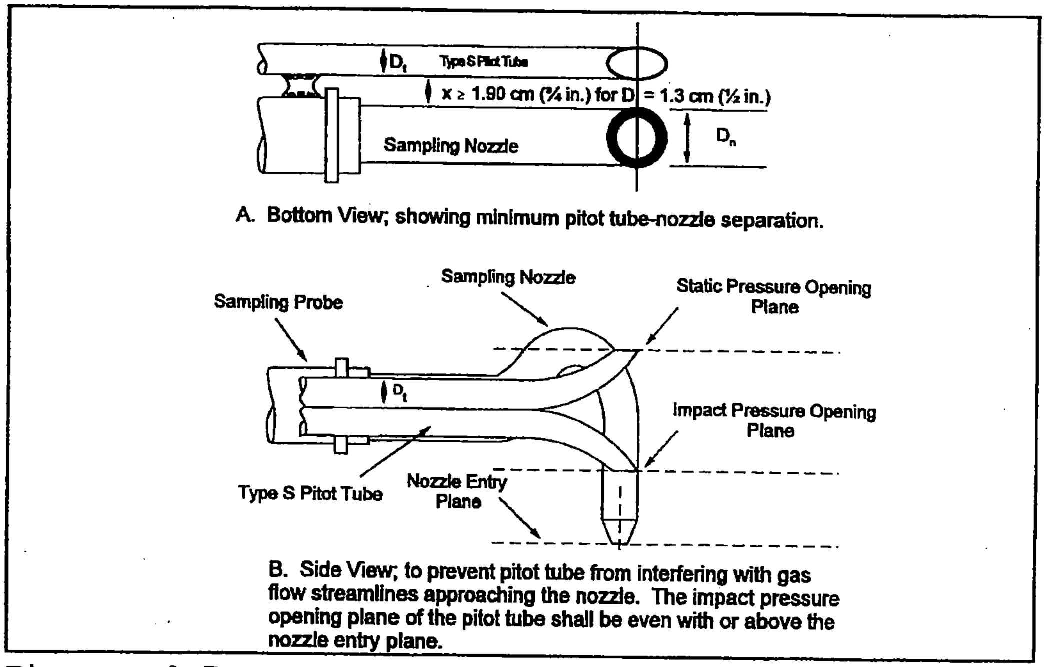
Figure 2-5. Standard pitot tube design specifications.

PLANT
DATE
RUN NO.
STACK DIA. OR DIMENSIONS, m (in.)
BAROMETRIC PRESS., mm Hg (in. Hg)
CROSS SECTIONAL AREA, m^2 (ft^2)
OPERATORS
PITOT TUBE I.D. NO.
AVG. COEFFICIENT, Cp =
LAST DATE CALIBRATED

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack temperature		P_g mm Hg (in. Hg)	$(\Delta p)^{1/2}$
		T_s, °C (°F)	T_s, °K (°R)		
	Average(1)				

Figure 2–6. Velocity Traverse Data



A. Bottom View; showing minimum pitot tube-nozzle separation.

B. Side View; to prevent pitot tube from interfering with gas flow streamlines approaching the nozzle. The impact pressure opening plane of the pitot tube shall be even with or above the nozzle entry plane.

Figure 2-7. Proper pitot tube-sampling nozzle configuration.

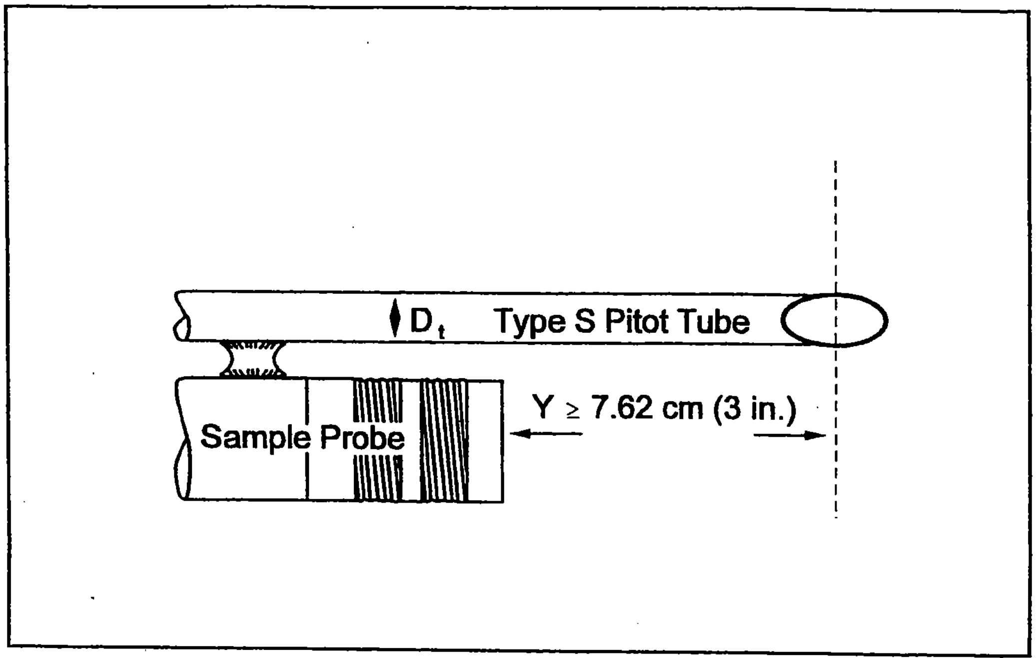


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in).

PITOT TUBE IDENTIFICATION NUMBER:
DATE:
CALIBRATED BY:

"A" SIDE CALIBRATION

Run No.	ΔP_{std} cm H_2O (in H_2O)	$\Delta P_{(s)}$ cm H_2O (in H_2O)	$C_{p(s)}$	Deviation $C_{p(s)}$—C_p(A)
1				
2				
3				
		$C_{p,\ avg}$ (SIDE A)		

"B" SIDE CALIBRATION

Run No.	ΔP_{std} cm H_2O (in H_2O)	$\Delta P_{(s)}$ cm H_2O (in H_2O)	$C_{p(s)}$	Deviation $C_{p(s)}$—C_p(B)
1				
2				
3				
		$C_{p,\ avg}$ (SIDE B)		

$$\sigma_{A\ or\ B} = \frac{\sum_{i=1}^{3} \left| C_{p(s)} - \bar{C}_{p(A\ or\ B)} \right|}{3} \qquad \text{Eq. 2-5}$$

[Cp, avg (side A)—Cp, avg (side B)]*
*Must be less than or equal to 0.01

Figure 2–9. Pitot Tube Calibration Data

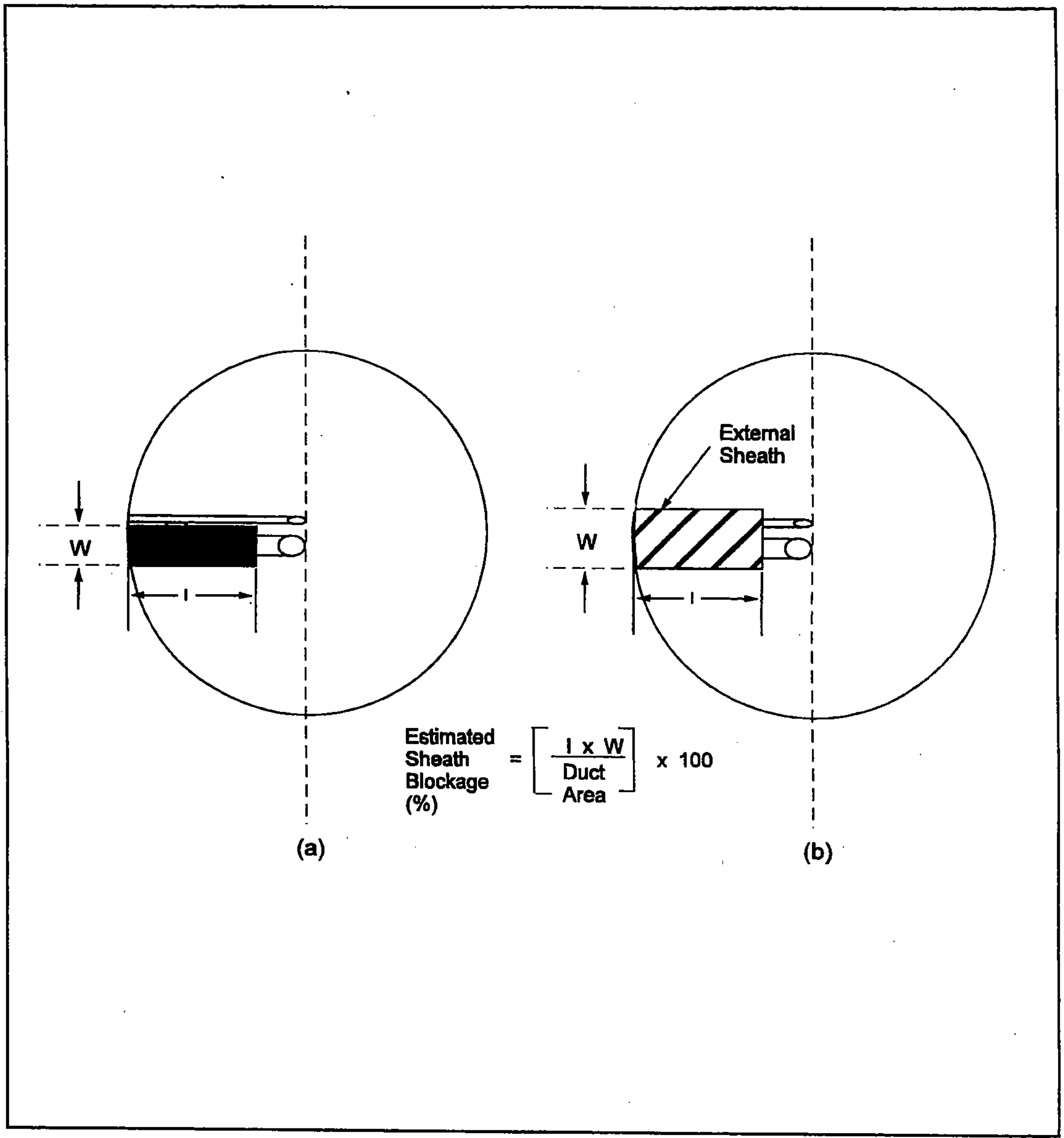


Figure 2-10. Projected-area models for typical pitot tube assemblies.

Method 2A—Direct Measurement of Gas Volume Through Pipes and Small Ducts

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2.

1.0 Scope and Application

1.1 This method is applicable for the determination of gas flow rates in pipes and small ducts, either in-line or at exhaust positions, within the temperature range of 0 to 50 °C (32 to 122 °F).

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas volume meter is used to measure gas volume directly. Temperature and pressure measurements are made to allow correction of the volume to standard conditions.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

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

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

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

Note: In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

6.3 Stopwatch. Capable of measurement to within 1 second.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection and Analysis

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

8.2 Leak Test.

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

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

8.3 Volume Measurement.

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

8.3.2 For sources with noncontinuous, non-steady emission flow rates, use the procedure in Section 8.3.1 with the addition of the following: Record all the meter parameters and the start and stop times corresponding to each process cyclical or noncontinuous event.

9.0 Quality Control

Section	Quality control measure	Effect
10.1–10.4	Sampling equipment calibration	Ensure accurate measurement of stack gas flow rate, sample volume.

10.0 Calibration and Standardization

10.1 Volume Meter.

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

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

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

10.1.4 The calibration shall be performed during at least three different flow rates. The calibration flow rates shall be about 0.3, 0.6, and 0.9 times the rated maximum flow rate of the test meter.

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

10.1.6 Calculate the test meter calibration coefficient as indicated in Section 12.2.

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

10.1.8 The procedure above shall be performed at least once for each volume meter. Thereafter, an abbreviated calibration check shall be completed following each field test. The calibration of the volume meter shall be checked with the meter pressure set at the average value encountered during the field test. Three calibration checks (runs) shall be performed using this average flow rate value. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of flow as described above.

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

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

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

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

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

12.1 Nomenclature.

f = Final reading.
i = Initial reading.
P_{bar} = Barometric pressure, mm Hg.
P_g = Average static pressure in volume meter, mm Hg.
Q_s = Gas flow rate, m^3/min, standard conditions.
s = Standard conditions, 20°C and 760 mm Hg.
T_r = Reference meter average temperature, °K (°R).
T_m = Test meter average temperature, °K (°R).
V_r = Reference meter volume reading, m^3.
V_m = Test meter volume reading, m^3.
Y_m = Test meter calibration coefficient, dimensionless.
θ = Elapsed test period time, min.

12.2 Test Meter Calibration Coefficient.

$$Y_m = \frac{\left(V_{r_f} - V_{r_i}\right) P_b T_{r(abs)}}{\left(V_{m_f} - V_{m_i}\right)\left(P_b + P_g\right) T_{m(abs)}} \qquad \text{Eq. 2A-1}$$

12.3 Volume.

$$Y_{m_s} = Y_m \left[\frac{\left(P_{bar} + P_g\right)\left(V_{m_f} - V_{m_i}\right)(293\ ^\circ\mathrm{K})}{\left(T_m\right)(760\ \mathrm{mmHg})} \right] \qquad \text{Eq. 2-2}$$

12.4 Gas Flow Rate.

$$Q_s = \frac{V_{m_s}}{\theta} \qquad \text{Eq. 2A-3}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. APTD–0576. March 1972.

2. Wortman, Martin, R. Vollaro, and P.R. Westlin. Dry Gas Volume Meter Calibrations. Source Evaluation Society Newsletter. Vol. 2, No. 2. May 1977.

3. Westlin, P.R., and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter. Vol. 3, No. 1. February 1978.

17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

Method 2B—Determination of Exhaust Gas Volume Flow Rate From Gasoline Vapor Incinerators

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 2A, Method 10, Method 25A, Method 25B.

1.0 Scope and Application

1.1 This method is applicable for the determination of exhaust volume flow rate from incinerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). It is assumed that the amount of auxiliary fuel is negligible.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Organic carbon concentration and volume flow rate are measured at the incinerator inlet using either Method 25A or Method 25B and Method 2A, respectively. Organic carbon, carbon dioxide (CO_2), and carbon monoxide (CO) concentrations are measured at the outlet using either Method 25A or Method 25B and Method 10, respectively. The ratio of total carbon at the incinerator inlet and outlet is multiplied by the inlet volume to determine the exhaust volume flow rate.

3.0 Definitions

Same as Section 3.0 of Method 10 and Method 25A.

4.0 Interferences

Same as Section 4.0 of Method 10.

5.0 Safety

5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Section 6.0 of Method 2A, Method 10, and Method 25A and/or Method 25B as applicable, with the addition of the following:

6.1 This analyzer must meet the specifications set forth in Section 6.1.2 of Method 10, except that the span shall be 15 percent CO_2 by volume.

7.0 Reagents and Standards

Same as Section 7.0 of Method 10 and Method 25A, with the following addition and exceptions:

7.1 Carbon Dioxide Analyzer Calibration. CO_2 gases meeting the specifications set forth in Section 7 of Method 6C are required.

7.2 Hydrocarbon Analyzer Calibration. Methane shall not be used as a calibration gas when performing this method.

7.3 Fuel Gas. If Method 25B is used to measure the organic carbon concentrations at both the inlet and exhaust, no fuel gas is required.

8.0 Sample Collection and Analysis

8.1 Pre-test Procedures. Perform all pre-test procedures (*e.g.*, system performance checks, leak checks) necessary to determine gas volume flow rate and organic carbon concentration in the vapor line to the incinerator inlet and to determine organic carbon, carbon monoxide, and carbon dioxide concentrations at the incinerator exhaust, as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable.

8.2 Sampling. At the beginning of the test period, record the initial parameters for the inlet volume meter according to the procedures in Method 2A and mark all of the recorder strip charts to indicate the start of the test. Conduct sampling and analysis as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable. Continue recording inlet organic and exhaust CO_2, CO, and organic concentrations throughout the test. During periods of process interruption and halting of gas flow, stop the timer and mark the recorder strip charts so that data from this interruption are not included in the calculations. At the end of the test period, record the final parameters for the inlet volume meter and mark the end on all of the recorder strip charts.

8.3 Post-test Procedures. Perform all post-test procedures (*e.g.*, drift tests, leak checks), as outlined in Method 2A, Method 10, and Method 25A and/or Method 25B as applicable.

9.0 Quality Control

Same as Section 9.0 of Method 2A, Method 10, and Method 25A.

10.0 Calibration and Standardization

Same as Section 10.0 of Method 2A, Method 10, and Method 25A.

Note: If a manifold system is used for the exhaust analyzers, all the analyzers and sample pumps must be operating when the analyzer calibrations are performed.

10.1 If an analyzer output does not meet the specifications of the method, invalidate the test data for the period. Alternatively, calculate the exhaust volume results using initial calibration data and using final calibration data and report both resulting volumes. Then, for emissions calculations, use the volume measurement resulting in the greatest emission rate or concentration.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

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

12.1 Nomenclature.

Co_c = Mean carbon monoxide concentration in system exhaust, ppm.

$(CO_2)_2$ = Ambient carbon dioxide concentration, ppm (if not measured during the test period, may be assumed to equal 300 ppm).

$(CO_2)_c$ = Mean carbon dioxide concentration in system exhaust, ppm.

HC_c = Mean organic concentration in system exhaust as defined by the calibration gas, ppm.

Hc_i = Mean organic concentration in system inlet as defined by the calibration gas, ppm.

K_c = Hydrocarbon calibration gas factor for the exhaust hydrocarbon analyzer, unitless [equal to the number of carbon atoms per molecule of the gas used to calibrate the analyzer (2 for ethane, 3 for propane, etc.)].

K_i = Hydrocarbon calibration gas factor for the inlet hydrocarbon analyzer, unitless.

V_{cs} = Exhaust gas volume, m^3.

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

Q_{es} = Exhaust gas volume flow rate, m^3/min.

Q_{is} = Inlet gas volume flow rate, m^3/min.

θ = Sample run time, min.

s = Standard conditions: 20 °C, 760 mm Hg.

12.2 Concentrations. Determine mean concentrations of inlet organics, outlet CO_2, outlet CO, and outlet organics according to the procedures in the respective methods and the analyzers' calibration curves, and for the time intervals specified in the applicable regulations.

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

$$V_{cs} = V_{is}\frac{K_i(HC_i)}{K_c(HC_c)+\left[(CO_2)_c-(CO_2)_a\right]+CO_c} \qquad \text{Eq. 2B-1}$$

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

$$Q_{cs} = \frac{V_{es}}{\Theta} \qquad \text{Eq. 2B-2}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Section 16.0 of Method 2A, Method 10, and Method 25A.

17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

Method 2C—Determination of Gas Velocity and Volumetric Flow Rate in Small Stacks or Ducts (Standard Pitot Tube)

Note: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2.

1.0 Scope and Application

1.1 This method is applicable for the determination of average velocity and volumetric flow rate of gas streams in small stacks or ducts. Limits on the applicability of this method are identical to those set forth in Method 2, Section 1.0, except that this method is limited to stationary source stacks or ducts less than about 0.30 meter (12 in.) in diameter, or 0.071 m^2 (113 $in.^2$) in cross-sectional area, but equal to or greater than about 0.10 meter (4 in.) in diameter, or 0.0081 m^2 (12.57 $in.^2$) in cross-sectional area.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

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

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Method 2, Section 6.0, with the exception of the following:

6.1 Standard Pitot Tube (instead of Type S). A standard pitot tube which meets the specifications of Section 6.7 of Method 2. Use a coefficient of 0.99 unless it is calibrated against another standard pitot tube with a NIST-traceable coefficient (see Section 10.2 of Method 2).

6.2 Alternative Pitot Tube. A modified hemispherical-nosed pitot tube (see Figure 2C–1), which features a shortened stem and enlarged impact and static pressure holes. Use a coefficient of 0.99 unless it is calibrated as mentioned in Section 6.1 above. This pitot tube is useful in particulate liquid droplet-laden gas streams when a "back purge" is ineffective.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection and Analysis

8.1 Follow the general procedures in Section 8.0 of Method 2, except conduct the measurements at the traverse points specified in Method 1A. The static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, adequate proof that the openings of the pitot tube have not plugged during the traverse period must be furnished; this can be done by taking the velocity head (Δp) heading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (within ±5 percent) the traverse is acceptable. Otherwise, reject the run. Note that if the Δp at the final traverse point is unsuitably low, another point may be selected. If "back purging" at regular intervals is part of the procedure, then take comparative Δp readings, as above, for the last two back purges at which suitably high Δp readings are observed.

9.0 Quality Control

Section	Quality control measure	Effect
10.0	Sampling equipment calibration	Ensure accurate measurement of stack gas velocity head.

10.0 Calibration and Standardization

Same as Method 2, Sections 10.2 through 10.4.

11.0 Analytical Procedure

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Calculations and Data Analysis

Same as Method 2, Section 12.0.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 2, Section 16.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

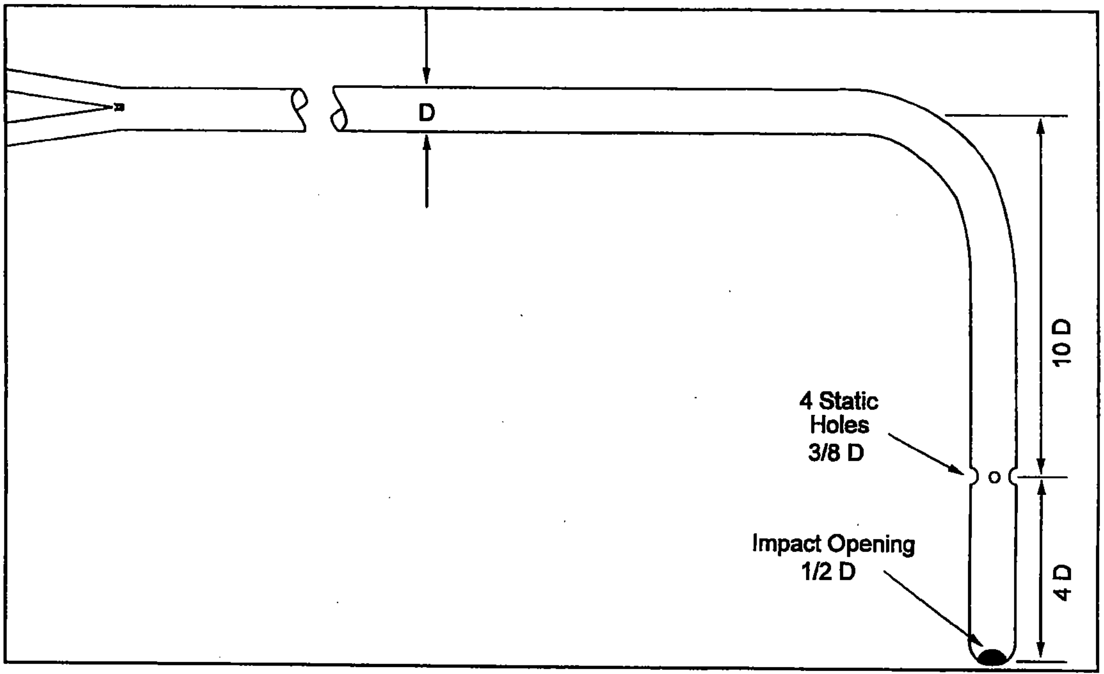


Figure 2C-1. Modified Hemispherical-Nosed Pitot Tube.

Method 2D—Measurement of Gas Volume Flow Rates in Small Pipes and Ducts

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (e.g., sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, and Method 2A.

1.0 Scope and Application

1.1 This method is applicable for the determination of the volumetric flow rates of gas streams in small pipes and ducts. It can be applied to intermittent or variable gas flows only with particular caution.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 All the gas flow in the pipe or duct is directed through a rotameter, orifice plate or similar device to measure flow rate or pressure drop. The device has been previously calibrated in a manner that insures its proper calibration for the gas being measured. Absolute temperature and pressure measurements are made to allow correction of volumetric flow rates to standard conditions.

3.0 Definitions. [Reserved]

4.0 Interferences. [Reserved]

5.0 Safety

5.1 This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

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

6.1 Gas Metering Rate or Flow Element Device. A rotameter, orifice plate, or other volume rate or pressure drop measuring device capable of measuring the stack flow rate to within ±5 percent. The metering device shall be equipped with a temperature gauge accurate to within ±2 percent of the minimum absolute stack temperature and a pressure gauge (accurate to within ±5 mm Hg). The capacity of the metering device shall be sufficient for the expected maximum and minimum flow rates at the stack gas conditions. The magnitude and variability of stack gas flow rate, molecular weight, temperature, pressure, dewpoint, and corrosive characteristics, and pipe or duct size are factors to consider in choosing a suitable metering device.

6.2 Barometer. Same as Method 2, Section 6.5.

6.3 Stopwatch. Capable of measurement to within 1 second.

7.0 Reagents and Standards. [Reserved]

8.0 Sample Collection and Analysis

8.1 Installation and Leak Check. Same as Method 2A, Sections 8.1 and 8.2, respectively.

8.2 Volume Rate Measurement.

8.2.1 Continuous, Steady Flow. At least once an hour, record the metering device flow rate or pressure drop reading, and the metering device temperature and pressure. Make a minimum of 12 equally spaced readings of each parameter during the test period. Record the barometric pressure at the beginning and end of the test period. Record the data on a table similar to that shown in Figure 2D–1.

8.2.2 Noncontinuous and Nonsteady Flow. Use volume rate devices with particular caution. Calibration will be affected by variation in stack gas temperature, pressure and molecular weight. Use the procedure in Section 8.2.1 with the addition of the following: Record all the metering device parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

9.0 Quality Control

Section	Quality control measure	Effect
10.0	Sampling equipment calibration	Ensure accurate measurement of stack gas flow rate or sample volume.

10.0 Calibration and Standardization

Same as Method 2A, Section 10.0, with the following exception:

10.1 Gas Metering Device. Same as Method 2A, Section 10.1, except calibrate the metering device with the principle stack gas to be measured (examples: air, nitrogen) against a standard reference meter. A calibrated dry gas meter is an acceptable reference meter. Ideally, calibrate the metering device in the field with the actual gas to be metered. For metering devices that have a volume rate readout, calculate the test metering device calibration coefficient, Y_m, for each run shown in Equation 2D–2 Section 12.3.

10.2 For metering devices that do not have a volume rate readout, refer to the manufacturer's instructions to calculate the V_{m2} corresponding to each V_r.

10.3 Temperature Gauge. Use the procedure and specifications in Method 2A, Section 10.2. Perform the calibration at a temperature that approximates field test conditions.

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

11.0 Analytical Procedure.

Sample collection and analysis are concurrent for this method (see Section 8.0).

12.0 Data Analysis and Calculations

12.1 Nomenclature.

P_{bar} = Barometric pressure, mm Hg (in. Hg).
P_m = Test meter average static pressure, mm Hg (in. Hg).
Q_r = Reference meter volume flow rate reading, m³/min (ft³/min).
Q_m = Test meter volume flow rate reading, m³/min (ft³/min).
T_r = Absolute reference meter average temperature, °K (°R).
T_m = Absolute test meter average temperature, °K (°R).
K_l = 0.3855 °K/mm Hg for metric units, = 17.65 °R/in. Hg for English units.

12.2 Gas Flow Rate.

$$Q_s = K_l Y_m Q_m \frac{(P_{bar} + P_m)}{T_m} \qquad \text{Eq. 2D-1}$$

12.3 Test Meter Device Calibration Coefficient. Calculation for testing metering device calibration coefficient, Y_m.

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

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

1. Spink, L.K. Principles and Practice of Flowmeter Engineering. The Foxboro Company. Foxboro, MA. 1967.
2. Benedict, R.P. Fundamentals of Temperature, Pressure, and Flow Measurements. John Wiley & Sons, Inc. New York, NY. 1969.
3. Orifice Metering of Natural Gas. American Gas Association. Arlington, VA. Report No. 3. March 1978. 88 pp.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Plant
Date
Run No.
Sample location
Barometric pressure (mm Hg):
Start
Finish
Operators
Metering device No.
Calibration coefficient
Calibration gas
Date to recalibrate

Time	Flow rate reading	Static Pressure [mm Hg (in. Hg)]	Temperature	
			°C (°F)	°K (°R)
Average				

Figure 2D–1. Volume Flow Rate Measurement Data

Method 2E—Determination of Landfill Gas Production Flow Rate

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of at least the following additional test methods: Methods 2 and 3C.

1.0 Scope and Application

1.1 Applicability. This method applies to the measurement of landfill gas (LFG) production flow rate from municipal solid waste landfills and is used to calculate the flow rate of nonmethane organic compounds (NMOC) from landfills.

1.2 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Extraction wells are installed either in a cluster of three or at five dispersed locations in the landfill. A blower is used to extract LFG from the landfill. LFG composition, landfill pressures, and orifice pressure differentials from the wells are measured and the landfill gas production flow rate is calculated.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Since this method is complex, only experienced personnel should perform the test. Landfill gas contains methane, therefore explosive mixtures may exist at or near the landfill. It is advisable to take appropriate safety precautions when testing landfills, such as refraining from smoking and installing explosion-proof equipment.

6.0 Equipment and Supplies

6.1 Well Drilling Rig. Capable of boring a 0.61 m (24 in.) diameter hole into the landfill to a minimum of 75 percent of the landfill depth. The depth of the well shall not extend to the bottom of the landfill or the liquid level.

6.2 Gravel. No fines. Gravel diameter should be appreciably larger than perforations stated in Sections 6.10 and 8.2.

6.3 Bentonite.

6.4 Backfill Material. Clay, soil, and sandy loam have been found to be acceptable.

6.5 Extraction Well Pipe. Minimum diameter of 3 in., constructed of polyvinyl chloride (PVC), high density polyethylene (HDPE), fiberglass, stainless steel, or other suitable nonporous material capable of transporting landfill gas.

6.6 Above Ground Well Assembly. Valve capable of adjusting gas flow, such as a gate, ball, or butterfly valve; sampling ports at the well head and outlet; and a flow measuring device, such as an in-line orifice meter or pitot tube. A schematic of the aboveground well head assembly is shown in Figure 2E–1.

6.7 Cap. Constructed of PVC or HDPE.

6.8 Header Piping. Constructed of PVC or HDPE.

6.9 Auger. Capable of boring a 0.15- to 0.23-m (6-to 9-in.) diameter hole to a depth equal to the top of the perforated section of the extraction well, for pressure probe installation.

6.10 Pressure Probe. Constructed of PVC or stainless steel (316), 0.025-m (1-in.). Schedule 40 pipe. Perforate the bottom two-thirds. A minimum requirement for perforations is slots or holes with an open area equivalent to four 0.006-m (¼-in.) diameter holes spaced 90° apart every 0.15 m (6 in.).

6.11 Blower and Flare Assembly. Explosion-proof blower, capable of extracting LFG at a flow rate of 8.5 m^3/min (300 ft^3/min), a water knockout, and flare or incinerator.

6.12 Standard Pitot Tube and Differential Pressure Gauge for Flow Rate Calibration with Standard Pitot. Same as Method 2, Sections 6.7 and 6.8.

6.13 Orifice Meter. Orifice plate, pressure tabs, and pressure measuring device to measure the LFG flow rate.

6.14 Barometer. Same as Method 4, Section 6.1.5.

6.15 Differential Pressure Gauge. Water-filled U-tube manometer or equivalent, capable of measuring within 0.02 mm Hg (0.01 in. H_2O), for measuring the pressure of the pressure probes.

7.0 Reagents and Standards. Not Applicable

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Placement of Extraction Wells. The landfill owner or operator may install a single cluster of three extraction wells in a test area or space five equal-volume wells over the landfill. The cluster wells are recommended but may be used only if the composition, age of the refuse, and the landfill depth of the test area can be determined.

8.1.1 Cluster Wells. Consult landfill site records for the age of the refuse, depth, and composition of various sections of the landfill. Select an area near the perimeter of the landfill with a depth equal to or greater than the average depth of the landfill and with the average age of the refuse between 2 and 10 years old. Avoid areas known to contain nondecomposable materials, such as concrete and asbestos. Locate the cluster wells as shown in Figure 2E–2.

8.1.1.1 The age of the refuse in a test area will not be uniform, so calculate a weighted average age of the refuse as shown in Section 12.2.

8.1.2 Equal Volume Wells. Divide the sections of the landfill that are at least 2 years old into five areas representing equal volumes. Locate an extraction well near the center of each area.

8.2 Installation of Extraction Wells. Use a well drilling rig to dig a 0.6 m (24 in.) diameter hole in the landfill to a minimum of 75 percent of the landfill depth, not to extend to the bottom of the landfill or the liquid level. Perforate the bottom two thirds of the extraction well pipe. A minimum requirement for perforations is holes or slots with an open area equivalent to 0.01-m (0.5-in.) diameter holes spaced 90° apart every 0.1 to 0.2 m (4 to 8 in.). Place the extraction well in the center of the hole and backfill with gravel to a level 0.30 m (1 ft) above the perforated section. Add a layer of backfill material 1.2 m (4 ft) thick. Add a layer of bentonite 0.9 m (3 ft) thick, and backfill the remainder of the hole with cover material or material equal in permeability to the existing cover material. The specifications for extraction well installation are shown in Figure 2E–3.

8.3 Pressure Probes. Shallow pressure probes are used in the check for infiltration of air into the landfill, and deep pressure probes are use to determine the radius of influence. Locate pressure probes along three radial arms approximately 120° apart at distances of 3, 15, 30, and 45 m (10, 50, 100, and 150 ft) from the extraction well. The tester has the option of locating additional pressure probes at distances every 15 m (50 feet) beyond 45 m (150 ft). Example placements of probes are shown in Figure 2E–4. The 15-, 30-, and 45-m, (50-, 100-, and 150-ft) probes from each well, and any additional probes located along the three radial arms (deep probes), shall extend to a depth equal to the top of the perforated section of the extraction wells. All other probes (shallow probes) shall extend to a depth equal to half the depth of the deep probes.

8.3.1 Use an auger to dig a hole, 0.15- to 0.23-m (6-to 9-in.) in diameter, for each pressure probe. Perforate the bottom two thirds of the pressure probe. A minimum requirement for perforations is holes or slots with an open area equivalent to four 0.006-m (0.25-in.) diameter holes spaced 90° apart every 0.15 m (6 in.). Place the pressure probe in the center of the hole and backfill with gravel to a level 0.30 m (1 ft) above the perforated section. Add a layer of backfill material at least 1.2 m (4 ft) thick. Add a layer of bentonite at least 0.3 m (1 ft) thick, and backfill the remainder of the hole with cover material or material equal in permeability to the existing cover material. The specifications for pressure probe installation are shown in Figure 2E–5.

8.4 LFG Flow Rate Measurement. Place the flow measurement device, such as an orifice meter, as shown in Figure 2E–1. Attach the wells to the blower and flare assembly. The individual wells may be ducted to a common header so that a single blower, flare assembly, and flow meter may be used. Use the procedures in Section 10.1 to calibrate the flow meter.

8.5 Leak-Check. A leak-check of the above ground system is required for accurate flow rate measurements and for safety. Sample LFG at the well head sample port and at the outlet sample port. Use Method 3C to determine nitrogen (N_2) concentrations. Determine the difference between the well head and outlet N_2 concentrations using the formula in Section 12.3. The system passes the leak-check if the difference is less than 10,000 ppmv.

8.6 Static Testing. Close the control valves on the well heads during static testing. Measure the gauge pressure (P_g) at each deep pressure probe and the barometric pressure (P_{bar}) every 8 hours (hr) for 3 days. Convert the gauge pressure of each deep pressure probe to absolute pressure using the equation in Section 12.4. Record as P_i (initial absolute pressure).

8.6.1 For each probe, average all of the 8-hr deep pressure probe readings (P_i) and record as P_{ia} (average absolute pressure). P_{ia} is used in Section 8.7.5 to determine the maximum radius of influence.

8.6.2 Measure the static flow rate of each well once during static testing.

8.7 Short-Term Testing. The purpose of short-term testing is to determine the maximum vacuum that can be applied to the wells without infiltration of ambient air into the landfill. The short-term testing is performed on one well at a time. Burn all LFG with a flare or incinerator.

8.7.1 Use the blower to extract LFG from a single well at a rate at least twice the static flow rate of the respective well measured in Section 8.6.2. If using a single blower and flare assembly and a common header system, close the control valve on the wells not being measured. Allow 24 hr for the system to stabilize at this flow rate.

8.7.2 Test for infiltration of air into the landfill by measuring the gauge pressures of the shallow pressure probes and using Method 3C to determine the LFG N_2 concentration. If the LFG N_2 concentration is less than 5 percent and all of the shallow probes have a positive gauge pressure, increase the blower vacuum by 3.7 mm Hg (2 in. H_2O), wait 24 hr, and repeat the tests for infiltration. Continue the above steps of increasing blower vacuum by 3.7 mm Hg (2 in. H_2O), waiting 24 hr, and testing for infiltration until the concentration of N_2 exceeds 5 percent or any of the shallow probes have a negative gauge pressure. When this occurs,reduce the blower vacuum to the maximum setting at which the N_2 concentration was less than 5 percent and the gauge pressures of the shallow probes are positive.

8.7.3 At this blower vacuum, measure atmospheric pressure (P_{bar}) every 8 hr for 24 hr, and record the LFG flow rate (Q_s) and the probe gauge pressures (P_f) for all of the probes. Convert the gauge pressures of the deep probes to absolute pressures for each 8-hr reading at Q_s as shown in Section 12.4.

8.7.4 For each probe, average the 8-hr deep pressure probe absolute pressure readings and record as P_{fa} (the final average absolute pressure).

8.7.5 For each probe, compare the initial average pressure (P_{ia}) from Section 8.6.1 to the final average pressure (P_{fa}). Determine the furthermost point from the well head along each radial arm where $P_{fa} \leq P_{ia}$. This distance is the maximum radius of influence (R_m), which is the distance from the well affected by the vacuum. Average these values to determine the average maximum radius of influence (R_{ma}).

8.7.6 Calculate the depth (D_{st}) affected by the extraction well during the short term test as shown in Section 12.6. If the computed value of D_{st} exceeds the depth of the landfill, set D_{st} equal to the landfill depth.

8.7.7 Calculate the void volume (V) for the extraction well as shown in Section 12.7.

8.7.8 Repeat the procedures in Section 8.7 for each well.

8.8 Calculate the total void volume of the test wells (V_v) by summing the void volumes (V) of each well.

8.9 Long-Term Testing. The purpose of long-term testing is to extract two void volumes of LFG from the extraction wells. Use the blower to extract LFG from the wells. If a single Blower and flare assembly and common header system are used, open all control valves and set the blower vacuum equal to the highest stabilized blower vacuum demonstrated by any individual well in Section 8.7. Every 8 hr, sample the LFG from the well head sample port, measure the gauge pressures of the shallow pressure probes, the blower vacuum, the LFG flow rate, and use the criteria for infiltration in Section 8.7.2 and Method 3C to test for infiltration. If infiltration is detected, do not reduce the blower vacuum, instead reduce the LFG flow rate from the well by adjusting the control valve on the well head. Adjust each affected well individually. Continue until the equivalent of two total void volumes (V_v) have been extracted, or until $V_t = 2V_v$.

8.9.1 Calculate V_t, the total volume of LFG extracted from the wells, as shown in Section 12.8.

8.9.2 Record the final stabilized flow rate as Q_f and the gauge pressure for each deep probe. If, during the long term testing, the flow rate does not stabilize, calculate Q_f by averaging the last 10 recorded flow rates.

8.9.3 For each deep probe, convert each gauge pressure to absolute pressure as in Section 12.4. Average these values and record as P_{sa}. For each probe, compare P_{ia} to P_{sa}. Determine the furthermost point from the well head along each radial arm where $P_{sa} \leq P_{ia}$. This distance is the stabilized radius of influence. Average these values to determine the average stabilized radius of influence (R_{sa}).

8.10 Determine the NMOC mass emission rate using the procedures in Section 12.9 through 12.15.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
10.1	LFG flow rate meter calibration	Ensures accurate measurement of LFG flow rate and sample volume

10.0 Calibration and Standardization

10.1 LFG Flow Rate Meter (Orifice) Calibration Procedure. Locate a standard pitot tube in line with an orifice meter. Use the procedures in Section 8, 12.5, 12.6, and 12.7 of Method 2 to determine the average dry gas volumetric flow rate for at least five flow rates that bracket the expected LFG flow rates, except in Section 8.1, use a standard pitot tube rather than a Type S pitot tube. Method 3C may be used to determine the dry molecular weight. It may be necessary to calibrate more than one orifice meter in order to bracket the LFG flow rates. Construct a calibration curve by plotting the pressure drops across the orifice meter for each flow rate versus the average dry gas volumetric flow rate in m³/min of the gas.

11.0 Procedures [Reserved]

12.0 Data Analysis and Calculations

12.1 Nomenclature.

A = Age of landfill, yr.
A_{avg} = Average age of the refuse tested, yr.
A_i = Age of refuse in the ith fraction, yr.
A_r = Acceptance rate, Mg/yr.
C_{NMOC} = NMOC concentration, ppmv as hexane ($C_{NMOC} = C_t/6$).
C_o = Concentration of N_2 at the outlet, ppmv.
C_t = NMOC concentration, ppmv (carbon equivalent) from Method 25C.
C_w = Concentration of N_2 at the wellhead, ppmv.
D = Depth affected by the test wells, m.
D_{st} = Depth affected by the test wells in the short-term test, m.
e = Base number for natural logarithms (2.718).
f = Fraction of decomposable refuse in the landfill.
f_i = Fraction of the refuse in the ith section.
k = Landfill gas generation constant, yr^{-1}.
L_o = Methane generation potential, m³/Mg.
L_o' = Revised methane generation potential to account for the amount of nondecomposable material in the landfill, m³/Mg.
M_i = Mass of refuse in the ith section, Mg.
M_r = Mass of decomposable refuse affected by the test well, Mg.
P_{bar} = Atmospheric pressure, mm Hg.
P_f = Final absolute pressure of the deep pressure probes during short-term testing, mm Hg.
P_{fa} = Average final absolute pressure of the deep pressure probes during short-term testing, mm Hg.
P_{gf} = final gauge pressure of the deep pressure probes, mm Hg.
P_{gi} = Initial gauge pressure of the deep pressure probes, mm Hg.
P_i = Initial absolute pressure of the deep pressure probes during static testing, mm Hg.
P_{ia} = Average initial absolute pressure of the deep pressure probes during static testing, mm Hg.
P_s = Final absolute pressure of the deep pressure probes during long-term testing, mm Hg.
P_{sa} = Average final absolute pressure of the deep pressure probes during long-term testing, mm Hg.
Q_f = Final stabilized flow rate, m³/min.
Q_i = LFG flow rate measured at orifice meter during the ith interval, m³/min.
Q_s = Maximum LFG flow rate at each well determined by short-term test, m³/min.
Q_t = NMOC mass emission rate, m³/min.
R_m = Maximum radius of influence, m.
R_{ma} = Average maximum radius of influence, m.
R_s = Stabilized radius of influence for an individual well, m.
R_{sa} = Average stabilized radius of influence, m.
t_i = Age of section i, yr.
t_t = Total time of long-term testing, yr.
t_{vi} = Time of the ith interval (usually 8), hr.
V = Void volume of test well, m³.
V_r = Volume of refuse affected by the test well, m³.
V_t = Total volume of refuse affected by the long-term testing, m³.
V_v = Total void volume affected by test wells, m³.
WD = Well depth, m.
ρ = Refuse density, Mg/m³ (Assume 0.64 Mg/m³ if data are unavailable).

12.2 Use the following equation to calculate a weighted average age of landfill refuse.

$$A_{avg} = \sum_{i=1}^{n} f_i A_i \qquad \text{Eq. 2E-1}$$

12.3 Use the following equation to determine the difference in N_2 concentrations (ppmv) at the well head and outlet location.

$$\text{Difference} = C_o - C_w \qquad \text{Eq. 2E-2}$$

12.4 Use the following equation to convert the gauge pressure (P_g) of each initial deep pressure probe to absolute pressure (P_i).

$$P_i = P_{bar} + P_{gi} \qquad \text{Eq. 2E-3}$$

12.5 Use the following equation to convert the gauge pressures of the deep probes to absolute pressures for each 8-hr reading at Q_s.

$$P_f = P_{bar} + P_{gf} \qquad \text{Eq. 2E-4}$$

12.6 Use the following equation to calculate the depth (D_{st}) affected by the extraction well during the short-term test.

$$D_{st} = WD + R_{ma} \qquad \text{Eq. 2E-5}$$

12.7 Use the following equation to calculate the void volume for the extraction well (V).

$$V = 0.40\, \Pi\, R_{ma}^{2} D_{st} \qquad \text{Eq. 2E-6}$$

12.8 Use the following equation to calculate V_t, the total volume of LFG extracted from the wells.

$$V_t = \sum_{i=1}^{n} 60\, Q_i t_{vi} \qquad \text{Eq. 2E-7}$$

12.9 Use the following equation to calculate the depth affected by the test well. If using cluster wells, use the average depth of the wells for WD. If the value of D is greater than the depth of the landfill, set D equal to the landfill depth.

$$D = WD + R_{sa} \qquad \text{Eq. 2E-8}$$

12.10 Use the following equation to calculate the volume of refuse affected by the test well.

$$V_r = R_{sa}^{2}\, \Pi\, D \qquad \text{Eq. 2E-9}$$

12.11 Use the following equation to calculate the mass affected by the test well.

$$M_r = V_r \rho \qquad \text{Eq. 2E-10}$$

12.12 Modify L_o to account for the nondecomposable refuse in the landfill.

$$L_o' = f L_o \qquad \text{Eq. 2E-11}$$

12.13 In the following equation, solve for k (landfill gas generation constant) by iteration. A suggested procedure is to select a value for k, calculate the left side of the equation, and if not equal to zero, select another value for k. Continue this process until the left hand side of the equation equals zero, ±0.001.

$$k_e{}^{-k}A_{avg} - \frac{Q_f}{2\,L_o'M_r} = 0 \qquad \text{Eq. 2E-12}$$

12.14 Use the following equation to determine landfill NMOC mass emission rate if the yearly acceptance rate of refuse has been consistent (10 percent) over the life of the landfill.

$$Q_t = 2\,L_o'A_r\left(1 - e^{-kA}\right)C_{NMOC}\left(3.595 \times 10^{-9}\right) \qquad \text{Eq. 2E-13}$$

12.15 Use the following equation to determine landfill NMOC mass emission rate if the acceptance rate has not been consistent over the life of the landfill.

$$Q_t = 2\,k\,L_o'C_{NMOC}\left(3.595 \times 10^{-9}\right)\sum_{i=1}^{n} M_i e^{-kt_i} \qquad \text{Eq. 2E-14}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Same as Method 2, Appendix A, 40 CFR Part 60.

2. Emcon Associates, Methane Generation and Recovery from Landfills. Ann Arbor Science, 1982.

3. The Johns Hopkins University, Brown Station Road Landfill Gas Resource Assessment, Volume 1: Field Testing and Gas Recovery Projections. Laurel, Maryland: October 1982.

4. Mandeville and Associates, Procedure Manual for Landfill Gases Emission Testing.

5. Letter and attachments from Briggum, S., Waste Management of North America, to Thorneloe, S., EPA. Response to July 28, 1988 request for additional information. August 18, 1988.

6. Letter and attachments from Briggum, S., Waste Management of North America, to Wyatt, S., EPA. Response to December 7, 1988 request for additional information. January 16, 1989.

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17.0 Tables, Diagrams, Flowcharts, and Validation Data

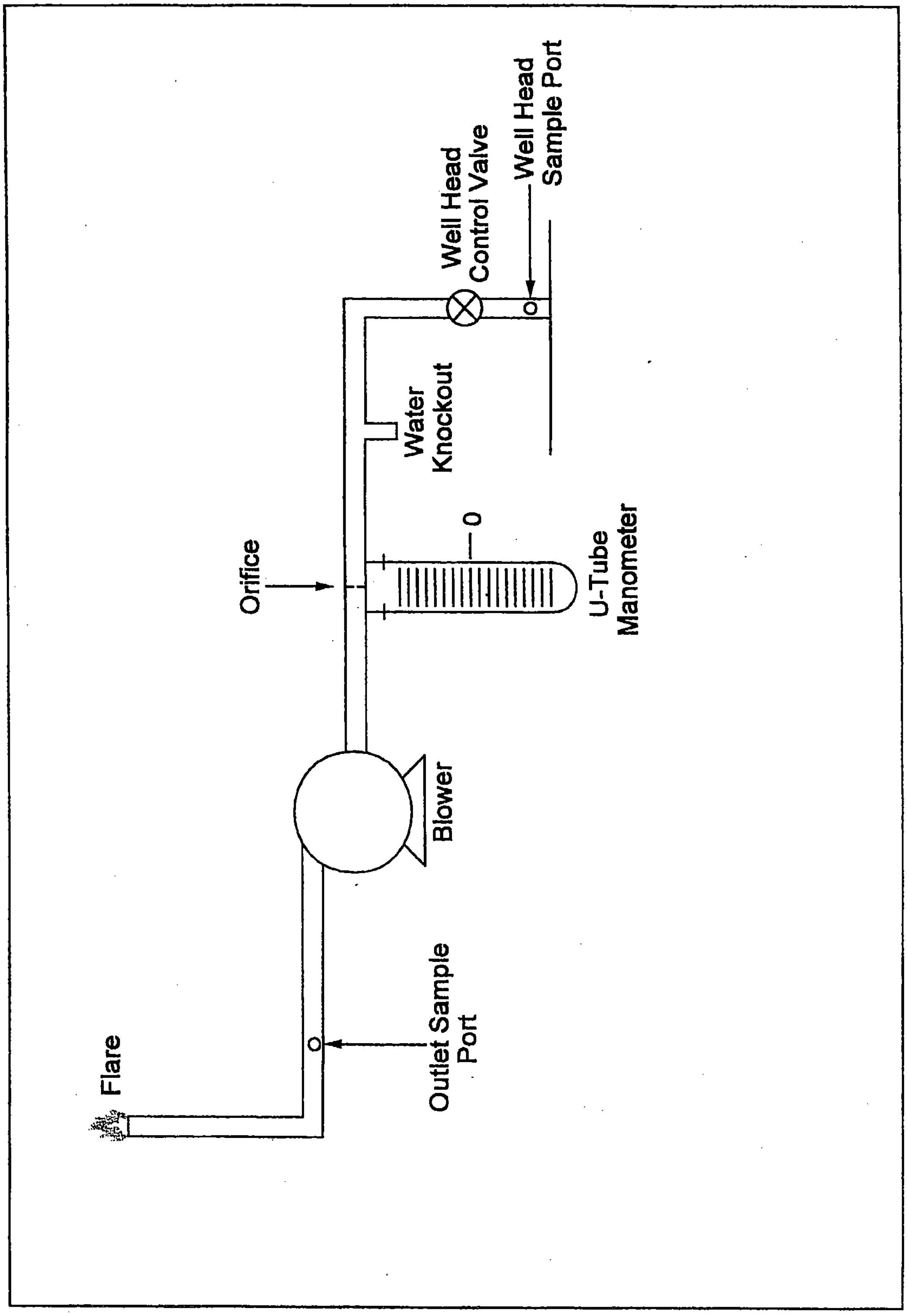

Figure 2E-1. Schematic of Aboveground Well Head Assembly.

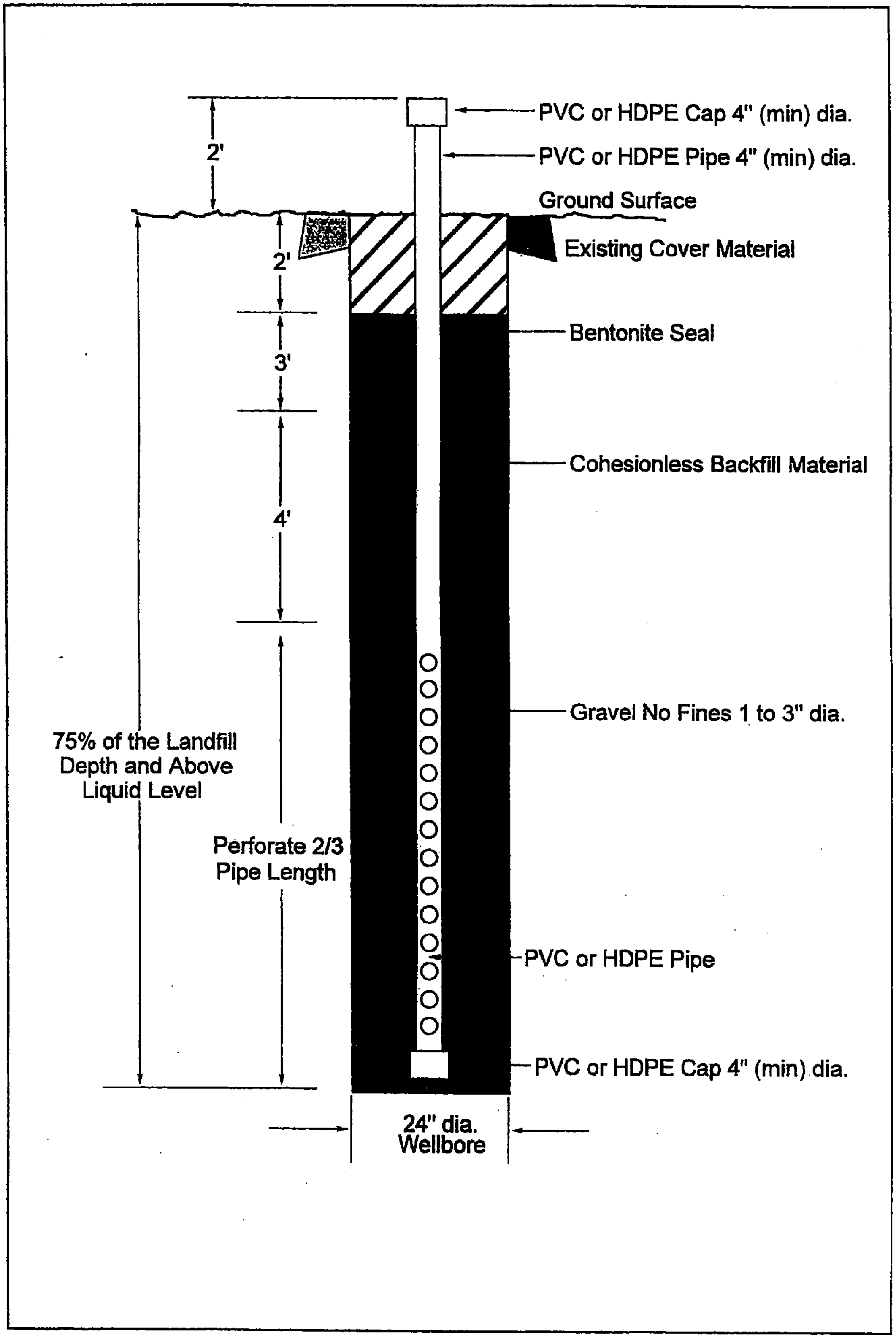

Figure 2E-3. Gas Extraction Well.

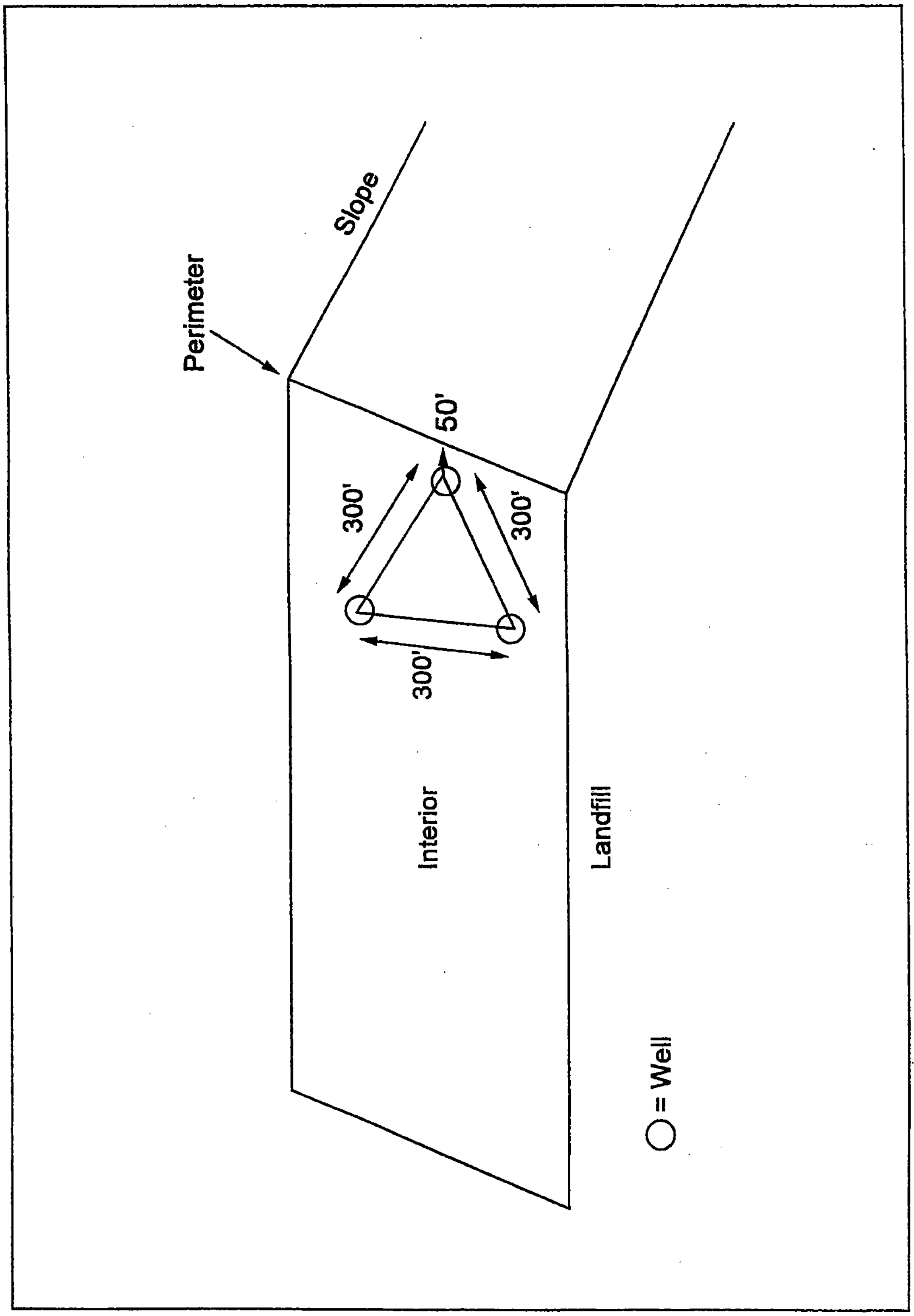

Figure 2E-2. Cluster Well Placement.

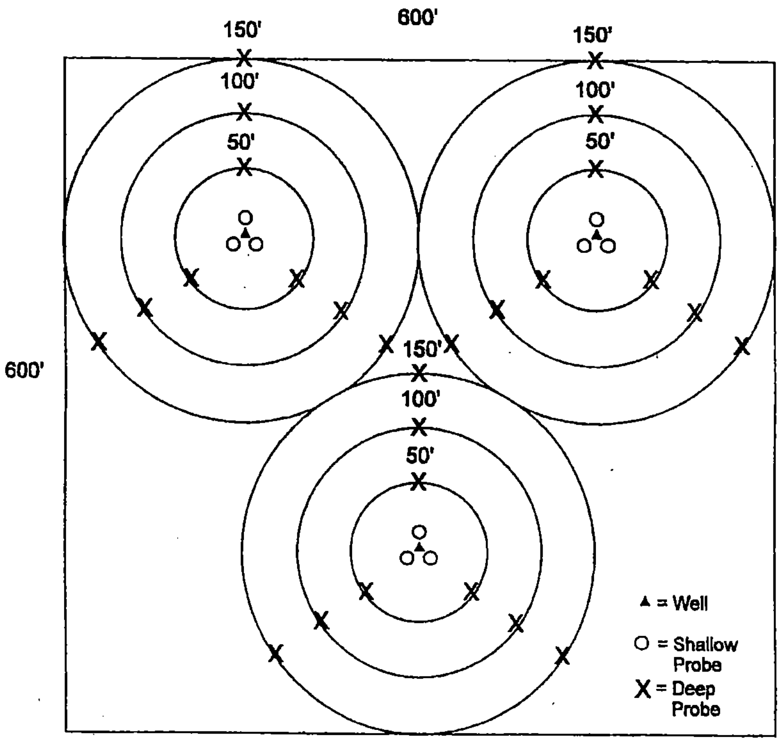

Figure 2E-4. Cluster Well Configuration.

