

unpaved parking lots, haul and access roads, vehicle track-out, and disturbed soil associated with these activities. Rule 310.01 is a rule designed to limit the emissions of fugitive dust or particulate matter from disturbed surfaces and vehicle use in open areas and vacant lots, unpaved roadways and parking lots, livestock activities, erosion-caused deposition of bulk material on paved roadways, and easements, rights-of-way, and access roads for utilities.

## II. EPA's Evaluation and Action

### A. How is EPA evaluating the rules?

SIP rules must be enforceable (*see* section 110(a) of the Act) and must not relax existing requirements (*see* sections 110(l) and 193). In addition, SIP rules must implement Reasonably Available Control Measures (RACM), including Reasonably Available Control Technology (RACT), in moderate PM nonattainment areas, and Best Available Control Measures (BACM), including Best Available Control Technology (BACT), in serious PM nonattainment areas (*see* CAA sections 189(a)(1) and 189(b)(1)). The MCAQD regulates a PM nonattainment area classified as serious (*see* 40 CFR part 81), so Rule 310 and Rule 310.01 must implement BACM.

Guidance and policy documents that we use to evaluate enforceability and RACM or BACM requirements consistently include the following:

1. "Issues Relating to VOC Regulation Cutpoints, Deficiencies, and Deviations; Clarification to Appendix D of November 24, 1987 **Federal Register** Notice." (Blue Book), notice of availability published in the May 25, 1988 **Federal Register**.

2. "Guidance Document for Correcting Common VOC & Other Rule Deficiencies," EPA Region 9, August 21, 2001 (the Little Bluebook).

3. "State Implementation Plans; General Preamble for the Implementation of Title I of the Clean Air Act Amendments of 1990," 57 FR 13498 (April 16, 1992); 57 FR 18070 (April 28, 1992).

4. "State Implementation Plans for Serious PM-10 Nonattainment Areas, and Attainment Date Waivers for PM-10 Nonattainment Areas Generally; Addendum to the General Preamble for the Implementation of Title I of the Clean Air Act Amendments of 1990," 59 FR 41998 (August 16, 1994).

5. "PM-10 Guideline Document," EPA 452/R-93-008, April 1993.

6. "Fugitive Dust Background Document and Technical Information Document for Best Available Control Measures," EPA 450/2-92-004, September 1992.

### B. Do the rules meet the evaluation criteria?

We believe these rules are consistent with the relevant policy and guidance. Our Technical Support Documents (TSD) on each rule has our detailed review and evaluation.

### C. EPA Recommendations To Further Improve the Rules

We have no recommendation at this time.

### D. Public Comment and Final Action

Because EPA believes the submitted rules fulfill all relevant requirements, we are proposing to fully approve them as described in section 110(k)(3) of the Act. We will accept comments from the public on this proposal for the next 30 days. Unless we receive convincing new information during the comment period, we intend to publish a final approval action that will incorporate these rules into the federally enforceable SIP.

## III. Statutory and Executive Order Reviews

Under the Clean Air Act, the Administrator is required to approve a SIP submission that complies with the provisions of the Act and applicable Federal regulations. 42 U.S.C. 7410(k); 40 CFR 52.02(a). Thus, in reviewing SIP submissions, EPA's role is to approve State choices, provided that they meet the criteria of the Clean Air Act. Accordingly, this action merely approves State law as meeting Federal requirements and does not impose additional requirements beyond those imposed by State law. For that reason, this action:

- Is not a "significant regulatory action" subject to review by the Office of Management and Budget under Executive Order 12866 (58 FR 51735, October 4, 1993);
- Does not impose an information collection burden under the provisions of the Paperwork Reduction Act (44 U.S.C. 3501 *et seq.*);
- Is certified as not having a significant economic impact on a substantial number of small entities under the Regulatory Flexibility Act (5 U.S.C. 601 *et seq.*);
- Does not contain any unfunded mandate or significantly or uniquely affect small governments, as described in the Unfunded Mandates Reform Act of 1995 (Pub. L. 104-4);
- Does not have Federalism implications as specified in Executive Order 13132 (64 FR 43255, August 10, 1999);
- Is not an economically significant regulatory action based on health or

safety risks subject to Executive Order 13045 (62 FR 19885, April 23, 1997);

- Is not a significant regulatory action subject to Executive Order 13211 (66 FR 28355, May 22, 2001);

- Is not subject to requirements of Section 12(d) of the National Technology Transfer and Advancement Act of 1995 (15 U.S.C. 272 note) because application of those requirements would be inconsistent with the Clean Air Act; and

- Does not provide EPA with the discretionary authority to address, as appropriate, disproportionate human health or environmental effects, using practicable and legally permissible methods, under Executive Order 12898 (59 FR 7629, February 16, 1994).

In addition, this rule does not have tribal implications as specified by Executive Order 13175 (65 FR 67249, November 9, 2000), because the SIP is not approved to apply in Indian country located in the State, and EPA notes that it will not impose substantial direct costs on tribal governments or preempt tribal law.

### List of Subjects in 40 CFR Part 52

Environmental protection, Air pollution control, Intergovernmental relations, Particulate matter, Reporting and recordkeeping requirements.

**Authority:** 42 U.S.C. 7401 *et seq.*

Dated: August 23, 2010.

