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Title 40—PROTECTION OF ENVIRONMENT

Chapter I—Environmental Protection Agency

SUBCHAPTER C—AIR PROGRAMS

PART 60—STANDARDS OF PERFORM-ANCE FOR NEW STATIONARY SOURCES

On August 17, 1971 (36 F.R. 15704) pursuant to section 111 of the Clean Air Act as amended, the Administrator proposed standards of performance for steam generators, portland c e m e n t plants, incinerators, nitric acid plants, and sulfuric acid plants. The proposed standards, applicable to sources the construction or modification of which was initiated after August 17, 1971, included emission limits for one or more of four pollutants (particulate matter, sulfur dioxide, nitrogen oxides, and sulfuric acid mist) for each source category. The proposal included requirements for performance testing, stack gas monitoring, record keeping and reporting, and procedures by which EPA will provide preconstruction review and determine the applicability of the standards to specific sources.

Interested parties were afforded an opportunity to participate in the rule making by submitting comments. A total of more than 200 interested parties, including Federal, State, and local agencies, citizens groups, and commercial and industrial organizations submitted comments. Following a review of the proposed regulations and consideration of the comments, the regulations, including the appendix, have been revised and are being promulgated today. The principal revisions are described below:

1. Particulate matter performance testing procedures have been revised to eliminate the requirement for impingers in the sampling train. Compliance will be based only on material collected in the dry filter and the probe preceding the filter. Emission limits have been adjusted as appropriate to reflect the change in test methods. The adjusted standards require the same degree of particulate control as the originally proposed standards.

2. Provisions have been added whereby alternative test methods can be used to determine compliance. Any person who proposes the use of an alternative method will be obliged to provide evidence that the alternative method is equivalent to the reference method.

3. The definition of modification, as it pertains to increases in production rate and changes of fuels, has been clarified. Increases in production rates up to design capacity will not be considered a modification nor will fuel switches if the equipment was/originally designed to accommodate such fuels. These provisions will eliminate inequities where equipment had been put into partial operation prior to the proposal of the standards.

4. The definition of a new source was clarified to include construction which

is completed within an organization as well as the more common situations where the facility is designed and constructed by a contractor.

5. The provisions regarding requests for EPA plan review and determination of construction or modification have been modified to emphasize that the submittal of such requests and attendant information is purely voluntary. Submittal of such a request will not bind the operator to supply further information; however, lack of sufficient information may prevent the Administrator from rendering an opinion. Further provisions have been added to the effect that information submitted voluntarily for such plan review or determination of applicability will be considered confidential, if the owner or operator requests such confidentiality.

6. Requirements for notifying the Administrator prior to commencing construction have been deleted. As proposed, the provision would have required notification prior to the signing of a contract for construction of a new source. Owners and operators still will be required to notify the Administrator 30 days prior to initial operation and to confirm the action within 15 days after startup.

7. Revisions were incorporated to permit compliance testing to be deferred up to 60 days after achieving the maximum production rate but no longer than 180 days after initial startup. The proposed regulation could have required testing within 60 days after startup but defined startup as the beginning of routine operation. Owners or operators will be required to notify the Administrator at least 10 days prior to compliance testing so that an EPA observer can be on hand. Procedures have been modified so that the equipment will have to be operated at maximum expected production rate, rather than rated capacity, during compliance tests.

8. The criteria for evaluating performance testing results have been simplified to eliminate the requirement that all values be within 35 percent of the average. Compliance will be based on the average of three repetitions conducted in the specified manner.

9. Provisions were added to require owners or operators of affected facilities to maintain records of compliance tests, monitoring equipment, pertinent analyses, feed rates, production rates, etc. for 2 years and to make such information available on request to the Administrator. Owners or operators will be required to summarize the recorded data daily and to convert recorded data into the applicable units of the standard.

10. Modifications were made to the visible emission standards for steam generators, cement plants, nitric acid plants, and sulfuric acid plants. The Ringelmann standards have been deleted; all limits will be based on opacity. In every case, the equivalent opacity will be at least as stringent as the proposed Ringelmann number. In addition, requirements have been altered for three of the source categories so that allowable emissions will be less than 10 percent opacity rather than 5 percent or less opacity. There were many comments

that observers could not accurately evaluate emissions of 5 percent opacity. In addition, drafting errors in the proposed visible emission limits for cement kilns and steam generators were corrected. Steam generators will be limited to visible emissions not greater than 20 percent opacity and cement kilns to not greater than 10 percent opacity.

11. Specifications for monitoring devices were clarified, and directives for calibration were included. The instruments are to be calibrated at least once a day, or more often if specified by the manufacturer. Additional guidance on the selection and use of such instruments will be provided at a later date.

12. The requirement for sulfur dioxide monitoring at steam generators was deleted for those sources which will achieve the standard by burning low-sulfur fuel, provided that fuel analysis is conducted and recorded daily. American Society for Testing and Materials sampling techniques are specified for coal and fuel oil.

13. Provisions were added to the steam generator standards to cover those instances where mixed fuels are burned. Allowable emissions will be determined by prorating the heat input of each fuel, however, in the case of sulfur dioxide, the provisions allow operators the option of burning low-sulfur fuels (probably natural gas) as a means of compliance.

14. Steam generators fired with lignite have been exempted from the nitrogen oxides limit. The revision was made in view of the lack of information on some types of lignite burning. When more information is developed, nitrogen oxides standards may be extended to lignite fired steam generators.

15. A provision was added to make it explicit that the sulfuric acid plant standards will not apply to scavenger acid plants. As stated in the background document, APTD 0711, which was issued at the time the proposed standards were published, the standards were not meant to apply to such operations, e.g., where sulfuric acid plants are used primarily to control sulfur dioxide or other sulfur compounds which would otherwise be vented into the atmosphere.

16. The regulation has been revised to provide that all materials submitted pursuant to these regulations will be directed to EPA's Office of General Enforcement.

17. Several other technical changes have also been made. States and interested parties are urged to make a careful reading of these regulations.

As required by section 111 of the Act, the standards of performance promulgated herein "reflect the degree of emission reduction which (taking into account the cost of achieving such reduction) the Administrator determines has been adequately demonstrated". The standards of performance are based on stationary source testing conducted by the Environmental Protection Agency and/or contractors and on data derived from various other sources, including the available technical literature. In the comments on the proposed standards, many questions were raised as to costs and

demonstrated capability of control systems to meet the standards. These comments have been evaluated and investigated, and it is the Administrator's judgment that emission control systems capable of meeting the standards have been adequately demonstrated and that the standards promulgated herein are achievable at reasonable costs.

The regulations establishing standards of performance for steam generators, incinerators, cement plants, nitric acid plants, and sulfuric acid plants are hereby promulgated effective on publication and apply to sources, the construction or modification of which was commenced after August 17, 1971.

Dated: December 16, 1971.

WILLIAM D. RUCKELSHAUS,
Administrator,
Environmental Protection Agency.

A new Part 60 is added to Chapter I, Title 40, Code of Federal Regulations, as follows:

AUTHORITY: The provisions of this Part 60 issued under sections 111, 114, Clean Air Act; Public Law 91–604, 84 Stat. 1713.

Subpart A—General Provisions

§ 60.1 Applicability.

The provisions of this part apply to the owner or operator of any stationary source, which contains an affected facility the construction or modification of which is commenced after the date of publication in this part of any proposed standard applicable to such facility.

§ 60.2 Definitions.

As used in this part, all terms not defined herein shall have the meaning given them in the Act:

(a) "Act" means the Clean Air Act (42 U.S.C. 1857 et seq., as amended by Public Law 91–604, 84 Stat. 1676).

(b) "Administrator" means the Administrator of the Environmental Protection Agency or his authorized representative.

(c) "Standard" means a standard of performance proposed or promulgated under this part.

(d) "Stationary source" means any building, structure, facility, or installation which emits or may emit any air pollutant.

(e) "Affected facility" means, with reference to a stationary source, any apparatus to which a standard is applicable.

(f) "Owner or operator" means any person who owns, leases, operates, controls, or supervises an affected facility or a stationary source of which an affected facility is a part.

(g) "Construction" means fabrication, erection, or installation of an affected facility.

(h) "Modification" means any physical change in, or change in the method of operation of, an affected facility which increases the amount of any air pollutant (to which a standard applies) emitted by such facility or which results in the emission of any air pollutant (to which a standard applies) not previously emitted, except that:

(1) Routine maintenance, repair, and replacement shall not be considered physical changes, and

(2) The following shall not be considered a change in the method of operation:

(i) An increase in the production rate, if such increase does not exceed the operating design capacity of the affected facility;

(ii) An increase in hours of operation;

(iii) Use of an alternative fuel or raw material if, prior to the date any standard under this part becomes applicable to such facility, as provided by § 60.1, the affected facility is designed to accommodate such alternative use.

(i) "Commenced" means that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a binding agreement or contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

(j) "Opacity" means the degree to which emissions reduce the transmission of light and obscure the view of an object in the background.

(k) "Nitrogen oxides" means all oxides of nitrogen except nitrous oxide, as measured by test methods set forth in this part.

(l) "Standard of normal conditions" means 70° Fahrenheit (21.1° centigrade) and 29.92 in. Hg (760 mm. Hg).

(m) "Proportional sampling" means sampling at a rate that produces a constant ratio of sampling rate to stack gas flow rate.

(n) "Isokinetic sampling" means sampling in which the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

(o) "Startup" means the setting in operation of an affected facility for any purpose.

§ 60.3 Abbreviations.

The abbreviations used in this part have the following meanings in both capital and lower case:

B.t.u.—British thermal unit.
cal.—calorie(s).
c.f.m.—cubic feet per minute.
CO_2—carbon dioxide.
g.—gram(s).
gr.—grain(s).
mg.—milligram(s).
mm.—millimeter(s).
l.—liter(s).
nm.—nanometer(s), 10^{-9} meter.
μg.—microgram(s), 10^{-6} gram.
Hg.—mercury.
in.—inch(es).
K—1,000.
lb.—pound(s).
ml.—milliliter(s).
No.—number.
%—percent.
NO—nitric oxide.
NO_2—nitrogen dioxide.
NO_x—nitrogen oxides.
N.m.³—normal cubic meter.
s.c.f.—standard cubic feet.
SO_2—sulfur dioxide.
H_2SO_4—sulfuric acid.
SO_3—sulfur trioxide.

ft.³—cubic feet.
ft.²—square feet.
min.—minute(s).
hr.—hour(s).

§ 60.4 Address.

All applications, requests, submissions, and reports under this part shall be submitted in triplicate and addressed to the Environmental Protection Agency, Office of General Enforcement, Waterside Mall SW., Washington, DC 20460.

§ 60.5 Determination of construction or modification.

When requested to do so by an owner or operator, the Administrator will make a determination of whether actions taken or intended to be taken by such owner or operator constitute construction or modification or the commencement thereof within the meaning of this part.

§ 60.6 Review of plans.

(a) When requested to do so by an owner or operator, the Administrator will review plans for construction or modification for the purpose of providing technical advice to the owner or operator.

(b) (1) A separate request shall be submitted for each affected facility.

(2) Each request shall (i) identify the location of such affected facility, and (ii) be accompanied by technical information describing the proposed nature, size, design, and method of operation of such facility, including information on any equipment to be used for measurement or control of emissions.

