#### List of Subjects in 33 CFR Part 165

Harbors, Marine safety, Navigation (water), Reporting and recordkeeping requirements, Security measures, and Waterways.

■ For the reasons discussed in the preamble, the Coast Guard proposes to amend 33 CFR part 165 as follows:

# PART 165—REGULATED NAVIGATION AREAS AND LIMITED ACCESS AREAS

■ 1. The authority citation for part 165 continues to read as follows:

**Authority:** 33 U.S.C. 1226, 1231; 46 U.S.C. Chapter 701; 50 U.S.C. 191, 195; 33 CFR 1.05–1, 6.04–1, 6.04–6, and 160.5; Public Law 107–295, 116 Stat. 2064; Department of Homeland Security Delegation No. 0170.1.

■ 2. Add temporary § 165.T–0046 to read as follows:

### § 165.T-0046 Trent River, New Bern, North Carolina.

- (a) Safety Zone. The safety zone includes all waters within a 150 feet radius of position 350°06′09″ North 77°02′15″ West, approximately one hundred yards west of the Trent River Swing Bridge, New Bern, North Carolina. All coordinates reference Datum NAD 1983.
  - (b) Definition:
- (1) As used in this section; Captain of the Port representative means any U.S. Coast Guard commissioned, warrant or petty officer who has been authorized by the Captain of the Port, North Carolina, to act on her behalf.
  - (c) Regulation:
- (1) In accordance with the general regulations in § 165.23 of this part, entry into this zone is prohibited unless authorized by the Captain of the Port, North Carolina or his designated representatives.
- (2) The operator of any vessel in the immediate vicinity of this safety zone shall:
- (i) Stop the vessel immediately upon being directed to do so by any commissioned, warrant or petty officer on shore or on board a vessel that is displaying a U.S. Coast Guard Ensign.
- (ii) Proceed as directed by any commissioned, warrant or petty officer on shore or on board a vessel that is displaying a U.S. Coast Guard Ensign.
- (3) The Captain of the Port and the Sector Duty Officer at Sector North Carolina can be contacted at (252) 247–4570.
- (4) The Captain of the Port representative enforcing the safety zone can be contacted on VHF–FM marine band radio, channel 13 (156.65Mhz) and channel 16 (156.8Mhz).

(d) Enforcement period. This section will be enforced from 7:30 p.m. to 9 p.m. on May 30, 2008.

Dated: May 16, 2008.

#### June E. Ryan,

Captain, U.S. Coast Guard, Captain of the Port North Carolina.

[FR Doc. E8–11937 Filed 5–28–08; 8:45 am] **BILLING CODE 4910–15–P** 

# ENVIRONMENTAL PROTECTION AGENCY

#### 40 CFR Part 51

[EPA-HQ-OAR-2007-0844, FRL-8572-1] RIN 2060-A039

# Method 207—Pre-Survey Procedure for Corn Wet-Milling Facility Emission Sources

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

ACTION: Direct final rule.

**SUMMARY:** The EPA is taking direct final action to add Method 207 to the promulgated test methods in 40 CFR Part 51. Appendix M contains recommended test methods that are provided for the States to use in their State Implementation Plans. Therefore, this method may be used as an alternative to existing test methods for measuring volatile organic compound (VOC) emissions. This pre-survey method was developed by the corn wetmilling industry specifically to VOC mass emissions from processes within the corn wet-milling industry. It provides a systematic approach to develop a specific list of target organic compounds and the appropriate methods to measure those target compounds during subsequent VOC emissions testing. After using the presurvey procedure, the tester will have sufficient information to design a comprehensive testing program using Method 18 and other appropriate methods to measure the mass of VOC emissions during the actual emissions testing. This method is an alternative to existing test methods and does not add any new reporting requirements to the reporting requirements that already exist. While it is an alternative method, it is the recommended method for measuring VOC mass emissions from corn wet-milling facilities.

