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# federal register

No. 159—Pt. II—1

FRANK M. NOONAN

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TUESDAY, AUGUST 17, 1971

WASHINGTON, D.C.

Volume 36 ■ Number 159

PART II



## ENVIRONMENTAL PROTECTION AGENCY

Standards of Performance for  
New Stationary Sources

Notice of Proposed Rule Making

FRANK M. NOONAN

# ENVIRONMENTAL PROTECTION AGENCY

[ 42 CFR Part 466 ]

## STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

### Proposed Standards for Five Categories

Pursuant to section 111 of the Clean Air Act, as amended, the Administrator published in the FEDERAL REGISTER of March 31, 1971 (36 F.R. 5931), an initial list of five stationary source categories which contribute significantly to air pollution which causes or contributes to the endangerment of public health or welfare. Publication of the list constituted an announcement of the Administrator's intention of establishing, under section 111, standards of performance applicable to new sources within the listed categories. The regulations proposed herein set forth the standards of performance applicable to the five categories. As required by section 111 of the Act, it is the Administrator's intent that standards of performance "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 set forth herein 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. Summaries of the pertinent data, including cost-effectiveness data, are available free of charge from the Office of Air Programs, Environmental Protection Agency, Research Triangle Park, N.C. 27711. Any additional data, including cost-effectiveness data, which can be obtained within the period for comment on these proposed regulations will be taken into consideration prior to promulgation of the standards of performance.

Pursuant to section 114 of the Act, these proposed regulations set forth requirements and establish procedures related to the implementation and enforcement of standards of performance. The test methods to be used in determining compliance with the standards of performance proposed herein are described in detail. It should be noted that the test method applicable to particulate emissions differs from the particulate test method of the American Society of Mechanical Engineers. The particulate test method set forth herein is designed to permit measurement of particulate matter under conditions approximating as closely as possible the conditions under which determination is made of the concentrations of particulate matter in ambient air.

Among other things, these regulations would also establish certain requirements for the installation, use, and maintenance of emission monitoring instruments; certain performance specifica-

tions applicable to such instruments are set forth. Additional guidance on the selection and use of such instruments will be provided at a later date.

These proposed regulations do not include provisions for implementation of section 111(d) of the Act, under which States would be expected to establish emission standards for existing stationary sources of certain pollutants. It is anticipated that such regulations will be published for comment at the time that the Administrator promulgates the standards of performance proposed herein.

Also omitted from the proposed regulations are provisions for delegations of authority to States under section 111(c). Nevertheless, it is the Administrator's intention to encourage States to assume the principal responsibility for enforcement of standards of performance. Toward this end, procedures for delegating authority will be established early next year, after the States have submitted their plans for implementation of national ambient air quality standards.

In accordance with section 117(f) of the Act, publication of these proposed standards was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies.

Interested persons may participate in this rule making by submitting written comments in triplicate to the Office of Air Programs, Environmental Protection Agency, Research Triangle Park, N.C. 27711, Attention: Mr. Goodwin. The Administrator will welcome comments on all aspects of the proposed regulations, including economic and technological issues, and on the proposed test methods. All relevant comments received not later than 45 days after the date of publication of this notice will be considered. Receipt of comments will be acknowledged, but the Office of Air Programs will not provide substantive responses to individual comments. The standards, modified if and as the Administrator deems appropriate after consideration of comments, will be promulgated no later than 90 days from the date of publication of this notice, as required by the Act.

This notice of proposed rule making is issued under the authority of section 111 and section 114 of the Clean Air Act; Public Law 91-604, 84 Stat. 1713.

Dated: August 3, 1971.

ROBERT W. FRI,  
Acting Administrator,  
Environmental Protection Agency.

A new Part 466 would be added to Chapter IV, Title 42, Code of Federal Regulations as follows:

### PART 466—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

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#### Subpart A—General Provisions

##### § 466.10 Applicability.

The provisions of this part apply to the owner or operator of any new source, the construction or modification of which is commenced after the date of publication in the FEDERAL REGISTER of proposed standards of performance which will be applicable to each source.

### § 466.11 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 of performance" or "standard" means a standard for emissions of air pollutants proposed or promulgated by the Administrator pursuant to section 111(b) of the Act.

(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 of performance is specifically 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) "New source" means any affected facility, the construction or modification of which is commenced after the publication in the FEDERAL REGISTER of proposed standards of performance which will be applicable to such facility.

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

(i) "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 emitted by such facility or which results in the emission of any air pollutant not previously emitted, except that:

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

(2) An increase in the production rate or hours of operation shall not be considered a change in the method of operation.

(j) "Commenced" means that an owner or operator and a contractor to, or affiliate of, such owner or operator have entered into a binding agreement or contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

(k) "Performance test" means measurements of emissions or other procedures used for the purpose of determining compliance with a standard of performance.

(l) "Start up of operation" means the beginning of routine operation of an affected facility.

(m) "Ringelmann Chart" means the chart published and described in the U.S. Bureau of Mines Information Circular 8333.

(n) "Particulate matter" means any material, other than uncombined water, which exists in a finely divided form as a liquid or solid at standard conditions, as measured by the test methods set forth in this part.

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

(p) "Visible emission" means an emission of greater than 5 percent opacity or 1/4 Ringelmann, as measured by test methods set forth in this part.

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

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

### § 466.12 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).  
CO<sub>2</sub>—Carbon dioxide.  
gm.—gram.  
gr.—grains.  
Hg—Mercury.  
in.—Inches.  
K—1,000.  
lb.—Pound(s).  
No.—Number.  
%—Percent.  
NO<sub>x</sub>—Nitrogen dioxide.  
NO<sub>x</sub>—Nitrogen oxides.  
NMC—Normal cubic meter.  
s.c.f.—Standard cubic feet.  
SO<sub>2</sub>—Sulfur dioxide.  
H<sub>2</sub>SO<sub>4</sub>—Sulfuric acid.  
SO<sub>3</sub>—Sulfur trioxide.

### § 466.13 Determination of construction or modification.

(a) Upon written application therefor 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.

(b) An application for a determination pursuant to this section shall be delivered to Office of Air Programs, Environmental Protection Agency, Research Triangle Park, N.C. 27711.

### § 466.14 Notification of commencement of construction or modification.

(a) Any person subject to the provisions of this part shall furnish the Administrator written notification in accordance with this section.

(1) A notification generally describing the nature of any intended construction or modification not less than 60 days prior to commencing such construction or modification, except that where such construction or modification is commenced after the initial publication of, but less than 60 days after the effective date of, this part, such notification shall be furnished within 30 days after such effective date.

(2) A notification of the anticipated date of startup of operation of a new source not more than 60 days or less than 30 days prior to such date.

(3) A notification of the actual date of startup of operation of a new source within 15 days after such date.

(b) Notifications required by this part shall be delivered to Office of Air Pro-

grams, Environmental Protection Agency, Research Triangle Park, N.C. 27711.

### § 466.15 Performance tests.

(a) Within 60 days after the startup of operation of a new source, and at intervals thereafter, as specified by the Administrator, the owner or operator 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 methods set forth in this part.

(c) The written reports required by this section shall be delivered to Office of Air Programs, Environmental Protection Agency, Research Triangle Park, N.C. 27711.

(d) The owner or operator of a new source shall provide, or cause to be provided, performance testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to such source.  
(2) Safe sampling platform(s).  
(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(e) Validity of performance test:

(1) Each performance test shall consist of three repetitions of the applicable test method.

(2) Except as otherwise provided by subparagraph (3) of this paragraph, a performance test shall not be considered valid unless the result of each repetition is within 35 percent of the average of the results of all three repetitions.

(3) If the result of one repetition deviates from such average by more than 35 percent, such result may be disregarded, provided that it is shown that such excessive deviation from the average was due to a specified error in testing or a specified upset in operation of the affected facility, and, in such case, the performance test shall be considered valid if the result of each of the other two repetitions is within 35 percent of the average of such two repetitions.

(4) For the purpose of determining compliance with an applicable standard of performance, the average of results of all repetitions which constitute a valid performance test shall apply.

### § 466.16 Review of plans.

(a) Upon written application therefor 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 application shall be submitted for each affected facility.