**Jared Blumenfeld,**

*Regional Administrator, Region IX.*

[FR Doc. 2010-21959 Filed 9-1-10; 8:45 am]

**BILLING CODE 6560-50-P**

## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Part 60

[EPA-HQ-OAR-2010-0115; FRL-9195-9]

RIN 2060-AQ23

### Method 16C for the Determination of Total Reduced Sulfur Emissions From Stationary Sources

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Proposed rule.

**SUMMARY:** This action proposes a method for measuring total reduced sulfur (TRS) emissions from stationary sources. The EPA is making this method available for general use as requested by a number of source testing companies since it has been allowed for use in the past on a case-by-case basis for kraft pulp mills and refineries. This proposed method would offer advantages over

current methods in that real-time data are acquired and testers are allowed to use analyzers and procedures for measuring TRS that are commonly used to measure sulfur dioxide (SO<sub>2</sub>). The proposed method would offer an alternative to methods that are currently required.

**DATES:** Comments must be received on or before November 1, 2010.

**ADDRESSES:** Submit your comments, identified by Docket ID No. EPA-HQ-OAR-2010-0115, by one of the following methods:

- *http://www.regulations.gov:* Follow the on-line instructions for submitting comments.

- *E-mail:* [a-and-r-docket@epa.gov](mailto:a-and-r-docket@epa.gov), attention Docket ID No. EPA-HQ-OAR-2010-0115.

- *Fax:* (202) 566-9744, attention Docket ID No. EPA-HQ-OAR-2010-0115.

- *Mail:* Method 16C for the Determination of Total Reduced Sulfur Emissions from Stationary Sources, Docket ID No. EPA-HQ-OAR-2010-0115. Environmental Protection Agency, Mailcode: 2822T, 1200 Pennsylvania Ave., NW., Washington, DC 20460. Please include a total of two copies.

- *Hand Delivery:* Deliver your comments to EPA Docket Center, Public Reading Room, EPA West, Room 3334, 1301 Constitution Ave., NW., Washington, DC 20460, attention Docket ID No. EPA-HQ-OAR-2010-0115. Such deliveries are only accepted during the Docket's normal hours of operation, and special arrangements should be made for deliveries of boxed information.

*Instructions:* Direct your comments to Docket ID No. EPA-HQ-OAR-2010-0115. EPA's policy is that all comments received will be included in the public docket without change and may be made available online at <http://www.regulations.gov>, including any personal information provided, unless the comment includes information claimed to be confidential business information (CBI) or other information whose disclosure is restricted by statute. Do not submit information that you consider to be CBI or otherwise protected through <http://www.regulations.gov> or e-mail. The <http://www.regulations.gov> Web site is an "anonymous access" system, which means EPA will not know your identity or contact information unless you provide it in the body of your comment. If you send an e-mail comment directly to EPA without going through <http://www.regulations.gov>, your e-mail address will be automatically captured and included as part of the comment that is placed in the public docket and

made available on the Internet. If you submit an electronic comment, EPA recommends that you include your name and other contact information in the body of your comment and with any disk or CD-ROM you submit. If EPA cannot read your comment due to technical difficulties and cannot contact you for clarification, EPA may not be able to consider your comment. Electronic files should avoid the use of special characters, any form of encryption, and be free of any defects or viruses. For additional information about EPA's public docket, visit the EPA Docket Center homepage at <http://www.epa.gov/epahome/dockets.htm>.

*Docket:* All documents in the docket are listed in the <http://www.regulations.gov> index. Although listed in the index, some information is not publicly available, e.g., CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will be publicly available only in hard copy. Publicly available docket materials are available either electronically in <http://www.regulations.gov> or in hard copy at the Method 16C for the Determination of Total Reduced Sulfur Emissions from Stationary Sources Docket, EPA/DC, EPA West, Room 3334, 1301 Constitution Ave., NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the Air Docket is (202) 566-1742.

**FOR FURTHER INFORMATION CONTACT:** Mr. Foston Curtis, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Air Quality Assessment Division (E143-02), Research Triangle Park, NC 27711; telephone number: (919) 541-1063; fax number: (919) 541-0516; and e-mail address: [curtis.foston@epa.gov](mailto:curtis.foston@epa.gov).

#### **SUPPLEMENTARY INFORMATION:**

##### **I. General Information**

###### *A. Does this action apply to me?*

Method 16C applies to the measurement of TRS at kraft pulp mills subject to Subpart BB of the New Source Performance Standards (NSPS). Currently, Methods 16, 16A, and 16B are allowed at these facilities. Method 16C would offer an additional alternative. The methods required under Subpart BB are sometimes used in special cases under the petroleum refineries NSPS (Subpart J). Method 16C may be applicable to other sources regulated by State and local regulations

that specify the use of Methods 16, 16A, or 16B if desired. The entities that are potentially affected by this proposal are included in the following table.

Category	NAICS <sup>a</sup>	Examples of regulated entities
Industry .....	324110	Petroleum Refineries.
Industry .....	322110	Kraft Pulp Mills.

<sup>a</sup>North American Industry Classification System.