(c) Neither a request for plans review nor advice furnished by the Administrator in response to such request shall (1) relieve an owner or operator of legal responsibility for compliance with any provision of this part or of any applicable State or local requirement, or (2) prevent the Administrator from implementing or enforcing any provision of this part or taking any other action authorized by the Act.

§ 60.7 Notification and record keeping.

(a) Any owner or operator subject to the provisions of this part shall furnish the Administrator written notification as follows:

(1) A notification of the anticipated date of initial startup of an affected facility not more than 60 days or less than 30 days prior to such date.

(2) A notification of the actual date of initial startup of an affected facility within 15 days after such date.

(b) Any owner or operator subject to the provisions of this part shall maintain for a period of 2 years a record of the occurrence and duration of any startup, shutdown, or malfunction in operation of any affected facility.

§ 60.8 Performance tests.

(a) Within 60 days after achieving the maximum production rate at which the affected facility will be operated, but not later than 180 days after initial startup of such facility and at such other times as may be required by the Administrator under section 114 of the Act, the owner or operator of such facility shall conduct performance test(s) and furnish the Administrator a written report of the results of such performance test(s).

(b) Performance tests shall be conducted and results reported in accordance with the test method set forth in this part or equivalent methods approved by the Administrator; or where the Administrator determines that emissions from the affected facility are not susceptible of being measured by such methods, the Administrator shall prescribe alternative test procedures for determining compliance with the requirements of this part.

(c) The owner or operator shall permit the Administrator to conduct performance tests at any reasonable time, shall cause the affected facility to be operated for purposes of such tests under such conditions as the Administrator shall specify based on representative performance of the affected facility, and shall make available to the Administrator such records as may be necessary to determine such performance.

(d) The owner or operator of an affected facility shall provide the Administrator 10 days prior notice of the performance test to afford the Administrator the opportunity to have an observer present.

(e) The owner or operator of an affected facility shall provide, or cause to be provided, performance testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to such facility.

(2) Safe sampling platform(s).

(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(f) Each performance test shall consist of three repetitions of the applicable test method. For the purpose of determining compliance with an applicable standard of performance, the average of results of all repetitions shall apply.

§ 60.9 Availability of information.

(a) Emission data provided to, or otherwise obtained by, the Administrator in accordance with the provisions of this part shall be available to the public.

(b) Except as provided in paragraph (a) of this section, any records, reports, or information provided to, or otherwise obtained by, the Administrator in accordance with the provisions of this part shall be available to the public, except that (1) upon a showing satisfactory to the Administrator by any person that such records, reports, or information, or particular part thereof (other than emission data), if made public, would divulge methods or processes entitled to protection as trade secrets of such person, the Administrator shall consider such records, reports, or information, or particular part thereof, confidential in accordance with the purposes of section 1905 of title 18 of the United States Code, except that such records, reports, or information, or particular part thereof, may be disclosed to other officers, employees, or authorized representatives of the United States concerned with carrying out the provisions of the Act or when relevant in any proceeding under the Act; and (2) information received by the Administrator solely for the purposes of §§ 60.5 and 60.6 shall not be disclosed if it is identified by the owner or operator as being a trade secret or commercial or financial information which such owner or operator considers confidential.

§ 60.10 State authority.

The provisions of this part shall not be construed in any manner to preclude any State or political subdivision thereof from:

(a) Adopting and enforcing any emission standard or limitation applicable to an affected facility, provided that such emission standard or limitation is not less stringent than the standard applicable to such facility.

(b) Requiring the owner or operator of an affected facility to obtain permits, licenses, or approvals prior to initiating construction, modification, or operation of such facility.

Subpart D—Standards of Performance for Fossil-Fuel Fired Steam Generators

§ 60.40 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each fossil fuel-fired steam generating unit of more than 250 million B.t.u. per hour heat input, which is the affected facility.

§ 60.41 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, and in Subpart A of this part.

(a) "Fossil fuel-fired steam generating unit" means a furnace or boiler used in the process of burning fossil fuel for the primary purpose of producing steam by heat transfer.

(b) "Fossil fuel" means natural gas, petroleum, coal and any form of solid, liquid, or gaseous fuel derived from such materials.

(c) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by Method 5.

§ 60.42 Standard for particulate matter.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter which is:

(a) In excess of 0.10 lb. per million B.t.u. heat input (0.18 g. per million cal.) maximum 2-hour average.

(b) Greater than 20 percent opacity, except that 40 percent opacity shall be permissible for not more than 2 minutes in any hour.

(c) Where the presence of uncombined water is the only reason for failure to meet the requirements of paragraph (b) of this section such failure shall not be a violation of this section.

§ 60.43 Standard for sulfur dioxide.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of sulfur dioxide in excess of:

(a) 0.80 lb. per million B.t.u. heat input (1.4 g. per million cal.), maximum 2-hour average, when liquid fossil fuel is burned.

(b) 1.2 lbs. per million B.t.u. heat input (2.2 g. per million cal.), maximum 2-hour average, when solid fossil fuel is burned.

(c) Where different fossil fuels are burned simultaneously in any combination, the applicable standard shall be determined by proration. Compliance shall be determined using the following formula:

$$\frac{y(0.80)+z(1.2)}{x+y+z}$$

where:

x is the percent of total heat input derived from gaseous fossil fuel and,
y is the percent of total heat input derived from liquid fossil fuel and,
z is the percent of total heat input derived from solid fossil fuel.

§ 60.44 Standard for nitrogen oxides.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of nitrogen oxides in excess of:

(a) 0.20 lb. per million B.t.u. heat input (0.36 g. per million cal.), maximum 2-hour average, expressed as NO_2, when gaseous fossil fuel is burned.

(b) 0.30 lb. per million B.t.u. heat input (0.54 g. per million cal.), maximum 2-hour average, expressed as NO_2, when liquid fossil fuel is burned.

(c) 0.70 lb. per million B.t.u. heat input (1.26 g. per million cal.), maximum 2-hour average, expressed as NO_2 when solid fossil fuel (except lignite) is burned.

(d) When different fossil fuels are burned simultaneously in any combination the applicable standard shall be determined by proration. Compliance shall be determined by using the following formula:

$$\frac{x(0.20)+y(0.30)+z(0.70)}{x+y+z}$$

where:

x is the percent of total heat input derived from gaseous fossil fuel and,
y is the percent of total heat input derived from liquid fossil fuel and,
z is the percent of total heat input derived from solid fossil fuel.

§ 60.45 Emission and fuel monitoring.

(a) There shall be installed, calibrated, maintained, and operated, in any fossil fuel-fired steam generating unit subject to the provisions of this part, emission monitoring instruments as follows:

(1) A photoelectric or other type smoke detector and recorder, except where gaseous fuel is the only fuel burned.

(2) An instrument for continuously monitoring and recording sulfur dioxide emissions, except where gaseous fuel is the only fuel burned, or where compliance is achieved through low sulfur fuels and representative sulfur analysis of fuels are conducted daily in accordance with paragraph (c) or (d) of this section.

(3) An instrument for continuously monitoring and recording emissions of nitrogen oxides.

(b) Instruments and sampling systems installed and used pursuant to this section shall be capable of monitoring emission levels within ±20 percent with a confidence level of 95 percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instruments; instruments shall be subjected to manufacturers recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method.

(c) The sulfur content of solid fuels, as burned, shall be determined in accordance with the following methods of the American Society for Testing and Materials.

(1) Mechanical sampling by Method D 2234065.

(2) Sample preparation by Method D 2013–65.

(3) Sample analysis by Method D 271–68.

(d) The sulfur content of liquid fuels, as burned, shall be determined in accordance with the American Society for Testing and Materials Methods D 1551–68, or D 129–64, or D 1552–64.

(e) The rate of fuel burned for each fuel shall be measured daily or at shorter intervals and recorded. The heating value and ash content of fuels shall be ascertained at least once per week and recorded. Where the steam generating unit is used to generate electricity, the average electrical output and the minimum and maximum hourly generation rate shall be measured and recorded daily.

(f) The owner or operator of any fossil fuel-fired steam generating unit subject to the provisions of this part shall maintain a file of all measurements required by this part. Appropriate measurements shall be reduced to the units of the applicable standard daily, and summarized monthly. The record of any such measurement(s) and summary shall be retained for at least 2 years following the date of such measurements and summaries.

§ 60.46 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of particulate matter, sulfur dioxide, and nitrogen oxides from fossil fuel-fired steam generating units.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum steam production rate at which such facility will be operated and while fuels or combinations of fuels representative of normal operation are being burned and under such other relevant conditions as the Administrator shall specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods approved by the Administrator shall be used as follows:

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5. Traversing during sampling by Method 5 shall be according to Method 1. The minimum sampling time shall be 2 hours, and minimum sampling volume shall be 60 ft.³ corrected to standard conditions on a dry basis.

(2) For each repetition, the SO_2 concentration shall be determined by using Method 6. The sampling site shall be the same as for determining volumetric flow rate. The sampling point in the duct shall be at the centroid of the cross section if the cross sectional area is less than 50 ft.² or at a point no closer to the walls than 3 feet if the cross sectional area is 50 ft.² or more. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 20 min. and minimum sampling volume shall be 0.75 ft.³ corrected to standard conditions. Two samples shall constitute one repetition and shall be taken at 1-hour intervals.

(3) For each repetition the NO_x concentration shall be determined by using Method 7. The sampling site and point shall be the same as for SO_2. The sampling time shall be 2 hours, and four samples shall be taken at 30-minute intervals.

(4) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to Method 1. Gas analysis shall be performed by Method 3, and moisture content shall be determined by the condenser technique of Method 5.

(d) Heat input, expressed in B.t.u. per hour, shall be determined during each 2-hour testing period by suitable fuel flow meters and shall be confirmed by a material balance over the steam generation system.

(e) For each repetition, emissions, expressed in lb./10³ B.t.u. shall be determined by dividing the emission rate in lb./hr. by the heat input. The emission rate shall be determined by the equation, $lb./hr. = Q_s \times c$ where, Q_s = volumetric flow rate of the total effluent in ft.³/hr. at standard conditions, dry basis, as determined in accordance with paragraph (c) (4) of this section.

(1) For particulate matter, c = particulate concentration in lb./ft.³, at determined in accordance with paragraph (c) (1) of this section, corrected to standard conditions, dry basis.

(2) For SO₂, c=SO_2 concentration in lb./ft.³, as determined in accordance with paragraph (c)(2) of this section, corrected to standard conditions, dry basis.

(3) For NOₓ, c=NO_x concentration in lb./ft.³, as determined in accordance with paragraph (c)(3) of this section, corrected to standard conditions, dry basis.

Subpart E—Standards of Performance for Incinerators

§ 60.50 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each incinerator of more than 50 tons per day charging rate, which is the affected facility.

§ 60.51 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Incinerator" means any furnace used in the process of burning solid waste for the primary purpose of reducing the volume of the waste by removing combustible matter.

(b) "Solid waste" means refuse, more than 50 percent of which is municipal type waste consisting of a mixture of paper, wood, yard wastes, food wastes, plastics, leather, rubber, and other combustibles, and noncombustible materials such as glass and rock.

(c) "Day" means 24 hours.

(d) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by Method 5. .

