On August 17, 1971, pursuant to section 111 of the Clean Air Act as amended, the Administrator proposed standards of performance for steam generators, Portland cement plants, incinerators, nitric acid plants, and sulfuric acid plants. The proposed standards, applicable to sources the construction or modification of which was initiated after August 17, 1971, included emission limits for one or more of the following pollutants: sulfur dioxide, nitrogen oxides, and sulfuric acid mist (for each source category). The proposed standards and applicable test methods were published in the Federal Register on September 24, 1971.

The provisions regarding requests for EPA plan review and determination of construction or modification have been revised as appropriate to reflect the change in the reference method. The adjusted standards as proposed in the original rulemaking will be obliged to provide evidence that the alternative method is equivalent to the reference method. Emission limits have been adjusted based only on material collected in the initial startup as the beginning of routine operation. Owners or operators will be required to notify the Administrator at least 10 days prior to compliance testing to permit emission limits to be within 5 percent of the average during the specified time period.

The definitions of modification, as it relates to the steam generators, were clarified, and directives for the monitoring equipment, pertinent analyses, feed rates, production rates, etc., for 2 years and to make such information available on request to the Administrator. Owners or operators will be required to summarize the recorded data daily and to convert recorded data into the applicable units of the standard.

The revised provisions were added to the steam generator standards to cover those instances where mixed fuels are burned. The revised provisions allow operators the option of burning low-sulfur fuels (probably natural gas) as a means of compliance. The provisions were added to the steam generator standards to cover those instances where mixed fuels are burned. The revised provisions allow operators the option of burning low-sulfur fuels (probably natural gas) as a means of compliance.
RULES AND REGULATIONS

Section 60.38 Standard for sulfur dioxide.
Section 60.39 Standard for acid mist.
Section 60.40 Emission monitoring.
Section 60.41 Test methods and procedures.

APPENDIX-TEST METHODS

Method 1-Sample and velocity traverses for stationary sources.
Method 2-Determination of stack gas velocity and volumetric flow rate (Type S pitot tube).
Method 3-Gas analysis for carbon dioxide, excess air, and dry molecular weight.
Method 4-Determination of moisture in stack gases.
Method 5-Determination of particulate emissions from stationary sources.
Method 6-Determination of sulfur dioxide emissions from stationary sources.
Method 7-Determination of nitrogen oxides emissions from stationary sources.
Method 8-Determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources.

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

Subpart A-General Provisions

§ 60.1 Applicability.

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

Subpart B-Standards of Performance for Fossil Fuel-Fired Steam Generators

§ 60.40 Applicability and designation of affected facility.
§ 60.41 Definitions.
§ 60.42 Standard for particulate matter.
§ 60.43 Standard for sulfur dioxide.
§ 60.44 Standard for sulfuric acid.
§ 60.45 Emission and fuel monitoring.
§ 60.46 Test methods and procedures.

Subpart C-Standards of Performance for Portland Cement Plants

§ 60.50 Applicability and designation of affected facility.
§ 60.51 Definitions.
§ 60.52 Standard for particulate matter.
§ 60.53 Monitoring of operations.
§ 60.54 Test methods and procedures.

Subpart D-Standards of Performance for Portland Cement Plants

§ 60.60 Applicability and designation of affected facility.
§ 60.61 Definitions.
§ 60.62 Standard for particulate matter.
§ 60.63 Monitoring of operations.
§ 60.64 Test methods and procedures.

Subpart E-Standards of Performance for Nitric Acid Plants

§ 60.70 Applicability and designation of affected facility.
§ 60.71 Definitions.
§ 60.72 Standard for nitrogen oxides.
§ 60.73 Emission monitoring.
§ 60.74 Test methods and procedures.

Subpart F-Standards of Performance for Sulfuric Acid Plants

§ 60.80 Applicability and designation of affected facility.
§ 60.81 Definitions.

(1) Routine maintenance, repair, and replacement shall not be considered physical changes, and
(2) The following shall not be considered a change in the method of operation:
(a) An increase in the production rate, if such increase does not exceed the operating design capacity of the affected facility;
(b) An increase in hours of operation; (c) Use of an alternative fuel or raw material if, prior to the date any standard under this part becomes applicable to such facility, as provided by § 60.1, the affected facility is designed to accommodate such alternative use.

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

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

(3) “Exhaust pollutants” means all oxides of nitrogen, except nitrous oxide, as measured by test methods set forth in this part.

(4) “Standard of normal conditions” means 70° Fahrenheit (21.1° centigrade) and 29.92 in. Hg (760 mm. Hg).

(5) “Proportional sampling” means sampling at a rate that produces a constant ratio of sampling rate to stack gas flow rate.

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

(7) “Startup” means the setting in operation of an affected facility for any purpose.

§ 60.3 Abbreviations.

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

B.t.u.—British thermal units.
cal.—calorie(s).
c.f.m.—cubic feet per minute.
C.O.—carbon dioxide.
c.g.—gram(s).
c.mm.—millimeter(s).
f.—foot(s).
f.t.u.—foot tons.
f.c.u.—foot cubic units.
f.m.—fathom(s).
f.t.—ton(s).
f.t.u.—foot tons.
Hg.—mercury.
In.—inch(es).
K.—kilogram(s).
Lb.—pound(s).
ml.—milliliter(s).
M.—mile(s).
Mr.—mile.
N.—number.
O.—percent.
N.O.—nitric oxide.
N.N.—nitrogen dioxide.
N.T.—normal cubic meter.
N.T.F.—normal ton.
O.—percent.
O.N.—nitric oxide.
O.N.—nitrogen dioxide.
N.T.—normal cubic meter.
N.T.F.—normal ton.
24777

FEDERAL REGISTER, VOL. 36, NO. 247—THURSDAY, DECEMBER 23, 1971

Dated: December 17, 1971.

