**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Parts 51, 60, 61, and 63**

**[EPA–HQ–OAR–2014–0292; FRL–9950–57–OAR]**

**RIN 2060–AS34**

# Revisions to Test Methods, Performance Specifications, and Testing Regulations for Air Emission Sources

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

**ACTION:** Final rule.

**SUMMARY:** This action promulgates technical and editorial corrections and revisions to regulations related to source testing of emissions. We have made corrections and updates to testing provisions, and added newly approved alternatives to existing testing regulations. These revisions will improve the quality of data and provide flexibility in the use of approved alternative procedures. The revisions do not impose any new substantive requirements on source owners or operators.

**DATES:** The final rule is effective on October 31, 2016. The incorporation by reference materials listed in the rule are approved by the Director of the Federal Register as of October 31, 2016.

**ADDRESSES:** The EPA has established a docket for this action under Docket ID No. EPA–HQ–OAR–2014–0292. All documents in the docket are listed on the *http://www.regulations.gov* Web site. Although listed in the index, some information is not publicly available, *e.g.,* confidential business information or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, is not placed on the Internet and will be publicly available only in hard copy. Publicly available docket materials are available electronically through *http://www.regulations.gov.*

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## I. General Information

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

The revisions promulgated in this final rule apply to a large number of industries that are already subject to the current provisions of 40 Code of Federal Regulations (CFR) parts 51, 60, 61, and 63. For example, Performance Specification 4A applies to municipal waste combustors and hazardous waste incinerators. We did not list all of the specific affected industries or their North American Industry Classification System (NAICS) codes herein since there are many affected sources. If you have any questions regarding the applicability of this action to a particular entity, consult either the air permitting authority for the entity or your EPA Regional representative as listed in 40 CFR 63.13.

### *B. What action is the Agency taking?*

We are promulgating technical and editorial corrections and revisions to regulations related to source testing of emissions. More specifically, we are correcting typographical and technical errors, updating obsolete testing procedures, adding approved testing alternatives, and clarifying testing requirements.

### *C. Judicial Review*

Under section 307(b)(1) of the Clean Air Act (CAA), judicial review of this final rule is available by filing a petition for review in the United States Court of Appeals for the District of Columbia Circuit by October 31, 2016. Under section 307(d)(7)(B) of the CAA, only an objection to this final rule that was raised with reasonable specificity during the period for public comment can be raised during judicial review. Moreover, under section 307(b)(2) of the CAA, the requirements that are the subject of this final rule may not be challenged later in civil or criminal proceedings brought by the EPA to enforce these requirements.

## II. Background

The revisions to test methods, performance specifications, and testing regulations were proposed in the **Federal Register** on September 8, 2015 (80 FR 54146). The public comment period ended December 9, 2015, and 42 comment letters were received from the public. Changes were made to this final rule based on the public comments.

## III. Summary of Amendments

### *A. Appendix M of Part 51*

In paragraph (4)(a) of appendix M to part 51, Methods 30A and 30B are added to the list of methods not requiring the use of audit samples.

### *B. Method 201A of Appendix M of Part 51*

In Method 201A, the constant in equation 9 is corrected from 0.07657 to 0.007657.

### *C. Method 202 of Appendix M of Part 51*

In Method 202, section 3.8 is added to incorporate ASTM E617–13 by reference. The first sentence in section 8.5.4.3 is revised by adding ''back half of the filterable PM filter holder.'' Also, in section 8.5.4.3, sentences inadvertently omitted in the proposed rule are re-inserted. In section 9.10, the erroneous statement ''You must purge the assembled train as described in sections 8.5.3.2 and 8.5.3.3.'' is corrected to reference section 8.5.3. Sections 10.3 and 10.4 are added to require calibration of the field balance used to weigh impingers and to require a multipoint calibration of the analytical balance. In section 10.3, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any better than one-half of the tolerance for the measurement. Sections 11.2.2.1, 11.2.2.2, 11.2.2.3, 11.2.2.4 and figure 7 are re-inserted.

### *D. Appendix P of Part 51*

In appendix P of part 51, section 3.3, the erroneous reference to section 2.1 of Performance Specification 2 of appendix B of part 60 is corrected to section 6.1. Also, in section 3.3, the reference to the National Bureau of Standards is changed to the National Institute of Standards and Technology. In section 5.1.3, the erroneous reference to paragraph 4.1.4 is changed to reflect the correct reference to paragraphs 3.1.4 and 3.1.5.

### *E. General Provisions (Subpart A) of Part 60*

In the General Provisions of part 60, section 60.8(f) is revised to require the reporting of specific emissions test data in test reports. These data elements are required regardless of whether the report is submitted electronically or in paper format. Note that revisions are made to the data elements (that were listed in the proposed rule) to provide clarity and to more appropriately define and limit the extent of elements reported for each test method included in a test report. These modifications ensure that emissions test reporting includes all data necessary to assess and assure the quality of the reported emissions data and that the reported information appropriately describes and identifies the specific unit covered by the emissions test report. Section 60.17(g) is revised to add ASTM D6911–15 to the list of incorporations by reference.

### *F. Standards of Performance for Stationary Spark Ignition Internal Combustion Engines (Subpart JJJJ) of Part 60*

We received a request for a public hearing on this rule. We held a hearing in Research Triangle Park, North Carolina on October 8, 2015. All comments received at that hearing were related to our proposed revisions to subpart JJJJ, and a transcript of that hearing is available in the rule docket [EPA–HQ–OAR–2014–0292]. We also received a substantial number of comments from the public, both supportive of and in opposition to the revisions that we proposed.

At issue is the use of specific methodologies in a manner allowing a tester to speciate the volatile organic compounds (VOC) in the emissions and, from those speciated measurements, calculate a total VOC emissions rate using Fourier Transform Infrared Spectroscopy (FTIR using Method 320 or ASTM D6348–03) or Method 18, a measurement methodology that makes use of a combination of capture and analytical approaches. We proposed to remove Method 320 and ASTM D6348–03 as options for measuring VOC emissions under subpart JJJJ due to the lack of a consistent, demonstrable, and validated approach to measuring total VOC emissions. This decision was primarily due to the lack of a discrete list of compounds identified as those constituting the total VOC for the sources affected by subpart JJJJ. We proposed to eliminate the option to use these measurement approaches and leave Method 25A itself, a total hydrocarbon measurement approach, as the sole means of determining compliance with the total VOC emissions limits in the rule. We are concerned that implementation of Methods 320, ASTM D6348–03, and Method 18 does not provide proper and consistent quality assurance (QA) for compliance demonstration with total VOC measurement as required under subpart JJJJ.

Several commenters stated that prohibiting the use of FTIR to measure VOC and leaving Method 25A as the sole means of demonstrating compliance would result in an increased cost to industry. The commenters reasoned that this would decrease the number of tests that could be conducted in a single day because Method 25A requires more time to set up and run. We did not find compelling support for this argument. A properly conducted emissions test using FTIR technology and Method 320 or ASTM D6348–03 takes several hours to conduct, including time for equipment setup including the same sampling probe and heated sample transport line requirements as Method 25A, warmup which takes the same amount of time as Method 25A, conducting appropriate calibration and spiking data quality assessments very similar in duration to the required Method 25A calibration, actual source sampling time to span three 1-hour periods, leak tests, and post-test QA procedures common to each method. While it is possible to conduct two such test runs in a single 12- to 14-hour day, it is likewise possible to conduct two such test runs with Method 25A in that same time frame.

Several commenters also remarked that using FTIR is less complex, easier, and quicker than using Method 25A, but we do not find this argument sufficiently compelling to reverse our proposed revisions. We understand that while an experienced spectroscopist can operate an FTIR with relative ease as compared to a novice, the process of quality assuring emissions data measured by FTIR in accordance with Method 320 or ASTM D6348–03 is not a trivial matter. Calibration checks and matrix spiking of target compounds, including the ''most difficult to recover'' compound (as required by Method 320), is both challenging and time consuming due to the need to rule out interferences that may be caused by the emissions gas matrix while working to individually quantify each VOC in that matrix. In summation, we do not agree that the use of FTIR for quantification of total VOC is quick, easy or less expensive to

conduct when compared with the use of Method 25A.

Several commenters provided information to the docket, and others stated individually during the public hearing that they have provided a list of VOC to the docket, or have compiled a list of VOC or recommend that EPA address the FTIR measurement issue through the agency providing a list of VOC that make up 95 percent of the emissions from natural gas-fired spark ignition (SI) engines. We agree with commenters that a list of VOC could be developed; however, we recognize that the list must represent total VOC (all the VOC that could be emitted from SI engines affected by subpart JJJJ), as that is the compliance requirement stated in the rule. We have not stated that 95 percent of the VOC emissions are the target goal for such a list. In a memo to the docket of this rule (Technical memorandum dated September 28, 2015, to Docket ID No. EPA–HQ–OAR–2014–0292 titled, ''Proposal to remove Methods 18, 320, and ASTM D6348–03 as Acceptable Methods for Measuring Total VOC Under 40 CFR 60, Subpart JJJJ''), we state that we are actively seeking sufficient documentation to create a complete list of VOC to support a speciated hydrocarbon measurement approach such as FTIR and/or Method 18. We received data from commenters that moves us toward compiling such a list, but we did not receive sufficient demonstration that all VOC were represented in that list. Additionally, while we received information on VOC present in well-operated and controlled engines, the data does not include VOC that may be present largely during, or only during, poor performance periods and could, thereby, serve as key indicators of engines that are not well-operated, well-controlled, or in compliance with the applicable standard. Therefore, we remain unable to define a complete list of VOC that would need to be quantified by a speciated measurement approach to demonstrate that total VOC were measured during a compliance test. Even so, we are swayed by arguments such as those made in support of speciated measurement approaches, specifically their ability to account for methane and ethane as separate quantifiable emissions.

Two commenters remarked that they do not believe that Method 25A is able to produce accurate total VOC values because there is an inherent issue with the ''difference or subtraction'' method when applied to compressed natural gas (CNG)-based emissions. We reviewed the data provided by the commenters in this respect and did not arrive at the same conclusion. Our review shows that the commenters appear to double-count some of the emissions in arriving at their results and do not present compelling evidence that demonstrates the ability of a hydrocarbon cutter to remove all ethane from the measured gas.

Two commenters stated that FTIR can measure real-time non-methane, non-ethane VOC. We agree that this speciated approach is capable of providing emissions data for methane, ethane, and other VOC in near-real-time.

One commenter recommended that we allow FTIR methods since FTIR is the only technology that can provide a mass emissions rate and since FTIR does not have a zero drift nor calibration drift problem like Method 25A. Subpart JJJJ requires the calculation of a mass emissions rate on a propane basis and Method 25A, calibrated with propane and using the molecular weight of propane (44.01 lb/lb-mol) for mass emissions calculations, is quite capable of providing a mass emissions rate appropriate for determination of compliance with the VOC standards in subpart JJJJ. In regard to zero drift, Method 25A has QA and quality control (QC) criteria to limit the acceptance of data where instrument drift is excessive.

Three commenters noted that we did not provide supporting data for proposing to disallow FTIR methods that have been allowed under subpart JJJJ for the past 7 years. We submitted a supporting memo to the docket (Technical memorandum dated September 28, 2015, to Docket ID No. EPA–HQ–OAR–2014–0292 titled, ''Proposal to Remove EPA Methods 18, 320, and ASTM D6348–03 as Acceptable Methods for Measuring Total VOC Under 40 CFR 60, Subpart JJJJ'') that provides the reasoning and justification for our proposal.