###### *B. What should I consider as I prepare my comments for EPA?*

1. *Submitting CBI.* Do not submit this information to EPA through <http://www.regulations.gov> or e-mail. Clearly mark any of the information that you claim to be CBI. For CBI information in a disk or CD-ROM that you mail to EPA, mark the outside of the disk or CD-ROM as CBI and then identify electronically within the disk or CD-ROM the specific information that is claimed as CBI. In addition to one complete version of the comment that includes information claimed as CBI, a copy of the comment that does not contain the information claimed as CBI must be submitted for inclusion in the public docket. Information so marked will not be disclosed except in accordance with procedures set forth in 40 CFR part 2.

2. *Tips for Preparing Your Comments.* When submitting comments, remember to:

- Identify the rulemaking by docket number and other identifying information (subject heading, **Federal Register** date and page number).
- Follow directions—The agency may ask you to respond to specific questions or organize comments by referencing a Code of Federal Regulations (CFR) part or section number.
- Explain why you agree or disagree, suggest alternatives, and substitute language for your requested changes.
- Describe any assumptions and provide any technical information and/or data that you used.
- If you estimate potential costs or burdens, explain how you arrived at your estimate in sufficient detail to allow for it to be reproduced.
- Provide specific examples to illustrate your concerns, and suggest alternatives.
- Explain your views as clearly as possible, avoiding the use of profanity or personal threats.
- Make sure to submit your comments by the comment period deadline identified.

*C. Where can I get a copy of this document?*

In addition to being available in the docket, an electronic copy of this proposed rule is also available on the Worldwide Web (WWW) through the Technology Transfer Network (TTN). Following the Administrator's signature, a copy of this proposed rule will be posted on the TTN's policy and guidance page for newly proposed or promulgated rules at the following address: <http://www.epa.gov/ttn/oarpg/>. The TTN provides information and technology exchange in various areas of air pollution control.

*D. How is this document organized?*

The information in this preamble is organized as follows:

- I. General Information
  - A. Does this action apply to me?
  - B. What should I consider as I prepare my comments for EPA?
  - C. Where can I get a copy of this document?
  - D. How is this document organized?
- II. Background and Summary of Method 16C
- III. Statutory and Executive Order Reviews
  - A. Executive Order 12866: Regulatory Planning and Review
  - B. Paperwork Reduction Act
  - C. Regulatory Flexibility Act
  - D. Unfunded Mandates Reform Act
  - E. Executive Order 13132: Federalism
  - F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments
  - G. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks
  - H. Executive Order 13211: Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use
  - I. National Technology Transfer Advancement Act
  - J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations

**II. Background and Summary of Method 16C**

The EPA is making Method 16C available for general use as requested by a number of source testing companies since it has been allowed on a case-by-case basis in the past. The proposed method would offer an alternative to methods that are currently required.

Method 16C uses the sampling procedures of Method 16A and the analytical procedures of Method 6C to measure TRS. Total reduced sulfur is defined as hydrogen sulfide, methyl mercaptan, dimethyl sulfide, and dimethyl disulfide. As described in Method 16A, the sample is collected from the source through a heated probe and immediately conditioned in a

citrate buffer scrubber. The conditioned sample is oxidized in a tube furnace to convert TRS to sulfur dioxide (SO<sub>2</sub>). The oxidized sample is then analyzed for SO<sub>2</sub> using a real-time SO<sub>2</sub> analyzer as prescribed in Method 6C. In this method, we are combining the proven combustion process of Method 16A with the analytical techniques currently used for SO<sub>2</sub> to form a new, improved method for measuring TRS.

This method would become available as an option for use in connection with the New Source Performance Standards for kraft pulp mills and possibly petroleum refineries. We have allowed its use in approximately four tests over the past 10 years on a case-by-case basis and, based on our experience, it is a good alternative. Method 16C offers advantages over currently required methods by supplying real-time data in the field using analyzers and procedures that are currently used for other pollutants. Performance checks are contained in the method to ensure that bias and calibration precision are periodically checked and maintained.

This rule will not require the use of Method 16C but will allow it as an alternative method at the discretion of the user. This method does not impact testing stringency; data are collected under the same conditions and time intervals as the current methods.

**III. Statutory and Executive Order Reviews**

*A. Executive Order 12866: Regulatory Planning and Review*

This action is not a "significant regulatory action" under the terms of Executive Order (EO) 12866 (58 FR 51735, October 4, 1993) and is therefore not subject to review under the EO.

*B. Paperwork Reduction Act*

This action does not impose an information collection burden under the provisions of the Paperwork Reduction Act, 44 U.S.C. 3501 et seq. Burden is defined at 5 CFR 1320.3(b). The method being proposed in this action does not add information collection requirements but makes an additional optional procedure available for use by affected parties.

*C. Regulatory Flexibility Act*

The Regulatory Flexibility Act (RFA) generally requires an agency to prepare a regulatory flexibility analysis of any rule subject to notice and comment rulemaking requirements under the Administrative Procedure Act or any other statute unless the agency certifies that the rule will not have a significant economic impact on a substantial

number of small entities. Small entities include small businesses, small organizations, and small governmental jurisdictions.

For purposes of assessing the impacts of today's rule on small entities, small entity is defined as (1) A small business as defined by the Small Business Administration's (SBA) regulations at 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population of less than 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field.

After considering the economic impacts of today's proposed rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. This rulemaking does not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard. As such, it will not present a significant economic impact on a substantial number of small businesses.

We continue to be interested in the potential impacts of the proposed rule on small entities and welcome comments on issues related to such impacts.