**DATES:** This direct final rule is effective on August 27, 2008 without further notice, unless EPA receives adverse comment by June 30, 2008. If EPA receives adverse comment, we will publish a timely withdrawal in the

**Federal Register** informing the public that this rule will not take effect.

**ADDRESSES:** Submit your comments, identified by Docket ID Number EPA–HQ–OAR–2007–0844, by one of the following methods:

- http://www.regulations.gov: Follow the online instructions for submitting comments.
  - E-mail: a-and-r-docket@epa.gov.
  - Fax: (202) 566-9744.
- Mail: Attention Docket ID No. EPA– HQ–OAR–2007–0844, U.S. Environmental Protection Agency, EPA West (Air Docket), Mail code: 2822T, 1200 Pennsylvania Avenue, NW., Washington, DC 20460. Please include a total of two copies.
- Hand Delivery: U.S. Environmental Protection Agency, EPA West (Air Docket), 1301 Constitution Avenue, Northwest, Room 3334, Washington, DC 20460, Attention Docket ID No. EPA–HQ–OAR–2007–0844. 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-2007-0844. 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 Air and Radiation Docket and Information Center, EPA/DC, EPA West Building, 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-

# FOR FURTHER INFORMATION CONTACT: Mr. Gary McAlister, Air Quality Assessment Division (E143–02), Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number: (919) 541–1062, e-mail address: mcalister.gary@epa.gov.

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#### I. General Information

A. Why Is EPA Using a Direct Final Rule?

The EPA is publishing this rule without a prior proposed rule because we view this as a noncontroversial

action and anticipate no adverse comment. This action adds a method to the list of recommended methods in 40 CFR Part 51, Appendix M. The method may be used as an alternative method to existing recommended methods, but it is not required to be used by any existing rule. In the "Proposed Rules" section of today's **Federal Register**, we are publishing a separate document that will serve as the proposed rule to add Method 207 to Appendix M in 40 CFR Part 51 if adverse comments are received on this direct final rule. We will not institute a second comment period on this action. Any parties interested in commenting must do so at this time. For Further information about commenting on this rule, see the ADDRESSES section of this document. If EPA receives adverse comment, we will publish a timely withdrawal in the Federal Register informing the public that this direct final rule will not take effect. We would address all public comments in any subsequent final rules based on the proposed rule.

#### B. Does This Action Apply to Me?

Method 207 affects/applies to the corn wet-milling industry and is used specifically to measure VOC mass emissions from processes within the corn wet-milling industry. Therefore, the categories and entities potentially regulated by this action include the following:

| Category                              | NAICS <sup>a</sup> | Examples of regulated entities                                                                             |
|---------------------------------------|--------------------|------------------------------------------------------------------------------------------------------------|
| IndustryState/local/tribal government |                    | Corn wet-milling. State, local, and tribal air quality management programs that regulate corn wet-milling. |

<sup>&</sup>lt;sup>a</sup> North American Industry Classification System.

#### C. Judicial Review

Under section 307(b)(1) of the Clean Air Act (CAA), judicial review of this direct final rule is available by filing a petition for review in the United States Court of Appeals for the District of Columbia Circuit by July 28, 2008. Only those objections to this final rule that were raised with reasonable specificity during the period for public comment may be raised during judicial review. Under section 307 (b)(2) of the CAA, the requirements that are the subject of this direct final rule may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

#### II. Background

A. What Is the Basis for This New Method?

The Method 207 Pre-Survey Procedure for Corn Wet-Milling Facility Emission Sources was developed in collaboration with the corn wet-milling industry (i.e., Corn Refiners Association (CRA)) specifically to measure volatile organic compound (VOC) mass emissions from processes within their facilities on an individual species basis. The pre-survey procedure provides a systematic approach to develop a specific list of target organic compounds and the appropriate sampling approach to collect those target compounds during subsequent VOC emissions testing. After using the new pre-survey procedure, the tester will have sufficient information to design a comprehensive

testing program using Method 18 and other appropriate methods to measure the mass of VOC emissions during the actual emissions testing. The CRA submitted their proposed procedures and supporting information to the EPA for review, and we concluded that it was an acceptable procedure for measuring VOC emissions from corn wet-milling facilities.