(2) Each application shall (i) identify the location of such affected facility, (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, and (iii) contain assurances that the applicant will provide any additional information requested by the Administrator.

## PROPOSED RULE MAKING

(c) Neither an application for plans review nor advice furnished by the Administrator in response to such an application 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.

(d) Applications for plans review by the Administrator shall be delivered to Office of Air Programs, Environmental Protection Agency, Research Triangle Park, N.C. 27711.

#### § 466.17 Implementation and enforcement.

After the date of promulgation of any performance standard prescribed by the Administrator, it shall be unlawful for any owner or operator of any new source to operate such source in violation of any standard of performance applicable to such source.

#### § 466.18 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) 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 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.

#### § 466.19 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 of performance 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 B—Standards of Performance for Fossil-Fuel Fired Steam Generators

#### § 466.20 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to fossil fuel-fired steam gen-

erators of more than 250 million B.t.u. per hour heat input.

(b) For purposes of § 466.11(e), that apparatus usually referred to as the furnace or boiler is the affected facility.

#### § 466.21 Definitions.

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

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

(b) "Fossil fuel" means any form of solid, liquid, or gaseous fuel containing carbon or hydrocarbons.

#### § 466.22 Standard for particulate matter.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of particulate matter in the effluent which is:

(a) In excess of 0.20 lb. per million B.t.u. heat input (0.36 gm. per million cal.) maximum 2-hour average.

(b) As dark as, or darker than, the shade designated as No. 1 on the Ringelmann Chart (equal to or greater than 20 percent opacity), except that a shade as dark as No. 2 on the Ringelmann Chart (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.

#### § 466.23 Standard for sulfur dioxide.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of sulfur dioxide in the effluent in excess of:

(a) 0.8 lb. per million B.t.u. heat input (1.4 gm. per million cal.), maximum 2-hour average, when liquid fuel is burned.

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

#### § 466.24 Standard for nitrogen oxides.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of nitrogen oxides in the effluent in excess of:

(a) 0.20 lb. per million B.t.u. heat input (0.36 gm. per million cal.), maximum 2-hour average, expressed as NO<sub>x</sub>, when gaseous fuel is burned.

(b) 0.30 lb. per million B.t.u. heat input (0.54 gm. per million cal.), maximum 2-hour average, expressed as NO<sub>x</sub>, when liquid fuel is burned.

(c) 0.70 lb. per million B.t.u. heat input (1.26 gm. per million cal.), maximum 2-hour average, expressed as NO<sub>x</sub>, when solid fuel is burned.

#### § 466.25 Emission and fuel monitoring.

(a) There shall be installed, calibrated, maintained, and operated, in any fossil fuel-fired steam generator subject to the provisions of this subpart, 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.

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

(b) Instruments installed and used pursuant to this section shall have a confidence level of at least 95 percent and be accurate within  $\pm 20$  percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instruments; instruments shall be calibrated at least once per year unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) The sulfur content of solid fuels, as burned, shall be determined in accordance with the American Society for Testing Materials Method D271-64, and the sulfur content of liquid fuels, as burned, shall be determined in accordance with the American Society for Testing Materials Methods D1551-65T or D129.

(d) The owner or operator of any fossil fuel-fired steam generator subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart and shall retain the record of any such measurement(s) for at least 1 year following the date of such measurements.

#### § 466.26 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 generators.

(b) All performance tests shall be conducted while the affected facility is operating at or above the steam production rate for which such facility was designed.

(c) Test methods set forth in the appendix to this part shall be used as follows:

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5 and traversing according to Method 1. The minimum sampling time shall be 2 hours, and minimum sampling volume shall be 25 ft.<sup>3</sup> corrected to standard conditions on a dry basis.

(2) For each repetition, the SO<sub>2</sub> concentration shall be determined by using Method 6. The sampling location shall be the same as for particulate matter. 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.<sup>2</sup> or at a point no closer to the walls than 3 feet if the cross sectional area is 50 ft.<sup>2</sup> 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.<sup>3</sup> corrected to standard conditions. Two samples shall constitute one repetition and shall be taken over a 2-hour period. Excess air shall be determined at the same sampling point by using the grab sample technique of Method 3.

(3) For each repetition the NO<sub>x</sub> concentration shall be determined by using Method 7. The sampling location and point shall be the same as for SO<sub>2</sub>. The sampling time shall be 2 hours, and four samples shall be taken during each 2-hour period. Excess air shall be determined at the same sampling point by using the grab sampling technique of Method 3.

(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.

(5) The average excess air shall be determined by using Method 3 and traversing according to Method 1.

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

(e) For each repetition, emissions, expressed in lb./10<sup>6</sup> 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,  $lb./hr. = Q \times C$ , where Q=volumetric flow rate of the total effluent in ft.<sup>3</sup>/hr. at standard conditions, dry basis, as determined in accordance with § 466.26(d)(4).

(1) For particulate matter, C=particulate concentration in lb./ft.<sup>3</sup>, as determined in accordance with § 466.26(d)(1), corrected to standard conditions, dry basis.

(2) For SO<sub>2</sub>, C=SO<sub>2</sub> concentration in lb./ft.<sup>3</sup>, as determined in accordance with § 466.26(d)(2), corrected to standard conditions, dry basis, multiplied by the excess air correction factor (100+percent excess air at sampling point)/(100+percent excess air, as determined in accordance with § 466.26(d)(2)).

(3) For NO<sub>x</sub>, C=NO<sub>x</sub> concentration in lb./ft.<sup>3</sup>, as determined in accordance with § 466.26(d)(3), corrected to standard conditions, dry basis, multiplied by the excess air correction factor.

#### Subpart C—Standards of Performance for Incinerators

##### § 466.30 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to incinerators of more than 50 tons per day charging rate.

(b) For purposes of § 466.11(e), that apparatus usually referred to as the furnace is the affected facility.

##### § 466.31 Definitions.

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

(a) "Incinerator" means any furnace and appurtenances thereto used in the process of burning solid waste for the primary purpose of removing the combustible matter from the waste.

(b) "Solid waste" means refuse, more than 50 percent of which is municipal type waste consisting of paper, wood, yard

wastes, food wastes, plastics, leather, rubber, and such other combustibles and noncombustible glass, rock, etc., which may be generated from residential and commercial operations and from small industrial sites.

(c) "Day" means 24 hours.

##### § 466.32 Standard for particulate matter.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of particulate matter in the effluent which is in excess of 0.03 gr./s.c.f. (0.23 gm./NM<sup>3</sup>) corrected to 12 percent CO<sub>2</sub>, maximum 2-hour average.

##### § 466.33 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 refuse charging rate for which such facility was designed.

(c) Test methods set forth in the appendix to this part shall be used as follows:

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5 and traversing according to Method 1. The minimum sampling time shall be 2 hours and the minimum sampling volume shall be 25 ft.<sup>3</sup> corrected to standard conditions on a dry basis.

(2) Gas analysis shall be performed by Method 3, and moisture content shall be determined by the condenser technique of Method 5.

(d) For each repetition particulate matter emissions, expressed in gr./s.c.f., shall be determined in accordance with § 466.33(d)(1) corrected to 12 percent CO<sub>2</sub>, dry basis.

#### Subpart D—Standards of Performance for Portland Cement Plants

##### § 466.40 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to portland cement plants.

(b) For purposes of § 466.11(e) those apparatus usually referred to as the 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 are the affected facilities.

##### § 466.41 Definitions.

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

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

##### § 466.42 Standard for particulate matter.

(a) No person subject to the provisions of this subpart shall cause or allow 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 Kgm. per metric ton), maximum 2-hour average.

(2) As dark as, or darker than, the shade considered to be No. 1/2 on the Ringelmann Chart (equal to or greater than 10 percent opacity), except that where the presence of uncombined water is the only reason for failure to meet the requirements of this subparagraph, such failure shall not be a violation of this section.

(b) No person subject to the provisions of this subpart shall cause or allow 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 (0.05 Kgm. per metric ton) maximum 2-hour average.

(2) A visible emission within the meaning of this part.

(c) No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of particulate matter from any affected facility other than the kiln and clinker cooler which is a visible emission within the meaning of this part.

##### § 466.43 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 production rate for which such facility was designed.