§ 60.52 Standard for particulate matter.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter which is in excess of 0.08 gr./s.c.f. (0.18 g./NM³) corrected to 12 percent CO_2, maximum 2-hour average.

§ 60.53 Monitoring of operations.

The owner or operator of any incinerator subject to the provisions of this part shall maintain a file of daily burning rates and hours of operation and any particulate emission measurements. The burning rates and hours of operation shall be summarized monthly. The record(s) and summary shall be retained for at least 2 years following the date of such records and summaries.

§ 60.54 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of particulate matter from incinerators.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum refuse charging rate at which such facility will be operated and the solid waste burned shall be representative of normal operation and under such other relevant conditions as the Administrator shall

specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods approved by the Administrator shall be used as follows:'

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5. Traversing during sampling by Method 5 shall be according to Method 1. The minimum sampling time shall be 2 hours and the minimum sampling volume shall be 60 ft.³ corrected to standard conditions on a dry basis.

(2) Gas analysis shall be performed using the integrated sample technique of Method 3, and moisture content shall be determined by the condenser technique of Method 5. If a wet scrubber is used, the gas analysis sample shall reflect flue gas conditions after the scrubber, allowing for the effect of carbon dioxide absorption.

(d) For each repetition particulate matter emissions, expressed in gr./s.c.f., shall be determined in accordance with paragraph (c)(1) of this section corrected to 12 percent CO_2, dry basis.

Subpart F—Standards of Performance for Portland Cement Plants

§ 60.60 Applicability and designation of affected facility.

The provisions of the subpart are applicable to the following affected facilities in portland cement plants: kiln, clinker cooler, raw mill system, finish mill system, raw mill dryer, raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging and bulk loading and unloading systems.

§ 60.61 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Portland cement plant" means any facility manufacturing portland cement by either the wet or dry process.

(b) "Particulate matter" means any finely divided liquid or solid material, other than uncombined water, as measured by Method 5.

§ 60.62 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter from the kiln which is:

(1) In excess of 0.30 lb. per ton of feed to the kiln (0.15 Kg. per metric ton), maximum 2-hour average.

(2) Greater than 10 percent opacity, except that where the presence of uncombined water is the only reason for failure to meet the requirements for this subparagraph, such failure shall not be a violation of this section.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner

or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter from the clinker cooler which is:

(1) In excess of 0.10 lb. per ton of feed to the kiln (0.050 Kg. per metric ton) maximum 2-hour average.

(2) 10 percent opacity or greater.

(c) On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter from any affected facility other than the kiln and clinker cooler which is 10 percent opacity or greater.

§ 60.63 Monitoring of operations.

The owner or operator of any portland cement plant subject to the provisions of this part shall maintain a file of daily production rates and kiln feed rates and any particulate emission measurements. The production and feed rates shall be summarized monthly. The record(s) and summary shall be retained for at least 2 years following the date of such records and summaries.

§ 60.64 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of particulate matter from portland cement plant kilns and clinker coolers.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum production rate at which such facility will be operated and under such other relevant conditions as the Administrator shall specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods approved by the Administrator shall be used as follows:

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5. Traversing during sampling by Method 5 shall be according to Method 1. The minimum sampling time shall be 2 hours and the minimum sampling volume shall be 60 ft.³ corrected to standard conditions on a dry basis.

(2) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to Method 1. Gas analysis shall be performed using the integrated sample technique of Method 3, and moisture content shall be determined by the condenser technique of Method 5.

(d) Total kiln feed (except fuels), expressed in tons per hour on a dry basis, shall be determined during each 2-hour testing period by suitable flow meters and shall be confirmed by a' material balance over the production system.

(e) For each repetition, particulate matter emissions, expressed in lb./ton of kiln feed shall be determined by dividing the emission rate in lb./hr. by the kiln feed. The emission rate shall be determined by the equation, lb./hr.=$Q_s \times c$,

where Q_s=volumetric flow rate of the total effluent in ft.³/hr. at standard conditions, dry basis, as determined in accordance with paragraph (c)(2) of this section, and, c=particulate concentration in lb./ft.³, as determined in accordance with paragraph (c)(1) of this section, corrected to standard conditions, dry basis.

Subpart G—Standards of Performance for Nitric Acid Plants

§ 60.70 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each nitric acid production unit, which is the affected facility.

§ 60.71 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Nitric acid production unit" means any facility producing weak nitric acid by either the pressure or atmospheric pressure process.

(b) "Weak nitric acid" means acid which is 30 to 70 percent in strength.

§ 60.72 Standard for nitrogen oxides.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of nitrogen oxides which are:

(a) In excess of 3 lbs. per ton of acid produced (1.5 kg. per metric ton), maximum 2-hour average, expressed as NO_2.

(b) 10 percent opacity or greater.

§ 60.73 Emission monitoring.

(a) There shall be installed, calibrated, maintained, and operated, in any nitric acid production unit subject to the provisions of this subpart, an instrument for continuously monitoring and recording emissions of nitrogen oxides.

(b) The instrument and sampling system installed and used pursuant to this section shall be capable of monitoring emission levels within ±20 percent with a confidence level of 95 percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subjected to manufacturers recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method.

(c) Production rate and hours of operation shall be recorded daily.

(d) The owner or operator of any nitric acid production unit subject to the provisions of this part shall maintain a file of all measurements required by this subpart. Appropriate measurements shall be reduced to the units of the standard daily and summarized monthly. The record of any such measurement and summary shall be retained for at least 2 years following the date of such measurements and summaries.

§ 60.74 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of nitrogen oxides from nitric acid production units.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum acid production rate at which such facility will be operated and under such other relevant conditions as the Administrator shall specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods as approved by the Administrator shall be used as follows:

(1) For each repetition the NO_x concentration shall be determined by using Method 7. The sampling site shall be selected according to Method 1 and the sampling point shall be the centroid of the stack or duct. The sampling time shall be 2 hours and four samples shall be taken at 30-minute intervals.

(2) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to Method 1. Gas analysis shall be performed by using the integrated sample technique of Method 3, and moisture content shall be determined by Method 4.

(d) Acid produced, expressed in tons per hour of 100 percent nitric acid, shall be determined during each 2-hour testing period by suitable flow meters and shall be confirmed by a material balance over the production system.

(e) For each repetition, nitrogen oxides emissions, expressed in lb./ton of 100 percent nitric acid, shall be determined by dividing the emission rate in lb./hr. by the acid produced. The emission rate shall be determined by the equation, lb./hr.=$Q_s \times c$, where Q_s=volumetric flow rate of the effluent in ft.³/hr. at standard conditions, dry basis, as determined in accordance with paragraph (c)(2) of this section, and c=NO_x concentration in lb./ft.³, as determined in accordance with paragraph (c)(1) of this section, corrected to standard conditions, dry basis.

Subpart H—Standards of Performance for Sulfuric Acid Plants

§ 60.80 Applicability and designation of affected facility.

The provisions of this subpart are applicable to each sulfuric acid production unit, which is the affected facility.

§ 60.81 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Sulfuric acid production unit" means any facility producing sulfuric acid by the contact process by burning elemental sulfur, alkylation acid, hydrogen sulfide, organic sulfides and mercaptans, or acid sludge, but does not include facilities where conversion to sulfuric acid is utilized primarily as a means of preventing emissions to the atmosphere of sulfur dioxide or other sulfur compounds.

(b) "Acid mist" means sulfuric acid mist, as measured by test methods set forth in this part.

§ 60.82 Standard for sulfur dioxide.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of sulfur dioxide in excess of 4 lbs. per ton of acid produced (2 kg. per metric ton), maximum 2-hour average.

§ 60.83 Standard for acid mist.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of acid mist which is:

(a) In excess of 0.15 lb. per ton of acid produced (0.075 kg. per metric ton), maximum 2-hour average, expressed as H_2SO_4.

(b) 10 percent opacity or greater.

§ 60.84 Emission monitoring.

(a) There shall be installed, calibrated, maintained, and operated, in any sulfuric acid production unit subject to the provisions of this subpart, an instrument for continuously monitoring and recording emissions of sulfur dioxide.

(b) The instrument and sampling system installed and used pursuant to this section shall be capable of monitoring emission levels within ±20 percent with a confidence level of 95 percent and shall be calibrated in accordance with the

method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subject to manufacturers recommended zero adjustment calibration procedures at least once per 24-hour operating period unless the manufacturer(s) specified or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method..

(c) Production rate and hours of operation shall be recorded daily.

(d) The owner or operator of any sulfuric acid production unit subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart. Appropriate measurements shall be reduced to the units of the applicable standard daily and summarized monthly. The record of any such measurement and summary shall be retained for at least 2 years following the date of such measurements and summaries.

§ 60.85 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determining emissions of acid mist and sulfur dioxide from sulfuric acid production units.

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum acid production rate at which such facility will be operated and under such other relevant conditions as the Administrator shall specify based on representative performance of the affected facility.

(c) Test methods set forth in the appendix to this part or equivalent methods as approved by the Administrator shall be used as follows:

(1) For each repetition the acid mist and SO_2 concentrations shall be determined by using Method 3 and traversing according to Method 1. The minimum sampling time shall be 2 hours, and minimum sampling volume shall be 40 ft.³ corrected to standard conditions.

(2) The volumetric flow rate of the total effluent shall be determined by using Method 2 and traversing according to Method 1. Gas analysis shall be performed by using the integrated sample technique of Method 3. Moisture content can be considered to be zero.

(d) Acid produced, expressed in tons per hour of 100 percent sulfuric acid shall be determined during each 2-hour testing period by suitable flow meters and shall be confirmed by a material balance over the production system.

(e) For each repetition acid mist and sulfur dioxide emissions, expressed in lb./ton of 100 percent sulfuric acid shall be determined by dividing the emission rate in lb./hr. by the acid produced. The emission rate, lb./hr.$=Q_s \times c$, where Q_s=volumetric flow rate of the effluent in ft.³/hr. at standard conditions, dry basis as determined in accordance with paragraph (c)(2) of this section, and c=acid mist and SO_2 concentrations in lb./ft.³ as determined in accordance with paragraph (c)(1) of this section, corrected to standard conditions, dry basis.

Appendix—Test Methods

Method 1—Sample and velocity traverses for stationary sources

1. Principle and Applicability.

1.1 Principle. A sampling site and the number of traverse points are selected to aid in the extraction of a representative sample.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

2. Procedure.

2.1 Selection of a sampling site and minimum number of traverse points.

2.1.1 Select a sampling site that is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. For rectangular cross section, determine an equivalent diameter from the following equation:

$$\text{equivalent diameter}=2\left(\frac{(\text{length})(\text{width})}{\text{length}+\text{width}}\right)$$

equation 1-1

2.1.2 When the above sampling site criteria can be met, the minimum number of traverse points is twelve (12).

2.1.3 Some sampling situations render the above sampling site criteria impractical. When this is the case, choose a convenient sampling location and use Figure 1-1 to determine the minimum number of traverse points. Under no conditions should a sampling point be selected within 1 inch of the stack wall. To obtain the number of traverse points for stacks or ducts with a diameter less than 2 feet, multiply the number of points obtained from Figure 1-1 by 0.67.