WILLIAM D. RUCKERSHAUS,
Administrator,
Environmental Protection Agency.
ft.—cubic feet.
ft. 2.—square feet.
min.—minute(s).
h.—hour(s).
§ 60.4 Address.
All applications, requests, submissions, and reports under this part shall be sub-
mitted in triplicate and addressed to the
Environmental Protection Agency, Office of
General Enforcement, Waterside Mall
SW., Washington, DC 20460.
§ 60.5 Determination of construction or
modification.
When requested to do so by an owner
or operator, the Administrator will make a
determination of whether actions taken
or intended to be taken by such owner or
operator constitute construction or modi-
fication or the commencement thereof
within the meaning of this part.
§ 60.6 Review of plans.
(a) When requested to do so by an
owner or operator, the Administrator will
review plans for construction or modi-
fication for the purpose of providing
technical advice to the owner or operator.
(b) (1) A separate request shall be
submitted for each affected facility.
(2) Each request shall (i) identify the
location of such affected facility, and (ii)
be accompanied by technical information
describing the proposed nature, size,
design, and method of operation of such
facility, including information on any
equipment to be used for measurement or
control of emissions.
(c) Neither a request for plans review
nor advice furnished by the Administra-
tor in response to such request shall (1)
relieve an owner or operator of legal
responsibility for compliance with any
provision of this part or of any applicable
State or local requirements, or (2) prevent
the Administrator from implementing or
enforcing any provision of this part or
taking any other action authorized by the
Act.
§ 60.7 Notification and record keeping.
(a) Any owner or operator subject to
the provisions of this part shall furnish
the Administrator written notification as
follows:
(1) A notification of the anticipated
date of initial startup of an affected
facility not more than 90 days or less
than 30 days prior to such date.
(2) A notification of the actual date
of initial startup of an affected facility
within 15 days after such date.
(b) Any owner or operator subject to
the provisions of this part shall maintain
for a period of 2 years a record of the
occurrence and duration of any startup,
shutdown, or malfunction in operation of
any affected facility.
§ 60.8 Performance tests.
(a) Within 60 days after achieving
the maximum production rate at which the
affected facility will be operated, but not
later than 180 days after initial startup
of such facility and at such other times
as may be required by the Administrator
under section 114 of the Act, the owner
or operator of such facility shall conduct
performance test(s) and furnish the Ad-
ministrator a written report of the results
of such performance test(s).
(b) Performance tests shall be con-
ducted and results reported in accord-
ance with the test method set forth in
this part or equivalent methods approved
by the Administrator; or where the Ad-
ministrator determines that emissions
from the affected facility are not sus-
ceptible of being measured by such
methods, the Administrator shall pre-
scribe alternative test procedures for
determining compliance with the
requirements of this part.
(c) The owner or operator shall permit
the Administrator to conduct performance
tests at any reasonable time, shall cause
the affected facility to be operated
for purposes of such tests under such
conditions as the Administrator shall
specify based on the performance of the
affected facility, and shall make available to the Administrator such records as may be necessary to
determine such performance.
(d) The owner or operator of an
affected facility shall provide the Ad-
ministrator 10 days prior notice of the
performance test to afford the Adminis-
trator the opportunity to have an
observer present.
(e) The owner or operator of an
affected facility shall provide, or cause to
be provided, performance testing facil-
ities as follows:
(1) Sampling ports adequate for test
methods applicable to such facility.
(2) Safe sampling platform(s).
(3) Safe access to sampling plat-
form(s).
(4) Utilities for sampling and testing
equipment.
(f) Each performance test shall con-
sist of three repetitions of the applicable
test procedure for determining compliance with an applicable
standard of performance, the average of
results of all repetitions shall apply.
§ 60.9 Availability of information.
(a) Emission data provided to, or
otherwise obtained by, the Administra-
tor in accordance with the provisions of
this part shall be available to the public, except that (1) upon a showing satisfactory
to the Administrator by any person that
such records, reports, or information, or
particular part thereof (other than
emission data), if made public, would
divulge methods or processes entitled to
protection as trade secrets or other
information from such materials.
(b) “Particulate matter” means any
finely divided liquid or solid material,
other than uncombined water, as meas-
ured by Method 5.
§ 60.10 Standard for particulate matter.
On and after the date on which the
performance test required to be con-
ducted by § 60.8 is initiated no owner
or operator subject to the provisions of
this part shall discharge or cause the
discharge into the atmosphere of par-
ticulate matter which is:
(a) In excess of 0.18 g./million
B.t.u. heat input (0.18 g./million cal.)
maximum 3-hour average for the
primary purposes of producing steam
by heat transfer.
(b) Greater than 20 percent opacity,
except that 40 percent opacity shall be
permissible for not more than 2 minutes
in any hour.
Where the presence of uncombined
water is the only reason for failure
unto the requirements of paragraph (b)
of this section such failure shall not be a violation of this section.
§ 60.43 Standard for sulfur dioxide.

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

(a) 0.08 lb. per million B.t.u. heat input (1.4 g. per million cal.), maximum 2-hour average, when liquid fossil fuel is burned.
(b) 1.2 lbs. per million B.t.u. heat input (2.2 g. per million cal.), maximum 2-hour average, when solid fossil fuel is burned.

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

\[ y(x.08)+z(1.2) \]

where:

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

§ 60.44 Standard for nitrogen oxides.

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

(a) 0.30 lb. per million B.t.u. heat input (0.56 g. per million cal.), maximum 2-hour average, expressed as NO\(_2\), when gaseous fossil fuel is burned.
(b) 0.30 lb. per million B.t.u. heat input (0.54 g. per million cal.), maximum 2-hour average, expressed as NO\(_2\), when liquid fossil fuel is burned.
(c) 0.70 lb. per million B.t.u. heat input (1.26 g. per million cal.), maximum 2-hour average, expressed as NO\(_2\), when solid fossil fuel (except lignite) is burned.
(d) When different fossil fuels are burned simultaneously in any combination the applicable standard shall be determined by proration. Compliance shall be determined by using the following formula:

\[ x(0.20)+y(0.30)+z(0.70) \]

where:

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

§ 60.45 Emission and fuel monitoring.

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

1. A photoelectric or other type smoke detector and recorder, except where gaseous fuel is the only fuel burned.
2. An instrument for continuously monitoring and recording sulfur dioxide emissions, except where gaseous fuel is the only fuel burned, or where compliance is achieved by the use of low sulfur fuels and representative sulfur analysis of fuels are conducted daily in accordance with paragraph (c) or (d) of this section.
3. An instrument for continuously monitoring and recording emissions of nitrogen oxides.