One commenter recommended that changes to subpart JJJJ test methods be proposed as a separate rulemaking under subpart JJJJ. We believe that we have the authority to make necessary or otherwise appropriate changes to a specific test procedure or pollutant measurement requirement in a rule through this periodic rulemaking.

One commenter agreed with our proposed position that FTIR should not be used to measure total VOC, but remarked that Method 18 should continue to be allowed since it allows direct measurement of VOC constituents using gas chromatography and does not rely on differential methods or require multiple test methods. We found the latter arguments and reasoning to be persuasive and compelling. Method 18 does contain provisions to screen and calibrate for VOC present in the emissions and thereby measure total VOC from a specific source. While this can be a complex and sometimes tedious undertaking, we recognize that it is an appropriate approach to measure total VOC from a specific source and are modifying the final rule language to reflect that this is allowable.

Two additional commenters agreed with our proposed position that the current FTIR methodologies are not adequately measuring total VOC. One of the commenters remarked that testers do not provide adequate total VOC results. The other commenter recommended only allowing FTIR if the QA is complete and accurate and if all VOC are proven to be accounted for. We are swayed by this commenter's support for complete QA/QC of data and stipulation that all VOC are proven to be accounted for. Although we do not currently possess sufficient data to compile a complete list of VOCs expected to be emitted from SI engines, we believe that where data with complete QA/QC are available, we may acquire sufficient data over time.

This action finalizes requirements to clarify the conduct of QA/QC procedures and report the QA/QC data with the emissions measurement data when applying Method 320 and ASTM D6348–03. We will revisit this decision and make a subsequent determination of the appropriateness for the use of Method 320 and/or ASTM–D6348 during the first risk and technology review evaluation for this sector.

In Table 2 of subpart JJJJ, the allowances to use Method 320 and ASTM D6348–03 are retained. The language requiring the reporting of specific QA/QC data when these test methods are used has been added to paragraph 60.4245(d).

The typographical error in the proposed Table 2 of subpart JJJJ is corrected; ''methane cutter'' is replaced with ''hydrocarbon cutter'' in paragraph (5) of section c.

*G. Method 1 of Appendix A–1 of Part 60*

In Method 1, section 11.2.1.2, the word ''istances'' is changed to ''distances'' in the second sentence, and the last two sentences in this section (inadvertently omitted in the proposed rule) are re-inserted. The second figure labeled Figure 1–2 is deleted because two figures labeled Figure 1–2 were inadvertently included.

*H. Method 2 of Appendix A–1 of Part 60*

In Method 2, instructions are given for conducting S-type pitot calibrations. Currently, the same equipment is commonly used for both Methods 2 and

2G (same S-type pitot), but the calibration procedure is slightly different in each method. Other key pieces that enhance the QA/QC of the calibrations are added to Method 2, and the amount of blockage allowed is reduced to improve calibration accuracy. To address these issues, changes are made to sections 6.7, 10.1.2.3, 10.1.3.4, 10.1.3.7, and 10.1.4.1.3 of Method 2. Sentences in section 6.7 (inadvertently omitted in the proposed rule) are re-inserted. In section 10.1.4.3, the erroneous reference to section 10.1.4.4 is corrected to section 12.4.4. The portion of Figure 2–10 labeled (b) is deleted because it is erroneous, and the label (a) is removed from the figure.

*I. Method 2G of Appendix A–2 of Part 60*

In Method 2G, instructions are given for conducting S-type pitot calibrations. Currently, the same equipment is commonly used for both Methods 2 and 2G (same S-type pitot), but the calibration procedure is slightly different in each method. Other key pieces that enhance the QA/QC of the calibrations are added to the method, and the amount of blockage allowed is reduced to tighten up calibration accuracy. Changes are made to sections 6.11.1, 6.11.2, 10.6.6, and 10.6.8 of Method 2G to address these issues. In section 10.6.6, the proposed language regarding recording rotational speed is revised based on a public comment.

*J. Method 3C of Appendix A–2 of Part 60*

In Method 3C, section 6.3 is revised to add subsections (6.3.1, 6.3.2, 6.3.3, 6.3.4, and 6.3.5) that clarify the requirements necessary to check analyzer linearity.

*K. Method 4 of Appendix A–3 of Part 60*

In Method 4, section 10.3 (Field Balance) is added to require calibration of the balance used to weigh impingers. In section 10.3, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any better than one-half of the tolerance for the measurement. Section 12.2.5, which gives another option for calculating the approximate moisture content, is added. Section 16.4 is revised to clarify that a fuel sample must be taken and analyzed to develop F-factors required by the alternative procedure. Also, in section 16.4, percent relative humidity is inadvertently defined as ''calibrated hydrometer acceptable''; the word ''hydrometer'' is replaced with ''hygrometer.''

*L. Method 5 of Appendix A–3 of Part 60*

In Method 5, we erroneously finalized the reference to the Isostack metering system in 79 FR 11228. Therefore, this reference from section 6.1.1.9 is removed. Broadly applicable test method determinations or letters of assessments, regarding whether specific alternative metering equipment meets the specifications of the method as was our intent in the ''Summary of Comments and Responses on Revisions to Test Methods and Testing Regulations'' (EPA–HQ–OAR–2010–0114–0045), will continue to be issued. In section 6.1.1.9, the parenthetical phrase ''(rechecked at least one point after each test)'' is removed since the requirements for temperature sensors are given in section 10.5 of Method 5. The phrase ''after ensuring that all joints have been wiped clean of silicone grease'' is removed from section 8.7.6.2.5. Sections 10.7 and 10.8 are added to require calibration of the balance used to weigh impingers and to require a multipoint calibration of the analytical balance. In section 10.7, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any better than one-half of the tolerance for the measurement. In section 10.8, the proposed language is revised to ''Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1 g and 5 g.''

*M. Method 5H of Appendix A–3 of Part 60*

In Method 5H, sections 10.4 and 10.5 are added to require calibration of the field balance used to weigh impingers and to require a multipoint calibration of the analytical balance. In section 10.4, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any better than one-half of the tolerance for the measurement. In section 10.5, the proposed language is revised to ''Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1 g and 5 g.''

*N. Method 5I of Appendix A–3 of Part 60*

In Method 5I, sections 10.1 and 10.2 are added to require calibration of the field balance used to weigh impingers and to require a multipoint calibration of the analytical balance. In section 10.1, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any more accurate than one-half of the tolerance for the measurement. In section 10.2, the proposed language is revised to ''Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1 g and 5 g.''

*O. Method 6C of Appendix A–4 of Part 60*

In Method 6C, the language detailing the methodology for performing interference checks in section 8.3 is revised to clarify and streamline the procedure. While we continue to believe that quenching can be an issue for fluorescence analyzers, the language regarding quenching that was promulgated on February 27, 2014, has raised many questions and is being removed. It is our opinion that the interference check, if done properly, using sulfur dioxide ($SO_2$) and both levels of carbon dioxide ($CO_2$) as specified in Table 7E–3 of Method 7E, will evaluate effects due to quenching. We will continue to evaluate data as it becomes available and propose additional language, as needed. However, if you believe that quenching is an issue, we recommend that you repeat the interference check using the $CO_2$ values specified in Table 7E–3 and an $SO_2$ value similar to your measured stack emissions.

*P. Method 7E of Appendix A–4 of Part 60*

In Method 7E, section 8.1.2, the requirements/specifications for the 3-point sampling line are revised to be consistent with Performance Specification 2; the new requirement is 0.4, 1.2, and 2.0 meters.

The language in section 8.2.7 regarding quenching that was promulgated on February 27, 2014, has raised many questions, and is being

removed at this time. It is our opinion that the interference check, if done properly, using the gas levels specified in Table 7E–3 of Method 7E, will evaluate analyzer bias. We will continue to evaluate data as it becomes available and propose additional language in the future as needed. However, if you feel that analyzer bias is an issue, we recommend that you repeat the interference check using calibration gas values similar to your measured stack emissions. The language in section 8.2.7 requiring that the interference check be performed periodically or after major repairs has also been removed to be consistent with the language found in section 8.2.7 (2), which states ''This interference test is valid for the life of the instrument unless major analytical components (*e.g.,* the detector) are replaced with different model parts.''

The word ''equations'' is replaced with ''equation'' in the sentence in section 12.8 that reads ''If desired, calculate the total $NO_X$ concentration with a correction for converter efficiency using equation 7E–8.''

We requested and received comments on the stratification test in Method 7E. We will consider the comments and propose changes in a future rulemaking.

*Q. Method 10 of Appendix A–4 of Part 60*

In Method 10, sections 6.2.5 and 8.4.2 are revised, and section 6.2.6 is added to clarify the types of sample tanks allowed for integrated sampling.

*R. Methods 10A and 10B of Appendix A–4 of Part 60*

Methods 10A and 10B are revised to allow the use of sample tanks as an alternative to flexible bags for sample collection.

*S. Method 15 of Appendix A–5 of Part 60*

In Method 15, section 8.3.2 is revised to clarify the calibrations that represent partial calibration.

*T. Method 16C of Appendix A–6 of Part 60*

In Method 16C, section 12.2, equation 16C–1 is revised to replace $C_v$ (manufacturer certified concentration of a calibration gas in ppmv $SO_2$) in the denominator with CS (calibration span in ppmv). The definition of CS is added to the nomenclature in section 12.1, and the definition of $C_v$ is retained in the nomenclature in section 12.1 because $C_v$ is in the numerator of equation 16C–1.

*U. Method 18 of Appendix A–6 of Part 60*

In Method 18, section 8.2.1.5.2.3 is removed because the General Provisions to Part 60 already include a requirement to analyze two field audit samples as described in section 9.2.

*V. Method 25C of Appendix A–7 of Part 60*

In Method 25C, section 9.1 is corrected to reference section 8.4.2 instead of section 8.4.1. Section 11.2 is deleted because the audit sample analysis is now covered under the General Provisions to Part 60. The nomenclature is revised in section 12.1, and equation 25C–2 is revised in section 12.3. Sections 12.4, 12.5, 12.5.1, and 12.5.2 are added to incorporate equations to correct sample concentrations for ambient air dilution. In section 12.5.2, the reference to equation 25C–4 is corrected to 25C–5.

*W. Method 26 of Appendix A–8 of Part 60*

In Method 26, section 13.3 is revised to indicate the correct method detection limit; the equivalent English unit for the metric quantity is added.

*X. Method 26A of Appendix A–8 of Part 60*

In Method 26A, language regarding minimizing chloride interferences is added to section 4.3. Also in section 4.3, the first sentence (inadvertently omitted in the proposed rule) is re-inserted.

Sections 6.1.7 and 8.1.5 are not changed in this final rule. The language in the proposed rule that revised the required probe and filter temperature requirements in sections 6.1.7 and 8.1.5 to allow a lower probe and filter temperature was an error.

In section 8.1.6, the typographical error, ''. . . between 120 and 134 °C (248 and 275 °F . . .''), is corrected to ''. . . between 120 and 134 °C (248 and 273 °F . . .'').

*Y. Method 29 of Appendix A–8 of Part 60*

In Method 29, section 8.2.9.3 is revised to require rinsing impingers containing permanganate with hydrogen chloride (HCl) to ensure consistency with the application of Method 29 across various stationary source categories and because there is evidence that HCl is needed to release the mercury (Hg) bound in the precipitate from the permanganate. Sections 10.4 and 10.5 are added to require calibration of the field balance used to weigh impingers and to require a multipoint calibration of the analytical balance. In section 10.4, the proposed language is revised to allow the use of a Class 6 tolerance weight (or better) in lieu of the proposed Class 3 (or better) tolerance weight for checking the field balance accuracy because the calibration weight does not need to be any better than one-half of the tolerance for the measurement.