2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location to the nearest upstream and downstream disturbances. Determine the corresponding number of traverse points for each distance from Figure 1-1. Select the higher of the two numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of 4, and for rectangular stacks the number follows the criteria of section 2.2.2.

2.2 Cross-sectional layout and location of traverse points.

2.2.1 For circular stacks locate the traverse points on at least two diameters according to Figure 1-2 and Table 1-1. The traverse axes shall divide the stack cross section into equal parts.
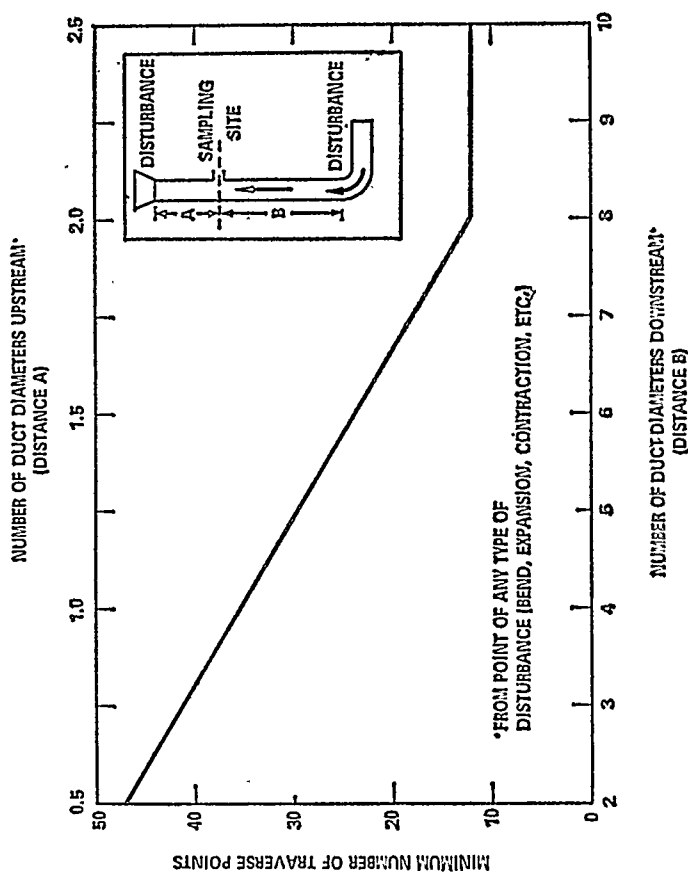

Figure 1-1. Minimum number of traverse points.

Table 1-1. Location of traverse points in circular stacks
(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.3	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	35.5	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.5	36.6	28.3	23.6	20.4	18.0	16.1
8				96.7	85.4	65.0	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	26.1	23.0
10					97.5	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	73.9	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											93.9	94.5
23												96.8
24												93.9

Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.
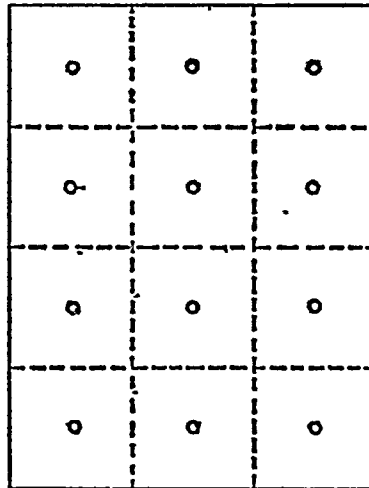

Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

2.2.2 For rectangular stacks divide the cross section into as many equal rectangular areas as traverse points, such that the ratio of the length to the width of the elemental areas is between one and two. Locate the traverse points at the centroid of each equal area according to Figure 1-3.

3. References.

Determining Dust Concentration in a Gas Stream, ASME Performance Test Code #27, New York, N.Y., 1957.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif. November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif. Bulletin WP-50, 1968.

Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1971, ASTM Designation D-2928-71.

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

1. Principle and applicability.

1.1 Principle. Stack gas velocity is determined from the gas density and from measurement of the velocity head using a Type S (Stauscheibe or reverse type) pitot tube.

1.2 Applicability. This method should be applied only when specified by the test procedure.

cedures for determining compliance with the New Source Performance Standards.

2. Apparatus.

2.1 Pitot tube—Type S (Figure 2-1), or equivalent, with a coefficient within ±5% over the working range.

2.2 Differential pressure gauge—Inclined manometer, or equivalent, to measure velocity head to within 10% of the minimum value.

2.3 Temperature gauge—Thermocouple or equivalent attached to the pitot tube to measure stack temperature to within 1.5% of the minimum absolute stack temperature.

2.4 Pressure gauge—Mercury-filled U-tube manometer, or equivalent, to measure stack pressure to within 0.1 in. Hg.

2.5 Barometer—To measure atmospheric pressure to within 0.1 in. Hg.

2.6 Gas analyzer—To analyze gas composition for determining molecular weight.

2.7 Pitot tube—Standard type, to calibrate Type S pitot tube.

3. Procedure.

3.1 Set up the apparatus as shown in Figure 2-1. Make sure all connections are tight and leak free. Measure the velocity head and temperature at the traverse points specified by Method 1.

3.2 Measure the static pressure in the stack.

3.3 Determine the stack gas molecular weight by gas analysis and appropriate calculations as indicated in Method 3.

4. Calibration.

4.1 To calibrate the pitot tube, measure the velocity head at some point in a flowing gas stream with both a Type S pitot tube and a standard type pitot tube with known coefficient. Calibration should be done in the laboratory and the velocity of the flowing gas stream should be varied over the normal working range. It is recommended that the calibration be repeated after use at each field site.

4.2 Calculate the pitot tube coefficient using equation 2-1.

$$C_{p_{test}} = C_{p_{std}} \sqrt{\frac{\Delta p_{std}}{\Delta p_{test}}} \quad \text{equation 2-1}$$

where:

$C_{p_{test}}$ = Pitot tube coefficient of Type S pitot tube.

$C_{p_{std}}$ = Pitot tube coefficient of standard type pitot tube (if unknown, use 0.99).

Δp_{std} = Velocity head measured by standard type pitot tube.

Δp_{test} = Velocity head measured by Type S pitot tube.

4.3 Compare the coefficients of the Type S pitot tube determined first with one leg and then the other pointed downstream. Use the pitot tube only if the two coefficients differ by no more than 0.01.

5. Calculations.

Use equation 2-2 to calculate the stack gas velocity.

$$(V_s)_{avg.} = K_p C_p (\sqrt{\Delta p})_{avg.} \sqrt{\frac{(T_s)_{avg.}}{P_s M_s}}$$

Equation 2-2

where:

$(V_s)_{avg.}$ = Stack gas velocity, feet per second (f.p.s.).

$K_p = 85.48 \frac{ft.}{sec.} \left(\frac{lb.}{lb. mole-°R}\right)^{1/2}$ when these units are used.

C_p = Pitot tube coefficient, dimensionless.

$(T_s)_{avg.}$ = Average absolute stack gas temperature, °R.

$(\sqrt{\Delta p})_{avg.}$ = Average velocity head of stack gas, inches H_2O (see Fig. 2-2).

P_s = Absolute stack gas pressure, inches Hg.

M_s = Molecular weight of stack gas (wet basis), lb./lb.-mole.

$$M_d(1-B_{wo})+18B_{wo}$$

M_d = Dry molecular weight of stack gas (from Method 3).

B_{wo} = Proportion by volume of water vapor in the gas stream (from Method 4).

Figure 2-2 shows a sample recording sheet for velocity traverse data. Use the averages in the last two columns of Figure 2-2 to determine the average stack gas velocity from Equation 2-2.

Use Equation 2-3 to calculate the stack gas volumetric flow rate.

$$Q_s = 3600 (1-B_{wo}) V_s A \left(\frac{T_{std}}{(T_s)_{avg.}}\right) \left(\frac{P_s}{P_{std}}\right)$$

Equation 2-3

where:

Q_s = Volumetric flow rate, dry basis, standard conditions, ft.³/hr.

A = Cross-sectional area of stack, ft.²

T_{std} = Absolute temperature at standard conditions, 530° R.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

PIPE COUPLING   TUBING ADAPTER
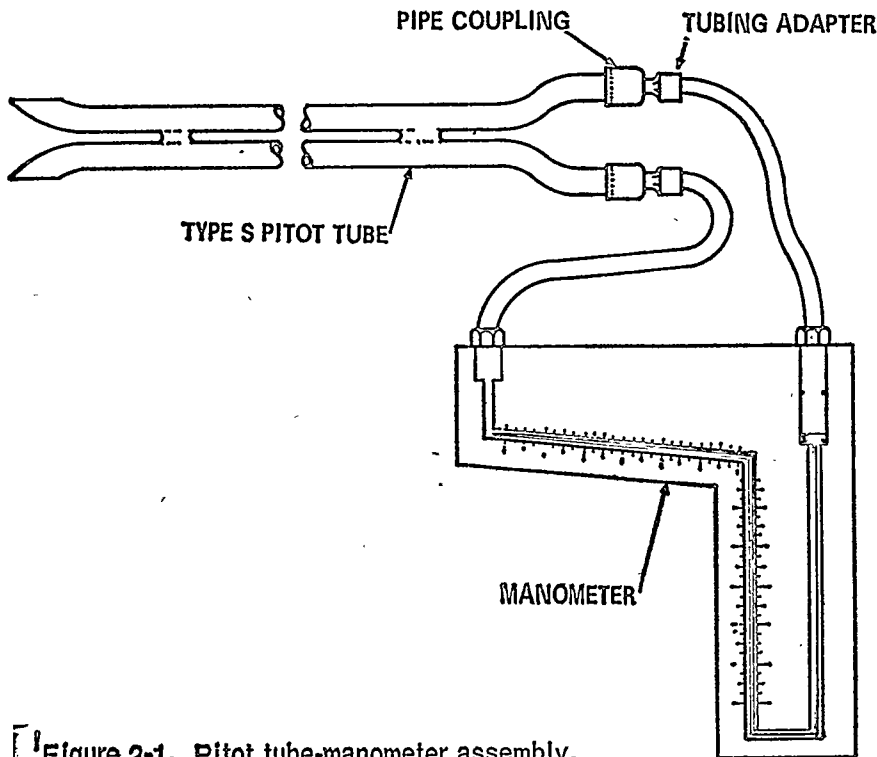
TYPE S PITOT TUBE

MANOMETER

Figure 2-1. Pitot tube-manometer assembly.

6. References.

Mark, L. S., Mechanical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1951.

Perry, J. H., Chemical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1960.

Shigehara, R. T., W. F. Todd, and W. S. Smith, Significance of Errors in Stack Sampling Measurements. Paper presented at the Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14–19, 1970.

Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa., 1971, ASTM Designation D-2928-71.

Vennard, J. K., Elementary Fluid Mechanics, John Wiley & Sons, Inc., New York, N.Y., 1947.

PLANT_____

DATE_____

RUN NO._____

STACK DIAMETER, in._____

BAROMETRIC. PRESSURE, in. Hg._____

STATIC PRESSURE IN STACK (P$_g$), in. Hg._____

OPERATORS_____
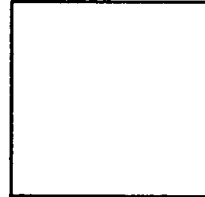
SCHEMATIC OF STACK
CROSS SECTION

Traverse point number	Velocity head, in. H_2O	$\sqrt{\Delta_P}$	Stack Temperature (T_S), °F
	AVERAGE:		

Figure 2-2. Velocity traverse data.

METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. *Principle and applicability.*

1.1 Principle. An integrated or grab gas sample is extracted from a sampling point and analyzed for its components using an Orsat analyzer.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. The test procedure will indicate whether a grab sample or an integrated sample is to be used.

2. *Apparatus.*

2.1 Grab sample (Figure 3-1).

2.1.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.1.2 Pump—One-way squeeze bulb, or equivalent, to transport gas sample to analyzer.

[1] Trade name.

2.2 *Integrated sample (Figure 3-2).*

2.2.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.2.2 Air-cooled condenser or equivalent—To remove any excess moisture.

2.2.3 Needle valve—To adjust flow rate.

2.2.4 Pump—Leak-free, diaphragm type, or equivalent, to pull gas.

2.2.5 Rate meter—To measure a flow range from 0 to 0.035 cfm.

2.2.6 Flexible bag—Tedlar,[1] or equivalent, with a capacity of 2 to 3 cu. ft. Leak test the bag in the laboratory before using.

2.2.7 Pitot tube—Type S, or equivalent, attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

2.3 *Analysis.*

2.3.1 Orsat analyzer, or equivalent.

3. *Procedure.*

3.1 Grab sampling.

3.1.1 Set up the equipment as shown in Figure 3-1, making sure all connections are leak-free. Place the probe in the stack at a sampling point and purge the sampling line.

3.1.2 Draw sample into the analyzer.

3.2 Integrated sampling.

3.2.1 Evacuate the flexible bag. Set up the equipment as shown in Figure 3-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are tight and that there are no leaks.

3.2.2 Sample at a rate proportional to the stack velocity.

3.3 Analysis.

3.3.1 Determine the CO, O, and CO concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than ten passes are necessary, replace the absorbing solution.

3.3.2 For grab sampling, repeat the sampling and analysis until three consecutive samples vary no more than 0.5 percent by volume for each component being analyzed.

3.3.3 For integrated sampling, repeat the analysis of the sample until three consecutive analyses vary no more than 0.2 percent by volume for each component being analyzed.

4. *Calculations.*

4.1 Carbon dioxide. Average the three consecutive runs and report the result to the nearest 0.1% CO₂.

4.2 Excess air. Use Equation 3-1 to calculate excess air, and average the runs. Report the result to the nearest 0.1% excess air.

$$\% \ EA = \frac{(\% \ O_2) - 0.5(\% \ CO)}{0.264(\% \ N_2) - (\% \ O_2) + 0.5(\% \ CO)} \times 100$$

<div align="right">equation 3-1</div>

where:

%EA=Percent excess air.
%O₂=Percent oxygen by volume, dry basis.
%N₂=Percent nitrogen by volume, dry basis.
%CO=Percent carbon monoxide by volume, dry basis.
0.264=Ratio of oxygen to nitrogen in air by volume.

4.3 Dry molecular weight. Use Equation 3-2 to calculate dry molecular weight and average the runs. Report the result to the nearest tenth.

$$M_d = 0.44(\% CO_2) + 0.32(\% O_2) + 0.28(\% N_2 + \% CO)$$

<div align="right">equation 3-2</div>

where:

M_d=Dry molecular weight, lb./lb-mole.
%CO₂=Percent carbon dioxide by volume, dry basis.
%O₂=Percent oxygen by volume, dry basis.
%N₂=Percent nitrogen by volume, dry basis.
0.44=Molecular weight of carbon dioxide divided by 100.
0.32=Molecular weight of oxygen divided by 100.
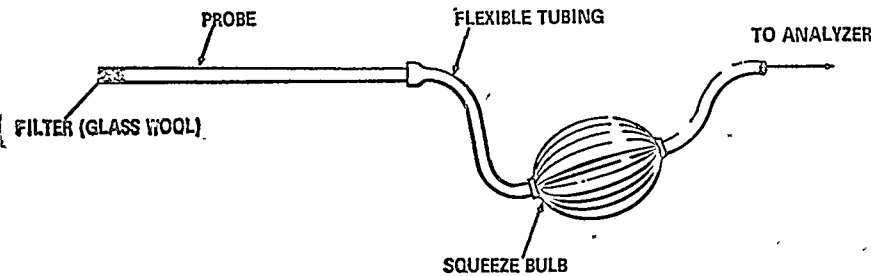0.28=Molecular weight of nitrogen and CO divided by 100.
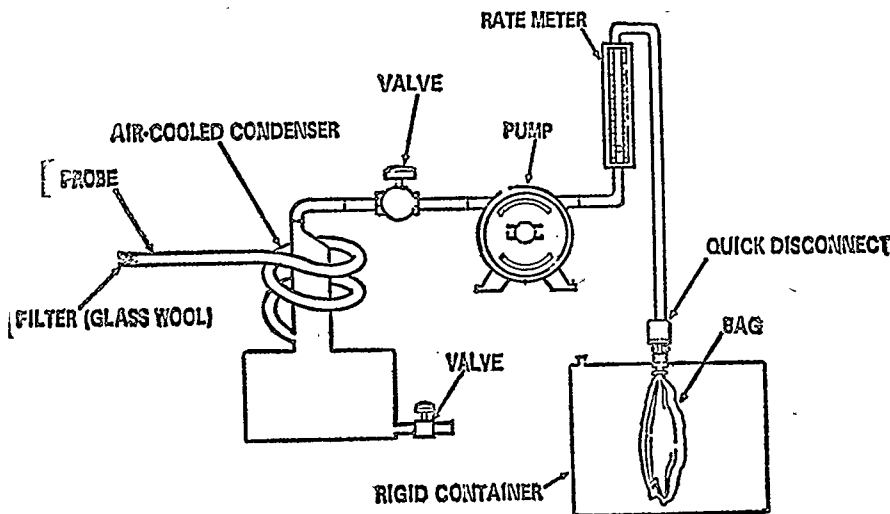

Figure 3-1. Grab-sampling train.

Figure 3-2. Integrated gas-sampling train.
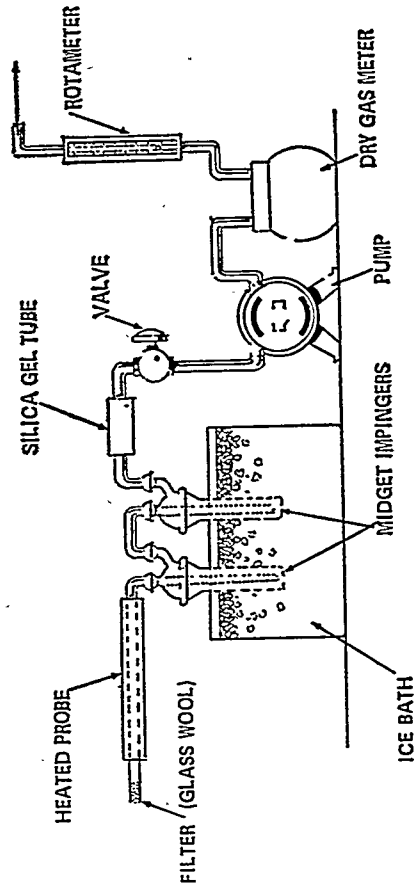
Figure 4-1. Moisture-sampling train.

LOCATION _____

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

COMMENTS _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), ft³	ROTAMETER SETTING ft³/min	METER TEMPERATURE, °F

Figure 4-2. Field moisture determination.

5. References.

Altshuller, A. P., et al., Storage of Gases and Vapors in Plastic Bags, Int. J. Air & Water Pollution, 6:75-81, 1963.

Conner, William D., and J. S. Nader, Air Sampling with Plastic Bags, Journal of the American Industrial Hygiene Association, 25:291-297, May-June 1964.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1968.

METHOD 4—DETERMINATION OF MOISTURE IN STACK GASES

1. Principle and applicability.

1.1 Principle. Moisture is removed from the gas stream, condensed, and determined volumetrically.

1.2 Applicability. This method is applicable for the determination of moisture in stack gas only when specified by test procedures for determining compliance with New Source Performance Standards. This method does not apply when liquid droplets are present in the gas stream[1] and the moisture is subsequently used in the determination of stack gas molecular weight.

Other methods such as drying tubes, wet bulb-dry bulb techniques, and volumetric condensation techniques may be used.

2. Apparatus.

2.1 Probe—Stainless steel or Pyrex[2] glass sufficiently heated to prevent condensation

and equipped with a filter to remove particulate matter.

2.2 Impingers—Two midget impingers, each with 30 ml. capacity, or equivalent.

2.3 Ice bath container—To condense moisture in impingers.

2.4 Silica gel tube (optional)—To protect pump and dry gas meter.

2.5 Needle valve—To regulate gas flow rate.

2.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through train.

2.7 Dry gas meter—To measure to within 1% of the total sample volume.

2.8 Rotameter—To measure a flow range from 0 to 0.1 c.f.m.

2.9 Graduated cylinder—25 ml.

2.10 Barometer—Sufficient to read to within 0.1 inch Hg.

2.11 Pitot tube—Type S, or equivalent, attached to probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

3. Procedure.

3.1 Place exactly 5 ml. distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-1. Leak check by plugging the inlet to the first impinger and drawing a vacuum. Insure that flow through the dry gas meter is less than 1% of the sampling rate.

3.2 Connect the probe and sample at a constant rate of 0.075 c.f.m. or at a rate proportional to the stack gas velocity. Continue sampling until the dry gas meter registers 1 cubic foot or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-2.

3.3 After collecting the sample, measure the volume increase to the nearest 0.5 ml.

4. Calculations.

4.1 Volume of water vapor collected.

$$V_{wc} = \frac{(V_f - V_i)\rho_{H_2O}RT_{std}}{P_{std}M_{H_2O}} = 0.0474\frac{ft^3}{ml.}(V_f - V_i)$$

equation 4-1

where:

V_{wc}=Volume of water vapor collected (standard conditions), cu. ft.

V_f=Final volume of impinger contents, ml.

V_i=Initial volume of impinger contents, ml.

R=Ideal gas constant, 21.83 inches

Hg—cu. ft./lb. mole-°R.

ρ_{H_2O}=Density of water, 1 g./ml.

T_{std}=Absolute temperature at standard conditions, 530° R.

P_{std}=Absolute pressure at standard conditions, 29.92 inches Hg.

M_{H_2O}=Molecular weight of water, 18 lb./lb.-mole.

[1] If liquid droplets are present in the gas stream, assume the stream to be saturated, determine the average stack gas temperature by traversing according to Method 1, and use a psychrometric chart to obtain an approximation of the moisture percentage.

[2] Trade name.

4.2 Gas volume.

$$V_{mo}=V_m\left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right)=$$

$$17.71\frac{°R}{in.\ Hg}\left(\frac{V_mP_m}{T_m}\right)\quad equation\ 4-2$$

where:

V_{mo} =Dry gas volume through meter at standard conditions, cu. ft.

V_m =Dry gas volume measured by meter, cu. ft.

P_m =Barometric pressure at the dry gas meter, inches Hg.

P_{std}=Pressure at standard conditions, 29.92 inches Hg.

T_{std}=Absolute temperature at standard conditions, 530° R.