(b) Instruments and sampling systems installed and used pursuant to this section shall be capable of monitoring emission levels within ±20 percent with a confidence level of 95 percent and shall be calibrated in accordance with the method prescribed by the manufacturer of such instruments; instruments shall be subjected to manufacturers recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable procedure shall be used to determine compliance with the provisions of this section if the cross sectional area is less than 50 ft\(^2\) or at a point no closer to the walls than 3 feet if the cross sectional area is greater than 50 ft\(^2\). The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 20 minutes and minimum sampling volume shall be 60 ft\(^3\), corrected to standard conditions on a dry basis.

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

\[ x(0.20)+y(0.30)+z(0.70) \]

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

\[ x(0.20)+y(0.30)+z(0.70) \]

§ 60.46 Test methods and procedures.

(a) The provisions of this section are applicable to performance tests for determination of performance of the unit operated and while fuels or combinations of fuels are being burned and under such other conditions as the Administrator shall specify based on representation of performance of the affected facility.

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

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

1. For each repetition, the average concentration of particulate matter shall be determined by using Method 5. The sampling site shall be the same as for determining volumetric flow rate. The sampling point shall be selected by the method called the concave section if the cross sectional area is less than 50 ft\(^2\) or at a point no closer to the walls than 3 feet if the cross sectional area is greater than 50 ft\(^2\). The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 30 minutes and minimum sampling volume shall be 0.75 ft\(^3\), corrected to standard conditions. Two samples shall constitute one repetition and shall be taken at 1-hour intervals.

2. For each repetition the SO\(_2\) concentration shall be determined by using Method 6. The sampling site shall be the same as for determining volumetric flow rate. The sampling point shall be selected by the method called the concave section if the cross sectional area is less than 10 ft\(^2\) or at a point no closer to the walls than 3 feet if the cross sectional area is greater than 10 ft\(^2\). The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The sampling time shall be 2 hours and minimum sampling volume shall be 0.75 ft\(^3\), corrected to standard conditions. Two samples shall constitute one repetition and shall be taken at 1-hour intervals.

3. For each repetition the NO\(_2\) concentration shall be determined by using Method 7. The sampling site and point shall be the same as for SO\(_2\). The sampling time shall be 2 hours and four samples shall be taken at 30-minute intervals.

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

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

6. For each repetition, emissions, expressed in lb./hr. 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, x where, Q, = volumetric flow rate of the total effluent in ft\(^3\)/hr. at standard conditions, dry basis, as determined in accordance with paragraph (c) or (d) of this section.

7. Particulate matter, c=particulate matter concentration in lb./ft\(^3\), is determined in accordance with paragraph (c) or (d) of this section, corrected to standard conditions, dry basis.
(2) For $SO_2$, $c_2$-$SO_2$ concentration in lb./ft.$^3$, as determined in accordance with paragraph (c) (2) of this section, corrected to standard conditions, dry basis.

(3) For $NO_x$, $c_2$-$NO_x$ concentration in lb./ft.$^3$, as determined in accordance with paragraph (c) (3) of this section, corrected to standard conditions, dry basis.

Subpart E—Standards of Performance for Incinerators

§ 60.50 Applicability and designation of affected facility.

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

§ 60.51 Definitions.

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

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

(b) "Solid waste" means refuse, more than 70 percent of which is municipal type waste consisting of a mixture of paper, wood, yard wastes, food wastes, plastics, leather, rubber, and other combustible, and noncombustible materials such as glass and rock.

(c) "Day" means 24 hours.

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

§ 60.52 Standard for particulate matter.

On and after the date on which the performance test required to be conducted by § 60.8 is initiated, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter which is in excess of 0.08 g./N$^1$V corrected to standard conditions, dry basis.

§ 60.53 Monitoring of operations.

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

Subpart F—Standards of Performance for Portland Cement Plants

§ 60.60 Applicability and designation of affected facility.

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

§ 60.61 Definitions.

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

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

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

§ 60.62 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is initiated, no owner or operator subject to the provisions of this part shall discharge or cause the discharge into the atmosphere of particulate matter which is greater than or equal to 0.10 lb./ton of feed to the kiln.

(b) The volumetric flow rate of the total effluent shall be determined by using Method 3 and normalizing according to Method 1. Gas analysis shall be performed using the integrated sample technique of Method 3, and moisture content shall be determined by the condenser technique of Method 5. If a wet scrubber is used, the gas analysis sample shall reflect flue gas conditions after the scrubber, allowing for the effect of carbon dioxide absorption.

(d) For each repetition, particulate matter emissions, expressed in g./s cf, shall be determined in accordance with paragraph (c) (1) of this section corrected to 12 percent CO$\text{2}$, dry basis.

§ 60.63 Monitoring of operations.

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

§ 60.64 Test methods and procedures.

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

(b) All performance tests shall be conducted while the affected facility is operating at or above the maximum allowable emission rates, as determined using the integrated sample technique of Method 3, and moisture content shall be determined by the condenser technique of Method 5.

(d) Total kiln feed (except fuel) expressed in tons per hour on a dry basis, shall be determined during each 2-hour 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 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. = $Q \times Q_x$,
Instruments shall be subjected to manufacturer(s) of such instrument, the recording emissions of nitrogen oxides. The provisions of this subpart are applicable to each nitric acid production unit, which is the affected facility.

§ 60.70 Applicability and designation of affected facility.

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

§ 60.71 Definitions.

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

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

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

§ 60.72 Standard for nitrogen oxides.

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

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

(b) 10 percent opacity or greater.

§ 60.73 Emission monitoring.

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

(b) The instrument and sampling system installed and used pursuant to this section shall be capable of monitoring emission levels within ±50 percent with a confidence level of 85 percent and shall be calibrated in accordance with the method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subjected to manufacturers recommended zero adjustment and calibration procedures at least once per 24-hour operating period unless the manufacturer(s) specifies or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method.

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

(d) The owner or operator of any nitric acid production unit subject to the provisions of this part shall maintain a file of all measurements required by this subpart. Appropriate measurements shall be reduced to the units of the standard daily and summarized monthly. The record of any such measurement shall be confirmed by the manufacturer(s) of preventing emissions of nitrogen oxides from nitric acid production units.

§ 60.74 Test methods and procedures.