*Z. Method 30A of Appendix A–8 of Part 60*

In Method 30A, the heading of section 8.1 is changed from ''Sample Point Selection'' to ''Selection of Sampling Sites and Sampling Points.''

*AA. Method 30B of Appendix A–8 of Part 60*

In Method 30B, the heading of section 8.1 is changed from ''Sample Point Selection'' to ''Selection of Sampling Sites and Sampling Points.'' In section 8.3.3.8, the reference to ASTM WK223 is changed to ASTM D6911–15, and the last two sentences in this section (inadvertently omitted in the proposed rule) are re-inserted.

*BB. Appendix B to Part 60—Performance Specifications*

In the index to appendix B to part 60, Performance Specification 16—Specifications and Test Procedures for Predictive Emission Monitoring Systems in Stationary Sources is added.

*CC. Performance Specification 1 of Appendix B of Part 60*

In Performance Specification 1, paragraph 8.1(2)(i) is revised in order to not limit the location of a continuous opacity monitoring system (COMS) to a point at least four duct diameters downstream and two duct diameters upstream from a control device or flow disturbance. Paragraph 8.1(2)(i) refers to paragraphs 8.1(2)(ii) and 8.1(2)(iii) for additional options.

*DD. Performance Specification 2 of Appendix B of Part 60*

In Performance Specification 2, the definition of span value is revised in section 3.11. The sentence, ''For spans less than 500 ppm, the span value may either be rounded upward to the next highest multiple of 10 ppm, or to the next highest multiple of 100 ppm such that the equivalent emissions concentration is not less than 30 percent of the selected span value.'', is added to section 3.11. Also, in section 6.1.1, the data recorder language is revised. In section 6.1.2, the term ''high-level'' is changed to ''span'' to be consistent with the definition of span value discussed above. In section 16.3.2, the characters ''|dverbar'' are replaced with $\overline{d}$ which is the average difference between

responses and the concentration/responses. In section 18, Table 2–2 is detached from Figure 2–1, and the figure is clearly labeled as ''Calibration Drift Determination.''

*EE. Performance Specification 3 of Appendix B of Part 60*

In Performance Specification 3, section 13.2 is revised to clarify how to calculate relative accuracy. The absolute value symbol is added to the proposed definition of absolute value of the mean of the differences.

*FF. Performance Specification 4A of Appendix B of Part 60*

In Performance Specification 4A, the response time test procedure in sections 8.3 and 8.3.1 is revised. In section 8.3.1, the next to the last sentence is re-worded to ''Repeat the entire procedure until you have three sets of data to determine the mean upscale and downscale response times.'' Also, the proposed response time requirement in section 13.3 is revised to 240 seconds.

*GG. Performance Specification 11 of Appendix B of Part 60*

In Performance Specification 11, equations 11–1 and 11–2 are revised in section 12.1, and the response range is used in lieu of the upscale value in section 13.1. In section 12.1, the sentence in paragraph (3) that was inadvertently omitted is re-inserted.

*HH. Performance Specification 15 of Appendix B of Part 60*

In Performance Specification 15, the statement, ''An audit sample is obtained from the Administrator,'' is deleted from paragraph 9.1.2. Also, in Performance Specification 15, reserved sections 14.0 and 15.0 are added.

*II. Performance Specification 16 of Appendix B of Part 60*

In Performance Specification 16, Table 16–1 is changed to be consistent with conventional statistical applications; the values listed in the column labelled $n-1$ (known as degrees of freedom) are corrected to coincide with standard t-tables, and the footnote is clarified. Section 12.2.3 is revised for selection of $n-1$ degrees of freedom.

*JJ. Procedure 2 of Appendix F of Part 60*

In Procedure 2, equations 2–2 and 2–3 in section 12.0 are revised to correctly define the denominator when calculating calibration drift. Also, equation 2–4 in section 12.0 is revised to correctly define the denominator when calculating accuracy. The proposed equation 2–4 is revised to:

$$\text{Accuracy} = \frac{|V_M - V_R|}{V_R} \times 100$$

*KK. General Provisions (Subpart A) of Part 61*

Section 61.13(e)(1)(i) of the General Provisions of Part 61 is revised to add Methods 30A and 30B to the list of methods not requiring the use of audit samples.

*LL. Method 107 of Appendix B of Part 61*

In Method 107, the term ''Geon'' is deleted from the heading in section 11.7.3.

*MM. General Provisions (Subpart A) of Part 63*

In the General Provisions of Part 63, section 63.7(c)(2)(iii)(A) is revised to add Methods 30A and 30B to the list of methods not requiring the use of audit samples.

Section 63.7(g)(2) is revised to require the reporting of specific emissions test data in test reports. These data elements are required regardless of whether the report is submitted electronically or in paper format. Revisions are made to the list of proposed data elements to provide clarity and to more appropriately define and limit the extent of elements reported for each test method included in a test report. These modifications ensure that emissions test reporting includes all data necessary to assess and assure the quality of the reported emissions data and that the reported information appropriately describes and identifies the specific unit covered by the emissions test report.

*NN. Method 320 of Appendix A of Part 63*

In Method 320, sections 13.1, 13.4, and 13.4.1 are revised to indicate the correct Method 301 reference.

## IV. Public Comments on the Proposed Rule

Forty-two comment letters were received on the proposed rule. The public comments and the agency's responses are summarized in the Summary of Comments and Responses document located in the docket for this rule. See the **ADDRESSES** section of this preamble.

## V. Statutory and Executive Order Reviews

*A. Executive Order 12866: Regulatory Planning and Review and Executive Order 13563: Improving Regulation and Regulatory Review*

This action is not a ''significant regulatory action'' under the terms of Executive Order (E.O.) 12866 (58 FR 51735, October 4, 1993) and is, therefore, not subject to review under Executive Orders 12866 and 13563 (76 FR 3821, January 21, 2011).

*B. Paperwork Reduction Act (PRA)*

This action does not impose an information collection burden under the PRA. This action does not add information collection requirements; it makes corrections and updates to existing testing methodology. In addition, this action clarifies performance testing requirements.

*C. Regulatory Flexibility Act (RFA)*

I certify that this action will not have a significant economic impact on a substantial number of small entities under the RFA. In making this determination, the impact of concern is any significant adverse economic impact on small entities. 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, has no net burden or otherwise has a positive economic effect on the small entities subject to the rule. This action will not impose emission measurement requirements beyond those specified in the current regulations, nor does it change any emission standard. We have, therefore, concluded that this action will have no net regulatory burden for all directly regulated small entities.

*D. Unfunded Mandates Reform Act (UMRA)*

This action does not contain any unfunded mandate as described in UMRA, 2 U.S.C. 1531–1538, and does not significantly or uniquely affect small governments. The action imposes no enforceable duty on any state, local or tribal governments or the private sector.

*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.

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

This action does not have tribal implications, as specified in Executive Order 13175. This action simply corrects and updates existing testing regulations. Thus, Executive Order 13175 does not apply to this action.

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

The EPA interprets Executive Order 13045 as applying only to those regulatory actions that concern environmental health or safety risks that the EPA has reason to believe may disproportionately affect children, per the definition of ''covered regulatory action'' in section 2–202 of the Executive Order. This action is not subject to Executive Order 13045 because it does not concern an environmental health risk or safety risk.

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

This action is not subject to Executive Order 13211, because it is not a significant regulatory action under Executive Order 12866.

*I. National Technology Transfer and Advancement Act (NTTAA) and 1 CFR Part 51*

This action involves technical standards. The EPA used ASTM D6911–15 for packaging and shipping samples in Method 30B. The ASTM D6911–15 standard provides guidance on the selection of procedures for proper packaging and shipment of environmental samples to the laboratory for analysis to ensure compliance with appropriate regulatory programs and protection of sample integrity during shipment.

The EPA used ASTM E617–13 for laboratory weights and precision mass standards in Methods 4, 5, 5H, 5I, 29, and 202. The ASTM E617–13 standard covers weights and mass standards used in laboratories for specific classes.

The ASTM D6911–15 and ASTM E617–13 standards were developed and adopted by the American Society for Testing and Materials (ASTM). These standards may be obtained from *http://www.astm.org* or from the ASTM at 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959.

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

The EPA believes that this action is not subject to Executive Order 12898 (59 FR 7629, February 16, 1994) because it does not establish an environmental health or safety standard. This action is a technical correction to previously promulgated regulatory actions and does not have an impact on human health or the environment.

*K. Congressional Review Act (CRA)*

This action is subject to the CRA, and the EPA will submit a rule report to each house of the Congress and to the Comptroller General of the United States. This action is not a ''major rule'' as defined by 5 U.S.C. 804(2).

**List of Subjects**

*40 CFR Part 51*

Environmental protection, Administrative practice and procedure, Air pollution control, Incorporation by reference, Reporting and recordkeeping requirements, Volatile organic compounds.

*40 CFR Part 60*

Environmental protection, Administrative practice and procedure, Air pollution control, Incorporation by reference, Volatile organic compounds.

*40 CFR Parts 61 and 63*

Environmental protection, Administrative practice and procedure, Air pollution control, Reporting and recordkeeping requirements.

Dated: August 5, 2016.

**Gina McCarthy,**

*Administrator.*

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

## PART 51—REQUIREMENTS FOR PREPARATION, ADOPTION, AND SUBMITTAL OF IMPLEMENTATION PLANS

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

**Authority:** 23 U.S.C. 101; 42 U.S.C. 7401–7671q.

■ 2. Amend appendix M to part 51 as follows:
■ a. Revise section 4.0a.
■ b. Revise section 12.5, equations 8 and 9, in Method 201A.
■ c. In Method 202:
■ i. Add section 3.8.
■ ii. Revise sections 8.5.4.3 and 9.10.
■ iii. Add sections 10.3, 10.4, 11.2.2.1, 11.2.2.2, 11.2.2.3, and 11.2.2.4.
■ iv. Add Figure 7 to section 18.0.

The additions and revisions read as follows:

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

* * * * *

*4.0 * * **

a. The source owner, operator, or representative of the tested facility shall obtain an audit sample, if commercially available, from an AASP for each test method used for regulatory compliance purposes. No audit samples are required for the following test methods: Methods 3A and 3C of appendix A–3 of part 60 of this chapter, Methods 6C, 7E, 9, and 10 of appendix A–4 of part 60, Methods 18 and 19 of appendix A–6 of part 60, Methods 20, 22, and 25A of appendix A–7 of part 60, Methods 30A and 30B of appendix A–8 of part 60, and Methods 303, 318, 320, and 321 of appendix A of part 63 of this chapter. If multiple sources at a single facility are tested during a compliance test event, only one audit sample is required for each method used during a compliance test. The compliance authority responsible for the compliance test may waive the requirement to include an audit sample if they believe that an audit sample is not necessary. ''Commercially available'' means that two or more independent AASPs have blind audit samples available for purchase. If the source owner, operator, or representative cannot find an audit sample for a specific method, the owner, operator, or representative shall consult the EPA Web site at the following URL, *http://www.epa.gov/ttn/emc,* to confirm whether there is a source that can supply an audit sample for that method. If the EPA Web site does not list an available audit sample at least 60 days prior to the beginning of the compliance test, the source owner, operator, or representative shall not be required to include an audit sample as part of the quality assurance program for the compliance test. When ordering an audit sample, the source owner, operator, or representative shall give the sample provider an estimate for the concentration of each pollutant that is emitted by the source or the estimated concentration of each pollutant based on the permitted level and the name, address, and phone number of the compliance authority. The source owner, operator, or representative shall report the results for the audit sample along with a summary of the emissions test results for the audited pollutant to the compliance authority and shall report the results of the audit sample to the AASP. The source owner, operator, or representative shall make both reports at the same time and in the same manner or shall report to the compliance authority first and then report to the AASP. If the method being audited is a method that allows the samples to be analyzed in the field, and the tester plans to analyze the samples in the field, the tester may analyze the audit samples prior to collecting the emission samples provided a representative of the compliance authority is present at the testing site. The tester may request and the compliance authority may grant a waiver to the requirement that a representative of the compliance authority must be present at the testing site during the field analysis of an audit sample. The source owner, operator, or representative may report the results of the audit sample to the compliance authority and then report the results of the audit sample to the AASP prior to collecting any emission samples. The test protocol and final test report shall document whether an audit sample was ordered and

utilized and the pass/fail results as applicable.