*D. Unfunded Mandates Reform Act*

This action contains no Federal mandates under the provisions of Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), 2 U.S.C. 1531–1538 for State, local, or tribal governments or the private sector. The action imposes no enforceable duty on any State, local or tribal governments or the private sector. Therefore, this action is not subject to the requirements of sections 202 or 205 of the UMRA. This action is also not subject to the requirements of section 203 of UMRA because it contains no regulatory requirements that might significantly or uniquely affect small governments. This action makes available a new optional method for measuring pollutants but adds no new requirements.

*E. Executive Order 13132: Federalism*

This action does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. This action simply makes an optional test method

available for affected sources who desire to use it. Thus, Executive Order 13132 does not apply to this action. In the spirit of Executive Order 13132, and consistent with EPA policy to promote communications between EPA and State and local governments, EPA specifically solicits comment on this proposed rule from State and local officials.

*F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments*

This action does not have tribal implications, as specified in Executive Order 13175 (65 FR 67249, November 9, 2000). This action makes available a new optional method for measuring pollutants but adds no new requirements. Thus, Executive Order 13175 does not apply to this action. EPA specifically solicits additional comment on this proposed action from tribal officials.

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

EPA interprets EO 13045 (62 FR 19885, April 23, 1997) as applying only to those regulatory actions that concern health or safety risks, such that the analysis required under section 5-501 of the EO has the potential to influence the regulation. This action is not subject to EO 13045 because it does not establish an environmental standard intended to mitigate health or safety risks.

*H. Executive Order 13211: Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use*

This action is not subject to Executive Order 13211 (66 FR 28355 (May 22, 2001)), because it is not a significant regulatory action under Executive Order 12866.

*I. National Technology Transfer and Advancement Act*

Section 12(d) of the National Technology Transfer and Advancement Act of 1995 ("NTTAA"), Public Law 104-113 (15 U.S.C. 272 note) directs EPA to use voluntary consensus

standards in its regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (e.g., materials specifications, test methods, sampling procedures, and business practices) that are developed or adopted by voluntary consensus standards bodies. NTTAA directs EPA to provide Congress, through OMB, explanations when the Agency decides not to use available and applicable voluntary consensus standards. This proposed rulemaking does not involve technical standards.

*J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations*

Executive Order (EO) 12898 (59 FR 7629 (Feb. 16, 1994)) establishes Federal executive policy on environmental justice. Its main provision directs Federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations and low-income populations in the United States.

EPA has determined that this proposed rule will not have disproportionately high and adverse human health or environmental effects on minority or low-income populations because it does not affect the level of protection provided to human health or the environment. This rule adds an optional test method and does not cause emission increases from regulated sources.

**Method 16C for the Determination of Total Reduced Sulfur Emissions From Stationary Sources**

**List of Subjects in 40 CFR Part 60**

Environmental protection, Air pollution control, Test methods and procedures, and Performance specifications.

Dated: August 26, 2010.

**Lisa P. Jackson,**  
Administrator.

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

**PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES**

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

**Authority:** 42 U.S.C. 7401-7601.

2. Add Method 16C to Appendix A-6 to read as follows:

**APPENDIX A-6 TO PART 60—TEST METHODS 16 THROUGH 18**

\* \* \* \* \*

**METHOD 16C—DETERMINATION OF TOTAL REDUCED SULFUR EMISSIONS FROM STATIONARY SOURCES**

*1.0 Scope and Application*

What is method 16C?

Method 16C is a procedure for measuring total reduced sulfur (TRS) in stationary source emissions using a continuous instrumental analyzer. Quality assurance and quality control requirements are included to assure that you, the tester, collect data of known quality. You must document your adherence to these specific requirements for equipment, supplies, sample collection and analysis, calculations, and data analysis. This method does not completely describe all equipment, supplies, and sampling and analytical procedures you will need but refers to other methods for some of the details. Therefore, to obtain reliable results, you should also have a thorough knowledge of these additional test methods which are found in appendix A to this part:

(a) Method 6C—Determination of Sulfur Dioxide Emissions from Stationary Sources (Instrumental Analyzer Procedure)

(b) Method 7E—Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)

(c) Method 16A—Determination of Total Reduced Sulfur Emissions from Stationary Sources (Impinger Technique)

1.1 Analytes. What does Method 16C determine?

Analyte	CAS No.
Total reduced sulfur including .....	N/A
Dimethyl disulfide (DMDS), [(CH <sub>3</sub> ) <sub>2</sub> S <sub>2</sub> ] .....	62-49-20
Dimethyl sulfide (DMS), [(CH <sub>3</sub> ) <sub>2</sub> S] .....	75-18-3
Hydrogen sulfide (H <sub>2</sub> S) .....	7783-06-4
Methyl mercaptan (MeSH), [CH <sub>4</sub> S] .....	74-93-1
Reported as: Sulfur dioxide (SO <sub>2</sub> ) .....	7449-09-5

1.2 Applicability. This method is applicable for determining TRS emissions

from recovery furnaces (boilers), lime kilns, and smelt dissolving tanks at kraft pulp

mills, and from other sources when specified in an applicable subpart of the regulations.

1.3 Data Quality Objectives. Adherence to the requirements described in Method 16C will enhance the quality of the data obtained from air pollutant sampling methods.