For the purposes of measuring VOC emissions from corn wet-milling facilities, all of the sampling procedures in Method 18 may be used, as well as an additional sampling procedure using water filled impingers to collect water soluble VOC. This sampling procedure is described in detail in Method 308 (40 CFR Part 63) and NCASI Method CI/SG/PULP-94.03. The resulting water samples should also be analyzed using the procedures in Method 308 or NCASI

Method CI/SG/PULP-94.03. If formaldehyde is a target compound, it may be collected with the water-filled impinger collection system, but the sample must be analyzed by procedures other than those in EPA Method 18. Examples of acceptable analytical procedures are those in Method 316 (40 CFR Part 63) or NCASI Method CI/SG/PULP-94.02.

## B. What Procedures Are Included in EPA Method 207?

In this action, we are amending Title V, Part 51, Appendix M of the Code of Federal Regulations (CFR) by adding a new measurement technique for VOC emissions from corn wet-milling facilities, referred to as "EPA Method 207—Pre-Survey Procedure for Corn Wet-Milling Facility Emission Sources." This method provides procedures for establishing analytes for subsequent EPA Method 18 testing to determine the total mass emissions of VOC from sources at corn wet-milling facilities. Objectives of the method include:

- (1) Identification of physical characteristics of the VOC contained in the effluent;
- (2) Determination of the appropriate Method 18 sampling approach to ensure efficient collection of all VOC present in the effluent;
- (3) Development of a specific list of target compounds to be quantified during the subsequent total VOC test program; and
- (4) Qualification of the list of target compounds as being a true representation of the total VOC.

The procedures call for using flame ionization detection in conjunction with various configurations of impingers, and other absorbents, or adsorbents to determine the best EPA Method 18 sampling train configuration for the assessment and capture of VOC. Volatile organic compound analytes present in the exhaust air from production processes located at corn wet-milling facilities typically fall into five general categories: Alcohols, aldehydes, acetate esters, ketones, and carboxylic acids, and typically contain fewer than six carbon atoms.

# 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 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). It adds a test method to the recommended methods in Appendix M of 40 CFR Part 51. This method is an alternative to existing test methods and does not add any new reporting requirements to the reporting requirements that already exist.

#### 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; or (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 this final rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. In determining whether a rule has a significant economic impact on a substantial number of small entities, the impact of concern is any significant adverse economic impact on a substantial number of small entities, since the primary purpose of the regulatory flexibility analyses is to identify and address regulatory alternatives "which minimize any significant economic impact of the rule on small entities." U.S.C. 603 and 604. Thus, an agency may certify that a rule will not have a significant economic impact on a substantial number of small entities if the rule relieves regulatory burden, or otherwise has a positive economic effect on all of the small entities subject to the rule. This action establishes voluntary alternative test procedures for satisfying the requirements of EPA Method 18,

Section 16 (pre-survey), which are used to determine the mass VOC emissions from processes within the corn wetmilling industry, by specifying the analytes for subsequent EPA Method 18 testing. This rule does not impose any new requirements or create impacts on small entities. Therefore, this action is not expected to have a significant economic impact on a substantial number of small entities.

#### D. Unfunded Mandates Reform Act

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

This rule contains no Federal mandates (under the regulatory provisions of Title II of the UMRA) for State, local, or tribal governments or the private sector. This rule imposes no enforceable duty on any State, local or tribal governments or the private sector.

EPA has determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments. This action adds a new test method for measuring VOC air emissions to the recommended methods in 40 CFR part 51. Because this method is an alternative method, its use is voluntary. It will not impose requirements on State, local governments, or tribal governments. Thus, this action is not subject to the requirements of sections 202 and 205 of the UMRA.

#### E. Executive Order 13132—Federalism

Executive Order 13132, entitled "Federalism" (64 FR 43255, August 10, 1999), requires the EPA to develop an accountable process to ensure "meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications." "Policies that have federalism implications" is defined in the Executive Order to include regulations that have "substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government."