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

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

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

(1) For each repetition the NOx concentration shall be determined by using Method 7. The sampling site shall be selected according to Method 1 and the sampling point shall be the centroid of the stack or duct. The sampling time shall be 3 hours and four samples shall be taken at 30-minute intervals.

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

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

(e) For each repetition, nitrogen oxides emissions, expressed in lb./ton of 100 percent nitric acid, shall be determined by dividing the emission rate in lb./hr., by the acid produced. The emission rate shall be determined by the equation, lb./hr. = Qe × NOx, where Qe = volumetric flow rate of the effluent in ft3/hr., at standard conditions, dry basis, as determined in accordance with paragraph (c) (2) of this section, and NOx concentration in lb./ft3, as determined in accordance with paragraph (d) (1) of this section, corrected to standard conditions, dry basis.

Subpart H—Standards for Performance of Sulfuric Acid Plants

§ 60.80 Applicability and designation of affected facility.

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

§ 60.81 Definitions.

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

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

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

§ 60.82 Standard for sulfur dioxide.

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

§ 60.83 Standard for acid mist.

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

(a) In excess of 0.15 lb. per ton of acid produced (0.076 kg. per metric ton), maximum 2-hour average, expressed as H2SO4.

(b) 10 percent opacity or greater.

§ 60.84 Emission monitoring.

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

(b) The instrument and sampling system installed and used pursuant to this section shall be capable of monitoring emission levels within ±20 percent with a confidence level of 85 percent and shall be calibrated in accordance with the
method(s) prescribed by the manufacturer(s) of such instrument, the instrument shall be subject to manufacturers recommended zero adjustment calibration procedures at least once per 24-hour operating period unless the manufacturer(s) specified or recommends calibration at shorter intervals, in which case such specifications or recommendations shall be followed. The applicable method specified in the appendix of this part shall be the reference method.

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

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

§ 60.85 Test methods and procedures.

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

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

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

(1) For each repetition the acid mist and SO₂ concentrations shall be determined by using Method 8 and traversing according to Method 1. The minimum sampling time shall be 2 hours, and minimum sampling volume shall be 40 ft³ corrected to standard conditions.

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

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

(e) For each repetition acid mist and sulfur dioxide emissions, expressed in lb./ton of 100 percent sulfuric acid shall be determined by dividing the emission rate in lb./hr. by the acid produced. The emission rate shall be determined by the equation, lb./hr. = Q₂ × C₂, where Q₂ = volumetric flow rate of the effluent in ft³/hr. at standard conditions, dry basis as determined in accordance with paragraph (c) (2) of this section, and C₂ = acid mist and SO₂ concentrations in lb./ft³ as determined in accordance with paragraph (c) (1) of this section, corrected to standard conditions, dry basis.

APPENDIX—Test Methods

METHOD 1—SAMPLE AND VELOCITY TRAVERSING FOR STATIONARY SOURCES

1. Principles and Applicability.

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

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

2. Procedure.

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

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

\[ \text{equivalent diameter} = \frac{\text{length} \times \text{width}}{\text{length} + \text{width}} \]  

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

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

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

2.2 Cross-sectional layout and location of traverse points.

2.2.1 For circular stacks locate the traverse points on at least two diameters according to Figure 1–1 and Table 1–1. The traverse axes shall divide the stack cross section into equal parts.
**Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.**

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

---

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

<table>
<thead>
<tr>
<th>Traverse point number on a diameter</th>
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<th>4</th>
<th>6</th>
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<th>10</th>
<th>12</th>
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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.

METHOD 2-DETEE=ATION

Volume, Dust and Mist Content of Gases, Source Testing Manual, Air Pollution Control

1. Principle and applicability.
1.1 Principle. Stack gas velocity is determined from the gas density and from measurement of the velocity head using the pitot tube.
1.2 Applicability. This method is applicable only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. Apparatus.
2.1 Pitot tube-Type S (Figure 2-1), or equivalent, with a coefficient within ±5% over the working range.
2.2 Differential pressure gauge—Inclined manometer, or equivalent, to measure velocity head to within 10% of the minimum value.
2.3 Temperature gauge—Thermocouple or equivalent attached to the pitot tube to measure stack temperature to within 1.5% of the minimum absolute stack temperature.
2.4 Pressure gauge—Mercury-filled U-tube manometer, or equivalent, to measure stack pressure to within 0.1 in. Hg.
2.5 Barometer—To measure atmospheric pressure to within 0.1 in. Hg.
2.6 Gas analyzer—To analyze gas composition for determining molecular weight.
2.7 Pitot tube—Standard type, to calibrate Type S pitot tube.

3. Procedure.
3.1 Set up the apparatus as shown in Figure 2-1. Make sure all connections are tight and leak free. Measure the velocity head and temperature at the traverse points specified by Method 1.
3.2 Measure the static pressure in the stack.
3.3 Determine the stack gas molecular weight by gas analysis and appropriate calculations as indicated in Method 3.

4.1 To calibrate the pitot tube, measure the velocity head at one point in a flowing gas stream with both a Type S pitot tube and a standard type pitot tube with known efficiency. Calibration should be done in the laboratory and the velocity of the flowing gas stream should be varied over the normal working range. It is recommended that the calibration be repeated after use at each field site.
4.2 Calculate the pitot tube coefficient using equation 2-1.

\[ C_{pitot} = \frac{\Delta P_{pitot}}{P_{mean}} \] (Equation 2-1)

where:
- \( C_{pitot} \) = Pitot tube coefficient of Type S pitot tube.
- \( \Delta P_{pitot} \) = Velocity head measured by standard type pitot tube.
- \( P_{mean} \) = Velocity head measured by Type S pitot tube.

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

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

\[ (V_{avg})_{stack} = \frac{C_{pitot} \Delta P_{mean}}{M_{gas}} \] (Equation 2-2)

where:
- \( (V_{avg})_{stack} \) = Stack gas velocity, feet per second (f.p.s.).
- \( C_{pitot} \) = Pitot tube coefficient, dimensionless.
- \( \Delta P_{mean} \) = Average absolute stack gas temperature, lb./hr.
- \( M_{gas} \) = Molecular weight of stack gas (from Method 3).
- \( M_{gas} \) = Proportion by volume of water vapor in the gas stream (from Method 4).

Figure 2-2 shows the sampling record chart for velocity traverse data. Use the average in the last two columns of Figure 2-2 to determine the average stack gas velocity from Equation 2-2.