* * * * *

**Method 201A—Determination of $PM_{10}$ and $PM_{2.5}$ Emissions From Stationary Sources (Constant Sampling Rate Procedure)**

* * * * *

12.5 * * *

For $N_{re}$ less than 3,162:

$$Q_{IV} = 0.0060639 \left[\frac{\mu}{C^{0.4242}}\right] \left[\frac{P_s M_w}{T_s}\right]^{-0.5759} \left[\frac{1}{D_{50}}\right]^{0.8481} \quad \text{(Eq. 8)}$$

For $N_{re}$ greater than 3,162:

$$Q_{IV} = 0.007657 \left[\frac{\mu}{C^{0.6205}}\right] \left[\frac{P_s M_w}{T_s}\right]^{-0.3795} \left[\frac{1}{D_{50}}\right]^{01.241} \quad \text{(Eq. 9)}$$

* * * * *

**Method 202—Dry Impinger Method for Determining Condensable Particulate Emissions From Stationary Sources**

* * * * *

3.8 *ASTM E617–13.* ASTM E617–13 ''Standard Specification for Laboratory Weights and Precisions Mass Standards,'' approved May 1, 2013, was developed and adopted by the American Society for Testing and Materials (ASTM). The standards cover weights and mass standards used in laboratories for specific classes. The ASTM E617–13 standard has been approved for incorporation by reference by the Director of the Office of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. The standard may be obtained from *http://www.astm.org* or from the ASTM at 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428–2959. All approved material is available for inspection at EPA WJC West Building, Room 3334, 1301 Constitution Ave. NW., Washington, DC 20460, telephone number 202–566–1744. It is also available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030 or go to *http://www.archives.gov/federal_register/code_of_federal_regulattions/ibr_locations.html.*

* * * * *

8.5.4.3 *CPM Container #2, Organic rinses.* Follow the water rinses of the back half of the filterable PM filter holder, probe extension, condenser, each impinger, and all of the connecting glassware and front half of the CPM filter with an acetone rinse. Recover the acetone rinse into a clean, leak-proof container labeled with test identification and ''CPM Container #2, Organic Rinses.'' Then repeat the entire rinse procedure with two rinses of hexane, and save the hexane rinses in the same container as the acetone rinse (CPM Container #2). Mark the liquid level on the jar.

* * * * *

9.10 Field Train Recovery Blank. You must recover a minimum of one field train blank for each source category tested at the facility. You must recover the field train blank after the first or second run of the test. You must assemble the sampling train as it will be used for testing. Prior to the purge, you must add 100 ml of water to the first impinger and record this data on Figure 4. You must purge the assembled train as described in section 8.5.3. You must recover field train blank samples as described in section 8.5.4. From the field sample weight, you will subtract the condensable particulate mass you determine with this blank train or 0.002 g (2.0 mg), whichever is less.

* * * * *

10.3 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' Class 6 (or better). Daily before use, the field balance must measure the weight within ± 0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.4 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5mg of the certified mass, perform corrective measures, and conduct the multipoint calibration before use.

* * * * *

11.2.2.1 Determine the inorganic fraction weight. Transfer the aqueous fraction from the extraction to a clean 500-ml or smaller beaker. Evaporate to no less than 10 ml liquid on a hot plate or in the oven at 105 °C and allow to dry at room temperature (not to exceed 30 °C (85 °F)). You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight. (See section 3.0 for a definition of constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of section 18) and proceed directly to section 11.2.3. If the residue cannot be weighed to constant weight, re-dissolve the residue in 100 ml of deionized distilled ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less and continue to section 11.2.2.2.

11.2.2.2 Use titration to neutralize acid in the sample and remove water of hydration. If used, calibrate the pH meter with the neutral and acid buffer solutions. Then titrate the sample with 0.1N $NH_4OH$ to a pH of 7.0, as indicated by the pH meter or colorimetric indicator. Record the volume of titrant used on the CPM Work Table (see Figure 6 of section 18).

11.2.2.3 Using a hot plate or an oven at 105 °C, evaporate the aqueous phase to approximately 10 ml. Quantitatively transfer the beaker contents to a clean, 50-ml pre-tared weighing tin and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing

anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight. (See section 3.0 for a definition of constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of section 18).

11.2.2.4 Calculate the correction factor to subtract the $NH_4^+$ retained in the sample using Equation 1 in section 12.

* * * * *

*18.0 Tables, Diagrams, Flowcharts and Validation Data*

* * * * *

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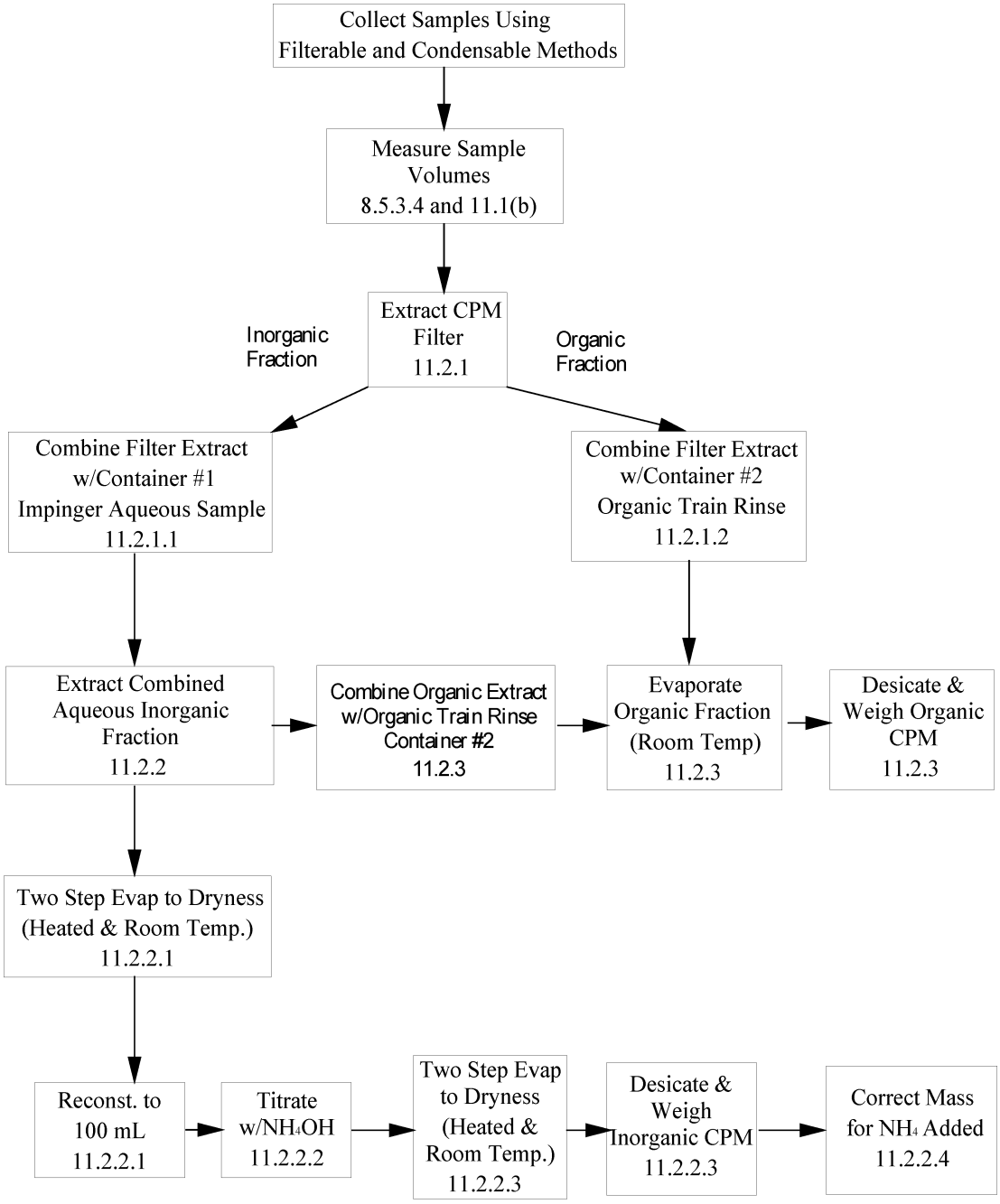


**Figure 7. CPM Sample Processing Flow Chart**

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* * * * *

■ 3. Revise sections 3.3 and 5.1.3 of appendix P to part 51 to read as follows:

**Appendix P to Part 51—Minimum Emission Monitoring Requirements**

* * * * *

3.3 *Calibration Gases.* For nitrogen oxides monitoring systems installed on fossil fuel-fired steam generators, the pollutant gas used to prepare calibration gas mixtures (section 6.1, Performance Specification 2, appendix B, part 60 of this chapter) shall be nitric oxide (NO). For nitrogen oxides monitoring systems

installed on nitric acid plants, the pollutant gas used to prepare calibration gas mixtures (section 6.1, Performance Specification 2, appendix B, part 60 of this chapter) shall be nitrogen dioxide ($NO_2$). These gases shall also be used for daily checks under paragraph 3.7 of this appendix as applicable. For sulfur dioxide monitoring systems installed on fossil fuel-fired steam generators or sulfuric acid plants, the pollutant gas used to prepare calibration gas mixtures (section 6.1, Performance Specification 2, appendix B, part 60 of this chapter) shall be sulfur dioxide ($SO_2$). Span and zero gases should be traceable to National Bureau of Standards reference gases whenever these reference gases are available. Every 6 months from date of manufacture, span and zero gases shall be reanalyzed by conducting triplicate analyses using the reference methods in appendix A, part 60 of this chapter as follows: for $SO_2$, use Reference Method 6; for nitrogen oxides, use Reference Method 7; and for carbon dioxide or oxygen, use Reference Method 3. The gases may be analyzed at less frequent intervals if longer shelf lives are guaranteed by the manufacturer.

* * * * *

5.1.3 The values used in the equations under paragraph 5.1 are derived as follows:

E = pollutant emission, g/million cal (lb/million BTU),

C = pollutant concentration, g/dscm (lb/dscf), determined by multiplying the average concentration (ppm) for each hourly period by $4.16 \times 10^{-5}$ M g/dscm per ppm ($2.64 \times 10^{-9}$ M lb/dscf per ppm) where M = pollutant molecular weight, g/g-mole (lb/lb-mole). M = 64 for sulfur dioxide and 46 for oxides of nitrogen.

$\%O_2$, $\%CO_2$ = Oxygen or carbon dioxide volume (expressed as percent) determined with equipment specified under paragraphs 3.1.4 and 3.1.5 of this appendix.

* * * * *

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

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

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

■ 5. In § 60.8, revise paragraph (f) to read as follows:

### § 60.8 Performance tests.

* * * * *

(f) Unless otherwise specified in the applicable subpart, each performance test shall consist of three separate runs using the applicable test method.