T_m =Absolute temperature at meter (°F+ 460), °R.

4.3 Moisture content.

$$B_{wo}=\frac{V_{wo}}{V_{wo}+V_{mo}}+B_{wm}=\frac{V_{wo}}{V_{wo}+V_{mo}}+(0.025)$$

equation 4-3

where:

B_{wo}=Proportion by volume of water vapor in the gas stream, dimensionless.

V_{wo} =Volume of water vapor collected (standard conditions), cu. ft.

V_{mo} =Dry gas volume through meter (standard conditions), cu. ft.

B_{wm}=Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

5. *References.*

Air Pollution Engineering Manual, Danielson, J. A. (ed.), U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999-AP-40, 1967.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and its weight is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling train. The design specifications of the particulate sampling train used by EPA (Figure 5-1) are described in APTD-0581. Commercial models of this train are available.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 600° F., Incoloy 825[1], or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 600° F. must have been approved by the Administrator.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

[1] Trade name.

2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to ±0.1 mg.

2.3.4 Trip balance—300 g. capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6-16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.
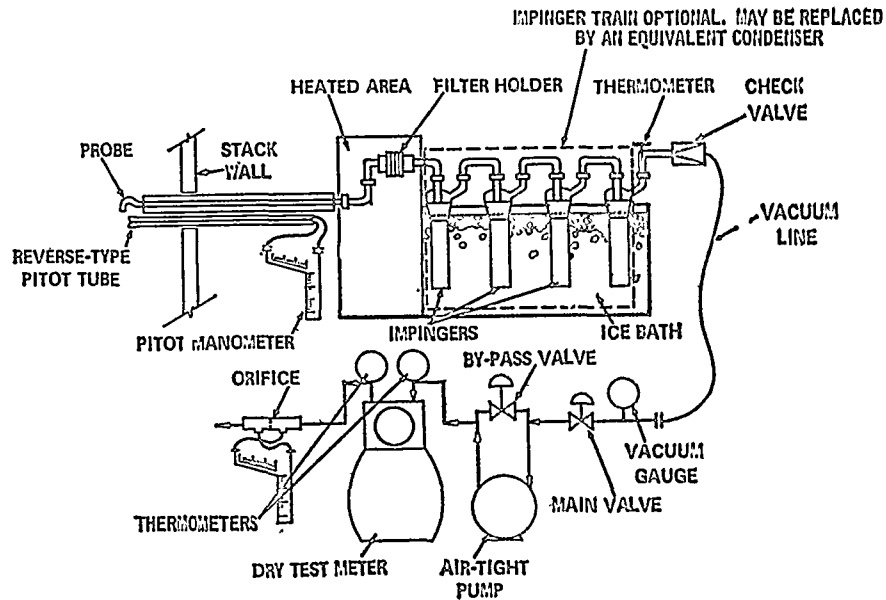
3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

IMPINGER TRAIN OPTIONAL. MAY BE REPLACED BY AN EQUIVALENT CONDENSER

Figure 5-1. Particulate-sampling train.

3.3.2 Desiccant—Drierite,[1] indicating.

4. *Procedure.*

4.1 Sampling

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Weigh to the nearest gram approximately 200 g. of silica gel. Label a filter of proper diameter, desiccate[2] for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add

[1] Trade name.
[2] Dry using Drierite[1] at 70° F.±10° F.

more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F., or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point, at least every 5 minutes, and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, 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. Sample for at least 5 minutes at each traverse point; sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APTD-0576 details the procedure for using these nomographs. Turn off the pump at the conclusion of each run and record the final readings. Remove the probe and nozzle from the stack and handle in accordance with the sample recovery process described in section 4.2.

PLANT_____
LOCATION_____
OPERATOR_____
DATE_____
RUN NO._____
SAMPLE BOX NO._____
METER BOX NO._____
METER ΔH_____
C FACTOR_____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE_____
BAROMETRIC PRESSURE_____
ASSUMED MOISTURE, %_____
HEATER BOX SETTING_____
PROBE LENGTH, m._____
NOZZLE DIAMETER, in._____
PROBE HEATER SETTING_____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (Ps), in. Hg	STACK TEMPERATURE (Ts), °F	VELOCITY HEAD (ΔPs),	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H2O	GAS SAMPLE VOLUME (Vm), ft³	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °F
							INLET (Tm in), °F	OUTLET (Tm out), °F		
TOTAL						Avg.	Avg.			
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

4.2 Sample recovery. Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in containers as follows:

Container No. 1. Remove the filter from its holder, place in this container, and seal.

Container No. 2. Place loose particulate matter and acetone washings from all sample-exposed surfaces prior to the filter in this container and seal. Use a razor blade, brush, or rubber policeman to lose adhering particles.

Container No. 3. Transfer the silica gel from the fourth impinger to the original container and seal. Use a rubber policeman as an aid in removing silica gel from the impinger.

4.3 Analysis. Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate, and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 2. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Weigh the spent silica gel and report to the nearest gram.

5. *Calibration.*

Use methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater. Recalibrate after each test series.

6. *Calculations.*

6.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.2 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 5-1.

$$V_{m\,std}=V_m\left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right)=$$

$$\left(17.71\ \frac{°R}{in.\ Hg}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

equation 5-1

where:

$V_{m\,std}$= Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m= Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std}= Absolute temperature at standard conditions, 530° R.

T_m= Average dry gas meter temperature, °R.

P_{bar}= Barometric pressure at the orifice meter, inches Hg.

ΔH= Average pressure drop across the orifice meter, inches H2O.

13.6= Specific gravity of mercury.

P_{std}= Absolute pressure at standard conditions, 29.92 inches Hg.

6.3 Volume of water vapor.

$$V_{w\,std}=V_{lc}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{RT_{std}}{P_{std}}\right)=\left(0.0474\ \frac{cu.\ ft.}{mL}\right)V_{lc}$$

equation 5-2

where:

$V_{w\,std}$= Volume of water vapor in the gas sample (standard conditions), cu. ft.

V_{lc}= Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

ρ_{H_2O}= Density of water, 1 g./mL.

M_{H_2O}= Molecular weight of water, 18 lb./lb.-mole.

R= Ideal gas constant, 21.83 inches Hg—cu. ft./lb.-mole-°R.

T_{std}= Absolute temperature at standard conditions, 530° R.

P_{std}= Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

$$B_{wo}=\frac{V_{w\,std}}{V_{m\,std}+V_{w\,std}}$$

equation 5-3

where:

B_{wo}= Proportion by volume of water vapor in the gas stream, dimensionless.

$V_{w\,std}$= Volume of water in the gas sample (standard conditions), cu. ft.

$V_{m\,std}$= Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 5-3).

6.6 Concentration.

6.6.1 Concentration in gr./s.c.f.

$$c'_s=\left(0.0154\frac{gr.}{mg.}\right)\left(\frac{M_n}{V_{m\,std}}\right)$$

equation 5-4

where:

c'_s= Concentration of particulate matter in stack gas, gr./s.c.f., dry basis.

M_n= Total amount of particulate matter collected, mg.

$V_{m\,std}$= Volume of gas sample through dry gas meter (standard conditions), cu. ft.

PLANT_____

DATE _____

RUN NO._____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			

	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):

$$\frac{INCREASE, g}{(1 \ g/ml)} = VOLUME \ WATER, ml$$

Figure 5-3. Analytical data.

6.6.2 Concentration in lb./cu. ft.

$$c_s = \frac{\left(\frac{1}{453,600} \frac{lb.}{mg.}\right) M_n}{V_{m_{std}}} = 2.205 \times 10^{-6} \frac{M_n}{V_{m_{std}}}$$

equation 5-5

where:

c_s = Concentration of particulate matter in stack gas, lb./s.c.f., dry basis. 453,600 = Mg/lb.

M_n = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.7 Isokinetic variation.

$$I = \frac{T_s\left[\frac{V_{l_o}(\rho_{H_2O})R}{M_{H_2O}} + \frac{V_m}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n} \times 100$$

$$= \frac{\left(1.667\frac{min.}{sec.}\right)\left[\left(0.00267\frac{in. \ Hg\text{-}cu. \ ft.}{ml.\text{-}°R}\right)V_{l_o} + \frac{V_m}{T_m}\cdot\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{\theta V_s P_s A_n}$$

Equation 5-6

where:
I = Percent of isokinetic sampling.
V_{l_o} = Total volume of liquid collected in impingers and silica gel (See Fig. 5-3), ml.
ρ_{H_2O} = Density of water, 1 g./ml.
R = Ideal gas constant, 21.83 inches Hg-cu. ft./lb. mole-°R.
M_{H_2O} = Molecular weight of water, 18 lb./lb.-mole.
V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.
T_m = Absolute average dry gas meter temperature (see Figure 5-2), °R.
P_{bar} = Barometric pressure at sampling site, inches Hg.
ΔH = Average pressure drop across the orifice (see Fig. 5-2), inches H2O.
T_s = Absolute average stack gas temperature (see Fig. 5-2), °R.
θ = Total sampling time, min.
V_s = Stack gas velocity calculated by Method 2, Equation 2-2, ft./sec.
P_s = Absolute stack gas pressure, inches Hg.
A_n = Cross-sectional area of nozzle, sq. ft.

6.8 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:

If $90\% \leq I \leq 110\%$, the results are acceptable, otherwise, reject the results and repeat the test.

7. Reference.

Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, Dec. 6, 1967.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0581.

Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-0576.

Smith, W. S., R. T. Shigehara, and W. F. Todd, A Method of Interpreting Stack Sampling Data, Paper presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.

Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment, APCA paper No. 67-119, 1967.

Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, 1967.

METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and applicability.

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The acid mist, including sulfur trioxide, is separated from the sulfur dioxide. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. Apparatus.

2.1 Sampling. See Figure 6-1.

2.1.1 Probe—Pyrex[1] glass, approximately 5 to 6 mm. ID, with a heating system to prevent condensation and a filtering medium to remove particulate matter including sulfuric acid mist.

2.1.2 Midget bubbler—One, with glass wool packed in top to prevent sulfuric acid mist carryover.

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

2.1.5 Drying tube—Packed with 6 to 16 mesh indicating-type silica gel, or equivalent, to dry the sample.

2.1.6 Valve—Needle valve, or equivalent, to adjust flow rate.

2.1.7 Pump—Leak-free, vacuum type.

2.1.8 Rate meter—Rotameter or equivalent, to measure a 0-10 s.c.f.h. flow range.

2.1.9 Dry gas meter—Sufficiently accurate to measure the sample volume within 1%.

2.1.10 Pitot tube—Type S, or equivalent,

[1] Trade names.

necessary only if a sample traverse is required, or if stack gas velocity varies with time.

2.2 Sample recovery.

2.2.1 Glass wash bottles—Two, 500 ml.
2.2.2 Polyethylene storage bottles—To store impinger samples.

2.3 Analysis.

3. Reagents.
3.1 Sampling.
3.1.1 Water—Deionized, distilled.
3.1.2 Isopropanol, 80%—Mix 80 ml. of isopropanol with 20 ml. of distilled water.
3.1.3 Hydrogen peroxide, 3%—dilute 100 ml. of 30% hydrogen peroxide to 1 liter with distilled water. Prepare fresh daily.