(c) Test methods set forth in the appendix to this part shall be used as follows:

(1) For each repetition, the average concentration of particulate matter shall be determined by using Method 5 and traversing according to Method 1. The minimum sampling time shall be 2 hours, and the minimum sampling volume shall be 25 ft.<sup>3</sup> 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 by Method 3, and moisture content shall be determined by the condenser technique of Method 5.

(d) Kiln feed, expressed in tons per hour on a dry basis, shall be determined 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,  $lb./hr. = Q \times C$ , where Q=volumetric flow rate of the total effluent in ft.<sup>3</sup>/hr. at standard conditions, dry basis, as determined in accordance with § 466.43(d)(2), and C=particulate concentration in lb./ft.<sup>3</sup>, as determined in accordance with § 466.43(d)(1), corrected to standard conditions, dry basis.

### Subpart E—Standards of Performance for Nitric Acid Plants

#### § 466.50 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to nitric acid plants.

(b) For purposes of § 466.11(e), the entire plant is the affected facility.

#### § 466.51 Definitions.

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

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

(b) "Weak nitric acid" means acid which is 50 to 70 percent in strength.

#### § 466.52 Standard for nitrogen oxides.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of nitrogen oxides in the effluent which are:

(a) In excess of 3 lbs. per ton of acid produced (1.5 Kgm. per metric ton), maximum 2-hour average, expressed as NO<sub>2</sub>.

(b) A visible emission within the meaning of this part.

#### § 466.53 Emission monitoring.

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

(b) The instrument installed and used pursuant to this section shall have a confidence level of at least 95 percent and be accurate within  $\pm 20$  percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instrument; the instrument shall be calibrated at least once per year unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) The owner or operator of any nitric acid plant subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart and shall retain the record of any such measurement for at least 1 year following the date of such measurement.

#### § 466.54 Test methods and procedures.

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

(b) All performance tests shall be conducted while the affected facility is operating at or above the acid product rate for which such facility was designed.

(c) Test methods set forth in the appendix to this part shall be used as follows:

(1) For each repetition the NO<sub>x</sub> concentration shall be determined by using Method 7. The sampling location 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 during each 2-hour period.

(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 Method 3, and moisture content shall be determined by Method 4.

(d) Acid produced, expressed in tons per hour of 100 percent weak 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 weak 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,  $lb./hr. = Q \times C$ , where Q=volumetric flow rate of the effluent in ft.<sup>3</sup>/hr. at standard conditions, dry basis, as determined in accordance with § 466.54(d)(2), and C=NO<sub>x</sub> concentration in lb./ft.<sup>3</sup>, as determined in accordance with § 466.54(d)(1), corrected to standard conditions, dry basis.

### Subpart F—Standards of Performance for Sulfuric Acid Plants

#### § 466.60 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to sulfur acid plants.

(b) For purposes of § 466.11(e) the entire plant is the affected facility.

#### § 466.61 Definitions.

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

(a) "Sulfuric acid plant" 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.

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

#### § 466.62 Standard for sulfur dioxide.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of sulfur dioxide in the effluent in excess of 4 lbs. per ton of acid produced (2 kgm. per metric ton), maximum 2-hour average.

#### § 466.63 Standard for acid mist.

No person subject to the provisions of this subpart shall cause or allow the discharge into the atmosphere of acid mist in the effluent which is:

(a) In excess of 0.15 lb. per ton of acid produced (0.075 Kgm. per metric ton), maximum 2-hour average, expressed as H<sub>2</sub>SO<sub>4</sub>.

(b) A visible emission within the meaning of this part.

#### § 466.64 Emission monitoring.

(a) There shall be installed, calibrated, maintained, and operated, in any sulfuric acid plant subject to the provisions of this subpart, an instrument for continu-

ously monitoring and recording emissions of sulfur dioxide.

(b) The instrument installed and used pursuant to this section shall have a confidence level of at least 95 percent and be accurate within  $\pm 20$  percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be calibrated at least once per year unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed.

(c) The owner or operator of any sulfuric acid plant subject to the provisions of this subpart shall maintain a file of all measurements required by this subpart and shall retain the record of any such measurement for at least 1 year following the date of such measurement.

#### § 466.65 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 plants.

(b) All performance tests shall be conducted while the affected facility is operating at or above the acid production rate for which such facility was designed.

(c) Test methods set forth in the appendix to this part shall be used as follows:

(1) For each repetition the acid mist and SO<sub>2</sub> concentrations shall be determined by using Method 8 and traversing according to Method 1. The sampling time shall be 2 hours, and sampling volume shall be 40 ft.<sup>3</sup> 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 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 sulfuric 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,  $lb./hr. = Q \times C$ , where Q=volumetric flow rate of the effluent in ft.<sup>3</sup>/hr. at standard conditions, dry basis, as determined in accordance with § 466.65(d)(2), and C=acid mist and SO<sub>2</sub> concentrations in lb./ft.<sup>3</sup> as determined in accordance with § 466.65(d)(1), 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

PROPOSED RULE MAKING

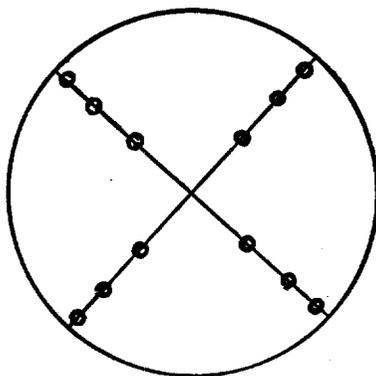


Figure 1-2. Cross section of circular stack showing location of traverse points on perpendicular diameters.

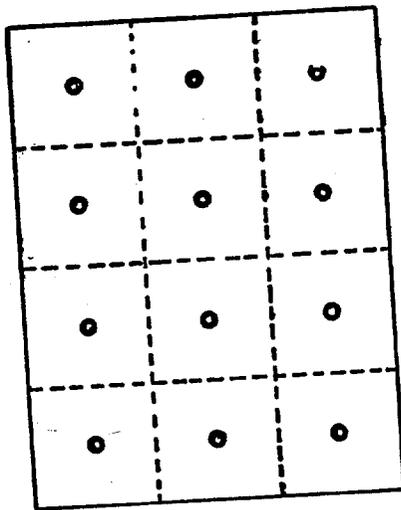


Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

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.

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 four, 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 two perpendicular diameters according to Figure 1-2 and Table 1-1.

New Source Performance Standards. 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 a rectangular cross section, determine an equivalent diameter from the following equation:

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

equation 1-1

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

NUMBER OF DUCT DIAMETERS UPSTREAM\* (DISTANCE A)

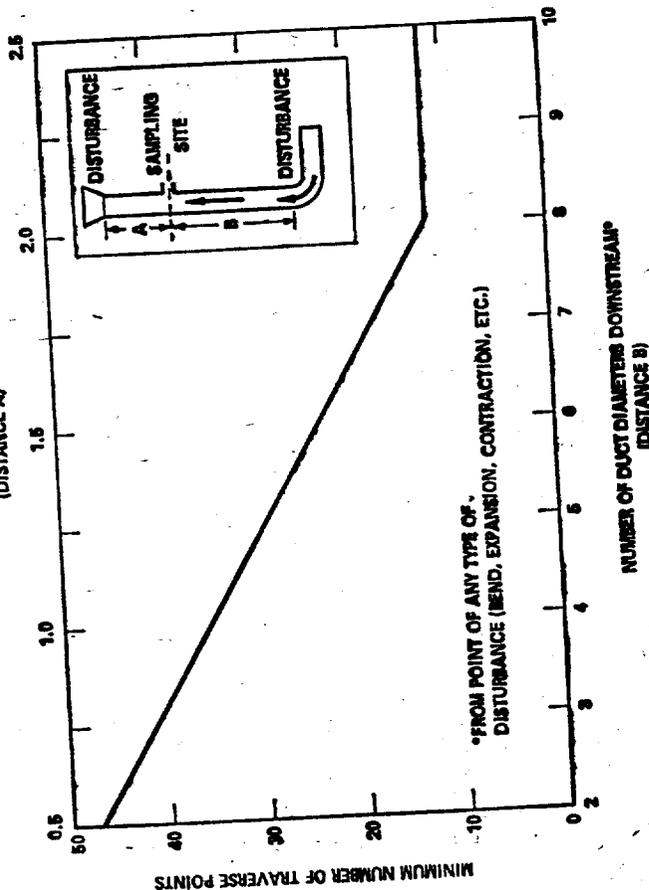


Figure 1-1. Minimum number of traverse points.