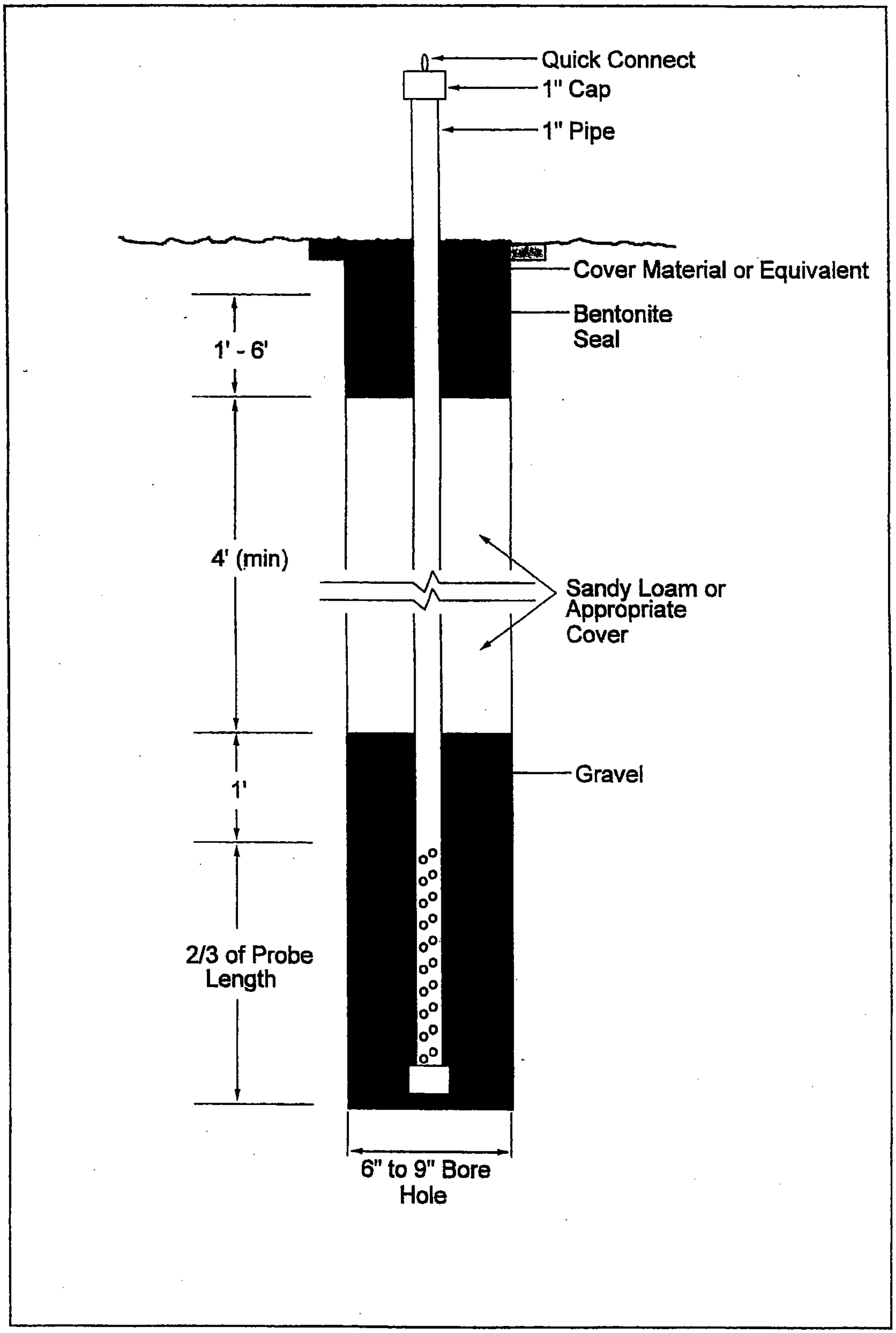

Figure 2E-5. Pressure Probe.

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Method 3—Gas Analysis for the Determination of Dry Molecular Weight

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should also have a thorough knowledge of Method 1.

1.0 Scope and Application

1.1 Analytes.

Analytes	CAS No.	Sensitivity
Oxygen (O_2)	7782–44–7	2,000 ppmv.
Nitrogen (N_2)	7727–37–9	N/A.
Carbon dioxide (CO_2)	124–38–9	2,000 ppmv.
Carbon monoxide (CO)	630–08–0	N/A.

1.2 Applicability. This method is applicable for the determination of CO_2 and O_2 concentrations and dry molecular weight of a sample from an effluent gas stream of a fossil-fuel combustion process or other process.

1.3 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point grab sampling method using an Orsat analyzer to analyze the individual grab sample obtained at each point; (2) a method for measuring either CO_2 or O_2 and using stoichiometric calculations to determine dry molecular weight; and (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, carbon monoxide (CO), and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

1.4 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas sample is extracted from a stack by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2 and percent O_2. For dry molecular weight determination, either an Orsat or a Fyrite analyzer may be used for the analysis.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 Several compounds can interfere, to varying degrees, with the results of Orsat or Fyrite analyses. Compounds that interfere with CO_2 concentration measurement include acid gases (*e.g.*, sulfur dioxide, hydrogen chloride); compounds that interfere with O_2 concentration measurement include unsaturated hydrocarbons (*e.g.*, acetone, acetylene), nitrous oxide, and ammonia. Ammonia reacts chemically with the O_2 absorbing solution, and when present in the effluent gas stream must be removed before analysis.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents.

5.2.1 A typical Orsat analyzer requires four reagents: a gas-confining solution, CO_2 absorbent, O_2 absorbent, and CO absorbent. These reagents may contain potassium hydroxide, sodium hydroxide, cuprous chloride, cuprous sulfate, alkaline pyrogallic acid, and/or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.

5.2.2 A typical Fyrite analyzer contains zinc chloride, hydrochloric acid, and either potassium hydroxide or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.

6.0 Equipment and Supplies

Note: As an alternative to the sampling apparatus and systems described herein, other sampling systems (*e.g.*, liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

6.1 Grab Sampling (See Figure 3–1).

6.1.1 Probe. Stainless steel or borosilicate glass tubing equipped with an in-stack or out-of-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials, resistant to temperature at sampling conditions and inert to all components of the gas stream, may be used for the probe. Examples of such materials may include aluminum, copper, quartz glass, and Teflon.

6.1.2 Pump. A one-way squeeze bulb, or equivalent, to transport the gas sample to the analyzer.

6.2 Integrated Sampling (Figure 3–2).

6.2.1 Probe. Same as in Section 6.1.1.

6.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser no greater than 250 ml that will not remove O_2, CO_2, CO, and N_2, to remove excess moisture which would interfere with the operation of the pump and flowmeter.

6.2.3 Valve. A needle valve, to adjust sample gas flow rate.

6.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rate meter.

6.2.5 Rate Meter. A rotameter, or equivalent, capable of measuring flow rate to ± 2 percent of the selected flow rate. A flow rate range of 500 to 1000 ml/min is suggested.

6.2.6 Flexible Bag. Any leak-free plastic (*e.g.*, Tedlar, Mylar, Teflon) or plastic-coated aluminum (*e.g.*, aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and duration of the test run. A capacity in the range of 55 to 90 liters (1.9 to 3.2 ft^3) is suggested. To leak-check the bag, connect it to a water manometer, and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to

5 to 10 cm (2 to 4 in.) H_2O and allow to stand overnight. A deflated bag indicates a leak.

6.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 30 cm (12 in.), for the flexible bag leak-check.

6.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm (30 in.) Hg, for the sampling train leak-check.

6.3 Analysis. An Orsat or Fyrite type combustion gas analyzer.

7.0 Reagents and Standards

7.1 Reagents. As specified by the Orsat or Fyrite-type combustion analyzer manufacturer.

7.2 Standards. Two standard gas mixtures, traceable to National Institute of Standards and Technology (NIST) standards, to be used in auditing the accuracy of the analyzer and the analyzer operator technique:

7.2.1. Gas cylinder containing 2 to 4 percent O_2 and 14 to 18 percent CO_2.

7.2.2. Gas cylinder containing 2 to 4 percent CO_2 and about 15 percent O_2.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Single Point, Grab Sampling Procedure.

8.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.0 m (3.3 ft), unless otherwise specified by the Administrator.

8.1.2 Set up the equipment as shown in Figure 3–1, making sure all connections ahead of the analyzer are tight. If an Orsat analyzer is used, it is recommended that the analyzer be leak-checked by following the procedure in Section 11.5; however, the leak-check is optional.

8.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point. Purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer, and immediately analyze it for percent CO_2 and percent O_2 according to Section 11.2.

8.2 Single-Point, Integrated Sampling Procedure.

8.2.1 The sampling point in the duct shall be located as specified in Section 8.1.1.

8.2.2 Leak-check (optional) the flexible bag as in Section 6.2.6. Set up the equipment as shown in Figure 3–2. Just before sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point. Purge the sampling line. Next, connect the bag, and make sure that all connections are tight.

8.2.3 Sample Collection. Sample at a constant rate (±10 percent). The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 28 liters (1.0 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

8.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite type combustion gas analyzer according to Section 11.3.

Note: When using an Orsat analyzer, periodic Fyrite readings may be taken to verify/confirm the results obtained from the Orsat.

8.3 Multi-Point, Integrated Sampling Procedure.

8.3.1 Unless otherwise specified in an applicable regulation, or by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1.

8.3.2 Follow the procedures outlined in Sections 8.2.2 through 8.2.4, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3–3.

9.0 Quality Control

Section	Quality control measure	Effect
8.2	Use of Fyrite to confirm Orsat results	Ensures the accurate measurement of CO_2 and O_2.
10.1	Periodic audit of analyzer and operator technique.	Ensures that the analyzer is operating properly and that the operator performs the sampling procedure correctly and accurately.
11.3	Replicable analyses of integrated samples	Minimizes experimental error.

10.0 Calibration and Standardization

10.1 Analyzer. The analyzer and analyzer operator's technique should be audited periodically as follows: take a sample from a manifold containing a known mixture of CO_2 and O_2, and analyze according to the procedure in Section 11.3. Repeat this procedure until the measured concentration of three consecutive samples agrees with the stated value ± 0.5 percent. If necessary, take corrective action, as specified in the analyzer users manual.

10.2 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

11.0 Analytical Procedure

11.1 Maintenance. The Orsat or Fyrite-type analyzer should be maintained and operated according to the manufacturers specifications.

11.2 Grab Sample Analysis. Use either an Orsat analyzer or a Fyrite-type combustion gas analyzer to measure O_2 and CO_2 concentration for dry molecular weight determination, using procedures as specified in the analyzer user's manual. If an Orsat analyzer is used, it is recommended that the Orsat leak-check, described in Section 11.5, be performed before this determination; however, the check is optional. Calculate the dry molecular weight as indicated in Section 12.0. Repeat the sampling, analysis, and calculation procedures until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

11.3 Integrated Sample Analysis. Use either an Orsat analyzer or a Fyrite-type combustion gas analyzer to measure O_2 and CO_2 concentration for dry molecular weight determination, using procedures as specified in the analyzer user's manual. If an Orsat analyzer is used, it is recommended that the Orsat leak-check, described in Section 11.5, be performed before this determination; however, the check is optional. Calculate the dry molecular weight as indicated in Section 12.0. Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

11.4 Standardization. A periodic check of the reagents and of operator technique should be conducted at least once every three series of test runs as outlined in Section 10.1.

11.5 Leak-Check Procedure for Orsat Analyzer. Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is as follows:

11.5.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.

11.5.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.

11.5.3 Record the meniscus position.

11.5.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

11.5.5 For the Orsat analyzer to pass the leak-check, two conditions must be met:

11.5.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

11.5.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

11.5.6 If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease any leaking stopcocks. Replace leaking rubber connections. After the analyzer is reassembled, repeat the leak-check procedure.

12.0 Calculations and Data Analysis

12.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).
$\%CO_2$ = Percent CO_2 by volume, dry basis.
$\%O_2$ = Percent O_2 by volume, dry basis.
$\%CO$ = Percent CO by volume, dry basis.
$\%N_2$ = Percent N_2 by volume, dry basis.
0.280 = Molecular weight of N_2 or CO, divided by 100.
0.320 = Molecular weight of O_2 divided by 100.
0.440 = Molecular weight of CO_2 divided by 100.

12.2 Nitrogen, Carbon Monoxide Concentration. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent.

12.3 Dry Molecular Weight. Use Equation 3–1 to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO) \qquad \text{Eq. 3-1}$$

Note: The above Equation 3–1 does not consider the effect on calculated dry molecular weight of argon in the effluent gas. The concentration of argon, with a molecular weight of 39.9, in ambient air is about 0.9 percent. A negative error of approximately 0.4 percent is introduced. The tester may choose to include argon in the analysis using procedures subject to approval of the Administrator.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

1. Altshuller, A.P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. *6*:75–81. 1963.

2. Conner, William D. and J.S. Nader. Air Sampling with Plastic Bags. Journal of the American Industrial Hygiene Association. *25*:291–297. 1964.

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4. Mitchell, W.J. and M.R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association. *26*:491–495. May 1976.

5. Shigehara, R.T., R.M. Neulicht, and W.S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. *4*(2):21–26. August 1976.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

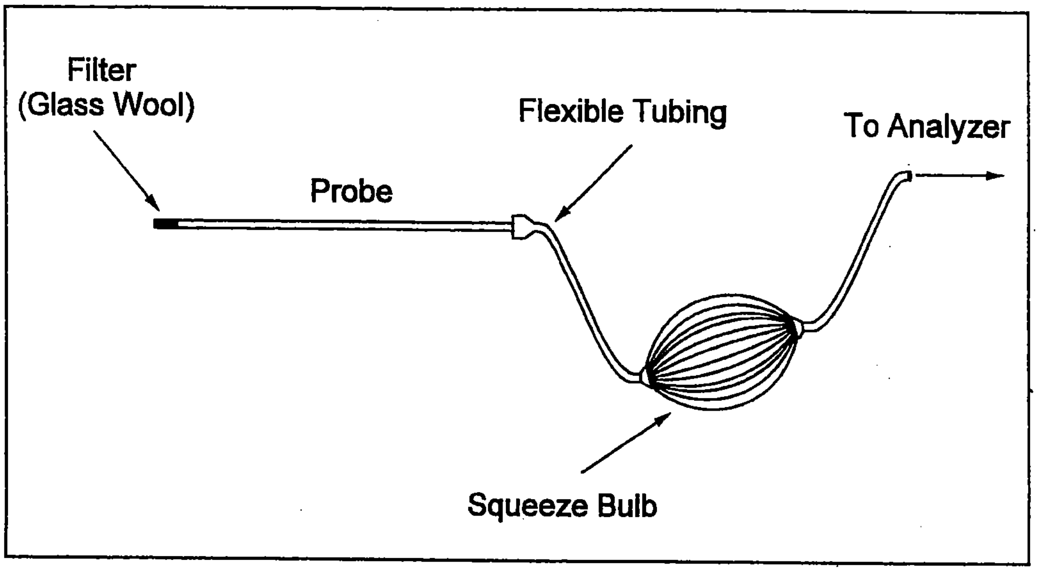

Figure 3-1. Grab-Sampling Train.

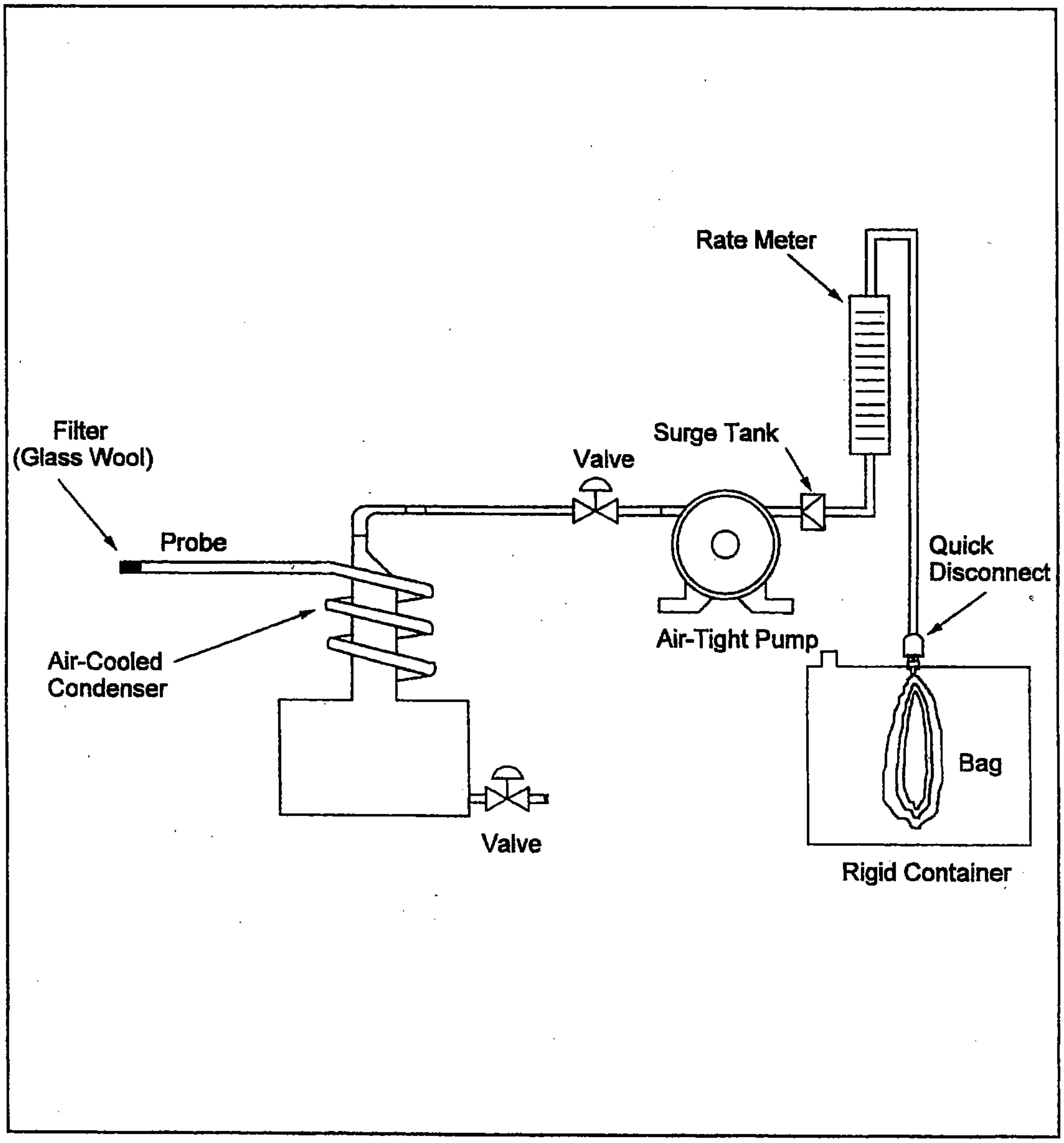

Figure 3-2. Integrated Gas-Sampling Train.

Time	Traverse point	Q (liter/min)	% Deviation[a]
Average			

[a] % Dev.=[(Q − Q_{avg})/Q_{avg}]×100 (Must be ≤±10%)

Figure 3–3. Sampling Rate Data

* * * * *

Method 3B—Gas Analysis for the Determination of Emission Rate Correction Factor or Excess Air

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1 and 3.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Oxygen (O_2)	7782–44–7	2,000 ppmv.
Carbon Dioxide (CO_2)	124–38–9	2,000 ppmv.
Carbon Monoxide (CO)	630–08–0	N/A.

1.2 Applicability. This method is applicable for the determination of O_2, CO_2, and CO concentrations in the effluent from fossil-fuel combustion processes for use in excess air or emission rate correction factor calculations. Where compounds other than CO_2, O_2, CO, and nitrogen (N_2) are present in concentrations sufficient to affect the results, the calculation procedures presented in this method must be modified, subject to the approval of the Administrator.

1.3 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) A multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point, and (2) a method using CO_2 or O_2 and stoichiometric calculations to determine excess air. These methods and modifications may be used, but are subject to the approval of the Administrator.

1.4 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas sample is extracted from a stack by one of the following methods: (1) Single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2, percent O_2, and, if necessary, percent CO using an Orsat combustion gas analyzer.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 Several compounds can interfere, to varying degrees, with the results of Orsat analyses. Compounds that interfere with CO2 concentration measurement include acid gases (*e.g.*, sulfur dioxide, hydrogen chloride); compounds that interfere with O_2 concentration measurement include unsaturated hydrocarbons (*e.g.*, acetone, acetylene), nitrous oxide, and ammonia. Ammonia reacts chemically with the O_2 absorbing solution, and when present in the effluent gas stream must be removed before analysis.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. A typical Orsat analyzer requires four reagents: a gas-confining solution, CO_2 absorbent, O_2 absorbent, and CO absorbent. These reagents may contain potassium hydroxide, sodium hydroxide, cuprous chloride, cuprous sulfate, alkaline pyrogallic acid, and/or chromous chloride. Follow manufacturer's operating instructions and observe all warning labels for reagent use.

6.0 Equipment and Supplies

Note: As an alternative to the sampling apparatus and systems described herein, other sampling systems (*e.g.*, liquid displacement) may be used, provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are, otherwise, capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

6.1 Grab Sampling and Integrated Sampling. Same as in Sections 6.1 and 6.2, respectively for Method 3.

6.2 Analysis. An Orsat analyzer only. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions. For Orsat maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

7.0 Reagents and Standards

7.1 Reagents. Same as in Method 3, Section 7.1.

7.2 Standards. Same as in Method 3, Section 7.2.

8.0 Sample Collection, Preservation, Storage, and Transport

Note: Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator. A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determinations, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight (see Method 3).

8.1 Single-Point, Grab Sampling and Analytical Procedure.

8.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.0 m (3.3 ft), unless otherwise specified by the Administrator.

8.1.2 Set up the equipment as shown in Figure 3–1 of Method 3, making sure all connections ahead of the analyzer are tight. Leak-check the Orsat analyzer according to the procedure described in Section 11.5 of Method 3. This leak-check is mandatory.

8.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer. For emission rate correction factor determinations, immediately analyze the sample for percent CO_2 or percent O_2, as outlined in Section 11.2. For excess air determination, immediately analyze the sample for percent CO_2, O_2, and CO, as outlined in Section 11.2, and calculate excess air as outlined in Section 12.2.

8.1.4 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 11.5 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak-test before and after the analysis.

8.2 Single-Point, Integrated Sampling and Analytical Procedure.

8.2.1 The sampling point in the duct shall be located as specified in Section 8.1.1.

8.2.2 Leak-check (mandatory) the flexible bag as in Section 6.2.6 of Method 3. Set up the equipment as shown in Figure 3–2 of Method 3. Just before sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag, and make sure that all connections are tight.

8.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 28 liters (1.0 ft^3) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

8.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Section 11.2).

8.3 Multi-Point, Integrated Sampling and Analytical Procedure.

8.3.1 Unless otherwise specified in an applicable regulation, or by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to Method 1.

8.3.2 Follow the procedures outlined in Sections 8.2.2 through 8.2.4, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3–3 of Method 3.

9.0 Quality Control

9.1 Data Validation Using Fuel Factor. Although in most instances, only CO_2 or O_2 measurement is required, it is recommended that both CO_2 and O_2 be measured to provide a check on the quality of the data. The data validation procedure of Section 12.3 is suggested.

Note: Since this method for validating the CO_2 and O_2 analyses is based on combustion of organic and fossil fuels and dilution of the gas stream with air, this method does not apply to sources that (1) remove CO_2 or O_2 through processes other than combustion, (2) add O_2 (*e.g.*, oxygen enrichment) and N_2 in proportions different from that of air, (3) add CO_2 (*e.g.*, cement or lime kilns), or (4) have no fuel factor, F_O, values obtainable (*e.g.*, extremely variable waste mixtures). This method validates the measured proportions of CO_2 and O_2 for fuel type, but the method does not detect sample dilution resulting from leaks during or after sample collection. The method is applicable for samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO_2 added or removed from the gas stream is not significant in relation to the total CO_2 concentration. The CO_2 concentrations from other types of scrubbers using only water or basic slurry can be significantly affected and would render the fuel factor check minimally useful.

10.0 Calibration and Standardization

10.1 Analyzer. The analyzer and analyzer operator technique should be audited periodically as follows: take a sample from a manifold containing a known mixture of CO_2 and O_2, and analyze according to the procedure in Section 11.3. Repeat this procedure until the measured concentration of three consecutive samples agrees with the stated value ±0.5 percent. If necessary, take corrective action, as specified in the analyzer users manual.

10.2 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

11.0 Analytical Procedure

11.1 Maintenance. The Orsat analyzer should be maintained according to the manufacturers specifications.

11.2 Grab Sample Analysis. To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.) Although in most cases, only CO_2 or O_2 concentration is required, it is recommended that both CO_2 and O_2 be measured, and that the procedure in Section 12.3 be used to validate the analytical data.

Note: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis.

11.3 Integrated Sample Analysis. The Orsat analyzer must be leak-checked (see Section 11.5 of Method 3) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 11.3.1 through 11.3.3) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air, as outlined in Section 12.2.

11.3.1 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, follow the procedure described in Section 11.2.

Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that the procedures in Section 12.3 be used to validate the analytical data.

11.3.2 Repeat the analysis until the following criteria are met:

11.3.2.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average three acceptable values of percent CO_2, and report the results to the nearest 0.2 percent.

11.3.2.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2, and report the results to the nearest 0.1 percent.

11.3.2.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO, and report the results to the nearest 0.1 percent.

11.3.3 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 11.5 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak-test before and after the analysis.

11.4 Standardization. A periodic check of the reagents and of operator technique should be conducted at least once every three series of test runs as indicated in Section 10.1.

12.0 Calculations and Data Analysis

12.1 Nomenclature. Same as Section 12.1 of Method 3 with the addition of the following:

%EA = Percent excess air.
0.264 = Ratio of O_2 to N_2 in air, v/v.

12.2 Percent Excess Air. Determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent CO, and percent O_2 from 100 percent. Calculate the percent excess air (if applicable) by substituting the appropriate values of percent O_2, CO, and N_2 into Equation 3B–1.

$$\%EA = \frac{\%O_2 - 0.5\ \%CO}{0.264\ \%N_2 - (\%O_2 - 0.5\ \%CO)} \times 100 \qquad \text{Eq. 3B-1}$$

Note: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternative methods, subject to approval of the Administrator, are required.

12.3 Data Validation When Both CO_2 and O_2 Are Measured.

12.3.1 Fuel Factor, F_o. Calculate the fuel factor (if applicable) using Equation 3B–2:

$$F_o = \frac{20.9 - \%O_2}{\%CO_2} \qquad \text{Eq. 3B-2}$$

Where:

$\%O_2$ = Percent O_2 by volume, dry basis.
$\%CO_2$ = Percent CO_2 by volume, dry basis.
20.9 = Percent O_2 by volume in ambient air.

If CO is present in quantities measurable by this method, adjust the O_2 and CO_2 values using Equations 3B–3 and 3B–4 before performing the calculation for F_o:

$$\%CO_2(\text{adj}) = \%CO_2 + \%CO \qquad \text{Eq. 3B-3}$$

$$\%O_2(\text{adj}) = \%O_2 - 0.5\ \%CO \qquad \text{Eq. 3B-4}$$

Where:

%CO = Percent CO by volume, dry basis.

12.3.2 Compare the calculated F_o factor with the expected F_o values. Table 3B–1 in Section 17.0 may be used in establishing acceptable ranges for the expected F_o if the fuel being burned is known. When fuels are burned in combinations, calculate the combined fuel F_d and F_c factors (as defined in Method 19, Section 12.2) according to the procedure in Method 19, Sections 12.2 and 12.3. Then calculate the F_o factor according to Equation 3B–5.

$$F_o = \frac{0.209\ F_d}{F_c} \qquad \text{Eq. 3B-5}$$

12.3.3 Calculated F_o values, beyond the acceptable ranges shown in this table, should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ±12 percent is appropriate for the F_o factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate; i.e., if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time consuming and costly.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

Same as Method 3, Section 16.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 3B–1.—F_o FACTORS FOR SELECTED FUELS

Fuel type	F_o range
Coal:	
Anthracite and lignite	1.016–1.130
Bituminous	1.083–1.230
Oil:	
Distillate	1.260–1.413
Residual	1.210–1.370
Gas:	
Natural	1.600–1.836
Propane	1.434–1.586
Butane	1.405–1.553
Wood	1.000–1.120
Wood bark	1.003–1.130

* * * * *

Method 4—Determination of Moisture Content in Stack Gases

Note: This method does not include all the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 5, and Method 6.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Water vapor (H_2O)	7732–18–5	N/A

1.2 Applicability. This method is applicable for the determination of the moisture content of stack gas.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

2.2 The method contains two possible procedures: a reference method and an approximation method.

2.2.1 The reference method is used for accurate determinations of moisture content (such as are needed to calculate emission data). The approximation method, provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content (*e.g.*, drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc.) are also acceptable.

2.2.2 The reference method is often conducted simultaneously with a pollutant emission measurement run. When it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent. These calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, to be capable of yielding results within one percent H_2O of the reference method.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 The moisture content of saturated gas streams or streams that contain water droplets, as measured by the reference method, may be positively biased. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to ±1 °C (2 °F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 8.1.1.1) during the reference method traverse, and calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) Using a psychrometric chart and making appropriate corrections if the stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternative methods, subject to the approval of the Administrator, shall be used.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Reference Method. A schematic of the sampling train used in this reference method is shown in Figure 4–1.

6.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and equipped with a filter, either in-stack (*e.g.*, a plug of glass wool inserted into the end of the probe) or heated out-of-stack (*e.g.*, as described in Method 5), to remove particulate matter. When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

6.1.2 Condenser. Same as Method 5, Section 6.1.1.8.

6.1.3 Cooling System. An ice bath container, crushed ice, and water (or equivalent), to aid in condensing moisture.

6.1.4 Metering System. Same as in Method 5, Section 6.1.1.9, except do not use sampling systems designed for flow rates higher than 0.0283 m^3/min (1.0 cfm). Other metering systems, capable of maintaining a constant sampling rate to within 10 percent and determining sample gas volume to within 2 percent, may be used, subject to the approval of the Administrator.

6.1.5 Barometer and Graduated Cylinder and/or Balance. Same as Method 5, Sections 6.1.2 and 6.2.5, respectively.

6.2. Approximation Method. A schematic of the sampling train used in this approximation method is shown in Figure 4–2.

6.2.1 Probe. Same as Section 6.1.1.

6.2.2 Condenser. Two midget impingers, each with 30-ml capacity, or equivalent.

6.2.3 Cooling System. Ice bath container, crushed ice, and water, to aid in condensing moisture in impingers.

6.2.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

6.2.5 Valve. Needle valve, to regulate the sample gas flow rate.

6.2.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

6.2.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to within 2 percent, and calibrated over the range of flow rates and conditions actually encountered during sampling.

6.2.8 Rate Meter. Rotameter, or equivalent, to measure the flow range from 0 to 3 liters/min (0 to 0.11 cfm).

6.2.9 Graduated Cylinder. 25-ml.

6.2.10 Barometer. Same as Method 5, Section 6.1.2.

6.2.11 Vacuum Gauge. At least 760-mm (30-in.) Hg gauge, to be used for the sampling leak check.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Reference Method. The following procedure is intended for a condenser system (such as the impinger system described in Section 6.1.1.8 of Method 5) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

8.1.1 Preliminary Determinations.

8.1.1.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.1.1.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m^3/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

8.1.2 Preparation of Sampling Train.

8.1.2.1 Place known volumes of water in the first two impingers; alternatively, transfer water into the first two impingers and record the weight of each impinger (plus water) to the nearest 0.5 g. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

8.1.2.2 Set up the sampling train as shown in Figure 4–1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of approximately 120 °C (248 °F), to prevent water condensation ahead of the condenser. Allow time for the temperatures to stabilize. Place crushed ice and water in the ice bath container.

8.1.3 Leak Check Procedures. It is recommended, but not required, that the volume metering system and sampling train be leak-checked as follows:

8.1.3.1 Metering System. Same as Method 5, Section 8.4.1.

8.1.3.2 Sampling Train. Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder), and pull a 380 mm (15 in.) Hg vacuum. A lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.020 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

8.1.4 Sampling Train Operation. During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on a data sheet similar to that shown in Figure 4–3. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point at least once during each time increment.

Note: When Method 4 is used concurrently with an isokinetic method (*e.g.*, Method 5) the sampling rate should be maintained at isokinetic conditions rather than 10 percent of constant rate.

8.1.4.1 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the silica gel outlet.

8.1.4.2 After collecting the sample, disconnect the probe from the first impinger (or from the filter holder), and conduct a leak check (mandatory) of the sampling train as described in Section 8.1.3.2. Record the leak rate. If the leakage rate exceeds the allowable rate, either reject the test results or correct the sample volume as in Section 12.3 of Method 5.

8.2 Approximation Method.

Note: The approximation method described below is presented only as a suggested method (see Section 2.0).

8.2.1 Place exactly 5 ml water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet. Then, plug the probe inlet and pull a vacuum of at least 250 mm (10 in.) Hg. Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0 to 40 ml/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable.

Note: Release the probe inlet plug slowly before turning off the pump.

8.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 liters/min (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft^3) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as indicated by Figure 4–4.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
Section 8.1.1.4	Leak rate of the sampling system cannot exceed four percent of the average sampling rate or 0.00057 m^3/min (0.20 cfm).	Ensures the accuracy of the volume of gas sampled. (Reference Method)
Section 8.2.1	Leak rate of the sampling system cannot exceed two percent of the average sampling rate.	Ensures the accuracy of the volume of gas sampled. (Approximation Method)

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Reference Method. Calibrate the metering system, temperature sensors, and barometer according to Method 5, Sections 10.3, 10.5, and 10.6, respectively.

10.2 Approximation Method. Calibrate the metering system and the barometer according to Method 6, Section 10.1 and Method 5, Section 10.6, respectively.

11.0 Analytical Procedure

11.1 Reference Method. Measure the volume of the moisture condensed in each of the impingers to the nearest ml. Alternatively, if the impingers were weighed prior to sampling, weigh the impingers after sampling and record the difference in weight to the nearest 0.5 g. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4–5), and calculate the moisture content, as described in Section 12.0.

11.2 Approximation Method. Combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.

12.0 Data Analysis and Calculations

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

12.1 Reference Method.

12.1.1 Nomenclature.

B_{ws} = Proportion of water vapor, by volume, in the gas stream.

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

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

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

R = Ideal gas constant, 0.06236 (mm Hg)(m^3)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft^3)/(lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_f = Final volume of condenser water, ml.

V_i = Initial volume, if any, of condenser water, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

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

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).

W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.

Y = Dry gas meter calibration factor.

ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

12.1.2 Volume of Water Vapor Condensed.

$$V_{wc(std)} = \frac{(V_f - V_i)\,\rho_w R\,T_{std}}{P_{std} M_W} = K_1 (V_f - V_i) \qquad \text{Eq. 4-1}$$

Where:

K_1 = 0.001333 m³/ml for metric units,
= 0.04706 ft³/ml for English units.

12.1.3 Volume of Water Collected in Silica Gel.

$$V_{wsg(std)} = \frac{(W_f - W_i)\,R\,T_{std}}{P_{std} M_W K_2} = K_3 (W_f - W_i) \qquad \text{Eq. 4-2}$$

Where:

K_2 = 1.0 g/g for metric units,
= 453.6 g/lb for English units.

K_3 = 0.001335 m³/g for metric units,
= 0.04715 ft³/g for English units.

12.1.4 Sample Gas Volume.

$$V_{m(std)} = \frac{V_m\,Y\,P_m T_{std}}{P_{std} T_m} = K_4 Y \frac{V_m P_m}{T_m} \qquad \text{Eq. 4-3}$$

Where:

K_4 = 0.3855 °K/mm Hg for metric units,
= 17.64 °R/in. Hg for English units.

Note: If the post-test leak rate (Section 8.1.4.2) exceeds the allowable rate, correct the value of Vm in Equation 4–3, as described in Section 12.3 of Method 5.

12.1.5 Moisture Content.

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} \qquad \text{Eq. 4-4}$$

12.1.6 Verification of Constant Sampling Rate. For each time increment, determine the ΔV_m. Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results, and repeat the run.

12.1.7 In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 4.1), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

12.2 Approximation Method. The approximation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

12.2.1 Nomenclature.

B_{wm} = Approximate proportion by volume of water vapor in the gas stream leaving the second impinger, 0.025.

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

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

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

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

R = Ideal gas constant, 0.06236 [(mm Hg)(m³)]/[(g-mole)(K)] for metric units and 21.85 [(in. Hg)(ft³)]/[(lb-mole)(°R)] for English units.

T_m = Absolute temperature at meter, °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

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

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

Y = Dry gas meter calibration factor.

ρ_w = Density of water, 0.09982 g/ml (0.002201 lb/ml).

12.2.2 Volume of Water Vapor Collected.

$$V_{wc(std)} = \frac{(V_f - V_i)\,\rho_w R\,T_{std}}{P_{std} M_W} = K_5 (V_f - V_i) \qquad \text{Eq. 4-5}$$

Where:

K_5 = 0.001333 m³/ml for metric units,
= 0.04706 ft³/ml for English units.

12.2.3 Sample Gas Volume.

$$V_{m(std)} = \frac{V_m\,Y\,P_m T_{std}}{P_{std} T_m} = K_6 Y \frac{V_m P_m}{T_m} \qquad \text{Eq. 4-6}$$

Where:

K_6 = 0.3855 °K/mm Hg for metric units,
= 17.64 °R/in. Hg for English units.

12.2.4 Approximate Moisture Content.

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025) \qquad \text{Eq. 4-7}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

17.0 References

1. Air Pollution Engineering Manual (Second Edition). Danielson, J.A. (ed.). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. Publication No. AP–40. 1973.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November 1963.

3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, CA. Bulletin WP–50. 1968.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

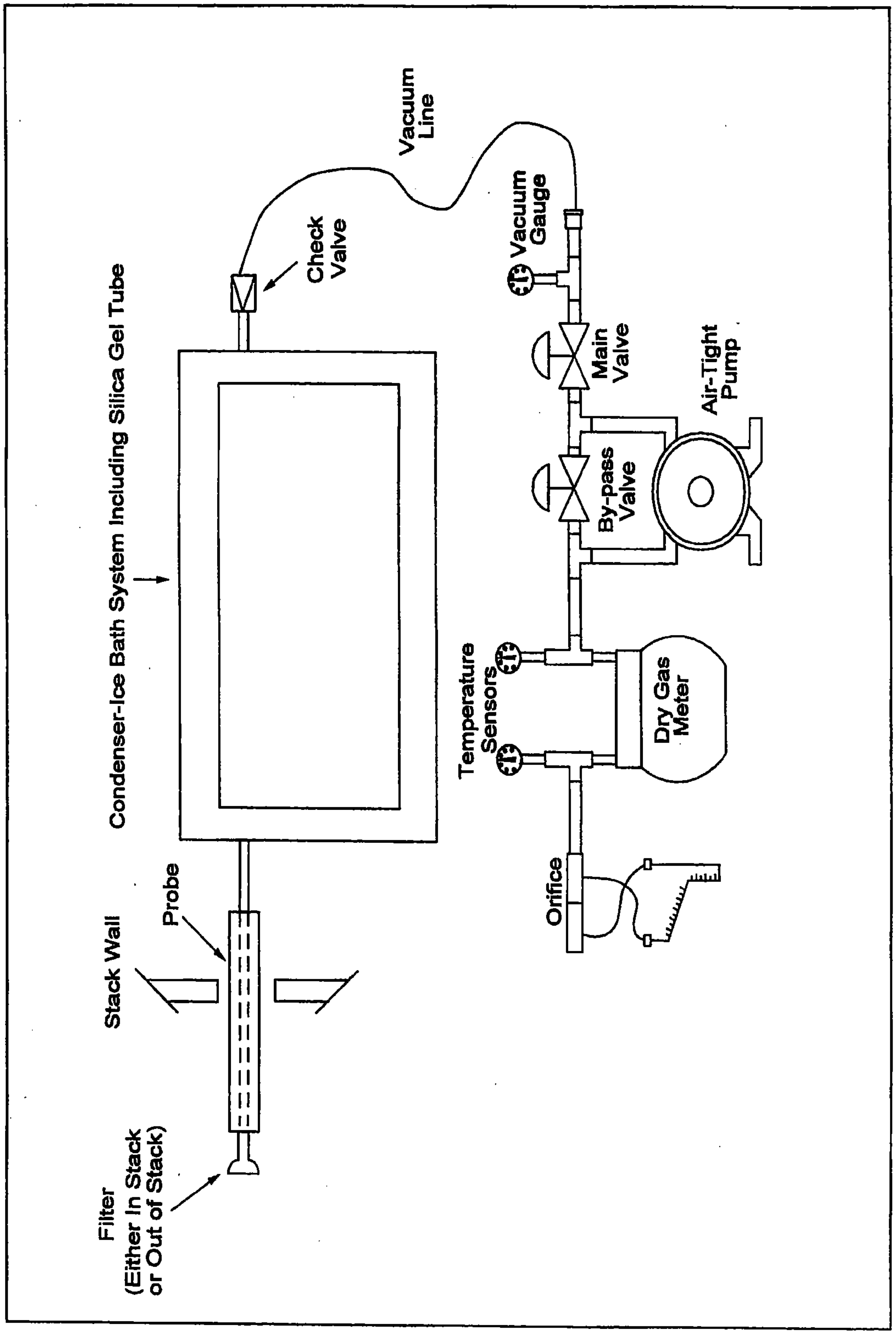

Figure 4-1. Moisture Sampling Train-Reference Method

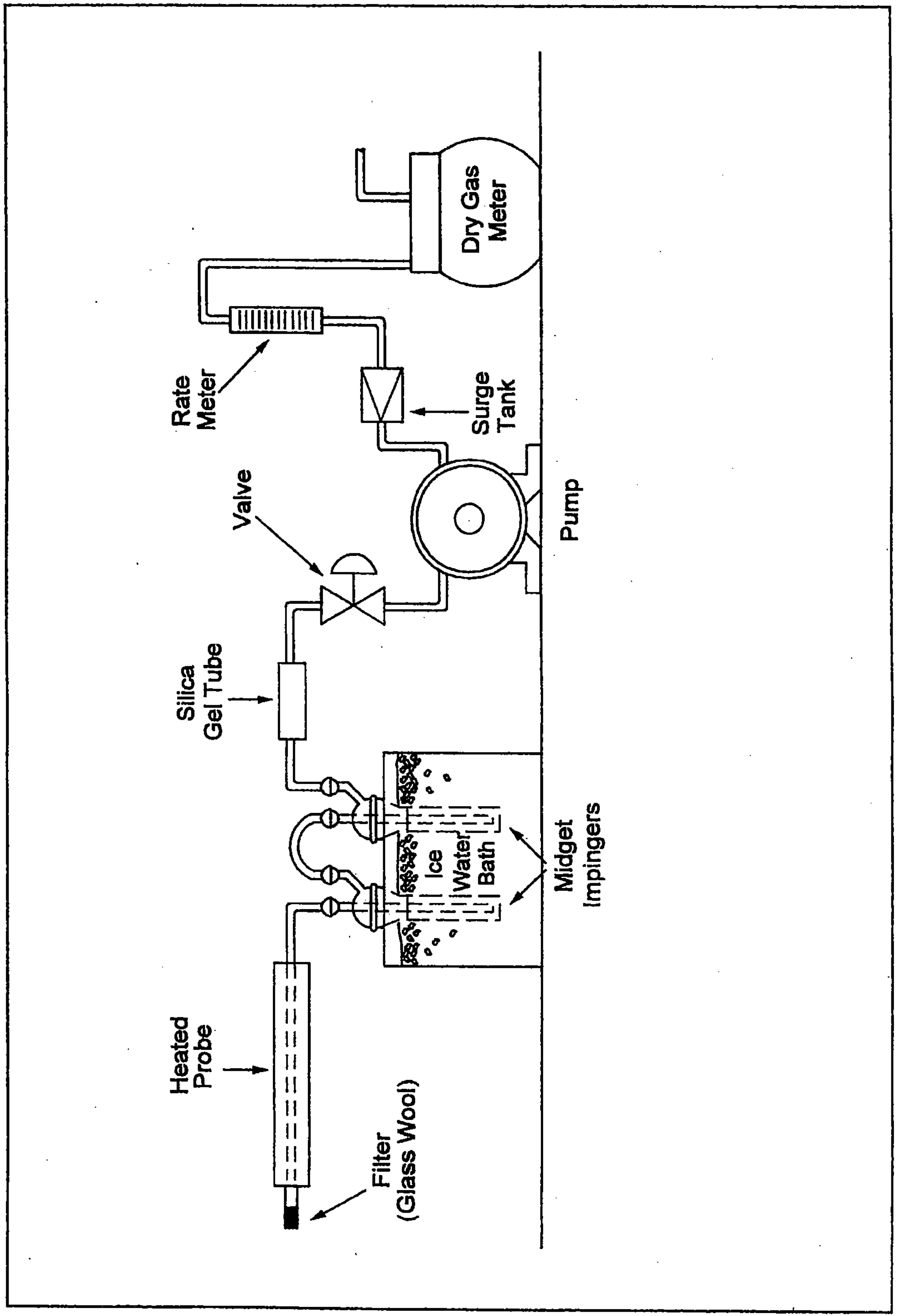

Figure 4-2. Moisture Sampling Train - Approximation Method.

Plant
Location
Operator
Date
Run No.
Ambient temperature
Barometric pressure
Probe Length

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Sampling time (Δ), min	Stack temperature °C (°F)	Pressure differential across orifice meter ΔH mm (in.) H_2O	Meter reading gas sample volume m^3 (ft^3)	ΔV_m m^3 (ft^3)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger °C (°F)
						Inlet Tm_{in} °C (°F)	Outlet Tm_{out} °C (°F)	
Average								

Location
Test
Date
Operator
Barometric pressure
Comments:

Figure 4–3. Moisture Determination—Reference Method

Clock time	Gas Volume through meter, (V_m), m^3 (ft^3)	Rate meter setting m^3/min (ft^3/min)	Meter temperature °C (°F)

Figure 4–4. Example Moisture Determination Field Data Sheet—Approximation Method

	Impinger volume, ml	Silica gel weight, g
Final Initial Difference		

Figure 4–5. Analytical Data—Reference Method

Method 5—Determination of Particulate Matter Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from stationary sources.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of 120 ± 14°C (248 ± 25°F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The PM mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5–1 in Section 18.0. Complete construction details are given in APTD–0581 (Reference 2 in Section 17.0); commercial models of this train are also available. For changes from APTD–0581 and for allowable modifications of the train shown in Figure 5–1, see the following subsections.

Note: The operating and maintenance procedures for the sampling train are described in APTD–0576 (Reference 3 in Section 17.0). Since correct usage is important in obtaining valid results, all users should read APTD–0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

6.1.1.1 Probe Nozzle. Stainless steel (316) or glass with a sharp, tapered leading edge. The angle of taper shall be ≤30°, and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the Administrator. A range of nozzle sizes suitable for isokinetic sampling should be available. Typical nozzle sizes range from 0.32 to 1.27 cm (⅛ to ½ in) inside diameter (ID) in increments of 0.16 cm (1/16 in). Larger nozzles sizes are also available if higher volume sampling trains are used. Each nozzle shall be calibrated, according to the procedures outlined in Section 10.1.

6.1.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a probe gas temperature during sampling of 120 ± 14 °C (248 ± 25 °F), or such other temperature as specified by an applicable subpart of the standards or as approved by the Administrator for a particular application. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD–0581 and utilizing the calibration curves of APTD–0576 (or calibrated according to the procedure outlined in APTD–0576) will be considered acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 °C (900 °F); quartz glass liners shall be used for temperatures between 480 and 900 °C (900 and 1,650 °F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate glass is 820 °C (1500°F), and for quartz glass it is 1500 °C (2700 °F). Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825 or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

6.1.1.3 Pitot Tube. Type S, as described in Section 6.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5–1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2–7) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 10.0 of Method 2.

6.1.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 6.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

6.1.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (*e.g.*, stainless steel, Teflon, or Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

6.1.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder of 120 ±14 °C (248 ±25 °F) during sampling, or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application.

6.1.1.7 Temperature Sensor. A temperature sensor capable of measuring temperature to within ±3 °C (5.4 °F) shall be installed so that the sensing tip of the temperature sensor is in direct contact with the sample gas, and the temperature around the filter holder can be regulated and monitored during sampling.

6.1.1.8 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 8.3.1), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A temperature sensor, capable of measuring temperature to within 1 °C (2 °F) shall be placed at the outlet of the fourth impinger for monitoring purposes. Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. An acceptable technique involves the measurement of condensed water either gravimetrically or volumetrically and the determination of the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20 °C (68 °F) and determining the weight gain. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

Note: If a determination of the PM collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

6.1.1.9 Metering System. Vacuum gauge, leak-free pump, temperature sensors capable of measuring temperature to within 3 °C (5.4 °F), dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5–1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall allow periodic checks of isokinetic rates.

6.1.1.10 Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD–0581 or APTD–0576 may be used provided that the specifications of this method are met.

6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in.).

Note: The barometric pressure reading may be obtained from a nearby National Weather Service station. In this case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be made at a rate of minus 2.5 mm Hg (0.1 in.) per 30 m (100 ft) elevation increase or plus 2.5 mm Hg (0.1 in) per 30 m (100 ft) elevation decrease.

6.1.3 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 6.3 and 6.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2–4). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature sensor need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

6.2 Sample Recovery. The following items are required for sample recovery:

6.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) constructed of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

6.2.2 Wash Bottles. Two Glass wash bottles are recommended. Alternatively, polyethylene wash bottles may be used. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

6.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

6.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

6.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml.

6.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

6.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

6.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

6.3 Sample Analysis. The following equipment is required for sample analysis:

6.3.1 Glass Weighing Dishes.

6.3.2 Desiccator.

6.3.3 Analytical Balance. To measure to within 0.1 mg.

6.3.4 Balance. To measure to within 0.5 g.

6.3.5 Beakers. 250 ml.

6.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

6.3.7 Temperature Sensor. To measure the temperature of the laboratory environment.

7.0 Reagents and Standards

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Method D 2986–71, 78, or 95a (incorporated by reference—see § 60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Reference 10 in Section 17.0 may be used to select the appropriate filter.

7.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

7.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water (to conform to ASTM D 1193–77 or 91 Type 3 (incorporated by reference—see § 60.17)) shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

7.1.4 Crushed Ice.

7.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

7.2 Sample Recovery. Acetone, reagent grade, ≤0.001 percent residue, in glass bottles, is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

7.3 Sample Analysis. The following reagents are required for sample analysis:

7.3.1 Acetone. Same as in Section 7.2.

7.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation. It is suggested that sampling equipment be maintained according to the procedures described in APTD–0576.

8.1.1 Place 200 to 300 g of silica gel in each of several air-tight containers. Weigh each container, including silica gel, to the nearest 0.5 g, and record this weight. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

8.1.2 Check filters visually against light for irregularities, flaws, or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or polyethylene petri dishes), and keep each filter in its identified container at all times except during sampling.

8.1.3 Desiccate the filters at 20 ± 5.6 °C (68 ± 10 °F) and ambient pressure for at least 24 hours. Weigh each filter (or filter and shipping container) at intervals of at least 6 hours to a constant weight (*i.e.*, ≤0.5 mg change from previous weighing). Record results to the nearest 0.1 mg. During each weighing, the period for which the filter is exposed to the laboratory atmosphere shall be less than 2 minutes. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

8.2 Preliminary Determinations.

8.2.1 Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak check of the pitot lines (see Method 2, Section 8.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 8.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

8.2.2 Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 8.3 of Method 2).

8.2.3 Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the required probe length.

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

8.2.5 The sampling time at each point shall be the same. It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

8.2.6 In some circumstances (*e.g.*, batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

8.3 Preparation of Sampling Train.

8.3.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin. Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

8.3.2 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

8.3.3 When glass probe liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F) or a heat-resistant string gasket when temperatures are higher. See APTD–0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as discussed above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.3.4 Set up the train as shown in Figure 5–1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD–0576) to avoid the possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

8.3.5 Place crushed ice around the impingers.

8.4 Leak-Check Procedures.

8.4.1 Leak Check of Metering System Shown in Figure 5–1. That portion of the sampling train from the pump to the orifice meter should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5–2): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing, and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

8.4.2 Pretest Leak Check. A pretest leak check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the following procedure should be used.

8.4.2.1 After the sampling train has been assembled, turn on and set the filter and probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

8.4.2.2 If a heat-resistant string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm (15 in.) Hg vacuum (see **Note** in Section 8.4.2.1). Then connect the probe to the train, and leak-check at approximately 25 mm (1 in.) Hg vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm (15 in.) Hg vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.020 cfm), whichever is less, are unacceptable.

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

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

8.4.3 Leak Checks During Sample Run. If, during the sampling run, a component (*e.g.*, filter assembly or impinger) change becomes necessary, a leak check shall be conducted immediately before the change is made. The leak check shall be done according to the procedure outlined in Section 8.4.2 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, either record the leakage rate and plan to correct the sample volume as shown in Section 12.3 of this method, or void the sample run.

Note: Immediately after component changes, leak checks are optional. If such leak checks are done, the procedure outlined in Section 8.4.2 above should be used.

8.4.4 Post-Test Leak Check. A leak check of the sampling train is mandatory at the conclusion of each sampling run. The leak check shall be performed in accordance with the procedures outlined in Section 8.4.2, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3 min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, either record the leakage rate and correct the sample volume as shown in Section 12.3 of this method, or void the sampling run.

8.5 Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120 ± 14 °C (248 ± 25 °F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

8.5.1 For each run, record the data required on a data sheet such as the one shown in Figure 5–3. Be sure to record the initial DGM reading. Record the DGM readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings indicated by Figure 5–3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

8.5.2 Clean the portholes prior to the test run to minimize the chance of collecting deposited material. To begin sampling, verify that the filter and probe heating systems are up to temperature, remove the nozzle cap, verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump, and adjust the flow to isokinetic conditions. Nomographs are available which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 ± 0.02, and the stack gas equivalent density [dry molecular weight (M_d)] is equal to 29 ± 4. APTD–0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Reference 7 in Section 17.0) are taken to compensate for the deviations.

8.5.3 When the stack is under significant negative pressure (*i.e.*, height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

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

8.5.5 Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

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

8.5.7 If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of the sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check (see Section 8.4.3). The total PM weight shall include the summation of the filter assembly catches.

8.5.8 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note: When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

8.5.9 At the end of the sample run, close the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final DGM meter reading, and conduct a post-test leak check, as outlined in Section 8.4.4. Also, leak-check the pitot lines as described in Method 2, Section 8.1. The lines must pass this leak check, in order to validate the velocity head data.

8.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 12.11) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Administrator for possible variance on the isokinetic rates.

8.7 Sample Recovery.

8.7.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

8.7.2 When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle, and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the filter holder, thereby drawing water from the impingers into the filter holder.

8.7.3 Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened, and cap it. Remove the umbilical cord from the last impinger, and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder, and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

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

8.7.5 Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used, and place it in a glass sample container labeled "acetone blank."

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

8.7.6.1 Container No. 1. Carefully remove the filter from the filter holder, and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the PM cake is inside the fold. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Seal the container.

8.7.6.2 Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Deionized distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator. In these cases, save a water blank, and follow the Administrator's directions on analysis. Perform the acetone rinse as follows:

8.7.6.2.1 Carefully remove the probe nozzle. Clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

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

8.7.6.2.3 Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible PM is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe.

8.7.6.2.4 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

8.7.6.2.5 After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to allow determination of whether leakage occurred during transport. Label the container to identify clearly its contents.

8.7.6.3 Container No. 3. Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container, and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in Section 11.2.3.

8.7.6.4 Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first three impingers to within 1 ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see NOTE, Section 6.1.1.8). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

8.8 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.1–10.6	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 Volume Metering System Checks. The following procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional.

9.2.1 Meter Orifice Check. Using the calibration data obtained during the calibration procedure described in Section 10.3, determine the ΔH@ for the metering system orifice. The ΔH@ is the orifice pressure differential in units of in. H_2O that correlates to 0.75 cfm of air at 528 °R and 29.92 in. Hg. The ΔH@ is calculated as follows:

$$\Delta H@ = 0.0319\ \Delta H \frac{T_M \theta^2}{P_{bar} Y^2 V_m^2}$$

Where:

ΔH = Average pressure differential across the orifice meter, in. H_2O.

T_m = Absolute average DGM temperature, °R.

P_{bar} = Barometric pressure, in. Hg.

θ = Total sampling time, min.

Y = DGM calibration factor, dimensionless.

V_m = Volume of gas sample as measured by DGM, dcf.

$0.0319 = (0.0567 \text{ in. Hg/°R})(0.75 \text{ cfm})^2$

9.2.1.1 Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (*i.e.*, pump, volume meter, and orifice) at the ΔH@ pressure differential for 10 minutes. Record the volume collected, the DGM temperature, and the barometric pressure. Calculate a DGM calibration check value, Y_c, as follows:

$$Y_c = \frac{10}{V_m}\left[\frac{0.0319\ T_m}{P_{bar}}\right]^{\frac{1}{2}}$$

where:

Y_c = DGM calibration check value, dimensionless.

10 = Run time, min.

9.2.1.2 Compare the Y_c value with the dry gas meter calibration factor Y to determine that: $0.97Y < Y_c < 1.03Y$. If the Y_c value is not within this range, the volume metering system should be investigated before beginning the test.

9.2.2 Calibrated Critical Orifice. A critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box, may be used as a check by following the procedure of Section 16.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory log of all calibrations.

10.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

10.2 Pitot Tube Assembly. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 10.1 of Method 2.

10.3 Metering System.

10.3.1 Calibration Prior to Use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5–4. The wet test meter should have a capacity of 30 liters/rev (1 ft^3/rev). A spirometer of 400 liters (14 ft^3) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the DGM. Also note the barometric pressure and the temperatures of the wet test meter, the inlet of the DGM, and the outlet of the DGM. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.14 m^3 (5 ft^3) at all orifice settings. Record all the data on a form similar to Figure 5–5 and calculate Y, the DGM calibration factor, and $\Delta H_{@}$, the orifice calibration factor, at each orifice setting as shown on Figure 5–5. Allowable tolerances for individual Y and $\Delta H_{@}$ values are given in Figure 5–5. Use the average of the Y values in the calculations in Section 12.0.

10.3.1.1 Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m^3/min (0.020 cfm). At the end of the run, take the difference of the measured wet test meter and DGM volumes. Divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.020 cfm).

10.3.2 Calibration After Use. After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the DGM calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as detailed in Section 10.3.1.

Note: Alternative procedures (*e.g.*, rechecking the orifice meter coefficient) may be used, subject to the approval of the Administrator.

10.3.3 Acceptable Variation in Calibration. If the DGM coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (*i.e.*, before or after) gives the lower value of total sample volume.

10.4 Probe Heater Calibration. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD–0576 can also be used. Probes constructed according to APTD–0581 need not be calibrated if the calibration curves in APTD–0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

Note: The probe heating system shall be calibrated before its initial use in the field.

10.5 Temperature Sensors. Use the procedure in Section 10.3 of Method 2 to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

10.6 Barometer. Calibrate against a mercury barometer.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5–6.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose PM from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight, and report the results to the nearest 0.1 mg. For the purposes of this section, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings. Alternatively, the sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours. Once the sample has cooled, weigh the sample, and use this weight as a final weight.

11.2.2 Container No. 2. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

11.2.3 Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

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

Note: The contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used, provided that they give equivalent results.