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

\[ Q_{stack} = 3600 \left( 1 - R_{dry} \right) V_{avg} \] (Equation 2-3)

where:
- \( Q_{stack} \) = Volumetric flow rate, dry basis, standard conditions, ft³/hr.
- \( V_{avg} \) = Average cross-sectional area of stack, ft².
- \( R_{dry} \) = Relative humidity at standard conditions, 50%.
- \( P_{-stack} \) = Absolute pressure at standard conditions, 29.92 inches Hg.
6. References.


<table>
<thead>
<tr>
<th>Plant</th>
<th>Date</th>
</tr>
</thead>
<tbody>
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</table>

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Stack Diameter, in.</th>
<th>Barometric Pressure, in. Hg.</th>
<th>Static Pressure in Stack (p_s), in. Hg.</th>
<th>Operators</th>
</tr>
</thead>
<tbody>
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<table>
<thead>
<tr>
<th>Traverse point number</th>
<th>Velocity head, in. H_2O</th>
<th>\sqrt{p}</th>
<th>Stack Temperature (T_s), °F</th>
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</thead>
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</table>

Average: \[ V_n \]

Figure 2-2. Velocity traverse data.
2.2 Integrated sample (Figure 3-2).
2.2.1 Probe—Stainless steel or Pyrex\* glass, equipped with a filter to remove particulate matter.
2.2.2 Air-cooled condenser or equivalent—To remove any excess moisture.
2.2.3 Needle valve—To adjust flow rate.
2.2.4 Pump—Leak-free, diaphragm type, or equivalent, to pull gas.
2.2.5 Flow meter—To measure a flow range from 0 to 0.035 cfm.
2.2.6 Flexible bag—Tedlar\* or equivalent, with a capacity of 2 to 3 cu. ft. Leak test the bag in the laboratory before using.
2.2.7 Pilot tube—Type S, or equivalent, attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.
2.3 Analysis.
2.3.1 Orsat analyzer, or equivalent.

3. Procedure.
3.1 Grab sampling.
3.1.1 Set up the equipment as shown in Figure 3-1, making sure all connections are leak-free. Place the probe in the stack at a sampling point and purge the sampling line.
3.1.2 Draw sample into the analyzer.
3.2 Integrated sampling.
3.2.1 Evacuate the flexible bag. Set up the equipment as shown in Figure 3-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are tight and that there are no leaks.
3.2.2 Sample at a rate proportional to the stack velocity.
3.3 Analysis.
3.3.1 Determine the CO, O\(_2\), and CO\(_2\) concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than ten passes are necessary, replace the absorbing solution.
3.3.2 For grab sampling, repeat the sampling and analysis until three consecutive samples vary no more than 0.5 percent by volume for each component being analyzed.
3.3.3 For integrated sampling, repeat the analysis of the sample until three consecutive analyses vary no more than 0.5 percent by volume for each component being analyzed.
4.1 Carbon dioxide. Average the three consecutive runs and report the result to the nearest 0.1% CO\(_2\).
4.2 Excess air. Use Equation 3-1 to calculate excess air, and average the runs. Report the result to the nearest 0.1% excess air.

\[
\% EA = \frac{(\% O_2) - 0.5(\% CO)}{\text{poth}} \times 100
\]

where:
\% EA = Percent excess air.
\% O\(_2\) = Percent oxygen by volume, dry basis.
\% N\(_2\) = 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.

\[
M_d = 0.44(\% CO) + 0.53(\% O_2) + 0.08(\% N_2 + \% CO)
\]

where:
M\(_d\) = Dry molecular weight, lb./lb-mole.
\% CO = Percent carbon dioxide by volume, dry basis.
\% O\(_2\) = Percent oxygen by volume, dry basis.
\% N\(_2\) = Percent nitrogen by volume, dry basis.
0.44 = Molecular weight of carbon dioxide divided by 100.
0.53 = Molecular weight of oxygen divided by 100.
0.08 = Molecular weight of nitrogen and CO divided by 100.
RULES AND REGULATIONS

5. References.

METHOD 4-DETERMINATION OF MOISTURE IN STACK GASES

1. Principle and applicability.
1.1 Principle. Moisture is removed from the gas stream, condensed, and determined volumetrically.
1.2 Applicability. This method is applicable for the determination of moisture in stack gas only when specified by test procedures for determining compliance with New Source Performance Standards. This method may not apply when liquid droplets are present in the gas stream and the moisture is subsequently added in the determination of stack gas molecular weight.

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

2. Apparatus.
2.1 Probe-Slotted steel or Pyrex® glass sufficiently heated to prevent condensation.

3. Procedure.
3.1 Place exactly 5 ml distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-1. Leak check by plugging the inlets to the first impinger and drawing a vacuum. Insure that flow through the dry gas meter is less than 1% of the sampling rate.
3.2 Connect the probe and sample at a constant rate of 0.054 cfm, or at a rate proportional to the stack gas velocity. Continue sampling until the dry gas meter registers 1 cubic foot or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-2.
3.3 After collecting the sample, measure the volume increase to the nearest 0.5 ml.

4.1 Volume of water vapor collected.

\[ V_{vc} = \frac{(V_f - V_i) p_{atm} R T_{atm}}{p_{atm} M_w} \]

where:
- \( V_{vc} \) = Volume of water vapor collected (standard conditions), ml
- \( V_f \) = Final volume of impinger contents, ml
- \( V_i \) = Initial volume of impinger contents, ml
- \( R \) = Ideal gas constant, 21.83 inches
- \( p_{atm} \) = Absolute pressure at standard conditions, 520° R.
- \( T_{atm} \) = Absolute temperature at standard conditions, 520° R.
- \( M_w \) = Molecular weight of water, 18 lb./

Figure 4-1. Moisture sampling train.

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Figure 4-2. Field moisture determination.