## 2.0 Summary of Method

2.1 An integrated gas sample is extracted from the stack. The SO<sub>2</sub> is removed selectively from the sample using a citrate buffer solution. The TRS compounds are then thermally oxidized to SO<sub>2</sub> and determined as SO<sub>2</sub> by an instrumental analyzer. This method is a combination of the sampling procedures of Method 16A and the analytical procedures of Method 6C (referenced in Method 7E), with minor modifications to facilitate their use together.

## 3.0 Definitions

*Analyzer calibration error, Calibration curve, Calibration gas, Low-level gas, Mid-level gas, High-level gas, Calibration drift, Calibration span, Data recorder, Direct calibration mode, Gas analyzer, Interference check, Measurement system, Response time, Run, System calibration mode, System performance check, and Test* are the same as used in Methods 16A and 6C.

## 4.0 Interferences

4.1 Reduced sulfur compounds other than those regulated by the emission standards, if present, may be measured by this method. Compounds like carbonyl sulfide, which is partially oxidized to SO<sub>2</sub> and may be present in a lime kiln exit stack, would be a positive interferent. Interferences may vary among instruments, and instrument-specific interferences must be evaluated through the interference check.

4.2 Particulate matter from the lime kiln stack gas (primarily calcium carbonate) can cause a negative bias if it is allowed to enter the citrate scrubber; the particulate matter will cause the pH to rise and H<sub>2</sub>S to be absorbed before oxidation. Proper use of the particulate filter, described in Section 6.1.3 of Method 16A, will eliminate this interference.

## 5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices before performing this test method.

5.2 Hydrogen Sulfide. Hydrogen sulfide is a flammable, poisonous gas with the odor of rotten eggs. Hydrogen sulfide is extremely hazardous and can cause collapse, coma, and death within a few seconds of one or two inhalations at sufficient concentrations. Low concentrations irritate the mucous membranes and may cause nausea, dizziness, and headache after exposure. It is the responsibility of the user of this test method to establish appropriate safety and health practices.

## 6.0 Equipment and Supplies

What do I need for the measurement system? The measurement system is similar to those applicable components in Methods 16A and 6C. An example measurement system is shown in Figure 16C-1 and

component parts are discussed below. Modifications to the apparatus are accepted provided the performance criteria in Section 13.0 are met.

6.1 Probe. Teflon tubing, 6.4-mm (1/4-in.) diameter, sequentially wrapped with heat-resistant fiber strips, a rubberized heat tape (plug at one end), and heat-resistant adhesive tape. A flexible thermocouple or other suitable temperature measuring device must be placed between the Teflon tubing and the fiber strips so that the temperature can be monitored to prevent softening of the probe. The probe must be sheathed in stainless steel to provide in-stack rigidity. A series of bored-out stainless steel fittings placed at the front of the sheath will prevent moisture and particulate from entering between the probe and sheath. A 6.4-mm (1/4-in.) Teflon elbow (bored out) must be attached to the inlet of the probe, and a 2.54 cm (1 in.) piece of Teflon tubing must be attached at the open end of the elbow to permit the opening of the probe to be turned away from the particulate stream; this will reduce the amount of particulate drawn into the sampling train. The probe is depicted in Figure 16A-2 of Method 16A.

6.2 Probe Brush. Nylon bristle brush with handle inserted into a 3.2-mm (1/8-in.) Teflon tubing. The Teflon tubing should be long enough to pass the brush through the length of the probe.

6.3 Particulate Filter. 50-mm Teflon filter holder and a 1- to 2- $\mu$ m porosity, Teflon filter (may be available through Savillex Corporation, 5325 Highway 101, Minnetonka, Minnesota 55343, or other suppliers of filters). The filter holder must be maintained in a hot box at a temperature sufficient to prevent moisture condensation. A temperature of 121 °C (250 °F) was found to be sufficient when testing a lime kiln under sub-freezing ambient conditions.

6.4 SO<sub>2</sub> Scrubber. Three 300-ml Teflon segmented impingers connected in series with flexible, thick-walled, Teflon tubing. (Impinger parts and tubing may be available through Savillex or other suppliers.) The first two impingers contain 100 ml of citrate buffer, and the third impinger is initially dry. The tip of the tube inserted into the solution should be constricted to less than 3 mm (1/8-in.) ID and should be immersed to a depth of at least 5 cm (2 in.).

6.5 Combustion Tube. Quartz glass tubing with an expanded combustion chamber 2.54 cm (1 in.) in diameter and at least 30.5 cm (12 in.) long. The tube ends should have an outside diameter of 0.6 cm (1/4-in.) and be at least 15.3 cm (6 in.) long. This length is necessary to maintain the quartz-glass connector near ambient temperature and thereby avoid leaks. Alternative combustion tubes are acceptable provided they are shown to combust TRS at concentrations encountered during tests.

6.6 Furnace. A furnace of sufficient size to enclose the combustion chamber of the combustion tube with a temperature regulator capable of maintaining the temperature at 800  $\pm$  100 °C (1472  $\pm$  180 °F). The furnace operating temperature should be checked with a thermocouple to ensure accuracy.

6.7 Sampling Pump. A leak-free pump is required to pull the sample gas through the

system at a flow rate sufficient to minimize the response time of the measurement system and constructed of material that is non-reactive to the gas it contacts. For dilution-type measurement systems, an eductor pump may be used to create a vacuum that draws the sample through a critical orifice at a constant rate.