12.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_a = Acetone blank residue concentration, mg/mg.
c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (gr/dscf).
I = Percent of isokinetic sampling.
L_1 = Individual leakage rate observed during the leak-check conducted prior to the first component change, m^3/min (ft^3/min)
L_a = Maximum acceptable leakage rate for either a pretest leak-check or for a leak-check following a component change; equal to 0.00057 m^3/min (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.
L_i = Individual leakage rate observed during the leak-check conducted prior to the "i^{th}" component change ($i = 1, 2, 3 \ldots n$), m^3/min (cfm).
L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).
m_a = Mass of residue of acetone after evaporation, mg.
m_n = Total amount of particulate matter collected, mg.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
P_s = Absolute stack gas pressure, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 ((mm Hg)(m^3))/((K)(g-mole)) {21.85 ((in. Hg) (ft^3))/((°R) (lb-mole))}.

T_m = Absolute average DGM temperature (see Figure 5–3), K (°R).
T_s = Absolute average stack gas temperature (see Figure 5–3), K (°R).
T_{std} = Standard absolute temperature, 293 K (528 °R).
V_a = Volume of acetone blank, ml.
V_{aw} = Volume of acetone used in wash, ml.
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5–6), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
V_s = Stack gas velocity, calculated by Method 2, Equation 2–7, using data obtained from Method 5, m/sec (ft/sec).
W_a = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration factor.
ΔH = Average pressure differential across the orifice meter (see Figure 5–4), mm H_2O (in. H_2O).
ρ_a = Density of acetone, mg/ml (see label on bottle).
ρ_w = Density of water, 0.9982 g/ml.(0.002201 lb/ml).
θ = Total sampling time, min.
θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.
θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5–3).

12.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using Equation 5–1.

$$V_{m(std)} = V_m Y \frac{T_{std}\left(P_{bar} + \frac{\Delta H}{13.6}\right)}{T_m P_{std}} \qquad \text{Eq. 5-1}$$

$$= K_1 V_m Y \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{T_m}$$

Where:

K_1 = 0.3858 °K/mm Hg for metric units, = 17.64 °R/in. Hg for English units.

Note: Equation 5–1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (*i.e.*, the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 5–1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5–1 with the expression:

$$\left(V_m - \left(L_p - L_a\right)\theta\right)$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5–1 by the expression:

$$\left[V_m - \left(L_1 - L_a\right)\theta_1 - \sum_{i=2}^{n}\left(L_i - L_a\right)\theta_i - \left(L_p - L_a\right)\theta_p\right]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

12.4 Volume of Water Vapor Condensed.

$$V_{w(std)} = V_{lc}\frac{\rho_w R\, T_{std}}{M_w P_{std}} \qquad \text{Eq. 5-2}$$

$$= K_2 V_{lc}$$

Where:

K_2 = 0.001333 m³/ml for metric units,
= 0.04706 ft³/ml for English units.

12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Eq. 5-3}$$

Note: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5–3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon the assumption of saturated conditions is given in Section 4.0 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5–3 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ± 1°C (2°F).

12.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a} \qquad \text{Eq. 5-4}$$

12.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a \qquad \text{Eq. 5-5}$$

12.8 Total Particulate Weight. Determine the total particulate matter catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 5–6).

Note: In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight. Refer to Section 8.5.8 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

12.9 Particulate Concentration.

$$C_s = \frac{K_3 m_n}{V_{m(std)}} \qquad \text{Eq. 5-6}$$

Where:

K_3 = 0.001 g/mg for metric units.

= 0.0154 gr/mg for English units.

12.10 Conversion Factors:

From	To	Multiply by
ft³	m³	0.02832
gr	mg	64.80004
gr/ft³	mg/m³	2288.4
mg	g	0.001
gr	lb	1.429×10^{-4}

12.11 Isokinetic Variation.

12.11.1 Calculation from Raw Data.

$$I = \frac{100\,T_s\left[K_4 V_{1c} + \frac{(V_m Y)}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{60\,\theta\, v_s P_s A_n} \qquad \text{Eq. 5-7}$$

Where:

K_4 = 0.003454 ((mm Hg)(m³))/((ml)(°K)) for metric units,

= 0.002669 ((in. Hg)(ft³))/((ml)(°R)) for English units.

12.11.2 Calculation from Intermediate Values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60\left(1 - B_{ws}\right)} \qquad \text{Eq. 5-8}$$

$$= K_5 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta\left(1 - B_{ws}\right)}$$

Where:

K_5 = 4.320 for metric units,

= 0.09450 for English units.

12.11.3 Acceptable Results. If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the PM results are low in comparison to the standard, and "I" is over 110 percent or less than 90 percent, the Administrator may opt to accept the results. Reference 4 in Section 17.0 may be used to make acceptability judgments. If "I" is judged to be unacceptable, reject the results, and repeat the sampling run.

12.12 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in Sections 12.3 and 12.4 of Method 2.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Dry Gas Meter as a Calibration Standard. A DGM may be used as a calibration standard for volume measurements in place of the wet test meter specified in Section 10.3, provided that it is calibrated initially and recalibrated periodically as follows:

16.1.1 Standard Dry Gas Meter Calibration.

16.1.1.1. The DGM to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity (*e.g.*, 3 liters/rev (0.1 ft³/rev)). A spirometer (400 liters (14 ft³) or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft³/rev) and capable of measuring volume to within 1.0 percent. Wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained.

16.1.1.2 Set up the components as shown in Figure 5–7. A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the DGM should be minimized (no greater than 100 mm H_2O (4 in. H_2O) at a flow rate of 30 liters/min (1 cfm)). This can be accomplished by using large diameter tubing connections and straight pipe fittings.

16.1.1.3 Collect the data as shown in the example data sheet (see Figure 5–8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

16.1.1.4 Calculate flow rate, Q, for each run using the wet test meter volume, V_W, and the run time, θ. Calculate the DGM coefficient, Y_{ds}, for each run. These calculations are as follows:

$$Q = K_1 \frac{P_{bar} V_w}{\left(T_w + T_{std}\right)\theta} \qquad \text{Eq. 5-9}$$

$$Y_{ds} = \frac{V_w\left(T_{ds} + T_{std}\right)P_{bar}}{V_{ds}\left(T_w + T_{std}\right)\left(P_{bar} + \frac{\Delta p}{13.6}\right)} \qquad \text{Eq. 5-10}$$

Where:

K_1 = 0.3858 °C/mm Hg for metric units=17.64 °F/in. Hg for English units.

V_W = Wet test meter volume, liter (ft³).

V_{ds} = Dry gas meter volume, liter (ft³).

T_{ds} = Average dry gas meter temperature, °C (°F).

T_{adj} = 273 °C for metric units = 460 °F for English units.

T_W = Average wet test meter temperature, °C (°F)

P_{bar} = Barometric pressure, mm Hg (in. Hg).
Δp = Dry gas meter inlet differential pressure, mm H_2O (in. H_2O).
θ = Run time, min.

16.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Y_{ds} values at each flow rate resulting in no less than five average meter coefficients, Y_{ds}.

16.1.1.6 Prepare a curve of meter coefficient, Y_{ds}, versus flow rate, Q, for the DGM. This curve shall be used as a reference when the meter is used to calibrate other DGMs and to determine whether recalibration is required.

16.1.2 Standard Dry Gas Meter Recalibration.

16.1.2.1 Recalibrate the standard DGM against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard DGM is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

16.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 30 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within 1.5 percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

16.2 Critical Orifices As Calibration Standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in Section 16.1, provided that they are selected, calibrated, and used as follows:

16.2.1 Selection of Critical Orifices.

16.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices (*i.e.*, a critical vacuum can be obtained, as described in Section 16.2.2.2.3). Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min (0.35 and 1.2 cfm) or the expected operating range. Two of the critical orifices should bracket the expected operating range. A minimum of three critical orifices will be needed to calibrate a Method 5 DGM; the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown in Table 5–1 in Section 18.0 give the approximate flow rates.

16.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13 by 20 mm sleeve type, into a ½-inch Swagelok (or equivalent) quick connect. Insert the needle into the stopper as shown in Figure 5–9.

16.2.2 Critical Orifice Calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in Section 6.1.1.9 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

16.2.2.1 Calibration of Meter Box. The critical orifices must be calibrated in the same configuration as they will be used (*i.e.*, there should be no connections to the inlet of the orifice).

16.2.2.1.1 Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero (*i.e.*, no detectable movement of the DGM dial shall be seen for 1 minute).

16.2.2.1.2 Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See Section 8.4.1 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

16.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in Section 10.3. Make sure that the wet test meter meets the requirements stated in Section 16.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

16.2.2.2 Calibration of Critical Orifices. Set up the apparatus as shown in Figure 5–10.

16.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

16.2.2.2.2 Leak check the system as in Section 16.2.2.1.1. The leakage rate shall be zero.

16.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, ΔH. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice. Orifices that do not reach a critical value shall not be used.

16.2.2.2.4 Obtain the barometric pressure using a barometer as described in Section 6.1.2. Record the barometric pressure, P_{bar}, in mm Hg (in. Hg).

16.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve ± 0.5 percent in K′ (see Eq. 5–11). Record the information listed in Figure 5–11.

16.2.2.2.6 Calculate K′ using Equation 5–11.

$$K^1 = \frac{K_1 V_m Y \left(P_{bar} + \frac{\Delta H}{13.6}\right) T_{amb}^{1/2}}{P_{bar} T_m \theta} \qquad \text{Eq. 5-11}$$

Where:

K′ = Critical orifice coefficient, $[m^3)(°K)^{1/2}]/$

[(mm Hg)(min)] {[(ft^3)(°R)$^{1/2}$)] [(in. Hg)(min)].

T_{amb} = Absolute ambient temperature, °K (°R).

Calculate the arithmetic mean of the K′ values. The individual K′ values should not differ by more than ±0.5 percent from the mean value.

16.2.3 Using the Critical Orifices as Calibration Standards.

16.2.3.1 Record the barometric pressure.

16.2.3.2 Calibrate the metering system according to the procedure outlined in Section 16.2.2. Record the information listed in Figure 5–12.

16.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y, using the equations below:

$$V_{m(std)} = \frac{K_1 V_m \left[P_{bar} + \left(\frac{\Delta H}{13.6}\right)\right]}{T_m} \qquad \text{Eq. 5-12}$$

$$V_{cr(std)} = K_1 \frac{P_{bar}\Theta}{\sqrt{T_{amb}}} \qquad \text{Eq. 5-13}$$

$$Y = \frac{V_{cr\,(std)}}{V_{m(std)}} \qquad \text{Eq. 5-14}$$

Where:

$V_{cr(std)}$ = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dscm (dscf).

K_1 = 0.3858 K/mm Hg for metric units

= 17.64 °R/in. Hg for English units.

16.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration factor, Y, at each of the flow rates should not differ by more than ± 2 percent from the average.

16.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking orifice 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to Section 16.2.2.

17.0 References.

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. December 6, 1967.

2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, NC. APTD–0581. April 1971.

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9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, PA. 1974. pp. 617–622.

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11. Westlin, P.R. and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter. *3*(1):17–30. February 1978.

12. Lodge, J.P., Jr., J.B. Pate, B.E. Ammons, and G.A. Swanson. The Use of Hypodermic Needles as Critical Orifices in Air Sampling. J. Air Pollution Control Association. *16*:197–200. 1966.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

Gauge/cm	Flow rate liters/min.	Gauge/cm	Flow rate liters/min.
12/7.6	32.56	14/2.5	19.54
12/10.2	30.02	14/5.1	17.27
13/2.5	25.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.61
13/10.2	20.67	15/10.2	10.48

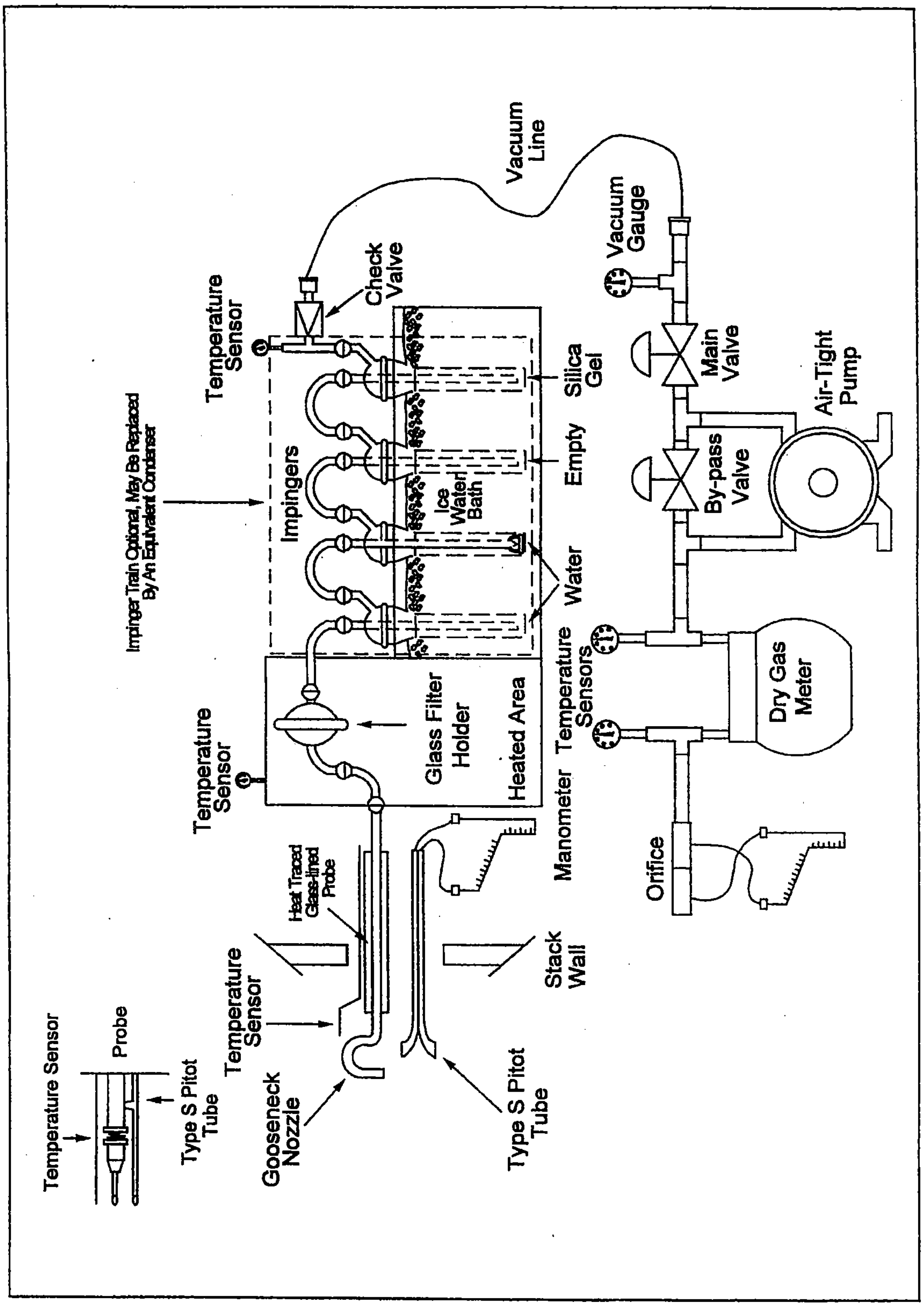


Figure 5-1. Particulate Sampling Train.

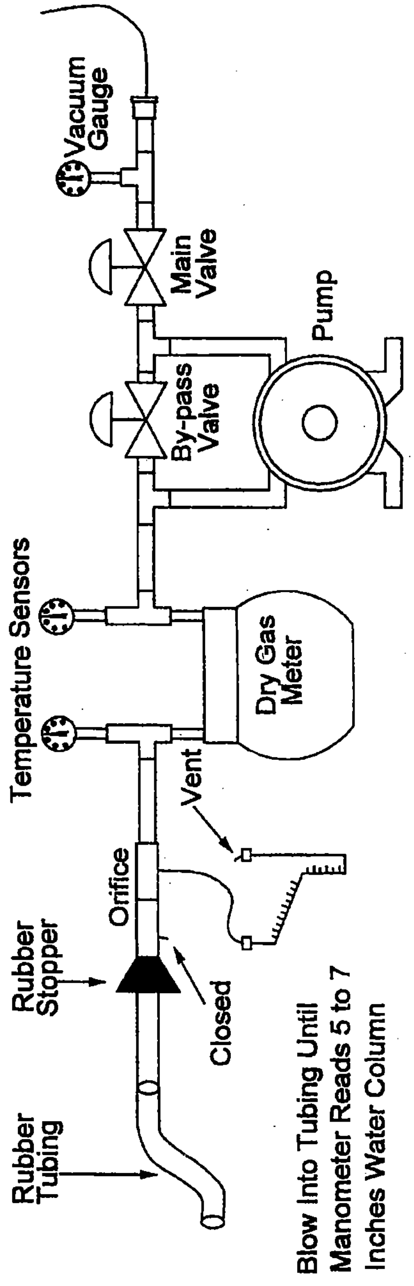


Figure 5-2. Leak Check of Meter Box.

Plant ____________________
Location ____________________
Operator ____________________
Date ____________________
Run No. ____________________
Sample box No. ____________________
Meter box No. ____________________
Meter $\Delta H@$ ____________________
C factor ____________________
Pitot tube coefficient, Cp ____________________

SCHEMATIC OF STACK CROSS SECTION

Ambient temperature ____________________
Barometric pressure ____________________
Assumed moisture, % ____________________
Probe length, (ft.) ____________________
Nozzle Identification No. ____________________
Average calibrated nozzle diameter, (in.) ____________________
Probe heater setting ____________________
Leak rate, (cfm) ____________________
Static pressure, (in. Hg) ____________________
Filter No. ____________________

Traverse point number	Sampling time	Vacuum	Stack temperature	Velocity head	Pressure differential across orifice meter	Gas meter reading	Gas sample temperature at dry gas meter		Filter temperature	Temperature of gas leaving condenser or last impinger
							Inlet	Outlet		
	min.	(in.Hg)	(T_s) (°F)	(ΔP_s) (in. H_2O)	(in. H_2O)	(ft^3)	(°F)	(°F)	(°F)	(°F)
Total							Avg.	Avg.		
Average							Avg.			

Figure 5-3. Particulate Field Data.

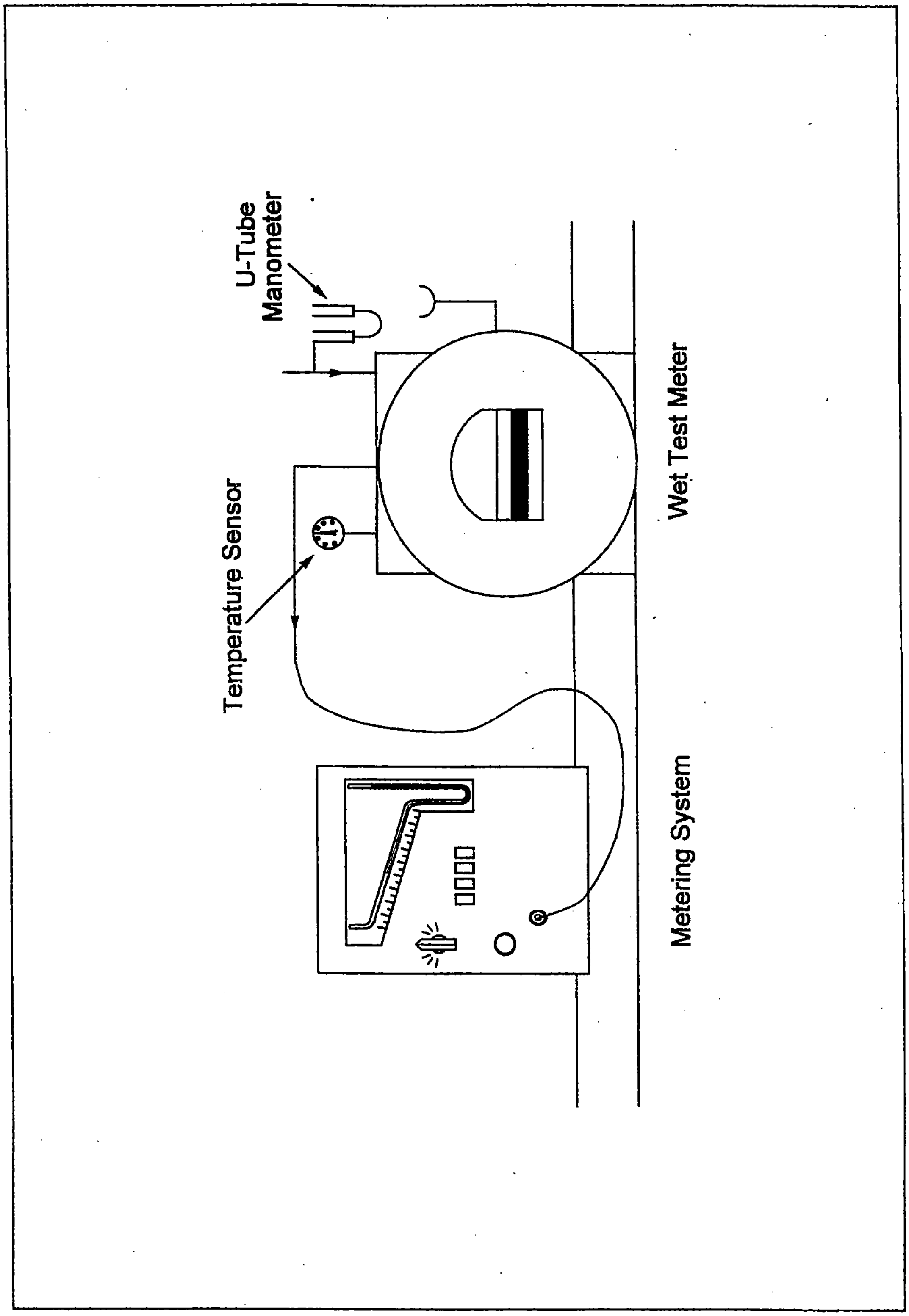


Figure 5-4. Equipment arrangement for metering system calibration.

Date: ____________ Metering System Identification: ________

Barometric pressure, P_b = ________ in. Hg

Orifice manometer setting ΔH in. H_2O	Spirometer (wet meter) gas volume, V_w ft^3	Dry gas meter volume, V_m, ft^3	Temperatures				Time θ min
			Spirometer (wet meter) T_w °F	Dry Gas Meter			
				Inlet T_i °F	Outlet T_o °F	Average T_m °F	

Calculations

ΔH in H_2O	Y				ΔH@		
Average							

Y = Ratio of reading of wet test meter to dry test meter; tolerance for individual values ± 0.02 from average.

$\Delta H_@$ = Orifice pressure differential that equates to 0.75 cfm of air @ 68°F and 29.92 inches of mercury, in. H_2O; tolerance for individual values ± 0.20 from average.

Figure 5-5. Example Data Sheet for Calibration of Metering System (English Units).

Plant
Date
Run No.
Filter No.
Amount liquid lost during transport
Acetone blank volume, m1
Acetone blank concentration, mg/mg (Equation 5–4)
Acetone wash blank, mg (Equation 5–5)

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			
2.			
Total: Less acetone blank. Weight of particulate matter.			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final Initial Liquid collected Total volume collected		g* ml

*Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

Figure 5–6. Analytical Data Sheet

$$\frac{\text{Increase, g}}{(1\text{g}/\text{ml})} = \text{Volume water, ml}$$

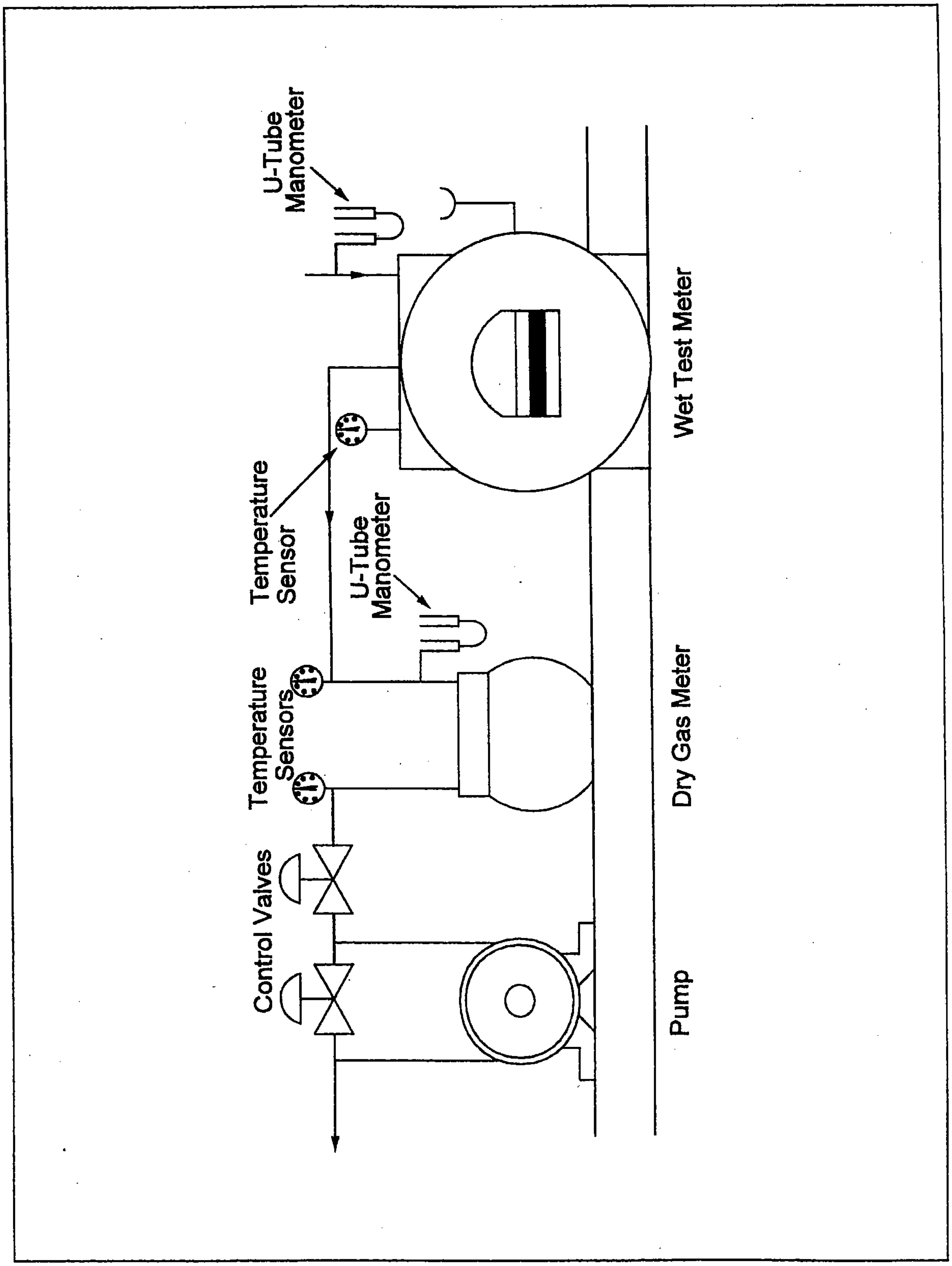


Figure 5-7. Equipment Arrangement for Dry Gas Meter Calibration.

Date: ______________________

Dry Gas Meter Identification: ______________

Barometric Pressure (P): ______________ in. Hg

Approximate Flow Rate ($\bar{Q}$) cfm	Spirometer (Wet Meter) Gas Volume (V_s) ft³	Dry Gas Meter Volume (V_{dg}) ft³	Temperatures				Dry Gas Meter Pressure (Δp) in. H_2O	Time (q) ft³	Flow Rate (Q) cfm	Meter Coefficient (Y_{ds})	Average Meter Coefficient ($\overline{Y_{ds}}$)
			Spirometer (Wet Meter) (t_s) °F	Dry Gas Meter							
				Inlet (t_i) °F	Outlet (t_o) °F	Average ($\bar{t}_d$) °F					
0.40											
0.60											
0.80											
1.00											
1.20											

$$Q = K_1 \frac{P_{bar} V_w}{(T_w + T_{std} Q)} \qquad Y_{ds} = \frac{V_w (T_{ds} + T_{std}) P_{bar}}{V_{ds} (T_w + T_{std}) (P_{bar} + \Delta P/13.6)}$$

Figure 5-8. Example Data Sheet for Calibration of a Standard Dry Gas Meter for Method 5 Sampling Equipment (English units).

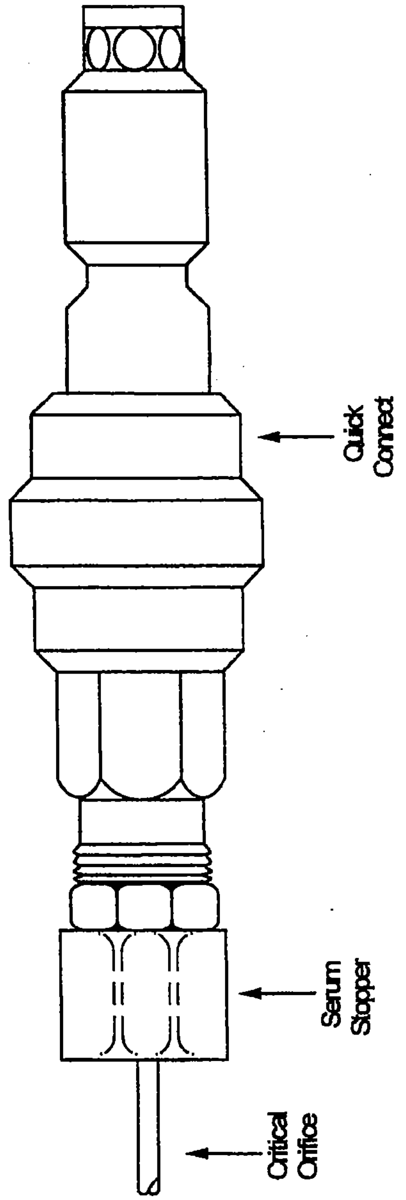


Figure 5-9. Critical Orifice Adaptation to Method 5 Metering System.

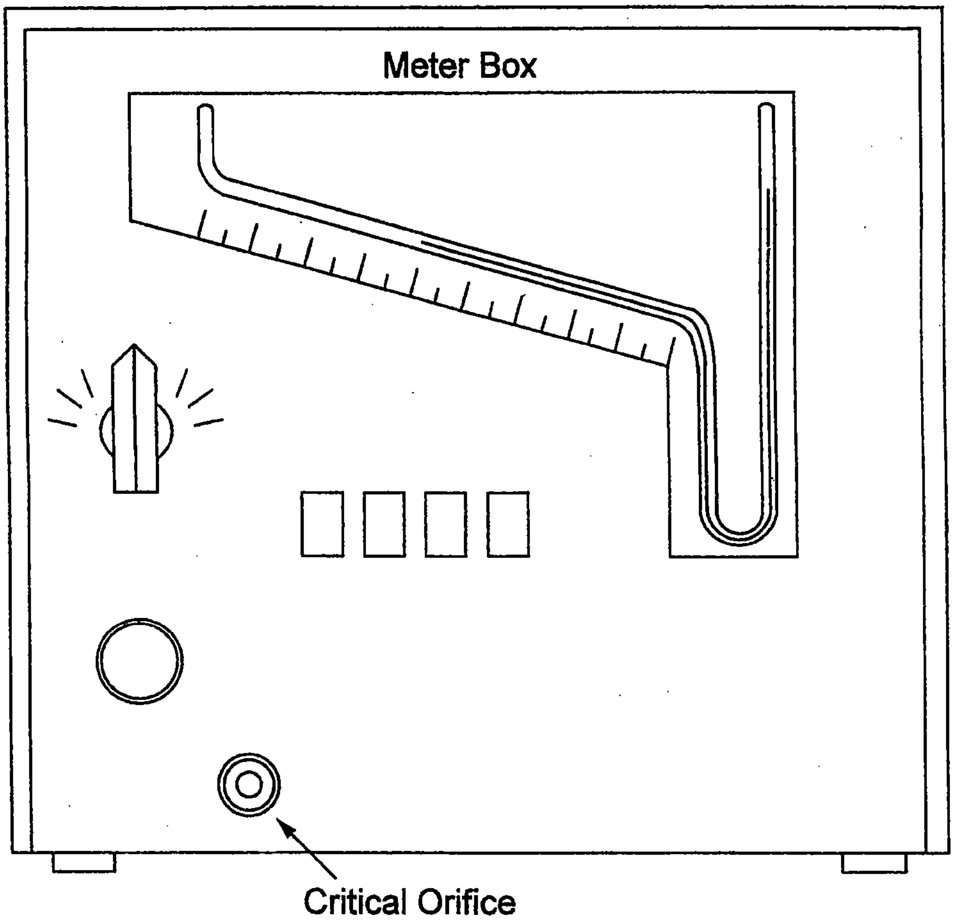


Figure 5-10. Apparatus Setup.

Date
Train ID
DGM cal. factor
Critical orifice ID

Dry gas meter		Run No.	
		1	2
Final reading	m^3 (ft^3)		
Initial reading	m^3 (ft^3)		
Difference, V^m	m^3 (ft^3)		
Inlet/Outlet			
Temperatures:	°C (°F)	/	/
Initial	°C (°F)	/	/
Final	min/sec	/	/
Av. Temeperature, t_m	min		
Time, θ			
Orifice man. rdg., ΔH	mm (in.) H_2		
Bar. pressure, P^{bar}	mm (in.) Hg		
Ambient temperature, t_{amb}	mm (in.) Hg		
Pump vacuum			
K′ factor			
Average			

Figure 5–11. Data sheet of determining K′ factor.

Date
Train ID
Critical orifice ID
Critical orifice K' factor

Dry gas meter		Run No.	
		1	2
Final reading	m^3 (ft^3)		
Initial reading	m^3 (ft^3)		
Difference, V_m	m^3 (ft^3)		
Inlet/outlet temperatures	°C (°F)	/	/
Initial	°C (°F)	/	/
Final	°C (°F)		
Avg. Temperature, t_m	min/sec	/	/
Time, θ	min		
Orifice man. rdg., ΔH	min		
Bar. pressure, P_{bar}	mm (in.) H_2O		
Ambient temperature, t_{amb}	mm (in.) Hg		
Pump vacuum	°C (°F)		
$V_{m(std)}$	mm (in.) Hg		
$V_{cr(std)}$	m^3 (ft^3)		
DGM cal. factor, Y	m^3 (ft^3)		

Figure 5–12. Data Sheet for Determining DGM Y Factor

Method 5A—Determination of Particulate Matter Emissions From the Asphalt Processing and Asphalt Roofing Industry

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Applications

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of 42 ± 10 °C (108 ± 18 °F). The PM mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as Method 5, Section 6.1, with the following exceptions and additions:

6.1.1 Probe Liner. Same as Method 5, Section 6.1.1.2, with the note that at high stack gas temperatures greater than 250 °C (480 °F), water-cooled probes may be required to control the probe exit temperature to 42 ± 10 °C (108 ± 18 °F).

6.1.2 Precollector Cyclone. Borosilicate glass following the construction details shown in Air Pollution Technical Document (APTD)–0581, "Construction Details of Isokinetic Source-Sampling Equipment" (Reference 2 in Method 5, Section 17.0).

Note: The cyclone shall be used when the stack gas moisture is greater than 10 percent, and shall not be used otherwise.

6.1.3 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling at 42 ± 10 °C (108 ± 18 °F).

6.2 Sample Recovery. The following items are required for sample recovery:

6.2.1 Probe-Liner and Probe-Nozzle Brushes, Graduated Cylinder and/or Balance, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as in Method 5, Sections 6.2.1, 6.2.5, 6.2.6, and 6.2.7, respectively.

6.2.2 Wash Bottles. Glass.

6.2.3 Sample Storage Containers. Chemically resistant 500-ml or 1,000-ml borosilicate glass bottles, with rubber-backed Teflon screw cap liners or caps that are constructed so as to be leak-free, and resistant to chemical attack by 1,1,1-trichloroethane (TCE). (Narrow-mouth glass bottles have been found to be less prone to leakage.)

6.2.4 Petri Dishes. Glass, unless otherwise specified by the Administrator.

6.2.5 Funnel. Glass.

6.3 Sample Analysis. Same as Method 5, Section 6.3, with the following additions:

6.3.1 Beakers. Glass, 250-ml and 500-ml.

6.3.2 Separatory Funnel. 100-ml or greater.

7.0. Reagents and Standards

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters, Silica Gel, Water, and Crushed Ice. Same as in Method 5, Sections 7.1.1, 7.1.2, 7.1.3, and 7.1.4, respectively.

7.1.2 Stopcock Grease. TCE-insoluble, heat-stable grease (if needed). This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used.

7.2 Sample Recovery. Reagent grade TCE, ≤0.001 percent residue and stored in glass bottles. Run TCE blanks before field use, and use only TCE with low blank values (≤0.001 percent). In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

7.3 Analysis. Two reagents are required for the analysis:

7.3.1 TCE. Same as in Section 7.2.

7.3.2 Desiccant. Same as in Method 5, Section 7.3.2.

8.0. Sample Collection, Preservation, Storage, and Transport

8.1. Pretest Preparation. Unless otherwise specified, maintain and calibrate all components according to the procedure described in APTD–0576, "Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment" (Reference 3 in Method 5, Section 17.0).

8.1.1 Prepare probe liners and sampling nozzles as needed for use. Thoroughly clean each component with soap and water followed by a minimum of three TCE rinses. Use the probe and nozzle brushes during at least one of the TCE rinses (refer to Section 8.7 for rinsing techniques). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.

8.1.2 Prepare silica gel portions and glass filters as specified in Method 5, Section 8.1.

8.2 Preliminary Determinations. Select the sampling site, probe nozzle, and probe length as specified in Method 5, Section 8.2. Select a total sampling time greater than or equal to the minimum total sampling time specified in the "Test Methods and Procedures" section of the applicable subpart of the regulations. Follow the guidelines outlined in Method 5, Section 8.2 for sampling time per point and total sample volume collected.

8.3 Preparation of Sampling Train. Prepare the sampling train as specified in Method 5, Section 8.3, with the addition of the precollector cyclone, if used, between the probe and filter holder. The temperature of the precollector cyclone, if used, should be maintained in the same range as that of the filter, *i.e.*, 42 ± 10 °C (108 ± 18 °F). Use no stopcock grease on ground glass joints unless grease is insoluble in TCE.

8.4 Leak-Check Procedures. Same as Method 5, Section 8.4.

8.5 Sampling Train Operation. Operate the sampling train as described in Method 5, Section 8.5, except maintain the temperature of the gas exiting the filter holder at 42 ± 10 °C (108 ± 18 °F).

8.6 Calculation of Percent Isokinetic. Same as Method 5, Section 8.6.

8.7 Sample Recovery. Same as Method 5, Section 8.7.1 through 8.7.6.1, with the addition of the following:

8.7.1 Container No. 2 (Probe to Filter Holder).

8.7.1.1 Taking care to see that material on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, precollector cyclone and collector flask (if used), and front half of the filter holder by washing these components with TCE and placing the wash in a glass container. Carefully measure the total amount of TCE used in the rinses. Perform the TCE rinses as described in Method 5, Section 8.7.6.2, using TCE instead of acetone.

8.7.1.2 Brush and rinse the inside of the cyclone, cyclone collection flask, and the front half of the filter holder. Brush and rinse each surface three times or more, if necessary, to remove visible PM.

8.7.2 Container No. 3 (Silica Gel). Same as in Method 5, Section 8.7.6.3.

8.7.3 Impinger Water. Same as Method 5, Section 8.7.6.4.

8.8 Blank. Save a portion of the TCE used for cleanup as a blank. Take 200 ml of this TCE directly from the wash bottle being used, and place it in a glass sample container labeled "TCE Blank."

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 A quality control (QC) check of the volume metering system at the field site is suggested before collecting the sample. Use the procedure outlined in Method 5, Section 9.2.

10.0 Calibration and Standardization

Same as Method 5, Section 10.0.

11.0 Analytical Procedures

11.1 Analysis. Record the data required on a sheet such as the one shown in Figure 5A–1. Handle each sample container as follows:

11.1.1 Container No. 1 (Filter). Transfer the filter from the sample container to a tared glass weighing dish, and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Rinse Container No. 1 with a measured amount of TCE, and analyze this rinse with the contents of Container No. 2. Weigh the filter to a constant weight. For the purpose of this analysis, the term "constant weight" means a difference of no more than 10 percent of the net filter weight or 2 mg (whichever is greater) between two consecutive weighings made 24 hours apart. Report the "final weight" to the nearest 0.1 mg as the average of these two values.

11.1.2 Container No. 2 (Probe to Filter Holder).

11.1.2.1 Before adding the rinse from Container No. 1 to Container No. 2, note the level of liquid in Container No. 2, and confirm on the analysis sheet whether leakage occurred during transport. If noticeable leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to correct the final results.

11.1.2.2 Add the rinse from Container No. 1 to Container No. 2 and measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Check to see whether there is any appreciable quantity of condensed water present in the TCE rinse (look for a boundary layer or phase separation). If the volume of condensed water appears larger than 5 ml, separate the oil-TCE fraction from the water fraction using a separatory funnel. Measure the volume of the water phase to the nearest ml; adjust the stack gas moisture content, if necessary (see Sections 12.3 and 12.4). Next, extract the water phase with several 25-ml portions of TCE until, by visual observation, the TCE does not remove any additional organic material. Transfer the remaining water fraction to a tared beaker and evaporate to dryness at 93 °C (200 °F), desiccate for 24 hours, and weigh to the nearest 0.1 mg.

11.1.2.3 Treat the total TCE fraction (including TCE from the filter container rinse and water phase extractions) as follows: Transfer the TCE and oil to a tared beaker, and evaporate at ambient temperature and pressure. The evaporation of TCE from the solution may take several days. Do not desiccate the sample until the solution reaches an apparent constant volume or until the odor of TCE is not detected. When it appears that the TCE has evaporated, desiccate the sample, and weigh it at 24-hour intervals to obtain a "constant weight" (as defined for Container No. 1 above). The "total weight" for Container No. 2 is the sum of the evaporated PM weight of the TCE-oil and water phase fractions. Report the results to the nearest 0.1 mg.

11.1.3 Container No. 3 (Silica Gel). This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance..

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

Note: In order to facilitate the evaporation of TCE liquid samples, these samples may be dried in a controlled temperature oven at temperatures up to 38 °C (100 °F) until the liquid is evaporated.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature. Same as Method 5, Section 12.1, with the following additions:

C_t = TCE blank residue concentration, mg/g.
m_t = Mass of residue of TCE blank after evaporation, mg.
V_{pc} = Volume of water collected in precollector, ml.
V_t = Volume of TCE blank, ml.
V_{tw} = Volume of TCE used in wash, ml.
W_t = Weight of residue in TCE wash, mg.
ρ_t = Density of TCE (see label on bottle), g/ml.

12.2 Dry Gas Meter Temperature, Orifice Pressure Drop, and Dry Gas Volume. Same as Method 5, Sections 12.2 and 12.3, except use data obtained in performing this test.

12.3 Volume of Water Vapor.

$$V_{w(std)} = K_2\left(V_{lc} + V_{pc}\right) \qquad \text{Eq. 5A-1}$$

Where:

K_2 = 0.001333 m^3/ml for metric units.
= 0.04706 ft^3/ml for English units.

12.4 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Eq. 5A-2}$$

Note: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger and precollector analysis (Equations 5A–1 and 5A–2) and a second from the assumption of saturated conditions. The lower of the two values of moisture content shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in Section 4.0 of Method 4. For the purpose of this method, the average stack gas temperature from Figure 5–3 of Method 5 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is within 1 °C (2 °F).

12.5 TCE Blank Concentration.

$$C_t = \frac{m_t}{V_t \rho_t} \qquad \text{Eq. 5A-3}$$

Note: In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

12.6 TCE Wash Blank.

$$W_t = C_t V_{tw} \rho_t \qquad \text{Eq. 5A-4}$$

12.7 Total PM Weight. Determine the total PM catch from the sum of the weights obtained from Containers 1 and 2, less the TCE blank.

12.8 PM Concentration.

$$c_s = K_3 \frac{m_n}{V_{m(std)}} \qquad \text{Eq. 5A-5}$$

Where:

K_3 = 0.001 g/mg for metric units
= 0.0154 gr/mg for English units

12.9 Isokinetic Variation. Same as in Method 5, Section 12.11.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 5, Section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Plant
Date
Run No.
Filter No.
Amount liquid lost during transport
Acetone blank volume, ml
Acetone blank concentration, mg/mg (Equation 5–4)
Acetone wash blank, mg (Equation 5–5)

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			
2.			
Total: Less acetone blank. Weight of particulate matter.			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final		
Initial		
Liquid collected		
Total volume collected	....	g* ml

*Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{(1\text{g/ml})} = \text{Volume water, ml}$$

Method 5B—Determination of Nonsulfuric Acid Particulate Matter Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5.

1.0 Scope and Application

1.1 Analyte. Nonsulfuric acid particulate matter. No CAS number assigned.

1.2 Applicability. This method is determining applicable for the determination of nonsulfuric acid particulate matter from stationary sources, only where specified by an applicable subpart of the regulations or where approved by the Administrator for a particular application.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of 160 ± 14 °C (320 ± 25 °F). The collected sample is then heated in an oven at 160 °C (320 °F) for 6 hours to volatilize any condensed sulfuric acid that may have been collected, and the nonsulfuric acid particulate mass is determined gravimetrically.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Method 5, Section 6.0, with the following addition and exceptions:

6.1 Sample Collection. The probe liner heating system and filter heating system must be capable of maintaining a sample gas temperature of 160 ± 14 °C (320 ± 25 °F).

6.2 Sample Preparation. An oven is required for drying the sample.

7.0 Reagents and Standards

Same as Method 5, Section 7.0.

8.0 Sample Collection, Preservation, Storage, and Transport.

Same as Method 5, with the exception of the following:

8.1 Initial Filter Tare. Oven dry the filter at 160 ± 5 °C (320 ± 10 °F) for 2 to 3 hours, cool in a desiccator for 2 hours, and weigh. Desiccate to constant weight to obtain the initial tare weight. Use the applicable specifications and techniques of Section 8.1.3 of Method 5 for this determination.

8.2 Probe and Filter Temperatures. Maintain the probe outlet and filter temperatures at 160 ± 14 °C (320 ± 25 °F).

9.0 Quality Control

Same as Method 5, Section 9.0.

10.0 Calibration and Standardization

Same as Method 5, Section 10.0.

11.0 Analytical Procedure

Same as Method 5, Section 11.0, except replace Section

11.2.2 With the following:

11.1 Container No. 2. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Then oven dry the probe and filter samples at a temperature of 160 ± 5 °C (320 ± 10 °F) for 6 hours. Cool in a desiccator for 2 hours, and weigh to constant weight. Report the results to the nearest 0.1 mg.

12.0 Data Analysis and Calculations

Same as in Method 5, Section 12.0.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 5, Section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

* * * * *

Method 5D—Determination of Particulate Matter Emissions from Positive Pressure Fabric Filters

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, Method 17.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability.

1.2.1 This method is applicable for the determination of PM emissions from positive pressure fabric filters. Emissions are determined in terms of concentration (mg/m^3 or gr/ft^3) and emission rate (kg/hr or lb/hr).

1.2.2 The General Provisions of 40 CFR part 60, § 60.8(e), require that the owner or operator of an affected facility shall provide performance testing facilities. Such performance testing facilities include sampling ports, safe sampling platforms, safe access to sampling sites, and utilities for testing. It is intended that affected facilities also provide sampling locations that meet the specification for adequate stack length and minimal flow disturbances as described in Method 1. Provisions for testing are often overlooked factors in designing fabric filters or are extremely costly. The purpose of this procedure is to identify appropriate alternative locations and procedures for sampling the emissions from positive pressure fabric filters. The requirements that the affected facility owner or operator provide adequate access to performance testing facilities remain in effect.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature at or above the exhaust gas temperature up to a nominal 120°C (248 ± 25°F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Section 6.0 of either Method 5 or Method 17.

7.0 Reagents and Standards

Same as Section 7.0 of either Method 5 or Method 17.

8.0 Sample Collection, Preservation, Storage, and Transport

Same Section 8.0 of either Method 5 or Method 17, except replace Section 8.2.1 of Method 5 with the following:

8.1 Determination of Measurement Site. The configuration of positive pressure fabric filter structures frequently are not amenable to emission testing according to the requirements of Method 1. Following are several alternatives for determining measurement sites for positive pressure fabric filters.

8.1.1 Stacks Meeting Method 1 Criteria. Use a measurement site as specified in Method 1, Section 11.1.

8.1.2 Short Stacks Not Meeting Method 1 Criteria. Use stack extensions and the procedures in Method 1. Alternatively, use flow straightening vanes of the "egg-crate" type (see Figure 5D–1). Locate the measurement site downstream of the straightening vanes at a distance equal to or greater than two times the average equivalent diameter of the vane openings and at least one-half of the overall stack diameter upstream of the stack outlet.

8.1.3 Roof Monitor or Monovent. (See Figure 5D–2). For a positive pressure fabric filter equipped with a peaked roof monitor, ridge vent, or other type of monovent, use a measurement site at the base of the monovent. Examples of such locations are shown in Figure 5D–2. The measurement site must be upstream of any exhaust point (*e.g.*, louvered vent).

8.1.4 Compartment Housing. Sample immediately downstream of the filter bags directly above the tops of the bags as shown in the examples in Figure 5D–2. Depending on the housing design, use sampling ports in the housing walls or locate the sampling equipment within the compartment housing.

8.2 Determination of Number and Location of Traverse Points. Locate the traverse points according to Method 1, Section 11.3. Because a performance test consists of at least three test runs and because of the varied configurations of positive pressure fabric filters, there are several schemes by which the number of traverse points can be determined and the three test runs can be conducted.

8.2.1 Single Stacks Meeting Method 1 Criteria. Select the number of traverse points according to Method 1. Sample all traverse points for each test run.

8.2.2 Other Single Measurement Sites. For a roof monitor or monovent, single compartment housing, or other stack not meeting Method 1 criteria, use at least 24 traverse points. For example, for a rectangular measurement site, such as a monovent, use a balanced 5×5 traverse point matrix. Sample all traverse points for each test run.

8.2.3 Multiple Measurement Sites. Sampling from two or more stacks or measurement sites may be combined for a test run, provided the following guidelines are met:

8.2.3.1 All measurement sites up to 12 must be sampled. For more than 12 measurement sites, conduct sampling on at least 12 sites or 50 percent of the sites, whichever is greater. The measurement sites sampled should be evenly, or nearly evenly, distributed among the available sites; if not, all sites are to be sampled.

8.2.3.2 The same number of measurement sites must be sampled for each test run.

8.2.3.3 The minimum number of traverse points per test run is 24. An exception to the 24-point minimum would be a test combining the sampling from two stacks meeting Method 1 criteria for acceptable stack length, and

Method 1 specifies fewer than 12 points per site.

8.2.3.4 As long as the 24 traverse points per test run criterion is met, the number of traverse points per measurement site may be reduced to eight.

8.2.3.5 Alternatively, conduct a test run for each measurement site individually using the criteria in Section 8.2.1 or 8.2.2 to determine the number of traverse points. Each test run shall count toward the total of three required for a performance test. If more than three measurement sites are sampled, the number of traverse points per measurement site may be reduced to eight as long as at least 72 traverse points are sampled for all the tests.

8.2.3.6 The following examples demonstrate the procedures for sampling multiple measurement sites.

8.2.3.6.1 Example 1: A source with nine circular measurement sites of equal areas may be tested as follows: For each test run, traverse three measurement sites using four points per diameter (eight points per measurement site). In this manner, test run number 1 will include sampling from sites 1,2, and 3; run 2 will include samples from sites 4, 5, and 6; and run 3 will include sites 7, 8, and 9. Each test area may consist of a separate test of each measurement site using eight points. Use the results from all nine tests in determining the emission average.

8.2.3.6.2 Example 2: A source with 30 rectangular measurement sites of equal areas may be tested as follows: For each of the three test runs, traverse five measurement sites using a 3x3 matrix of traverse points for each site. In order to distribute the sampling evenly over all the available measurement sites while sampling only 50 percent of the sites, number the sites consecutively from 1 to 30 and sample all the even numbered (or odd numbered) sites. Alternatively, conduct a separate test of each of 15 measurement sites using Section 8.2.1 or 8.2.2 to determine the number and location of traverse points, as appropriate.

8.2.3.6.3 Example 3: A source with two measurement sites of equal areas may be tested as follows: For each test of three test runs, traverse both measurement sites, using Section 8.2.3 in determining the number of traverse points. Alternatively, conduct two full emission test runs for each measurement site using the criteria in Section 8.2.1 or 8.2.2 to determine the number of traverse points.

8.2.3.7 Other test schemes, such as random determination of traverse points for a large number of measurement sites, may be used with prior approval from the Administrator.

8.3 Velocity Determination.

8.3.1 The velocities of exhaust gases from positive pressure baghouses are often too low to measure accurately with the type S pitot tube specified in Method 2 (*i.e.*, velocity head <1.3 mm H_2O (0.05 in. H_2O)). For these conditions, measure the gas flow rate at the fabric filter inlet following the procedures outlined in Method 2. Calculate the average gas velocity at the measurement site as shown in Section 12.2 and use this average velocity in determining and maintaining isokinetic sampling rates.

8.3.2 Velocity determinations to determine and maintain isokinetic rates at measurement sites with gas velocities within the range measurable with the type S pitot tube (*i.e.*, velocity head greater than 1.3 mm H_2O (0.05 in. H_2O)) shall be conducted according to the procedures outlined in Method 2.

8.4 Sampling. Follow the procedures specified in Sections 8.1 through 8.6 of Method 5 or Sections 8.1 through 8.25 in Method 17 with the exceptions as noted above.

8.5 Sample Recovery. Follow the procedures specified in Section 8.7 of Method 5 or Section 8.2 of Method 17.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.0, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Same as Section 10.0 of either Method 5 or Method 17.

11.0 Analytical Procedure

Same as Section 11.0 of either Method 5 or Method 17.

12.0 Data Analysis and Calculations

Same as Section 12.0 of either Method 5 or Method 17 with the following exceptions:

12.1 Nomenclature.

A_o = Measurement site(s) total cross-sectional area, m^2 (ft^2).
$\bar{C}$ or C_{avg} = Average concentration of PM for all n runs, mg/scm (gr/scf).
Q_i = Inlet gas volume flow rate, m^3/sec (ft^3/sec).
m_i = Mass collected for run i of n, mg (gr).
T_o = Average temperature of gas at measurement site, °K (°R).
T_i = Average temperature of gas at inlet, °K (°R).
Vol_i = Sample volume collected for run i of n, scm (scf).
$\bar{v}$ = Average gas velocity at the measurement site(s), m/s (ft/s)
Q_o = Total baghouse exhaust volumetric flow rate, m^3/sec (ft^3/sec).
Q_d = Dilution air flow rate, m^3/sec (ft^3/sec).
Tamb = Ambient Temperature, (°K).

12.2 Average Gas Velocity. When following Section 8.3.1, calculate the average gas velocity at the measurement site as follows:

$$\bar{v} = \frac{Q_o}{A_o} \qquad \text{Eq. 5D-1}$$

12.3 Volumetric Flow Rate. Total volumetric flow rate may be determined as follows:

$$Q_o = Q_i + Q_d \qquad \text{Eq. 5D-2}$$

12.4 Dilution Air Flow Rate.

$$Q_d = \frac{Q_i \left(T_i - T_o\right)}{T_o - T_{amb}} \qquad \text{Eq. 5D-3}$$

12.5 Average PM Concentration. For multiple measurement sites, calculate the average PM concentration as follows:

$$C_{avg} \text{ or } \bar{C} = \frac{\sum_{i=1}^{n} m_i}{\sum_{i=1}^{n} Vol_i} \qquad \text{Eq. 5D-4}$$

13.0 Method Performance. [Reserved]

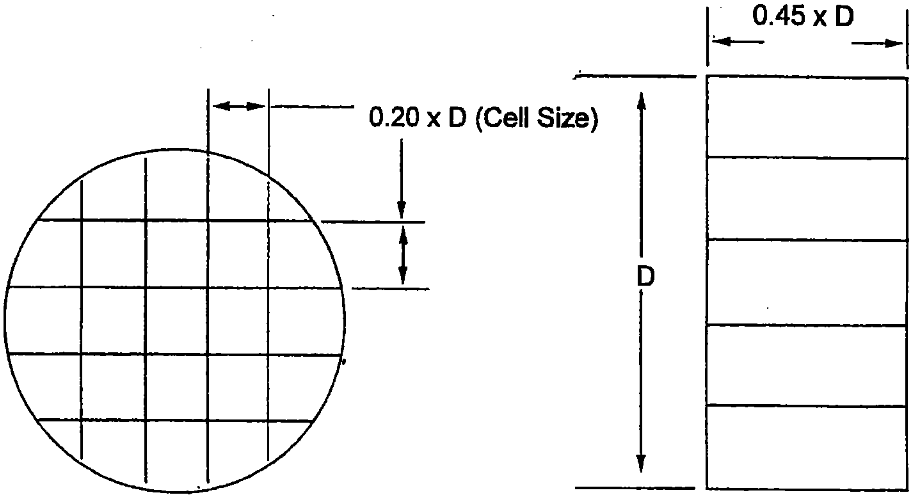
14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

Same as Method 5, Section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data



NOTE: Position Straighteners So That Cell Sides Are Located Approximately 45° From Traverse Diameters.

Figure 5D-1. Example of Flow Straightening Vanes.

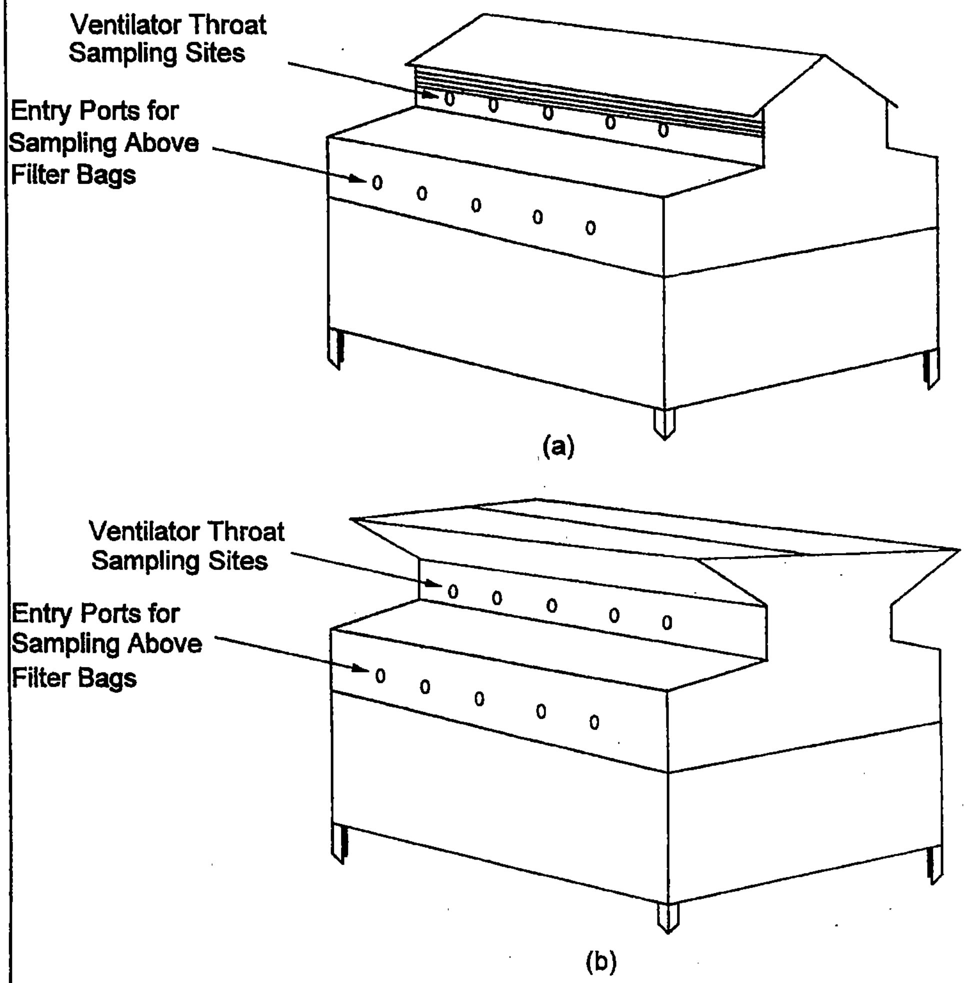


Figure 5D-2. Acceptable Sampling Site Locations for: (a) Peaked Roof; and (b) Ridge Vent Type Fabric Filters

Method 5E—Determination of Particulate Matter Emissions From the Wool Fiberglass Insulation Manufacturing Industry

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Applications

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from wool fiberglass insulation manufacturing sources.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and is collected either on a glass fiber filter maintained at a temperature in the range of 120 ± 14°C (248 ± 25°F) and in impingers in solutions of 0.1 N sodium hydroxide (NaOH). The filtered particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water. The condensed PM collected in the impinger solutions is determined as total organic carbon (TOC) using a nondispersive infrared type of analyzer. The sum of the filtered PM mass and the condensed PM is reported as the total PM mass.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Hydrochloric Acid (HCl). Highly toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent in air can be lethal in minutes. Will react with metals, producing hydrogen.