This final rule 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. Because this method is an alternative method, its use is voluntary. It will not impose substantial direct compliance costs on State or local governments, nor will it preempt State law. Thus, Executive Order 13132 does not apply to this rule.

#### F. Executive Order 13175—Consultation and Coordination With Indian Tribal Governments

Executive Order 13175, entitled "Consultation and Coordination with Indian Tribal Governments" (65 FR 67249, November 9, 2000), requires EPA to develop an accountable process to ensure "meaningful and timely input by tribal officials in the development of regulatory policies that have tribal implications." This final rule does not have tribal implications, as specified in Executive Order 13175. The final action would add a test method that could be used as an alternative to existing methods. It does not add any new requirements and does not affect VOC emissions or air quality. Thus, Executive Order 13175 does not apply to this action.

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

EPA interprets Executive Order 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 Executive Order has the potential to influence the regulation. This action is not subject to Executive Order 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 rule is not subject to Executive Order 13211, "Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use" (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, section 12(d) (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.

The rulemaking involves technical standards. Therefore, the Agency conducted a search to identify potentially applicable voluntary consensus standards. However, we identified no such standards, and none were brought to our attention in comments. Therefore, EPA has decided to use Method 207 to measure mass VOC emissions from processes within the corn-wet milling industry. This method provides a systematic approach to develop a specific list of target organic compounds and the appropriate methods to measure those target compounds during subsequent VOC emissions testing.

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

Executive Order 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 final 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 action adds a new test method for measuring VOC air emissions to the recommended methods in 40 CFR Part 51. It does not change any existing rules that limit VOC air emission limits.

#### K. Congressional Review Act

The Congressional Review Act, 5 U.S.C. 801 et seq., as added by the Small **Business Regulatory Enforcement** Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the Federal Register. A Major rule cannot take effect until 60 days after it is published in the **Federal Register**. This action is not a "major rule" as defined by 5 U.S.C. 804(2). This rule will be effective on August 27, 2008.

#### List of Subjects in 40 CFR Part 51

Environmental protection, Administrative practice and procedure, Air pollution control, Intergovernmental relations, Volatile organic compounds.

Dated: May 21, 2008. **Stephen L. Johnson**, *Administrator*.

■ For reasons stated in the preamble, title 40, chapter I of the Code of Federal Regulations is amended as follows:

#### PART 51—[AMENDED]

■ 1. The authority citation for part 51 continues to read as follows:

**Authority:** 23 U.S.C. 101; 42 U.S.C. 7401, *et seq.* 

■ 2. Part 51 is amended in appendix M by adding Method 207 in numerical order to read as follows:

#### Appendix M to Part 51—Recommended Test Methods for State Implementation Plans

\* \* \* \* \*

#### METHOD 207—PRE-SURVEY PROCEDURE FOR CORN WET-MILLING FACILITY EMISSION SOURCES

- 1.0 Scope and Application
- 1.1 *Analyte*. Total gaseous organic compounds.
- 1.2 Applicability. This pre-survey method is intended for use at corn wet-milling (CWM) facilities to satisfy the requirements of Method 18, Section 16 (Pre-survey). This procedure establishes the analytes for subsequent Method 18 testing to determine the total mass emissions of VOCs from sources at CWM facilities. The specific objectives of the pre-survey procedure are:
- 1.2.1 Identify the physical characteristics of the VOC contained in the effluent.
- 1.2.2 Determine the appropriate Method 18 sampling approach to ensure efficient collection of all VOC present in the effluent.
- 1.2.3 Develop a specific list of target compounds to be quantified during the subsequent total VOC test program.
- 1.2.4 Qualify the list of target compounds as being a true representation of the total VOC.
- 1.3 Range. The lower and upper ranges of this procedure are determined by the sensitivity of the flame ionization detector (FID) instruments used. Typically, gas detection limits for the VOCs will be on the order of 1–5 ppmv, with the upper limit on the order of 100,000 ppmv.