6.8 Calibration Gas Manifold. The calibration gas manifold must allow the introduction of calibration gases either directly to the gas analyzer in direct calibration mode or into the measurement system, at the probe, in system calibration mode, or both, depending upon the type of system used. In system calibration mode, the system must be able to flood the sampling probe and vent excess gas. Alternatively, calibration gases may be introduced at the calibration valve following the probe. Maintain a constant pressure in the gas manifold. For in-stack dilution-type systems, a gas dilution subsystem is required to transport large volumes of purified air to the sample probe and a probe controller is needed to maintain the proper dilution ratio.

6.9 Sample Gas Manifold. The sample gas manifold diverts a portion of the sample to the analyzer, delivering the remainder to the by-pass discharge vent. The manifold should also be able to introduce calibration gases directly to the analyzer. The manifold must be made of material that is non-reactive to SO<sub>2</sub> and be configured to safely discharge the bypass gas.

6.10 SO<sub>2</sub> Analyzer. You must use an instrument that uses an ultraviolet, non-dispersive infrared, fluorescence, or other detection principle to continuously measure SO<sub>2</sub> in the gas stream provided it meets the performance specifications in Section 13.0.

6.11 Data Recording. A strip chart recorder, computerized data acquisition system, digital recorder, or data logger for recording measurement data must be used.

## 7.0 Reagents and Standards

**Note:** Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. When such specifications are not available, the best available grade must be used.

7.1 Water. Deionized distilled water must conform to ASTM Specification D 1193-77 or 91 Type 3 (incorporated by reference—see § 60.17). The KMnO<sub>4</sub> test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.2 Citrate Buffer. Dissolve 300 g of potassium citrate (or 284 g of sodium citrate) and 41 g of anhydrous citric acid in 1 liter of water (200 ml is needed per test). Adjust the pH to between 5.4 and 5.6 with potassium citrate or citric acid, as required.

7.3 Calibration Gas. Refer to Section 7.1 of Method 7E (as applicable) for the calibration gas requirements. Example calibration gas mixtures are listed below.

- SO<sub>2</sub> in nitrogen (N<sub>2</sub>).
- SO<sub>2</sub> in air.
- SO<sub>2</sub> and CO<sub>2</sub> in N<sub>2</sub>.
- SO<sub>2</sub> and O<sub>2</sub> in N<sub>2</sub>.
- SO<sub>2</sub>/CO<sub>2</sub>/O<sub>2</sub> gas mixture in N<sub>2</sub>.
- CO<sub>2</sub>/NO<sub>x</sub> gas mixture in N<sub>2</sub>.

(g) CO<sub>2</sub>/SO<sub>2</sub>/NO<sub>x</sub> gas mixture in N<sub>2</sub>. For fluorescence-based analyzers, the O<sub>2</sub> and CO<sub>2</sub> concentrations of the calibration gases as introduced to the analyzer must be within 1 percent (absolute) O<sub>2</sub> and 1 percent (absolute) CO<sub>2</sub> of the O<sub>2</sub> and CO<sub>2</sub> concentrations of the effluent samples as introduced to the analyzer. Alternatively, for fluorescence-based analyzers, use calibration blends of SO<sub>2</sub> in air and the nomographs provided by the vendor to determine the quenching correction factor (the effluent O<sub>2</sub> and CO<sub>2</sub> concentrations must be known). This requirement does not apply to ambient-level fluorescence analyzers that are used in conjunction with sample dilution systems.

7.4 System Performance Check Gas. You must use hydrogen sulfide (100 ppmv or less) in nitrogen, stored in aluminum cylinders with concentration certified by the manufacturer.

**Note:** Alternatively, hydrogen sulfide recovery gas generated from a permeation device gravimetrically calibrated and certified at some convenient operating temperature may be used. The permeation rate of the device must be such that at the appropriate dilution gas flow rate, an H<sub>2</sub>S concentration can be generated in the range of the stack gas or within 20 percent of the emission standard.

7.5 Interference Check. Examples of test gases for the interference check are listed in Table 7E-3 of Method 7E.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pre-sampling Tests. Before measuring emissions, perform the following procedures:  
 (a) Calibration gas verification,  
 (b) Calibration error test,  
 (c) System performance check,  
 (d) Verification that the interference check has been satisfied.

8.1.1 Calibration Gas Verification. Obtain a certificate from the gas manufacturer documenting the quality of the gas. Confirm that the manufacturer certification is complete and current. Ensure that your calibration gas certifications have not expired. This documentation should be available on-site for inspection. To the extent practicable, select a high-level gas concentration that will result in the measured emissions being between 20 and 100 percent of the calibration span.

8.1.2 Analyzer Calibration Error Test. After you have assembled, prepared, and calibrated your sampling system and analyzer, you must conduct a 3-point analyzer calibration error test before the first run and again after any failed system

performance check or failed drift test to ensure the calibration is acceptable. Introduce the low-, mid-, and high-level calibration gases sequentially to the analyzer in direct calibration mode. For each calibration gas, calculate the analyzer calibration error using Equation 16C-1 in Section 12.2. The calibration error for the low-, mid-, and high-level gases must not exceed 5 percent or 0.5 ppmv. If the calibration error specification is not met, take corrective action and repeat the test until an acceptable 3-point calibration is achieved.