5.2.2 Sodium Hydroxide (NaOH). Causes severe damage to eye tissues and to skin. Inhalation causes irritation to nose, throat, and lungs. Reacts exothermically with limited amounts of water.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as Method 5, Section 6.1, with the exception of the following:

6.1.1 Probe Liner. Same as described in Section 6.1.1.2 of Method 5 except use only borosilicate or quartz glass liners.

6.1.2 Filter Holder. Same as described in Section 6.1.1.5 of Method 5 with the addition of a leak-tight connection in the rear half of the filter holder designed for insertion of a temperature sensor used for measuring the sample gas exit temperature.

6.2 Sample Recovery. Same as Method 5, Section 6.2, except three wash bottles are needed instead of two and only glass storage bottles and funnels may be used.

6.3 Sample Analysis. Same as Method 5, Section 6.3, with the additional equipment for TOC analysis as described below:

6.3.1 Sample Blender or Homogenizer. Waring type or ultrasonic.

6.3.2 Magnetic Stirrer.

6.3.3 Hypodermic Syringe. 0- to 100-μl capacity.

6.3.4 Total Organic Carbon Analyzer. Rosemount Model 2100A analyzer or equivalent and a recorder.

6.3.5 Beaker. 30-ml.

6.3.6 Water Bath. Temperature controlled.

6.3.7 Volumetric Flasks. 1000-ml and 500-ml.

7.0 Reagents and Standards

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. Same as Method 5, Section 7.1, with the addition of 0.1 N NaOH (Dissolve 4 g of NaOH in water and dilute to 1 liter).

7.2 Sample Recovery. Same as Method 5, Section 7.2, with the addition of the following:

7.2.1 Water. Deionized distilled to conform to ASTM Specification D 1193–77 or 91 Type 3 (incorporated by reference—see § 60.17). The potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.2.2 Sodium Hydroxide. Same as described in Section 7.1.

7.3 Sample Analysis. Same as Method 5, Section 7.3, with the addition of the following:

7.3.1 Carbon Dioxide-Free Water. Distilled or deionized water that has been freshly boiled for 15 minutes and cooled to room temperature while preventing exposure to ambient air by using a cover vented with an Ascarite tube.

7.3.2 Hydrochloric Acid. HCl, concentrated, with a dropper.

7.3.3 Organic Carbon Stock Solution. Dissolve 2.1254 g of dried potassium biphthalate ($HOOCC_6H_4COOK$) in CO_2-free water, and dilute to 1 liter in a volumetric flask. This solution contains 1000 mg/L organic carbon.

7.3.4 Inorganic Carbon Stock Solution. Dissolve 4.404 g anhydrous sodium carbonate (Na_2CO_3.) in about 500 ml of CO_2-free water in a 1-liter volumetric flask. Add 3.497 g anhydrous sodium bicarbonate ($NaHCO_3$) to the flask, and dilute to 1 liter with CO_2 -free water. This solution contains 1000 mg/L inorganic carbon.

7.3.5 Oxygen Gas. CO_2 -free.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation and Preliminary Determinations. Same as Method 5, Sections 8.1 and 8.2, respectively.

8.2 Preparation of Sampling Train. Same as Method 5, Section 8.3, except that 0.1 N NaOH is used in place of water in the impingers. The volumes of the solutions are the same as in Method 5.

8.3 Leak-Check Procedures, Sampling Train Operation, Calculation of Percent Isokinetic. Same as Method 5, Sections 8.4 through 8.6, respectively.

8.4 Sample Recovery. Same as Method 5, Sections 8.7.1 through 8.7.4, with the addition of the following:

8.4.1 Save portions of the water, acetone, and 0.1 N NaOH used for cleanup as blanks. Take 200 ml of each liquid directly from the wash bottles being used, and place in glass sample containers labeled "water blank," "acetone blank," and "NaOH blank," respectively.

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

8.4.2.1 Container No. 1. Same as Method 5, Section 8.7.6.1.

8.4.2.2 Container No. 2. Use water to rinse the sample nozzle, probe, and front half of the filter holder three times in the manner described in Section 8.7.6.2 of Method 5 except that no brushing is done. Put all the water wash in one container, seal, and label.

8.4.2.3 Container No. 3. Rinse and brush the sample nozzle, probe, and front half of the filter holder with acetone as described for Container No. 2 in Section 8.7.6.2 of Method 5.

8.4.2.4 Container No. 4. Place the contents of the silica gel impinger in its original container as described for Container No. 3 in Section 8.7.6.3 of Method 5.

8.4.2.5 Container No. 5. Measure the liquid in the first three impingers and record the volume or weight as described for the Impinger Water in Section 8.7.6.4 of Method 5. Do not discard this liquid, but place it in a sample container using a glass funnel to aid in the transfer from the impingers or graduated cylinder (if used) to the sample container. Rinse each impinger thoroughly with 0.1 N NaOH three times, as well as the graduated cylinder (if used) and the funnel, and put these rinsings in the same sample container. Seal the container and label to clearly identify its contents.

8.5 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

9.0 Quality Control.

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.3, 10.0	Sampling equipment leak-check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1.2, 11.2.5.3	Repetitive analyses	Ensures precise measurement of total carbon and inorganic carbon concentration of samples, blank, and standards.
10.1.4	TOC analyzer calibration	Ensures linearity of analyzer response to standards.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Same as Method 5, Section 10.0, with the addition of the following procedures for calibrating the total organic carbon analyzer:

10.1 Preparation of Organic Carbon Standard Curve.

10.1.1 Add 10 ml, 20 ml, 30 ml, 40 ml, and 50 ml of the organic carbon stock solution to a series of five 1000-ml volumetric flasks. Add 30 ml, 40 ml, and 50 ml of the same solution to a series of three 500-ml volumetric flasks. Dilute the contents of each flask to the mark using CO_2-free water. These flasks contain 10, 20, 30, 40, 50, 60, 80, and 100 mg/L organic carbon, respectively.

10.1.2 Use a hypodermic syringe to withdraw a 20- to 50-µl aliquot from the 10 mg/L standard solution and inject it into the total carbon port of the analyzer. Measure the peak height. Repeat the injections until three consecutive peaks are obtained within 10 percent of their arithmetic mean. Repeat this procedure for the remaining organic carbon standard solutions.

10.1.3 Calculate the corrected peak height for each standard by deducting the blank correction (see Section 11.2.5.3) as follows:

$$\text{Corrected Peak Height} = A - B \qquad \text{Eq. 5E-1}$$

Where:

A = Peak height of standard or sample, mm or other appropriate unit.

B = Peak height of blank, mm or other appropriate unit.

10.1.4 Prepare a linear regression plot of the arithmetic mean of the three consecutive peak heights obtained for each standard solution against the concentration of that solution. Calculate the calibration factor as the inverse of the slope of this curve. If the product of the arithmetic mean peak height for any standard solution and the calibration factor differs from the actual concentration by more than 5 percent, remake and reanalyze that standard.

10.2 Preparation of Inorganic Carbon Standard Curve. Repeat the procedures outlined in Sections 10.1.1 through 10.1.4, substituting the inorganic carbon stock solution for the organic carbon stock solution, and the inorganic carbon port of the analyzer for the total carbon port.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5–6 of Method 5.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Same as Method 5, Section 11.2.1, except that the filters must be dried at 20 ± 6 °C (68 ± 10 °F) and ambient pressure.

11.2.2 Containers No. 2 and No. 3. Same as Method 5, Section 11.2.2, except that evaporation of the samples must be at 20 ± 6 °C (68 ± 10 °F) and ambient pressure.

11.2.3 Container No. 4. Same as Method 5, Section 11.2.3.

11.2.4 "Water Blank" and "Acetone Blank" Containers. Determine the water and acetone blank values following the procedures for the "Acetone Blank" container in Section 11.2.4 of Method 5. Evaporate the samples at ambient temperature (20 ± 6 °C (68 ± 10 °F)) and pressure.

11.2.5 Container No. 5. For the determination of total organic carbon, perform two analyses on successive identical samples, *i.e.*, total carbon and inorganic carbon. The desired quantity is the difference between the two values obtained. Both analyses are based on conversion of sample carbon into carbon dioxide for measurement by a nondispersive infrared analyzer. Results of analyses register as peaks on a strip chart recorder.

11.2.5.1 The principal differences between the operating parameters for the two channels involve the combustion tube packing material and temperature. In the total carbon channel, a high temperature (950 °C (1740 °F)) furnace heats a Hastelloy combustion tube packed with cobalt oxide-impregnated asbestos fiber. The oxygen in the carrier gas, the elevated temperature, and the catalytic effect of the packing result in oxidation of both organic and inorganic carbonaceous material to CO_2, and steam. In the inorganic carbon channel, a low temperature (150 °C (300 °F)) furnace heats a glass tube containing quartz chips wetted with 85 percent phosphoric acid. The acid liberates CO_2 and steam from inorganic carbonates. The operating temperature is below that required to oxidize organic matter. Follow the manufacturer's instructions for assembly, testing, calibration, and operation of the analyzer.

11.2.5.2 As samples collected in 0.1 N NaOH often contain a high measure of inorganic carbon that inhibits repeatable determinations of TOC, sample pretreatment is necessary. Measure and record the liquid volume of each sample (or impinger contents). If the sample contains solids or immiscible liquid matter, homogenize the sample with a blender or ultrasonics until satisfactory repeatability is obtained. Transfer a representative portion of 10 to 15 ml to a 30-ml beaker, and acidify with about 2 drops of concentrated HCl to a pH of 2 or less. Warm the acidified sample at 50 °C (120 °F) in a water bath for 15 minutes.

11.2.5.3 While stirring the sample with a magnetic stirrer, use a hypodermic syringe to withdraw a 20-to 50-µl aliquot from the beaker. Analyze the sample for total carbon and calculate its corrected mean peak height according to the procedures outlined in Sections 10.1.2 and 10.1.3. Similarly analyze an aliquot of the sample for inorganic carbon. Repeat the analyses for all the samples and for the 0.1 N NaOH blank.

11.2.5.4 Ascertain the total carbon and inorganic carbon concentrations (C_{TC} and C_{IC}, respectively) of each sample and blank by comparing the corrected mean peak heights for each sample and blank to the appropriate standard curve.

Note: If samples must be diluted for analysis, apply an appropriate dilution factor.

12.0 Data Analysis and Calculations

Same as Method 5, Section 12.0, with the addition of the following:

12.1 Nomenclature.

C_c = Concentration of condensed particulate matter in stack gas, gas dry basis, corrected to standard conditions, g/dscm (gr/dscf).
C_{IC} = Concentration of condensed TOC in the liquid sample, from Section 11.2.5, mg/L.
C_t = Total particulate concentration, dry basis, corrected to standard conditions, g/dscm (gr/dscf).
C_{TC} = Concentration of condensed TOC in the liquid sample, from Section 11.2.5, mg/L.
C_{TOC} = Concentration of condensed TOC in the liquid sample, mg/L.
m_{TOC} = Mass of condensed TOC collected in the impingers, mg.
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, from Section 12.3 of Method 5, dscm (dscf).
V_s = Total volume of liquid sample, ml.

12.2 Concentration of Condensed TOC in Liquid Sample.

$$C_{TOC} = C_{TC} - C_{IC} \qquad \text{Eq. 5E-2}$$

12.3 Mass of Condensed TOC Collected.

$$m_{TOC} = 0.001\, C_{TOC} V_s \qquad \text{Eq. 5E-3}$$

Where:

0.001 = Liters per milliliter.

12.4 Concentration of Condensed Particulate Material.

$$C_c = K_4 m_{TOC}/V_{m(std)} \qquad \text{Eq. 5E-4}$$

Where:

K_4 = 0.001 g/mg for metric units.
= 0.0154 gr/mg for English units.

12.5 Total Particulate Concentration.

$$C_t = C_s + C_c \qquad \text{Eq. 5E-4}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References.

Same as Section 17.0 of Method 5, with the addition of the following:

1. American Public Health Association, American Water Works Association, Water Pollution Control Federation. Standard Methods for the Examination of Water and Wastewater. Fifteenth Edition. Washington, D.C. 1980.

17.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

Method 5F—Determination of Nonsulfate Particulate Matter Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Applications

1.1 Analyte. Nonsulfate particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of nonsulfate PM emissions from stationary sources. Use of this method must be specified by an applicable subpart of the standards, or approved by the Administrator for a particular application.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a filter maintained at a temperature in the range 160 ± 14 °C (320 ± 25 °F). The collected sample is extracted with water. A portion of the extract is analyzed for sulfate content by ion chromatography. The remainder is neutralized with ammonium hydroxide (NH_4OH), dried, and weighed. The weight of sulfate in the sample is calculated as ammonium sulfate ($(NH_4)_2SO_4$), and is subtracted from the total particulate weight; the result is reported as nonsulfate particulate matter.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection and Recovery. Same as Method 5, Sections 6.1 and 6.2, respectively.

6.2 Sample Analysis. Same as Method 5, Section 6.3, with the addition of the following:

6.2.1 Erlenmeyer Flasks. 125-ml, with ground glass joints.

6.2.2 Air Condenser. With ground glass joint compatible with the Erlenmeyer flasks.

6.2.3 Beakers. 600-ml.

6.2.4 Volumetric Flasks. 1-liter, 500-ml (one for each sample), 200-ml, and 50-ml (one for each sample and standard).

6.2.5 Pipet. 5-ml (one for each sample and standard).

6.2.6 Ion Chromatograph. The ion chromatograph should have at least the following components.

6.2.6.1 Columns. An anion separation column or other column capable of resolving the sulfate ion from other species present and a standard anion suppressor column. Suppressor columns are produced as proprietary items; however, one can be produced in the laboratory using the resin available from BioRad Company, 32nd and Griffin Streets, Richmond, California. Other systems which do not use suppressor columns may also be used.

6.2.6.2 Pump. Capable of maintaining a steady flow as required by the system.

6.2.6.3 Flow Gauges. Capable of measuring the specified system flow rate.

6.2.6.4 Conductivity Detector.

6.2.6.5 Recorder. Compatible with the output voltage range of the detector.

7.0 Reagents and Standards

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. Same as Method 5, Section 7.1.

7.2 Sample Recovery. Same as Method 5, Section 7.2, with the addition of the following:

7.2.1 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91 Type 3 (incorporated by reference—see § 60.17). The potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.3 Analysis. Same as Method 5, Section 7.3, with the addition of the following:

7.3.1 Water. Same as in Section 7.2.1.

7.3.2 Stock Standard Solution, 1 mg $(NH_4)_2SO_4$/ml. Dry an adequate amount of primary standard grade ammonium sulfate ($(NH_4)_2SO_4$) at 105 to 110 °C (220 to 230 °F) for a minimum of 2 hours before preparing the standard solution. Then dissolve exactly 1.000 g of dried $(NH_4)_2SO_4$ in water in a 1-liter volumetric flask, and dilute to 1 liter. Mix well.

7.3.3 Working Standard Solution, 25 μg $(NH_4)_2SO_4$/ml. Pipet 5 ml of the stock standard solution into a 200-ml volumetric flask. Dilute to 200 ml with water.

7.3.4 Eluent Solution. Weigh 1.018 g of sodium carbonate (Na_2CO_3) and 1.008 g of sodium bicarbonate ($NaHCO_3$), and dissolve in 4 liters of water. This solution is 0.0024 M Na_2CO_3/0.003 M $NaHCO_3$. Other eluents appropriate to the column type and capable of resolving sulfate ion from other species present may be used.

7.3.5 Ammonium Hydroxide. Concentrated, 14.8 M.

7.3.6 Phenolphthalein Indicator. 3,3-Bis(4-hydroxyphenyl)-1-(3H)-isobenzofuranone. Dissolve 0.05 g in 50 ml of ethanol and 50 ml of water.

8.0 Sample Collection, Preservation, Storage, and Transport

Same as Method 5, Section 8.0, with the exception of the following:

8.1 Sampling Train Operation. Same as Method 5, Section 8.5, except that the probe outlet and filter temperatures shall be maintained at 160 ± 14 °C (320 ± 25 °F).

8.2 Sample Recovery. Same as Method 5, Section 8.7, except that the recovery solvent shall be water instead of acetone, and a clean filter from the same lot as those used during testing shall be saved for analysis as a blank.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures

Section	Quality control measure	Effect
8.3, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1.2, 11.2.5.3	Repetitive analyses	Ensures precise measurement of total carbon and inorganic carbon concentration of samples, blank, and standards.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Same as Method 5, Section 10.0, with the addition of the following:

10.1 Determination of Ion Chromatograph Calibration Factor S. Prepare a series of five standards by adding 1.0, 2.0, 4.0, 6.0, and 10.0 ml of working standard solution (25 μg/ml) to a series of five 50-ml volumetric flasks. (The standard masses will equal 25, 50, 100, 150, and 250 μg.) Dilute each flask to the mark with water, and mix well. Analyze each standard according to the chromatograph manufacturer's instructions. Take peak height measurements with symmetrical peaks; in all other cases, calculate peak areas. Prepare or calculate a linear regression plot of the standard masses in μg (x-axis) versus their responses (y-axis). From this line, or equation, determine the slope and calculate its reciprocal which is the calibration factor, S. If any point deviates from the line by more than 7 percent of the concentration at that point, remake and reanalyze that standard. This deviation can be determined by multiplying S times the response for each standard. The resultant concentrations must not differ by more than 7 percent from each known standard mass (*i.e.*, 25, 50, 100, 150, and 250 μg).

10.2 Conductivity Detector. Calibrate according to manufacturer's specifications prior to initial use.

11.0 Analytical Procedure

11.1 Sample Extraction.

11.1.1 Note on the analytical data sheet, the level of the liquid in the container, and whether any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

11.1.2 Cut the filter into small pieces, and place it in a 125-ml Erlenmeyer flask with a ground glass joint equipped with an air condenser. Rinse the shipping container with water, and pour the rinse into the flask. Add additional water to the flask until it contains about 75 ml, and place the flask on a hot plate. Gently reflux the contents for 6 to 8 hours. Cool the solution, and transfer it to a 500-ml volumetric flask. Rinse the Erlenmeyer flask with water, and transfer the rinsings to the volumetric flask including the pieces of filter.

11.1.3 Transfer the probe rinse to the same 500-ml volumetric flask with the filter sample. Rinse the sample bottle with water, and add the rinsings to the volumetric flask. Dilute the contents of the flask to the mark with water.

11.1.4 Allow the contents of the flask to settle until all solid material is at the bottom of the flask. If necessary, remove and centrifuge a portion of the sample.

11.1.5 Repeat the procedures outlined in Sections 11.1.1 through 11.1.4 for each sample and for the filter blank.

11.2 Sulfate (SO_4) Analysis.

11.2.1 Prepare a standard calibration curve according to the procedures outlined in Section 10.1.

11.2.2 Pipet 5 ml of the sample into a 50-ml volumetric flask, and dilute to 50 ml with water. (Alternatively, eluent solution may be used instead of water in all sample, standard, and blank dilutions.) Analyze the set of standards followed by the set of samples, including the filter blank, using the same injection volume used for the standards.

11.2.3 Repeat the analyses of the standards and the samples, with the standard set being done last. The two peak height or peak area responses for each sample must agree within 5 percent of their arithmetic mean for the analysis to be valid. Perform this analysis sequence on the same day. Dilute any sample and the blank with equal volumes of water if the concentration exceeds that of the highest standard.

11.2.4 Document each sample chromatogram by listing the following analytical parameters: injection point, injection volume, sulfate retention time, flow rate, detector sensitivity setting, and recorder chart speed.

11.3 Sample Residue.

11.3.1 Transfer the remaining contents of the volumetric flask to a tared 600-ml beaker or similar container. Rinse the volumetric flask with water, and add the rinsings to the tared beaker. Make certain that all particulate matter is transferred to the beaker. Evaporate the water in an oven at 105 °C (220 °F) until only about 100 ml of water remains. Remove the beakers from the oven, and allow them to cool.

11.3.2 After the beakers have cooled, add five drops of phenolphthalein indicator, and then add concentrated ammonium hydroxide until the solution turns pink. Return the samples to the oven at 105 °C (220 °F), and evaporate the samples to dryness. Cool the samples in a desiccator, and weigh the samples to constant weight.

12.0 Data Analysis and Calculations

Same as Method 5, Section 12.0, with the addition of the following:

12.1 Nomenclature.

C_W = Water blank residue concentration, mg/ml.

F = Dilution factor (required only if sample dilution was needed to reduce the concentration into the range of calibration).

H_S = Arithmetic mean response of duplicate sample analyses, mm for height or mm2 for area.

H_b = Arithmetic mean response of duplicate filter blank analyses, mm for height or mm2 for area.

m_b = Mass of beaker used to dry sample, mg.

m_f = Mass of sample filter, mg.

m_n = Mass of nonsulfate particulate matter in the sample as collected, mg.

m_s = Mass of ammonium sulfate in the sample as collected, mg.

m_t = Mass of beaker, filter, and dried sample, mg.

m_w = Mass of residue after evaporation of water blank, mg.

S = Calibration factor, µg/mm.

V_b = Volume of water blank, ml.

V_S = Volume of sample collected, 500 ml.

12.2 Water Blank Concentration.

$$C_w = \frac{m_w}{V_b} \qquad \text{Eq. 5F-1}$$

12.3 Mass of Ammonium Sulfate.

$$m_s = \frac{(99)\,S\,(H_s - H_b)}{(1000)}F \qquad \text{Eq. 5F-2}$$

Where:

100 = Aliquot factor, 495 ml/5 ml

1000 = Constant, µg/mg

12.4 Mass of Nonsulfate Particulate Matter.

$$m_n = m_t - m_b - m_s - m_f - V_s C_w \qquad \text{Eq. 5F-3}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 The following procedure may be used as an alternative to the procedure in Section 11.0

16.1.1 Apparatus. Same as for Method 6, Sections 6.3.3 to 6.3.6 with the following additions.

16.1.1.1 Beakers. 250-ml, one for each sample, and 600-ml.

16.1.1.2 Oven. Capable of maintaining temperatures of 75 ± 5 °C (167 ± 9 °F) and 105 ± 5 °C (221 ± 9 °F).

16.1.1.3 Buchner Funnel.

16.1.1.4 Glass Columns. 25-mm x 305-mm (1-in. x 12-in.) with Teflon stopcock.

16.1.1.5 Volumetric Flasks. 50-ml and 500-ml, one set for each sample, and 100-ml, 200-ml, and 1000-ml.

16.1.1.6 Pipettes. Two 20-ml and one 200-ml, one set for each sample, and 5-ml.

16.1.1.7 Filter Flasks. 500-ml.

16.1.1.8 Polyethylene Bottle. 500-ml, one for each sample.

16.1.2 Reagents. Same as Method 6, Sections 7.3.2 to 7.3.5 with the following additions:

16.1.2.1 Water, Ammonium Hydroxide, and Phenolphthalein. Same as Sections 7.2.1, 7.3.5, and 7.3.6 of this method, respectively.

16.1.2.2 Filter. Glass fiber to fit Buchner funnel.

16.1.2.3 Hydrochloric Acid (HCl), 1 m. Add 8.3 ml of concentrated HCl (12 M) to 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

16.1.2.4 Glass Wool.

16.1.2.5 Ion Exchange Resin. Strong cation exchange resin, hydrogen form, analytical grade.

16.1.2.6 pH Paper. Range of 1 to 7.

16.1.3 Analysis.

16.1.3.1 Ion Exchange Column Preparation. Slurry the resin with 1 M HCl in a 250-ml beaker, and allow to stand overnight. Place 2.5 cm (1 in.) of glass wool in the bottom of the glass column. Rinse the slurried resin twice with water. Resuspend the resin in water, and pour sufficient resin into the column to make a bed 5.1 cm (2 in.) deep. Do not allow air bubbles to become entrapped in the resin or glass wool to avoid channeling, which may produce erratic results. If necessary, stir the resin with a glass rod to remove air bubbles, after the column has been prepared, never let the liquid level fall below the top of the upper glass wool plug. Place a 2.5-cm (1-in.) plug of glass wool on top of the resin. Rinse the column with water until the eluate gives a pH of 5 or greater as measured with pH paper.

16.1.3.2 Sample Extraction. Followup the procedure given in Section 11.1.3 except do not dilute the sample to 500 ml.

16.1.3.3 Sample Residue.

16.1.3.3.1 Place at least one clean glass filter for each sample in a Buchner funnel, and rinse the filters with water. Remove the filters from the funnel, and dry them in an oven at 105 ± 5° C (221 ± 9 °F); then cool in a desiccator. Weigh each filter to constant weight according to the procedure in Method 5, Section 11.0. Record the weight of each filter to the nearest 0.1 mg.

16.1.3.3.2 Assemble the vacuum filter apparatus, and place one of the clean, tared glass fiber filters in the Buchner funnel. Decant the liquid portion of the extracted sample (Section 16.1.3.2) through the tared glass fiber filter into a clean, dry, 500-ml filter flask. Rinse all the particulate matter remaining in the volumetric flask onto the glass fiber filter with water. Rinse the particulate matter with additional water. Transfer the filtrate to a 500-ml volumetric flask, and dilute to 500 ml with water. Dry the filter overnight at 105 ± 5° C (221 ± 9°F), cool in a desiccator, and weigh to the nearest 0.1 mg.

16.1.3.3.3 Dry a 250-ml beaker at 75 ± 5° C (167 ± 9° F), and cool in a desiccator; then weigh to constant weight to the nearest 0.1 mg. Pipette 200 ml of the filtrate that was saved into a tared 250-ml beaker; add five drops of phenolphthalein indicator and sufficient concentrated ammonium hydroxide to turn the solution pink. Carefully evaporate the contents of the beaker to dryness at 75 ± 5° C (167 ± 9° F). Check for dryness every 30 minutes. Do not continue to bake the sample once it has dried. Cool the sample in a desiccator, and weigh to constant weight to the nearest 0.1 mg.

16.1.3.4 Sulfate Analysis. Adjust the flow rate through the ion exchange column to 3 ml/min. Pipette a 20-ml aliquot of the filtrate onto the top of the ion exchange column, and collect the eluate in a 50-ml volumetric flask. Rinse the column with two 15-ml portions of water. Stop collection of the eluate when the volume in the flask reaches 50-ml. Pipette a 20-ml aliquot of the eluate into a 250-ml Erlenmeyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink end point using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger. Perform the ion exchange and titration procedures on duplicate portions of the filtrate. Results should agree within 5 percent. Regenerate or replace the ion exchange resin after 20 sample aliquots have been analyzed or if the end point of the titration becomes unclear.

Note: Protect the 0.0100 N barium perchlorate solution from evaporation at all times.

16.1.3.5 Blank Determination. Begin with a sample of water of the same volume as the samples being processed and carry it through the analysis steps described in Sections 16.1.3.3 and 16.1.3.4. A blank value larger than 5 mg should not be subtracted from the final particulate matter mass. Causes for large blank values should be investigated and any problems resolved before proceeding with further analyses.

16.1.4 Calibration. Calibrate the barium perchlorate solutions as in Method 6, Section 10.5.

16.1.5 Calculations.

16.1.5.1 Nomenclature. Same as Section 12.1 with the following additions:

m_a = Mass of clean analytical filter, mg.
m_d = Mass of dissolved particulate matter, mg.
m_c = Mass of beaker and dissolved particulate matter after evaporation of filtrate, mg.
m_p = Mass of insoluble particulate matter, mg.
m_r = Mass of analytical filter, sample filter, and insoluble particulate matter, mg.
m_{bk} = Mass of nonsulfate particulate matter in blank sample, mg.
m_n = Mass of nonsulfate particulate matter, mg.
m_s = Mass of Ammonium sulfate, mg.
N = Normality of $Ba(ClO_4)$ titrant, meq/ml.
V_a = Volume of aliquot taken for titration, 20 ml.
V_c = Volume of titrant used for titration blank, ml.
V_d = Volume of filtrate evaporated, 200 ml.
V_c = Volume of eluate collected, 50 ml.
V_f = Volume of extracted sample, 500 ml.
V_i = Volume of filtrate added to ion exchange column, 20 ml.
V_t = Volume of $Ba(C10_4)_2$ titrant, ml.
W = Equivalent weight of ammonium sulfate, 66.07 mg/meq.

16.1.5.2 Mass of Insoluble Particulate Matter.

$$m_p = m_r - m_a - m_f \qquad \text{Eq. 5F-4}$$

16.1.5.3 Mass of Dissolved Particulate Matter.

$$m_d = \left(m_e - \left(V_f/V_d\right)m_b\right) \qquad \text{Eq. 5F-5}$$

16.1.5.4 Mass of Ammonium Sulfate.

$$m_s = \frac{\left(V_t - V_c\right) N\, W\, V_e V_f}{V_a V_i} \qquad \text{Eq. 5F-6}$$

16.1.5.5 Mass of Nonsulfate Particulate Matter.

$$m_n = m_p + m_d - m_s - m_{bk} \qquad \text{Eq. 5F-7}$$

17.0 References

Same as Method 5, Section 17.0, with the addition of the following:

1. Mulik, J.D. and E. Sawicki. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 2, 1979.
2. Sawicki, E., J.D. Mulik, and E. Wittgenstein. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 1. 1978.
3. Siemer, D.D. Separation of Chloride and Bromide from Complex Matrices Prior to Ion Chromatographic Determination. Analytical Chemistry 52(12): 1874–1877. October 1980.
4. Small, H., T.S. Stevens, and W.C. Bauman. Novel Ion Exchange Chromatographic Method Using Conductimetric Determination. Analytical Chemistry. *47*(11):1801. 1975.

18.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

Method 5G—Determination of Particulate Matter Emissions From Wood Heaters (Dilution Tunnel Sampling Location)

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 4, Method 5, Method 5H, and Method 28.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from wood heaters.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 The exhaust from a wood heater is collected with a total collection hood, and is combined with ambient dilution air. Particulate matter is withdrawn proportionally from a single point in a sampling tunnel, and is collected on two glass fiber filters in series. The filters are maintained at a temperature of no greater than 32 °C (90 °F). The particulate mass is determined gravimetrically after the removal of uncombined water.

2.2 There are three sampling train approaches described in this method: (1) One dual-filter dry sampling train operated at about 0.015 m^3/min (0.5 cfm), (2) One dual-filter plus impingers sampling train operated at about 0.015 m^3/min (0.5 cfm), and (3) two dual-filter dry sampling trains operated simultaneously at any flow rate. Options

(2) and (3) are referenced in Section 16.0 of this method. The dual-filter dry sampling train equipment and operation, option (1), are described in detail in this method.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. The sampling train configuration is shown in Figure 5G–1 and consists of the following components:

6.1.1.1 Probe. Stainless steel (*e.g.*, 316 or grade more corrosion resistant) or glass about 9.5 mm (3/8 in.) I.D., 0.6 m (24 in.) in length. If made of stainless steel, the probe shall be constructed from seamless tubing.

6.1.1.2 Pitot Tube. Type S, as described in Section 6.1 of Method 2. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Method 2, Section 10. Alternatively, a standard pitot may be used as described in Method 2, Section 6.1.2.

6.1.1.3 Differential Pressure Gauge. Inclined manometer or equivalent device, as described in Method 2, Section 6.2. One manometer shall be used for velocity head (Δp) readings and another (optional) for orifice differential pressure readings (ΔH).

6.1.1.4 Filter Holders. Two each made of borosilicate glass, stainless steel, or Teflon, with a glass frit or stainless steel filter support and a silicone rubber, Teflon, or Viton gasket. The holder design shall provide a positive seal against leakage from the outside or around the filters. The filter holders shall be placed in series with the backup filter holder located 25 to 100 mm (1 to 4 in.) downstream from the primary filter holder. The filter holder shall be capable of holding a filter with a 100 mm (4 in.) diameter, except as noted in Section 16.

6.1.1.5 Filter Temperature Monitoring System. A temperature sensor capable of measuring temperature to within ± 3 °C (± 5 °F). The sensor shall be installed at the exit side of the front filter holder so that the sensing tip of the temperature sensor is in direct contact with the sample gas or in a thermowell as shown in Figure 5G–1. The temperature sensor shall comply with the calibration specifications in Method 2, Section 10.3. Alternatively, the sensing tip of the temperature sensor may be installed at the inlet side of the front filter holder.

6.1.1.6 Dryer. Any system capable of removing water from the sample gas to less than 1.5 percent moisture (volume percent) prior to the metering system. The system shall include a temperature sensor for demonstrating that sample gas temperature exiting the dryer is less than 20 °C (68 °F).

6.1.1.7 Metering System. Same as Method 5, Section 6.1.1.9.

6.1.2 Barometer. Same as Method 5, Section 6.1.2.

6.1.3 Dilution Tunnel Gas Temperature Measurement. A temperature sensor capable of measuring temperature to within ± 3 °C (± 5 °F).

6.1.4 Dilution Tunnel. The dilution tunnel apparatus is shown in Figure 5G–2 and consists of the following components:

6.1.4.1 Hood. Constructed of steel with a minimum diameter of 0.3 m (1 ft) on the large end and a standard 0.15 to 0.3 m (0.5 to 1 ft) coupling capable of connecting to standard 0.15 to 0.3 m (0.5 to 1 ft) stove pipe on the small end.

6.1.4.2 90° Elbows. Steel 90° elbows, 0.15 to 0.3 m (0.5 to 1 ft) in diameter for connecting mixing duct, straight duct and optional damper assembly. There shall be at least two 90° elbows upstream of the sampling section (see Figure 5G–2).

6.1.4.3 Straight Duct. Steel, 0.15 to 0.3 m (0.5 to 1 ft) in diameter to provide the ducting for the dilution apparatus upstream of the sampling section. Steel duct, 0.15 m (0.5 ft) in diameter shall be used for the sampling section. In the sampling section, at least 1.2 m (4 ft) downstream of the elbow, shall be two holes (velocity traverse ports) at 90° to each other of sufficient size to allow entry of the pitot for traverse measurements. At least 1.2 m (4 ft) downstream of the velocity traverse ports, shall be one hole (sampling port) of sufficient size to allow entry of the sampling probe. Ducts of larger diameter may be used for the sampling section, provided the specifications for minimum gas velocity and the dilution rate range shown in Section 8 are maintained. The length of duct from the hood inlet to the sampling ports shall not exceed 9.1 m (30 ft).

6.1.4.4 Mixing Baffles. Steel semicircles (two) attached at 90° to the duct axis on opposite sides of the duct midway between the two elbows upstream of sampling section. The space between the baffles shall be about 0.3 m (1 ft).

6.1.4.5 Blower. Squirrel cage or other fan capable of extracting gas from the dilution tunnel of sufficient flow to maintain the velocity and dilution rate specifications in Section 8 and exhausting the gas to the atmosphere.

6.2 Sample Recovery. The following items are required for sample recovery: probe brushes, wash bottles, sample storage containers, petri dishes, and funnel. Same as Method 5, Sections 6.2.1 through 6.2.4, and 6.2.8, respectively.

6.3 Sample Analysis. The following items are required for sample analysis: glass weighing dishes, desiccator, analytical balance, beakers (250-ml or smaller), hygrometer, and temperature sensor. Same as Method 5, Sections 6.3.1 through 6.3.3 and 6.3.5 through 6.3.7, respectively.

7.0 Reagents and Standards

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters. Glass fiber filters with a minimum diameter of 100 mm (4 in.), without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. Gelman A/E 61631 has been found acceptable for this purpose.

7.1.2 Stopcock Grease. Same as Method 5, Section 7.1.5. 7.2 Sample Recovery. Acetone-reagent grade, same as Method 5, Section 7.2.

7.3 Sample Analysis. Two reagents are required for the sample analysis:

7.3.1 Acetone. Same as in Section 7.2.

7.3.2 Desiccant. Anhydrous calcium sulfate, calcium chloride, or silica gel, indicating type.

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Dilution Tunnel Assembly and Cleaning. A schematic of a dilution tunnel is shown in Figure 5G–2. The dilution tunnel dimensions and other features are described in Section 6.1.4. Assemble the dilution tunnel, sealing joints and seams to prevent air leakage. Clean the dilution tunnel with an appropriately sized wire chimney brush before each certification test.

8.2 Draft Determination. Prepare the wood heater as in Method 28, Section 6.2.1. Locate the dilution tunnel hood centrally over the wood heater stack exhaust. Operate the dilution tunnel blower at the flow rate to be used during the test run. Measure the draft imposed on the wood heater by the dilution tunnel (*i.e.*, the difference in draft measured with and without the dilution tunnel operating) as described in Method 28, Section 6.2.3. Adjust the distance between the top of the wood heater stack exhaust and the dilution tunnel hood so that the dilution tunnel induced draft is less than 1.25 Pa (0.005 in. H_2O). Have no fire in the wood heater, close the wood heater doors, and open fully the air supply controls during this check and adjustment.

8.3 Pretest Ignition. Same as Method 28, Section 8.7.

8.4 Smoke Capture. During the pretest ignition period, operate the dilution tunnel and visually monitor the wood heater stack exhaust. Operate the wood heater with the doors closed and determine that 100 percent of the exhaust gas is collected by the dilution tunnel hood. If less than 100 percent of the wood heater exhaust gas is collected, adjust the distance between the wood heater stack and the dilution tunnel hood until no visible exhaust gas is escaping. Stop the pretest ignition period, and repeat the draft determination procedure described in Section 8.2.

8.5 Velocity Measurements. During the pretest ignition period, conduct a velocity traverse to identify the point of average velocity. This single point shall be used for measuring velocity during the test run.

8.5.1 Velocity Traverse. Measure the diameter of the duct at the velocity traverse port location through both ports. Calculate the duct area using the average of the two diameters. A pretest leak-check of pitot lines as in Method 2, Section 8.1, is recommended. Place the calibrated pitot tube at the centroid of the stack in either of the velocity traverse ports. Adjust the damper or similar device on the blower inlet until the velocity indicated by the pitot is approximately 220 m/min (720 ft/min). Continue to read the Δp and temperature until the velocity has remained constant (less than 5 percent change) for 1 minute. Once a constant velocity is obtained at the centroid of the duct, perform a velocity traverse as outlined in Method 2, Section 8.3 using four points per traverse as outlined in Method 1. Measure the Δp and tunnel temperature at each traverse point and record the readings. Calculate the total gas flow rate using calculations contained in Method 2, Section 12. Verify that the flow rate is 4 ± 0.40 dscm/min (140 ± 14 dscf/min); if not, readjust the damper, and repeat the velocity traverse. The moisture may be assumed to be 4 percent (100 percent relative humidity at 85 °F). Direct moisture measurements (*e.g.*, according to Method 4) are also permissible.

Note: If burn rates exceed 3 kg/hr (6.6 lb/hr), dilution tunnel duct flow rates greater than 4 dscm/min (140 dscfm) and sampling section duct diameters larger than 150 mm (6 in.) are allowed. If larger ducts or flow rates are used, the sampling section velocity shall be at least 220 m/min (720 fpm). In order to ensure measurable particulate mass catch, it is recommended that the ratio of the average mass flow rate in the dilution tunnel to the average fuel burn rate be less than 150:1 if larger duct sizes or flow rates are used.

8.5.2 Testing Velocity Measurements. After obtaining velocity traverse results that meet the flow rate requirements, choose a point of average velocity and place the pitot and temperature sensor at that location in the duct. Alternatively, locate the pitot and the temperature sensor at the duct centroid and calculate a velocity correction factor for the centroidal position. Mount the pitot to ensure no movement during the test run and seal the port holes to prevent any air leakage. Align the pitot opening to be parallel with the duct axis at the measurement point. Check that this condition is maintained during the test run (about 30-minute intervals). Monitor the temperature and velocity during the pretest ignition period to ensure that the proper flow rate is maintained. Make adjustments to the dilution tunnel flow rate as necessary.

8.6 Pretest Preparation. Same as Method 5, Section 8.1.

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

Using a tweezer or clean disposable surgical gloves, place one labeled (identified) and weighed filter in each of the filter holders. Be sure that each filter is properly centered and that the gasket is properly placed so as to prevent the sample gas stream from circumventing the filter. Check each filter for tears after assembly is completed.

Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct. Set up the train as shown in Figure 5G–1.

8.8 Leak-Check Procedures.

8.8.1 Leak-Check of Metering System Shown in Figure 5G–1. That portion of the sampling train from the pump to the orifice meter shall be leak-checked prior to initial use and after each certification or audit test. Leakage after the pump will result in less volume being recorded than is actually sampled. Use the procedure described in Method 5, Section 8.4.1. Similar leak-checks shall be conducted for other types of metering systems (*i.e.*, without orifice meters).

8.8.2 Pretest Leak-Check. A pretest leak-check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the procedures outlined in Method 5, Section 8.4.2 should be used. A vacuum of 130 mm Hg (5 in. Hg) may be used instead of 380 mm Hg (15 in. Hg).

8.8.3 Post-Test Leak-Check. A leak-check of the sampling train is mandatory at the conclusion of each test run. The leak-check shall be performed in accordance with the procedures outlined in Method 5, Section 8.4.2. A vacuum of 130 mm Hg (5 in. Hg) or the highest vacuum measured during the test run, whichever is greater, may be used instead of 380 mm Hg (15 in. Hg).

8.9 Preliminary Determinations. Determine the pressure, temperature and the average velocity of the tunnel gases as in Section 8.5. Moisture content of diluted tunnel gases is assumed to be 4 percent for making flow rate calculations; the moisture content may be measured directly as in Method 4.

8.10 Sampling Train Operation. Position the probe inlet at the stack centroid, and block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream. Be careful not to bump the probe into the stack wall when removing or inserting the probe through the porthole; this minimizes the chance of extracting deposited material.

8.10.1 Begin sampling at the start of the test run as defined in Method 28, Section 8.8.1. During the test run, maintain a sample flow rate proportional to the dilution tunnel flow rate (within 10 percent of the initial proportionality ratio) and a filter holder temperature of no greater than 32 °C (90 °F). The initial sample flow rate shall be approximately 0.015 m^3/min (0.5 cfm).

8.10.2 For each test run, record the data required on a data sheet such as the one shown in Figure 5G–3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment and when sampling is halted. Take other readings as indicated on Figure 5G–3 at least once each 10 minutes during the test run. Since the manometer level and zero may drift because of vibrations and temperature changes, make periodic checks during the test run.

8.10.3 For the purposes of proportional sampling rate determinations, data from calibrated flow rate devices, such as glass rotameters, may be used in lieu of incremental dry gas meter readings. Proportional rate calculation procedures must be revised, but acceptability limits remain the same.

8.10.4 During the test run, make periodic adjustments to keep the temperature between (or upstream of) the filters at the proper level. Do not change sampling trains during the test run.

8.10.5 At the end of the test run (see Method 28, Section 6.4.6), turn off the coarse adjust valve, remove the probe from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 8.8.2. Also, leak-check the pitot lines as described in Method 2, Section 8.1; the lines must pass this leak-check in order to validate the velocity head data.

8.11 Calculation of Proportional Sampling Rate. Calculate percent proportionality (see Section 12.7) to determine whether the run was valid or another test run should be made.

8.12 Sample Recovery. Same as Method 5, Section 8.7, with the exception of the following:

8.12.1 An acetone blank volume of about 50-ml or more may be used.

8.12.2 Treat the samples as follows:

8.12.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, Section 8.7.6.1. The filters may be stored either in a single container or in separate containers. Use the sum of the filter tare weights to determine the sample mass collected.

8.12.2.3 Container No. 2.

8.12.2.3.1 Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe and filter holders by washing and brushing these components with acetone and placing the wash in a labeled glass container. At least three cycles of brushing and rinsing are required.

8.12.2.3.2 Between sampling runs, keep brushes clean and protected from contamination.

8.12.2.3.3 After all acetone washings and particulate matter have been collected in the sample containers, tighten the lids on the sample containers so that the acetone will not leak out when transferred to the laboratory weighing area. Mark the height of the fluid levels to determine whether leakage occurs during transport. Label the containers clearly to identify contents.

8.13 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

Note: Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.8, 10.1–10.4	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.5	Analytical balance calibration	Ensure accurate and precise measurement of collected particulate.
16.2.5	Simultaneous, dual-train sample collection	Ensure precision of measured particulate concentration.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory record of all calibrations.

10.1 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Method 2, Section 10.1, prior to the first certification test and checked semiannually, thereafter. A standard pitot need not be calibrated but shall be inspected and cleaned, if necessary, prior to each certification test.

10.2 Volume Metering System.

10.2.1 Initial and Periodic Calibration. Before its initial use and at least semiannually thereafter, calibrate the volume metering system as described in Method 5, Section 10.3.1, except that the wet test meter with a capacity of 3.0 liters/rev (0.1 ft^3/rev) may be used. Other liquid displacement systems accurate to within ±1 percent, may be used as calibration standards.

Note: Procedures and equipment specified in Method 5, Section 16.0, for alternative calibration standards, including calibrated dry gas meters and critical orifices, are allowed for calibrating the dry gas meter in the sampling train. A dry gas meter used as a calibration standard shall be recalibrated at least once annually.

10.2.2 Calibration After Use. After each certification or audit test (four or more test runs conducted on a wood heater at the four burn rates specified in Method 28), check calibration of the metering system by performing three calibration runs at a single, intermediate flow rate as described in Method 5, Section 10.3.2.

Note: Procedures and equipment specified in Method 5, Section 16.0, for alternative calibration standards are allowed for the post-test dry gas meter calibration check.

10.2.3 Acceptable Variation in Calibration. If the dry gas meter coefficient values obtained before and after a certification test differ by more than 5 percent, the certification test shall either be voided and repeated, or calculations for the certification test shall be performed using whichever meter coefficient value (*i.e.*, before or after) gives the lower value of total sample volume.

10.3 Temperature Sensors. Use the procedure in Method 2, Section 10.3, to calibrate temperature sensors before the first certification or audit test and at least semiannually, thereafter.

10.4 Barometer. Calibrate against a mercury barometer before the first certification test and at least semiannually, thereafter. If a mercury barometer is used, no calibration is necessary. Follow the manufacturer's instructions for operation.

10.5 Analytical Balance. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first certification test and semiannually, thereafter. Before each certification test, audit the balance by weighing at least one calibration weight (class F) that corresponds to 50 to 150 percent of the weight of one filter. If the scale cannot reproduce the value of the calibration weight to within 0.1 mg, conduct the multipoint calibration before use.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5G–4. Use the same analytical balance for determining tare weights and final sample weights.

11.2 Handle each sample container as follows:

11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, Section 11.2.1.

11.2.2 Container No. 2. Same as Method 5, Section 11.2.2, except that the beaker may be smaller than 250 ml.

11.2.3 Acetone Blank Container. Same as Method 5, Section 11.2.4, except that the beaker may be smaller than 250 ml.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

B_{ws} = Water vapor in the gas stream, proportion by volume (assumed to be 0.04).

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

E = Particulate emission rate, g/hr (lb/hr).

E_{adj} = Adjusted particulate emission rate, g/hr (lb/hr).

L_a = Maximum acceptable leakage rate for either a pretest or post-test leak-check, equal to 0.00057 m^3/min (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

m_a = Mass of residue of acetone blank after evaporation, mg.

m_{aw} = Mass of residue from acetone wash after evaporation, mg.

m_n = Total amount of particulate matter collected, mg.

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

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

PR = Percent of proportional sampling rate.

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

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

Q_{sd} = Average gas flow rate in dilution tunnel, calculated as in Method 2, Equation 2–8, dscm/hr (dscf/hr).

T_m = Absolute average dry gas meter temperature (see Figure 5G–3), °K (°R).

T_{mi} = Absolute average dry gas meter temperature during each 10-minute interval, i, of the test run, °K (°R).

T_s = Absolute average gas temperature in the dilution tunnel (see Figure 5G–3), °K (°R).

T_{si} = Absolute average gas temperature in the dilution tunnel during each 10 minute interval, i, of the test run, °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_a = Volume of acetone blank, ml.

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

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

V_{mi} = Volume of gas sample as measured by dry gas meter during each 10-minute interval, i, of the test run, dcm.

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

V_s = Average gas velocity in the dilution tunnel, calculated by Method 2, Equation 2–7, m/sec (ft/sec). The dilution tunnel dry gas molecular weight may be assumed to be 29 g/g mole (lb/lb mole).

V_{si} = Average gas velocity in dilution tunnel during each 10-minute interval, i, of the test run, calculated by Method 2, Equation 2–7, m/sec (ft/sec).

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter, if used (see Figure 5G–2), mm H^2O (in. H^2O).

U = Total sampling time, min.

10 = 10 minutes, length of first sampling period.

13.6 = Specific gravity of mercury.

100 = Conversion to percent.

12.2 Dry Gas Volume. Same as Method 5, Section 12.2, except that component changes are not allowable.

12.3 Solvent Wash Blank.

$$m_{aw} = \frac{m_a V_{aw}}{V_a} \qquad \text{Eq. 5G-1}$$

12.4 Total Particulate Weight. Determine the total particulate catch, mn, from the sum of the weights obtained from Container Nos. 1, 1A, and 2, less the acetone blank (see Figure 5G–4).

12.5 Particulate Concentration.

$$c_s = K_2 \frac{m_n}{V_{m(std)}} \qquad \text{Eq. 5G-2}$$

Where:

K_2 = 0.001 g/mg for metric units.

= 0.0154 gr/mg for English units.

12.6 Particulate Emission Rate.

$$E = C_s Q_{sd} \qquad \text{Eq. 5G-3}$$

Note: Particulate emission rate results produced using the sampling train described in Section 6 and shown in Figure 5G–1 shall be adjusted for reporting purposes by the following method adjustment factor:

$$E_{adj} = K_3 E^{0.83} \qquad \text{Eq. 5G-4}$$

Where:

K_3 = constant, 1.82 for metric units.

= constant, 0.643 for English units.

12.7 Proportional Rate Variation. Calculate PR for each 10-minute interval, i, of the test run.

$$PR = \left(\frac{\theta \left(V_{mi} V_s \, T_m T_{si} \right)}{10 \left(V_m \, V_{si} T_s T_{mi} \right)} \right) \times 100 \qquad \text{Eq. 5G-5}$$

Alternate calculation procedures for proportional rate variation may be used if other sample flow rate data (e.g., orifice flow meters or rotameters) are monitored to maintain proportional sampling rates. The proportional rate variations shall be calculated for each 10-minute interval by comparing the stack to nozzle velocity ratio for each 10-minute interval to the average stack to nozzle velocity ratio for the test run. Proportional rate variation may be calculated for intervals shorter than 10 minutes with appropriate revisions to Equation 5G–5. If no more than 10 percent of the PR values for all the intervals exceed 90 percent ≤ PR ≤ 110 percent, and if no PR value for any interval exceeds 80 percent ≤ PR ≤ 120 percent, the results are acceptable. If the PR values for the test run are judged to be unacceptable, report the test run emission results, but do not include the results in calculating the weighted average emission rate, and repeat the test run.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Method 5H Sampling Train. The sampling train and sample collection, recovery, and analysis procedures described in Method 5H, Sections 6.1.1, 7.1, 7.2, 8.1, 8.10, 8.11, and 11.0, respectively, may be used in lieu of similar sections in Method 5G.

Operation of the Method 5H sampling train in the dilution tunnel is as described in Section 8.10 of this method. Filter temperatures and condenser conditions are as described in Method 5H. No adjustment to the measured particulate matter emission rate (Equation 5G–4, Section 12.6) is to be applied to the particulate emission rate measured by this alternative method.

16.2 Dual Sampling Trains. Two sampling trains may be operated simultaneously at sample flow rates other than that specified in Section 8.10, provided that the following specifications are met.

16.2.1 Sampling Train. The sampling train configuration shall be the same as specified in Section 6.1.1, except the probe, filter, and filter holder need not be the same sizes as specified in the applicable sections. Filter holders of plastic materials such as Nalgene or polycarbonate materials may be used (the Gelman 1119 filter holder has been found suitable for this purpose). With such materials, it is recommended that solvents not be used in sample recovery. The filter face velocity shall not exceed 150 mm/sec (30 ft/min) during the test run. The dry gas meter shall be calibrated for the same flow rate range as encountered during the test runs. Two separate, complete sampling trains are required for each test run.

16.2.2 Probe Location. Locate the two probes in the dilution tunnel at the same level (see Section 6.1.4.3). Two sample ports are necessary. Locate the probe inlets within the 50 mm (2 in.) diameter centroidal area of the dilution tunnel no closer than 25 mm (1 in.) apart.

16.2.3 Sampling Train Operation. Operate the sampling trains as specified in Section 8.10, maintaining proportional sampling rates and starting and stopping the two sampling trains simultaneously. The pitot values as described in Section 8.5.2 shall be used to adjust sampling rates in both sampling trains.

16.2.4 Recovery and Analysis of Sample. Recover and analyze the samples from the two sampling trains separately, as specified in Sections 8.12 and 11.0, respectively.

16.2.4.1 For this alternative procedure, the probe and filter holder assembly may be weighed without sample recovery (use no solvents) described above in order to determine the sample weight gains. For this approach, weigh the clean, dry probe and filter holder assembly upstream of the front filter (without filters) to the nearest 0.1 mg to establish the tare weights. The filter holder section between the front and second filter need not be weighed. At the end of the test run, carefully clean the outside of the probe, cap the ends, and identify the sample (label). Remove the filters from the filter holder assemblies as described for container Nos. 1 and 1A in Section 8.12.2.1. Reassemble the filter holder assembly, cap the ends, identify the sample (label), and transfer all the samples to the laboratory weighing area for final weighing. Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

16.2.4.2 For this alternative procedure, filters may be weighed directly without a petri dish. If the probe and filter holder assembly are to be weighed to determine the sample weight, rinse the probe with acetone to remove moisture before desiccating prior to the test run. Following the test run, transport the probe and filter holder to the desiccator, and uncap the openings of the probe and the filter holder assembly. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

16.2.5 Calculations. Calculate an emission rate (Section 12.6) for the sample from each sampling train separately and determine the average emission rate for the two values. The two emission rates shall not differ by more than 7.5 percent from the average emission rate, or 7.5 percent of the weighted average emission rate limit in the applicable subpart of the regulations, whichever is greater. If this specification is not met, the results are unacceptable. Report the results, but do not include the results in calculating the weighted average emission rate. Repeat the test run until acceptable results are achieved, report the average emission rate for the acceptable test run, and use the average in calculating the weighted average emission rate.

17.0 References

Same as Method 5, Section 17.0, References 1 through 11, with the addition of the following:

1. Oregon Department of Environmental Quality. Standard Method for Measuring the Emissions and Efficiencies of Woodstoves. June 8, 1984. Pursuant to Oregon Administrative Rules Chapter 340, Division 21.

2. American Society for Testing and Materials. Proposed Test Methods for Heating Performance and Emissions of Residential Wood-fired Closed Combustion-Chamber Heating Appliances. E–6 Proposal P 180. August 1986.

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18.0 Tables, Diagrams, Flowcharts, and Validation Data

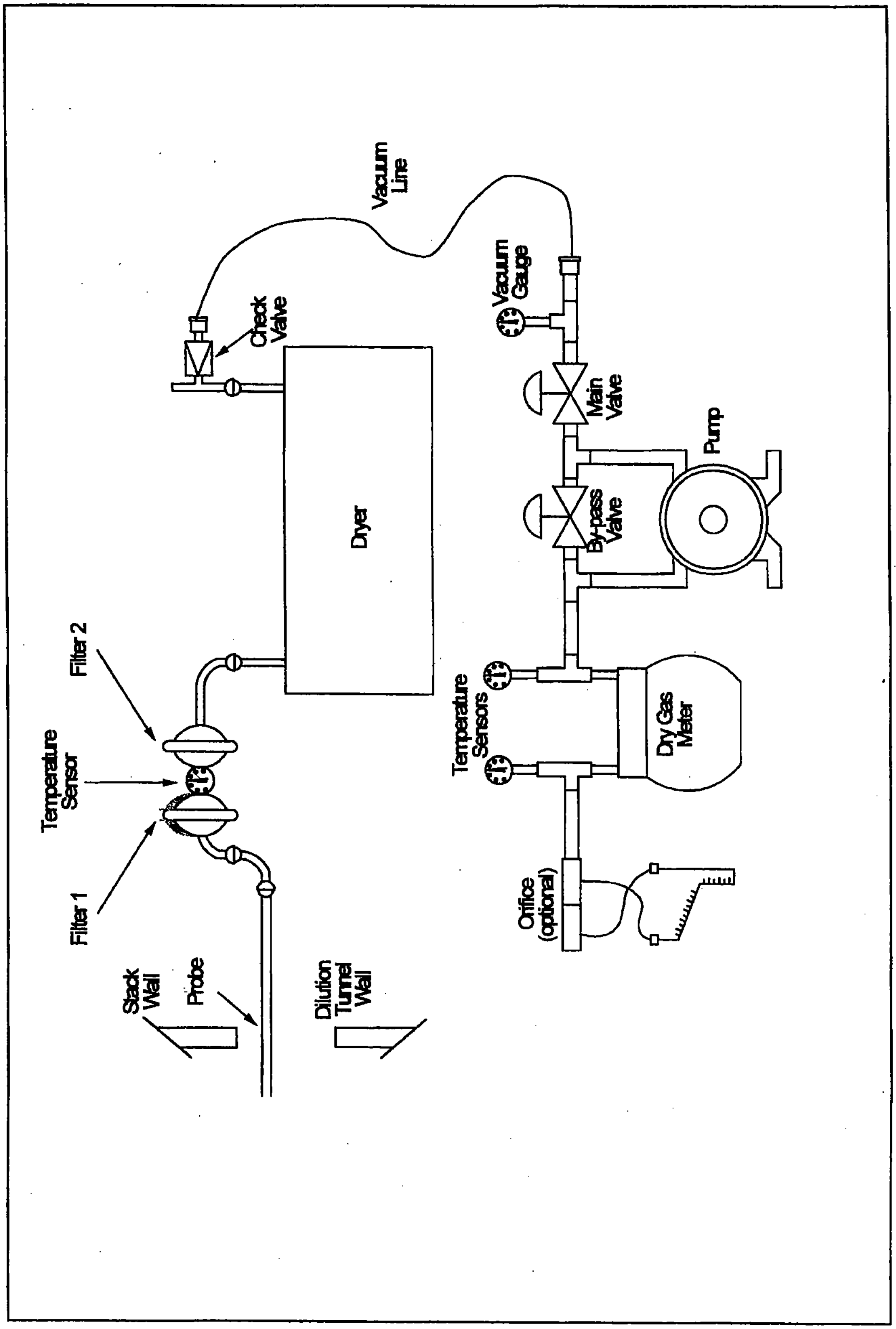


Figure 5G-1. Method 5G Sampling Train.