- not be used in the case of nondirectional flow.
2. Apparatus.
- 2.1 Pitot tube—Type S (Figure 2-1), or equivalent.
- 2.2 Differential pressure gauge—Inclined manometer, or equivalent, to measure velocity head to within 10 percent of the minimum valve.
- 2.3. Temperature gauge—Thermocouples, bimetallic thermometers, liquid filled systems, or equivalent, to measure stack temperature to within 1.5 percent 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 at the traverse points specified by Method 1.
- 3.2 Measure the temperature of the stack gas. If the total temperature variation with time is less than 50° F., a point measurement will suffice. Otherwise, conduct a temperature traverse.
- 3.3 Measure the static pressure in the stack.
- 3.4 Determine the stack gas molecular weight by gas analysis and appropriate calculation as indicated in Method 3.

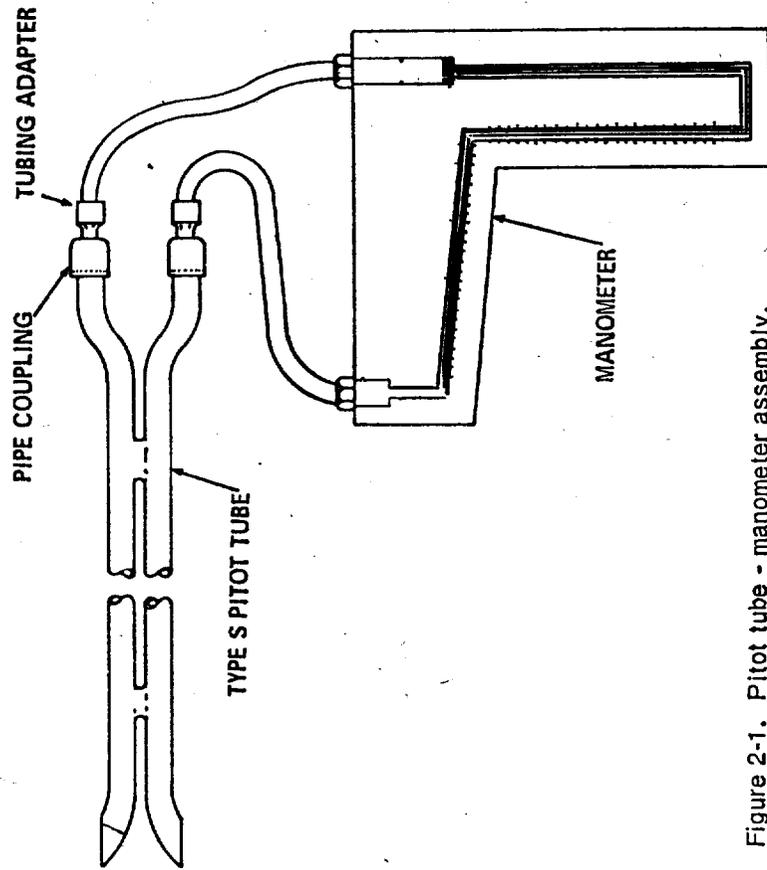


Figure 2-1. Pitot tube - manometer assembly.

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. The velocity of the flowing gas stream should be within the normal working range.

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									
	6	8	10	12	14	16	18	20	22	24
1	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4	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	14.4
8	96.7	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4	17.4
9	91.8	82.3	73.1	62.5	38.2	30.6	26.1	23.0	20.4	18.4
10	97.5	88.2	79.9	71.7	61.8	38.8	31.5	27.2	24.0	21.6
11	93.3	85.4	78.0	70.4	61.2	39.3	32.3	28.0	24.8	22.4
12	97.9	90.1	83.1	76.4	69.4	60.7	39.8	32.3	28.0	24.8
13	94.3	87.5	81.2	75.0	68.5	60.2	40.2	32.7	28.4	25.2
14	98.2	91.5	85.4	79.6	73.9	67.7	40.6	33.1	28.8	25.6
15	95.1	89.1	83.5	78.2	72.8	67.0	41.0	33.5	29.2	26.0
16	98.4	92.5	87.1	82.0	77.0	71.4	41.4	33.9	29.6	26.4
17	95.6	90.3	85.4	80.6	75.4	70.0	41.8	34.3	30.0	26.8
18	98.6	93.3	88.4	83.9	78.8	73.4	42.2	34.7	30.4	27.2
19	96.1	91.3	86.8	81.9	77.0	71.6	42.6	35.1	30.8	27.6
20	99.7	94.0	89.5	84.9	80.0	74.6	43.0	35.5	31.2	28.0
21	96.5	92.1	88.9	84.5	80.0	74.6	43.4	35.9	31.6	28.4
22	98.9	94.5	90.9	86.5	82.0	76.6	43.8	36.3	32.0	28.8
23	96.8	93.9	90.1	86.1	81.6	76.2	44.2	36.7	32.4	29.2
24	99.9	96.8	93.9	89.9	85.4	80.0	44.6	37.1	32.8	29.6

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

METHOD 2—DETERMINATION OF STACK GAS VELOCITY (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.
- 1.3 Applicability. This method should be applied only when specified by the test procedures for determining compliance with New Source Performance Standards. Being a directional instrument, a pitot tube should

- 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, 1967.
- Devorckin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles. November 1963.
- Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles. Bulletin WP-50. 1968.



## PROPOSED RULE MAKING

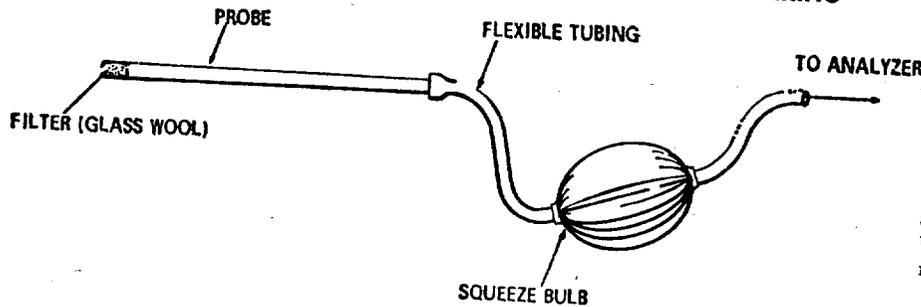


Figure 3-1. Grab-sampling train.

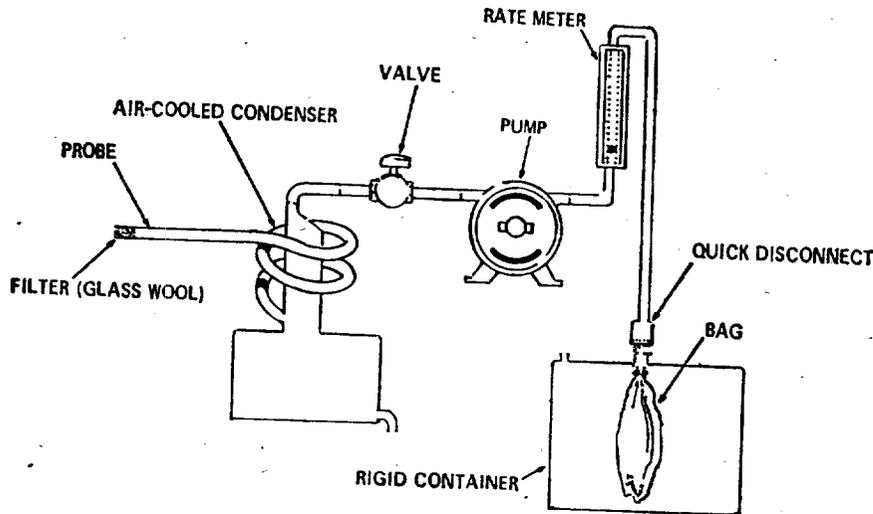


Figure 3-2. Integrated gas-sampling train.