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<th>CLOCK TIME</th>
<th>GAS VOLUME THROUGH IMPINGERS, ft³</th>
<th>ROTAMETER SETTING, ft³/min</th>
<th>METER TEMPERATURE, °F</th>
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</table>
RULERS AND REGULATIONS

2.1.1 Filter Holder—Pyrex† glass with heating system capable of maintaining minimum temperature of 250° F.

2.1.2 Impingers/Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 0.5-inch OD glass tube extending to half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

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

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

2.2 Sample recovery.

2.3 Probe brush—At least 16 inches long as possible.

2.3.1 Probe—Glass with a 1.5 inch OD glass tube extending to half inch from the bottom of the flask.

2.3.3 Desiccant—Drierite1, indicating.

3.1 Sampling.

3.2 Sample Recovery.

3.3 Analyzing.

3.3.1 Water.

3.3.2 Gas weighing dishes.

3.3.3 Desiccator.

3.3.4 Analytical balance—To measure to ±0.1 mg.

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

3.3.6 Reagents.

3.3.7 Sampling.

3.3.8 Sampling accessories.

3.4 Standard.

3.5 Water.

3.5.1 Cracked ice.

3.5.2 Sample recovery.

3.5.3 Water.

3.5.4 Glass wash bottles—Two.

3.5.5 Glass sample storage containers.

3.5.6 Graduated cylinders—250 ml.

3.5.7 Analysis.

3.5.8 Analytical balance—To measure to ±0.1 mg.

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

3.5.10 Reagents.

3.5.11 Sampling.

3.5.12 Samplng accessories.

3.5.13 Water.

3.5.14 Glass weighing dishes.

3.5.15 Desiccator.

3.5.16 Analytical balance—To measure to ±0.1 mg.

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

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4.3 Sample recovery. Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetonite used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in the example sheet shown in Figure 4.3. Analysis. Record the data required on the example sheet shown in Figure 4.4. Handle each sample container as follows:

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

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

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

4.3 Analysis. Record the data required on the example sheet shown in Figure 4.4. Handle each sample container as follows:

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

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

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

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

6. Calculations.

6.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 4.5).

6.2 Correction of sample volume measured by the dry gas meter to standard conditions (10°F, 29.92 inches Hg) by using Equation 5-1:

\[ V_{\text{astd}} = V_a \left( \frac{T_{\text{std}}}{T_m} \right) \left( \frac{P_{\text{bar}} + \frac{15}{T_m}}{15} \right) \]

where:

- \( V_a \) = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.
- \( T_m \) = Absolute temperature at standard conditions, 630°F.
- \( P_{\text{bar}} \) = Barometric pressure at the orifice meter, inches Hg.
- \( \Delta P \) = Average pressure drop across the orifice meter, inches Hg.
- \( M_a \) = Molecular weight of air, 28.964.
- \( M_h \) = Molecular weight of water, 18.015.
- \( R \) = Ideal gas constant, 154.85 cm Hg · cm · mol · °K.
- \( P_{\text{bar}} \) = Absolute pressure at standard conditions, 29.92 inches Hg.
- \( T_m \) = Absolute temperature at standard conditions, 630°F.

6.3 Volume of water vapor.

\[ V_{\text{wstd}} = \frac{V_a \left( \frac{P_{\text{bar}} + \frac{15}{T_m}}{15} \right)}{0.0474 \text{ cu. ft.}} \]

where:

- \( V_{\text{wstd}} \) = Volume of water vapor in the gas sample (standard conditions), cu. ft.
- \( V_a \) = Total volume of gas collected in impingers and silica gel (see Figure 4.3), cu. ft.
- \( P_{\text{bar}} \) = Barometric pressure at the orifice meter, inches Hg.
- \( T_m \) = Absolute temperature at standard conditions, 630°F.

6.4 Moisture content.

\[ B_m = \frac{V_{\text{wstd}}}{V_a} \]

where:

- \( B_m \) = Percentage by volume of water vapor in the gas stream, percent.
- \( V_{\text{wstd}} \) = Volume of water vapor in the gas sample (standard conditions), cu. ft.
- \( V_a \) = Total volume of gas collected in impingers and silica gel (see Figure 4.3), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 4.6).

6.6 Concentration.

6.6.1 Concentration in gr./cu. ft.

\[ c_a = \left( \frac{0.0154 \text{ gr.}}{\text{cu. ft.}} \right) \frac{M_p}{V_{\text{std}}} \]

where:

- \( c_a \) = Concentration of particulate matter in stack gas, gr./cu. ft.
- \( M_p \) = Total weight of particulate matter collected, gr.
- \( V_{\text{std}} \) = Volume of gas sample through dry gas meter (standard conditions), cu. ft.
where:

- Percent of isokinetic sampling.
- Total volume of liquid collected in impingers and silica gel (see Figs. 5-3). mg
- Density of water, 1 g/ml.
- Ideal gas constant, 21.23 inches Hg cu. ft./lb.-mol.
- Molecular weight of water, 18 lb./lb.-mol.
- Volume of gas sample through dry gas meter (meter conditioned), cu. ft.
- Absolute average dry gas meter temperature (see Figs. 5-3).°R.
- Total sampling time, min.
- Total sampling time, sec. (see Fig. 5-3), °R.
- Absolute average stack gas temperature (see Figs. 5-3).°R.
- Total sampling time, sec.
- Absolute average stack gas temperature (see Fig. 5-3).°R.
- Cross-sectional area of stack, cu. ft.
- Cross-sectional area of stack, cu. ft.

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

If 0.05 ≤ I ≤ 110%, the results are acceptable, otherwise, reject the results and repeat the test.

7. Reference.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, APTD-030.


METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

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

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

2. Apparatus.

2.1 Sampling. See Figure 5-1.

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

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

2.1.3 Glass wool.

2.1.4 Midget impingers.—Three.

2.1.5 Drying tube.—Packed with 0 to 10 mesh indicating-type silica gel, or equivalent, to dry the sample.

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

2.1.7 Pump.—Electric, vacuum type.

2.1.8 Dry gas meter.—Rotometer or equivalent, to measure a 0-10 c.f.m. flow range.

2.1.9 Dry gas meter.—Sufficiently accurate to measure the sample volume within 1%. 2.1.10 Pilot tube.—Type B, or equivalent.

1 Trade names.
2.5.1 Pipettes—Transfer type, 5 mL and 10 mL, glass (0.1 mL divisions) and 25 mL, glass (0.2 mL divisions).

2.5.2 Volumetric flasks—50 mL, 100 mL, and 1000 mL.

2.5.3 burettes—5 mL and 10 mL.