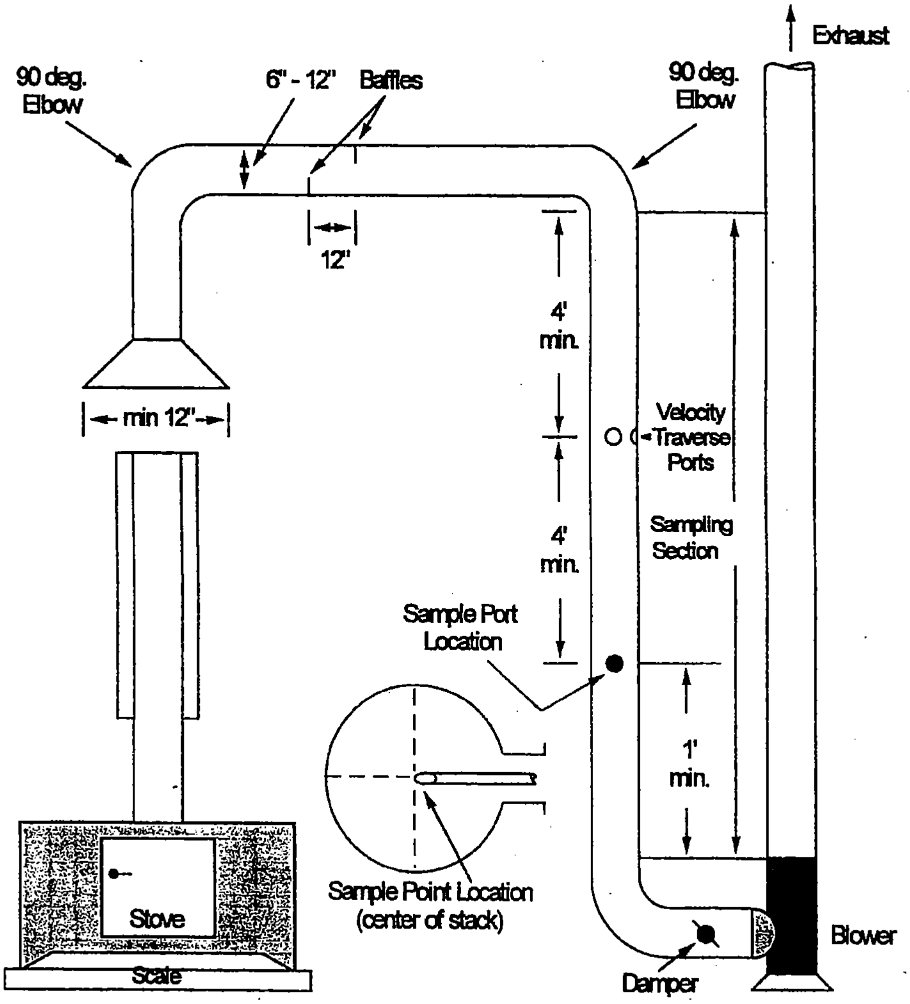


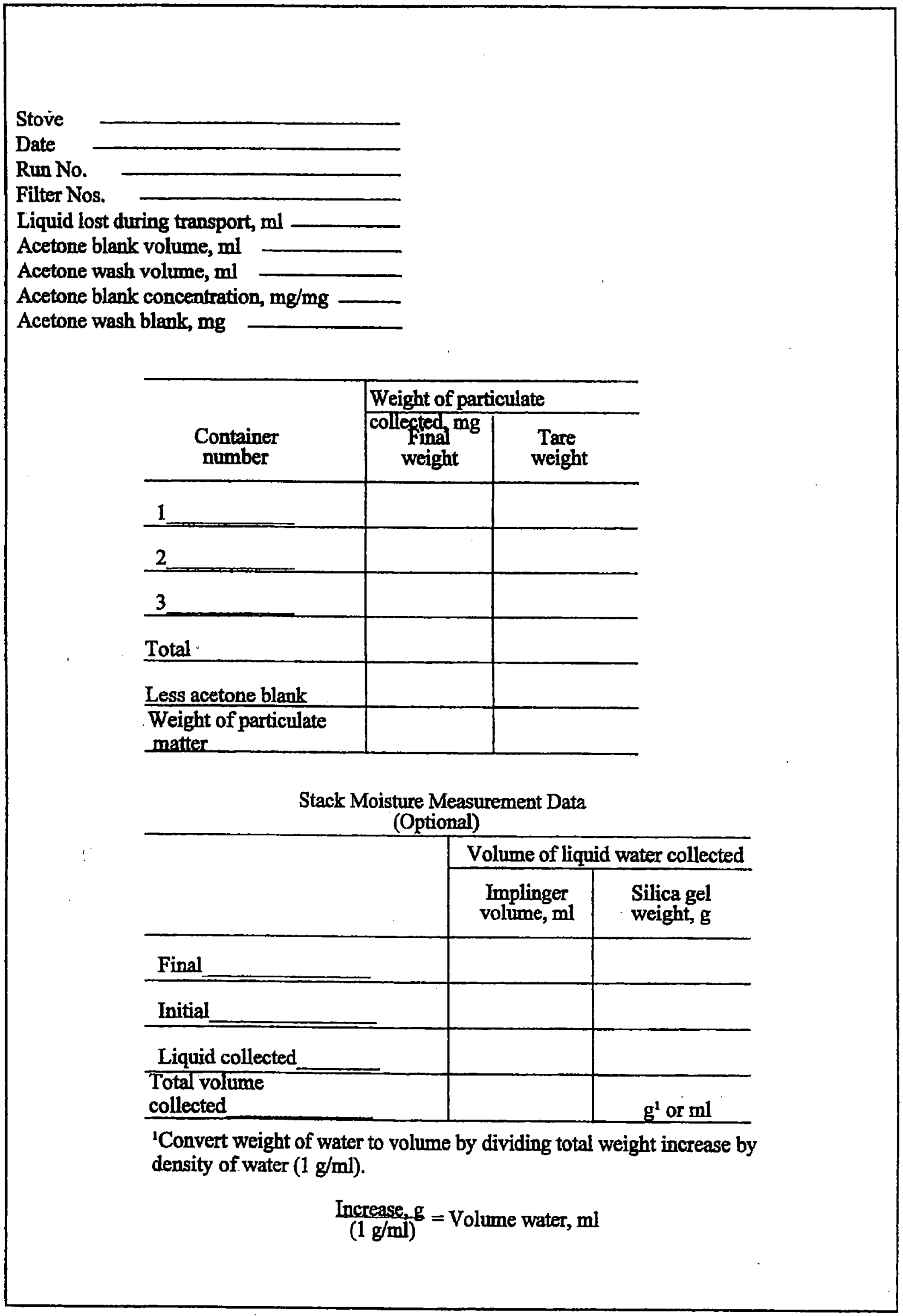
Figure 5G-2. Suggested Construction Details of the Dilution Tunnel.

Stove ____________
Test Method ____________
Operator ____________
Date ____________
Run No. ____________
Start Time ____________
Stop Time ____________
Sample Box No. ____________
Meter Box No. ____________
Meter ΔH @ (optional) ____________

Pitot tube coefficient, C_p ____________
Room temperature, °C (°F) ____________
Barometric pressure mb (in. Hg) ____________
Measured or assumed moisture, % ____________
Final leak rate, m^3/min, (cfm) ____________
Probe liner material ____________
Draft or static pressure, mm H_2O (in. H_2O) ____________
Filter No. ____________

Clock time	Test run time (e) min.	Vacuum mm Hg (in. Hg)	Tunnel temp (T_S) °C (°F)	Velocity head (ΔP_S) mm (in. H_2O)	Sample flow rate indicator (orifice meter optional) mm H_2O (in. H_2O)	Gas meter volume m^3 (ft^3)	Gas sample temp at dry gas meter		Filter holder temp °C (°F)	Temperature of gas leaving dryer or last impinger °C (°F)
							Inlet °C (°F)	Outlet °C (°F)		
Total							Avg.	Avg.		
Average							Avg.			

Figure 5G-3. Sampling Data Sheet.



Stove ____________________

Date ____________________

Run No. ____________________

Filter Nos. ____________________

Liquid lost during transport, ml __________

Acetone blank volume, ml __________

Acetone wash volume, ml __________

Acetone blank concentration, mg/mg ______

Acetone wash blank, mg __________

Container number	Weight of particulate collected, mg	
	Final weight	Tare weight
1		
2		
3		
Total		
Less acetone blank		
Weight of particulate matter		

Stack Moisture Measurement Data
(Optional)

	Volume of liquid water collected	
	Implinger volume, ml	Silica gel weight, g
Final		
Initial		
Liquid collected		
Total volume collected		g[1] or ml

[1] Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{(1\text{ g/ml})} = \text{Volume water, ml}$$

Figure 5G-4. Analysis Data Sheet.

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Method 5H—Determination of Particulate Matter Emissions From Wood Heaters From a Stack Location

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 2, Method 3, Method 5, Method 5G, Method 6, Method 6C, Method 16A, and Method 28.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM and condensible emissions from wood heaters.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate matter is withdrawn proportionally from the wood heater exhaust and is collected on two glass fiber filters separated by impingers immersed in an ice water bath. The first filter is maintained at a temperature of no greater than 120 °C (248 °F). The second filter and the impinger system are cooled such that the temperature of the gas exiting the second filter is no greater than 20 °C (68 °F). The particulate mass collected in the probe, on the filters, and in the impingers is determined gravimetrically after the removal of uncombined water.

3.0 Definitions

Same as in Method 6C, Section 3.0.

4.0 Interferences. [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. The sampling train configuration is shown in Figure 5H–1. Same as Method 5, Section 6.1.1, with the exception of the following:

6.1.1.1 Probe Nozzle. The nozzle is optional; a straight sampling probe without a nozzle is an acceptable alternative.

6.1.1.2 Probe Liner. Same as Method 5, Section 6.1.1.2, except that the maximum length of the sample probe shall be 0.6 m (2 ft) and probe heating is optional.

6.1.1.3 Filter Holders. Two each of borosilicate glass, with a glass frit or stainless steel filter support and a silicone rubber, Teflon, or Viton gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter. The front filter holder shall be attached immediately at the outlet of the probe and prior to the first impinger. The second filter holder shall be attached on the outlet of the third impinger and prior to the inlet of the fourth (silica gel) impinger.

6.1.2 Barometer. Same as Method 5, Section 6.2.

6.1.3 Stack Gas Flow Rate Measurement System. A schematic of an example test system is shown in Figure 5H–2. The flow rate measurement system consists of the following components:

6.1.3.1 Sample Probe. A glass or stainless steel sampling probe.

6.1.3.2 Gas Conditioning System. A high density filter to remove particulate matter and a condenser capable of lowering the dew point of the gas to less than 5 °C (40 °F). Desiccant, such as Drierite, may be used to dry the sample gas. Do not use silica gel.

6.1.3.3 Pump. An inert (*e.g.*, Teflon or stainless steel heads) sampling pump capable of delivering more than the total amount of sample required in the manufacturer's instructions for the individual instruments. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (*e.g.*, precision rotameter, pressure gauge downstream of all flow controls) shall be provided at the analyzer. The requirements for measuring and controlling the analyzer flow rate are not applicable if data are presented that demonstrate that the analyzer is insensitive to flow variations over the range encountered during the test.

6.1.3.4 Carbon Monoxide (CO) Analyzer. Any analyzer capable of providing a measure of CO in the range of 0 to 10 percent by volume at least once every 10 minutes.

6.1.3.5 Carbon Dioxide (CO_2) Analyzer. Any analyzer capable of providing a measure of CO_2 in the range of 0 to 25 percent by volume at least once every 10 minutes.

Note: Analyzers with ranges less than those specified above may be used provided actual concentrations do not exceed the range of the analyzer.

6.1.3.6 Manifold. A sampling tube capable of delivering the sample gas to two analyzers and handling an excess of the total amount used by the analyzers. The excess gas is exhausted through a separate port.

6.1.3.7 Recorders (optional). To provide a permanent record of the analyzer outputs.

6.1.4 Proportional Gas Flow Rate System. To monitor stack flow rate changes and provide a measurement that can be used to adjust and maintain particulate sampling flow rates proportional to the stack gas flow rate. A schematic of the proportional flow rate system is shown in Figure 5H–2 and consists of the following components:

6.1.4.1 Tracer Gas Injection System. To inject a known concentration of sulfur dioxide (SO_2) into the flue. The tracer gas injection system consists of a cylinder of SO_2, a gas cylinder regulator, a stainless steel needle valve or flow controller, a nonreactive (stainless steel and glass) rotameter, and an injection loop to disperse the SO_2 evenly in the flue.

6.1.4.2 Sample Probe. A glass or stainless steel sampling probe.

6.1.4.3 Gas Conditioning System. A combustor as described in Method 16A, Sections 6.1.5 and 6.1.6, followed by a high density filter to remove particulate matter, and a condenser capable of lowering the dew point of the gas to less than 5 °C (40 °F). Desiccant, such as Drierite, may be used to dry the sample gas. Do not use silica gel.

6.1.4.4 Pump. Same as described in Section 6.1.3.3.

6.1.4.5 SO_2 Analyzer. Any analyzer capable of providing a measure of the SO_2 concentration in the range of 0 to 1,000 ppm by volume (or other range necessary to measure the SO_2 concentration) at least once every 10 minutes.

6.1.4.6 Recorder (optional). To provide a permanent record of the analyzer outputs.

Note: Other tracer gas systems, including helium gas systems, are acceptable for determination of instantaneous proportional sampling rates.

6.2 Sample Recovery. Same as Method 5, Section 6.2.

6.3 Sample Analysis. Same as Method 5, Section 6.3, with the addition of the following:

6.3.1 Separatory Funnel. Glass or Teflon, 500-ml or greater.

7.0 Reagents and Standards

7.1 Sample Collection. Same as Method 5, Section 7.1, including deionized distilled water.

7.2 Sample Recovery. Same as Method 5, Section 7.2.

7.3 Sample Analysis. The following reagents and standards are required for sample analysis:

7.3.1 Acetone. Same as Method 5 Section 7.2.

7.3.2 Dichloromethane (Methylene Chloride). Reagent grade, <0.001 percent residue in glass bottles.

7.3.3 Desiccant. Anhydrous calcium sulfate, calcium chloride, or silica gel, indicating type.

7.3.4 Cylinder Gases. For the purposes of this procedure, span value is defined as the upper limit of the range specified for each analyzer as described in Section 6.1.3.4 or 6.1.3.5. If an analyzer with a range different from that specified in this method is used, the span value shall be equal to the upper limit of the range for the analyzer used (see **Note** in Section 6.1.3.5).

7.3.4.1 Calibration Gases. The calibration gases for the CO_2, CO, and SO_2 analyzers shall be CO_2 in nitrogen (N_2), CO in N_2, and SO_2 in N_2, respectively. CO_2 and CO calibration gases may be combined in a single cylinder. Use three calibration gases as specified in Method 6C, Sections 7.2.1 through 7.2.3.

7.3.4.2 SO_2 Injection Gas. A known concentration of SO_2 in N_2. The concentration must be at least 2 percent SO_2 with a maximum of 100 percent SO_2.

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Pretest Preparation. Same as Method 5, Section 8.1.

8.2 Calibration Gas and SO_2 Injection Gas Concentration Verification, Sampling System Bias Check, Response Time Test, and Zero and Calibration Drift Tests. Same as Method 6C, Sections 8.2.1, 8.2.3, 8.2.4, and 8.5, respectively, except that for verification of CO and CO_2 gas concentrations, substitute Method 3 for Method 6.

8.3 Preliminary Determinations.

8.3.1 Sampling Location. The sampling location for the particulate sampling probe shall be 2.45 ± 0.15 m (8 ± 0.5 ft) above the platform upon which the wood heater is placed (*i.e.*, the top of the scale).

8.3.2 Sampling Probe and Nozzle. Select a nozzle, if used, sized for the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain proportional sampling rates. During the run, do not change the nozzle size. Select a suitable probe liner and probe length to effect minimum blockage.

8.4 Preparation of Particulate Sampling Train. Same as Method 5, Section 8.3, with the exception of the following:

8.4.1 The train should be assembled as shown in Figure 5H–1.

8.4.2 A glass cyclone may not be used between the probe and filter holder.

8.5 Leak-Check Procedures.

8.5.1 Leak-Check of Metering System Shown in Figure 5H–1. That portion of the sampling train from the pump to the orifice meter shall be leak-checked after each certification or audit test. Use the procedure described in Method 5, Section 8.4.1.

8.5.2 Pretest Leak-Check. A pretest leak-check of the sampling train is recommended, but not required. If the pretest leak-check is conducted, the procedures outlined in Method 5, Section 8.5.2 should be used. A vacuum of 130 mm Hg (5 in. Hg) may be used instead of 380 mm Hg (15 in. Hg).

8.5.2 Leak-Checks During Sample Run. If, during the sampling run, a component (*e.g.*, filter assembly or impinger) change becomes necessary, conduct a leak-check as described in Method 5, Section 8.4.3.

8.5.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be performed in accordance with the procedures outlined in Method 5, Section 8.4.4, except that a vacuum of 130 mm Hg (5 in. Hg) or the greatest vacuum measured during the test run, whichever is greater, may be used instead of 380 mm Hg (15 in. Hg).

8.6 Tracer Gas Procedure. A schematic of the tracer gas injection and sampling systems is shown in Figure 5H–2.

8.6.1 SO_2 Injection Probe. Install the SO_2 injection probe and dispersion loop in the stack at a location 2.9 ± 0.15 m (9.5 ± 0.5 ft) above the sampling platform.

8.6.2 SO_2 Sampling Probe. Install the SO_2 sampling probe at the centroid of the stack at a location 4.1 ± 0.15 m (13.5 ± 0.5 ft) above the sampling platform.

8.7 Flow Rate Measurement System. A schematic of the flow rate measurement system is shown in Figure 5H–2. Locate the flow rate measurement sampling probe at the centroid of the stack at a location 2.3 ± 0.3 m (7.5 ± 1 ft) above the sampling platform.

8.8 Tracer Gas Procedure. Within 1 minute after closing the wood heater door at the start of the test run (as defined in Method 28, Section 8.8.1), meter a known concentration of SO_2 tracer gas at a constant flow rate into the wood heater stack. Monitor the SO_2 concentration in the stack, and record the SO_2 concentrations at 10-minute intervals or more often. Adjust the particulate sampling flow rate proportionally to the SO_2 concentration changes using Equation 5H–6 (*e.g.*, the SO_2 concentration at the first 10-minute reading is measured to be 100 ppm; the next 10 minute SO_2 concentration is measured to be 75 ppm: the particulate sample flow rate is adjusted from the initial 0.15 cfm to 0.20 cfm). A check for proportional rate variation shall be made at the completion of the test run using Equation 5H–10.

8.9 Volumetric Flow Rate Procedure. Apply stoichiometric relationships to the wood combustion process in determining the exhaust gas flow rate as follows:

8.9.1 Test Fuel Charge Weight. Record the test fuel charge weight (wet) as specified in Method 28, Section 8.8.2. The wood is assumed to have the following weight percent composition: 51 percent carbon, 7.3 percent hydrogen, 41 percent oxygen. Record the wood moisture for each fuel charge as described in Method 28, Section 8.6.5. The ash is assumed to have negligible effect on associated C, H, and O concentrations after the test burn.

8.9.2 Measured Values. Record the CO and CO_2 concentrations in the stack on a dry basis every 10 minutes during the test run or more often. Average these values for the test run. Use as a mole fraction (*e.g.*, 10 percent CO_2 is recorded as 0.10) in the calculations to express total flow (see Equation 5H–6).

8.10 Sampling Train Operation.

8.10.1 For each run, record the data required on a data sheet such as the one shown in Figure 5H–3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings as indicated on Figure 5H–3 at least once each 10 minutes during the test run.

8.10.2 Remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the probe is properly positioned. Position the nozzle, if used, facing into gas stream, or the probe tip in the 50 mm (2 in.) centroidal area of the stack.

8.10.3 Be careful not to bump the probe tip into the stack wall when removing or inserting the probe through the porthole; this minimizes the chance of extracting deposited material.

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

8.10.5 Begin sampling at the start of the test run as defined in Method 28, Section 8.8.1, start the sample pump, and adjust the sample flow rate to between 0.003 and 0.014 m^3/min (0.1 and 0.5 cfm). Adjust the sample flow rate proportionally to the stack gas flow during the test run according to the procedures outlined in Section 8. Maintain a proportional sampling rate (within 10 percent of the desired value) and a filter holder temperature no greater than 120 °C (248 °F).

8.10.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level. Add more ice to the impinger box and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet.

8.10.7 If the pressure drop across the filter becomes too high, making proportional sampling difficult to maintain, either filter may be replaced during a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see Section 8.5.2). The total particulate weight shall include the summation of all filter assembly catches. The total time for changing sample train components shall not exceed 10 minutes. No more than one component change is allowed for any test run.

8.10.8 At the end of the test run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 8.5.3.

8.11 Sample Recovery. Same as Method 5, Section 8.7, with the exception of the following:

8.11.1 Blanks. The volume of the acetone blank may be about 50-ml, rather than 200-ml; a 200-ml water blank shall also be saved for analysis.

8.11.2 Samples.

8.11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, Section 8.7.6.1. The filters may be stored either in a single container or in separate containers.

8.11.2.2 Container No. 2. Same as Method 5, Section 8.7.6.2, except that the container should not be sealed until the impinger rinse solution is added (see Section 8.10.2.4).

8.11.2.3 Container No. 3. Treat the impingers as follows: Measure the liquid which is in the first three impingers to within 1-ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Transfer the water from the first, second, and third impingers to a glass container. Tighten the lid on the sample container so that water will not leak out.

8.11.2.4 Rinse impingers and graduated cylinder, if used, with acetone three times or more. Avoid direct contact between the acetone and any stopcock grease or collection of any stopcock grease in the rinse solutions. Add these rinse solutions to sample Container No. 2.

8.11.2.5 Container No. 4. Same as Method 5, Section 8.7.6.3

8.12 Sample Transport. Whenever possible, containers should be transferred in such a way that they remain upright at all times.

Note: Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.2	Sampling system bias check	Ensures that bias introduced by measurement system, minus analyzer, is no greater than 3 percent of span.
8.2	Analyzer zero and calibration drift tests	Ensures that bias introduced by drift in the measurement system output during the run is no greater than 3 percent of span.
8.5, 10.1, 12.13	Sampling equipment leak-check and calibration; proportional sampling rate verification.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1	Analytical balance calibration	Ensure accurate and precise measurement of collected particulate.
10.3	Analyzer calibration error check	Ensures that bias introduced by analyzer calibration error is no greater than 2 percent of span.

9.2 Volume Metering System Checks. Same as Method 5, Section 9.2.

10.0 Calibration and Standardization

Note: Maintain a laboratory record of all calibrations.

10.1 Volume Metering System, Temperature Sensors, Barometer, and Analytical Balance. Same as Method 5G, Sections 10.2 through 10.5, respectively.

10.2 SO_2 Injection Rotameter. Calibrate the SO_2 injection rotameter system with a soap film flowmeter or similar direct volume measuring device with an accuracy of 2 percent. Operate the rotameter at a single reading for at least three calibration runs for 10 minutes each. When three consecutive calibration flow rates agree within 5 percent, average the three flow rates, mark the rotameter at the calibrated setting, and use the calibration flow rate as the SO_2 injection flow rate during the test run. Repeat the rotameter calibration before the first certification test and semiannually thereafter.

10.3. Gas Analyzers. Same as Method 6C, Section 10.0.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5H–4.

11.2 Handle each sample container as follows:

11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, Section 11.2.1.

11.2.2 Container No. 2. Same as Method 5, Section 11.2.2, except that the beaker may be smaller than 250-ml.

11.2.3 Container No. 3. Note the level of liquid in the container and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Determination of sample leakage is not applicable if sample recovery and analysis occur in the same room. Measure the liquid in this container either volumetrically to within 1-ml or gravimetrically to within 0.5 g. Transfer the contents to a 500-ml or larger separatory funnel. Rinse the container with water, and add to the separatory funnel. Add 25-ml of dichloromethane to the separatory funnel, stopper and vigorously shake 1 minute, let separate and transfer the dichloromethane (lower layer) into a tared beaker or evaporating dish. Repeat twice more. It is necessary to rinse Container No. 3 with dichloromethane. This rinse is added to the impinger extract container. Transfer the remaining water from the separatory funnel to a tared beaker or evaporating dish and evaporate to dryness at 104 °C (220 °F). Desiccate and weigh to a constant weight. Evaporate the combined impinger water extracts at ambient temperature and pressure. Desiccate and weigh to a constant weight. Report both results to the nearest 0.1 mg.

11.2.4 Container No. 4. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

11.2.5 Acetone Blank Container. Same as Method 5, Section 11.2.4, except that the beaker may be smaller than 250 ml.

11.2.6 Dichloromethane Blank Container. Treat the same as the acetone blank.

11.2.7 Water Blank Container. Transfer the water to a tared 250 ml beaker and evaporate to dryness at 104 °C (220 °F). Desiccate and weigh to a constant weight.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

a = Sample flow rate adjustment factor.
BR = Dry wood burn rate, kg/hr (lb/hr), from Method 28, Section 8.3.
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
E = Particulate emission rate, g/hr (lb/hr).
ΔH = Average pressure differential across the orifice meter (see Figure 5H–1), mm H_2O (in. H_2O).
L_a = Maximum acceptable leakage rate for either a post-test leak-check or for a leak-check following a component change; equal to 0.00057 cmm (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.
L_l = Individual leakage rate observed during the leak-check conducted before a component change, cmm (cfm).
L_p = Leakage rate observed during the post-test leak-check, cmm (cfm).
m_n = Total amount of particulate matter collected, mg.
M_a = Mass of residue of solvent after evaporation, mg.
N_C = Grams of carbon/gram of dry fuel (lb/lb), equal to 0.0425.
N_T = Total dry moles of exhaust gas/kg of dry wood burned, g-moles/kg (lb-moles/lb).
PR = Percent of proportional sampling rate.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in.Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in.Hg).
Q_{sd} = Total gas flow rate, dscm/hr (dscf/hr).
S_l = Concentration measured at the SO_2 analyzer for the first 10-minute interval, ppm.
S_i = Concentration measured at the SO_2 analyzer for the "ith" 10 minute interval, ppm.
T_m = Absolute average dry gas meter temperature (see Figure 5H–3), °K (°R).
T_{std} = Standard absolute temperature, 293 °K (528 °R).
V_a = volume of solvent blank, ml.
V_{aw} = Volume of solvent used in wash, ml.
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5H–4), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{mi(std)}$ = Volume of gas sample measured by the dry gas meter during the "ith" 10-minute interval, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
W_a = Weight of residue in solvent wash, mg.
Y = Dry gas meter calibration factor.
Y_{CO} = Measured mole fraction of CO (dry), average from Section 8.2, g/g-mole (lb/lb-mole).
Y_{CO2} = Measured mole fraction of CO_2 (dry), average from Section 8.2, g/g-mole (lb/lb-mole).
Y_{HC} = Assumed mole fraction of HC (dry), g/g-mole (lb/lb-mole); = 0.0088 for catalytic wood heaters; = 0.0132 for non-catalytic wood heaters; = 0.0080 for pellet-fired wood heaters.
10 = Length of first sampling period, min.
13.6 = Specific gravity of mercury.
100 = Conversion to percent.
θ = Total sampling time, min.
θ_l = Sampling time interval, from the beginning of a run until the first component change, min.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5H–3).

12.3 Dry Gas Volume. Same as Method 5, Section 12.3.

12.4 Volume of Water Vapor.

$$V_{w(std)} = K_2 V_{lc} \qquad \text{Eq. 5H-1}$$

Where:

K_2 = 0.001333 m^3/ml for metric units.
K_2 = 0.04707 ft^3/ml for English units.

12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Eq. 5H-2}$$

12.6 Solvent Wash Blank.

$$W_a = \frac{M_a V_{aw}}{V_a} \qquad \text{Eq. 5H-3}$$

12.7 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1, 2, 3, and 4 less the appropriate solvent blanks (see Figure 5H–4).

Note: Refer to Method 5, Section 8.5 to assist in calculation of results involving two filter assemblies.

12.8 Particulate Concentration.

$$C_s = \frac{0.001 g}{mg} \cdot \frac{m_n}{V_{m(std)}} \qquad \text{Eq. 5H-4}$$

12.9 Sample Flow Rate Adjustment.

$$a = \frac{S_l}{S_i} \qquad \text{Eq. 5H-5}$$

12.10 Carbon Balance for Total Moles of Exhaust Gas (dry)/kg of Wood Burned in the Exhaust Gas.

$$N_T = \frac{K_3 N_C}{Y_{CO_2} + Y_{CO} + Y_{HC}} \qquad \text{Eq. 5H-6}$$

Where:

K_3 = 1000 g/kg for metric units.
K_3 = 1.0 lb/lb for English units.

Note: The NO_X/SO_X portion of the gas is assumed to be negligible.

12.11 Total Stack Gas Flow Rate.

$$Q_{sd} = K_4 N_T BR \qquad \text{Eq. 5H-7}$$

Where:

K_4 = 0.02406 dscm/g-mole for metric units.
K_4 = 384.8 dscf/lb-mole for English units.

12.12 Particulate Emission Rate.

$$E = C_s Q_{sd} \qquad \text{Eq. 5H-8}$$

12.13 Proportional Rate Variation. Calculate PR for each 10-minute interval, i, of the test run.

$$PR = \frac{\theta\, S_i V_{mi(std)}}{10 \sum_{i=1}^{N} \left[S_i V_{mi(std)} \right]} \times 100 \qquad \text{Eq. 5H-9}$$

12.14 Acceptable Results. If no more than 15 percent of the PR values for all the intervals fall outside the range 90 percent ≤ PR ≤ 110 percent, and if no PR value for any interval falls outside the range 75 ≤ PR ≤ 125 percent, the results are acceptable. If the PR values for the test runs are judged to be unacceptable, report the test run emission results, but do not include the test run results in calculating the weighted average emission rate, and repeat the test.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

Same as Method 5G, Section 17.0.

BILLING CODE 6560–50–P

17.0 Tables, Diagrams, Flowcharts, and Validation Data

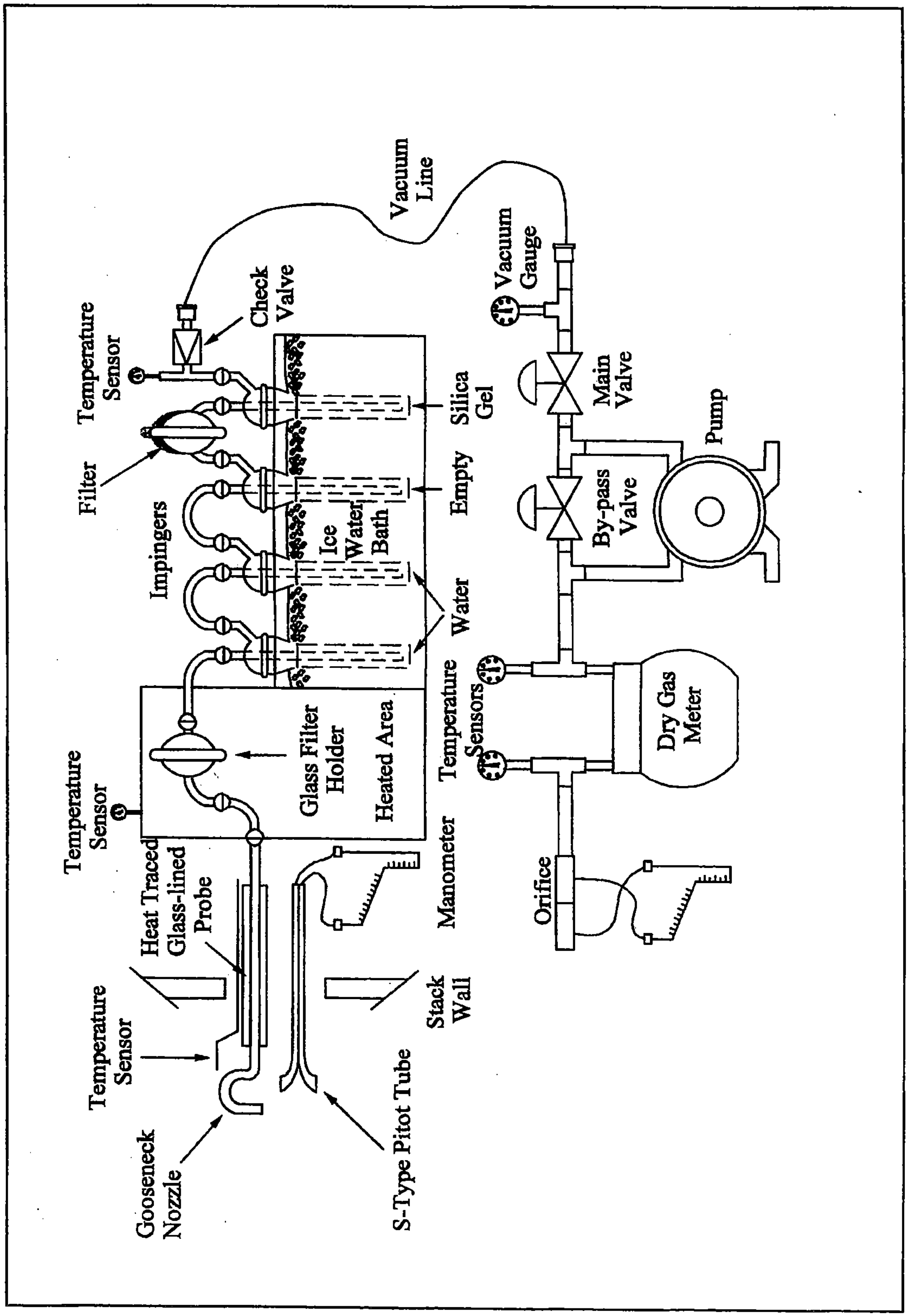


Figure 5H-1. Sampling Train.

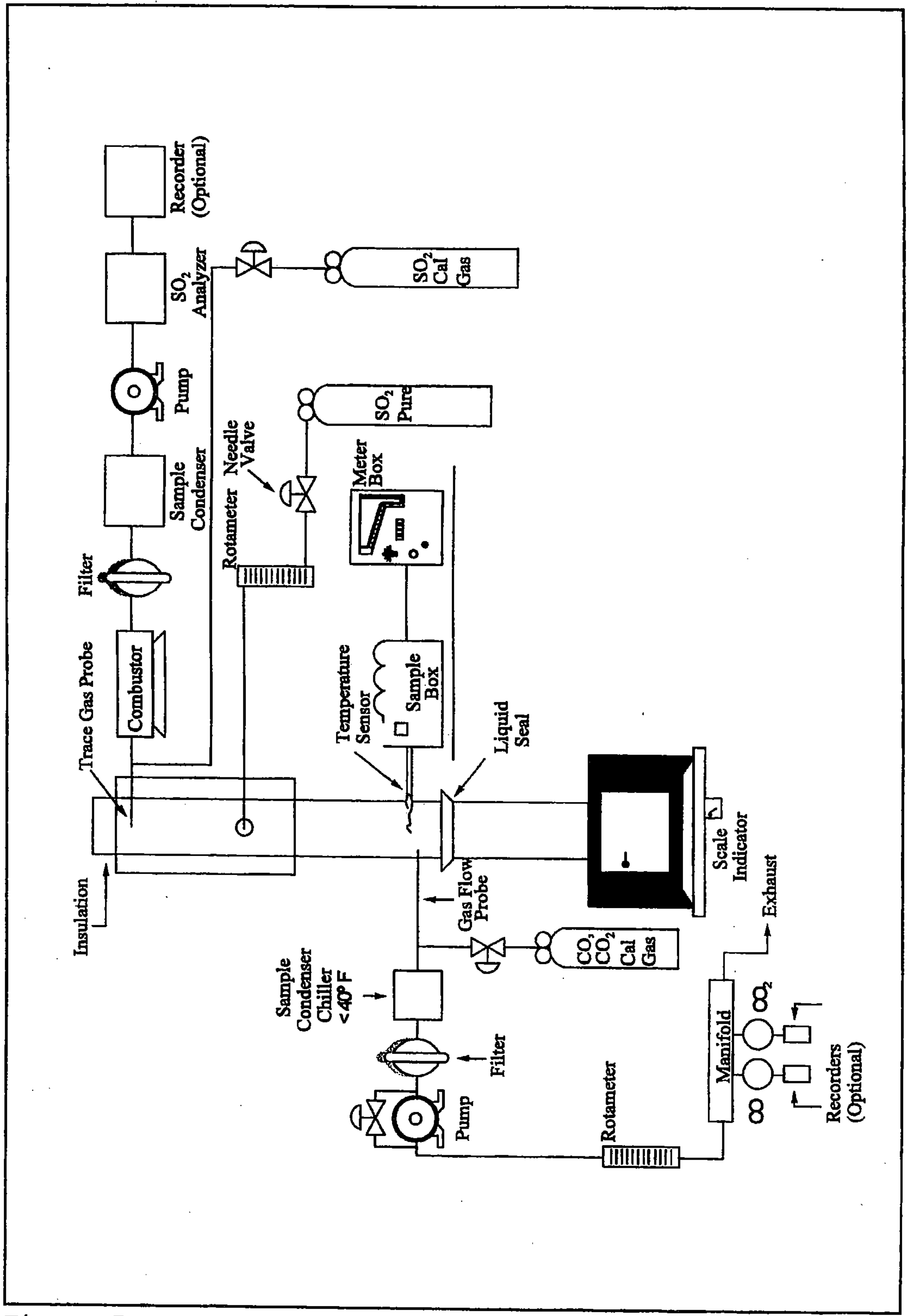


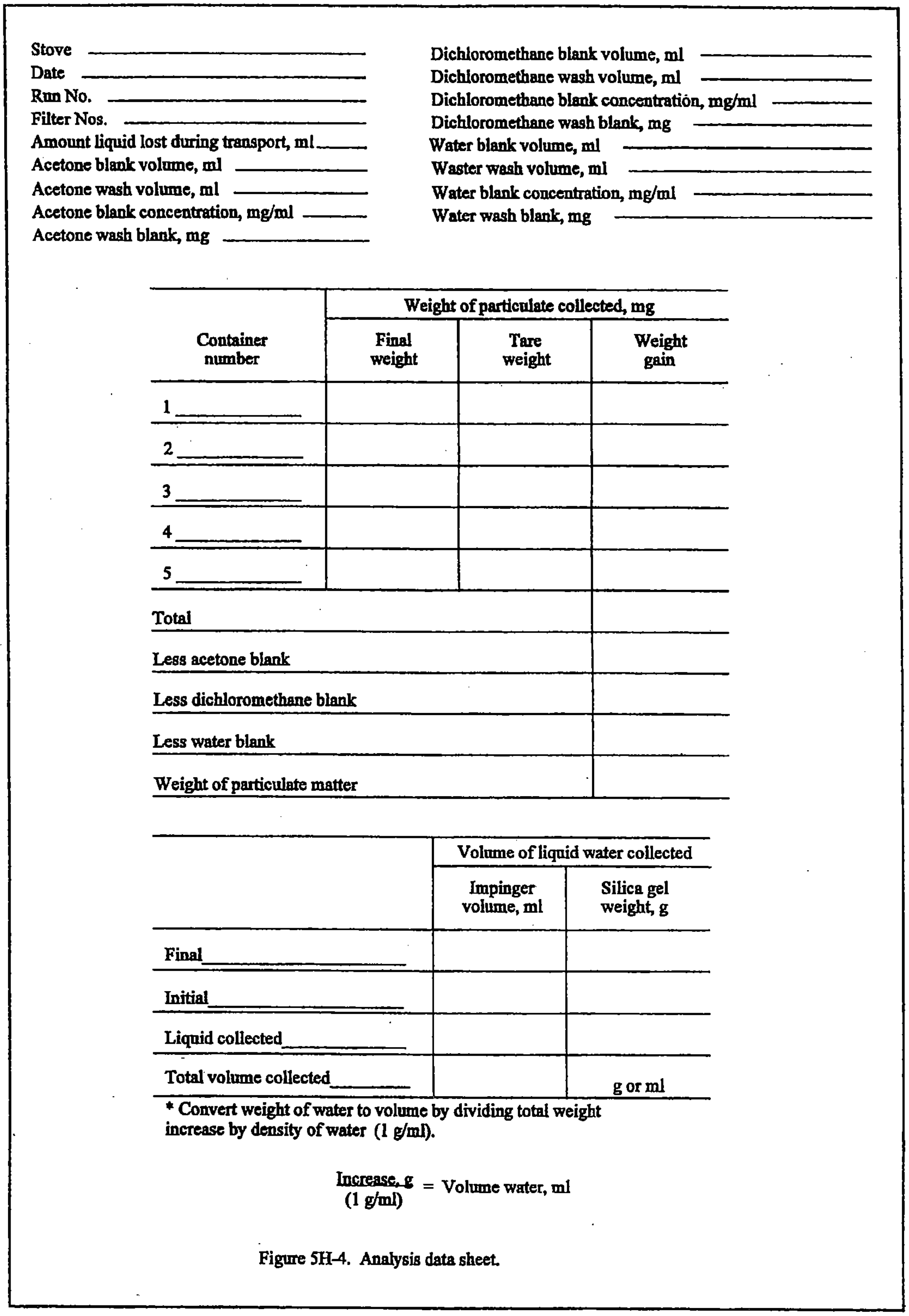
Figure 5H-2. Test System Schematic.

Stove ______
Test Method ______
Operator ______
Date ______
Run No. ______
Start Time ______
Stop Time ______
Sample Box No. ______
Meter Box No. ______
Meter $\Delta H_@$ (Optional) ______
C Factor ______

Pitot tube coefficient, Cp ______
Room temperature, C°, (°F) ______
Barometric pressure mb (in. Hg) ______
Measured or assumed moisture, % ______
Nozzle identification No. ______
Average calibrated nozzle diameter, mm (in.) ______
Final leak rate, m^3/min, (cfm) ______
Probe liner material ______
Draft or static pressure, mm H_2O (in. H_2O) ______
Filter Nos. ______

Clock time	Test run time	Vacuum	Fuel temperature	Flow in Flue	Volume sample in period	Gas meter volume	Gas sample temperature at dry gas meter		Front filter holder temperature	Temperature of gas leaving dryer or last impinger
	(q). min.	mm Hg (in. Hg)	(T_s) °C (°F)	m^3/min (ft^3/min)	m^3(ft^3)	m^3 (ft^3)	°C (°F)	°C (°F)	°C (°F)	°C (°F)
Total							Avg.	Avg.		
Average							Avg.			

Figure 5H-3. Sampling Data Sheet.



Stove ____________
Date ____________
Run No. ____________
Filter Nos. ____________
Amount liquid lost during transport, ml ____
Acetone blank volume, ml ____________
Acetone wash volume, ml ____________
Acetone blank concentration, mg/ml ______
Acetone wash blank, mg ____________

Dichloromethane blank volume, ml ____________
Dichloromethane wash volume, ml ____________
Dichloromethane blank concentration, mg/ml ________
Dichloromethane wash blank, mg ____________
Water blank volume, ml ____________
Waster wash volume, ml ____________
Water blank concentration, mg/ml ____________
Water wash blank, mg ____________

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1 ____________			
2 ____________			
3 ____________			
4 ____________			
5 ____________			
Total			
Less acetone blank			
Less dichloromethane blank			
Less water blank			
Weight of particulate matter			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final ____________		
Initial ____________		
Liquid collected ____________		
Total volume collected ________		g or ml

* Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{(1\ \text{g/ml})} = \text{Volume water, ml}$$

Figure 5H-4. Analysis data sheet.

Figure 5H-4. Analysis Data Sheet.

Method 6—Determination of Sulfur Dioxide Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, and Method 8.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
SO_2	7449–09–5	3.4 mg SO_2/m^3 $(2.12 \times 10)_{-7}$ lb/ft_3

1.2 Applicability. This method applies to the measurement of sulfur dioxide (SO^2) emissions from stationary sources.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2. 0 Summary of Method

2.1 A gas sample is extracted from the sampling point in the stack. The SO^2 and the sulfur trioxide, including those fractions in any sulfur acid mist, are separated. The SO^2 fraction is measured by the barium-thorin titration method.

3.0 Definitions. [Reserved]

4.0 Interferences

4.1 Free Ammonia. Free ammonia interferes with this method by reacting with SO^2 to form particulate sulfite and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and/or noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator are required. One approved alternative is listed in Reference 13 of Section 17.0.

4.2 Water-Soluble Cations and Fluorides. The cations and fluorides are removed by a glass wool filter and an isopropanol bubbler; therefore, they do not affect the SO_2 analysis. When samples are collected from a gas stream with high concentrations of metallic fumes (*i.e.*, very fine cation aerosols) a high-efficiency glass fiber filter must be used in place of the glass wool plug (*i.e.*, the one in the probe) to remove the cation interferent.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations before performing this test method.

5.2 Corrosive reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water for at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burns as thermal burns.

5.2.1 Hydrogen Peroxide (H_2O_2). Irritating to eyes, skin, nose, and lungs. 30% H_2O_2 is a strong oxidizing agent. Avoid contact with skin, eyes, and combustible material. Wear gloves when handling.

5.2.2 Sodium Hydroxide (NaOH). Causes severe damage to eyes and skin. Inhalation causes irritation to nose, throat, and lungs. Reacts exothermically with limited amounts of water.

5.2.3 Sulfuric Acid (H_2SO_4). Rapidly destructive to body tissue. Will cause third degree burns. Eye damage may result in blindness. Inhalation may be fatal from spasm of the larynx, usually within 30 minutes. May cause lung tissue damage with edema. 1 mg/m^3 for 8 hours will cause lung damage or, in higher concentrations, death. Provide ventilation to limit inhalation. Reacts violently with metals and organics.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. A schematic of the sampling train is shown in Figure 6–1. The sampling equipment described in Method 8 may be substituted in place of the midget impinger equipment of Method 6. However, the Method 8 train must be modified to include a heated filter between the probe and isopropanol impinger, and the operation of the sampling train and sample analysis must be at the flow rates and solution volumes defined in Method 8. Alternatively, SO_2 may be determined simultaneously with particulate matter and moisture determinations by either (1) replacing the water in a Method 5 impinger system with a 3 percent H_2O_2 solution, or (2) replacing the Method 5 water impinger system with a Method 8 isopropanol-filter-H_2O_2 system. The analysis for SO_2 must be consistent with the procedure of Method 8. The Method 6 sampling train consists of the following components:

6.1.1.1 Probe. Borosilicate glass or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6 mm (0.25 in.) inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated out-of-stack) to remove particulate matter, including sulfuric acid mist. A plug of glass wool is a satisfactory filter.

6.1.1.2 Bubbler and Impingers. One midget bubbler with medium-coarse glass frit and borosilicate or quartz glass wool packed in top (see Figure 6–1) to prevent sulfuric acid mist carryover, and three 30-ml midget impingers. The midget bubbler and midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage. A midget impinger may be used in place of the midget bubbler.

Note: Other collection absorbers and flow rates may be used, subject to the approval of the Administrator, but the collection efficiency must be shown to be at least 99 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added and analyzed separately. This extra absorber must not contain more than 1 percent of the total SO_2.

6.1.1.3 Glass Wool. Borosilicate or quartz.

6.1.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

6.1.1.5 Temperature Sensor. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1 °C (2 °F).

6.1.1.6 Drying Tube. Tube packed with 6- to 16- mesh indicating-type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump. If silica gel is previously used, dry at 177 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

6.1.1.7 Valve. Needle valve, to regulate sample gas flow rate.

6.1.1.8 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and rate meter to negate the pulsation effect of the diaphragm pump on the rate meter.

6.1.1.9 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1 liter/min (0.035 cfm).

6.1.1.10 Volume Meter. Dry gas meter (DGM), sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature sensor (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3 °C (5.4 °F). A critical orifice may be used in place of the DGM specified in this section provided that it is selected, calibrated, and used as specified in Section 16.0.

6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). See the **Note** in Method 5, Section 6.1.2.

6.1.3 Vacuum Gauge and Rotameter. At least 760-mm Hg (30-in. Hg) gauge and 0- to 40-ml/min rotameter, to be used for leak-check of the sampling train.

6.2 Sample Recovery. The following items are needed for sample recovery:

6.2.1 Wash Bottles. Two polyethylene or glass bottles, 500-ml.

6.2.2 Storage Bottles. Polyethylene bottles, 100-ml, to store impinger samples (one per sample).

6.3 Sample Analysis. The following equipment is needed for sample analysis:

6.3.1 Pipettes. Volumetric type, 5-ml, 20-ml (one needed per sample), and 25-ml sizes.

6.3.2 Volumetric Flasks. 100-ml size (one per sample) and 1000-ml size.

6.3.3 Burettes. 5- and 50-ml sizes.

6.3.4 Erlenmeyer Flasks. 250-ml size (one for each sample, blank, and standard).

6.3.5 Dropping Bottle. 125-ml size, to add indicator.

6.3.6 Graduated Cylinder. 100-ml size.

6.3.7 Spectrophotometer. To measure absorbance at 352 nm.

7.0 Reagents and Standards

Note: Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Water. Deionized distilled to conform to ASTM Specification D 1193–77 or 91 Type 3 (incorporated by reference—see § 60.17). The $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.1.2 Isopropanol, 80 Percent by Volume. Mix 80 ml of isopropanol with 20 ml of water.

7.1.2.1 Check each lot of isopropanol for peroxide impurities as follows: Shake 10 ml of isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of water. After 1 minute, read the absorbance at 352 nm on a spectrophotometer using a 1-cm path length. If absorbance exceeds 0.1, reject alcohol for use.

7.1.2.2 Peroxides may be removed from isopropanol by redistilling or by passage through a column of activated alumina; however, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Rejection of contaminated lots may, therefore, be a more efficient procedure.

7.1.3 Hydrogen Peroxide (H_2O_2), 3 Percent by Volume. Add 10 ml of 30 percent H_2O_2 to 90 ml of water. Prepare fresh daily.

7.1.4 Potassium Iodide Solution, 10 Percent Weight by Volume (w/v). Dissolve 10.0 g of KI in water, and dilute to 100 ml. Prepare when needed.

7.2 Sample Recovery. The following reagents are required for sample recovery:

7.2.1 Water. Same as in Section 7.1.1.

7.2.2 Isopropanol, 80 Percent by Volume. Same as in Section 7.1.2.

7.3 Sample Analysis. The following reagents and standards are required for sample analysis:

7.3.1 Water. Same as in Section 7.1.1.

7.3.2 Isopropanol, 100 Percent.

7.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of water.

7.3.4 Barium Standard Solution, 0.0100 N. Dissolve 1.95 g of barium perchlorate trihydrate [$Ba(ClO_4)_2\ 3H_2O$] in 200 ml water, and dilute to 1 liter with isopropanol. Alternatively, 1.22 g of barium chloride dihydrate [$BaCl_2\ 2H_2O$] may be used instead of the barium perchlorate trihydrate. Standardize as in Section 10.5.

7.3.5 Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to ±0.0002 N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

7.3.6 Quality Assurance Audit Samples. When making compliance determinations, audit samples, if available must be obtained from the appropriate EPA Regional Office or from the responsible enforcement authority and analyzed in conjunction with the field samples.

Note: The responsible enforcement authority should be notified at least 30 days prior to the test date to allow sufficient time for sample delivery.

8.0 Sample Collection, Preservation, Storage and Transport

8.1 Preparation of Sampling Train. Measure 15 ml of 80 percent isopropanol into the midget bubbler and 15 ml of 3 percent H_2O_2 into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6–1. Adjust the probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

8.2 Sampling Train Leak-Check Procedure. A leak-check prior to the sampling run is recommended, but not required. A leak-check after the sampling run is mandatory. The leak-check procedure is as follows:

8.2.1 Temporarily attach a suitable (*e.g.*, 0- to 40- ml/min) rotameter to the outlet of the DGM, and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate in excess of 2 percent of the average sampling rate is not acceptable.

Note: Carefully (*i.e.*, slowly) release the probe inlet plug before turning off the pump.

8.2.2 It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. To leak-check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm Hg (10 in. Hg), plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum should remain stable for at least 30 seconds.

If performed prior to the sampling run, the pump leak-check shall precede the leak-check of the sampling train described immediately above; if performed after the sampling run, the pump leak-check shall follow the sampling train leak-check.

8.2.3 Other leak-check procedures may be used, subject to the approval of the Administrator.

8.3 Sample Collection.

8.3.1 Record the initial DGM reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rate meter. Maintain this constant rate (± 10 percent) during the entire sampling run.

8.3.2 Take readings (DGM volume, temperatures at DGM and at impinger outlet, and rate meter flow rate) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C (68 °F) or less.

8.3.3 At the conclusion of each run, turn off the pump, remove the probe from the stack, and record the final readings. Conduct a leak-check as described in Section 8.2. (This leak-check is mandatory.) If a leak is detected, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for the leakage.

8.3.4 Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate. Clean ambient air can be provided by passing air through a charcoal filter or through an extra midget impinger containing 15 ml of 3 percent H_2O_2. Alternatively, ambient air without purification may be used.

8.4 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment. Rinse the three midget impingers and the connecting tubes with water, and add the rinse to the same storage container. Mark the fluid level. Seal and identify the sample container.

9.0 Quality Control

Section	Quality control measure	Effect
7.1.2	Isopropanol check	Ensure acceptable level of peroxide impurities in isopropanol.
8.2, 10.1–10.4	Sampling equipment leak-check and calibration	Ensure accurate measurement of stack gas flow rate, sample volume.
10.5	Barium standard solution standardization	Ensure precision of normality determination.
11.2.3	Replicate titrations	Ensure precision of titration determinations
11.3	Audit sample analysis	Evaluate analyst's technique and standards preparation.

10.0 Calibration and Standardization

10.1 Volume Metering System.

10.1.1 Initial Calibration.

10.1.1.1 Before its initial use in the field, leak-check the metering system (drying tube, needle valve, pump, rate meter, and DGM) as follows: Place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 250 mm Hg (10 in. Hg). Plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum must remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

10.1.1.2 Remove the drying tube, and calibrate the metering system (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet-test meter (*e.g.*, 1 liter per revolution) to the inlet of the needle valve. Make three independent calibration runs, using at least five revolutions of the DGM per run. Calculate the calibration factor Y (wet-test meter calibration volume divided by the DGM volume, both volumes adjusted to the same reference temperature and pressure) for each run, and average the results (Y_i). If any Y-value deviates by more than 2 percent from (Y_i), the metering system is unacceptable for use. If the metering system is acceptable, use (Y_i) as the calibration factor for subsequent test runs.

10.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check using the procedures outlined in Section 10.1.1.2, except that three or more revolutions of the DGM may be used, and only two independent runs need be made. If the average of the two post-test calibration factors does not deviate by more than 5 percent from Y_i, then Y_i is accepted as the DGM calibration factor (Y), which is used in Equation 6–1 to calculate collected sample volume (see Section 12.2). If the deviation is more than 5 percent, recalibrate the metering system as in Section 10.1.1, and determine a post-test calibration factor (Y_f). Compare Y_i and Y_f; the smaller of the two factors is accepted as the DGM calibration factor. If recalibration indicates that the metering system is unacceptable for use, either void the test run or use methods, subject to the approval of the Administrator, to determine an acceptable value for the collected sample volume.

10.1.3 DGM as a Calibration Standard. A DGM may be used as a calibration standard for volume measurements in place of the wet-test meter specified in Section 10.1.1.2, provided that it is calibrated initially and recalibrated periodically according to the same procedures outlined in Method 5, Section 10.3 with the following exceptions: (a) the DGM is calibrated against a wet-test meter having a capacity of 1 liter/rev (0.035 ft^3/rev) or 3 liters/rev (0.1 ft^3/rev) and having the capability of measuring volume to within 1 percent; (b) the DGM is calibrated at 1 liter/min (0.035 cfm); and (c) the meter box of the Method 6 sampling train is calibrated at the same flow rate.

10.2 Temperature Sensors. Calibrate against mercury-in-glass thermometers.

10.3 Rate Meter. The rate meter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instructions.

10.4 Barometer. Calibrate against a mercury barometer.

10.5 Barium Standard Solution. Standardize the barium perchlorate or chloride solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added. Run duplicate analyses. Calculate the normality using the average of duplicate analyses where the titrations agree within 1 percent or 0.2 ml, whichever is larger.

11.0 Analytical Procedure

11.1 Sample Loss Check. Note level of liquid in container and confirm whether any sample was lost during shipment; note this finding on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

11.2 Sample Analysis.

11.2.1 Transfer the contents of the storage container to a 100-ml volumetric flask, dilute to exactly 100 ml with water, and mix the diluted sample.

11.2.2 Pipette a 20-ml aliquot of the diluted sample into a 250-ml Erlenmeyer flask and add 80 ml of 100 percent isopropanol plus two to four drops of thorin indicator. While stirring the solution, titrate to a pink endpoint using 0.0100 N barium standard solution.

11.2.3 Repeat the procedures in Section 11.2.2, and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger.

Note: Protect the 0.0100 N barium standard solution from evaporation at all times.

11.3 Audit Sample Analysis.

11.3.1 When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample, if available, must be analyzed.

11.3.2 Concurrently analyze the audit sample and the compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation.

11.3.3 The same analyst, analytical reagents, and analytical system must be used for the compliance samples and the audit sample. If this condition is met, duplicate auditing of subsequent compliance analyses for the same enforcement agency within a 30-day period is waived. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of separate enforcement agencies, unless prior arrangements have been made with both enforcement agencies.

11.4 Audit Sample Results.

11.4.1 Calculate the audit sample concentrations and submit results using the instructions provided with the audit samples.

11.4.2 Report the results of the audit samples and the compliance determination samples along with their identification numbers, and the analyst's name to the responsible enforcement authority. Include this information with reports of any subsequent compliance analyses for the same enforcement authority during the 30-day period.

11.4.3 The concentrations of the audit samples obtained by the analyst must agree within 5 percent of the actual concentration. If the 5 percent specification is not met, reanalyze the compliance and audit samples, and include initial and reanalysis values in the test report.

11.4.4 Failure to meet the 5-percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.

12.0 Data Analysis and Calculations

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

12.1 Nomenclature.

C_a = Actual concentration of SO_2 in audit sample, mg/dscm.

C_d = Determined concentration of SO_2 in audit sample, mg/dscm.

C_{SO_2} = Concentration of SO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf).

N = Normality of barium standard titrant, meq/ml.

P_{bar} = Barometric pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29:92 in. Hg).

RE = Relative error of QA audit sample analysis, percent

T_m = Average DGM absolute temperature, °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_a = Volume of sample aliquot titrated, ml.

V_m = Dry gas volume as measured by the DGM, dcm (dcf).

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

V_{soln} = Total volume of solution in which the SO*2* sample is contained, 100 ml.

V_t = Volume of barium standard titrant used for the sample (average of replicate titration), ml.

V_{tb} = Volume of barium standard titrant used for the blank, ml.

Y = DGM calibration factor.

12.2 Dry Sample Gas Volume, Corrected to Standard Conditions.

$$V_{m(std)} = \frac{\left(V_m Y\, T_{std}\, P_{bar}\right)}{\left(T_m\, P_{std}\right)} = \frac{K_1 Y\, V_m\, P_{bar}}{T_m} \qquad \text{Eq. 6-1}$$

Where:

K_1 = 0.3855 °K/mm Hg for metric units.

K_1 = 17.65 °R/in. Hg for English units.

12.3 SO_2 Concentration.

$$C_{SO_2} = \frac{K_2\, N\left(V_t - V_{tb}\right)\left(V_{soln}/V_a\right)}{V_{m(std)}} \qquad \text{Eq. 6-2}$$

Where:

K_2 = 32.03 mg SO_2/meq for metric units.

K_2 = 7.061×10^{-5} lb SO_2/meq for English units.

12.4 Relative Error for QA Audit Samples.

$$RE = \frac{100\left(C_d - C_a\right)}{C_a} \qquad \text{Eq. 6-3}$$

13.0 Method Performance

13.1 Range. The minimum detectable limit of the method has been determined to be 3.4 mg SO_2/m^3 (2.12×10^{-7} lb/ft^3). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m^3 (0.005 lb/ft^3) of SO_2 can be collected efficiently at a rate of 1.0 liter/min (0.035 cfm) for 20 minutes in two midget impingers, each containing 15 ml of 3 percent H_2O_2. Based on theoretical calculations, the upper concentration limit in a 20 liter (0.7 ft^3) sample is about 93,300 mg/m^3 (0.00583 lb/ft^3).

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Nomenclature. Same as Section 12.1, with the following additions:

B_{wa} = Water vapor in ambient air, proportion by volume.

M_a = Molecular weight of the ambient air saturated at impinger temperature, g/g-mole (lb/lb-mole).

M_s = Molecular weight of the sample gas saturated at impinger temperature, g/g-mole (lb/lb-mole).

P_c = Inlet vacuum reading obtained during the calibration run, mm Hg (in. Hg).
P_{sr} = Inlet vacuum reading obtained during the sampling run, mm Hg (in. Hg).
$\overline{Q}_{std}$ = Volumetric flow rate through critical orifice, scm/min (scf/min).
Q_{std} = Average flow rate of pre-test and post-test calibration runs, scm/min (scf/min).
T_{amb} = Ambient absolute temperature of air, °K (°R).
V_{sb} = Volume of gas as measured by the soap bubble meter, m³ (ft³).
$V_{sb(std)}$ = Volume of gas as measured by the soap bubble meter, corrected to standard conditions, scm (scf).
θ = Soap bubble travel time, min.
θ_s = Time, min.