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 gas velocity.

3.3 Analysis.

3.3.1 Determine the CO<sub>2</sub>, O<sub>2</sub>, and CO concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than 10 passes are necessary, replace the absorbing solution.

3.3.2 For integrated sampling, repeat the analysis until three consecutive runs 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 result to the nearest 0.1 percent CO<sub>2</sub>.

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

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

equation 3-1

where:

% EA = Percent excess air.

% O<sub>2</sub> = Percent oxygen by volume, dry basis.

% N<sub>2</sub> = 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.

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

Equation 3-2

where:

M<sub>d</sub> = Dry molecular weight, lb./lb.-mole.

% CO<sub>2</sub> = Percent carbon dioxide by volume, dry basis.

% O<sub>2</sub> = Percent oxygen by volume, dry basis.

% N<sub>2</sub> = 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 divided by 100.

## 5. References

- Altshuler, 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, November 1963.

## 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 gravimetrically.

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.<sup>2</sup>

Other methods such as drying tubes, wet bulb-dry bulb techniques, and volumetric condensation techniques may be used subject to the approval of the Administrator.

## 2. Apparatus.

2.1 Probe—Stainless steel or Pyrex<sup>1</sup> 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—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 percent of the total sample volume.

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

2.9 Balance—Capable of measuring to the nearest 0.1 g.

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

2.11 Pilot 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 about 5 ml. distilled water in each impinger and weigh the impinger and contents to the nearest 0.1 g. 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 percent 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 not to exceed 0.075 c.f.m. Continue sampling until the dry gas meter registers 1 cu. ft. or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter reading as required by Figure 4-2.

3.3 After collecting the sample, weigh the impingers and their contents again to the nearest 0.1 g.

<sup>1</sup> Trade name.

<sup>2</sup> If liquid droplets are present in the gas stream, assume the stream to be saturated, determine the average stack gas temperature (Method 1), and use a psychrometric chart to obtain an approximation of the moisture percentage.

4. Calculations.

4.1 Volume of water collected.

$$V_{wc} = \frac{(W_f - W_i)RT_{std}}{P_{std}M_w} = (0.0474 \frac{ft.^3}{g.})(W_f - W_i)$$

equation 4-1

where:

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

$W_f$  = Final weight of impingers and contents, g.  
 $W_i$  = Initial weight of impingers and contents, g.  
 $R$  = Ideal gas constant, 21.83-in. Hg-cu. ft./lb. mole-° R.  
 $T_{std}$  = Absolute temperature at standard conditions, 530° R.  
 $P_{std}$  = Pressure at standard conditions, 29.92 in. Hg.  
 $M_w$  = Molecular weight of water, 18 lb./lb. mole.

4.2 Gas volume.

$$V_{mc} = V_m \left( \frac{P_m}{P_{std}} \right) \left( \frac{T_{std}}{T_m} \right) = (17.71 \frac{°R}{in. Hg}) \frac{V_m P_m}{T_m}$$

equation 4-2

where:

$V_{mc}$  = 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, in. Hg.  
 $P_{std}$  = Pressure at standard conditions, 29.92-in. Hg.  
 $T_{std}$  = Absolute temperature at standard conditions, 530° R.  
 $T_m$  = Absolute temperature at meter (°F. + 460), °R.

4.3 Moisture content.

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

equation 4-3

where:

$B_{wo}$  = Proportion by volume of water vapor in the gas stream, dimensionless.  
 $V_{wc}$  = Volume of water vapor collected (standard conditions), cu. ft.  
 $V_{mc}$  = 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<sup>1</sup> glass with a heating system capable of maintaining a gas temperature of 250° F. at the exit end during sampling. When temperature or length limitations are encountered, 316 stainless steel, or equivalent, may be used, as approved by the Administrator.

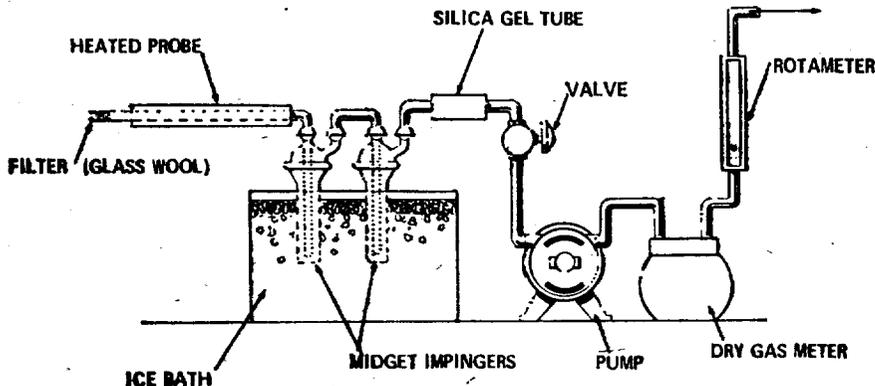


Figure 4-1. Moisture-sampling train.

LOCATION \_\_\_\_\_ COMMENTS \_\_\_\_\_  
 TEST \_\_\_\_\_  
 DATE \_\_\_\_\_  
 OPERATOR \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), ft <sup>3</sup>	ROTAMETER SETTING, ft <sup>3</sup> /min	METER TEMPERATURE, °F

Figure 4-2. Field moisture determination.

## PROPOSED RULE MAKING

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

2.1.4 Filter holder—Pyrex<sup>1</sup> glass with heating system capable of maintaining any temperature to a maximum of 225° F.

2.1.5 Impingers—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by re-

placing the tip with a 1/4-inch ID glass tube extending to 1/4-inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

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

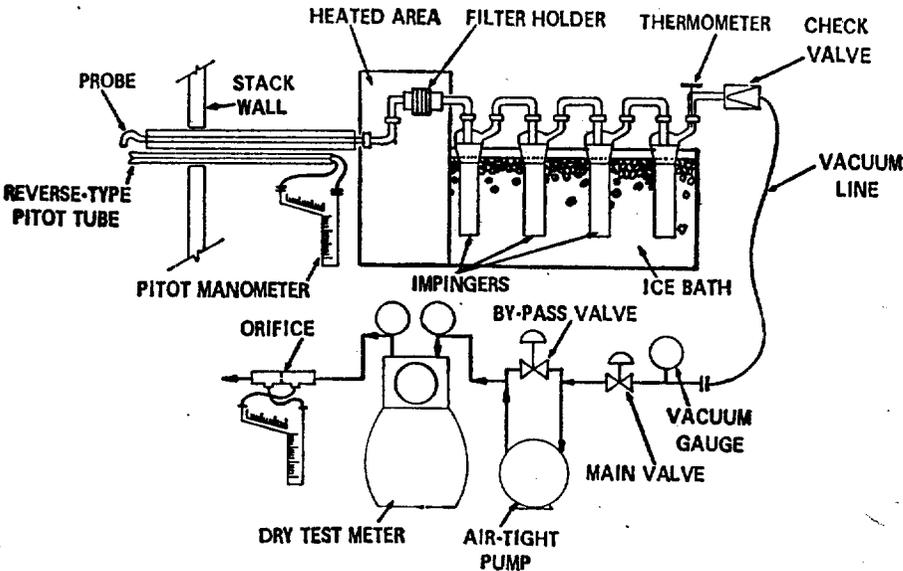


Figure 5-1. Particulate-sampling train.

2.1.7 Barometer—To measure atmospheric pressure to  $\pm 0.1$  in. 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  $\pm 0.1$  mg.

2.3.4 Beakers—250 ml.

<sup>1</sup> Trade name.

2.3.5 Separatory funnels—500 ml. and 1,000 ml.

2.3.6 Trip balance—300 g. capacity, to measure to  $\pm 0.05$  g.

2.3.7 Graduated cylinder—25 ml.

3. Reagents.

3.1 Sampling

3.1.1 Filters—Glass fiber, MSA 1106 BE, or equivalent, numbered for identification and preweighed.

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

3.1.3 Water—Deionized, distilled.

3.1.4 Crushed ice.

3.2 Sample recovery

3.2.1 Water—Deionized, distilled.

3.2.2 Acetone—Reagent grade.

3.3 Analysis

3.3.1 Water—Deionized, distilled.

3.3.2 Chloroform—Reagent grade.

3.3.3 Ethyl ether—Reagent grade.

3.3.4 Desiccant—Drierite,<sup>1</sup> 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<sup>2</sup> for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50 percent. 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. Save a portion of the water for use as a blank in the sample analysis. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging 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 more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.