3.2 Sample recovery.
3.2.1 Water—Deionized, distilled.
3.2.2 Isopropanol, 80%.

3.3 Analysis.
3.3.1 Water—Deionized, distilled.
3.3.2 Isopropanol.
3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.
3.3.4 Barium perchlorate (0.01 N)—Dissolve 1.95 g. of barium perchlorate [Ba(ClO₄)₂·3H₂O] in 200 ml. distilled water

2.3.1
Pipettes—Transfer type, 5 ml. and 10 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. division).
2.3.2 Volumetric flasks—50 ml., 100 ml., and 1,000 ml.
2.3.3 Burettes—5 ml. and 50 ml.
2.3.4 Erlenmeyer flask—125 ml.

and dilute to 1 liter with isopropanol. Standardize with sulfuric acid. Barium chloride may be used.
3.3.5 Sulfuric acid standard (0.01 N)—Purchase or standardize to ±0.0002 N against 0.01N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

4. Procedure.
4.1 Sampling.
4.1.1 Preparation of collection train. Pour 15 ml. of 80% isopropanol into the midget bubbler and 15 ml. of 3% hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Leak check the sampling train at the sampling site by plugging the probe inlet and pulling a 10 inches Hg vacuum. A leakage rate not in excess of 1% of the sampling rate is acceptable. Carefully release the probe inlet plug and turn off the pump. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.
4.1.2 Sample collection. Adjust the sample flow rate proportional to the stack gas

Figure 6-1. SO₂ sampling train.

velocity. Take readings at least every five minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the tip of the probe at the first sampling point and start the pump. Sample proportionally throughout the run. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

4.2 Sample recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a polyethylene shipment bottle. Rinse the three midget impingers and the connecting tubes with distilled water and add these washings to the same storage container.

4.3 Sample analysis. Transfer the contents of the storage container to a 50 ml. volumetric flask. Dilute to the mark with deionized, distilled water. Pipette a 10 ml. aliquot of this solution into a 125 ml. Erlenmeyer flask. Add 40 ml. of isopropanol and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.01 N barium perchlorate. Run a blank with each series of samples.

5. Calibration.
5.1 Use standard methods and equipment which have been approved by the Administrator to calibrate the rotameter, pitot tube, dry gas meter, and probe heater.

5.2 Standardize the barium perchlorate against 25 ml. of standard sulfuric acid containing 100 ml. of isopropanol.

6. Calculations.
6.1 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F. and 29.92 inches Hg) by using equation 6-1.

$$V_{m\,std} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) =$$

$$17.71 \frac{°R}{\text{in. Hg}} \left(\frac{V_m P_{bar}}{T_m}\right) \quad \text{equation 6-1}$$

where:

$V_{m\,std}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfur dioxide concentration.

$$C_{SO_2} = \left(7.05 \times 10^{-5} \frac{lb.-l.}{g.-ml.}\right) \frac{(V_t - V_{tb})N\left(\frac{V_{soln}}{V_a}\right)}{V_{m\,std}} \quad \text{equation 6-2}$$

where:

C_{SO_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

7.05×10^{-5} = Conversion factor, including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

N = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfur dioxide, 50 ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m\,std}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 6-1.

7. References.

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 999-AP-13, Cincinnati, Ohio, 1965.

Corbett, P. F., The Determination of SO₂ and SO₃ in Flue Gases, Journal of the Institute of Fuel, 24:237-243, 1961.

Matty, R. L. and E. K. Diehl, Measuring Flue-Gas SO₂ and SO₃, Power 101:94-97, November, 1957.

Patton, W. F. and J. A. Brink, Jr., New Equipment and Techniques for Sampling Chemical Process Gases, J. Air Pollution Control Association, 13, 162 (1963).

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and applicability.
1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except

nitrous oxide, are measure colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable for the measurement of nitrogen oxides from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling. See Figure 7-1.

2.1.1 Probe—Pyrex[1] glass, heated, with filter to remove particulate matter. Heating is unnecessary if the probe remains dry during the purging period.

2.1.2 Collection flask—Two-liter, Pyrex,[1] round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

―――――――――
[1] Trade name.

2.1.3 Flask valve—T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature gauge—Dial-type thermometer, or equivalent, capable of measuring 2° F. intervals from 25° to 125° F.

2.1.5 Vacuum line—Tubing capable of withstanding a vacuum of 3 inches Hg absolute pressure, with "T" connection and T-bore stopcock, or equivalent.

2.1.6 Pressure gauge—U-tube manometer, 36 inches, with 0.1-inch divisions, or equivalent.

2.1.7 Pump—Capable of producing a vacuum of 3 inches Hg absolute pressure.

2.1.8 Squeeze bulb—One way.

2.2 Sample recovery.

2.2.1 Pipette or dropper.

2.2.2 Glass storage containers—Cushioned for shipping.
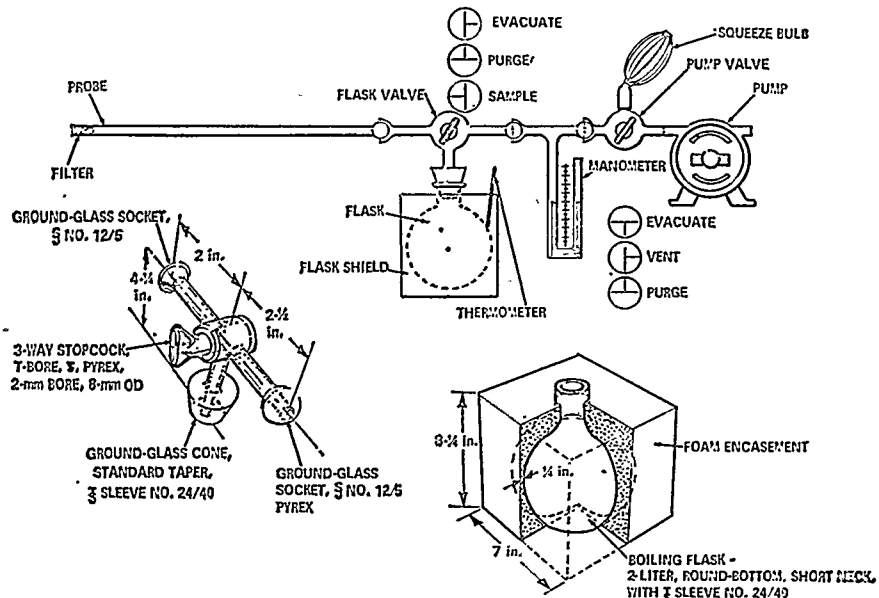
positions. Evacuate the flask to at least 3 inches Hg absolute pressure. Turn the pump valve to its "vent" position and turn off the pump. Check the manometer for any fluctuation in the mercury level. If there is a visible change over the span of one minute, check for leaks. Record the initial volume, temperature, and barometric pressure. Turn the flask valve to its "purge" position, and then do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and flask valve area, heat the probe and purge until the condensation disappears. Then turn the pump valve to its "vent" position. Turn the flask valve to its "sample" position and allow sample to enter the flask for about 15 seconds. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for 5 minutes.

4.2 Sample recovery.

4.2.1 Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer, open the valve from the flask to the manometer, and record the flask pressure and temperature along with the barometric pressure. Transfer the flask contents to a container for shipment or to a 250 ml. beaker for analysis. Rinse the flask with two portions of distilled water (approximately 10 ml.) and add rinse water to the sample. For a blank use 25 ml. of absorbing solution and the same volume of distilled water as used in rinsing the flask. Prior to shipping or analysis, add sodium hydroxide (1N) dropwise into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

4.3 Analysis.

4.3.1 If the sample has been shipped in a container, transfer the contents to a 250 ml. beaker using a small amount of distilled water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml. phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a glass rod. Make sure the solution contacts all the residue. Add 1 ml. distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml. distilled water, mix well by stirring, and add concentrated ammonium hydroxide dropwise with constant stirring until alkaline to litmus paper. Transfer the solution to a 100 ml. volumetric flask and wash the beaker three times with 4 to 5 ml. portions of distilled water. Dilute to the mark and mix thoroughly. If the sample contains solids, transfer a portion of the solution to a clean, dry centrifuge tube, and centrifuge, or filter a portion of the solution. Measure the absorbance of each sample at 420 nm. using the blank solution as a zero. Dilute the sample and the blank with a suitable amount of distilled water if absorbance falls outside the range of calibration.

5. *Calibration.*

5.1 Flask volume. Assemble the flask and flask valve and fill with water to the stopcock. Measure the volume of water to ±10 ml. Number and record the volume on the flask.

5.2 Spectrophotometer. Add 0.0 to 16.0 ml. of standard solution to a series of beakers. To each beaker add 25 ml. of absorbing solution and add sodium hydroxide (1N) dropwise until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of section 4.3 to collect enough data to draw a calibration curve of concentration in μg. NO₂ per sample versus absorbance.

6. *Calculations.*

6.1 Sample volume.

Figure 7-1. Sampling train, flask valve, and flask.

2.2.3 Glass wash bottle.

2.3 Analysis.

2.3.1 Steam bath.

2.3.2 Beakers or casseroles—250 ml., one for each sample and standard (blank).

2.3.3 Volumetric pipettes—1, 2, and 10 ml.

2.3.4 Transfer pipette—10 ml. with 0.1 ml. divisions.

2.3.5 Volumetric flask—100 ml., one for each sample, and 1,000 ml. for the standard (blank).

2.3.6 Spectrophotometer—To measure absorbance at 420 nm.

2.3.7 Graduated cylinder—100 ml. with 1.0 ml. divisions.

2.3.8 Analytical balance—To measure to 0.1 mg.

3. *Reagents.*

3.1 Sampling.

3.1.1 Absorbing solution—Add 2.8 ml. of concentrated H₂SO₄ to 1 liter of distilled water. Mix well and add 6 ml. of 3 percent hydrogen peroxide. Prepare a fresh solution weekly and do not expose to extreme heat or direct sunlight.

3.2 Sample recovery.

3.2.1 Sodium hydroxide (1N)—Dissolve 40 g. NaOH in distilled water and dilute to 1 liter.

3.2.2 Red litmus paper.

3.2.3 Water—Deionized, distilled.

3.3 Analysis.

3.3.1 Fuming sulfuric acid—15 to 18% by weight free sulfur trioxide.

3.3.2 Phenol—White solid reagent grade.

3.3.3 Sulfuric acid—Concentrated reagent grade.

3.3.4 Standard solution—Dissolve 0.5495 g. potassium nitrate (KNO₃) in distilled water and dilute to 1 liter. For the working standard solution, dilute 10 ml. of the resulting solution to 100 ml. with distilled water. One ml. of the working standard solution is equivalent to 25 μg. nitrogen dioxide.

3.3.5 Water—Deionized, distilled.

3.3.6 Phenoldisulfonic acid solution—Dissolve 25 g. of pure white phenol in 150 ml. concentrated sulfuric acid on a steam bath. Cool, add 75 ml. fuming sulfuric acid, and heat at 100° C. for 2 hours. Store in a dark, stoppered bottle.

4. *Procedure.*

4.1 Sampling.

4.1.1 Pipette 25 ml. of absorbing solution into a sample flask. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Turn the flask valve and the pump valve to their "evacuate"

$$V_{sc}=\frac{T_{std}(V_f-V_a)}{P_{std}}\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)=\left(17.71\ \frac{°R}{in.\ H_g}\right)(V_f-25\ ml.)\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)\quad \text{Equation 7-1}$$

where:

V_{sc}=Sample volume at standard conditions (dry basis), ml.