16.2 Critical Orifices for Volume and Rate Measurements. A critical orifice may be used in place of the DGM specified in Section 6.1.1.10, provided that it is selected, calibrated, and used as follows:

16.2.1 Preparation of Sampling Train. Assemble the sampling train as shown in Figure 6–2. The rate meter and surge tank are optional but are recommended in order to detect changes in the flow rate.

Note: The critical orifices can be adapted to a Method 6 type sampling train as follows: Insert sleeve type, serum bottle stoppers into two reducing unions. Insert the needle into the stoppers as shown in Figure 6–3.

16.2.2 Selection of Critical Orifices.

16.2.2.1 The procedure that follows describes the use of hypodermic needles and stainless steel needle tubings, which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices, (*i.e.*, a critical vacuum can be obtained) as described in this section. Select a critical orifice that is sized to operate at the desired flow rate. The needle sizes and tubing lengths shown in Table 6–1 give the following approximate flow rates.

16.2.2.2 Determine the suitability and the appropriate operating vacuum of the critical orifice as follows: If applicable, temporarily attach a rate meter and surge tank to the outlet of the sampling train, if said equipment is not present (see Section 16.2.1). Turn on the pump and adjust the valve to give an outlet vacuum reading corresponding to about half of the atmospheric pressure. Observe the rate meter reading. Slowly increase the vacuum until a stable reading is obtained on the rate meter. Record the critical vacuum, which is the outlet vacuum when the rate meter first reaches a stable value. Orifices that do not reach a critical value must not be used.

16.2.3 Field Procedures.

16.2.3.1 Leak-Check Procedure. A leak-check before the sampling run is recommended, but not required. The leak-check procedure is as follows: Temporarily attach a suitable (*e.g.*, 0–40 ml/min) rotameter and surge tank, or a soap bubble meter and surge tank to the outlet of the pump. Plug the probe inlet, pull an outlet vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter or bubble meter. A leakage rate in excess of 2 percent of the average sampling rate ($\overline{Q}_{std}$) is not acceptable. Carefully release the probe inlet plug before turning off the pump.

16.2.3.2 Moisture Determination. At the sampling location, prior to testing, determine the percent moisture of the ambient air using the wet and dry bulb temperatures or, if appropriate, a relative humidity meter.

16.2.3.3 Critical Orifice Calibration. At the sampling location, prior to testing, calibrate the entire sampling train (*i.e.*, determine the flow rate of the sampling train when operated at critical conditions). Attach a 500-ml soap bubble meter to the inlet of the probe, and operate the sampling train at an outlet vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. Record the information listed in Figure 6–4. Calculate the standard volume of air measured by the soap bubble meter and the volumetric flow rate using the equations below:

$$V_{sb(std)} = V_{sb}\left(T_{std}/T_{amb}\right)\left(P_{bar}/P_{std}\right) \qquad \text{Eq. 6-4}$$

$$Q_{std} = \frac{V_{sb(std)}}{\theta} \qquad \text{Eq. 6-5}$$

16.2.3.4 Sampling.

16.2.3.4.1 Operate the sampling train for sample collection at the same vacuum used during the calibration run. Start the watch and pump simultaneously. Take readings (temperature, rate meter, inlet vacuum, and outlet vacuum) at least every 5 minutes. At the end of the sampling run, stop the watch and pump simultaneously.

16.2.3.4.2 Conduct a post-test calibration run using the calibration procedure outlined in Section 16.2.3.3. If the Q_{std} obtained before and after the test differ by more than 5 percent, void the test run; if not, calculate the volume of the gas measured with the critical orifice using Equation 6–6 as follows:

$$V_{m(std)} = \frac{\overline{Q}_{std}\ \theta_s\left(1-B_{wa}\right)\left(P_{bar}+P_{sr}\right)}{\left(P_{bar}+P_c\right)} \qquad \text{Eq. 6-6}$$

16.2.3.4.3 If the percent difference between the molecular weight of the ambient air at saturated conditions and the sample gas is more that ± 3 percent, then the molecular weight of the gas sample must be considered in the calculations using the following equation:

$$_{(std)} = \frac{\overline{Q}_{std}\ \theta_s\left(1-B_{wa}\right)\left(P_{bar}+P_{sr}\right)\left(M_a/M_s\right)^{1}}{\left(P_{bar}+P_c\right)} \qquad \text{Eq. 6-7}$$

Note: A post-test leak-check is not necessary because the post-test calibration run results will indicate whether there is any leakage.

16.2.3.4.4 Drain the ice bath, and purge the sampling train using the procedure described in Section 8.3.4.

16.3 Elimination of Ammonia Interference. The following alternative procedures must be used in addition to those specified in the method when sampling at sources having ammonia emissions.

16.3.1 Sampling. The probe shall be maintained at 275 °C (527 °F) and equipped with a high-efficiency in-stack filter (glass fiber) to remove particulate matter. The filter material shall be unreactive to SO_2. Whatman 934AH (formerly Reeve Angel 934AH) filters treated as described in Reference 10 in Section 17.0 of Method 5 is an example of a filter that has been shown to work. Where alkaline particulate matter and condensed moisture are present in the gas stream, the filter shall be heated above the moisture dew point but below 225 °C (437 °F).

16.3.2 Sample Recovery. Recover the sample according to Section 8.4 except for discarding the contents of the midget bubbler. Add the bubbler contents, including the rinsings of the bubbler with water, to a separate polyethylene bottle from the rest of the sample. Under normal testing conditions where sulfur trioxide will not be present significantly, the tester may opt to delete the midget bubbler from the sampling train. If an approximation of the sulfur trioxide concentration is desired, transfer the contents of the midget bubbler to a separate polyethylene bottle.

16.3.3 Sample Analysis. Follow the procedures in Sections 11.1 and 11.2, except add 0.5 ml of 0.1 N HCl to the Erlenmeyer flask and mix before adding the indicator. The following analysis procedure may be used for an approximation of the sulfur trioxide concentration. The accuracy of the calculated concentration will depend upon the ammonia to SO_2 ratio and the level of oxygen present in the gas stream. A fraction of the SO_2 will be counted as sulfur trioxide as the ammonia to SO_2 ratio and the sample oxygen content increases. Generally, when this ratio is 1 or less and the oxygen content is in the range of 5 percent, less than 10 percent of the SO_2 will be counted as sulfur trioxide. Analyze the peroxide and isopropanol sample portions separately. Analyze the peroxide portion as described above. Sulfur trioxide is determined by difference using sequential titration of the isopropanol portion of the sample. Transfer the contents of the isopropanol storage container to a 100-ml volumetric flask, and dilute to exactly 100 ml with water. Pipette a 20-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 0.5 ml of 0.1 N HCl, 80 ml of 100 percent isopropanol, and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes that agree within 1 percent or 0.2 ml, whichever is larger. Use this volume in Equation 6–2 to determine the sulfur trioxide concentration. From the flask containing the remainder of the isopropanol sample, determine the fraction of SO_2 collected in the bubbler by pipetting 20-ml aliquots into 250-ml Erlenmeyer flasks. Add 5 ml of 3 percent H_2O_2, 100 ml of 100 percent isopropanol, and two to four drips of thorin indicator, and titrate as before. From this titration volume, subtract the titrant volume determined for sulfur trioxide, and add the titrant volume determined for the peroxide portion. This final volume constitutes V_t, the volume of barium perchlorate used for the SO_2 sample.

17.0 References

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18.0 Tables, Diagrams, Flowcharts and Validation Data

TABLE 6–1.—APPROXIMATE FLOW RATES FOR VARIOUS NEEDLE SIZES

Needle size (gauge)	Needle length (cm)	Flow rate (ml/min)
21	7.6	1,100
22	2.9	1,000
22	3.8	900
23	3.8	500
23	5.1	450
24	3.2	400

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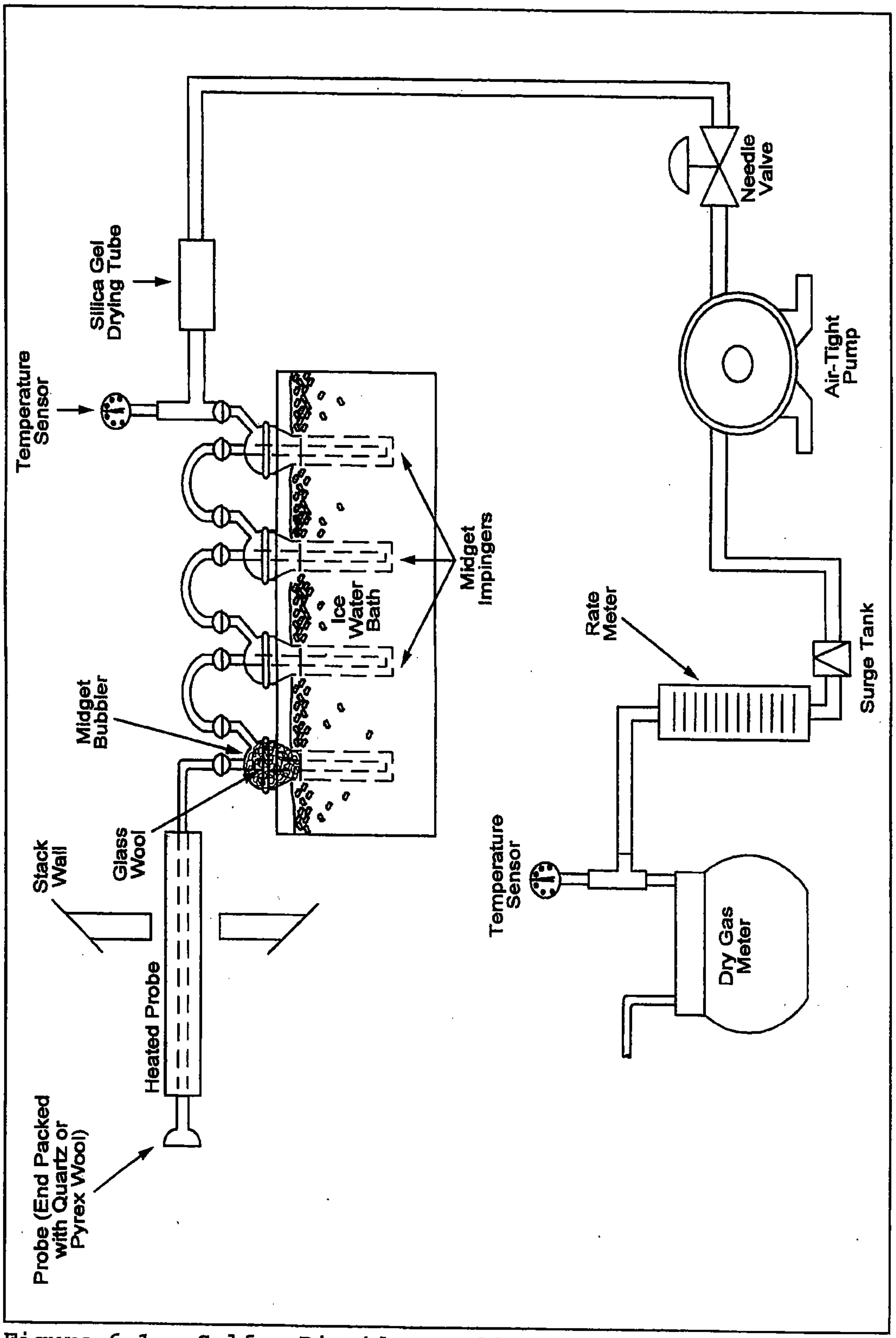

Figure 6-1. Sulfur Dioxide Sampling Train.

the original endpoint was in error. It is recommended that persons conducting this test perform several titrations to be able to correctly identify the endpoint. The importance of this should be recognized because the results of this analytical procedure are extremely sensitive to errors in titration.

16.10 Sample Analysis. Sample treatment is similar to the blank treatment. Before detaching the stems from the bottoms of the impingers, add 20.0 ml of 0.01 N iodine solution through the stems of the impingers holding the zinc acetate solution, dividing it between the two (add about 15 ml to the first impinger and the rest to the second). Add 2 ml HCl solution through the stems, dividing it as with the iodine. Disconnect the sampling line, and store the impingers for 30 minutes. At the end of 30 minutes, rinse the impinger stems into the impinger bottoms. Titrate the impinger contents with 0.01 N $Na_2S_2O_3$. Do not transfer the contents of the impinger to a flask because this may result in a loss of iodine and cause a positive bias.

16.11 Post-test Orifice Calibration. Conduct a post-test critical orifice calibration run using the calibration procedures outlined in Section 16.12.4. If the Q_{std} obtained before and after the test differs by more than 5 percent, void the sample; if not, proceed to perform the calculations.

16.12 Calibrations and Standardizations.

16.12.1 Rotameter and Barometer. Same as Method 11, Sections 10.1.3 and 10.1.4.

16.12.2 $Na_2S_2O_3$ Solution, 0.1 N. Standardize the 0.1 N $Na_2S_2O_3$ solution as follows: To 80 ml water, stirring constantly, add 1 ml concentrated H_2SO_4, 10.0 ml of 0.100 N $KH(IO_3)_2$ and 1 g potassium iodide. Titrate immediately with 0.1 N $Na_2S_2O_3$ until the solution is light yellow. Add 3 ml starch solution, and titrate until the blue color just disappears. Repeat the titration until replicate analyses agree within 0.05 ml. Take the average volume of $Na_2S_2O_3$ consumed to calculate the normality to three decimal figures using Equation 16A–5.

16.12.3 Iodine Solution, 0.01 N. Standardize the 0.01 N iodine solution as follows: Pipet 20.0 ml of 0.01 N iodine solution into a 125-ml Erlenmeyer flask. Titrate with standard 0.01 N $Na_2S_2O_3$ solution until the solution is light yellow. Add 3 ml starch solution, and continue titrating until the blue color just disappears. If the normality of the iodine tested is not 0.010, add a few ml of 0.1 N iodine solution if it is low, or a few ml of water if it is high, and standardize again. Repeat the titration until replicate values agree within 0.05 ml. Take the average volume to calculate the normality to three decimal figures using Equation 16A–6.

16.12.4 Critical Orifice. Calibrate the critical orifice using the sampling train shown in Figure 16A–4 but without the H_2S cylinder and vent rotameter. Connect the soap bubble meter to the Teflon line that is connected to the first impinger. Turn on the pump, and adjust the needle valve until the vacuum is higher than the critical vacuum determined in Section 16.7.4. Record the time required for gas flow to equal the soap bubble meter volume (use the 100-ml soap bubble meter for gas flow rates below 100 ml/min, otherwise use the 500-ml soap bubble meter). Make three runs, and record the data listed in Table 16A–1. Use these data to calculate the volumetric flow rate of the orifice.

16.13 Calculations.

16.13.1 Nomenclature.

B_{wa} = Fraction of water vapor in ambient air during orifice calibration.

C_{H_2S} = H_2S concentration in cylinder gas, ppmv.

$$K_2 = 32.03\frac{mg}{meq}\,\frac{24.05L}{mole}\,\frac{1\,mole}{64.06g}\,\frac{1g}{10^3mg}\,\frac{10^3mL}{L}\,\frac{10^3\mu L}{1mL}$$
$$= \frac{12025\mu L}{meq}$$

M_a = Molecular weight of ambient air saturated at impinger temperature, g/g-mole.

M_s = Molecular weight of sample gas (nitrogen) saturated at impinger temperature, g/g-mole.

Note: (For tests carried out in a laboratory where the impinger temperature is 25 °C, M_a = 28.5 g/g-mole and M_s = 27.7 g/g-mole.)

N_I = Normality of standard iodine solution (0.01 N), g-eq/liter.

N_T = Normality of standard $Na_2S_2O_3$ solution (0.01 N), g-eq/liter.

P_{bar} = Barometric pressure, mm Hg.

P_{std} = Standard absolute pressure, 760 mm Hg.

Q_{std} = Average volumetric flow rate through critical orifice, liters/min.

T_{amb} = Absolute ambient temperature, °K.

T_{std} = Standard absolute temperature, 293 °K.

θ_s = Sampling time, min.

θ_{sb} = Time for soap bubble meter flow rate measurement, min.

$V_{m(std)}$ = Sample gas volume measured by the critical orifice, corrected to standard conditions, liters.

V_{sb} = Volume of gas as measured by the soap bubble meter, ml.

$V_{sb(std)}$ = Volume of gas as measured by the soap bubble meter, corrected to standard conditions, liters.

V_I = Volume of standard iodine solution (0.01 N) used, ml.

V_T = Volume of standard $Na_2S_2O_3$ solution (0.01 N) used, ml.

V_{TB} = Volume of standard $Na_2S_2O_3$ solution (0.01 N) used for the blank, ml.

16.13.2 Normality of Standard $Na_2S_2O_3$ Solution (0.1 N).

$$N_T = \frac{1}{mL\,Na_2S_2O_3\,consumed} \quad \text{Eq. 16A-5}$$

16.13.3 Normality of Standard Iodine Solution (0.01 N).

$$N_I = \frac{N_T V_T}{V_I} \qquad \text{Eq. 16A-6}$$

16.13.4 Sample Gas Volume.

$$V_{m(std)} = \overline{Q}_{std}\Theta_s\left(1 - B_{wa}\right)\frac{M_a}{M_b} \qquad \text{Eq. 16A-7}$$

16.13.5 Concentration of H_2S in the Gas Cylinder.

17.0 References

$$C_{H_2S} = \frac{K N_T \left(V_{TB} - V_T\right)}{V_{m(std)}} \qquad \text{Eq. 16A-8}$$

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18.0 Tables, Diagrams, Flowcharts, and Validation Data

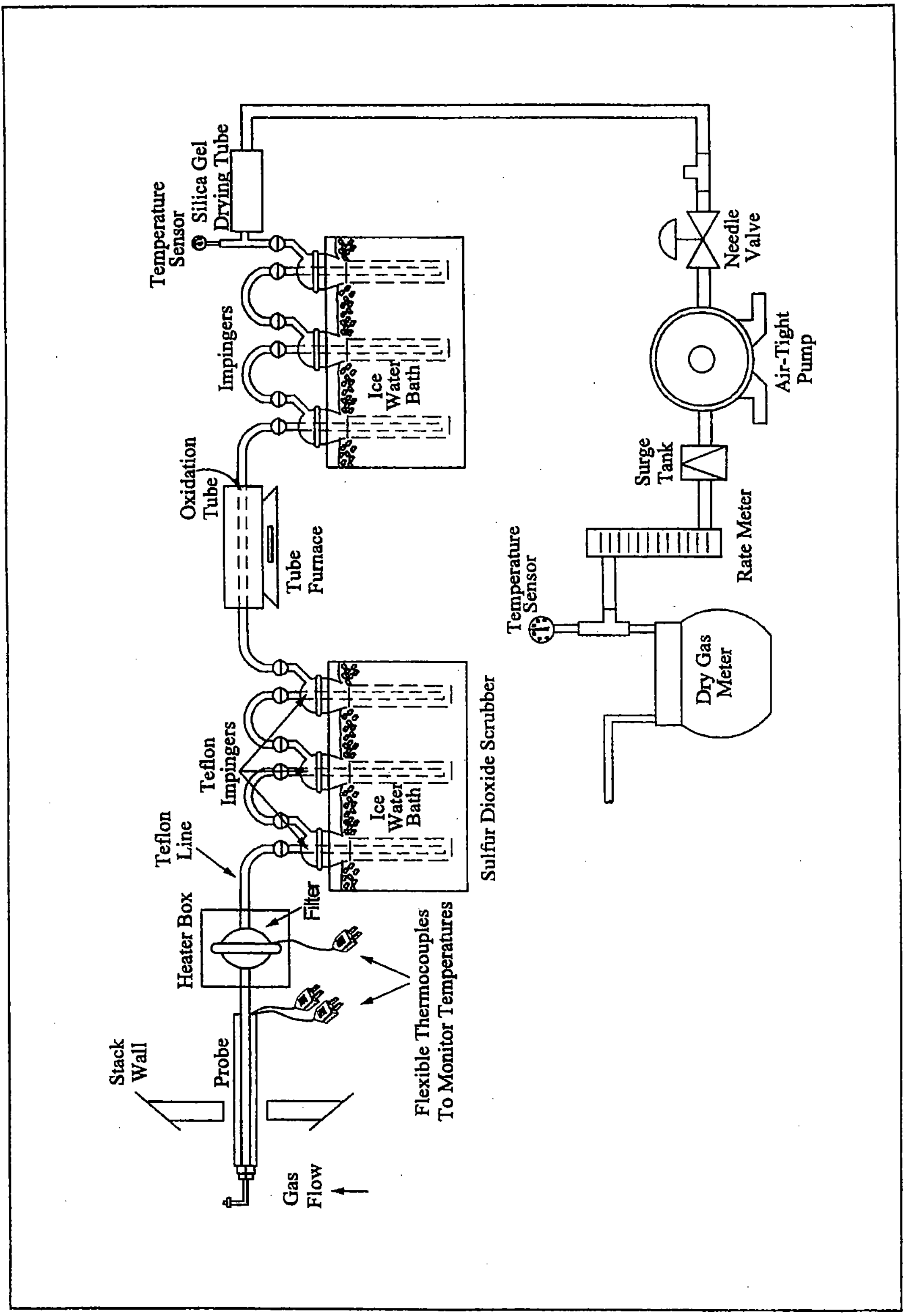

Figure 16A-1. Sampling Train.

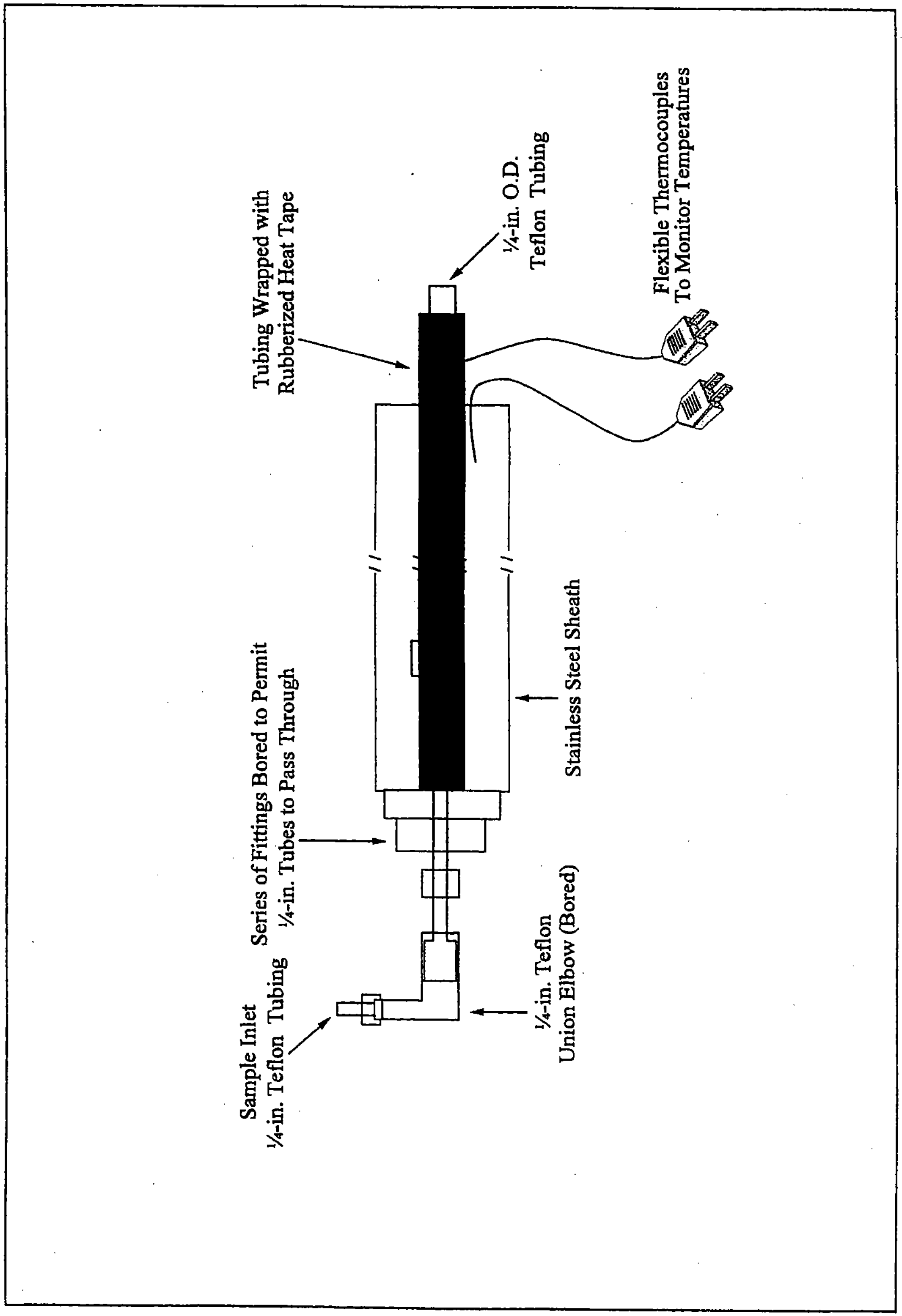

Figure 16A-2. Angled Sampling Probe.

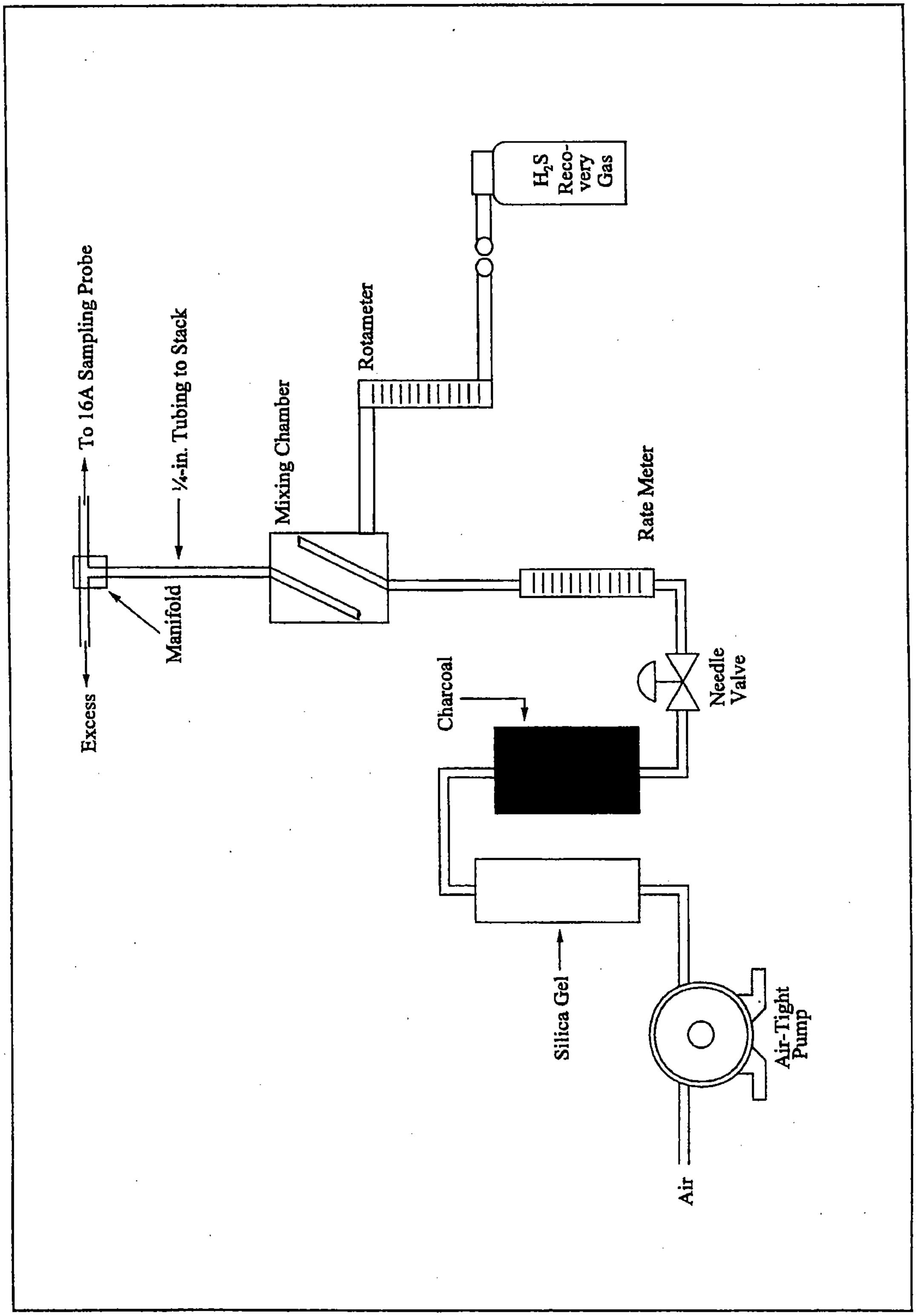

Figure 16A-3. Recovery Gas Dilution System.

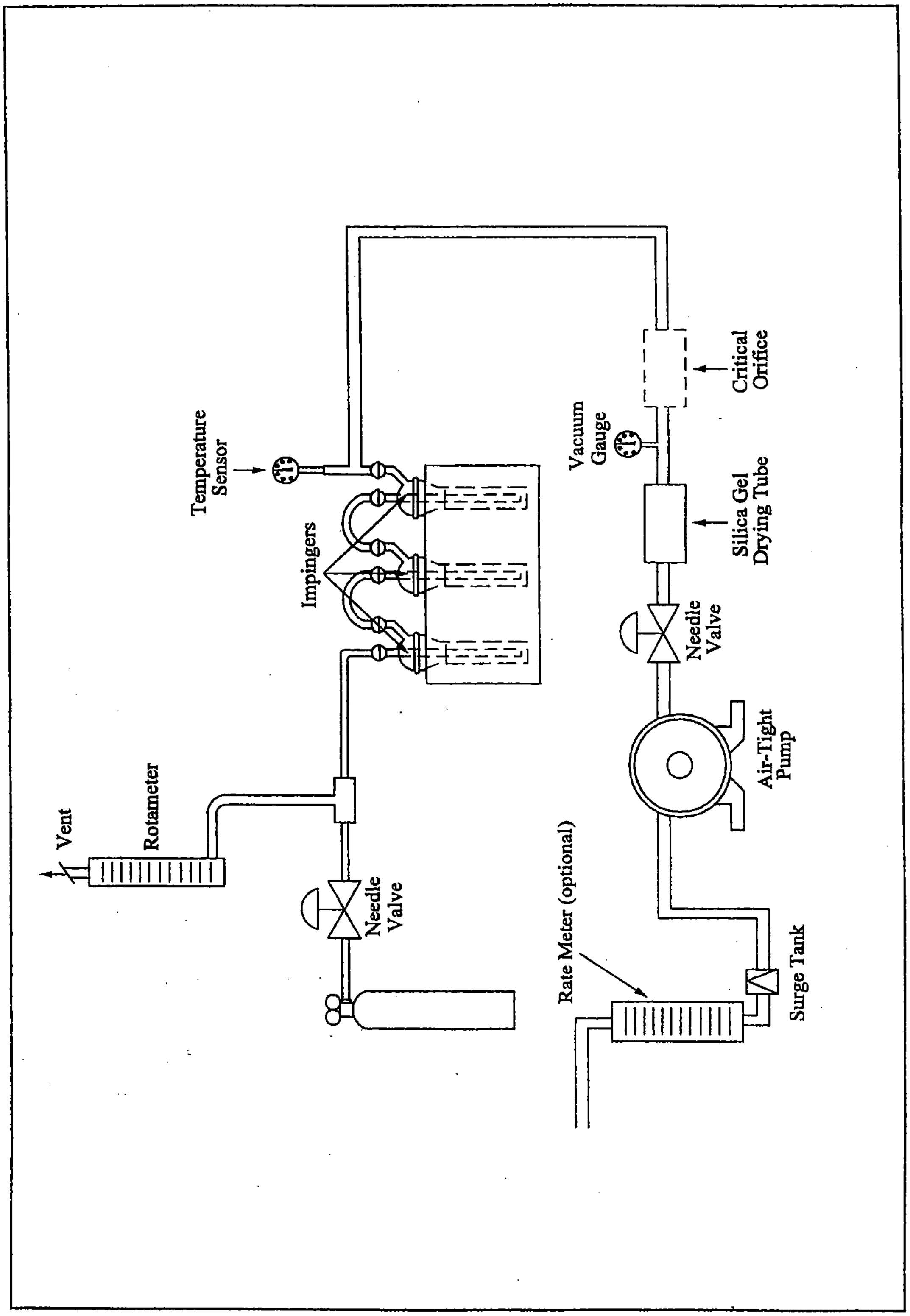

Figure 16A-4. Recovery Check Gas Sampling Train.

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Date
Critical orifice ID
Soap bubble meter volume, Vsb liters
Time, θsb
Run no. 1 min sec
Run no. 2 min sec
Run no. 3 min sec
Average min sec
Covert the seconds to fraction of minute:
Time = min + Sec/60 = min
Barometric pressure, P_{bar} = mm Hg
Ambient temperature, t_{amb} = 273 + °C = °K = mm Hg. (This should be approximately 0.4 times barometric pressure.)
Pump vacuum,

$$V_{sb(std)} = \frac{V_{sb}\,T_{std}\,P_{bar}\left(10^{-3}\right)}{T_{amb}\,P_{std}}$$

$$= \text{----------} \text{ liters}$$

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

$$= \text{----------} \text{ liters/min}$$

Table 16A–1. Critical Orifice Calibration Data

Method 16B—Determination of Total Reduced Sulfur Emissions From Stationary Sources

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a knowledge of at least the following additional test methods: Method 6C, Method 16, and Method 16A.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.
Total reduced sulfur (TRS) including:	N/A
Dimethyl disulfide (DMDS), [$(CH_3)_2S_2$]	62–49–20
Dimethyl sulfide (DMS), [$(CH_3)_2S$]	75–18–3
Hydrogen sulfide (H_2S)	7783–06–4
Methyl mercaptan (MeSH), [CH_4S]	74–93–1
Reported as: Sulfur dioxide (SO_2)	7449–09–5

1.2 Applicability. This method is applicable for determining TRS emissions from recovery furnaces (boilers), lime kilns, and smelt dissolving tanks at kraft pulp mills, and from other sources when specified in an applicable subpart of the regulations. The flue gas must contain at least 1 percent oxygen for complete oxidation of all TRS to SO_2.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 An integrated gas sample is extracted from the stack. The SO_2 is removed selectively from the sample using a citrate buffer solution. The TRS compounds are then thermally oxidized to SO_2 and analyzed as SO_2 by gas chromatography (GC) using flame photometric detection (FPD).

3.0 Definitions. [Reserved]

4.0 Interferences

4.1 Reduced sulfur compounds other than those regulated by the emission standards, if present, may be measured by this method. Therefore, carbonyl sulfide, which is partially oxidized to SO_2 and may be present in a lime kiln exit stack, would be a positive interferant.

4.2 Particulate matter from the lime kiln stack gas (primarily calcium carbonate) can cause a negative bias if it is allowed to enter the citrate scrubber; the particulate matter will cause the pH to rise and H_2S to be absorbed before oxidation. Proper use of the particulate filter, described in Section 6.1.3 of Method 16A, will eliminate this interference.

4.3 Carbon monoxide (CO) and carbon dioxide (CO_2) have substantial desensitizing effects on the FPD even after dilution. Acceptable systems must demonstrate that they have eliminated this interference by some procedure such as eluting these compounds before the SO_2. Compliance with this requirement can be demonstrated by submitting chromatograms of calibration gases with and without CO_2 in diluent gas. The CO_2 level should be approximately 10 percent for the case with CO_2 present. The two chromatograms should show agreement within the precision limits of Section 13.0.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hydrogen Sulfide (H_2S). A flammable, poisonous gas with the odor of rotten eggs. H_2S is extremely hazardous and can cause collapse, coma, and death within a few seconds of one or two inhalations at sufficient concentrations. Low concentrations irritate the mucous membranes and may cause nausea, dizziness, and headache after exposure.

6.0 Equipment and Supplies

6.1 Sample Collection. The sampling train is shown in Figure 16B–1. Modifications to the apparatus are accepted provided the system performance check in Section 8.4.1 is met.

6.1.1 Probe, Probe Brush, Particulate Filter, SO_2 Scrubber, Combustion Tube, and Furnace. Same as in Method 16A, Sections 6.1.1 to 6.1.6.

6.1.2 Sampling Pump. Leakless Teflon-coated diaphragm type or equivalent.

6.2 Analysis.

6.2.1 Dilution System (optional), Gas Chromatograph, Oven, Temperature Gauges, Flow System, Flame Photometric Detector, Electrometer, Power Supply, Recorder, Calibration System, Tube Chamber, Flow System, and Constant Temperature Bath. Same as in Method 16, Sections 6.2.1, 6.2.2, and 6.3.

6.2.2 Gas Chromatograph Columns. Same as in Method 16, Section 6.2.3. Other columns with demonstrated ability to resolve SO_2 and be free from known interferences are acceptable alternatives. Single column systems such as a 7-ft Carbsorb B HT 100 column have been found satisfactory in resolving SO_2 from CO_2.

7.0 Reagents and Standards

Same as in Method 16, Section 7.0, except for the following:

7.1 Calibration Gas. SO_2 permeation tube gravimetrically calibrated and certified at some convenient operating temperature. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquefied gaseous substance is enclosed. The enclosed gas permeates through the tubing wall at a constant rate. When the temperature is constant, calibration gases covering a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. In place of SO_2 permeation tubes, cylinder gases containing SO_2 in nitrogen may be used for calibration. The cylinder gas concentration must be verified according to Section 8.2.1 of Method 6C. The calibration gas is used to calibrate the GC/FPD system and the dilution system.

7.2 Recovery Check Gas.

7.2.1 Hydrogen sulfide (100 parts per million by volume (ppmv) or less) in nitrogen, stored in aluminum cylinders. Verify the concentration by Method 11, the procedure discussed in Section 16.0 of Method 16A, or gas chromatography where the instrument is calibrated with an H_2S permeation tube as described below. For the wet-chemical methods, the standard deviation should not exceed 5 percent on at least three 20-minute runs.

7.2.2 Hydrogen sulfide recovery gas generated from a permeation device gravimetrically calibrated and certified at some convenient operation temperature may be used. The permeation rate of the device must be such that at a dilution gas flow rate of 3 liters/min (64 ft^3/hr), an H_2S concentration in the range of the stack gas or within 20 percent of the emission standard can be generated.

7.3 Combustion Gas. Gas containing less than 50 ppbv reduced sulfur compounds and less than 10 ppmv total hydrocarbons. The gas may be generated from a clean-air system that purifies ambient air and consists of the following components: diaphragm pump, silica gel drying tube, activated charcoal tube, and flow rate measuring device. Gas from a compressed air cylinder is also acceptable.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Procedures. Same as in Method 15, Section 8.1.

8.2 Sample Collection. Before any source sampling is performed, conduct a system performance check as detailed in Section 8.4.1 to validate the sampling train components and procedures. Although this test is optional, it would significantly reduce the possibility of rejecting tests as a result of failing the post-test performance check. At the completion of the pretest system performance check, insert the sampling probe into the test port making certain that no dilution air enters the stack though the port. Condition the entire system with sample for a minimum of 15 minutes before beginning analysis. If the sample is diluted, determine the dilution factor as in Section 10.4 of Method 15.

8.3 Analysis. Inject aliquots of the sample into the GC/FPD analyzer for analysis. Determine the concentration of SO_2 directly from the calibration curves or from the equation for the least-squares line.

8.4. Post-Test Procedures

8.4.1 System Performance Check. Same as in Method 16A, Section 8.5. A sufficient number of sample injections should be made so that the precision requirements of Section 13.2 are satisfied.

8.4.2 Determination of Calibration Drift. Same as in Method 15, Section 8.3.2.

9.0 Quality Control

Section	Quality control measure	Effect
8.2, 8.3	System performance check	Ensure validity of sampling train components and analytical procedure.
8.1	Sampling equipment leak-check and calibration	Ensure accurate measurement of stack gas flow rate, sample volume.
10.0	Analytical calibration	Ensure precision of analytical results within 5 percent.

10.0 Calibration

Same as in Method 16, Section 10, except SO_2 is used instead of H_2S.

11.0 Analytical Procedure

11.1 Sample collection and analysis are concurrent for this method (see section 8.3).

12.0 Data Analysis and Calculations

12.1 Nomenclature.

C_{SO2} = Sulfur dioxide concentration, ppmv.

C_{TRS} = Total reduced sulfur concentration as determined by Equation 16B–1, ppmv.

d = Dilution factor, dimensionless.
N = Number of samples.

12.2 SO_2 Concentration. Determine the concentration of SO_2, C_{SO_2}, directly from the calibration curves. Alternatively, the concentration may be calculated using the equation for the least-squares line.

12.3 TRS Concentration.

$$C_{TRS} = C_{SO_2} d \qquad \text{Eq. 16B-1}$$

12.4 Average TRS Concentration

$$\text{Avg.}\, C_{TRS} = \frac{\sum_{i=1}^{n} C_{TRS}}{N} \qquad \text{Eq. 16B-2}$$

13.0 Method Performance.

13.1 Range and Sensitivity. Coupled with a GC using a 1-ml sample size, the maximum limit of the FPD for SO_2 is approximately 10 ppmv. This limit is extended by diluting the sample gas before analysis or by reducing the sample aliquot size. For sources with emission levels between 10 and 100 ppm, the measuring range can be best extended by reducing the sample size.

13.2 GC/FPD Calibration and Precision. A series of three consecutive injections of the sample calibration gas, at any dilution, must produce results which do not vary by more than 5 percent from the mean of the three injections.

13.3 Calibration Drift. The calibration drift determined from the mean of the three injections made at the beginning and end of any run or series of runs within a 24-hour period must not exceed 5 percent.

13.4 System Calibration Accuracy. Losses through the sample transport system must be measured and a correction factor developed to adjust the calibration accuracy to 100 percent.

13.5 Field tests between this method and Method 16A showed an average difference of less than 4.0 percent. This difference was not determined to be significant.

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Same as in Method 16, Section 16.0.
2. National Council of the Paper Industry for Air and Stream Improvement, Inc, A Study of TRS Measurement Methods. Technical Bulletin No. 434. New York, NY. May 1984. 12p.
3. Margeson, J.H., J.E. Knoll, and M.R. Midgett. A Manual Method for TRS Determination. Draft available from the authors. Source Branch, Quality Assurance Division, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

BILLING CODE 6560–50–P

17.0 Tables, Diagrams, Flowcharts, and Validation Data

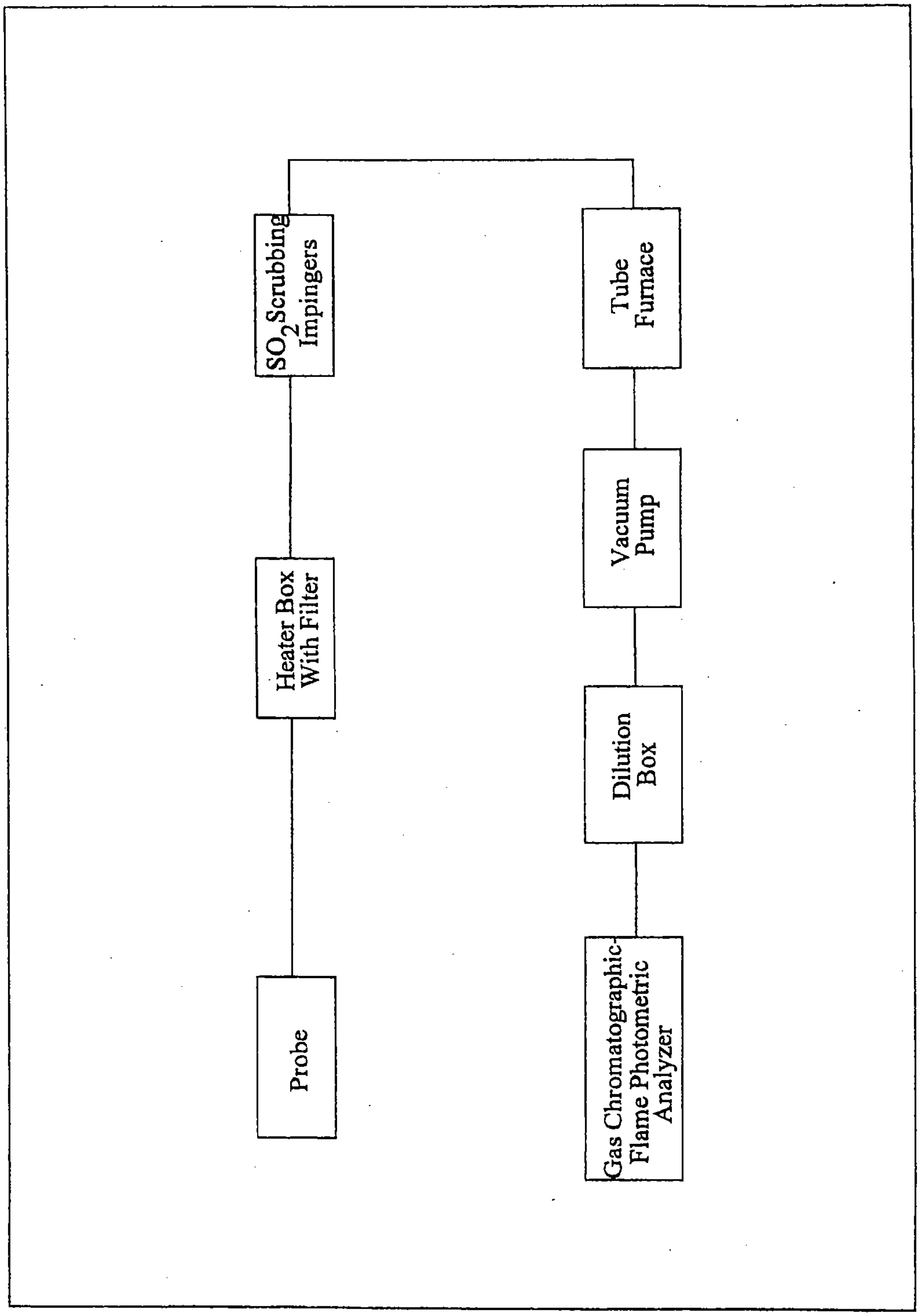

Figure 16B-1. Method 16B Sampling Train.

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Method 17—Determination of Particulate Matter Emissions From Stationary Sources

Note: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

Note: Particulate matter is not an absolute quantity. It is a function of temperature and pressure. Therefore, to prevent variability in PM emission regulations and/or associated test methods, the temperature and pressure at which PM is to be measured must be carefully defined. Of the two variables (*i.e.*, temperature and pressure), temperature has the greater effect upon the amount of PM in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible. In Method 5, 120 °C (248 °F) is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standard, PM is defined with respect to temperature. In order to maintain a collection temperature of 120 °C (248 °F), Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where PM concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and the heating systems, and to sample at stack temperature.

1.2 Applicability. This method is applicable for the determination of PM emissions, where PM concentrations are known to be independent of temperature over the normal range of temperatures characteristic of emissions from a specified source category. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 8.1.2).

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The PM mass is determined gravimetrically after the removal of uncombined water.

3.0 Definitions

Same as Method 5, Section 3.0.

4.0 Interferences. [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 17–1. The sampling train components and operation and maintenance are very similar to Method 5, which should be consulted for details.

6.1.1 Probe Nozzle, Differential Pressure Gauge, Metering System, Barometer, Gas Density Determination Equipment. Same as in Method 5, Sections 6.1.1, 6.1.4, 6.1.8, 6.1.9, and 6.1.10, respectively.

6.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel. If a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. Other holder and gasket materials may be used, subject to the approval of the Administrator. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.

6.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

6.1.4 Pitot Tube. Same as in Method 5, Section 6.1.3.

6.1.4.1 It is recommended (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 10 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17–1). Note that the 1.9 cm (¾-in.) free-space between the nozzle and pitot tube shown in Figure 17–1, is based on a 1.3 cm (½-in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (¾-in.) with the largest sized nozzle in place.

6.1.4.2 Source-sampling assemblies that do not meet the minimum spacing requirements of Figure 17–1 (or the equivalent of these requirements, *e.g.*, Figure 2–4 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Administrator.

6.1.5 Condenser. It is recommended that the impinger system or alternatives described in Method 5 be used to determine the moisture content of the stack gas. Flexible tubing may be used between the probe extension and condenser. Long tubing lengths may affect the moisture determination.

6.2 Sample Recovery. Probe-liner and probe-nozzle brushes, wash bottles, glass sample storage containers, petri dishes, graduated cylinder and/or balance, plastic storage containers, funnel and rubber policeman, funnel. Same as in Method 5, Sections 6.2.1 through 6.2.8, respectively.

6.3 Sample Analysis. Glass weighing dishes, desiccator, analytical balance, balance, beakers, hygrometer, temperature sensor. Same as in Method 5, Sections 6.3.1 through 6.3.7, respectively.

7.0 Reagents and Standards

7.1 Sampling. Filters, silica gel, water, crushed ice, stopcock grease. Same as in Method 5, Sections 7.1.1, 7.1.2, 7.1.3, 7.1.4, and 7.1.5, respectively. Thimble glass fiber filters may also be used.

7.2 Sample Recovery. Acetone (reagent grade). Same as in Method 5, Section 7.2.

7.3 Sample Analysis. Acetone and Desiccant. Same as in Method 5, Sections 7.3.1 and 7.3.2, respectively.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Sampling.

8.1.1 Pretest Preparation. Same as in Method 5, Section 8.1.1.

8.1.2 Preliminary Determinations. Same as in Method 5, Section 8.1.2, except as follows: Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17–2. Calculate the estimated cross-section blockage, as shown in Figure 17–2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options exist: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Administrator (see also Reference 1 in Section 17.0). Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

8.1.3 Preparation of Sampling Train. Same as in Method 5, Section 8.1.3, except the following: Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Assemble the train as in Figure 17–1, using a very light coat of silicone grease on all ground glass joints and greasing only the outer portion (see APTD–0576) to avoid possibility of contamination by the silicone grease. Place crushed ice around the impingers.

8.1.4 Leak-Check Procedures. Same as in Method 5, Section 8.1.4, except that the filter holder is inserted into the stack during the sampling train leak-check. To do this, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream.

8.1.5 Sampling Train Operation. The operation is the same as in Method 5. Use a data sheet such as the one shown in Figure 5–3 of Method 5, except that the filter holder temperature is not recorded.

8.1.6 Calculation of Percent Isokinetic. Same as in Method 5, Section 12.11.

8.2 Sample Recovery.

8.2.1 Proper cleanup procedure begins as soon as the probe extension assembly is removed from the stack at the end of the sampling period. Allow the assembly to cool.

8.2.2 When the assembly can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, forcing condenser water backward.

8.2.3 Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate, if present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser; cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

8.2.4 Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

8.2.5 Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone from the wash bottle being used and place it in a glass sample container labeled "acetone blank." Inspect the train prior to and during disassembly and not any abnormal conditions. Treat the sample as discussed in Method 5, Section 8.2.

9.0 Quality Control. [Reserved]

10.0 Calibration and Standardization

The calibrations of the probe nozzle, pitot tube, metering system, temperature sensors, and barometer are the same as in Method 5, Sections 10.1 through 10.3, 10.5, and 10.6, respectively.

11.0 Analytical Procedure

Same as in Method 5, Section 11.0. Analytical data should be recorded on a form similar to that shown in Figure 5–6 of Method 5.

12.0 Data Analysis and Calculations.

Same as in Method 5, Section 12.0.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

Same as in Method 5, Section 16.0.

17.0 References

Same as in Method 5, Section 17.0, with the addition of the following:

1. Vollaro, R.F. Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, NC. November 1976.

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18.0 Tables, Diagrams, Flowcharts, and Validation Data

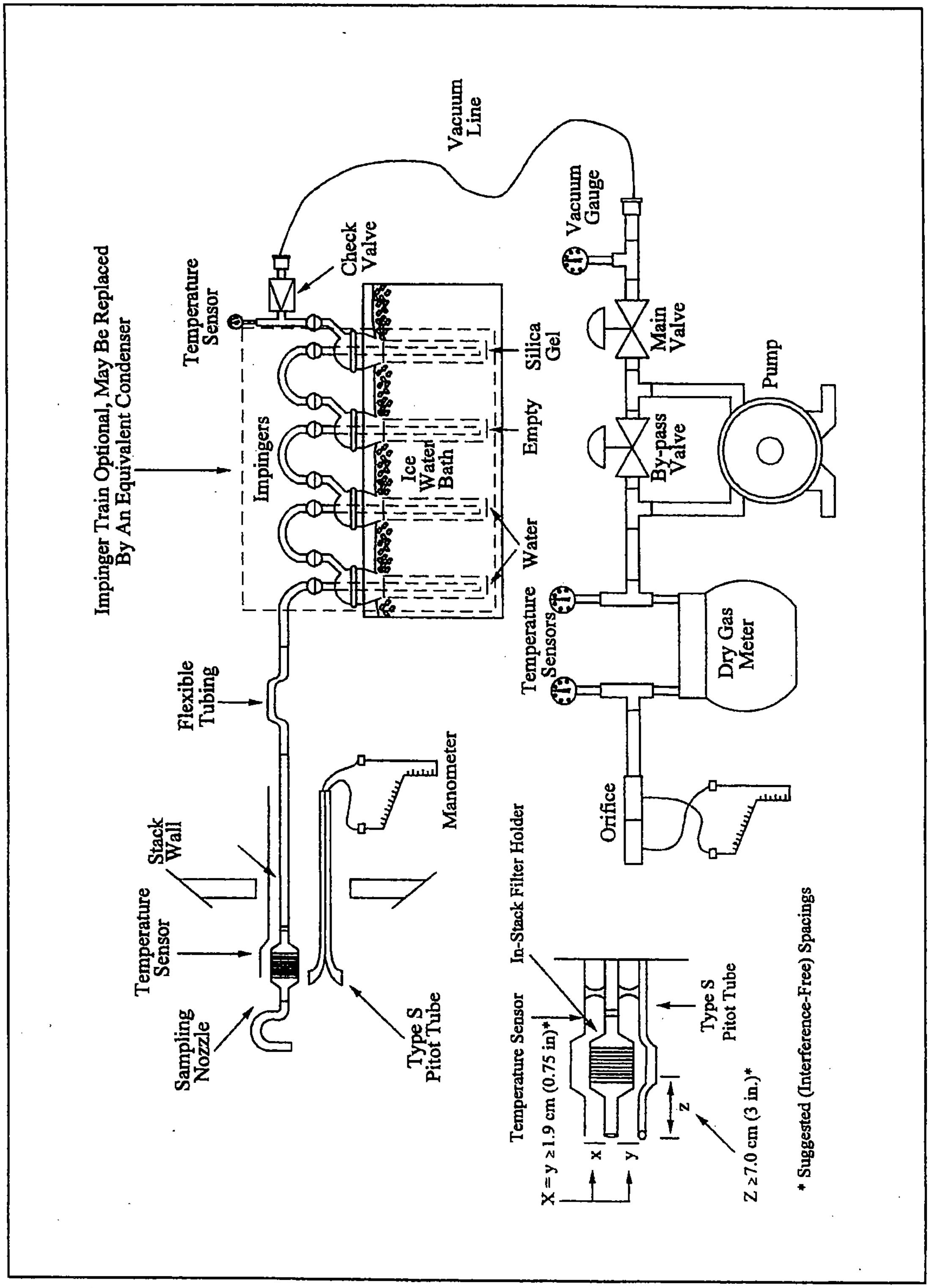
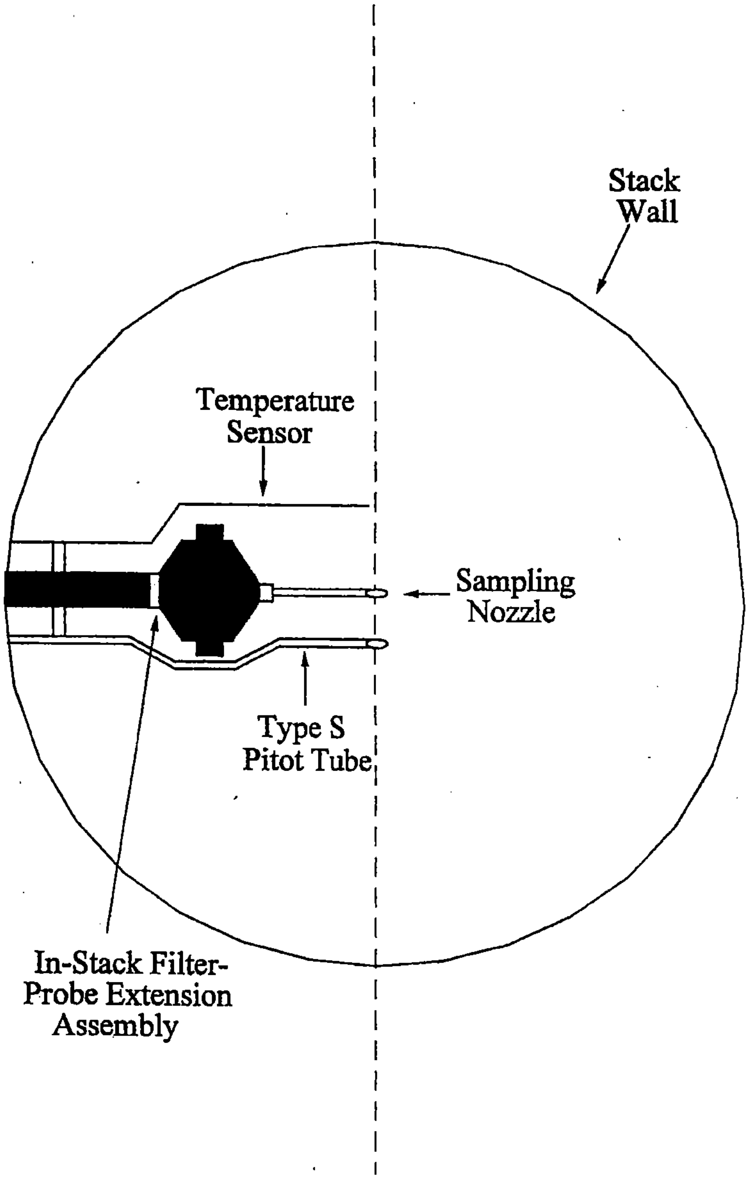


Figure 17-1. Particulate Matter Sampling Train with In-Stack Filter.



$$\text{Estimated Blockage (\%)} = \left[\frac{\text{Shaded Area}}{\text{Duct Area}}\right] \times 100$$

Figure 17-2. Projected-Area Model of Cross-Section Blockage (approximate average for a sample traverse) Caused by an In-Stack Filter Holder-Probe Extension Assembly.

BILLING CODE 6560-50-C

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

Note: This method is not inclusive with respect to specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3.

Note: 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.

1.0 Scope and Application

1.1 Analyte. Total gaseous organic compounds.

1.2 Applicability.

1.2.1 This method is designed to measure gaseous organics emitted from an industrial source. While designed for ppm level sources, some detectors are quite capable of detecting compounds at ambient levels, *e.g.*, ECD, ELCD, and helium ionization detectors. Some other types of detectors are evolving such that the sensitivity and applicability may well be in the ppb range in only a few years.

1.2.2 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.

1.3 Range. The lower range of this method is determined by the sampling system; adsorbents may be used to concentrate the sample, thus lowering the limit of detection below the 1 part per million (ppm) typically achievable with direct interface or bag sampling. The upper limit is governed by GC detector saturation or column overloading; the upper range can be extended by dilution of sample with an inert gas or by using smaller volume gas sampling loops. The upper limit can also be governed by condensation of higher boiling compounds.

1.4 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.

2.0 Summary of Method

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

3.0 Definitions. [Reserved]

4.0 Interferences

4.1 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.

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

4.3 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.

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

4.5 The gas chromatograph run time must be sufficient to clear all eluting peaks from the column before proceeding to the next run (in order to prevent sample carryover).

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method. The analyzer users manual should be consulted for specific precautions to be taken with regard to the analytical procedure.