#### 2.0 Summary of Method

**Note:** Method 6, Method 18, and Method 25A as cited in this method refer to the methods in 40 CFR Part 60, Appendix A.

This procedure calls for using an FIA in conjunction with various configurations of impingers, and other absorbents, or adsorbents to determine the best EPA Method 18 sampling train configuration for the assessment and capture of VOCs. VOC compounds present in the exhaust gas from processes located at CWM facilities fall into five general categories: Alcohols, aldehydes, acetate esters, ketones, and carboxylic acids, and typically contain fewer than six carbon atoms. This pre-survey protocol characterizes and identifies the VOC species present. Since it is qualitative in nature, quantitative performance criteria do not apply.

#### 3.0 Definitions

3.1 Calibration drift means the difference in the measurement system response to a mid-level calibration gas before and after a stated period of operation during which no

- unscheduled maintenance, repair, or adjustment took place.
- 3.2 Calibration error means the difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.
- 3.3 Calibration gas means a known concentration of a gas in an appropriate diluent gas.
- 3.4 Measurement system means the equipment required for the determination of the gas concentration. The system consists of the following major subsystems:
- 3.4.1 Sample interface means that portion of a system used for one or more of the following: Sample acquisition, sample transportation, sample conditioning, or protection of the analyzer(s) from the effects of the stack effluent.
- 3.4.2 Organic analyzer means that portion of the measurement system that senses the gas to be measured and generates an output proportional to its concentration.
- 3.5 Response time means the time interval from a step change in pollutant concentration at the inlet to the emission measurement system to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.
- 3.6 Span Value means the upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations. The span value is established in the applicable regulation and is usually 1.5 to 2.5 times the applicable emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100 percent of the recorder scale.
- 3.7 Zero drift means the difference in the measurement system response to a zero level calibration gas before or after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.
- 4.0 Interferences [Reserved]
- 5.0 Safety [Reserved]
- 6.0 Equipment and Supplies
- 6.1 Organic Concentration Analyzer. A flame ionization analyzer (FIA) with heated detector block and sample handling system, meeting the requirements of USEPA Method 25A.
- 6.2 Heated Sampling System. A sampling system consisting of a stainless steel probe with particulate filter, Teflon® sample line, and sampling pump capable of moving 1.0 l/ min through the sample probe and line. The entire system from probe tip to FIA analyzer must have the capability to maintain all sample-wetted parts at a temperature >120°C. A schematic of the heated sampling system and impinger train is shown in Figure 1 of this method.
- 6.3 Impinger Train. EPA Method 6 type, comprised of three midget impingers with appropriate connections to the sampling system and FIA system. The impinger train may be chilled in an ice bath or maintained at a set temperature in a water bath as indicated by the operator's knowledge of the source and the compounds likely to be

- present. Additional impingers or larger impingers may be used for high moisture sources.
  - 6.4 Adsorbent tubes.
- 6.4.1 Silica gel, SKC Type 226–22 or equivalent, with appropriate end connectors and holders.
- 6.4.2 Activated carbon, SKC Type 226–84 or equivalent, with appropriate end connectors and holders.
- 6.5 Tedlar bag. 24 liter, w/ Roberts valve, for GC/MS analysis of "breakthrough" VOC fraction as needed.
- 7.0 Reagents and Standards
- 7.1 Organic-free water, HPLC, or pharmaceutical grade.
- 7.2 Calibration Gases. The calibration gases for the gas analyzer shall be propane in air or propane in nitrogen. If organic compounds other than propane are used, the appropriate corrections for response factor must be available and applied to the results. Calibration gases shall be prepared in accordance with the procedure listed in Citation 2 of section 16. Additionally, the manufacturer of the cylinder must provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than  $\pm 2$  percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems (Test Method 205, 40 CFR Part 51, Appendix M), may be used with prior approval of the Administrator.
- 7.3 Fuel. A 40 percent H2/60 percent N2 or He gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.
- 7.4 Zero Gas. High purity air with less than 0.1 parts per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.
- 7.5 Low-level Calibration Gas. An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value.
- 7.6 Mid-level Calibration Gas. An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value.
- 7.7 High-level Calibration Gas. An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value.
- 8.0 Sample Collection, Preservation and Storage
- 8.1 Configuration. The configuration of the pre-survey sampling system is provided in Figure 1. This figure shows the primary components of the sampling system needed to conduct a VOC survey. A dual-channel analyzer is beneficial, but not necessary. Only a single channel is indicated in the figure.
- 8.2 Sampling. The pre-survey system should be set up and calibrated with the targeted sampling flow rate that will be used during Method 18 VOC sampling. The targeted flow rate for capture of most