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. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APID-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.

<sup>2</sup> Dry using Drierite<sup>1</sup> at 70°  $\pm$  10° F.



PROPOSED RULE MAKING

PLANT \_\_\_\_\_  
 DATE \_\_\_\_\_  
 RUN NO. \_\_\_\_\_

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
3a*			
3b**			
5			
TOTAL			

\*3a- ORGANIC EXTRACT FRACTION.  
 \*\*3b - RESIDUAL WATER FRACTION.

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

\* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER. (1 g/ml):

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

Figure 5-3. Analytical data.

Container No. 5. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate, dry to a constant weight, and report the results to the nearest 0.5 mg.

5. Calibration.

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

6. Calculations.

6.1 Sample concentration method.

6.1.1 Average dry gas meter temperature. See data sheet (Figure 5-2).

6.1.2 Dry gas volume. Correct the sample

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

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

$$\left( 17.71 \frac{^{\circ}\text{R}}{\text{in. Hg}} \right) (V_m) \left( \frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} \right)$$

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, in. Hg.

$\Delta H$  = Pressure drop across the orifice meter, in H<sub>2</sub>O.

13.6 = Specific gravity of mercury.

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

6.1.3 Volume of Water vapor.

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

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.

$\rho_{H_2O}$  = Density of water, 1 g./ml.

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

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

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

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

6.1.4 Total gas volume.

$$V_{total} = V_{m, std} + V_{w, std}$$

equation 5-3

where:

$V_{total}$  = Total volume of gas sample (standard conditions), cu. ft.

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

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

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

6.1.6 Concentration.

$$c_s' = \left( 0.0154 \frac{\text{gr.}}{\text{mg.}} \right) \left( \frac{M_a}{V_{total}} \right)$$

equation 5-4

where:

$c_s'$  = Concentration of particulate matter in stack gas (Sample Concentration Method), gr./s.c.f.

$M_a$  = Total amount of particulate matter collected, mg.

$V_{total}$  = Total volume of gas sample (standard conditions), cu. ft.

6.2 Ratio of area method.

6.2.1 Stack gas velocity. Collect the necessary data as detailed in Method 2. Correct the

stack gas velocity to standard conditions (29.92 in. Hg, 530° R.) as follows:

$$V_{s,1d} = V_s \left( \frac{P_s}{P_{s,1d}} \right) \left( \frac{T_{s,1d}}{T_s} \right) = \left( \frac{17.71}{\text{in. Hg}} \right) \left( \frac{P_s}{P_{s,1d}} \right) \left( \frac{V_s P_s}{T_s} \right) \text{ equation 5-5}$$

where:

$V_{s,1d}$  = Stack gas velocity at standard conditions, ft./sec.

$$C_s = \frac{M_s A_s}{Q_s A_s} = \frac{M_s}{A_s V_{s,1d}} = \left( \frac{2.57 \times 10^{-4} \text{ gr.} = \text{min.}}{\text{mg.} = \text{sec.}} \right) \left( \frac{M_s}{\theta V_{s,1d} A_s} \right) \text{ equation 5-6}$$

where:

$C_s$  = Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

$M_s$  = Particulate mass flow rate through the stack (standard conditions), mass/time.

$Q_s$  = Volumetric flow rate of gas stream through the stack (standard conditions), volume/time.

$$I = \frac{C_s}{C_s} \times 100 = \frac{T_s \left[ \frac{V_1 \rho_{H_2O} R}{M_{H_2O}} + \frac{V_2}{T_s} (P_{bar} + \frac{\Delta H}{13.6}) \right] \times 100}{(1.667 \frac{\text{min.}}{\text{sec.}}) \left[ (0.00267 \frac{\text{in. Hg-cu. ft.}}{\text{ml.} \cdot \text{°R.}}) V_{10} + \frac{V_2}{T_s} (P_{bar} + \frac{\Delta H}{13.6}) \right] T_s} \theta V_s P_s A_s$$

where:

$I$  = Percent of isokinetic sampling.

$C_s$  = Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

$C_s'$  = Concentration of particulate matter in the stack gas (Sample Concentration Method), gr./s.c.f.

$V_1$  = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

$\rho_{H_2O}$  = Density of water, 1 g./ml.

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

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

$V_2$  = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

$T_s$  = Absolute average dry gas meter temperature (see Figure 5-2), °R.

$P_{bar}$  = Barometric pressure at sampling site, in Hg.

$\Delta H$  = Average pressure drop across the orifice (see Figure 5-2), in H<sub>2</sub>O.

$T_s$  = Absolute average stack gas temperature (see Figure 5-3), °R.

$V_s$  = Stack gas velocity calculated by Method 2, Equation 2-3, ft./sec.

$P_s$  = Absolute stack gas pressure, in. Hg.

$P_{s,1d}$  = Absolute pressure at standard conditions, 29.92 in. Hg.

$T_{s,1d}$  = Absolute temperature at standard conditions, 530° R.

$T_s$  = Absolute stack gas temperature (average), °R.

6.2.2 Concentration.

$$C_s = \frac{M_s}{Q_s A_s} = \left( \frac{2.57 \times 10^{-4} \text{ gr.} = \text{min.}}{\text{mg.} = \text{sec.}} \right) \left( \frac{M_s}{\theta V_{s,1d} A_s} \right) \text{ equation 5-6}$$

$M_s$  = Total amount of particulate matter collected by train, mg.

$\theta$  = Total sampling time, min.

$A_s$  = Cross-sectional area of stack, sq. ft.

$A_{s,1d}$  = Cross-sectional area of nozzle, sq. ft.

$V_{s,1d}$  = Stack gas velocity at standard conditions, ft./sec.

6.3 Isokinetic variation.

equation 5-7

$\theta$  = Total sampling time, min.

$V_s$  = Stack gas velocity calculated by Method 2, Equation 2-3, ft./sec.

$P_s$  = Absolute stack gas pressure, in. Hg.

$A_s$  = Cross-sectional area of nozzle, sq. ft.

6.4 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:  
If 82 percent < I < 120 percent, the results are acceptable; otherwise, reject the results and repeat the test.

6.5 Average particulate concentration. If the criteria for acceptability are met, calculate the average concentration of particulate in the stack from the following equation:

$$C_s = \frac{C_s + C_s'}{2} \text{ Equation 5-8}$$

where:

$C_s$  = Average particulate concentration in the stack gas, gr./s.c.f.

$C_s'$  = Concentration of particulate matter in the stack gas (Ratio of Area Method), gr./s.c.f.

$C_s'$  = Concentration of particulate matter in the stack gas (Sample Concentration Method), gr./s.c.f.

7. References.  
Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. Dec. 6, 1987.

Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency, APTD-0561. 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, 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, 1987.

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, and 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 to:

PROBE (END PACKED WITH QUARTZ OR PYREX WOOL)

STACK WALL

MIDGET WOOL

GLASS WOOD

MIDGET BUBBLER MIDGET IMPINGERS

SILICA GEL DRYING TUBE

TYPE S PITOT TUBE

PITOT MANOMETER

ICE BATH

ROTAMETER

DRY GAS METER

PUMP

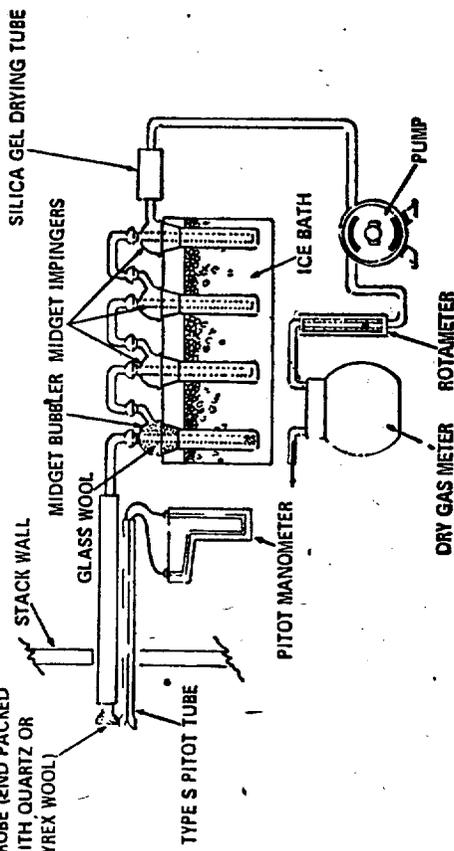


Figure 6-1. SO<sub>2</sub> sampling train.