(1) Each run shall be conducted for the time and under the conditions specified in the applicable standard. For the purpose of determining compliance with an applicable standard, the arithmetic means of results of the three runs shall apply. In the event that a sample is accidentally lost or conditions occur in which one of the three runs must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sample train, extreme meteorological conditions, or other circumstances, beyond the owner or operator's control, compliance may, upon the Administrator's approval, be determined using the arithmetic mean of the results of the two other runs.

(2) Contents of report (electronic or paper submitted copy). Unless otherwise specified in a relevant standard or test method, or as otherwise approved by the Administrator in writing, the report for a performance test shall include the elements identified in paragraphs (f)(2)(i) through (vi) of this section.

(i) General identification information for the facility including a mailing address, the physical address, the owner or operator or responsible official (where applicable) and his/her email address, and the appropriate Federal Registry System (FRS) number for the facility.

(ii) Purpose of the test including the applicable regulation(s) requiring the test, the pollutant(s) and other parameters being measured, the applicable emission standard and any process parameter component, and a brief process description.

(iii) Description of the emission unit tested including fuel burned, control devices, and vent characteristics; the appropriate source classification code (SCC); the permitted maximum process rate (where applicable); and the sampling location.

(iv) Description of sampling and analysis procedures used and any modifications to standard procedures, quality assurance procedures and results, record of process operating conditions that demonstrate the applicable test conditions are met, and values for any operating parameters for which limits were being set during the test.

(v) Where a test method requires you record or report, the following shall be included: Record of preparation of standards, record of calibrations, raw data sheets for field sampling, raw data sheets for field and laboratory analyses, chain-of-custody documentation, and example calculations for reported results.

(vi) Identification of the company conducting the performance test including the primary office address, telephone number, and the contact for this test program including his/her email address.

* * * * *

■ 6. In § 60.17:

■ a. Revise paragraph (h)(180).

■ b. Redesignate paragraphs (h)(200) through (h)(206) as paragraphs (h)(202) through (h)(208).

■ c. Redesignate paragraphs (h)(190) through (h)(199) as (h)(191) through (h)(200).

■ d. Add new paragraphs (h)(190) and (h)(201).

The additions and revisions read as follows:

### § 60.17 Incorporations by reference.

* * * * *

(h) * * *

(180) ASTM D6348–03, Standard Test Method for Determination of Gaseous Compounds by Extractive Direct Interface Fourier Transform Infrared (FTIR) Spectroscopy, (Approved October 1, 2003), IBR approved for § 60.73a(b), table 7 to subpart IIII, table 2 to subpart JJJJ, and § 60.4245(d).

* * * * *

(190) ASTM D6911–15, Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis, approved January 15, 2015, IBR approved for appendix A–8: Method 30B.

* * * * *

(201) ASTM E617–13, Standard Specification for Laboratory Weights and Precision Mass Standards, approved May 1, 2013, IBR approved for appendix A–3: Methods 4, 5, 5H, 5I, and appendix A–8: Method 29.

* * * * *

## Subpart JJJJ—Standards of Performance for Stationary Spark Ignition Internal Combustion Engines

■ 7. Revise § 60.4245(d) to read as follows:

### § 60.4245 What are my notification, reporting, and recordkeeping requirements if I am an owner or operator of a stationary SI internal combustion engine?

* * * * *

(d) Owners and operators of stationary SI ICE that are subject to performance testing must submit a copy of each performance test as conducted in § 60.4244 within 60 days after the test has been completed. Performance test reports using EPA Method 18, EPA Method 320, or ASTM D6348–03 (incorporated by reference—see 40 CFR 60.17) to measure VOC require reporting of all QA/QC data. For Method 18, report results from sections 8.4 and 11.1.1.4; for Method 320, report results from sections 8.6.2, 9.0, and 13.0; and for ASTM D6348–03 report results of all QA/QC procedures in Annexes 1–7.

* * * * *

■ 8. Revise Table 2 to subpart JJJJ of part 60 to read as follows:

TABLE 2 TO SUBPART JJJJ OF PART 60—REQUIREMENTS FOR PERFORMANCE TESTS

[As stated in § 60.4244, you must comply with the following requirements for performance tests within 10 percent of 100 percent peak (or the highest achievable) load]

| For each | Complying with the requirement to | You must | Using | According to the following requirements |
|---|---|---|---|---|
| 1. Stationary SI internal combustion engine demonstrating compliance according to § 60.4244. | a. limit the concentration of $NO_X$ in the stationary SI internal combustion engine exhaust. | i. Select the sampling port location and the number/location of traverse points at the exhaust of the stationary internal combustion engine;. | (1) Method 1 or 1A of 40 CFR part 60, appendix A–1, if measuring flow rate. | (a) Alternatively, for $NO_X$, $O_2$, and moisture measurement, ducts ≤6 inches in diameter may be sampled at a single point located at the duct centroid and ducts >6 and ≤12 inches in diameter may be sampled at 3 traverse points located at 16.7, 50.0, and 83.3% of the measurement line ('3-point long line'). If the duct is >12 inches in diameter *and* the sampling port location meets the two and half-diameter criterion of Section 11.1.1 of Method 1 of 40 CFR part 60, Appendix A, the duct may be sampled at '3-point long line'; otherwise, conduct the stratification testing and select sampling points according to Section 8.1.2 of Method 7E of 40 CFR part 60, Appendix A. |
| | | ii. Determine the $O_2$ concentration of the stationary internal combustion engine exhaust at the sampling port location;. | (2) Method 3, 3A, or 3B [b] of 40 CFR part 60, appendix A–2 or ASTM Method D6522–00 (Reapproved 2005) [a d]. | (b) Measurements to determine $O_2$ concentration must be made at the same time as the measurements for $NO_X$ concentration. |
| | | iii. If necessary, determine the exhaust flowrate of the stationary internal combustion engine exhaust;. | (3) Method 2 or 2C of 40 CFR part 60, appendix A–1 or Method 19 of 40 CFR part 60, appendix A–7. | |
| | | iv. If necessary, measure moisture content of the stationary internal combustion engine exhaust at the sampling port location; and. | (4) Method 4 of 40 CFR part 60, appendix A–3, Method 320 of 40 CFR part 63, appendix A [e], or ASTM Method D6348–03 [d e]. | (c) Measurements to determine moisture must be made at the same time as the measurement for $NO_X$ concentration. |
| | | v. Measure $NO_X$ at the exhaust of the stationary internal combustion engine; if using a control device, the sampling site must be located at the outlet of the control device. | (5) Method 7E of 40 CFR part 60, appendix A–4, ASTM Method D6522–00 (Reapproved 2005) [a d], Method 320 of 40 CFR part 63, appendix A [e], or ASTM Method D6348–03 [d e]. | (d) Results of this test consist of the average of the three 1-hour or longer runs. |
| | b. limit the concentration of CO in the stationary SI internal combustion engine exhaust. | i. Select the sampling port location and the number/location of traverse points at the exhaust of the stationary internal combustion engine;. | (1) Method 1 or 1A of 40 CFR part 60, appendix A–1, if measuring flow rate. | (a) Alternatively, for CO, $O_2$, and moisture measurement, ducts ≤6 inches in diameter may be sampled at a single point located at the duct centroid and ducts >6 and ≤12 inches in diameter may be sampled at 3 traverse points located at 16.7, 50.0, and 83.3% of the measurement line ('3-point long line'). If the duct is >12 inches in diameter *and* the sampling port location meets the two and half-diameter criterion of Section 11.1.1 of Method 1 of 40 CFR part 60, Appendix A, the duct may be sampled at '3-point long line'; otherwise, conduct the stratification testing and select sampling points according to Section 8.1.2 of Method 7E of 40 CFR part 60, Appendix A. |

TABLE 2 TO SUBPART JJJJ OF PART 60—REQUIREMENTS FOR PERFORMANCE TESTS—Continued

[As stated in § 60.4244, you must comply with the following requirements for performance tests within 10 percent of 100 percent peak (or the highest achievable) load]

| For each | Complying with the requirement to | You must | Using | According to the following requirements |
|---|---|---|---|---|
| | | ii. Determine the $O_2$ concentration of the stationary internal combustion engine exhaust at the sampling port location;. | (2) Method 3, 3A, or 3B[b] of 40 CFR part 60, appendix A–2 or ASTM Method D6522–00 (Reapproved 2005)[a d]. | (b) Measurements to determine $O_2$ concentration must be made at the same time as the measurements for CO concentration. |
| | | iii. If necessary, determine the exhaust flowrate of the stationary internal combustion engine exhaust;. | (3) Method 2 or 2C of 40 CFR 60, appendix A–1 or Method 19 of 40 CFR part 60, appendix A–7. | |
| | | iv. If necessary, measure moisture content of the stationary internal combustion engine exhaust at the sampling port location; and. | (4) Method 4 of 40 CFR part 60, appendix A–3, Method 320 of 40 CFR part 63, appendix A[e], or ASTM Method D6348–03[d e]. | (c) Measurements to determine moisture must be made at the same time as the measurement for CO concentration. |
| | | v. Measure CO at the exhaust of the stationary internal combustion engine; if using a control device, the sampling site must be located at the outlet of the control device. | (5) Method 10 of 40 CFR part 60, appendix A4, ASTM Method D6522–00 (Reapproved 2005)[a d e], Method 320 of 40 CFR part 63, appendix A[e], or ASTM Method D6348–03[d e]. | (d) Results of this test consist of the average of the three 1-hour or longer runs. |
| | c. limit the concentration of VOC in the stationary SI internal combustion engine exhaust. | i. Select the sampling port location and the number/location of traverse points at the exhaust of the stationary internal combustion engine;. | (1) Method 1 or 1A of 40 CFR part 60, appendix A–1, if measuring flow rate. | (a) Alternatively, for VOC, $O_2$, and moisture measurement, ducts ≤6 inches in diameter may be sampled at a single point located at the duct centroid and ducts >6 and ≤12 inches in diameter may be sampled at 3 traverse points located at 16.7, 50.0, and 83.3% of the measurement line ('3-point long line'). If the duct is >12 inches in diameter *and* the sampling port location meets the two and half-diameter criterion of Section 11.1.1 of Method 1 of 40 CFR part 60, Appendix A, the duct may be sampled at '3-point long line'; otherwise, conduct the stratification testing and select sampling points according to Section 8.1.2 of Method 7E of 40 CFR part 60, Appendix A. |
| | | ii. Determine the $O_2$ concentration of the stationary internal combustion engine exhaust at the sampling port location;. | (2) Method 3, 3A, or 3B[b] of 40 CFR part 60, appendix A–2 or ASTM Method D6522–00 (Reapproved 2005)[a d]. | (b) Measurements to determine $O_2$ concentration must be made at the same time as the measurements for VOC concentration. |
| | | iii. If necessary, determine the exhaust flowrate of the stationary internal combustion engine exhaust;. | (3) Method 2 or 2C of 40 CFR 60, appendix A–1 or Method 19 of 40 CFR part 60, appendix A–7. | |
| | | iv. If necessary, measure moisture content of the stationary internal combustion engine exhaust at the sampling port location; and. | (4) Method 4 of 40 CFR part 60, appendix A–3, Method 320 of 40 CFR part 63, appendix A[e], or ASTM Method D6348–03[d e]. | (c) Measurements to determine moisture must be made at the same time as the measurement for VOC concentration. |

TABLE 2 TO SUBPART JJJJ OF PART 60—REQUIREMENTS FOR PERFORMANCE TESTS—Continued

[As stated in § 60.4244, you must comply with the following requirements for performance tests within 10 percent of 100 percent peak (or the highest achievable) load]

| For each | Complying with the requirement to | You must | Using | According to the following requirements |
|---|---|---|---|---|
| | | v. Measure VOC at the exhaust of the stationary internal combustion engine; if using a control device, the sampling site must be located at the outlet of the control device. | (5) Methods 25A and 18 of 40 CFR part 60, appendices A–6 and A–7, Method 25A with the use of a hydrocarbon cutter as described in 40 CFR 1065.265, Method 18 of 40 CFR part 60, appendix A–6 [c] [e], Method 320 of 40 CFR part 63, appendix A [e], or ASTM Method D6348–03 [d] [e]. | (d) Results of this test consist of the average of the three 1-hour or longer runs. |

[a] Also, you may petition the Administrator for approval to use alternative methods for portable analyzer.