T_{std}=Absolute temperature at standard conditions, 530° R.

P_{std}=Pressure at standard conditions, 29.92 inches Hg.

V_x=Volume of flask and valve, ml.

V_a=Volume of absorbing solution, 25 ml.

P_f=Final absolute pressure of flask, inches Hg.

P_i=Initial absolute pressure of flask, inches Hg.

T_f=Final absolute temperature of flask, °R.

T_i=Initial absolute temperature of flask, °R.

6.2 Sample concentration. Read μg. NO₂ for each sample from the plot of μg. NO₂ versus absorbance.

$$C=\left(\frac{m}{V_{sc}}\right)\left(\frac{1\ lb.}{cu.\ ft.}\middle/ 1.6\times10^4\frac{\mu g.}{ml.}\right)=\left(6.2\times10^{-8}\frac{lb./s.c.f.}{\mu g./ml.}\right)\left(\frac{m}{V_{sc}}\right)\quad \text{equation 7-2}$$

where:

C=Concentration of NO_x as NO_2 (dry basis), lb./s.c.f.

m=Mass of NO_2 in gas sample, μg.

V_{sc}=Sample volume at standard conditions (dry basis), ml.

7. *References.*

Standard Methods of Chemical Analysis. 6th ed. New York, D. Van Nostrand Co., Inc., 1962, vol. 1, p. 329-330.

Standard Method of Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenoldisulfonic Acid Procedure). In: 1968 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1968, ASTM Designation D-1608-60, p. 725-729.

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METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. A gas sample is extracted from a sampling point in the stack and the acid mist including sulfur trioxide is separated from sulfur dioxide. Both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable to determination of sulfuric acid mist (including sulfur trioxide) and sulfur dioxide from stationary sources only when specified by the test procedures for determining

compliance with the New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling. See Figure 8-1. Many of the design specifications of this sampling train are described in APTD-0581.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system to prevent visible condensation during sampling.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

2.1.4 Filter holder—Pyrex[1] glass.

2.1.5 Impingers—Four as shown in Figure 8-1. The first and third are of the Greenburg-Smith design with standard tip. The second and fourth are of the Greenburg-Smith design, modified by replacing the standard tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the impinger flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inch Hg.

[1] Trade name.

2.2 Sample recovery.

2.2.1 Wash bottles—Two.

2.2.2 Graduated cylinders—250 ml., 500 ml.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Pipette—25 ml., 100 ml.

2.3.2 Burette—50 ml.

2.3.3 Erlenmeyer flask—250 ml.

2.3.4 Graduated cylinder—100 ml.

2.3.5 Trip balance—300 g. capacity, to measure to ±0.05 g.

2.3.6 Dropping bottle—to add indicator solution.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA type 1106 BH, or equivalent, of a suitable size to fit in the filter holder.

3.1.2 Silica gel—Indicating type, 6-16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water—Deionized, distilled.

3.1.4 Isopropanol, 80%—Mix 800 ml. of isopropanol with 200 ml. of deionized, distilled water.

3.1.5 Hydrogen peroxide, 3%—Dilute 100 ml. of 30% hydrogen peroxide to 1 liter with deionized, distilled water.

3.1.6 Crushed ice.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01N)—Dissolve 1.95 g. of barium perchlorate [Ba(ClO₄)₂·3 H₂O] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

3.3.5 Sulfuric acid standard (0.01N)—Purchase or standardize to ± 0.0002 N against 0.01 N NaOH which has previously been standardized against primary standard potassium acid phthalate.

4. *Procedure.*

4.1 Sampling.

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Place 100 ml. of 80% isopropanol in the first impinger, 100 ml. of 3% hydrogen peroxide in both the second and third impingers, and about 200 g. of silica gel in the fourth impinger. Retain a portion of the reagents for use as blank solutions. Assemble the train without the probe as shown in Figure 8-1 with the filter between the first and second impingers. Leak check the sampling train at the sampling site by plugging the inlet to the first impinger and pulling a 15-inch Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 inches Hg is acceptable. Attach the probe and turn on the probe heating system. Adjust the probe heater setting during sampling to prevent any visible condensation. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.

4.1.3 Train operation. For each run, record the data required on the example sheet shown in Figure 8-2. Take readings at each sampling point at least every 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Start the pump and immediately adjust the flow to isokinetic conditions. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the
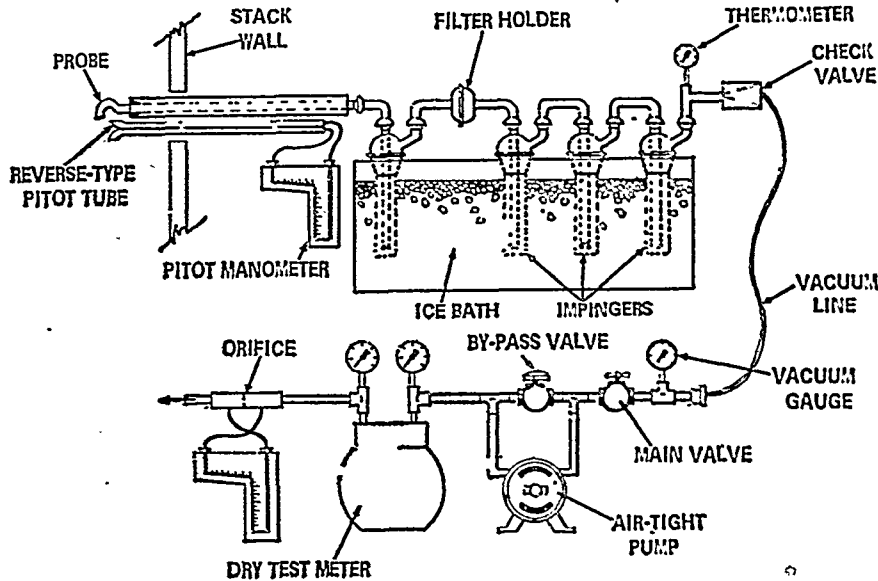
Figure 8-1. Sulfuric acid mist sampling train.

rapid adjustment of the sampling rate without other computations. APTD-0576 details the procedure for using these nomographs. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH@ _____
C FACTOR _____

AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
HEATER BOX SETTING _____
PROBE LENGTH, m. _____
NOZZLE DIAMETER, in. _____
PROBE HEATER SETTING _____

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_s), in. Hg.	STACK TEMPERATURE (T_s), °F.	VELOCITY HEAD (ΔP_s), in. H2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H2O	GAS SAMPLE VOLUME (V_m), ft.3	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
							INLET $(T_{m\,in})$, °F	OUTLET $(T_{m\,out})$, °F			
TOTAL											
AVERAGE							Avg.	Avg.	Avg.	Avg.	

Figure 8-2. Field data.

4.2 Sample recovery.

4.2.1 Transfer the isopropanol from the first impinger to a 250 ml. graduated cylinder. Rinse the probe, first impinger, and all connecting glassware before the filter with 80% isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml. with 80% isopropanol. Add the filter to the solution, mix, and transfer to a suitable storage container. Transfer the solution from the second and third impingers to a 500 ml. graduated cylinder. Rinse all glassware between the filter and silica gel impinger with deionized, distilled water and add this rinse water to the cylinder. Dilute to a volume of 500 ml. with deionized, distilled water. Transfer the solution to a suitable storage container.

4.3 Analysis.

4.3.1 Shake the container holding isopropanol and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100 ml. aliquot of sample into a 250 ml. Erlenmeyer flask and add 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Make sure to record volumes. Repeat the titration with a second aliquot of sample. Shake the container holding the contents of the second and third impingers. Pipette a 25 ml. aliquot of sample into a 250 ml. Erlenmeyer flask. Add 100 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Repeat the titration with a second aliquot of sample. Titrate the blanks in the same manner as the samples.

5. Calibration.

5.1 Use standard methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater.

5.2 Standardize the barium perchlorate with 25 ml. of standard sulfuric acid containing 100 ml. of isopropanol.

6. Calculations.

6.1 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 8-1.

$$V_{m_{std}} = V_m\left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71\frac{°R}{in.\ Hg}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

equation 8-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

V_m = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

T_{std} = Absolute temperature at standard conditions, 530° R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, inches Hg.

ΔH = Pressure drop across the orifice meter, inches H2O.

13.6 = Specific gravity of mercury.

P_{std} = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfuric acid concentration.

$$C_{H_2SO_4} = \left(1.08\times10^{-4}\frac{lb.-l.}{g.-ml.}\right)\frac{(V_t - V_{tb})(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

equation 8-2

where:

$C_{H_2SO_4}$ = Concentration of sulfuric acid at standard conditions, dry basis, lb./cu. ft.

1.08×10^{-4} = Conversion factor including the number of grams per gram equivalent of sulfuric acid (49 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

N = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfuric acid (first impinger and filter), ml.

V_a = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8-1.

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = \left(7.05\times10^{-5}\frac{lb.-l.}{g.-ml.}\right)\frac{(V_t - V_{tb})(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

equation 8-3

where:

C_{SO_2} = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

7.05×10^{-5} = Conversion factor including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

N = Normality of barium perchlorate titrant, g.-eq./l.

V_{soln} = Total solution volume of sulfur dioxide (second and third impingers), ml.

V_a = Volume of sample aliquot titrated, ml.

7. References.

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 999-AP-13, Cincinnati, Ohio, 1965.

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Shell Development Co. Analytical Department, Determination of Sulfur Dioxide and Sulfur Trioxide in Stack Gases, Emeryville Method Series, 4516/59a.

METHOD 9—VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 Principle. The relative opacity of an emission from a stationary source is determined visually by a qualified observer.

1.2 Applicability. This method is applicable for the determination of the relative opacity of visible emissions from stationary sources only when specified by test procedures for determining compliance with the New Source Performance Standards.

2. *Procedure.*

2.1 The qualified observer stands at approximately two stack heights, but not more than a quarter of a mile from the base of the stack with the sun to his back. From a vantage point perpendicular to the plume, the observer studies the point of greatest opacity in the plume. The data required in

Figure 9-1 is recorded every 15 to 30 seconds to the nearest 5% opacity. A minimum of 25 readings is taken.

3. *Qualifications.*

3.1 To certify as an observer, a candidate must complete a smokereading course conducted by EPA, or equivalent; in order to certify the candidate must assign opacity readings in 5% increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent on any one reading and an average error not to exceed 7.5 percent in each category. The smoke generator used to qualify the observers must be equipped with a calibrated smoke indicator or light transmission meter located in the source stack if the smoke generator is to determine the actual opacity of the emissions. All qualified observers must pass this test every 6 months in order to remain certified.

4. *Calculations.*

4.1 Determine the average opacity.

5. *References.*

Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Chapter 2, Schedule 6, Regulation 4, Prohibition, Rule 50, 17 p.

Kudluk, Rudolf, Ringelmann Smoke Chart, U.S. Department of Interior, Bureau of Mines, Information Circular No. 8333, May 1967.

Figure 9-1. Field data.

[FR Doc.71-18824 Filed 12-22-71;8:45 am]