## PROPOSED RULE MAKING

2.3.1 Pipettes—Transfer type, 5 ml. and 10 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. divisions).

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.

## 3. Reagents.

## 3.1 Sampling.

3.1.1 Water—Deionized, distilled.

3.1.2 Isopropanol, 80 percent—Mix 80 ml. of isopropanol with 20 ml. of distilled water.

3.1.3 Hydrogen peroxide, 3 percent—dilute 100 ml. of 30 percent hydrogen peroxide with 900 ml. of distilled water. Prepare fresh daily.

## 3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80 percent.

## 3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(*o*-arsenophenylazo)-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 [ $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ ] 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 against a primary standard to  $\pm 0.0002N$ .

## 4. Procedure.

## 4.1 Sampling.

4.1.1 Preparation of collection train. Pour 15 ml. of 80 percent isopropanol into the midget bubbler and 15 ml. of 3 percent hydrogen peroxide to 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-in. Hg vacuum. A leakage rate not in excess of 1 percent 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 as velocity. Take readings at least every 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle with the tip pointing directly into the gas stream 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 the purging period. 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 to a 125-ml. erlenmeyer flask. Add 40 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate to a pink endpoint using 0.01N barium perchlorate. Run a blank with each series of samples.

## 5. Calibration.

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

5.2 Standardize the sulfuric acid with potassium acid phthalate as a primary standard. 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. and 29.92 in. Hg) by using Equation 6-1.

$$V_{m, std} = V_m \left( \frac{T_{std}}{T_m} \right) \left( \frac{P_{bar}}{P_{std}} \right) = \left( 17.71 \frac{^{\circ}\text{R}}{\text{in. Hg}} \right) \frac{V_m P_{bar}}{T_m} \quad \text{equation 6-1}$$

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, in. Hg.

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

6.2 Sulfur dioxide concentration.

$C_{SO_2}$  =

$$\left( 7.05 \times 10^{-5} \frac{\text{lb.-l.}}{\text{g.-ml.}} \right) \frac{(V_t - V_b)(N) \left( \frac{V_{soi}}{V_s} \right)}{V_{m, std}} \quad \text{equation 6-2}$$

where:

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

$7.05 \times 10^{-5}$  = 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.

$V_b$  = Volume of barium perchlorate titrant used for the blank, ml.

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

$V_{soi}$  = Total solution volume of sulfur dioxide, ml.

$V_s$  = Volume of sample aliquot titrated, ml.

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

## 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  $\text{SO}_2$  and  $\text{SO}_3$  in Flue Gases. Journal of the Institute of Fuel. 24:237-243, 1961.

Matty, R. E. and E. K. Diehl. Measuring Flue-Gas  $\text{SO}_2$  and  $\text{SO}_3$ . Power 101:94-97, November 1957.

Patton, W. F. and J. A. Brink, Jr. New Equipment and Techniques for Sampling Chemical Process Gases. Paper presented at the 55th Annual Meeting of APCA, Chicago, Ill. May 20-24, 1962.

## 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 measured 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<sup>1</sup> 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<sup>1</sup> round bottom with short neck and 24/40 standard taper opening, protected against implosion or breakage.

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-in. 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-in. 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.

<sup>1</sup> Trade name.

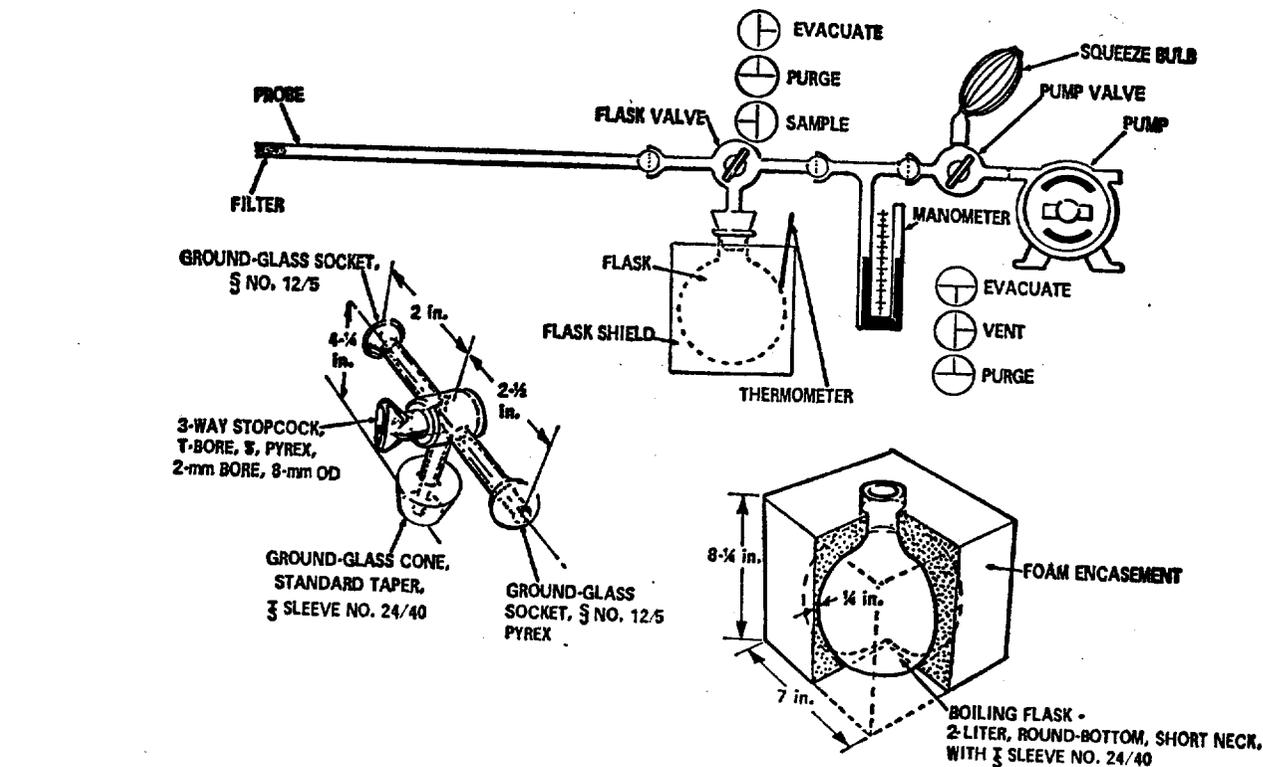


Figure 7-1. Sampling train, flask valve, and flask.

- 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 420m $\mu$ .
- 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 H<sub>2</sub>SO<sub>4</sub> 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 18N 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 (KNO<sub>3</sub>) 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  $\mu$ g. nitrogen dioxide.

- 3.3.5 Water—Deionized, distilled.
- 3.3.6 Phenol-disulfonic 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" positions. Evacuate the flask to at least 3-in. 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 1 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 sit 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 water (approximately 10 ml.) and add to the same amount of rinse water as in the sample. For a blank use 25 ml. of absorbing solution and the solution in a container for shipment or in a 250-ml. beaker for analysis. 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 water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml. phenol-disulfonic acid solution to the dried residue and triturate thoroughly with a glass rod. Make sure the solution contacts all the residue. Add 1 ml. water and 4 drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional 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 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 m $\mu$  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  $\pm$ 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 26 ml. of absorbing solution and add sodium hydroxide (1N) dropwise until alkaline to litmus paper

$$V_{s0} = \frac{T_{100}(V_1 - V_2)}{P_{100}} \left( \frac{P_1}{T_1} - \frac{P_2}{T_2} \right) (V_1 - 25 \text{ ml.}) \left( \frac{P_1}{T_1} - \frac{P_2}{T_2} \right) \text{ equation 7-1}$$

where:

- $V_{s0}$  = Sample volume at standard conditions (dry basis), ml.
- $T_{100}$  = Absolute temperature at standard conditions, 530° R.
- $P_{100}$  = Pressure at standard conditions, 29.92 in. Hg.
- $V_1$  = Volume of flask and valve, ml.
- $V_2$  = Volume of absorbing solution, 26 ml.
- $P_1$  = Final absolute pressure of flask, in. Hg.
- $P_2$  = Initial absolute pressure of flask, in. Hg.
- $T_1$  = Final absolute temperature of flask, °R.
- $T_2$  = Initial absolute temperature of flask, °R.