2.5.4 Erlenmeyer flask—100 mL.

3. Reagents.

3.1 Sampling.

3.1.1 Water—Deionized distilled.

3.1.2 Isopropanol, 90%—Mix 60 mL of isopropanol with 20 mL of distilled water.

3.1.3 Hydrogen peroxide, 35%—Dilute 100 mL of 30% hydrogen peroxide to 1 liter with distilled water. Prepare fresh daily.

3.2 Sample recovery.

3.2.1 Water—Deionized distilled.

3.2.2 Isopropanol, 90%.

3.3 Analysis.

3.3.1 Water—Deionized distilled.

3.3.2 Propylene.

3.3.3 Thorin indicator—1-(p-tosylphenoxy)2-naphthyl-3,6-disulfo acid, disodium salt (or equivalent). Dissolve 0.2 g in 100 mL distilled water.

3.4 Barium perchlorate (0.01 N)—Dissolve 1.05 g of barium perchlorate \([\text{Ba(ClO}_4]_2\) in 200 mL distilled water.

4. Procedure.

4.1 Sample preparation.

4.1.1 Preparation of the collection train. Four 15 mL of 35% isopropanol into the midget bubbler and 15 mL of 35% hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Load the collection train at the sampling site by plugging the probe inlet and pushing a 10 inches Hg vacuum. A leakage rate not in excess of 1% of the sampling rate is acceptable. Carefully release the probe inlet plug and turn off the pump. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or lower.

4.1.2 Sample collection. Adjust the sample flow rate proportional to the stack gas velocity. Take readings at least every five minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the tip of the probe at the first sampling point and start the pump. Sample proportionally throughout the run. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 10 minutes.

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

4.3 Sample analysis. Transfer the contents of the storage container to a 60 mL volumetric flask. Dilute to the mark with deionized distilled water. Pipette a 10 mL aliquot of this solution into a 125 mL Erlenmeyer flask. Add 40 mL of isopropanol and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.01 N barium perchlorate. Run a blank with each series of samples.

5. Calibration.

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

5.2 Standardize the barium perchlorate against 5 mL of standard sulfuric acid containing 100 mL of isopropanol.

6. Calculations.

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

\[
V_{\text{std}} = \frac{V_{\text{inlet}}}{T_{\text{inlet}}/T_{\text{std}}} = \frac{V_{\text{inlet}}}{(70 + 469.8) / 298.15}
\]

6.2 Barometric pressure at the calibration point (inches Hg).

6.3 Absolute pressure at standard conditions.

6.4 Average dry gas meter temperature (°R).

6.5 Barometric pressure at the calibration point (inches Hg).

6.6 Absolute pressure at standard conditions.

6.7 Sulfur dioxide concentration.

\[
\text{mg/m}^3 = \frac{(V_{\text{inlet}} - V_{\text{b}}) N V_{\text{std}}}{V_{\text{inlet}}}
\]

7. References.


8. Principle and applicability.

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

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nitrous oxide, are measured colorimetrically using the phenolsulfuric acid (FDS) 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® glass, heated, with filter to remove particulate matter. Heating is unnecessary if the probe remains dry during the sampling period.

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

Trade name.

2.3 Procedure.

2.3.1 Absorbing solution—Add 2.8 ml of concentrated H$_2$SO$_4$ 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.

2.3.2 Sample recovery.

2.3.2.1 Pipette 25 ml of absorbing solution into a glass storage container—Cushioned for shipping.

2.3.3 Drain the contents of each sample and standard (blank).

2.3.4 Spectrophotometer—To measure absorption at 420 nm.

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

2.3.6 Transfer pipette—10 ml with 0.1-inch divisions.

2.3.7 Sample recovery.

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

2.3.9 Temperature—Dial-type thermometer, or equivalent, capable of measuring 4°F to 120°F.

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

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

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

2.3.13 Squeeze bulb—One way.

2.3.14 Sample recovery.

2.3.15 Pipette or dropper.

2.3.16 Glass storage containers—Cushioned for shipping.

2.3.17 Flask valve to its "vent" position and disconnect the flask from the sampling train. Shake the flask for 5 minutes.

2.4 Sample recovery.

2.4.1 Let the flask set for a minimum of 16 hours and then shake the contents for 3 minutes. Connect the flask to a mercury filled U-tube manometer, open the valve from the flask to the manometer, and record the flask pressure and temperature along with the barometric pressure. Transfer the flask contents to a container for shipment or to a 250 ml beaker for analysis. Rinse the flask with two portions of distilled water (approximately 10 ml.) and add rinse water to the sample. For a blank use 25 ml of absorbing solution and the same volume of distilled water as used in rinsing the flask. Prior to shipping or analysis, add sodium hydroxide (13/4) dropwise into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

3. Analysis.

3.1 Absorbing solution—Add 0.025 g of sodium hydroxide (1N) to 25 ml of absorbing solution and the same volume of distilled water.

3.2 Water—Deionized, distilled.

3.3 Phenol—White solid reagent grade.

3.4 Standard solution—Dissolve 0.5485 g of potassium nitrate (KNO$_3$) in distilled water and dilute to 1 liter. For the working standard dilute 10 ml of the resulting solution to 100 ml with distilled water. One ml of the working standard is equivalent to 25 µg nitrogen dioxide.

4. Procedure.

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a glass storage container and 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 a least 3 inches Hg absolute pressure. Turn the pump valve to its "vent" position and turn off the pump. Check the manometer for any drop in the measurement in the mercury level. If there is a visible change over the span of one minute, discard the sample.

4.2 Sample recovery.

4.2.1 Drain the contents of each sample and standard (blank).

4.3 Analysis.

4.3.1 Neutralize the sample contents to a container for shipment or to a 250 ml beaker for analysis. Rinse the flask with two portions of distilled water (approximately 10 ml.) and add rinse water to the sample. For a blank use 25 ml of absorbing solution and the same volume of distilled water as used in rinsing the flask. Prior to shipping or analysis, add sodium hydroxide (13/4) dropwise into both the sample and the blank until alkaline to litmus paper. Transfer the solution to a 100 ml beaker and wash the beaker three times with 4 to 5 ml portions of distilled water. Evacuate the solution to dryness on a steam bath and then cool. Add 2 ml phenolsulfuric acid solution to the dried residue and triturate thoroughly with a glass rod. Make sure the solution contains all the residue. Add 1 ml distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml distilled water, mix well by stirring, and add concentrated ammonium hydroxide dropwise with constant stirring until alkaline to litmus paper. Transfer the solution to a 100 ml beaker and neutralize the sample and the blank with a suitable amount of distilled water if absorbance falls outside the range of calibration.