expected VOC species is 400 cc/min. Since most FIA analyzers do not specifically allow for adjusting the total sample flow rate (only the back pressure), it may be necessary to insert a flow control valve at the sample inlet to the FIA. The total sample flow can be measured at the FIA bypass, since only a small fraction of the sample flow is diverted to analysis portion of the instrument.

The sampling system configuration shown in Figure 1 is operated using the process flow diagram provided in Figure 2. As noted in the process flowchart, the initial sampling media consists of the three midget impingers. The attenuation of the VOC sample stream is evaluated to determine if 95 percent or greater attenuation (capture) of the VOCs present has been achieved. The flow diagram specifies successive adjustments to the sampling media that are utilized to increase VOC capture.

A one-hour test of the final sampling configuration is performed using fresh media to ensure that significant breakthrough does not occur. Additional sampling media (more water, silica or carbon tubes) may be added to ensure that breakthrough is not occurring for the full duration of a test run.

If 95 percent or greater attenuation has not been achieved after inserting all indicated media, the most likely scenario is that methane is present. This is easily checked by collecting a sample of this final bypass sample stream and analyzing for methane. There are other VOC compounds which could also penetrate the media. Their identification by gas chromatography followed by mass spectrometry would be required if the breakthrough cannot be accounted for by the presence of methane.

#### 9.0 Quality Control

9.1 Blanks. A minimum of one method blank shall be prepared and analyzed for each sample medium employed during a presurvey testing field deployment to assess the effect of media contamination. Method blanks are prepared by assembling and charging the sample train with reagents, then recovering and preserving the blanks in the same manner as the test samples. Method blanks and test samples are stored, transported and analyzed in identical fashion as the test samples.

9.2 Synthetic Sample (optional). A synthetic sample may be used to assess the performance of the VOC characterization apparatus with respect to specific compounds. The synthetic sample is prepared by injecting appropriate volume(s) of the compounds of interest into a Tedlar bag containing a known volume of zero air or nitrogen. The contents of the bag are allowed to equilibrate, and the bag is connected to the sampling system. The sampling system, VOC characterization apparatus and FIA are operated normally to determine the performance of the system with respect to the VOC compounds present in the synthetic sample.

#### 10.0 Calibration and Standardization

10.1 Calibration. The FIA equipment is able to be calibrated for almost any range of total organic concentrations. For high concentrations of organics (>1.0 percent by volume as propane), modifications to most commonly available analyzers are necessary. One accepted method of equipment modification is to decrease the size of the sample to the analyzer through the use of a smaller diameter sample capillary. Direct and continuous measurement of organic concentration is a necessary consideration when determining any modification design.

#### 11.0 Procedure

11.1 Analytical Procedure. Upon completion of the pre-survey sampling, the sample fractions are to be analyzed by an appropriate chromatographic technique. (Ref: Method 18) The resulting chromatograms must be reviewed to ensure that the ratio of

known peak area to total peak area is 95% or greater. It should be noted that if formaldehyde is a suspected analyte, it must be quantitated separately using a different analytical technique.

#### 12.0 Data Analysis and Calculations

Chromatogram peaks will be ranked from greatest area to least area using peak integrator output. The area of all peaks will then be totaled, and the proportion of each peak area to the total area will be calculated. Beginning with the highest ranked area, each peak will be identified and the area added to previous areas until the cumulative area comprises at least 95% of the total area. The VOC compounds generating those identified peaks will comprise the compound list to be used in Method 18 testing of the subject source.