6.2 Sample concentration. Read  $\mu\text{g. NO}_2$  for each sample from the plot  $\mu\text{g. NO}_2$  versus absorbance.

$$c = \left( \frac{m}{V_s} \right) \left( \frac{1 \text{ lb./cu. ft.}}{1.6 \times 10^4 \mu\text{g./ml.}} \right) = \left( 6.2 \times 10^{-4} \frac{\text{lb./s.c.f.}}{\mu\text{g./ml.}} \right) \left( \frac{m}{V_s} \right) \text{ equation 7-2}$$

where:

- $c$  = Concentration of  $\text{NO}_2$  as  $\text{NO}_2$  (dry basis), lb./s.c.f.
- $m$  = Mass of  $\text{NO}_2$  in gas sample,  $\mu\text{g.}$
- $V_s$  = Sample volume at standard conditions (dry basis), ml.

7. References.

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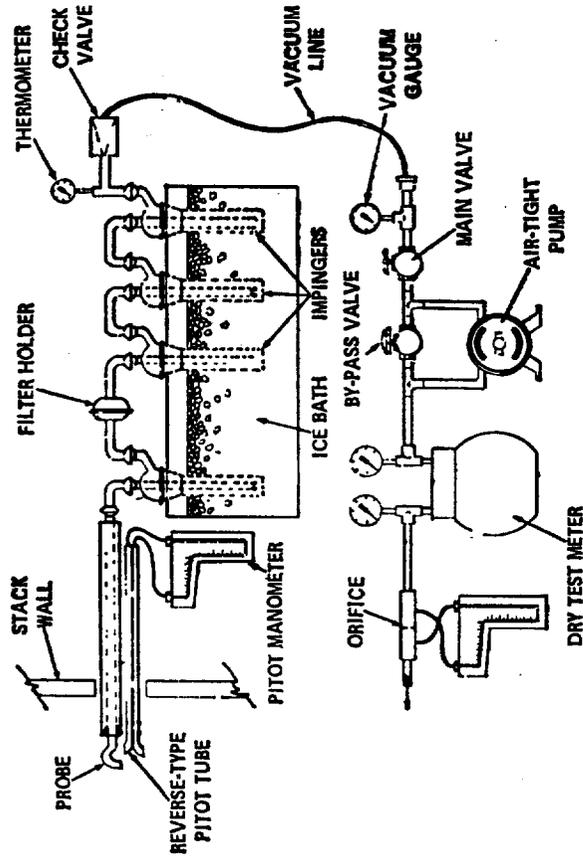


Figure 8-1. Sulfuric acid mist sampling train.

- 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  $\pm 0.05$  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 percent—Mix 800 ml. of isopropanol with 200 ml. of deionized, distilled water.
    - 3.1.5 Hydrogen peroxide, 3 percent—Dilute 100 ml. of 30 percent hydrogen peroxide with 900 ml. deionized, distilled water.
- 3.1.6 Crushed ice.
  - 3.2 Sample recovery.
    - 3.2.1 Water—Deionized, distilled.
    - 3.2.2 Isopropanol, 80 percent.
    - 3.3 Analysis.
      - 3.3.1 Water—Deionized, distilled.
      - 3.3.2 Isopropanol.
      - 3.3.3 Thorin indicator—1 - (o - arsono-phenylazo)-2-naphthol-3, 6-disulfonic acid, sodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.
      - 3.3.4 Barium perchlorate (0.01N)—Dissolve 1.96 g. of barium perchlorate [ $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ ] 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 against a primary standard to  $\pm 0.0002N$ .
  - 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 percent isopropanol in the first impinger, 100 ml. of 3 percent hydrogen peroxide in both the second and third impingers,



6.2 Sulfuric acid concentration

$$c_{H_2SO_4} = \left( 1.08 \times 10^{-4} \frac{\text{lb.-l.}}{\text{g.-ml.}} \right) \frac{(V_t - V_{tb})(N) \left( \frac{V_{soln}}{V_s} \right)}{V_{std}}$$

equation 8-2

where:

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

$1.08 \times 10^{-4}$  = 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 blank, ml.

$N$  = Normality of barium perchlorate titrant, q.-equivalent/liter.

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

$V_s$  = Volume of sample aliquot titrated, ml.

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

6.3 Sulfur dioxide concentration

$$c_{SO_2} = \left( 7.05 \times 10^{-5} \frac{\text{lb.-l.}}{\text{g.-ml.}} \right) \frac{(V_t - V_{tb})(N) \left( \frac{V_{soln}}{V_s} \right)}{V_{std}}$$

equation 8-3

where:

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

$7.05 \times 10^{-5}$  = 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.

$V_s$  = Volume of sample aliquot titrated, ml.

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

7. References.

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METHOD 9—DETERMINATION OF THE OPTICAL DENSITY OR OPACITY OF VISIBLE EMISSIONS FROM STATIONARY SOURCES

1. Principle and applicability.

1.1 Principle. The relative optical density or 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 optical density or opacity of visible emissions from stationary sources only when specified by test procedures for determining compliance with New Source Performance Standards.

2. Procedures.

2.1 Black or gray smoke. The qualified observer stands at least 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 optical density in the plume. Record the data required in Figure 9-1 every 15 to

30 seconds. Read to the nearest quarter Ringelmann number.

2.2 Smoke other than gray or black. The qualified observer goes through the same procedure as depicted in section 2.1 except readings of opacity are recorded instead of Ringelmann numbers.

3. Qualifications.

3.1 To qualify as an observer, a candidate must complete a smoke-reading course conducted by OAP, or equivalent. The candidate must be able to assign Ringelmann numbers to the nearest quarter of a Ringelmann number to 25 different smoke plumes, with an error not to exceed 15 percent on any one reading and an average error not to exceed 7.5 percent on all 25 readings. The smoke generator used to train the observers should be equipped with a calibrated smoke indicator or light transmission meter located in the source stack of the smoke generator to determine the actual Ringelmann number or opacity of the emissions. All qualified observers must pass this test every year in order to be recertified.

3.2 To pass the test for certification for observing opacity, one must assign opacity readings in 5 percent increments to 25 different plumes, with an error not to exceed 15 percent on any one reading and an average error not to exceed 7.5 percent on all 25 readings. All qualified observers must pass this test every year in order to be recertified.

4. Calculations.

4.1 Ringelmann number. Determine the average reading.

4.2 Opacity. Determine the average reading.

TIME	Ringelmann Number					Opacity				
	0	¼	½	¾	1	0	¼	½	¾	1
0						30				
1						31				
2						32				
3						33				
4						34				
5						35				
6						36				
7						37				
8						38				
9						39				
10						40				
11						41				
12						42				
13						43				
14						44				
15						45				
16						46				
17						47				
18						48				
19						49				
20						50				
21						51				
22						52				
23						53				
24						54				
25						55				
26						56				
27						57				
28						58				
29						59				

Observation date \_\_\_\_\_

Plant \_\_\_\_\_

Stack location \_\_\_\_\_

Observer \_\_\_\_\_

Date \_\_\_\_\_

Time \_\_\_\_\_

Distance to stack \_\_\_\_\_

Wind direction \_\_\_\_\_

Wind speed \_\_\_\_\_

Sum of numbers recorded \_\_\_\_\_

Total number of readings \_\_\_\_\_

Ringelmann no. : Sum of nos. recorded

Total no. readings

Opacity : Sum of nos. recorded

Total no. readings

Figure 9-1. Field data.

5. References.

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[FR Doc.71-11438 Filed 8-16-71;8:45 am]