[b] You may use ASME PTC 19.10–1981, Flue and Exhaust Gas Analyses, for measuring the $O_2$ content of the exhaust gas as an alternative to EPA Method 3B. AMSE PTC 19.10–1981 incorporated by reference, see 40 CFR 60.17

[c] You may use EPA Method 18 of 40 CFR part 60, appendix A–6, provided that you conduct an adequate pre-survey test prior to the emissions test, such as the one described in OTM 11 on EPA's Web site (*http://www.epa.gov/ttn/emc/prelim/otm11.pdf*).

[d] Incorporated by reference; see 40 CFR 60.17.

[e] You must meet the requirements in § 60.4245(d).

■ 9. In appendix A–1 to part 60:

■ a. Revise section 11.2.1.2 in Method 1.

■ b. Remove Figure 1–2 in section 17.0 after the table entitled ''Table 1–1 Cross-Section Layout for Rectangular Stacks'' in Method 1.

■ c. Revise sections 6.7, 10.1.2.3, 10.1.3.4, 10.1.3.7, 10.1.4.1.3, 10.1.4.3, and Figure 2–10 in section 17.0 in Method 2.

The revisions read as follows:

## Appendix A–1 to Part 60—Test Methods 1 Through 2F

* * * * *

### Method 1-Sample and Velocity Traverses for Stationary Sources

* * * * *

11.2.1.2 When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1–1. Before referring to the figure, however, determine the distances from the measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1–1 the minimum number of traverse points that corresponds:

(1) To the number of duct diameters upstream; and

(2) To the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks, the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1–1.

* * * * *

### Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)

* * * * *

6.7 Calibration Pitot Tube. Calibration of the Type S pitot tube requires a standard pitot tube for a reference. When calibration of the Type S pitot tube is necessary (see Section 10.1), a standard pitot tube shall be used for a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained directly from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, (301) 975–2002; or by calibration against another standard pitot tube with a NIST-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in sections 6.7.1 through 6.7.5 below and illustrated in Figure 2–5 (see also References 7, 8, and 17 in section 17.0) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of 0.99 ±0.01.

* * * * *

10.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 910 m/min (3,000 ft/min). This velocity must be constant with time to guarantee constant and steady flow during the entire period of calibration. A centrifugal fan is recommended for this purpose, as no flow rate adjustment for back pressure of the fan is allowed during the calibration process. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 910 m/min (3,000 ft/min) will generally be valid to ±3 percent for the measurement of velocities above 300 m/min (1,000 ft/min) and to ±6 percent for the measurement of velocities between 180 and 300 m/min (600 and 1,000 ft/min). If a more precise correlation between the pitot tube coefficient (Cp) and velocity is desired, the flow system should have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,500 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see References 9 and 14 in section 17.0 for details).

* * * * *

10.1.3.4 Read $\Delta p_{std}$, and record its value in a data table similar to the one shown in Figure 2–9. Remove the standard pitot tube from the duct, and disconnect it from the manometer. Seal the standard entry port. Make no adjustment to the fan speed or other wind tunnel volumetric flow control device between this reading and the corresponding Type S pitot reading.

* * * * *

10.1.3.7 Repeat Steps 10.1.3.3 through 10.1.3.6 until three pairs of $\Delta p$ readings have been obtained for the A side of the Type S pitot tube, with all the paired observations conducted at a constant fan speed (no changes to fan velocity between observed readings).

* * * * *

10.1.4.1.3 For Type S pitot tube combinations with complete probe assemblies, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area interference and blockage and yield incorrect coefficient values (Reference 9 in section 17.0). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary, but no closer to the outer wall of the wind tunnel than 4 inches. The maximum allowable blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area (Figure

2–10a). If the pitot and/or probe assembly blocks more than 2 percent of the cross-sectional area at an insertion point only 4 inches inside the wind tunnel, the diameter of the wind tunnel must be increased.

* * * * *

10.1.4.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube needs to be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2–2 or 2–3, however, and must have an average deviation ($\sigma$) value of 0.01 or less (see section 12.4.4).

* * * * *

*17.0 Tables, Diagrams, Flowcharts, and Validation Data*

* * * * *

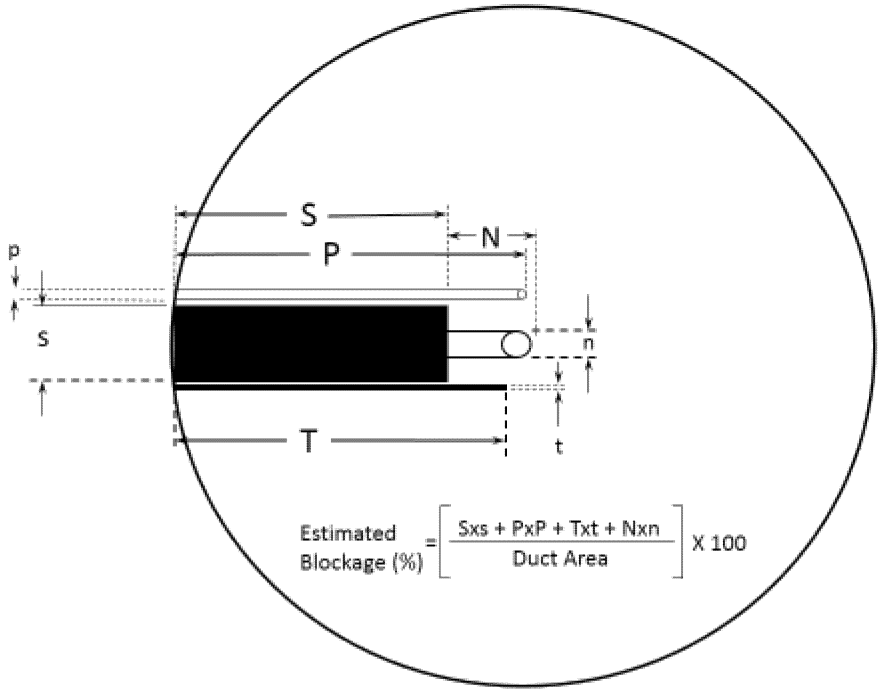


Figure 2-10. Projected-area model for a typical pitot tube assembly.

* * * * *

■ 10. In appendix A–2 to part 60:
■ a. Revise sections 6.11.1, 6.11.2, 10.6.6, and 10.6.8 in Method 2G.
■ b. Revise section 6.3 in Method 3C.
■ c. Add sections 6.3.1, 6.3.2, 6.3.3, 6.3.4, and 6.3.5 in Method 3C.

The revisions and additions read as follows:

## Appendix A–2 to Part 60—Test Methods 2G Through 3C

* * * * *

### Method 2G—Determination of Stack Gas Velocity and Volumetric Flow Rate With Two-Dimensional Probes

* * * * *

6.11.1 Test section cross-sectional area. The flowing gas stream shall be confined within a circular, rectangular, or elliptical duct. The cross-sectional area of the tunnel must be large enough to ensure fully developed flow in the presence of both the calibration pitot tube and the tested probe. The calibration site, or ''test section,'' of the wind tunnel shall have a minimum diameter of 30.5 cm (12 in.) for circular or elliptical duct cross-sections or a minimum width of 30.5 cm (12 in.) on the shorter side for rectangular cross-sections. Wind tunnels shall meet the probe blockage provisions of this section and the qualification requirements prescribed in section 10.1. The projected area of the portion of the probe head, shaft, and attached devices inside the wind tunnel during calibration shall represent no more than 2 percent of the cross-sectional area of the tunnel. If the pitot and/or probe assembly blocks more than 2 percent of the cross-sectional area at an insertion point only 4 inches inside the wind tunnel, the diameter of the wind tunnel must be increased.

6.11.2 Velocity range and stability. The wind tunnel should be capable of achieving and maintaining a constant and steady velocity between 6.1 m/sec and 30.5 m/sec (20 ft/sec and 100 ft/sec) for the entire calibration period for each selected calibration velocity. The wind tunnel shall produce fully developed flow patterns that are stable and parallel to the axis of the duct in the test section.

* * * * *

10.6.6 Read the differential pressure from the calibration pitot tube ($\Delta P_{std}$), and record its value. Read the barometric pressure to within ±2.5 mm Hg (±0.1 in. Hg) and the temperature in the wind tunnel to within 0.6 °C (1 °F). Record these values on a data form similar to Table 2G–8. Record the rotational speed of the fan or indicator of wind tunnel velocity control (damper setting, variac rheostat, etc.) and make no adjustment to fan speed or wind tunnel velocity control between this observation and the Type S probe reading.

* * * * *

10.6.8 Take paired differential pressure measurements with the calibration pitot tube and tested probe (according to sections 10.6.6 and 10.6.7). The paired measurements in each replicate can be made either simultaneously (*i.e.,* with both probes in the wind tunnel) or by alternating the measurements of the two probes (*i.e.,* with only one probe at a time in the wind tunnel). Adjustments made to the fan speed or other changes to the system designed to change the air flow velocity of the wind tunnel between observation of the calibration pitot tube ($\Delta P_{std}$) and the Type S pitot tube invalidates the reading and the observation must be repeated.

* * * * *

### Method 3C—Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen From Stationary Sources

* * * * *

6.3 Analyzer Linearity Check and Calibration. Perform this test before sample analysis.

6.3.1 Using the gas mixtures in section 5.1, verify the detector linearity over the range of suspected sample concentrations with at least three concentrations per compound of interest. This initial check may also serve as the initial instrument calibration.

6.3.2 You may extend the use of the analyzer calibration by performing a single-point calibration verification. Calibration verifications shall be performed by triplicate injections of a single-point standard gas. The concentration of the single-point calibration must either be at the midpoint of the calibration curve or at approximately the source emission concentration measured during operation of the analyzer.

6.3.3 Triplicate injections must agree within 5 percent of their mean, and the average calibration verification point must agree within 10 percent of the initial calibration response factor. If these calibration verification criteria are not met, the initial calibration described in section 6.3.1, using at least three concentrations, must be repeated before analysis of samples can continue.

6.3.4 For each instrument calibration, record the carrier and detector flow rates, detector filament and block temperatures, attenuation factor, injection time, chart speed, sample loop volume, and component concentrations.

6.3.5 Plot a linear regression of the standard concentrations versus area values to obtain the response factor of each compound. Alternatively, response factors of uncorrected component concentrations (wet basis) may be generated using instrumental integration.

**Note:** Peak height may be used instead of peak area throughout this method.

* * * * *

■ 11. In appendix A–3 to part 60:
■ a. Add sections 10.3 and 12.2.5 in Method 4.
■ b. Revise section 16.4 in Method 4.
■ c. Revise sections 6.1.1.9 and 8.7.6.2.5 in Method 5.
■ d. Add sections 10.7 and 10.8 in Method 5.
■ e. Add sections 10.4 and 10.5 in Method 5H.
■ f. Add sections 10.1 and 10.2 in Method 5I.