5. Calibration.

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

5.2 Spectrophotometer. Add 0.0 to 1.60 ml of standard solution to a series of beakers. To each beaker add 25 ml of absorbing solution and sodium hydroxide (1N) dropwise until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of section 4.2 to collect enough data to draw a calibration curve of concentration in µg/N per sample versus absorbance.

6. Calculations.

6.1 Sample volume.
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2.2 Sample recovery.
2.2.1 Wash bottle—Two.
2.2.2 Graduated cylinder—250 ml, 600 ml.
2.2.3 Glass sample storage container.
2.2.4 Graduated cylinder—250 ml.
2.3 Analysis.
2.3.1 Pipette—25 ml, 100 ml.
2.3.2 Burdick—60 ml.
2.3.3 Erlenmeyer flask—250 ml.
2.3.4 Graduated cylinder—100 ml.
2.3.5 Trip balance—200 g. capacity, to measure to 0.001 g.
2.3.6 Dropping bottle—to add indicator solution.
2.3.7 Reagents.
3.1 Sampling.
3.1.1 Filters—Glass fiber, N8A type 1103
3.1.2 Silica gel—Indicating type, 6-16 mesh,detect at 180°F, (60° C.) for 5 hours.
3.1.3 Water—Distilled, distilled.
3.1.4 Excrepant, E:57—Stir 500 ml. of
3.2 Water—Distilled, distilled.
3.2.1 Water—Distilled, distilled.
3.2.2 Excrepant, E:57.
3.2.3 Analysis.
3.2.4 Water—Distilled, distilled.
3.2.5 Excrepant.
3.2.6 Thorium indicator—1-(o-aminophenyl)-2-(o-aminophenyl) benzene,
3.2.7 Water, 1 g. in 100 ml. distilled water.
3.3 Sulfuric acid standard (0.01N)—Dilute
3.3.4 Sulfuric acid standard.
3.3.5 Sulfuric acid standard.
4. Procedure.
4.1 Sampling.
4.1.1 Air sampling.
4.1.2 Preparation of collection train.
4.1.3 Train operation. For each run, rec
4.2 Procedure.
4.2.1 Washing the collection train.
4.2.2 Prior to using or cleaning the collection train, clean it thoroughly with dilute sulfuric acid to remove any clogging materials. The train is then cleaned with hot water and dried before being placed in storage.

Figure 8-1. Sulfuric acid mist sampling train.

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rapid adjustment of the sampling rate without other computations. Anthropometric data details the procedure for using these nomographs. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 10 minutes.

<table>
<thead>
<tr>
<th>PHASE</th>
<th>LOCATION</th>
<th>CARRIER</th>
<th>DATA</th>
<th>ACTION</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</table>

\[ V_{\text{std}} = V_{\text{a}} \left( \frac{T_{\text{std}}}{T_{\text{a}}} \right) \left( \frac{P_{\text{bar}} + \Delta H}{P_{\text{std}}} \right) = \left( \frac{17.71}{\text{in. Hg}} \right) V_{\text{a}} \left( \frac{P_{\text{bar}} + \Delta H}{P_{\text{std}}} \right) \]  

\[ T_{\text{a}} = \text{Average dry gas meter temperature}, \]
\[ P_{\text{bar}} = \text{Barometric pressure at the stack outlet, inches Hg}, \]
\[ \Delta H = \text{Pressure drop across the gas meter, inches Hg}, \]
\[ P_{\text{std}} = \text{Absolute pressure at standard conditions}, \]  

6.3 Sulfur dioxide concentration.

\[ (V_{\text{i}} - V_{\text{th}}) \left( \frac{V_{\text{std}}}{V_{\text{a}}} \right) \]  

\[ C_{\text{SO}_2} = \left( 7.05 \times 10^{-5} \right) \left( \frac{V_{\text{std}}}{V_{\text{a}}} \right) \left( \frac{V_{\text{std}}}{V_{\text{a}}} \right) \]  

\[ N = \text{Normality of barium perchlorate titrant, g-equiv./l}, \]
\[ V_{\text{std}} = \text{Total solution volume of sulfur dioxide, first impinger and filter, ml}, \]
\[ V_{\text{a}} = \text{Volume of sample aliquot titrated, ml}, \]
\[ V_{\text{std}} = \text{Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8-1}, \]

6.3 Sulfur dioxide concentration.

\[ C_{\text{SO}_2} = \left( 7.05 \times 10^{-5} \right) \left( \frac{V_{\text{std}}}{V_{\text{a}}} \right) \left( \frac{V_{\text{std}}}{V_{\text{a}}} \right) \]  

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

7. References.


Enslen, Robert, Construction Details of Pollution Source Sampling Equipment, Environmental Protection Agency, Air Pollution Control Office Publication No. APFD-2531.

RULES AND REGULATIONS

**Method 9—Visual Determination of the Opacity of Emissions from Stationary Sources**

1. **Principle and applicability.**

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

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

2. **Procedure.**

2.1 The qualified observer stands at approximately two stack heights, but not more than a quarter of a mile from the base of the stack with the sun to his back. From a vantage point perpendicular to the plume, the observer studies the point of greatest opacity in the plume. The data required in Figure 9-1 is recorded every 15 to 60 seconds to the nearest 0.5 opacity. A minimum of 23 readings is taken.

3. **Qualifications.**

3.1 To certify as an observer, a candidate must complete a smoke reading course conducted by EPA, or equivalent; in order to certify the candidate must assign opacity readings in 0.5-inch increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent on any one reading and an average error not to exceed 10 percent in each category. The smoke generator used to qualify the observers must be equipped with a calibrated smoke indicator or light transmission meter located in the source stack; if the smoke generator is to determine the actual opacity of the emissions. All qualified observers must pass this test every 6 months in order to remain certified.

4. **Calculations.**

4.1 Determine the average opacity.

5. **References.**

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


![Figure 9-1. Field data.](image)