6.0 Equipment and Supplies

6.1 Equipment needed for the presurvey sampling procedure can be found in Section 16.1.1.

6.2 Equipment needed for the integrated bag sampling and analysis procedure can be found in Section 8.2.1.1.1.

6.3 Equipment needed for direct interface sampling and analysis can be found in Section 8.2.2.1.

6.4 Equipment needed for the dilution interface sampling and analysis can be found in Section 8.2.3.1.

6.5 Equipment needed for adsorbent tube sampling and analysis can be found in Section 8.2.4.1.

7.0 Reagents and Standards

7.1 Reagents needed for the presurvey sampling procedure can be found in Section 16.1.2.

7.2 Quality Assurance Audit Samples. When making compliance determinations, and upon availability, an audit sample may be obtained from the appropriate EPA Regional Office or from the responsible enforcement authority.

Note: The responsible enforcement autority should be notified at least 30 days prior to the test date to allow sufficient time for sample delivery.

8.0 Sample Collection, Preservation, Storage, and Transport

8.2 Final Sampling and Analysis Procedure. Considering safety (flame hazards) and the source conditions, select an appropriate sampling and analysis procedure (Section 8.2.1, 8.2.2, 8.2.3 or 8.2.4). 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.

8.2.1 Integrated Bag Sampling and Analysis.

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

8.2.1.1.1 Apparatus.

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

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

8.2.1.1.1.3 Needle Valve. To control gas flow.

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

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

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

8.2.1.1.2 Sampling Procedure. To obtain a sample, assemble the sample train as shown in Figure 18–9. Leak-check both the bag and the container. Connect the vacuum line from the needle valve to the Teflon sample line from the probe. Place the end of the probe at the centroid of the stack or at a point no closer to the walls than 1 m, and start the pump. Set the flow rate so that the final volume of the sample is approximately 80 percent of the bag capacity. 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. Record the time lapsed between sample collection and analysis, and then conduct the recovery procedure in Section 8.4.2.

8.2.1.2 Direct Pump Sampling Procedure. Follow 8.2.1.1, except place the pump and needle valve between the probe and the bag. Use a pump and needle valve constructed of inert material not affected by the stack gas. Leak-check the system, and then purge with stack gas before connecting to the previously evacuated bag.

8.2.1.3 Explosion Risk Area Bag Sampling Procedure. Follow 8.2.1.1 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.

8.2.1.4 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.

8.2.1.4.1 First Alternative Procedure. Heat the box containing the sample bag to 120 °C (±5 °C). 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 120 °C (±5 °C) 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.

8.2.1.4.2 Second Alternative 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 10.1.2.2), but eliminate the midget 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 before sampling each bag through dilution and analysis of gases of known concentration.

8.2.1.5 Analysis of Bag Samples.

8.2.1.5.1 Apparatus. Same as Section 8.1. A minimum of three gas standards are required.

8.2.1.5.2 Procedure.

8.2.1.5.2.1 Establish proper GC operating conditions as described in Section 10.2, and record all data listed in Figure 18–7. Prepare the GC so that gas can be drawn through the sample valve. Flush the sample loop with calibration gas mixture, and activate the valve (sample pressure at the inlet to the GC introduction valve should be similar during calibration as during actual sample analysis). Obtain at least three chromatograms for the mixture. The results are acceptable when the peak areas for the three injections agree to within 5 percent of their average. If they do not agree, run additional samples or correct the analytical techniques until this requirement is met. Then analyze the other two calibration mixtures in the same manner. Prepare a calibration curve as described in Section 10.2.

8.2.1.5.2.2 Analyze the two field audit samples as described in Section 9.2 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.

8.2.1.5.2.3 Analyze the three source gas samples by connecting each bag to the sampling valve with a piece of Teflon tubing identified with that bag. Analyze each bag sample three times. Record the data in Figure 18–11. If certain items do not apply, use the notation "N.A." If the bag has been maintained at an elevated temperature as described in Section 8.2.1.4, determine the stack gas water content by Method 4. After all samples have been analyzed, repeat the analysis of the mid-level calibration gas for each compound. Compare the average response factor of the pre- and post-test analysis for each compound. If they differ by >5percent, analyze the other calibration gas levels for that compound, and prepare a calibration curve using all the pre- and post-test calibration gas mixture values. If the two response factor averages (pre- and post-test) differ by less than 5 percent from their mean value, the tester has the option of using only the pre-test calibration curve to generate the concentration values.

8.2.1.6 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.) If the bag has been maintained at an elevated temperature as described in Section 8.2.1.4, determine the stack gas water content by Method 4.

8.2.1.7 Audit Gas Analysis. Immediately prior to the analysis of the stack gas samples, perform audit analyses as described in Section 9.2.

8.2.1.8 Emission Calculations. From the calibration curve described in Section 8.2.1.5, select the value of C_s that corresponds to the peak area. Calculate the concentration C_c in ppm, dry basis, of each organic in the sample using Equation 18–5 in Section 12.6.

8.2.2 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 falls within the linear range of the detector. Adhere to all safety requirements with this method.

8.2.2.1 Apparatus.

8.2.2.1.1 Probe. Constructed of stainless steel, Pyrex glass, or Teflon tubing as dictated by duct temperature and reactivity of target compounds. A filter or glass wool plug may be needed if particulate is present in the stack gas. If necessary, heat the probe with heating tape or a special heating unit capable of maintaining a temperature greater than 110 °C.

8.2.2.1.2 Sample Lines. 6.4-mm OD (or other diameter as needed) Teflon lines, heat-traced to prevent condensation of material (greater than 110 °C).

8.2.2.1.3 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.

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

8.2.2.1.5 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.

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

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

8.2.2.1.8 Flowmeter. Of suitable range to measure sampling rate.

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

8.2.2.1.10 Gas Cylinders. Carrier gas, oxygen and fuel as needed to run GC and detector.

8.2.2.1.11 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.

8.2.2.1.12 Recorder/Integrator. To record results.

8.2.2.2 Procedure. Calibrate the GC using the procedures in Section 8.2.1.5.2.1. To obtain a stack gas 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 target temperature as indicated on the thermocouple readout device, control the heating to maintain a temperature greater than 110 °C. Conduct a 3-point calibration of the GC by analyzing each gas mixture in triplicate. Generate a calibration curve. Place the inlet of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and draw source gas into the probe, heated line, and sample loop. After thorough flushing, analyze the stack gas sample using the same conditions as for the calibration gas mixture. For each run, sample, analyze, and record five consecutive samples. A test consists of three runs (five samples per run times three runs, for a total of fifteen samples). After all samples have been analyzed, repeat the analysis of the mid-level calibration gas for each compound. For each calibration standard, compare the pre- and post-test average response factors (RF) for each compound. If the two calibration RF values (pre- and post-analysis) differ by more than 5 percent from their mean value, then analyze the other calibration gas levels for that compound and determine the stack gas sample concentrations by comparison to both calibration curves (this is done by preparing a calibration curve using all the pre and post-test calibration gas mixture values). If the two calibration RF values differ by less than 5 percent from their mean value, the tester has the option of using only the pre-test calibration curve to generate the concentration values. 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.

8.2.2.3 Determination of Stack Gas Moisture Content. Use Method 4 to measure the stack gas moisture content.

8.2.2.4 Quality Assurance. Same as Section 8.2.1.7. Introduce the audit gases in the sample line immediately following the probe.

8.2.2.5 Emission Calculations. Same as Section 8.2.1.8.

8.2.3 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 8.2.2, 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.

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

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

8.2.3.1.2 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.

8.2.3.1.3 Valves. Two Teflon three-way valves, suitable for connecting to Teflon tubing.

8.2.3.1.4 Flowmeters. Two, for measurement of diluent gas.

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

8.2.3.1.6 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. 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.

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

8.2.3.2 Procedure.

8.2.3.2.1 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, or to a point no closer to the walls than 1 m. Measure the source temperature, and adjust all heating units to a temperature 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds (greater than 110 °C). Calibrate the GC through the dilution system by following the procedures in Section 8.2.1.5.2.1. Determine the concentration of the diluted calibration gas using the dilution factor and the certified concentration of the calibration gas. Record the pertinent data on the data sheet shown in Figure 18–11.

8.2.3.2.2 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.

8.2.3.2.3 Analyze the 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.

8.2.3.3 Determination of Stack Gas Moisture Content. Same as Section 8.2.2.3.

8.2.3.4 Quality Assurance. Same as Section 8.2.2.4.

8.2.3.5 Emission Calculations. Same as section 8.2.2.5, with the dilution factor applied.

8.2.4 Adsorption Tube Procedure. Any commercially available adsorbent is allowed for the purposes of this method, as long as the recovery study criteria in Section 8.4.3 are met. Help in choosing the adsorbent may be found by calling the distributor, or the tester may refer to National Institute for Occupational Safety and Health (NIOSH) methods for the particular organics to be sampled. For some adsorbents, the principal interferent will be water vapor. If water vapor is thought to be a problem, the tester may place a midget impinger in an ice bath before the adsorbent tubes. If this option is chosen, the water catch in the midget impinger shall be analyzed for the target compounds. Also, the spike for the recovery study (in Section 8.4.3) shall be conducted in both the midget impinger and the adsorbent tubes. The combined recovery (add the recovered amount in the impinger and the adsorbent tubes to calculate R) shall then meet the criteria in Section 8.4.3.

Note: Post-test leak-checks are not allowed for this technique since this can result in sample contamination.

8.2.4.1 Additional Apparatus. The following items (or equivalent) are suggested.

8.2.4.1.1 Probe. 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-of-stack, heated to stack temperature) to remove particulate matter. In most instances, a plug of glass wool is a satisfactory filter.

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

8.2.4.1.3 Leakless Sample Pump. Flow controlled, constant rate pump, with a set of limiting (sonic) orifices.

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

8.2.4.1.5 Stopwatch. To time sampling and pump rate calibration.

8.2.4.1.6 Adsorption Tubes. Precleaned adsorbent, with mass of adsorbent to be determined by calculating breakthrough volume and expected concentration in the stack.

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

8.2.4.1.8 Rotameter. O to 100 cc/min, to detect changes in flow rate during sampling.

8.2.4.2 Sampling and Analysis.

8.2.4.2.1 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.

8.2.4.2.2 Use a sample probe, if required, to obtain the sample at the centroid of the duct, or at a point no closer to the walls than 1 m. Minimize the length of flexible tubing between the probe and adsorption tubes. Several adsorption tubes can be connected in series, if the extra adsorptive capacity is needed. Adsorption tubes should be maintained vertically during the test in order to prevent channeling. 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. 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. Calibrate the instrument. Analyze the audit samples (see Section 16.1.4.3), then the emission samples.

8.2.4.3 Standards and Calibration. If using thermal desorption, obtain calibration gases using the procedures in Section 10.1. If using solvent extraction, prepare liquid standards in the desorption solvent. 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 using the procedures in Section 8.2.1.5.2.1.

8.2.4.4 Quality Assurance.

8.2.4.4.1 Determine the recovery efficiency of the pollutants of interest according to Section 8.4.3.

8.2.4.4.2 Determination of Sample Collection Efficiency (Optional). If sample breakthrough is thought to be a problem, a routine procedure for determining breakthrough is to analyze the primary and backup portions of the adsorption tubes separately. If the backup portion exceeds 10 percent of the total amount (primary and back-up), it is usually a sign of sample breakthrough. For the purposes of this method, only the recovery efficiency value (Section 8.4.3) is used to determine the appropriateness of the sampling and analytical procedure.

8.2.4.4.3 Volume Flow Rate Checks. Perform this check immediately after sampling with all sampling train components in place. Use the bubble-tube flowmeter to measure the pump volume flow rate with the orifice used in the test sampling, and record 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.

8.2.4.4.4 Calculations. 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 according to the applicable procedure in Section 8.4.3. Report results as ppm by volume, dry basis.

8.3 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.

8.4 Recovery Study. After conducting the presurvey and identifying all of the pollutants of interest, conduct the appropriate recovery study during the test based on the sampling system chosen for the compounds of interest.

8.4.1 Recovery Study for Direct Interface or Dilution Interface Sampling. If the procedures in Section 8.2.2 or 8.2.3 are to be used to analyze the stack gas, conduct the calibration procedure as stated in Section 8.2.2.2 or 8.2.3.2, as appropriate. Upon successful completion of the appropriate calibration procedure, attach the mid-level calibration gas for at least one target compound to the inlet of the probe or as close as possible to the inlet of the probe, but before the filter. Repeat the calibration procedure by sampling and analyzing the mid-level calibration gas through the entire sampling and analytical system in triplicate. The mean of the calibration gas response sampled through the probe shall be within 10 percent of the analyzer response. If the difference in the two means is greater than 10 percent, check for leaks throughout the sampling system and repeat the analysis of the standard through the sampling system until this criterion is met.

8.4.2 Recovery Study for Bag Sampling.

8.4.2.1 Follow the procedures for the bag sampling and analysis in Section 8.2.1. After analyzing all three bag samples, choose one of the bag samples and tag this bag as the spiked bag. Spike the chosen bag sample with a known mixture (gaseous or liquid) of all of the target pollutants. The theoretical concentration, in ppm, of each spiked compound in the bag shall be 40 to 60 percent of the average concentration measured in the three bag samples. If a target compound was not detected in the bag samples, the concentration of that compound to be spiked shall be 5 times the limit of detection for that compound. Store the spiked bag for the same period of time as the bag samples collected in the field. After the appropriate storage time has passed, analyze the spiked bag three times. Calculate the average fraction recovered (R) of each spiked target compound with the equation in Section 12.7.

8.4.2.2 For the bag sampling technique to be considered valid for a compound, $0.70 \le R \le 1.30$. If the R value does not meet this criterion for a target compound, the sampling technique is not acceptable for that compound, and therefore another sampling technique shall be evaluated for acceptance (by repeating the recovery study with another sampling technique). Report the R value in the test report and correct all field measurements with the calculated R value for that compound by using the equation in Section 12.8.

8.4.3 Recovery Study for Adsorption Tube Sampling. If following the adsorption tube procedure in Section 8.2.4, conduct a recovery study of the compounds of interest during the actual field test. Set up two identical sampling trains. Collocate the two sampling probes in the stack. The probes shall be placed in the same horizontal plane, where the first probe tip is 2.5 cm from the outside edge of the other. One of the sampling trains shall be designated the spiked train and the other the unspiked train. Spike all of the compounds of interest (in gaseous or liquid form) onto the adsorbent tube(s) in the spiked train before sampling. The mass of each spiked compound shall be 40 to 60 percent of the mass expected to be collected with the unspiked train. Sample the stack gas into the two trains simultaneously. Analyze the adsorbents from the two trains utilizing identical analytical procedures and instrumentation. Determine the fraction of spiked compound recovered (R) using the equations in Section 12.9.

8.4.3.1 Repeat the procedure in Section 8.4.3 twice more, for a total of three runs. In order for the adsorbent tube sampling and analytical procedure to be acceptable for a compound, $0.70 \le R \le 1.30$ (R in this case is the average of three runs). If the average R value does not meet this criterion for a target compound, the sampling technique is not acceptable for that compound, and therefore another sampling technique shall be evaluated for acceptance (by repeating the recovery study with another sampling technique). Report the R value in the test report and correct all field measurements with the calculated R value for that compound by using the equation in Section 12.8.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures

Section	Quality control measure	Effect
8.4.1	Recovery study for direct interface or dilution interface sampling.	Ensure that there are no significant leaks in the sampling system.
8.4.2	Recovery study for bag sampling	Demonstrate that proper sampling/analysis procedures were selected.
8.4.3	Recovery study for adsorption tube sampling	Demonstrate that proper sampling/analysis procedures were selected.

9.2 Quality Assurance for Laboratory Procedures. Immediately after the preparation of the calibration curves, the analysis audit described in 40 CFR Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis," should be performed if audit materials are available. The information required to document the analysis of the audit samples has been included on the example data sheets shown in Figures 18–3 and 18–7. The audit analyses should agree with the certified audit concentrations within 10 percent. Audit sample results shall be submitted according to directions provided with the audit samples.

10.0 Calibration and Standardization.

10.1 Calibration Standards. Obtain calibration gas standards for each target compound to be analyzed. Commercial cylinder gases certified by the manufacturer to be accurate to within 1 percent of the certified label value are preferable, although cylinder gases certified by the manufacturer to 2 percent accuracy are allowed. Another option allowed by this method is for the tester to obtain high concentration certified cylinder gases and then use a dilution system meeting the requirements of Test Method 205, 40 CFR Part 51, Appendix M to make multi-level calibration gas standards. Prepare or obtain enough calibration standards so that there are three different concentrations of each organic compound expected to be measured in the source sample. For each organic compound, select those concentrations that bracket the concentrations expected in the source samples. A calibration standard may contain more than one organic compound. If samples are collected in adsorbent tubes and extracted using solvent extraction, prepare or obtain standards in the same solvent used for the sample extraction procedure. Verify the stability of all standards for the time periods they are used.

10.2 Preparation of Calibration Curves.

10.2.1 Establish proper GC conditions, then flush the sampling loop for 30 seconds. Allow the sample loop pressure to equilibrate to atmospheric pressure, and activate the injection valve. Record the standard concentration, attenuator factor, injection time, chart speed, retention time, peak area, sample loop temperature, column temperature, and carrier gas flow rate. Analyze each standard in triplicate.

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

11.0 Analytical Procedures

11.1 Analysis Development

11.1.1 Selection of GC Parameters

11.1.1.1 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.

Note: Most column manufacturers keep excellent records on 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.

11.1.1.2 Preliminary GC Adjustment. Using the standards and column obtained in Section 11.1.1.1, perform initial tests to determine appropriate GC conditions that provide good resolution and minimum analysis time for the compounds of interest.

11.1.1.3 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.

11.1.1.4 Presurvey Sample Analysis.

11.1.1.4.1 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.

11.1.1.4.2 Use the GC conditions determined by the procedure of Section 11.1.1.2 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.

11.1.1.4.3 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 10.2) to obtain an estimate of the concentrations.

11.1.1.4.4 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.

12.0 Data Analysis and Calculations

12.1 Nomenclature.

B_{ws} = Water vapor content of the bag sample or stack gas, proportion by volume.

C_s = Concentration of the organic from the calibration curve, ppm.

G_v = Gas volume or organic compound injected, ml.

L_v = Liquid volume of organic injected, µl.

M = Molecular weight of organic, g/g-mole.

m_s = Total mass of compound measured on adsorbent with spiked train (µg).

m_u = Total mass of compound measured on adsorbent with unspiked train (µg).

m_v = Mass per volume of spiked compound measured (µg/L).

P_i = Barometric or absolute sample loop pressure at time of sample analysis, mm Hg.

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

P_r = Reference pressure, the barometric pressure or absolute sample loop pressure recorded during calibration, mm Hg.

P_s = Absolute pressure of syringe before injection, mm Hg.

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

q_{c1} = Flow rate of the calibration gas to be diluted in stage 1.

q_{c2} = Flow rate of the calibration gas to be diluted in stage 2.

q_d = Diluent gas flow rate.

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

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

s = Theoretical concentration (ppm) of spiked target compound in the bag.

S = Theoretical mass of compound spiked onto adsorbent in spiked train (µg).

t = Measured average concentration (ppm) of target compound and source sample (analysis results subsequent to bag spiking)

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

T_m = Absolute temperature of dry gas meter, °K.

T_s = Absolute temperature of syringe before injection, °K.

u = Source sample average concentration (ppm) of target compound in the bag (analysis results before bag spiking).

V_m = Gas volume indicated by dry gas meter, liters.

v_s = volume of stack gas sampled with spiked train (L).

v_u = volume of stack gas sampled with unspiked train (L).

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

Y = Dry gas meter calibration factor, dimensionless.

µl = Liquid organic density as determined, g/ml.

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

1000 = Conversion factor, ml/liter.

10^6 = Conversion to ppm.

12.2 Calculate the concentration, C_s, in ppm using the following equation:

$$C_s = \frac{10^6\left(\overline{X}q_c\right)}{q_c + q_d} \qquad \text{Eq. 18-1}$$

12.3 Calculate the concentration, C_s, in ppm of the organic in the final gas mixture using the following equation:

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

12.4 Calculate each organic standard concentration, C_s, in ppm using the following equation:

$$C_s = \frac{G_v \times 10^6 \frac{293}{T_s} \frac{P_s}{760}}{V_m Y \frac{293}{T_m} \frac{P_m}{760} 1000} \quad \text{Eq. 18-3}$$

$$= \frac{G_v \times 10^3 \frac{P_s}{T_s} \frac{T_m}{P_m}}{V_m Y}$$

12.5 Calculate each organic standard concentration, C_s, in ppm using the following equation:

$$C_s = \frac{\frac{L_v}{M} \rho \left(24.055 \times 10^6 \right)}{V_m Y \frac{293}{T_m} \frac{P_m}{760} 1000} = 6.24 \times 10^4 \frac{L_v \rho T_m}{M V_m Y P_m} \quad \text{Eq. 18-4}$$

12.6 Calculate the concentration, C_c, in ppm, dry basis, of each organic is the sample using the following equation:

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r \left(1 - B_{ws} \right)} \quad \text{Eq. 18-5}$$

12.7 Calculate the average fraction recovered (R) of each spiked target compound using the following equation:

$$R = \frac{t - u}{s} \quad \text{Eq. 18-6}$$

12.8 Correct all field measurements with the calculated R value for that compound using the following equation:

$$\text{Reported Result} = \frac{\text{Measured Concentration (ppm)}}{R} \quad \text{Eq. 18-7}$$

12.9 Determine the mass per volume of spiked compound measured using the following equation:

$$m_v = \frac{m_s}{V_s} - \frac{m_u}{V_u} \quad \text{Eq. 18-8}$$

12.10 Calculate the fraction of spiked compound recovered, R, using the following equation:

$$R = \frac{m_v \times v_s}{S} \quad \text{Eq. 18-9}$$

13.0 Method Performance

13.1 Since a potential sample may contain a variety of compounds from various sources, a specific precision limit for the analysis of field samples is impractical. Precision in the range of 5 to 10 percent relative standard deviation (RSD) is typical for gas chromatographic techniques, 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. Triplicate analyses of calibration standards fall within 5 percent of their mean value.

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

(c) Recovery. After developing an appropriate sampling and analytical system for the pollutants of interest, conduct the procedure in Section 8.4. Conduct the appropriate recovery study in Section 8.4 at each sampling point where the method is being applied. Submit the data and results of the recovery procedure with the reporting of results under Section 8.3.

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Optional Presurvey and Presurvey Sampling.

Note: Presurvey screening is optional. Presurvey sampling should be conducted for sources where the target pollutants are not known from previous tests and/or process knowledge.

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

16.1.1 Apparatus. This apparatus list also applies to Sections 8.2 and 11.

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

16.1.1.2 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.

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

16.1.1.4 Flow Meter. To measure flow rates.

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

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

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

16.1.1.8 Tubing Fittings. To plumb GC and gas cylinders.

16.1.1.9 Septa. For syringe injections.

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

16.1.1.11 Soap Film Flowmeter. To determine flow rates.

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

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

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

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

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

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

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

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

16.1.1.20 Barometer. To measure barometric pressure.

16.1.2 Reagents.

16.1.2.1 Water. Deionized distilled.

16.1.2.2 Methylene chloride.

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

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

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

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

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

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

16.1.3 Sampling.

16.1.3.1 Collection of Samples with Glass Sampling Flasks. Presurvey samples may 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 chloride (or other non-target pollutant solvent, or heat and humidified air). Clean all glass ports with a soap solution, then rinse with tap and deionized distilled water. Place the flask in a cool glass annealing furnace, and apply heat up to 500 °C. Maintain at this temperature for 1 hour. After this time period, shut off and open the furnace to allow the flask to cool. Return the stopcocks 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.

16.1.3.1.1 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 and of sufficient length to reach the centroid of the duct to be sampled. Insert a glass wool plug in the enlarged end of the probe to remove particulate matter. Attach the other end of the probe to the tee with a short piece of 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.

16.1.3.1.2 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 8.3.3.1.1. Place the filter end of the probe at the centroid of the duct, or at a point no closer to the walls than 1 m, and apply suction with the bulb to completely purge the probe and flask. After the flask has been purged, close off the stopcock near the suction bulb, and then close off 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.

16.1.3.2 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 8.2.1.

16.1.3.3 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.

16.1.4 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.

16.1.5 Collection of Presurvey Samples with Adsorption Tube. Follow Section 8.2.4 for presurvey sampling.

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Volume 1—017–033–00267–3/$13
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Volume 5—017–033–00349–1/$10
Volume 6—017–033–00369–6/$9
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18.0 Tables, Diagrams, Flowcharts, and Validation Data

I. Name of company
Date
Address

Contracts
Phone
Process to be sampled

Duct or vent to be sampled

II. Process description

Raw material

Products

Operating cycle
Check: Batch Continuous Cyclic
Timing of batch or cycle

Best time to test

III. Sampling site
A. Description
Site decription
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 H_2O, Data source
Moisture content %, Data source
Particulate content , Data source

Gaseous components
N_2 % Hydrocarbons ppm
O_2 %
CO %
CO_2 %
SO_2 %

Hydrocarbon components
ppm
ppm
ppm
ppm
ppm
ppm

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. Preliminary Survey Data Sheet

Components to be analyzed and 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 and Retention time and Attenuation

Figure 18–2. Chromatographic Conditions Data Sheet

FIGURE 18–3. PREPARATION OF STANDARDS IN TEDLAR BAGS AND CALIBRATION CURVE

	Standards		
	Mixture #1	Mixture #2	Mixture #3
Standards Preparation Data:			
Organic:			
Bag number or identification			
Dry gas meter calibration factor			
Final meter reading (liters)			
Initial meter reading (liters)			
Metered volume (liters)			
Average meter temperature (°K)			
Average meter pressure, gauge (mm Hg)			
Average atmospheric perssure (mm Hg)			
Average meter pressure, absolute (mm Hg)			
Syringe temperature (°K) (see Section 10.1.2.1)			
Syringe pressure, absolute (mm Hg) (see Section 10.1.2.1)			
Volume of gas in syringe (ml) (Section 10.1.2.1)			
Density of liquid organic (g/ml) (Section 10.1.2.1)			
Volume of liquid in syringe (ml) (Section 10.1.2.1)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Carrier gas flow rate (ml/min)			
Column temperature:			
Initial (°C)			
Rate change (°C/min)			
Final (°C)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24 hour clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Organic retention time (min)			
Attenuation factor			
Peak height (mm)			
Peak area (mm_2)			
Peak area * attenuation factor (mm_2)			
Calculated concentration (ppm) (Equation 18–3 or 18–4)			

Plot peak area * attenuation factor against calculated concentration to obtain calibration curve.

Flowmeter number or identification
Flowmeter Type
Method: Bubble meter Spirometer Wet test meter
Readings at laboratory conditions:
Laboratory temperature (T_{lab}) °K
Laboratory barometric pressure (P_{lab}) mm Hg
Flow data:

FLOWMETER

Reading (as marked)	Temp. (°K)	Pressure (absolute)

CALIBRATION DEVICE

Time (min)	Gas volume [a]	Flow rate [b]

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

[b] Convert to standard conditions (20 °C and 760 mm Hg). Plot flowmeter reading against flow rate (standard conditions), and draw a smooth curve. If the flowmeter being calibrated is a rotameter or other flow device that is viscosity dependent, it may be necessary to generate a "family" of calibration curves that cover the operating pressure and temperature ranges of the flowmeter. While the following technique should be verified before application, it may be possible to calculate flow rate reading for rotameters at standard conditions Q_{std} as follows:

$$Q_{std} = Q_{lab}\left(\frac{760 \times T_{lab}}{P_{lab} \times 293}\right)^{1/2}$$

Flow rate (laboratory conditions)	Flow rate (STD conditions)

Figure 18–4. Flowmeter Calibration

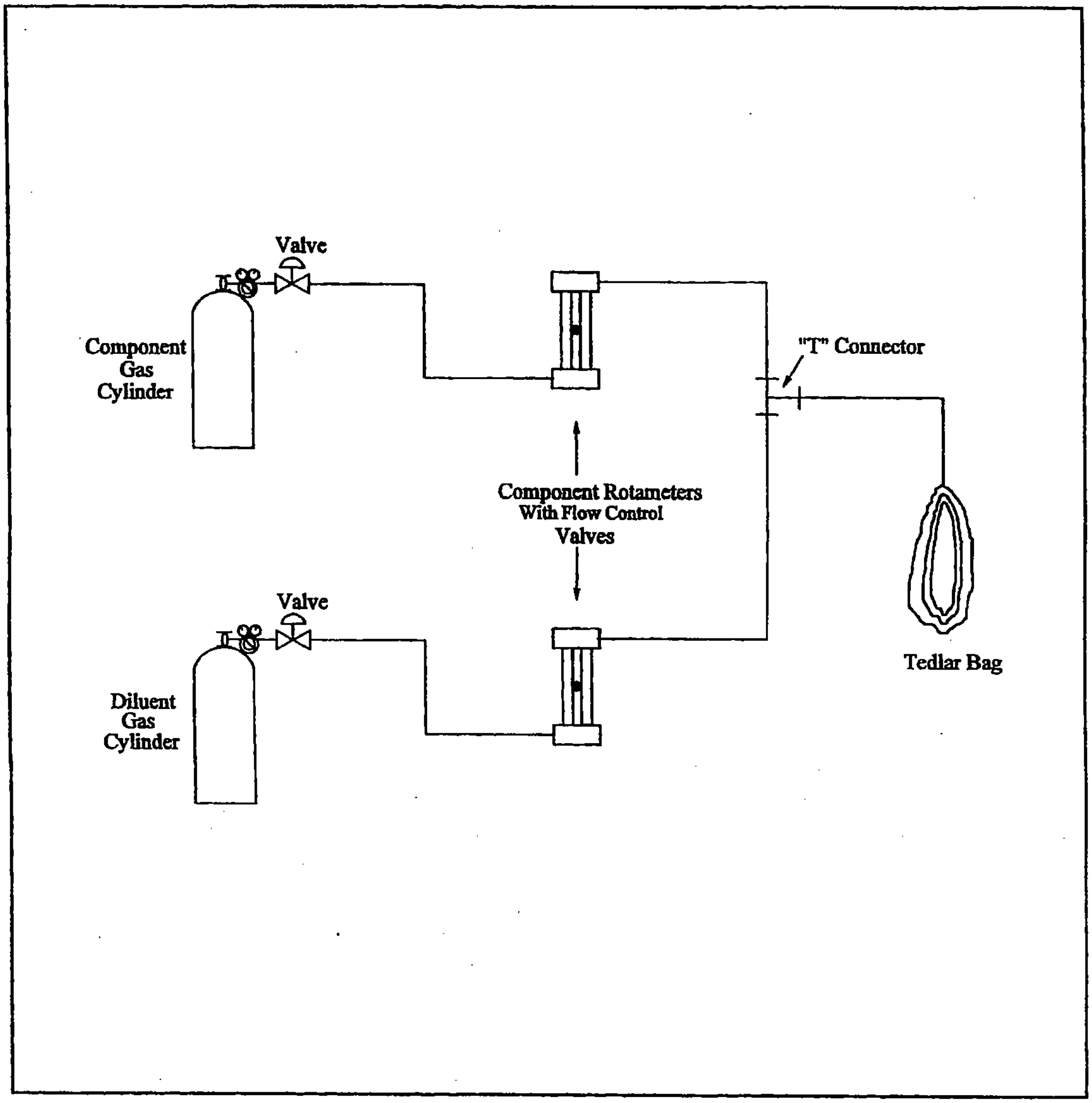

Figure 18-5. Single-Stage Calibration Gas Dilution System.

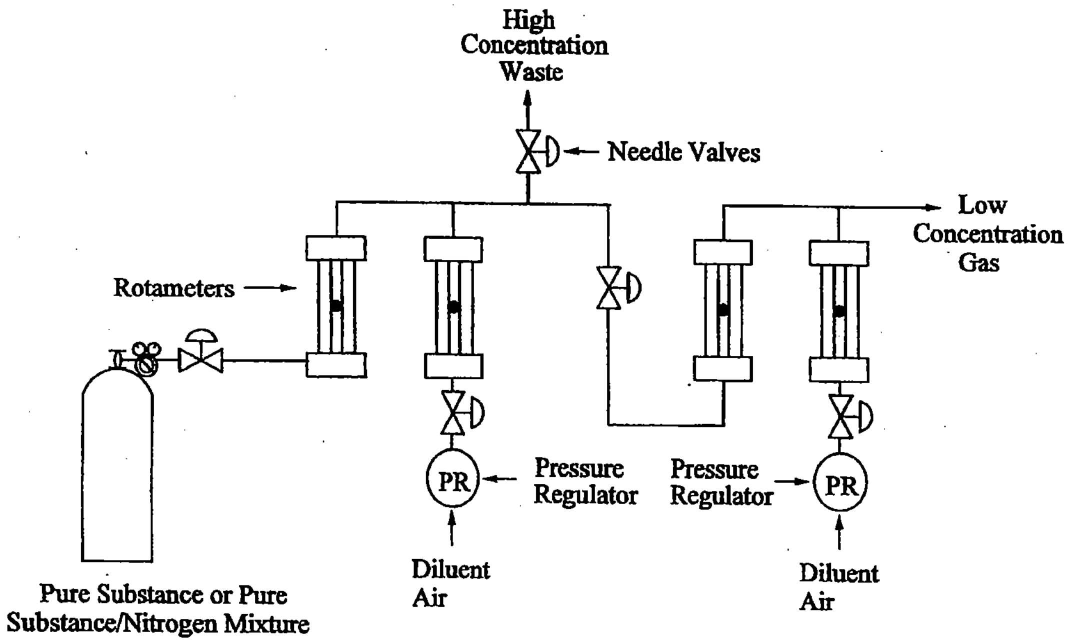

Figure 18-6. Two-Stage Dilution Apparatus.

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PREPARATION OF STANDARDS BY DILUTION OF CYLINDER STANDARD

[Cylinder Standard: Organic ——— Certified Concentration ——— ppm]

Standards preparation data:	Date:		
	Mixture 1	Mixture 2	Mixture 3
Stage 1:			
Standard gas flowmeter reading.			
Diluent gas flowmeter reading			
Laboratory temperature (°K)			
Barometric pressure (mm Hg)			
Flowmeter gage pressure (mm Hg)			
Flow rate cylinder gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions (ml/min)			
Calculated concentration (ppm)			
Stage 2 (if used):			
Standard gas flowmeter reading			
Diluent gas flowmeter reading			
Flow rate Stage 1 gas at standard conditions (ml/min)			
Flow rate diluent gas at standard conditions			

PREPARATION OF STANDARDS BY DILUTION OF CYLINDER STANDARD—Continued

[Cylinder Standard: Organic ——— Certified Concentration ——— ppm]

Standards preparation data:	Date:		
	Mixture 1	Mixture 2	Mixture 3
Calculated concentration (ppm)			
GC Operating Conditions:			
Sample loop volume (ml)			
Sample loop temperature (°C)			
Carrier gas flow rate (ml/min)			
Column temperature:			
Initial (°C)			
Program rate (°C/min)			
Final (°C)			
Organic Peak Identification and Calculated Concentrations:			
Injection time (24-hour clock)			
Distance to peak (cm)			
Chart speed (cm/min)			
Retention time (min)			
Attenuation factor			
Peak area (mm^2)			
Peak area *attenuation factor			

Plot peak area *attenuation factor against calculated concentration to obtain calibration curve.

Figure 18–7. Standards Prepared by Dilution of Cylinder Standard

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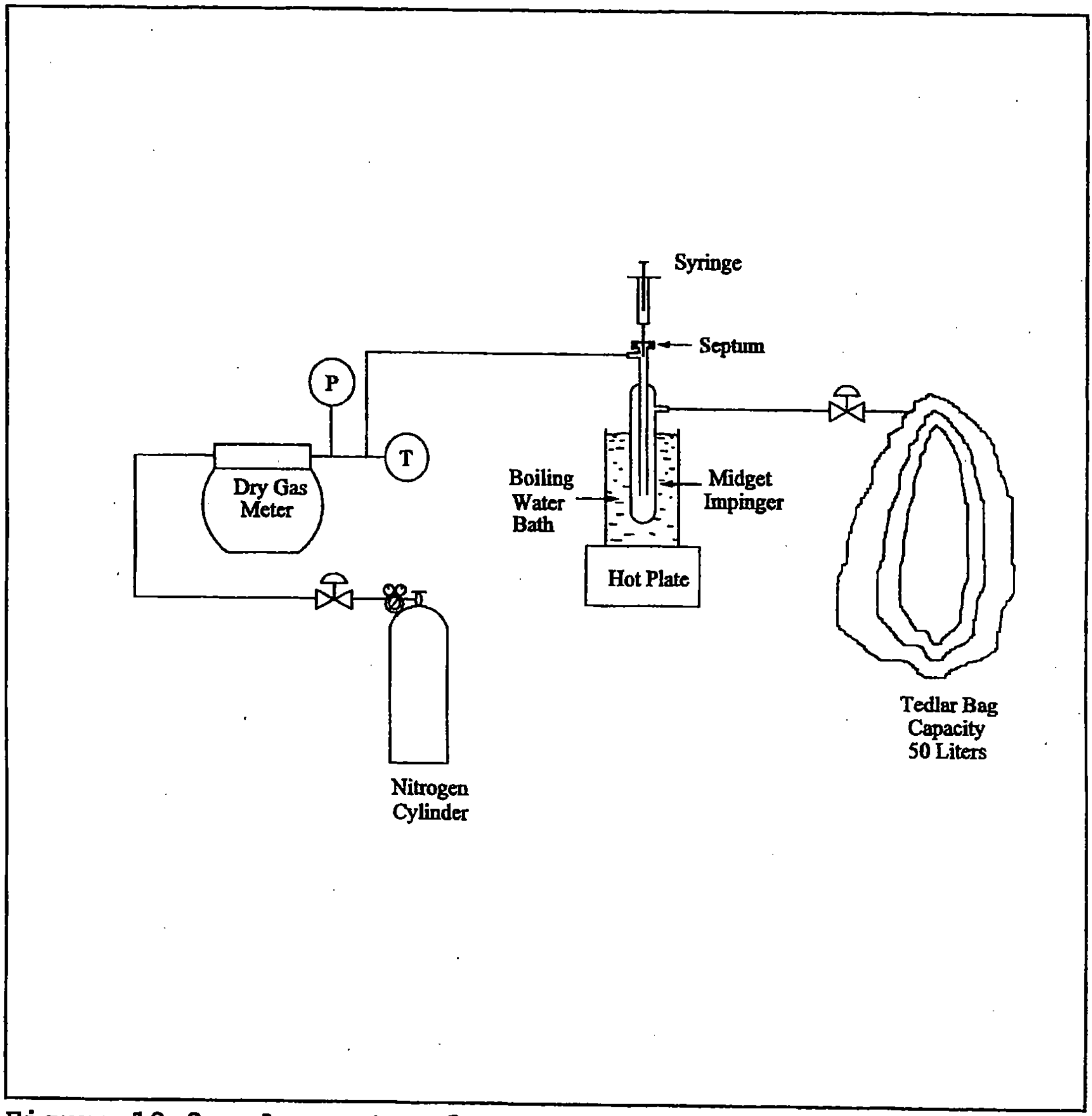


Figure 18-8. Apparatus for Preparation of Liquid Materials.

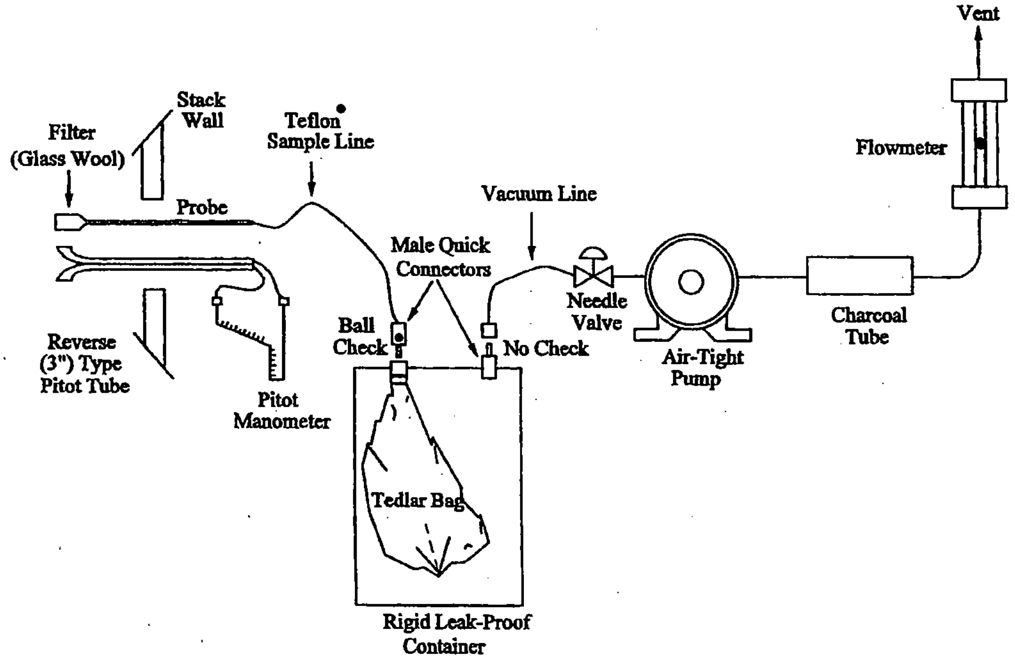


Figure 18-9. Integrated Bag Sampling Train.

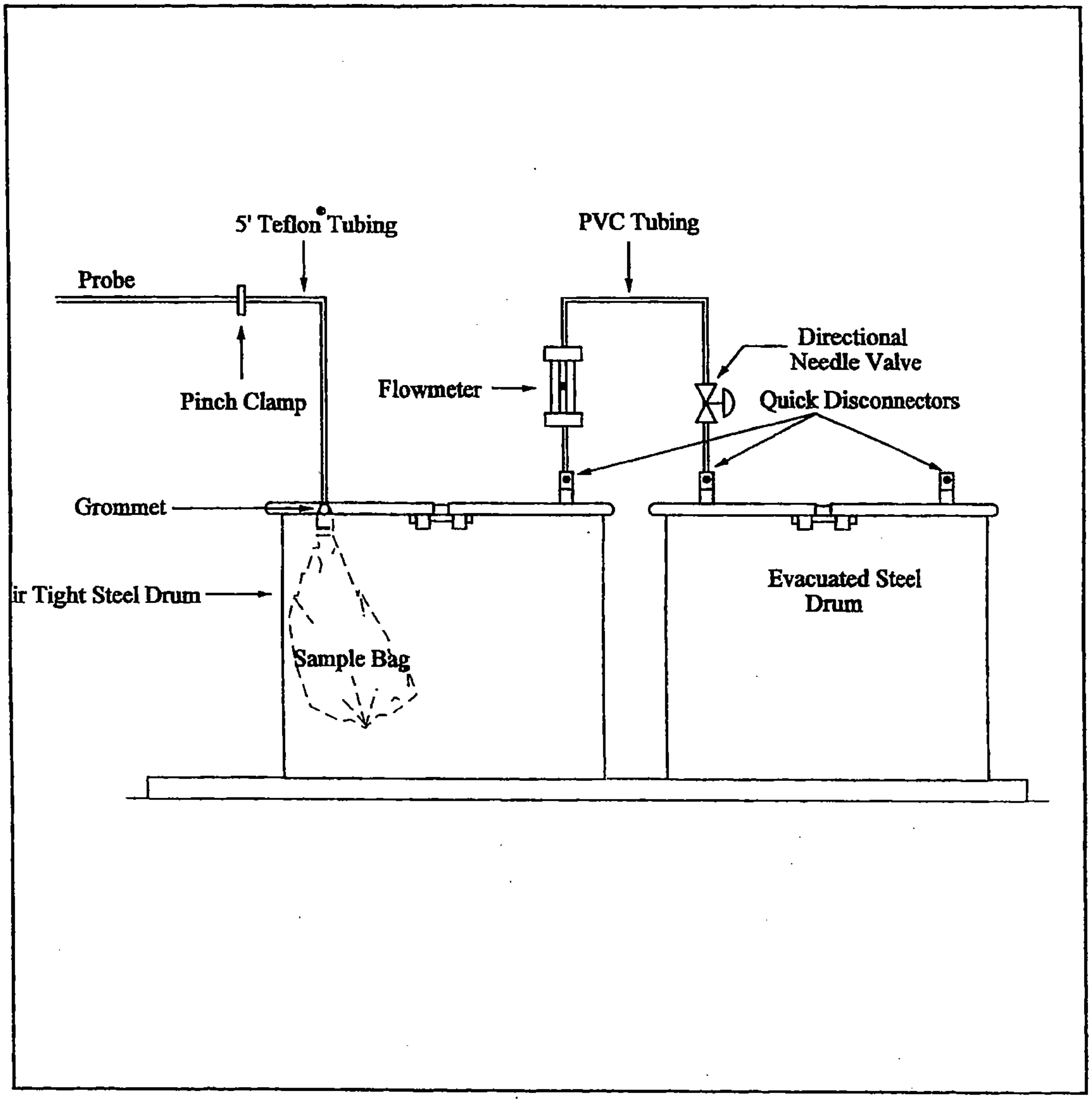


Figure 18-9a. Explosion Risk Gas Sampling Method.

BILLING CODE 6560-50-C

PLANT DATE SITE

	Sample 1	Sample 2	Sample 3
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

PLANT DATE LOCATION

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

PLANT ——— DATE ——— LOCATION ———Continued

Source pressure ("Hg) ....
Absolute source pressure (mm) ....
Sampling rate (liter/min) ....
Sample loop volume (ml) ....
Sample loop temperature (°C) ....
Columnar temperature:
  Initial (°C) time (min) ....
  Program rate (°C/min) ....
  Final (°C)/time (min) ....
Carrier gas flow rate (ml/min) ....
Detector temperature (°C) ....
Injection time (24-hour basis) ....
Chart Speed (mm/min) ....
Dilution gas flow rate (ml/min) ....
Dilution gas used (symbol) ....
Dilution ratio ....

2. FIELD ANALYSIS DATA—CALIBRATION GAS

2. [Run No. Time]

Components	Area	Attenuation	A x A Factor	Conc. (ppm)

Figure 18–11. Field Analysis Data Sheets

BILLING CODE 6560-50-P

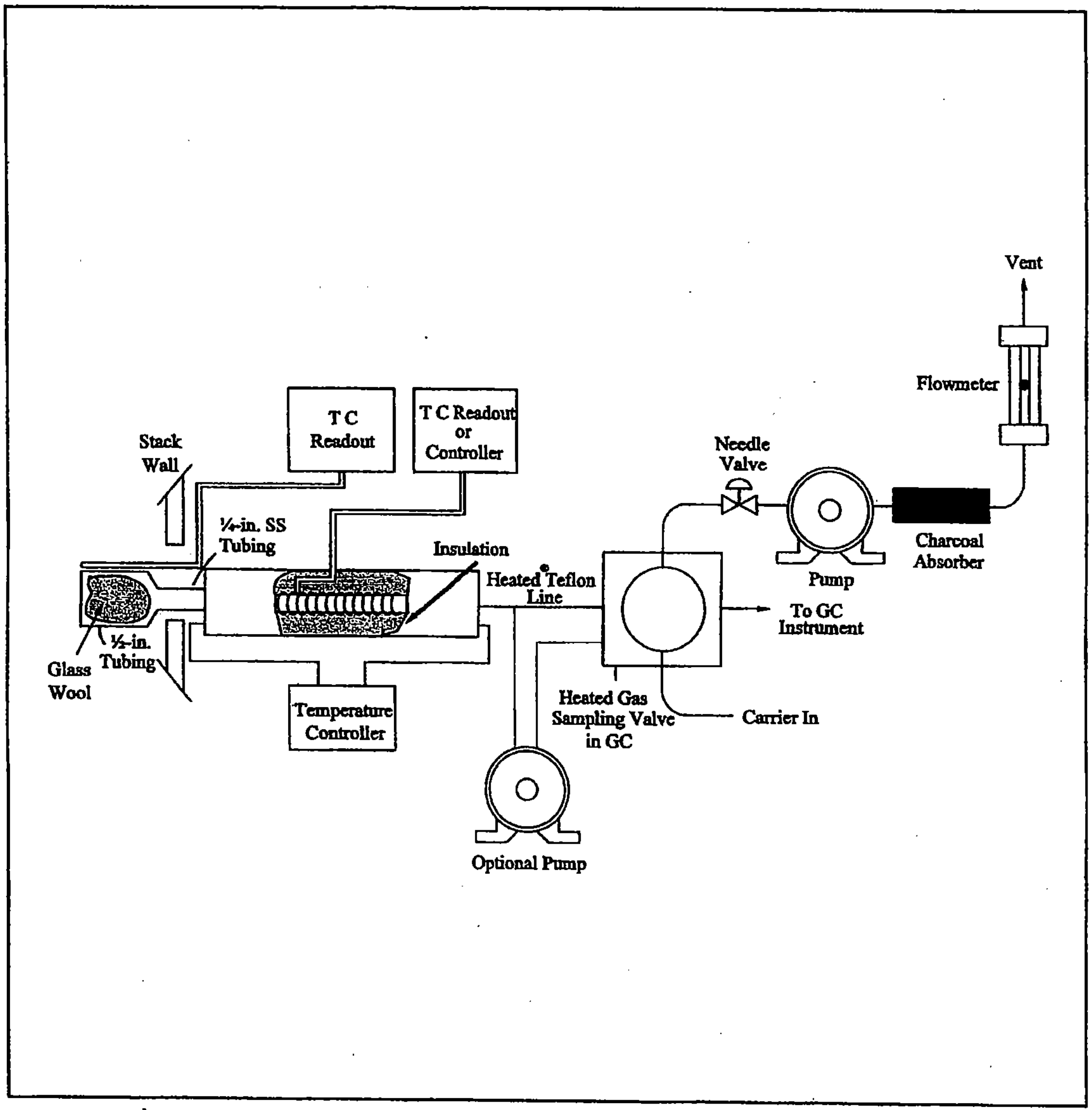


Figure 18-12. Direct Interface Sampling System.

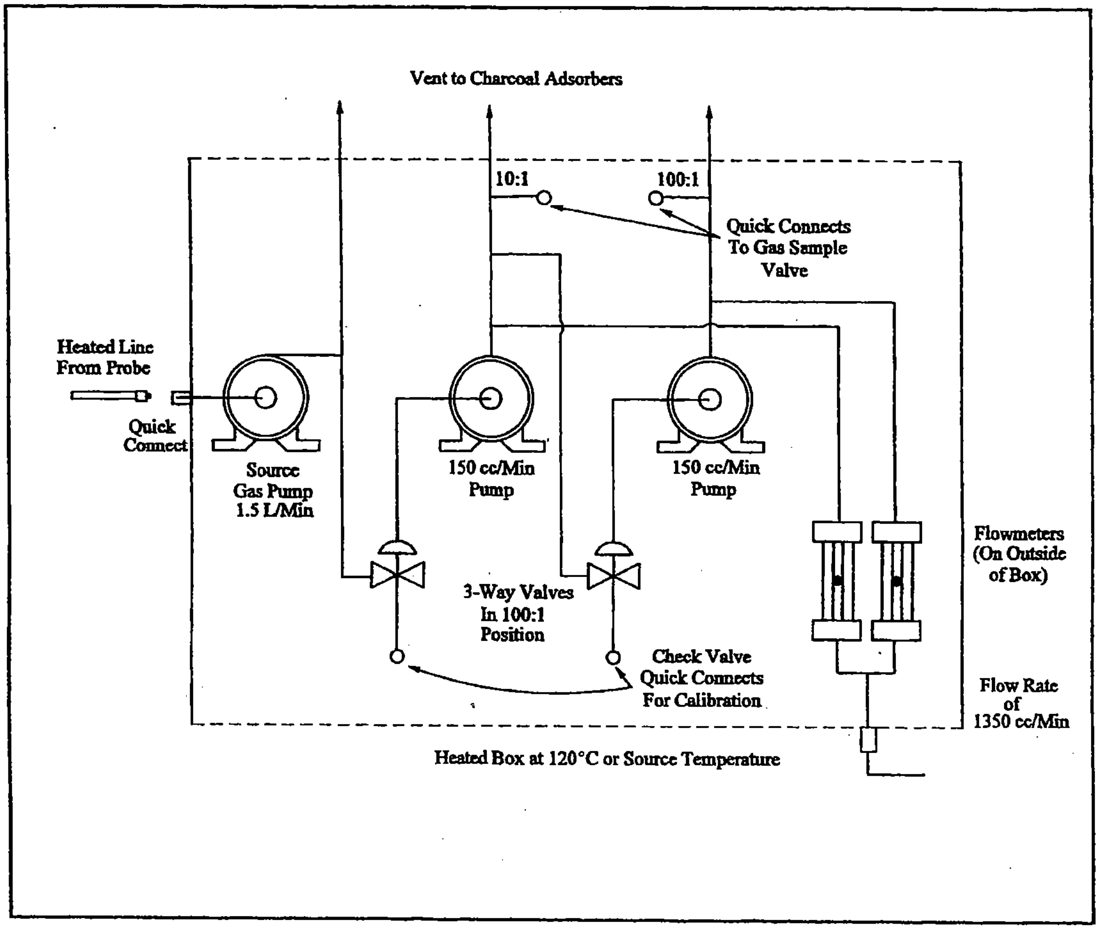


Figure 18-13. Schematic Diagram of the Heated Box Required for Dilution of Sample Gas.

BILLING CODE 6560-50-C

GASEOUS ORGANIC SAMPLING AND ANALYSIS CHECK LIST

[Respond with initials or number as appropriate]

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 ☐

GASEOUS ORGANIC SAMPLING AND ANALYSIS CHECK LIST—Continued

[Respond with initials or number as appropriate]

B. Calibration curve prepared ☐
Number of components ☐
Number of concentrations per component (3 required) ☐

Gaseous Organic Sampling and Analysis Data

Plant
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 pressure (mm Hg)		
Sampling rate (ml/min)		
Sample loop volume (ml)		
Sample loop temperature (°C)		
Sample collection time (24-hr basis)		
Column 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

Method 19—Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide, and Nitrogen Oxide Emission Rates

1.0 Scope and Application

1.1 Analytes. This method provides data reduction procedures relating to the following pollutants, but does not include any sample collection or analysis procedures.

Analyte	CAS No.	Sensitivity
Nitrogen oxides (NO_X), including:		
Nitric oxide (NO)	10102–43–9	N/A
Nitrogen dioxide (NO_2)	10102–44–0.	
Particulate matter (PM)	None assigned	N/A
Sulfur dioxide (SO_2)	7499–09–05	N/A

1.2 Applicability. Where specified by an applicable subpart of the regulations, this method is applicable for the determination of (a) PM, SO_2, and NO_X emission rates; (b) sulfur removal efficiencies of fuel pretreatment and SO_2 control devices; and (c) overall reduction of potential SO_2 emissions.

2.0 Summary of Method

2.1 Emission Rates. Oxygen (O_2) or carbon dioxide (CO_2) concentrations and appropriate F factors (ratios of combustion gas volumes to heat inputs) are used to calculate pollutant emission rates from pollutant concentrations.

2.2 Sulfur Reduction Efficiency and SO_2 Removal Efficiency. An overall SO_2 emission reduction efficiency is computed from the efficiency of fuel pretreatment systems, where applicable, and the efficiency of SO_2 control devices.

2.2.1 The sulfur removal efficiency of a fuel pretreatment system is determined by fuel sampling and analysis of the sulfur and heat contents of the fuel before and after the pretreatment system.

2.2.2 The SO_2 removal efficiency of a control device is determined by measuring the SO_2 rates before and after the control device.

2.2.2.1 The inlet rates to SO_2 control systems (or, when SO_2 control systems are not used, SO_2 emission rates to the atmosphere) are determined by fuel sampling and analysis.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety [Reserved]

6.0 Equipment and Supplies [Reserved]

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Storage, and Transport [Reserved]

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization [Reserved]

11.0 Analytical Procedures [Reserved]

12.0 Data Analysis and Calculations

12.1 Nomenclature

B_{wa} = Moisture fraction of ambient air, percent.
B_{ws} = Moisture fraction of effluent gas, percent.
%C = Concentration of carbon from an ultimate analysis of fuel, weight percent.
C_d = Pollutant concentration, dry basis, ng/scm (lb/scf)
$\%CO_{2d}$,$\%CO_{2w}$ = Concentration of carbon dioxide on a dry and wet basis, respectively, percent.
C_w = Pollutant concentration, wet basis, ng/scm (lb/scf).
D = Number of sampling periods during the performance test period.
E = Pollutant emission rate, ng/J (lb/million Btu).
E_a = Average pollutant rate for the specified performance test period, ng/J (lb/million Btu).
E_{ao}, E_{ai} = Average pollutant rate of the control device, outlet and inlet, respectively, for the performance test period, ng/J (lb/million Btu).
E_{bi} = Pollutant rate from the steam generating unit, ng/J (lb/million Btu)
E_{bo} = Pollutant emission rate from the steam generating unit, ng/J (lb/million Btu).
E_{ci} = Pollutant rate in combined effluent, ng/J (lb/million Btu).
E_{co} = Pollutant emission rate in combined effluent, ng/J (lb/million Btu).
E_d = Average pollutant rate for each sampling period (*e.g.*, 24-hr Method 6B sample or 24-hr fuel sample) or for each fuel lot (*e.g.*, amount of fuel bunkered), ng/J (lb/million Btu).
E_{di} = Average inlet SO_2 rate for each sampling period d, ng/J (lb/million Btu)
E_g = Pollutant rate from gas turbine, ng/J (lb/million Btu).
E_{ga} = Daily geometric average pollutant rate, ng/J (lbs/million Btu) or ppm corrected to 7 percent O_2.
E_{jo},E_{ji} = Matched pair hourly arithmetic average pollutant rate, outlet and inlet, respectively, ng/J (lb/million Btu) or ppm corrected to 7 percent O_2.
E_h = Hourly average pollutant, ng/J (lb/million Btu).
E_{hj} = Hourly arithmetic average pollutant rate for hour "j," ng/J (lb/million Btu) or ppm corrected to 7 percent O_2.
EXP = Natural logarithmic base (2.718) raised to the value enclosed by brackets.
F_d, F_w, F_c = Volumes of combustion components per unit of heat content, scm/J (scf/million Btu).
GCV = Gross calorific value of the fuel consistent with the ultimate analysis, kJ/kg (Btu/lb).
GCV_p, GCV_r = Gross calorific value for the product and raw fuel lots, respectively, dry basis, kJ/kg (Btu/lb).
%H = Concentration of hydrogen from an ultimate analysis of fuel, weight percent.
H = Total number of operating hours for which pollutant rates are determined in the performance test period.
H_b = Heat input rate to the steam generating unit from fuels fired in the steam generating unit, J/hr (million Btu/hr).
H_g = Heat input rate to gas turbine from all fuels fired in the gas turbine, J/hr (million Btu/hr).
$\%H_2O$ = Concentration of water from an ultimate analysis of fuel, weight percent.
H_r = Total numbers of hours in the performance test period (*e.g.*, 720 hours for 30-day performance test period).
K = Conversion factor, 10^{-5} (kJ/J)/(%) [10^6 Btu/million Btu].
K_c = (9.57 scm/kg)/% [(1.53 scf/lb)/%].
K_{cc} = (2.0 scm/kg)/% [(0.321 scf/lb)/%].
K_{hd} = (22.7 scm/kg)/% [(3.64 scf/lb)/%].
K_{hw} = (34.74 scm/kg)/% [(5.57 scf/lb)/%].
K_n = (0.86 scm/kg)/% [(0.14 scf/lb)/%].
K_o = (2.85 scm/kg)/% [(0.46 scf/lb)/%].
K_s = (3.54 scm/kg)/% [(0.57 scf/lb)/%].
K_w = (1.30 scm/kg)/% [(0.21 scf/lb)/%].
ln = Natural log of indicated value.
L_p,L_r = Weight of the product and raw fuel lots, respectively, metric ton (ton).
%N = Concentration of nitrogen from an ultimate analysis of fuel, weight percent.
N = Number of fuel lots during the averaging period.
n = Number of fuels being burned in combination.
n_d = Number of operating hours of the affected facility within the performance test period for each E_d determined.
n_t = Total number of hourly averages for which paired inlet and outlet pollutant rates are available within the 24-hr midnight to midnight daily period.
%O = Concentration of oxygen from an ultimate analysis of fuel, weight percent.
$\%O_{2d}$, $\%O_{2w}$ = Concentration of oxygen on a dry and wet basis, respectively, percent.
P_s = Potential SO_2 emissions, percent.
$\%R_f$ = SO_2 removal efficiency from fuel pretreatment, percent.
$\%R_g$ = SO_2 removal efficiency of the control device, percent.
$\%R_{ga}$ = Daily geometric average percent reduction.
$\%R_o$ = Overall SO_2 reduction, percent.
%S = Sulfur content of as-fired fuel lot, dry basis, weight percent.
S_e = Standard deviation of the hourly average pollutant rates for each performance test period, ng/J (lb/million Btu).
$\%S_f$ = Concentration of sulfur from an ultimate analysis of fuel, weight percent.
S_i = Standard deviation of the hourly average inlet pollutant rates for each performance test period, ng/J (lb/million Btu).
S_o = Standard deviation of the hourly average emission rates for each performance test period, ng/J (lb/million Btu).
$\%S_p$, $\%S_r$ = Sulfur content of the product and raw fuel lots respectively, dry basis, weight percent.
$t_{0.95}$ = Values shown in Table 19–3 for the indicated number of data points n.
X_k = Fraction of total heat input from each type of fuel k.