The revisions and additions read as follows:

**Appendix A–3 to Part 60-Test Methods 4 Through 5I**

* * * * *

**Method 4—Determination of Moisture Content in Stack Gases**

* * * * *

10.3 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference-see 40 CFR 60.17) Class 6 (or better). Daily, before use, the field balance must measure the weight within ± 0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

* * * * *

12.2.5 Using F-factors to determine approximate moisture for estimating moisture content where no wet scrubber is being used, for the purpose of determining isokinetic sampling rate settings with no fuel sample, is acceptable using the average $F_c$ or $F_d$ factor from Method 19 (see Method 19, section 12.3.1). If this option is selected, calculate the approximate moisture as follows:

$B_{ws} = B_H + B_A + B_F$

Where:

$B_A$ = Mole Fraction of moisture in the ambient air.

$$B_A = \frac{\%RH}{100 * P_{Bar}} * 10^{\left[6.6912 - \left(\frac{3144}{T + 390.86}\right)\right]}$$

$B_F$ = Mole fraction of moisture from free water in the fuel.

$$B_F = \left[\frac{0.0036W^2 + 0.075W}{100}\right]\left[\frac{20.9 - O_2}{20.9}\right]$$

$B_H$ = Mole fraction of moisture from the hydrogen in the fuel.

$$B_H = \left[1 - \frac{F_d}{F_w}\right]\frac{(20.9 - O_2)}{20.9}$$

$B_{ws}$ = Mole fraction of moisture in the stack gas.

$F_d$ = Volume of dry combustion components per unit of heat content at 0 percent oxygen, dscf/$10^6$ Btu (scm/J). See Table 19–2 in Method 19.

$F_w$ = Volume of wet combustion components per unit of heat content at 0 percent oxygen, wet. scf/$10^6$ Btu (scm/J). See Table 19–2 in Method 19.

%RH = Percent relative humidity (calibrated hygrometer acceptable), percent.

$P_{Bar}$ = Barometric pressure, in. Hg.

T = Ambient temperature, °F.

W = Percent free water by weight, percent.

$O_2$ = Percent oxygen in stack gas, dry basis, percent.

* * * * *

16.4 Using F-factors to determine moisture is an acceptable alternative to Method 4 for a combustion stack not using a scrubber, and where a fuel sample is taken during the test run and analyzed for development of an $F_d$ factor (see Method 19, section 12.3.2), and where stack $O_2$ content is measured by Method 3A or 3B during each test run. If this option is selected, calculate the moisture content as follows:

$B_{ws} = B_H + B_A + B_F$

Where:

$B_A$ = Mole fraction of moisture in the ambient air.

$$B_A = \frac{\%RH}{100\ P_{Bar}}\left[10^{\left[6.6912-\left(\frac{3144}{T+390.86}\right)\right]}\right]$$

**Note:** Values of $B_A$ should be between 0.00 and 0.06 with common values being about 0.015.

$B_F$ = Mole fraction of moisture from free water in the fuel.

$$B_F = \left[\frac{0.0036\ W^2 + 0.075\ W}{100}\right]\left[\frac{20.9 - O_2}{20.9}\right]$$

**Note:** Free water in fuel is minimal for distillate oil and gases, such as propane and natural gas, so this step may be omitted for those fuels.

$B_H$ = Mole fraction of moisture from the hydrogen in the fuel.

$$B_H = \left(1 - \frac{F_d}{F_w}\right)\frac{(20.9 - O_2)}{20.9}$$

$B_{ws}$ = Mole fraction of moisture in the stack gas.

$F_d$ = Volume of dry combustion components per unit of heat content at 0 percent oxygen, dscf/$10^6$ Btu (scm/J). Develop a test specific $F_d$ value using an integrated fuel sample from each test run and Equation 19–13 in section 12.3.2 of Method 19.

$F_w$ = Volume of wet combustion components per unit of heat content at 0 percent oxygen, wet scf/$10^6$ Btu (scm/J). Develop a test specific $F_w$ value using an integrated fuel sample from each test run and Equation 19–14 in section 12.3.2 of Method 19.

%RH = Percent relative humidity (calibrated hygrometer acceptable), percent.

$P_{Bar}$ = Barometric pressure, in. Hg.

T = Ambient temperature, °F.

W = Percent free water by weight, percent.

$O_2$ = Percent oxygen in stack gas, dry basis, percent.

* * * * *

**Method 5—Determination of Particulate Matter Emissions From Stationary Sources**

* * * * *

6.1.1.9 Metering System. Vacuum gauge, leak-free pump, calibrated temperature sensors, dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5–1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall allow periodic checks of isokinetic rates.

* * * * *

8.7.6.2.5 Clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to allow determination of whether leakage occurred during transport. Label the container to clearly identify its contents.

* * * * *

10.7 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 6 (or better). Daily before use, the field balance must measure the weight within ±0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.8 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures, and conduct the multipoint calibration before use.

* * * * *

**Method 5H—Determination of Particulate Matter Emissions From Wood Heaters From a Stack Location**

* * * * *

10.4 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 6 (or better). Daily before use, the field balance must measure the weight within ± 0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.5 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures, and conduct the multipoint calibration before use.

* * * * *

**Method 5I—Determination of Low Level Particulate Matter Emissions From Stationary Sources**

* * * * *

10.1 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 6 (or better). Daily, before use, the field balance must measure the weight within ±0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.2 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or

between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures and conduct the multipoint calibration before use.

* * * * *

■ 12. In appendix A–4 to part 60:
■ a. Revise section 8.3 in Method 6C.
■ b. Revise sections 8.1.2, 8.2.7, and 12.8 in Method 7E.
■ c. Revise sections 6.2.5 and 8.4.2 in Method 10.
■ d. Add section 6.2.6 in Method 10.
■ e. Revise sections 6.1.6, 6.1.7, 6.1.8, 6.1.9, 6.1.10, 8.1, 8.2.1 and 8.2.3 in Method 10A.
■ f. Add section 6.1.11 in Method 10A.
■ g. Revise section 6.1 in Method 10B.

The revisions and additions read as follows:

## Appendix A–4 to Part 60—Test Methods 6 Through 10B

* * * * *

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

* * * * *

*8.3 Interference Check.* You must follow the procedures of section 8.2.7 of Method 7E to conduct an interference check, substituting $SO_2$ for $NO_X$ as the method pollutant. For dilution-type measurement systems, you must use the alternative interference check procedure in section 16 and a co-located, unmodified Method 6 sampling train.

* * * * *

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

* * * * *

8.1.2 Determination of Stratification. Perform a stratification test at each test site to determine the appropriate number of sample traverse points. If testing for multiple pollutants or diluents at the same site, a stratification test using only one pollutant or diluent satisfies this requirement. A stratification test is not required for small stacks that are less than 4 inches in diameter. To test for stratification, use a probe of appropriate length to measure the $NO_X$ (or pollutant of interest) concentration at 12 traverse points located according to Table 1–1 or Table 1–2 of Method 1. Alternatively, you may measure at three points on a line passing through the centroidal area. Space the three points at 16.7, 50.0, and 83.3 percent of the measurement line. Sample for a minimum of twice the system response time (see section 8.2.6) at each traverse point. Calculate the individual point and mean $NO_X$ concentrations. If the concentration at each traverse point differs from the mean concentration for all traverse points by no more than: ±5.0 percent of the mean concentration; or ±0.5 ppm (whichever is less restrictive), the gas stream is considered unstratified, and you may collect samples from a single point that most closely matches the mean. If the 5.0 percent or 0.5 ppm criterion is not met, but the concentration at each traverse point differs from the mean concentration for all traverse points by not more than: ±10.0 percent of the mean concentration; or ±1.0 ppm (whichever is less restrictive), the gas stream is considered to be minimally stratified and you may take samples from three points. Space the three points at 16.7, 50.0, and 83.3 percent of the measurement line. Alternatively, if a 12-point stratification test was performed and the emissions were shown to be minimally stratified (all points within ± 10.0 percent of their mean or within ±1.0 ppm), and if the stack diameter (or equivalent diameter, for a rectangular stack or duct) is greater than 2.4 meters (7.8 ft), then you may use 3-point sampling and locate the three points along the measurement line exhibiting the highest average concentration during the stratification test at 0.4, 1.2 and 2.0 meters from the stack or duct wall. If the gas stream is found to be stratified because the 10.0 percent or 1.0 ppm criterion for a 3-point test is not met, locate 12 traverse points for the test in accordance with Table 1–1 or Table 1–2 of Method 1.

* * * * *

8.2.7 *Interference Check.* Conduct an interference response test of the gas analyzer prior to its initial use in the field. If you have multiple analyzers of the same make and model, you need only perform this alternative interference check on one analyzer. You may also meet the interference check requirement if the instrument manufacturer performs this or a similar check on an analyzer of the same make and model of the analyzer that you use and provides you with documented results.

(1) You may introduce the appropriate interference test gases (that are potentially encountered during a test; see examples in Table 7E–3) into the analyzer separately or as mixtures. Test the analyzer with the interference gas alone at the highest concentration expected at a test source and again with the interference gas and $NO_X$ at a representative $NO_X$ test concentration. For analyzers measuring $NO_X$ greater than 20 ppm, use a calibration gas with a $NO_X$ concentration of 80 to 100 ppm and set this concentration equal to the calibration span. For analyzers measuring less than 20 ppm $NO_X$, select an NO concentration for the calibration span that reflects the emission levels at the sources to be tested, and perform the interference check at that level. Measure the total interference response of the analyzer to these gases in ppmv. Record the responses and determine the interference using Table 7E–4. The specification in section 13.4 must be met.

(2) A copy of this data, including the date completed and signed certification, must be available for inspection at the test site and included with each test report. This interference test is valid for the life of the instrument unless major analytical components (*e.g.,* the detector) are replaced with different model parts. If major components are replaced with different model parts, the interference gas check must be repeated before returning the analyzer to service. If major components are replaced, the interference gas check must be repeated before returning the analyzer to service. The tester must ensure that any specific technology, equipment, or procedures that are intended to remove interference effects are operating properly during testing.

* * * * *

12.8 $NO_2$—NO Conversion Efficiency Correction. If desired, calculate the total $NO_X$ concentration with a correction for converter efficiency using Equation 7E–8.

$$NO_{x\,Corr} = NO + \left(\frac{(NO_x - NO)}{Eff_{NO_2}} x\ 100\right) \qquad \text{Eq. 7E-8}$$

* * * * *

### Method 10—Determination of Carbon Monoxide Emissions From Stationary Sources (Instrumental Analyzer Procedure)

* * * * *

6.2.5 Flexible Bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 $ft^3$). (Verify through the manufacturer that the Tedlar alternative is suitable for CO and make this verified information available for inspection.) Leak-test the bag in the laboratory before using by evacuating with a pump followed by a dry gas meter. When the evacuation is complete, there should be no flow through the meter.

6.2.6 Sample Tank. Stainless steel or aluminum tank equipped with a pressure indicator with a minimum volume of 4 liters.

* * * * *

8.4.2 Integrated Sampling. Evacuate the flexible bag or sample tank. Set up the equipment as shown in Figure 10–1 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak-free. Sample at a rate proportional to the stack velocity. If needed, the $CO_2$ content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing an ascarite $CO_2$ removal tube used and computing $CO_2$ concentration from the gas volume sampled and the weight gain of the tube. Data may be recorded on a form similar to Table 10–1. If a sample tank is used for sample collection, follow procedures similar to those in sections 8.1.2, 8.2.3, 8.3, and 12.4 of Method 25 as appropriate to

prepare the tank, conduct the sampling, and correct the measured sample concentration.