8.1.3 System Performance Check. Same as in Method 16A, Section 8.5, except samples need not be 30 minutes in duration, and the TRS sample concentration measured between system performance checks is corrected by the average of the two system performance samples. System performance checks are conducted before sampling begins (optional) and after each sample run (mandatory).

8.1.4 Interference Check. Same as in Method 7E, Section 8.2.7.

8.2 Measurement System Preparation.  
 8.2.1 For the SO<sub>2</sub> scrubber, measure 100 ml of citrate buffer into the first and second impingers; leave the third impinger empty. Immerse the impingers in an ice bath, and locate them as close as possible to the filter heat box. The connecting tubing should be free of loops. Maintain the probe and filter temperatures sufficiently high to prevent moisture condensation, and monitor with a suitable temperature sensor. Prepare the oxidation furnace and maintain at 800 ± 100 °C (1472 ± 180 °F).

8.2.2 Citrate Scrubber Conditioning Procedure. Condition the citrate buffer scrubbing solution by pulling stack gas through the Teflon impingers as described in Section 8.4.1.

8.3 Pretest Procedures. After the complete measurement system has been set up at the site and deemed to be operational, the following procedures must be completed before sampling is initiated.

8.3.1 Leak-Check. Appropriate leak-check procedures must be employed to verify the integrity of all components, sample lines, and connections. For components upstream of the sample pump, attach the probe end of the sample line to a manometer or vacuum gauge, start the pump and pull a vacuum greater than 50 mm (2 in.) Hg, close off the pump outlet, and then stop the pump and ascertain that there is no leak for 1 minute. For components after the pump, apply a slight positive pressure and check for leaks by applying a liquid (detergent in water, for example) at each joint. Bubbling indicates the

presence of a leak. As an alternative to the initial leak-test, the system performance check in Section 8.3.2 may be performed to verify the integrity of components.

8.3.2 Initial System Performance Check. A system performance check using the test gas (Section 7.4) is required prior to testing to validate the sampling train components and procedure.

8.4 Sample Collection and Analysis  
 8.4.1 After performing the required pretest procedures described in Section 8.1, insert the sampling probe into the test port ensuring that no dilution air enters the stack through the port. Condition the sampling system and citrate buffer solution for a minimum of 15 minutes before beginning analysis. (This preconditioning may not be necessary if the initial system performance check is performed.) Begin the sampling and analysis. Determine the concentration of SO<sub>2</sub> for the prescribed sample or run time. Method 16 defines a test run as sampling over a period of not less than 3 hours or more than 6 hours when testing kraft pulp mills. For Method 16C to be consistent with Method 16, a run may be obtained by: (1) sampling for three 60-minute intervals or (2) sampling for a 3-hour interval. (Three runs constitute a test.)

8.5 Post-Run Evaluations  
 8.5.1 System Performance Check. Perform a post-run system performance check (Section 8.5 of Method 16A) before replacing the citrate buffer solution and particulate filter and before the probe is cleaned. The check results must not exceed the 100 ± 20 percent limit set forth in Section 13.3. If this limit is exceeded, the intervening run or runs are considered invalid. However, if the recovery efficiency is not in the 100 ± 20 percent range, but the results do not affect the compliance or noncompliance status of the affected facility, the Administrator may decide to accept the results of the compliance test.

8.5.2 Calibration Drift. After a run or series of runs, not to exceed a 24-hour period after initial calibration, perform a calibration drift test using a calibration gas (preferably the level that best approximates the sample concentration) in direct calibration mode. This drift must not differ from the manufacturer certified concentration of the gas by more than 3 percent or 0.5 ppm. If the drift exceeds this limit, the intervening run or runs are considered valid, but a new analyzer calibration test must be performed and passed before continuing sampling.

9.0 Quality Control

Section	Quality control measure	Effect
8.1 .....	Sampling equipment leak-check and calibration.	Ensures accurate measurement of sample gas flow rate, sample volume.
8.1.2 .....	Analyzer calibration error .....	Establishes initial calibration accuracy within 2%.
8.3.2, 8.5.1 .....	System performance check .....	Ensures accuracy of sampling/analytical procedure within 20%.
8.5.2 .....	Calibration drift test .....	Ensures a stable calibration within 3%.
10.0 .....	Interference check .....	Checks for analytical interferences.

### 10.0 Calibration

10.1 Calibrate the system using the gases described in Section 7.3. The initial 3-point calibration error test as described in Section 8.1.2 is required and must meet the specifications in Section 13 before you start the test. We recommend you conduct an initial system performance test described in Section 8.1.4 as well before the test to validate the sampling components and procedures before sampling. After the test commences, a system performance check is required after each run. You must include a copy of the manufacturer's certification of the calibration gases used in the testing as part of the test report. This certification must include the 13 documentation requirements in the EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards, September 1997, as amended August 25, 1999.

#### 11.0 Analytical Procedure

Because sample collection and analysis are performed together (see Section 8.0), additional discussion of the analytical procedure is not necessary.

#### 12.0 Calculations and Data Analysis

##### 12.1 Nomenclature

ACE = Analyzer calibration error, percent of calibration span.

$B_{WO}$  = Fraction of volume of water vapor in the gas stream.

CD = Calibration drift, percent.

$C_{Dir}$  = Measured concentration of a calibration gas (low, mid, or high) when introduced in direct calibration mode, ppmv.