12.2 Emission Rates of PM, SO_2, and NO_x. Select from the following sections the applicable procedure to compute the PM, S_{O2}, or NO_x emission rate (E) in ng/J (lb/million Btu). The pollutant concentration must be in ng/scm (lb/scf) and the F factor must be in scm/J (scf/million Btu). If the pollutant concentration (C) is not in the appropriate units, use Table 19–1 in Section 17.0 to make the proper conversion. An F factor is the ratio of the gas volume of the products of combustion to the heat content of the fuel. The dry F factor (F_d) includes all components of combustion less water, the wet F factor (F_w) includes all components of combustion, and the carbon F factor (F_c) includes only carbon dioxide.

Note: Since F_w factors include water resulting only from the combustion of hydrogen in the fuel, the procedures using F_w factors are not applicable for computing E from steam generating units with wet scrubbers or with other processes that add water (*e.g.*, steam injection).

12.2.1 Oxygen-Based F Factor, Dry Basis. When measurements are on a dry basis for both O ($\%O_{2d}$) and pollutant (C_d) concentrations, use the following equation:

$$E = C_dF_d \frac{20.9}{(20.9 - \%O_{2d})} \qquad \text{Eq. 19-1}$$

12.2.2 Oxygen-Based F Factor, Wet Basis. When measurements are on a wet basis for both O_2 ($\%O_{2w}$) and pollutant (C_w) concentrations, use either of the following:

12.2.2.1 If the moisture fraction of ambient air (B_{wa}) is measured:

$$E = C_wF_w \frac{20.9}{[20.9(1 - B_{wa}) - \%O_{2w}]} \qquad \text{Eq. 19-2}$$

Instead of actual measurement, B_{wa} may be estimated according to the procedure below.

NOTE: The estimates are selected to ensure that negative errors will not be larger than −1.5 percent. However, positive errors, or over-estimation of emissions by as much as 5 percent may be introduced depending upon the geographic location of the facility and the associated range of ambient moisture.

12.2.2.1.1 B_{wa} = 0.027. This value may be used at any location at all times.

12.2.2.1.2 B_{wa} = Highest monthly average of B_{wa} that occurred within the previous calendar year at the nearest Weather Service Station. This value shall be determined annually and may be used as an estimate for the entire current calendar year.

12.2.2.1.3 B_{wa} = Highest daily average of Bwa that occurred within a calendar month at the nearest Weather Service Station, calculated from the data from the past 3 years. This value shall be computed for each month and may be used as an estimate for the current respective calendar month.

12.2.2.2 If the moisture fraction (B_{ws}) of the effluent gas is measured:

$$E = C_wF_d \frac{20.9}{[20.9(1 - B_{ws}) - \%O_{2w}]} \qquad \text{Eq. 19-3}$$

12.2.3 Oxygen-Based F Factor, Dry/Wet Basis.

12.2.3.1 When the pollutant concentration is measured on a wet basis (C_w) and O_2 concentration is measured on a dry basis ($\%O_{2d}$), use the following equation:

$$E = \frac{(C_wF_d)(20.9)}{(1 - B_{ws})(20.9 - \%O_{2d})} \qquad \text{Eq. 19-4}$$

12.2.3.2 When the pollutant concentration is measured on a dry basis (C_d) and the O_2 concentration is measured on a wet basis ($\%O_{2w}$), use the following equation:

$$E = \frac{\dfrac{C_dF_d 20.9}{(20.9 - \%O_{2w})}}{(1 - B_{ws})} \qquad \text{Eq. 19-5}$$

12.2.4 Carbon Dioxide-Based F Factor, Dry Basis. When measurements are on a dry basis for both CO_2 ($\%CO_{2d}$) and pollutant (C_d) concentrations, use the following equation:

$$E = C_dF_c \frac{100}{\%CO_{2d}} \qquad \text{Eq. 19-6}$$

12.2.5 Carbon Dioxide-Based F Factor, Wet Basis. When measurements are on a wet basis for both CO_2 ($\%CO_{2w}$) and pollutant (C_w) concentrations, use the following equation:

$$E = C_wF_c \frac{100}{\%CO_{2w}} \qquad \text{Eq. 19-7}$$

12.2.6 Carbon Dioxide-Based F Factor, Dry/Wet Basis.

12.2.6.1 When the pollutant concentration is measured on a wet basis (C_w) and CO_2 concentration is measured on a dry basis ($\%CO_{2d}$), use the following equation:

$$E = \frac{C_wF_c}{(1 - B_{ws})} \frac{100}{\%CO_{2d}} \qquad \text{Eq. 19-8}$$

12.2.6.2 When the pollutant concentration is measured on a dry basis (C_d) and CO_2 concentration is measured on a wet basis ($\%CO_{2w}$), use the following equation:

$$E = C_dF_c(1 - B_{ws}) \frac{100}{\%CO_{2w}} \qquad \text{Eq. 19-9}$$

12.2.7 Direct-Fired Reheat Fuel Burning. The effect of direct-fired reheat fuel burning (for the purpose of raising the temperature of the exhaust effluent from wet scrubbers to above the moisture dew-point) on emission rates will be less than 1.0 percent and, therefore, may be ignored.

12.2.8 Combined Cycle-Gas Turbine Systems. For gas turbine-steam generator combined cycle systems, determine the emissions from the steam generating unit or the percent reduction in potential SO_2 emissions as follows:

12.2.8.1 Compute the emission rate from the steam generating unit using the following equation:

$$E_{bo} = E_{co} + \frac{H_g}{H_b}(E_{co} - E_g) \qquad \text{Eq. 19-10}$$

12.2.8.1.1 Use the test methods and procedures section of 40 CFR Part 60, Subpart GG to obtain E_{co} and E_g. Do not use F_w factors for determining E_g or E_{co}. If an SO_2 control device is used, measure E_{co} after the control device.

12.2.8.1.2 Suitable methods shall be used to determine the heat input rates to the steam generating units (H_b) and the gas turbine (H_g).

12.2.8.2 If a control device is used, compute the percent of potential SO_2 emissions (P_s) using the following equations:

$$E_{bi} = E_{ci} - \frac{H_g}{H_b}(E_{ci} - E_g) \qquad \text{Eq. 19-11}$$

$$P_s = 100\left(1 - \frac{E_{bo}}{E_{bi}}\right) \qquad \text{Eq. 19-12}$$

NOTE: Use the test methods and procedures section of Subpart GG to obtain E_{ci} and E_g. Do not use F_w factors for determining E_g or E_{ci}.

12.3 F Factors. Use an average F factor according to Section 12.3.1 or determine an applicable F factor according to Section 12.3.2. If combined fuels are fired, prorate the applicable F factors using the procedure in Section 12.3.3.

12.3.1 Average F Factors. Average F factors (F_d, F_w, or F_c) from Table 19–2 in Section 17.0 may be used.

12.3.2 Determined F Factors. If the fuel burned is not listed in Table 19–2 or if the owner or operator chooses to determine an F factor rather than use the values in Table 19–2, use the procedure below:

12.3.2.1 Equations. Use the equations below, as appropriate, to compute the F factors:

$$F_d = \frac{K\left(K_{hd}\%H + K_c\%C + K_s\%S + K_n\%N - K_o\%O\right)}{GCV} \qquad \text{Eq. 19-13}$$

$$F_w = \frac{K\left[K_{hw}\%H + K_c\%C + K_s\%S + K_n\%N - K_o\%O + K_w\%H_2O\right]}{GCV_w} \qquad \text{Eq. 19-14}$$

$$F_c = \frac{K\left(K_{cc}\%C\right)}{GCV} \qquad \text{Eq. 19-15}$$

Note: Omit the $\%H_2O$ term in the equations for F_w if %H and %O include the unavailable hydrogen and oxygen in the form of H_2O.)

12.3.2.2 Use applicable sampling procedures in Section 12.5.2.1 or 12.5.2.2 to obtain samples for analyses.

12.3.2.3 Use ASTM D 3176–74 or 89 (all cited ASTM standards are incorporated by reference—see § 60.17) for ultimate analysis of the fuel.

12.3.2.4 Use applicable methods in Section 12.5.2.1 or 12.5.2.2 to determine the heat content of solid or liquid fuels. For gaseous fuels, use ASTM D 1826–77 or 94 (incorporated by reference—see § 60.17) to determine the heat content.

12.3.3 F Factors for Combination of Fuels. If combinations of fuels are burned, use the following equations, as applicable unless otherwise specified in an applicable subpart:

$$F_d = \sum_{k=1}^{n}\left(X_k F_{dk}\right) \qquad \text{Eq. 19-16}$$

$$F_w = \sum_{k=1}^{n}\left(X_k F_{wk}\right) \qquad \text{Eq. 19-17}$$

$$F_c = \sum_{k=1}^{n}\left(X_k F_{ck}\right) \qquad \text{Eq. 19-18}$$

12.4 Determination of Average Pollutant Rates.

12.4.1 Average Pollutant Rates from Hourly Values. When hourly average pollutant rates (E_h), inlet or outlet, are obtained (*e.g.*, CEMS values), compute the average pollutant rate (E_a) for the performance test period (*e.g.*, 30 days) specified in the applicable regulation using the following equation:

$$E_a = \frac{1}{H}\sum_{j=1}^{n} E_{hj} \qquad \text{Eq. 19-19}$$

12.4.2 Average Pollutant Rates from Other than Hourly Averages. When pollutant rates are determined from measured values representing longer than 1-hour periods (*e.g.*, daily fuel sampling and analyses or Method 6B values), or when pollutant rates are determined from combinations of 1-hour and longer than 1-hour periods (*e.g.*, CEMS and Method 6B values), compute the average pollutant rate (E_a) for the performance test period (*e.g.*, 30 days) specified in the applicable regulation using the following equation:

$$E_a = \frac{\sum_{j=1}^{D}\left(n_d E_d\right)_j}{\sum_{j=1}^{D} n_{dj}} \qquad \text{Eq. 19-20}$$

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

$$E_{ga} = \exp\left[\frac{1}{n_t}\sum_{j=1}^{n_t}\left[\ln\left(E_{hj}\right)\right]\right] \qquad \text{Eq. 19-21}$$

12.5 Determination of Overall Reduction in Potential Sulfur Dioxide Emission.

12.5.1 Overall Percent Reduction. Compute the overall percent SO_2 reduction ($\%R_o$) using the following equation:

$$\%R_o = 100\left[1.0 - \left(1.0 - \frac{\%R_f}{100}\right)\left(1.0 - \frac{\%R_g}{100}\right)\right] \qquad \text{Eq. 19-22}$$

12.5.2 Pretreatment Removal Efficiency (Optional). Compute the SO_2 removal efficiency from fuel pretreatment ($\%R_f$) for the averaging period (*e.g.*, 90 days) as specified in the applicable regulation using the following equation:

$$\%R_f = 100\left[1.0 - \frac{\sum_{j=1}^{N}\left(\frac{\%S_{pj}}{GCV_{pj}}\right)L_{pj}}{\sum_{j=1}^{N}\left(\frac{\%S_{rj}}{GCV_{rj}}\right)L_{rj}}\right] \qquad \text{Eq. 19-23}$$

Note: In calculating $\%R_f$, include %S and GCV values for all fuel lots that are not pretreated and are used during the averaging period.

12.5.2.1 Solid Fossil (Including Waste) Fuel/Sampling and Analysis.

Note: For the purposes of this method, raw fuel (coal or oil) is the fuel delivered to the desulfurization (pretreatment) facility. For oil, the input oil to the oil desulfurization process (*e.g.*, hydrotreatment) is considered to be the raw fuel.

12.5.2.1.1 Sample Increment Collection. Use ASTM D 2234–76, 96, 97a, or 98 (incorporated by reference—see § 60.17), Type I, Conditions A, B, or C, and systematic spacing. As used in this method, systematic spacing is intended to include evenly spaced increments in time or increments based on equal weights of coal passing the collection area. As a minimum, determine the number and weight of increments required per gross sample representing each coal lot according to Table 2 or Paragraph 7.1.5.2 of ASTM D 2234. Collect one gross sample for each lot of raw coal and one gross sample for each lot of product coal.

12.5.2.1.2 ASTM Lot Size. For the purpose of Section 12.5.2 (fuel pretreatment), the lot size of product coal is the weight of product coal from one type of raw coal. The lot size of raw coal is the weight of raw coal used to produce one lot of product coal. Typically, the lot size is the weight of coal processed in a 1-day (24-hour) period. If more than one type of coal is treated and produced in 1 day, then gross samples must be collected and analyzed for each type of coal. A coal lot size equaling the 90-day quarterly fuel quantity for a steam generating unit may be used if representative sampling can be conducted for each raw coal and product coal.

Note: Alternative definitions of lot sizes may be used, subject to prior approval of the Administrator.

12.5.2.1.3 Gross Sample Analysis. Use ASTM D 2013–72 or 86 to prepare the sample, ASTM D 3177–75 or 89 or ASTM D 4239–85, 94, or 97 to determine sulfur content (%S), ASTM D 3173–73 or 87 to determine moisture content, and ASTM D 2015–77 (Reapproved 1978) or 96, D 3286–85 or 96, or D 5865–98 to determine gross calorific value (GCV) (all standards cited are incorporated by reference—see § 60.17 for acceptable versions of the standards) on a dry basis for each gross sample.

12.5.2.2 Liquid Fossil Fuel-Sampling and Analysis. See Note under Section 12.5.2.1.

12.5.2.2.1 Sample Collection. Follow the procedures for continuous sampling in ASTM D 270 or D 4177–95 (incorporated by reference—see § 60.17) for each gross sample from each fuel lot.

12.5.2.2.2 Lot Size. For the purpose of Section 12.5.2 (fuel pretreatment), the lot size of a product oil is the weight of product oil from one pretreatment facility and intended as one shipment (ship load, barge load, etc.). The lot size of raw oil is the weight of each crude liquid fuel type used to produce a lot of product oil.

Note: Alternative definitions of lot sizes may be used, subject to prior approval of the Administrator.

12.5.2.2.3 Sample Analysis. Use ASTM D 129–64, 78, or 95, ASTM D 1552–83 or 95, or ASTM D 4057–81 or 95 to determine the sulfur content (%S) and ASTM D 240–76 or 92 (all standards cited are incorporated by reference—see § 60.17) to determine the GCV of each gross sample. These values may be assumed to be on a dry basis. The owner or operator of an affected facility may elect to determine the GCV by sampling the oil combusted on the first steam generating unit operating day of each calendar month and then using the lowest GCV value of the three GCV values per quarter for the GCV of all oil combusted in that calendar quarter.

12.5.2.3 Use appropriate procedures, subject to the approval of the Administrator, to determine the fraction of total mass input derived from each type of fuel.

12.5.3 Control Device Removal Efficiency. Compute the percent removal efficiency ($\%R_g$) of the control device using the following equation:

$$\%R_g = 100\left(1.0 - \frac{E_{ao}}{E_{ai}}\right) \qquad \text{Eq. 19-24}$$

12.5.3.1 Use continuous emission monitoring systems or test methods, as appropriate, to determine the outlet SO_2 rates and, if appropriate, the inlet SO_2 rates. The rates may be determined as hourly (E_h) or other sampling period averages (E_d). Then, compute the average pollutant rates for the performance test period (E_{ao} and E_{ai}) using the procedures in Section 12.4.

12.5.3.2 As an alternative, as-fired fuel sampling and analysis may be used to determine inlet SO_2 rates as follows:

12.5.3.2.1 Compute the average inlet SO_2 rate (E_{di}) for each sampling period using the following equation:

$$E_{di} = K\frac{\%S}{GCV} \qquad \text{Eq. 19-25}$$

Where:

$$K = 2\times10^7\left(\frac{\text{ng } SO_2}{\%S}\right)\left(\frac{(kJ)}{J}\right)\left(\frac{1}{\text{kg coal}}\right)\left[2\times10^4\left(\frac{\text{lb } SO_2}{\%S}\right)\left(\frac{\text{Btu}}{\text{million Btu}}\right)\left(\frac{1}{\text{lb coal}}\right)\right]$$

After calculating E_{di}, use the procedures in Section 12.4 to determine the average inlet SO_2 rate for the performance test period (E_{ai}).

12.5.3.2.2 Collect the fuel samples from a location in the fuel handling system that provides a sample representative of the fuel bunkered or consumed during a steam generating unit operating day. For the purpose of as-fired fuel sampling under Section 12.5.3.2 or Section 12.6, the lot size for coal is the weight of coal bunkered or consumed during each steam generating unit operating day. The lot size for oil is the weight of oil supplied to the "day" tank or consumed during each steam generating unit operating day. For reporting and calculation purposes, the gross sample shall be identified with the calendar day on which sampling began. For steam generating unit operating days when a coal-fired steam generating unit is operated without coal being added to the bunkers, the coal analysis from the previous "as bunkered" coal sample shall be used until coal is bunkered again. For steam generating unit operating days when an oil-fired steam generating unit is operated without oil being added to the oil "day" tank, the oil analysis from the previous day shall be used until the "day" tank is filled again. Alternative definitions of fuel lot size may be used, subject to prior approval of the Administrator.

12.5.3.2.3 Use ASTM procedures specified in Section 12.5.2.1 or 12.5.2.2 to determine %S and GCV.

12.5.4 Daily Geometric Average Percent Reduction from Hourly Values. The geometric average percent reduction ($\%R_{ga}$) is computed using the following equation:

$$\%R_{ga} = 100\left[1 - \text{EXP}\left(\frac{1}{n_t}\sum_{j=1}^{n_t} \ln\frac{E_{jo}}{E_{ji}}\right)\right] \quad \text{Eq. 19-26}$$

NOTE: The calculation includes only paired data sets (hourly average) for the inlet and outlet pollutant measurements.

12.6 Sulfur Retention Credit for Compliance Fuel. If fuel sampling and analysis procedures in Section 12.5.2.1 are being used to determine average SO_2 emission rates (E_{as}) to the atmosphere from a coal-fired steam generating unit when there is no SO_2 control device, the following equation may be used to adjust the emission rate for sulfur retention credits (no credits are allowed for oil-fired systems) (E_{di}) for each sampling period using the following equation:

$$E_{di} = 0.97K\,\frac{\%S}{GDV} \quad \text{Eq. 19-27}$$

Where:

$$K = 2\times10^7\left(\frac{\text{ng SO}_2}{\%S}\right)\left(\frac{\text{kJ}}{\text{J}}\right)\left(\frac{1}{\text{kg coal}}\right)\left[2\times10^4\left(\frac{\text{lb SO}_2}{\%S}\right)\left(\frac{\text{Btu}}{\text{million Btu}}\right)\left(\frac{1}{\text{lb coal}}\right)\right]$$

After calculating E_{di}, use the procedures in Section 12.4.2 to determine the average SO_2 emission rate to the atmosphere for the performance test period (E_{ao}).

12.7 Determination of Compliance When Minimum Data Requirement Is Not Met.

12.7.1 Adjusted Emission Rates and Control Device Removal Efficiency. When the minimum data requirement is not met, the Administrator may use the following adjusted emission rates or control device removal efficiencies to determine compliance with the applicable standards.

12.7.1.1 Emission Rate. Compliance with the emission rate standard may be determined by using the lower confidence limit of the emission rate (E_{ao}*) as follows:

$$E_{ao}^* = E_{ao} - t_{0.95}S_o \quad \text{Eq. 19-28}$$

12.7.1.2 Control Device Removal Efficiency. Compliance with the overall emission reduction ($\%R_o$) may be determined by using the lower confidence limit of the emission rate (E_{ao}*) and the upper confidence limit of the inlet pollutant rate (E_{ai}*) in calculating the control device removal efficiency ($\%R_g$) as follows:

$$\%R_g = 100\left(1.0 - \frac{E_{ao}^*}{E_{ai}^*}\right) \quad \text{Eq. 19-29}$$

$$E_{ai}^* = E_{ai} + t_{0.95}S_i \quad \text{Eq. 19-30}$$

12.7.2 Standard Deviation of Hourly Average Pollutant Rates. Compute the standard deviation (S_e) of the hourly average pollutant rates using the following equation:

$$S_e = \sqrt{\frac{1}{H} - \frac{1}{H_r}}\sqrt{\frac{\sum_{j=1}^{H}\left(E_{hj} - E_a\right)^2}{H-1}} \quad \text{Eq. 19-31}$$

Equation 19–19 through 19–31 may be used to compute the standard deviation for both the outlet (S_o) and, if applicable, inlet (S_i) pollutant rates.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References [Reserved]

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 19–1.—CONVERSION FACTORS FOR CONCENTRATION

From	To	Multiply by
g/scm	ng/scm	10^9
mg/scm	ng/scm	10^6
lb/scf	ng/scm	1.602×10^{13}
ppm SO_2	ng/scm	2.66×10^6
ppm NO_x	ng/scm	1.912×10^6
ppm SO_2	lb/scf	1.660×10^{-7}
ppm NO_x	lb/scf	1.194×10^{-7}

TABLE 19–2.—F FACTORS FOR VARIOUS FUELS[1]

Fuel Type	F_d		F_w		F_c	
	dscm/J	dscf/10^6 Btu	wscm/J	wscf/10^6 Btu	scm/J	scf/10^6 Btu
Coal:						
Anthracite[2]	2.71×10^{-7}	10,100	2.83×10^{-7}	10,540	0.530×10^{-7}	1,970
Bituminus[2]	2.63×10^{-7}	9,780	2.86×10^{-7}	10,640	0.484×10^{-7}	1,800
Lignite	2.65×10^{-7}	9,860	3.21×10^{-7}	11,950	0.513×10^{-7}	1,910
Oil[3]	2.47×10^{-7}	9,190	2.77×10^{-7}	10,320	0.383×10^{-7}	1,420
Gas:.						
Natural	2.34×10^{-7}	8,710	2.85×10^{-7}	10,610	0.287×10^{-7}	1,040
Propane	2.34×10^{-7}	8,710	2.74×10^{-7}	10,200	0.321×10^{-7}	1,190
Butane	2.34×10^{-7}	8,710	2.79×10^{-7}	10,390	0.337×10^{-7}	1,250
Wood	2.48×10^{-7}	9,240			0.492×10^{-7}	1,830
Wood Bark	2.58×10^{-7}	9,600			0.516×10^{-7}	1,920
Municipal	2.57×10^{-7}	9,570			0.488×10^{-7}	1,820
Solid Waste						

[1] Determined at standard conditions: 20 °C (68 °F) and 760 mm Hg (29.92 in Hg)
[2] As classified according to ASTM D 388.
[3] Crude, residual, or distillate.

TABLE 19–3.—VALUES FOR $T_{0.95}$*

n[1]	$t_{0.95}$	n[1]	$t_{0.95}$	n[1]	$t_{0.95}$
2	6.31	8	1.89	22–26	1.71
3	2.42	9	1.86	27–31	1.70
4	2.35	10	1.83	32–51	1.68
5	2.13	11	1.81	52–91	1.67
6	2.02	12–16	1.77	92–151	1.66
7	1.94	17–21	1.73	152 or more	1.65

[1] The values of this table are corrected for n-1 degrees of freedom. Use n equal to the number (H) of hourly average data points.

* * * * *

Method 21—Determination of Volatile Organic Compound Leaks

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.
Volatile Organic Compounds (VOC).	No CAS number assigned.

1.2 Scope. This method is applicable for the determination of VOC leaks from process equipment. These sources include, but are not limited to, valves, flanges and other connections, pumps and compressors, pressure relief devices, process drains, open-ended valves, pump and compressor seal system degassing vents, accumulator vessel vents, agitator seals, and access door seals.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A portable instrument is used to detect VOC leaks from individual sources. The instrument detector type is not specified, but it must meet the specifications and performance criteria contained in Section 6.0. A leak definition concentration based on a reference compound is specified in each applicable regulation. This method is intended to locate and classify leaks only, and is not to be used as a direct measure of mass emission rate from individual sources.

3.0 Definitions

3.1 *Calibration gas* means the VOC compound used to adjust the instrument meter reading to a known value. The calibration gas is usually the reference compound at a known concentration approximately equal to the leak definition concentration.

3.2 *Calibration precision* means the degree of agreement between measurements of the same known value, expressed as the relative percentage of the average difference between the meter readings and the known concentration to the known concentration.

3.3 *Leak definition concentration* means the local VOC concentration at the surface of a leak source that indicates that a VOC emission (leak) is present. The leak definition is an instrument meter reading based on a reference compound.

3.4 *No detectable emission* means a local VOC concentration at the surface of a leak source, adjusted for local VOC ambient concentration, that is less than 2.5 percent of the specified leak definition concentration. that indicates that a VOC emission (leak) is not present.

3.5 *Reference compound* means the VOC species selected as the instrument calibration basis for specification of the leak definition concentration. (For example, if a leak definition concentration is 10,000 ppm as methane, then any source emission that results in a local concentration that yields a meter reading of 10,000 on an instrument meter calibrated with methane would be classified as a leak. In this example, the leak definition concentration is 10,000 ppm and the reference compound is methane.)

3.6 *Response factor* means the ratio of the known concentration of a VOC compound to the observed meter reading when measured using an instrument calibrated with the reference compound specified in the applicable regulation.

3.7 *Response time* means the time interval from a step change in VOC concentration at the input of the sampling system to the time at which 90 percent of the corresponding final value is reached as displayed on the instrument readout meter.

4.0 Interferences. [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hazardous Pollutants. Several of the compounds, leaks of which may be determined by this method, may be irritating or corrosive to tissues (*e.g.*, heptane) or may be toxic (*e.g.*, benzene, methyl alcohol). Nearly all are fire hazards. Compounds in emissions should be determined through familiarity with the source. Appropriate precautions can be found in reference documents, such as reference No. 4 in Section 16.0.

6.0 Equipment and Supplies

A VOC monitoring instrument meeting the following specifications is required:

6.1 The VOC instrument detector shall respond to the compounds being processed. Detector types that may meet this requirement include, but are not limited to, catalytic oxidation, flame ionization, infrared absorption, and photoionization.

6.2 The instrument shall be capable of measuring the leak definition concentration specified in the regulation.

6.3 The scale of the instrument meter shall be readable to ±2.5 percent of the specified leak definition concentration.

6.4 The instrument shall be equipped with an electrically driven pump to ensure that a sample is provided to the detector at a constant flow rate. The nominal sample flow rate, as measured at the sample probe tip, shall be 0.10 to 3.0 l/min (0.004 to 0.1 ft^3/min) when the probe is fitted with a glass wool plug or filter that may be used to prevent plugging of the instrument.

6.5 The instrument shall be equipped with a probe or probe extension or sampling not to exceed 6.4 mm (¼ in) in outside diameter, with a single end opening for admission of sample.

6.6 The instrument shall be intrinsically safe for operation in explosive atmospheres as defined by the National Electrical Code by the National Fire Prevention Association or other applicable regulatory code for operation in any explosive atmospheres that may be encountered in its use. The instrument shall, at a minimum, be intrinsically safe for Class 1, Division 1 conditions, and/or Class 2, Division 1 conditions, as appropriate, as defined by the example code. The instrument shall not be operated with any safety device, such as an exhaust flame arrestor, removed.

7.0 Reagents and Standards

7.1 Two gas mixtures are required for instrument calibration and performance evaluation:

7.1.1 Zero Gas. Air, less than 10 parts per million by volume (ppmv) VOC.

7.1.2 Calibration Gas. For each organic species that is to be measured during individual source surveys, obtain or prepare a known standard in air at a concentration approximately equal to the applicable leak definition specified in the regulation.

7.2 Cylinder Gases. If cylinder calibration gas mixtures are used, they must be analyzed and certified by the manufacturer to be within 2 percent accuracy, and a shelf life must be specified. Cylinder standards must be either reanalyzed or replaced at the end of the specified shelf life.

7.3 Prepared Gases. Calibration gases may be prepared by the user according to any accepted gaseous preparation procedure that will yield a mixture accurate to within 2 percent. Prepared standards must be replaced each day of use unless it is demonstrated that degradation does not occur during storage.

7.4 Mixtures with non-Reference Compound Gases. Calibrations may be performed using a compound other than the reference compound. In this case, a conversion factor must be determined for the alternative compound such that the resulting meter readings during source surveys can be converted to reference compound results.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Instrument Performance Evaluation. Assemble and start up the instrument according to the manufacturer's instructions for recommended warmup period and preliminary adjustments.

8.1.1 Response Factor. A response factor must be determined for each compound that is to be measured, either by testing or from reference sources. The response factor tests are required before placing the analyzer into service, but do not have to be repeated at subsequent intervals.

8.1.1.1 Calibrate the instrument with the reference compound as specified in the applicable regulation. Introduce the calibration gas mixture to the analyzer and record the observed meter reading. Introduce zero gas until a stable reading is obtained. Make a total of three measurements by alternating between the calibration gas and zero gas. Calculate the response factor for each repetition and the average response factor.

8.1.1.2 The instrument response factors for each of the individual VOC to be measured shall be less than 10 unless otherwise specified in the applicable regulation. When no instrument is available that meets this specification when calibrated with the reference VOC specified in the applicable regulation, the available instrument may be calibrated with one of the VOC to be measured, or any other VOC, so long as the instrument then has a response factor of less than 10 for each of the individual VOC to be measured.

8.1.1.3 Alternatively, if response factors have been published for the compounds of interest for the instrument or detector type, the response factor determination is not required, and existing results may be referenced. Examples of published response factors for flame ionization and catalytic oxidation detectors are included in References 1–3 of Section 17.0.

8.1.2 Calibration Precision. The calibration precision test must be completed prior to placing the analyzer into service and at subsequent 3-month intervals or at the next use, whichever is later.

8.1.2.1 Make a total of three measurements by alternately using zero gas and the specified calibration gas. Record the meter readings. Calculate the average algebraic difference between the meter readings and the known value. Divide this average difference by the known calibration value and multiply by 100 to express the resulting calibration precision as a percentage.

8.1.2.2 The calibration precision shall be equal to or less than 10 percent of the calibration gas value.

8.1.3 Response Time. The response time test is required before placing the instrument into service. If a modification to the sample pumping system or flow configuration is made that would change the response time, a new test is required before further use.

8.1.3.1 Introduce zero gas into the instrument sample probe. When the meter reading has stabilized, switch quickly to the specified calibration gas. After switching, measure the time required to attain 90 percent of the final stable reading. Perform this test sequence three times and record the results. Calculate the average response time.

8.1.3.2 The instrument response time shall be equal to or less than 30 seconds. The instrument pump, dilution probe (if any), sample probe, and probe filter that will be used during testing shall all be in place during the response time determination.

8.2 Instrument Calibration. Calibrate the VOC monitoring instrument according to Section 10.0.

8.3 Individual Source Surveys.

8.3.1 Type I—Leak Definition Based on Concentration. Place the probe inlet at the surface of the component interface where leakage could occur. Move the probe along the interface periphery while observing the instrument readout. If an increased meter reading is observed, slowly sample the interface where leakage is indicated until the maximum meter reading is obtained. Leave the probe inlet at this maximum reading location for approximately two times the instrument response time. If the maximum observed meter reading is greater than the leak definition in the applicable regulation, record and report the results as specified in the regulation reporting requirements. Examples of the application of this general technique to specific equipment types are:

8.3.1.1 Valves. The most common source of leaks from valves is the seal between the stem and housing. Place the probe at the interface where the stem exits the packing gland and sample the stem circumference. Also, place the probe at the interface of the packing gland take-up flange seat and sample the periphery. In addition, survey valve housings of multipart assembly at the surface of all interfaces where a leak could occur.

8.3.1.2 Flanges and Other Connections. For welded flanges, place the probe at the outer edge of the flange-gasket interface and sample the circumference of the flange. Sample other types of nonpermanent joints (such as threaded connections) with a similar traverse.

8.3.1.3 Pumps and Compressors. Conduct a circumferential traverse at the outer surface of the pump or compressor shaft and seal interface. If the source is a rotating shaft, position the probe inlet within 1 cm of the shaft-seal interface for the survey. If the housing configuration prevents a complete traverse of the shaft periphery, sample all accessible portions. Sample all other joints on the pump or compressor housing where leakage could occur.

8.3.1.4 Pressure Relief Devices. The configuration of most pressure relief devices prevents sampling at the sealing seat interface. For those devices equipped with an enclosed extension, or horn, place the probe inlet at approximately the center of the exhaust area to the atmosphere.

8.3.1.5 Process Drains. For open drains, place the probe inlet at approximately the center of the area open to the atmosphere. For covered drains, place the probe at the surface of the cover interface and conduct a peripheral traverse.

8.3.1.6 Open-ended Lines or Valves. Place the probe inlet at approximately the center of the opening to the atmosphere.

8.3.1.7 Seal System Degassing Vents and Accumulator Vents. Place the probe inlet at approximately the center of the opening to the atmosphere.

8.3.1.8 Access door seals. Place the probe inlet at the surface of the door seal interface and conduct a peripheral traverse.

8.3.2 Type II—"No Detectable Emission". Determine the local ambient VOC concentration around the source by moving the probe randomly upwind and downwind at a distance of one to two meters from the source. If an interference exists with this determination due to a nearby emission or leak, the local ambient concentration may be determined at distances closer to the source, but in no case shall the distance be less than 25 centimeters. Then move the probe inlet to the surface of the source and determine the concentration as outlined in Section 8.3.1. The difference between these concentrations determines whether there are no detectable emissions. Record and report the results as specified by the regulation. For those cases where the regulation requires a specific device installation, or that specified vents be ducted or piped to a control device, the existence of these conditions shall be visually confirmed. When the regulation also requires that no detectable emissions exist, visual observations and sampling surveys are required. Examples of this technique are:

8.3.2.1 Pump or Compressor Seals. If applicable, determine the type of shaft seal. Perform a survey of the local area ambient VOC concentration and determine if detectable emissions exist as described in Section 8.3.2.

8.3.2.2 Seal System Degassing Vents, Accumulator Vessel Vents, Pressure Relief Devices. If applicable, observe whether or not the applicable ducting or piping exists. Also, determine if any sources exist in the ducting or piping where emissions could occur upstream of the control device. If the required ducting or piping exists and there are no sources where the emissions could be vented to the atmosphere upstream of the control device, then it is presumed that no detectable emissions are present. If there are sources in the ducting or piping where emissions could be vented or sources where leaks could occur, the sampling surveys described in Section 8.3.2 shall be used to determine if detectable emissions exist.

8.3.3 Alternative Screening Procedure.

8.3.3.1 A screening procedure based on the formation of bubbles in a soap solution that is sprayed on a potential leak source may be used for those sources that do not have continuously moving parts, that do not have surface temperatures greater than the boiling point or less than the freezing point of the soap solution, that do not have open areas to the atmosphere that the soap solution cannot bridge, or that do not exhibit evidence of liquid leakage. Sources that have these conditions present must be surveyed using the instrument technique of Section 8.3.1 or 8.3.2.

8.3.3.2 Spray a soap solution over all potential leak sources. The soap solution may be a commercially available leak detection solution or may be prepared using concentrated detergent and water. A pressure sprayer or squeeze bottle may be used to dispense the solution. Observe the potential leak sites to determine if any bubbles are formed. If no bubbles are observed, the source is presumed to have no detectable emissions or leaks as applicable. If any bubbles are observed, the instrument techniques of Section 8.3.1 or 8.3.2 shall be used to determine if a leak exists, or if the source has detectable emissions, as applicable.

9.0 Quality Control

Section	Quality control measure	Effect
8.1.2	Instrument calibration precision check	Ensure precision and accuracy, respectively, of instrument response to standard.
10.0	Instrument calibration.	

10.0 Calibration and Standardization

10.1 Calibrate the VOC monitoring instrument as follows. After the appropriate warmup period and zero internal calibration procedure, introduce the calibration gas into the instrument sample probe. Adjust the instrument meter readout to correspond to the calibration gas value.

Note: If the meter readout cannot be adjusted to the proper value, a malfunction of the analyzer is indicated and corrective actions are necessary before use.

11.0 Analytical Procedures. [Reserved]

12.0 Data Analyses and Calculations. [Reserved]

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Dubose, D.A., and G.E. Harris. Response Factors of VOC Analyzers at a Meter Reading of 10,000 ppmv for Selected Organic Compounds. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA 600/2–81051. September 1981.

2. Brown, G.E., *et al.* Response Factors of VOC Analyzers Calibrated with Methane for Selected Organic Compounds. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA 600/2–81–022. May 1981.

3. DuBose, D.A. *et al.* Response of Portable VOC Analyzers to Chemical Mixtures. U.S. Environmental Protection Agency, Research Triangle Park, NC. Publication No. EPA 600/2–81–110. September 1981.

4. Handbook of Hazardous Materials: Fire, Safety, Health. Alliance of American Insurers. Schaumberg, IL. 1983.

17.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

Method 22—Visual Determination of Fugitive Emissions From Material Sources and Smoke Emissions From Flares

Note: This method is not inclusive with respect to observer certification. Some material is incorporated by reference from Method 9.

1.0 Scope and Application

This method is applicable for the determination of the frequency of fugitive emissions from stationary sources, only as specified in an applicable subpart of the regulations. This method also is applicable for the determination of the frequency of visible smoke emissions from flares.

2.0 Summary of Method

2.1 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.

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

2.3 This method determines the amount of time that visible emissions occur during the observation period (*i.e.*, the accumulated emission time). This method does not require that the opacity of emissions be determined. Since this procedure requires only the determination of whether visible emissions occur and does not require the determination of opacity levels, observer certification according to the procedures of Method 9 is not required. However, it is necessary that the observer is knowledgeable with respect to the general procedures for determining the presence of visible emissions. At a minimum, the observer must be trained and knowledgeable regarding the effects of background contrast, ambient lighting, observer position relative to lighting, wind, and the presence of uncombined water (condensing water vapor) on the visibility of emissions. This training is to be obtained from written materials found in References 1 and 2 or from the lecture portion of the Method 9 certification course.

3.0 Definitions

3.1 *Emission frequency* means the percentage of time that emissions are visible during the observation period.

3.2 *Emission time* means the accumulated amount of time that emissions are visible during the observation period.

3.3 *Fugitive emissions* means emissions generated by an affected facility which is not collected by a capture system and is released to the atmosphere. This includes emissions that (1) escape capture by process equipment exhaust hoods; (2) are emitted during material transfer; (3) are emitted from buildings housing material processing or handling equipment; or (4) are emitted directly from process equipment.

3.4 *Observation period* means the accumulated time period during which observations are conducted, not to be less than the period specified in the applicable regulation.

3.5 *Smoke emissions* means a 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.

4.0 Interferences

4.1 Occasionally, fugitive emissions from sources other than the affected facility (*e.g.*, road dust) may prevent a clear view of the affected facility. This may particularly be a problem during periods of high wind. If the view of the potential emission points is obscured to such a degree that the observer questions the validity of continuing observations, then the observations shall be terminated, and the observer shall clearly note this fact on the data form.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment

6.1 Stopwatches (two). Accumulative type with unit divisions of at least 0.5 seconds.

6.2 Light Meter. Light meter capable of measuring illuminance in the 50 to 200 lux range, required for indoor observations only.

7.0 Reagents and Supplies. [Reserved]

8.0 Sample Collection, Preservation, Storage, and Transfer. [Reserved]

9.0 Quality Control. [Reserved]

10.0 Calibration and Standardization. [Reserved]

11.0 Analytical Procedure

11.1 Selection of Observation Location. Survey the affected facility, or the building or structure housing the process to be observed, and determine the locations of potential emissions. If the affected facility is located inside a building, determine an observation location that is consistent with the requirements of the applicable regulation (*i.e.*, outside observation of emissions escaping the building/structure or inside observation of emissions directly emitted from the affected facility process unit). Then select a position that enables a clear view of the potential emission point(s) of the affected facility or of the building or structure housing the affected facility, as appropriate for the applicable subpart. A position at least 4.6 m (15 feet), but not more than 400 m (0.25 miles), from the emission source is recommended. For outdoor locations, select a position where the sunlight is not shining directly in the observer's eyes.

11.2 Field Records.

11.2.1 Outdoor Location. Record the following information on the field data sheet (Figure 22–1): Company name, industry, process unit, observer's name, observer's affiliation, and date. Record also the estimated wind speed, wind direction, and sky condition. Sketch the process unit being observed, and note the observer location relative to the source and the sun. Indicate the potential and actual emission points on the sketch.

11.2.2 Indoor Location. Record the following information on the field data sheet (Figure 22–2): Company name, industry, process unit, observer's name, observer's affiliation, and date. Record as appropriate the type, location, and intensity of lighting on the data sheet. Sketch the process unit being observed, and note the observer location relative to the source. Indicate the potential and actual fugitive emission points on the sketch.

11.3 Indoor Lighting Requirements. For indoor locations, use a light meter to measure the level of illumination at a location as close to the emission source(s) as is feasible. An illumination of greater than 100 lux (10 foot candles) is considered necessary for proper application of this method.

11.4 Observations.

11.4.1 Procedure. Record the clock time when observations begin. Use one stopwatch to monitor the duration of the observation period. Start this stopwatch when the observation period begins. If the observation period is divided into two or more segments by process shutdowns or observer rest breaks (see Section 11.4.3), stop the stopwatch when a break begins and restart the stopwatch without resetting it when the break ends. Stop the stopwatch at the end of the observation period. The accumulated time indicated by this stopwatch is the duration of observation period. When the observation period is completed, record the clock time. During the observation period, continuously watch the emission source. Upon observing an emission (condensed water vapor is not considered an emission), start the second accumulative stopwatch; stop the watch when the emission stops. Continue this procedure for the entire observation period. The accumulated elapsed time on this stopwatch is the total time emissions were visible during the observation period (*i.e.*, the emission time.)

11.4.2 Observation Period. Choose an observation period of sufficient length to meet the requirements for determining compliance with the emission standard in the applicable subpart of the regulations. When the length of the observation period is specifically stated in the applicable subpart, it may not be necessary to observe the source for this entire period if the emission time required to indicate noncompliance (based on the specified observation period) is observed in a shorter time period. In other words, if the regulation prohibits emissions for more than 6 minutes in any hour, then observations may (optional) be stopped after an emission time of 6 minutes is exceeded. Similarly, when the regulation is expressed as an emission frequency and the regulation prohibits emissions for greater than 10 percent of the time in any hour, then observations may (optional) be terminated after 6 minutes of emission are observed since 6 minutes is 10 percent of an hour. In any case, the observation period shall not be less than 6 minutes in duration. In some cases, the process operation may be intermittent or cyclic. In such cases, it may be convenient for the observation period to coincide with the length of the process cycle.

11.4.3 Observer Rest Breaks. Do not observe emissions continuously for a period of more than 15 to 20 minutes without taking a rest break. For sources requiring observation periods of greater than 20 minutes, the observer shall take a break of not less than 5 minutes and not more than 10 minutes after every 15 to 20 minutes of observation. If continuous observations are desired for extended time periods, two observers can alternate between making observations and taking breaks.

11.5 Recording Observations. Record the accumulated time of the observation period on the data sheet as the observation period duration. Record the accumulated time emissions were observed on the data sheet as the emission time. Record the clock time the observation period began and ended, as well as the clock time any observer breaks began and ended.

12.0 Data Analysis and Calculations

If the applicable subpart requires that the emission rate be expressed as an emission frequency (in percent), determine this value as follows: Divide the accumulated emission time (in seconds) by the duration of the observation period (in seconds) or by any minimum observation period required in the applicable subpart, if the actual observation period is less than the required period, and multiply this quotient by 100.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

1. Missan, R., and A. Stein. Guidelines for Evaluation of Visible Emissions Certification, Field Procedures, Legal Aspects, and Background Material. EPA Publication No. EPA–340/1–75–007. April 1975.

2. Wohlschlegel, P., and D.E. Wagoner. Guideline for Development of a Quality Assurance Program: Volume IX— Visual Determination of Opacity Emissions from Stationary Sources. EPA Publication No. EPA–650/4–74–005i. November 1975.

BILLING CODE 6560–50–P

17.0 Tables, Diagrams, Flowcharts, and Validation Data

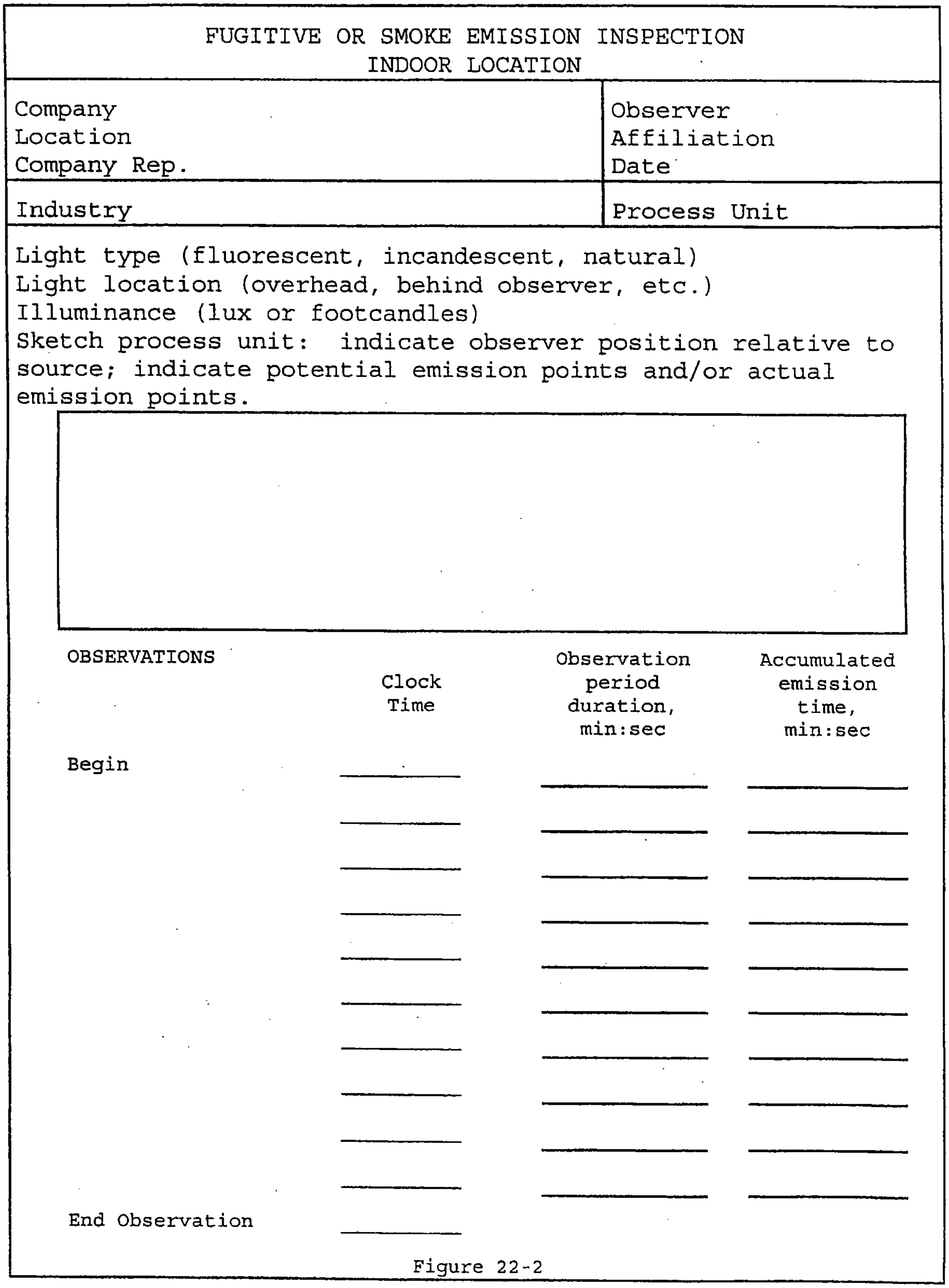
FUGITIVE OR SMOKE EMISSION INSPECTION
OUTDOOR LOCATION

Company Location Company Rep.	Observer Affiliation Date
Sky Conditions Precipitation	Wind Direction Wind Speed
Industry	Process Unit

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
Begin Observation			
End Observation			

Figure 22-1



FUGITIVE OR SMOKE EMISSION INSPECTION
INDOOR LOCATION

Company Location Company Rep.	Observer Affiliation 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
Begin			
End Observation			

Figure 22-2

BILLING CODE 6560-50-C

* * * * *

Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.
Volatile organic compounds Water.	No CAS Number assigned 7732–18–5

1.2 Applicability. This method is applicable for the determination of volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or other related surface coatings.

1.3 Precision and Bias. Intra-and inter-laboratory analytical precision statements are presented in Section 13.1. No bias has been identified.

2.0 Summary of Method

2.1 Standard methods are used to determine the volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or other related surface coatings.

3.0 Definitions

3.1 *Waterborne coating* means any coating which contains more than 5 percent water by weight in its volatile fraction.

3.2 *Multicomponent coatings* are coatings that are packaged in two or more parts, which are combined before application. Upon combination a coreactant from one part of the coating chemically reacts, at ambient conditions, with a coreactant from another part of the coating.

3.3 *Ultraviolet (UV) radiation-cured coatings* are coatings which contain unreacted monomers that are polymerized by exposure to ultraviolet light.

4.0 Interferences. [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

5.2 Hazardous Components. Several of the compounds that may be contained in the coatings analyzed by this method may be irritating or corrosive to tissues (e.g., heptane) or may be toxic (e.g., benzene, methyl alcohol). Nearly all are fire hazards. Appropriate precautions can be found in reference documents, such as Reference 3 of Section 16.0.

6.0 Equipment and Supplies

The equipment and supplies specified in the ASTM methods listed in Sections 6.1 through 6.6 (incorporated by reference—see § 60.17 for acceptable versions of the methods) are required:

6.1 ASTM D 1475–60, 80, or 90, Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products.

6.2 ASTM D 2369–81, 87, 90, 92, 93, or 95, Standard Test Method for Volatile Content of Coatings.

6.3 ASTM D 3792–79 or 91, Standard Test Method for Water Content of Water Reducible Paints by Direct Injection into a Gas Chromatograph.

6.4 ASTM D 4017–81, 90, or 96a, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method.

6.5 ASTM 4457–85 91, Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph.

6.6 ASTM D 5403–93, Standard Test Methods for Volatile Content of Radiation Curable Materials.

7.0 Reagents and Standards

7.1 The reagents and standards specified in the ASTM methods listed in Sections 6.1 through 6.6 are required.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Follow the sample collection, preservation, storage, and transport procedures described in Reference 1 of Section 16.0.

9.0 Quality Control

9.1 Reproducibility

Note: Not applicable to UV radiation-cured coatings). The variety of coatings that may be subject to analysis makes it necessary to verify the ability of the analyst and the analytical procedures to obtain reproducible results for the coatings tested. Verification is accomplished by running duplicate analyses on each sample tested (Sections 11.2 through 11.4) and comparing the results with the intra-laboratory precision statements (Section 13.1) for each parameter.

9.2 Confidence Limits for Waterborne Coatings. Because of the inherent increased imprecision in the determination of the VOC content of waterborne coatings as the weight percent of water increases, measured parameters for waterborne coatings are replaced with appropriate confidence limits (Section 12.6). These confidence limits are based on measured parameters and inter-laboratory precision statements.

10.0 Calibration and Standardization

10.1 Perform the calibration and standardization procedures specified in the ASTM methods listed in Sections 6.1 through 6.6.

11.0 Analytical Procedure

Additional guidance can be found in Reference 2 of Section 16.0.

11.1 Non Thin-film Ultraviolet Radiation-cured (UV radiation-cured) Coatings.

11.1.1 Volatile Content. Use the procedure in ASTM D 5403 to determine the volatile matter content of the coating except the curing test described in NOTE 2 of ASTM D 5403 is required.

11.1.2 Water Content. To determine water content, follow Section 11.3.2.

11.1.3 Coating Density. To determine coating density, follow Section 11.3.3.

11.1.4 Solids Content. To determine solids content, follow Section 11.3.4.

11.1.5 To determine if a coating or ink can be classified as a thin-film UV cured coating or ink, use the equation in Section 12.2. If C is less than 0.2 g and A is greater than or equal to 225 cm^2 (35 in^2) then the coating or ink is considered a thin-film UV radiation-cured coating and ASTM D 5403 is not applicable.

Note: As noted in Section 1.4 of ASTM D 5403, this method may not be applicable to radiation curable materials wherein the volatile material is water.

11.2 Multi-component Coatings.

11.2.1 Sample Preparation.

11.2.1.1 Prepare about 100 ml of sample by mixing the components in a storage container, such as a glass jar with a screw top or a metal can with a cap. The storage container should be just large enough to hold the mixture. Combine the components (by weight or volume) in the ratio recommended by the manufacturer. Tightly close the container between additions and during mixing to prevent loss of volatile materials. However, most manufacturers mixing instructions are by volume. Because of possible error caused by expansion of the liquid when measuring the volume, it is recommended that the components be combined by weight. When weight is used to combine the components and the manufacturer's recommended ratio is by volume, the density must be determined by Section 11.3.3.

11.2.1.2 Immediately after mixing, take aliquots from this 100 ml sample for determination of the total volatile content, water content, and density.

11.2.2 Volatile Content. To determine total volatile content, use the apparatus and reagents described in ASTM D2369 Sections 3 and 4 (incorporated by reference—see § 60.17 for the approved versions of the standard), respectively, and use the following procedures:

11.2.2.1 Weigh and record the weight of an aluminum foil weighing dish. Add 3 ± 1 ml of suitable solvent as specified in ASTM D2369 to the weighing dish. Using a syringe as specified in ASTM D2369, weigh to 1 mg, by difference, a sample of coating into the weighing dish. For coatings believed to have a volatile content less than 40 weight percent, a suitable size is 0.3 + 0.10 g, but for coatings believed to have a volatile content greater than 40 weight percent, a suitable size is 0.5 ± 0.1 g.

Note: If the volatile content determined pursuant to Section 12.4 is not in the range corresponding to the sample size chosen repeat the test with the appropriate sample size. Add the specimen dropwise, shaking (swirling) the dish to disperse the specimen completely in the solvent. If the material forms a lump that cannot be dispersed, discard the specimen and prepare a new one. Similarly, prepare a duplicate. The sample shall stand for a minimum of 1 hour, but no more than 24 hours prior to being oven cured at 110 ± 5°C (230 ± 9°F) for 1 hour.

11.2.2.2 Heat the aluminum foil dishes containing the dispersed specimens in the forced draft oven for 60 min at 110 ± 5°C (230 ± 9°F). Caution—provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.

11.2.2.3 Remove the dishes from the oven, place immediately in a desiccator, cool to ambient temperature, and weigh to within 1 mg.

11.2.2.4 Run analyses in pairs (duplicate sets) for each coating mixture until the criterion in Section 11.4 is met. Calculate W_V following Equation 24–2 and record the arithmetic average.

11.2.3 Water Content. To determine water content, follow Section 11.3.2.

11.2.4 Coating Density. To determine coating density, follow Section 11.3.3.

11.2.5 Solids Content. To determine solids content, follow Section 11.3.4.

11.2.6 Exempt Solvent Content. To determine the exempt solvent content, follow Section 11.3.5.

Note: For all other coatings (*i.e.*, water-or solvent-borne coatings) not covered by multicomponent or UV radiation-cured coatings, analyze as shown below:

11.3 Water-or Solvent-borne coatings.

11.3.1 Volatile Content. Use the procedure in ASTM D 2369 to determine the volatile matter content (may include water) of the coating.

11.3.1.1 Record the following information:

W_1 = weight of dish and sample before heating, g
W_2 = weight of dish and sample after heating, g
W_3 = sample weight, g.

11.3.1.2 Calculate the weight fraction of the volatile matter (W_v) for each analysis as shown in Section 12.3.

11.3.1.3 Run duplicate analyses until the difference between the two values in a set is less than or equal to the intra-laboratory precision statement in Section 13.1.

11.3.1.4 Record the arithmetic average (W_v).

11.3.2 Water Content. For waterborne coatings only, determine the weight fraction of water (W_w) using either ASTM D 3792 or ASTM D 4017.

11.3.2.1 Run duplicate analyses until the difference between the two values in a set is less than or equal to the intra-laboratory precision statement in Section 13.1.

11.3.2.2 Record the arithmetic average (w_w).

11.3.3 Coating Density. Determine the density (Dc, kg/l) of the surface coating using the procedure in ASTM D 1475.

11.3.3.1 Run duplicate analyses until each value in a set deviates from the mean of the set by no more than the intra-laboratory precision statement in Section 13.1.

11.3.3.2 Record the arithmetic average (D_c).

11.3.4 Solids Content. Determine the volume fraction (V_s) solids of the coating by calculation using the manufacturer's formulation.

11.3.5 Exempt Solvent Content. Determine the weight fraction of exempt solvents (W_E) by using ASTM Method D4457. Run a duplicate set of determinations and record the arithmetic average (W_E).

11.4 Sample Analysis Criteria. For W_v and W_w, run duplicate analyses until the difference between the two values in a set is less than or equal to the intra-laboratory precision statement for that parameter. For D_c, run duplicate analyses until each value in a set deviates from the mean of the set by no more than the intra-laboratory precision statement. If, after several attempts, it is concluded that the ASTM procedures cannot be used for the specific coating with the established intra-laboratory precision (excluding UV radiation-cured coatings), the U.S. Environmental Protection Agency (EPA) will assume responsibility for providing the necessary procedures for revising the method or precision statements upon written request to: Director, Emissions, Monitoring, and Analysis Division, MD–14, Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

12.0 Calculations and Data Analysis

12.1 Nomenclature.

A = Area of substrate, cm^2, (in^2).
C = Amount of coating or ink added to the substrate, g.
D_c = Density of coating or ink, g/cm^3 (g/in^3).
F = Manufacturer's recommended film thickness, cm (in).
W_o = Weight fraction of nonaqueous volatile matter, g/g.
W_s = Weight fraction of solids, g/g.
W_v = Weight fraction of the volatile matter, g/g.
W_w = Weight fraction of the water, g/g.

12.2 To determine if a coating or ink can be classified as a thin-film UV cured coating or ink, use the following equation:

$$C = F A D_c \qquad \text{Eq. 24-1}$$

12.3 Calculate W_v for each analysis as shown below:

$$W_v = \frac{W_1 - W_2}{W_3} \qquad \text{Eq. 24-2}$$

12.4 Nonaqueous Volatile Matter.

12.4.1 Solvent-borne Coatings.

$$W_o = W_v \qquad \text{Eq. 24-3}$$

12.4.2 Waterborne Coatings.

$$W_o = W_v - W_w \qquad \text{Eq. 24-4}$$

12.4.3 Coatings Containing Exempt Solvents.

$$W_o = W_v - W_E - W_w \qquad \text{Eq. 24-5}$$

12.5 Weight Fraction Solids.

$$W_s = 1 - W_v \qquad \text{Eq. 24-6}$$

12.6 Confidence Limit Calculations for Waterborne Coatings. To calculate the lower confidence limit, subtract the appropriate inter-laboratory precision value from the measured mean value for that parameter. To calculate the upper confidence limit, add the appropriate inter-laboratory precision value to the measured mean value for that parameter. For W_v and D_c, use the lower confidence limits; for W_w, use the upper confidence limit. Because W_s is calculated, there is no adjustment for this parameter.