- 13.0 Method Performance [Reserved]
- 14.0 Pollution Prevention [Reserved]
- 15.0 Waste Management [Reserved]
- 16.0 References
- 16.1 CFR 40 Part 60, Appendix A, Method 18, Measurement of Gaseous Organic Compound Emissions by Gas Chromatography.
- 16.2 CFR 40 Part 60, Appendix A, Method 25A, Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer.
- 16.2 CFR 40 Part 60, Appendix A, Method 6, Determination of Sulfur Dioxide Emissions from Stationary Sources.
- 16.3 National Council for Air and Stream Improvement (NCASI), Method CI/WP–98.01 "Chilled Impinger Method for Use at Wood Products Mills to Measure Formaldehyde, Methanol, and Phenol.
- 17. Tables, Diagrams, Flowcharts, and Validation Data

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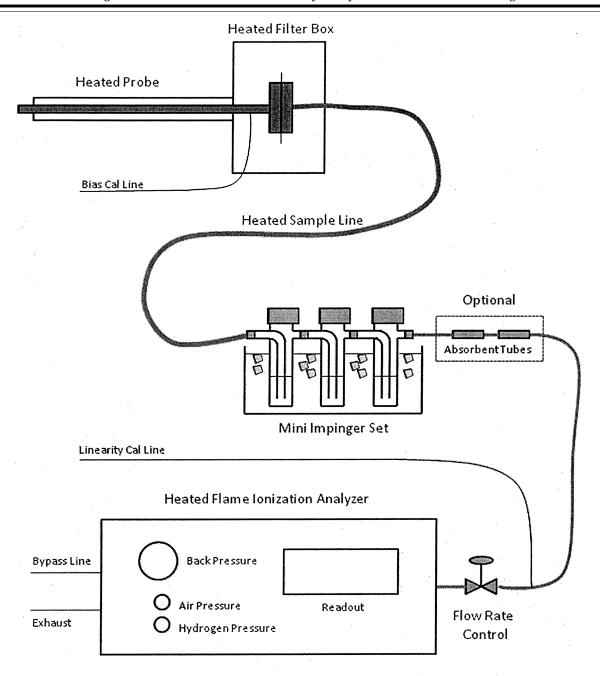
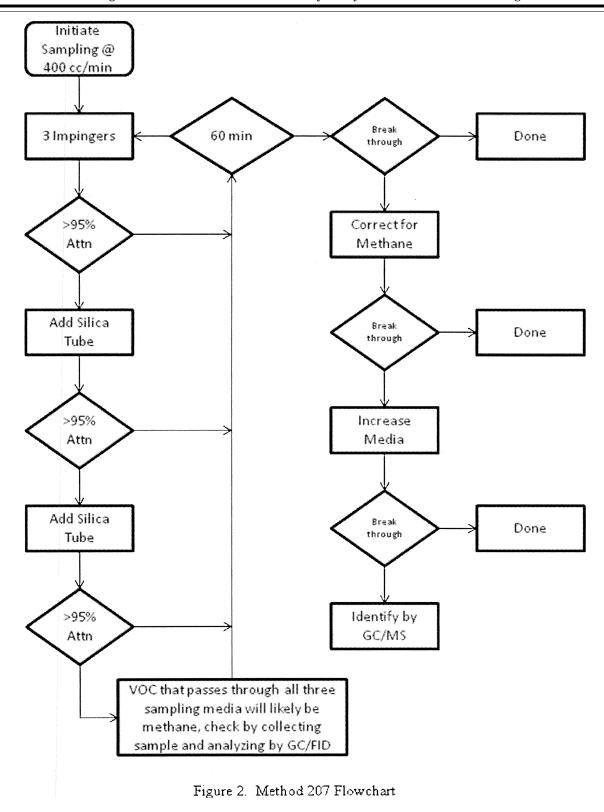


Figure 1. Pre-Survey Sampling System



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