$C_{H2S}$  = Concentration of the system performance check gas, ppmv  $H_2S$ .

$C_S$  = Measured concentration of the system performance gas when introduced in system calibration mode, ppmv  $H_2S$ .

$C_V$  = Manufacturer certified concentration of a calibration gas (low, mid, or high), ppmv  $SO_2$ .

$C_{SO2}$  = Sample  $SO_2$  concentration, ppmv.

$C_{TRS}$  = Total reduced sulfur concentration corrected for system performance and adjusted to dry conditions, ppmv.

SP = System performance, percent.

12.2 Analyzer Calibration Error. Use Equation 16C-1 to calculate the analyzer calibration error for the low-, mid-, and high-level calibration gases.

$$ACE = \frac{C_{Dir} - C_V}{C_V} \times 100 \quad \text{Eq. 16C-1}$$

12.3 System Performance. Use Equation 16C-2 to calculate the system performance.

$$SP = \frac{C_S - C_{H2S}}{C_{H2S}} \times 100 \quad \text{Eq. 16C-2}$$

12.4 Calibration Drift. Use Equation 16C-3 to calculate the calibration drift at a single concentration level after a run or series of runs (not to exceed a 24-hr period) from initial calibration. Compare the calibration gas response to the original response obtained for the gas in the initial analyzer calibration test ( $ACE_i$ ).

$$CD = |ACE_i - ACE_n| \quad \text{Eq. 16C-3}$$

12.5 TRS Concentration as  $SO_2$ . For each sample or test run, calculate the arithmetic average of  $SO_2$  concentration values (e.g., 1-minute averages). Then calculate the sample TRS concentration using Equation 16C-4.

$$C_{TRS} = \frac{\overline{C_{SO2}}}{1 - |SP| - B_{WO}} \quad \text{Eq. 16C-4}$$

#### 13.0 Method Performance

13.1 Analyzer Calibration Error. At each calibration gas level (low, mid, and high), the calibration error must either not exceed 5.0 percent of the calibration gas concentration or  $|C_s - C_v|$  must be  $\leq 0.5$  ppmv.

13.2 System Performance. The system performance check result must be within 20 percent of the system performance gas concentration. Alternatively, the results are acceptable if  $|C_s - C_{dir}|$  is  $\leq 0.5$  ppmv.

13.3 Calibration Drift. The calibration drift at the end of any run or series of runs within a 24-hour period must not differ by more than 3 percent from the original ACE at that level or  $|ACE_i - ACE_n|$  must not exceed 0.5 ppmv.

13.4 Interference Check. For the analyzer, the total interference response (i.e., the sum of the interference responses of all tested gaseous components) must not be greater than 2.50 percent of the calibration span. The results are also acceptable if the sum of the responses does not exceed 0.5 ppmv for a calibration span of 5 to 10 ppmv, or 0.2 ppmv for a calibration span  $< 5$  ppmv.

#### 14.0 Pollution Prevention [Reserved]

#### 15.0 Waste Management [Reserved]

#### 16.0 References

1. The references are the same as in Section 16.0 of Method 16, Section 17.0 of Method 16A, and Section 17.0 of Method 6C.

2. National Council of the Paper Industry for Air and Stream Improvement, Inc., A Study of TRS Measurement Methods. Technical Bulletin No. 434. New York, NY. May 1984. 12p.

3. Margeson, J.H., J.E. Knoll, and M.R. Midgett. A Manual Method for TRS Determination. Draft available from the authors. Source Branch, Quality Assurance Division, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

#### 17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

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## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Part 140

[EPA-R09-OW-2010-0438; FRL-9196-3]

RIN 2009-AA04

### Marine Sanitation Devices (MSDs): Proposed Regulation To Establish a No Discharge Zone (NDZ) for California State Marine Waters

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Proposed rule.

**SUMMARY:** EPA is proposing to establish a No Discharge Zone (NDZ) for sewage discharges from: Large passenger vessels; and oceangoing vessels of 300 gross tons or more (referred to throughout this proposed rule as "Large oceangoing vessels") with two days or more sewage holding capacity to California State marine waters pursuant to Section 312(f)(4)(A) of the Clean Water Act (CWA), 33 U.S.C. 1322(f)(4)(A). This action is being taken in response to an April 5, 2006 application from the California State Water Resources Control Board (State) requesting establishment of this NDZ. Under Section 312(f)(4)(A), if EPA determines upon application by a State that the protection and enhancement of the quality of specified waters within such State requires such a prohibition, then EPA shall by regulation completely prohibit the discharge of any sewage (whether treated or not) from a vessel into such waters. California State marine waters would be defined as the territorial sea measured from the baseline, as determined in accordance with the Convention on the Territorial Sea and the Contiguous Zone, and extending seaward a distance of three miles, and would also include all enclosed bays and estuaries subject to tidal influences from the Oregon border to the Mexican border. (Federal Clean Water Act Section 502(8)). State marine waters also extend three miles from State islands, including the Farallones and the Northern and Southern Channel Islands. A map of California State marine waters can be obtained or viewed at the EPA's Web site at <http://www.epa.gov/region9/water/no-discharge/overview.html>, or by calling (415) 972-3476. It should be noted that effective March 2009, the National Oceanic and Atmospheric Administration (NOAA) established prohibitions on the discharge of sewage from large vessels in waters within the boundaries of the four National Marine