* * * * *

**Method 10A—Determination of Carbon Monoxide Emissions in Certifying Continuous Emission Monitoring Systems at Petroleum Refineries**

* * * * *

6.1.6 Flexible Bag. Tedlar, or equivalent, with a capacity of 10 liters (0.35 $ft^3$) and equipped with a sealing quick-connect plug. The bag must be leak-free according to section 8.1. For protection, it is recommended that the bag be enclosed within a rigid container.

6.1.7 Sample Tank. Stainless steel or aluminum tank equipped with a pressure indicator with a minimum volume of 10 liters.

6.1.8 Valves. Stainless-steel needle valve to adjust flow rate, and stainless-steel 3-way valve, or equivalent.

6.1.9 $CO_2$ Analyzer. Fyrite, or equivalent, to measure $CO_2$ concentration to within 0.5 percent.

6.1.10 Volume Meter. Dry gas meter, capable of measuring the sample volume under calibration conditions of 300 ml/min (0.01 $ft^3$/min) for 10 minutes.

6.1.11 Pressure Gauge. A water filled U-tube manometer, or equivalent, of about 30 cm (12 in.) to leak-check the flexible bag.

* * * * *

8.1 Sample Bag or Tank Leak-Checks. While a leak-check is required after bag or sample tank use, it should also be done before the bag or sample tank is used for sample collection. The tank should be leak-checked according to the procedure specified in section 8.1.2 of Method 25. The bag should be leak-checked in the inflated and deflated condition according to the following procedure:

* * * * *

8.2.1 Evacuate and leak check the sample bag or tank as specified in section 8.1. Assemble the apparatus as shown in Figure 10A–1. Loosely pack glass wool in the tip of the probe. Place 400 ml of alkaline permanganate solution in the first two impingers and 250 ml in the third. Connect the pump to the third impinger, and follow this with the surge tank, rate meter, and 3-way valve. Do not connect the bag or sample tank to the system at this time.

* * * * *

8.2.3 Purge the system with sample gas by inserting the probe into the stack and drawing the sample gas through the system at 300 ml/min ±10 percent for 5 minutes. Connect the evacuated bag or sample tank to the system, record the starting time, and sample at a rate of 300 ml/min for 30 minutes, or until the bag is nearly full, or the sample tank reaches ambient pressure. Record the sampling time, the barometric pressure, and the ambient temperature. Purge the system as described above immediately before each sample.

* * * * *

**Method 10B—Determination of Carbon Monoxide Emissions from Stationary Sources**

* * * * *

6.1. Sample Collection. Same as in Method 10A, section 6.1 (paragraphs 6.1.1 through 6.1.11).

* * * * *

■ 13. Revise section 8.3.2 in Method 15 of appendix A–5 to part 60 to read as follows:

## Appendix A–5 to Part 60-Test Methods 11 Through 15A

* * * * *

**Method 15—Determination of Hydrogen Sulfide, Carbonyl Sulfide, and Carbon Disulfide Emissions From Stationary Sources**

* * * * *

8.3.2 Determination of Calibration Drift. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in section 10.0. Only $H_2S$ (or other permeant) need be used to recalibrate the GC/FPD analysis system and the dilution system. Partial recalibration may be performed at the midlevel calibration gas concentration or at a concentration measured in the samples but not less than the lowest calibration standard used in the initial calibration. Compare the calibration curves obtained after the runs to the calibration curves obtained under section 10.3. The calibration drift should not exceed the limits set forth in section 13.4. If the drift exceeds this limit, the intervening run or runs should be considered invalid. As an option, the calibration data set that gives the highest sample values may be chosen by the tester.

* * * * *

■ 14. In appendix A–6 to part 60:

■ a. Revise sections 12.1 and 12.2 in Method 16C.

■ b. Remove section 8.2.1.5.2.3 in Method 18.

The revisions 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**

* * * * *

12.1 Nomenclature.

ACE = Analyzer calibration error, percent of calibration span.

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 $SO_2$.

$C_{SO2}$ = Unadjusted sample $SO_2$ concentration, ppmv.

$C_{TRS}$ = Total reduced sulfur concentration corrected for system performance, ppmv.

CS = Calibration span, ppmv.

DF = Dilution system (if used) dilution factor, dimensionless.

SP = System performance, percent.

12.2 Analyzer Calibration Error. For non-dilution systems, use Equation 16C–1 to calculate the analyzer calibration error for the low-, mid-, and high-level calibration gases.

$$ACE = \frac{C_{Dir} - C_v}{CS} X\ 100 \qquad \text{Eq. 16C-1}$$

* * * * *

■ 15. In appendix A–7 to part 60:

■ a. Revise sections 9.1, 12.1, and 12.3 in Method 25C.

■ b. Remove section 11.2 in Method 25C.

■ c. Add sections 12.4, 12.5, 12.5.1 and 12.5.2 in Method 25C.

The revisions and additions read as follows:

## Appendix A–7 to Part 60—Test Methods 19 Through 25E

* * * * *

**Method 25C—Determination of Nonmethane Organic Compounds (NMOC) in Landfill Gases**

* * * * *

9.1 Miscellaneous Quality Control Measures.

| Section | Quality control measure | Effect |
|---|---|---|
| 8.4.2 ..... | Verify that landfill gas sample contains less than 20 percent $N_2$ or 5 percent $O_2$. | Ensures that ambient air was not drawn into the landfill gas sample and gas was sampled from an appropriate location. |

| Section | Quality control measure | Effect |
|---|---|---|
| 10.1, 10.2 ..... | NMOC analyzer initial and daily performance checks .... | Ensures precision of analytical results. |

* * * * *

12.1 Nomenclature

$B_w$ = Moisture content in the sample, fraction.

$C_{N2}$ = $N_2$ concentration in the diluted sample gas.

$C_{mN2}$ = Measured $N_2$ concentration, fraction in landfill gas.

$C_{mOx}$ = Measured Oxygen concentration, fraction in landfill gas.

$C_{Ox}$ = Oxygen concentration in the diluted sample gas.

$C_t$ = Calculated NMOC concentration, ppmv C equivalent.

$C_{tm}$ = Measured NMOC concentration, ppmv C equivalent.

$P_b$ = Barometric pressure, mm Hg.

$P_t$ = Gas sample tank pressure after sampling, but before pressurizing, mm Hg absolute.

$P_{tf}$ = Final gas sample tank pressure after pressurizing, mm Hg absolute.

$P_{ti}$ = Gas sample tank pressure after evacuation, mm Hg absolute.

$P_w$ = Vapor pressure of $H_2O$ (from Table 25C–1), mm Hg.

r = Total number of analyzer injections of sample tank during analysis (where j = injection number, 1 . . . r).

$T_t$ = Sample tank temperature at completion of sampling, °K.

$T_{ti}$ = Sample tank temperature before sampling, °K.

$T_{tf}$ = Sample tank temperature after pressuring, °K.

* * * * *

12.3 Nitrogen Concentration in the landfill gas. Use equation 25C–2 to calculate the measured concentration of nitrogen in the original landfill gas.

$$C_{N2} = \left[\frac{\left(\frac{P_{tf}}{T_{tf}}\right)}{\left(\left(\frac{P_t}{T_t}\right)-\left(\frac{P_{ti}}{T_{ti}}\right)\right)}\right] C_{mN2} \qquad \text{Eq.25C-2}$$

12.4 Oxygen Concentration in the landfill gas. Use equation 25C–3 to calculate the measured concentration of oxygen in the original landfill gas.

$$C_{Ox} = \left[\frac{\left(\frac{P_{tf}}{T_{tf}}\right)}{\left(\left(\frac{P_t}{T_t}\right)-\left(\frac{P_{ti}}{T_{ti}}\right)\right)}\right] C_{mOx} \qquad \text{Eq.25C-3}$$

12.5 You must correct the NMOC Concentration for the concentration of nitrogen or oxygen based on which gas or gases passes the requirements in section 9.1.

12.5.1 NMOC Concentration with nitrogen correction. Use Equation 25C–4 to calculate the concentration of NMOC for each sample tank when the nitrogen concentration is less than 20 percent.

$$C_t = \frac{\frac{P_{tf}}{T_{tf}}}{\left(\frac{P_t}{T_t}-\frac{P_{ti}}{T_{ti}}\right)\left(1-\frac{99}{78}C_{N2}\right)-B_w} \frac{1}{r}\sum_{j=1}^{r} C_{tm(j)} \qquad \text{Eq.25C-4}$$

12.5.2 NMOC Concentration with oxygen correction. Use Equation 25C–5 to calculate the concentration of NMOC for each sample tank if the landfill gas oxygen is less than 5 percent and the landfill gas nitrogen concentration is greater than 20 percent.

$$C_t = \frac{\frac{P_{tf}}{T_{tf}}}{\left(\frac{P_t}{T_t}-\frac{P_{ti}}{T_{ti}}\right)\left(1-\frac{99}{21}C_{Ox}\right)-B_w} \frac{1}{r}\sum_{j=1}^{r} C_{tm(j)} \qquad \text{Eq.25C-5}$$

* * * * *

■ 16. In appendix A–8 to Part 60:

■ a. Revise section 13.3 in Method 26.

■ b. Revise sections 4.3 and 8.1.6 in Method 26A.

■ c. Revise section 8.2.9.3 in Method 29.

■ d. Add sections 10.4 and 10.5 in Method 29.

■ e. Revise the section heading for section 8.1 in Method 30A.

■ f. Revise the section heading for section 8.1, and revise 8.3.3.8 in Method 30B.

The revisions and additions read as follows:

## Appendix A–8 to Part 60—Test Methods 26 Through 30B

* * * * *

### Method 26—Determination of Hydrogen Chloride Emissions From Stationary Sources

* * * * *

13.3 Detection Limit. A typical IC instrumental detection limit for $Cl^-$ is 0.2 μg/ml. Detection limits for the other analyses should be similar. Assuming 50 ml liquid recovered from both the acidified impingers, and the basic impingers, and 0.12 dscm (4.24 dscf) of stack gas sampled, then the analytical detection limit in the stack gas would be about 0.05 ppm for HCl and $Cl_2$, respectively.

* * * * *

### Method 26A—Determination of Hydrogen Halide and Halogen Emissions From Stationary Sources Isokinetic Method

* * * * *

4.3 High concentrations of nitrogen oxides ($NO_X$) may produce sufficient nitrate ($NO_3^-$) to interfere with measurements of very low $Br^-$ levels. Dissociating chloride salts (*e.g.,* ammonium chloride) at elevated temperatures interfere with halogen acid measurement in this method. Maintaining particulate probe/filter temperatures between 120 °C and 134 °C (248 °F and 273 °F) minimizes this interference.

* * * * *

8.1.6 Post-Test Moisture Removal (Optional). When the optional cyclone is included in the sampling train or when liquid is visible on the filter at the end of a sample run even in the absence of a cyclone, perform the following procedure. Upon completion of the test run, connect the ambient air conditioning tube at the probe inlet and operate the train with the filter heating system between 120 and 134 °C (248 and 273 °F) at a low flow rate (*e.g.,* ΔH = 1 in. $H_2O$) to vaporize any liquid and hydrogen halides in the cyclone or on the filter and pull them through the train into the impingers. After 30 minutes, turn off the flow, remove the conditioning tube, and examine the cyclone and filter for any visible liquid. If liquid is visible, repeat this step for 15 minutes and observe again. Keep repeating until the cyclone is dry.

**Note:** It is critical that this procedure is repeated until the cyclone is completely dry.

* * * * *

### Method 29—Determination of Metals Emissions From Stationary Sources

* * * * *

8.2.9.3 Wash the two permanganate impingers with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled No. 5C containing 200 ml of water. First, place 200 ml of water in the container. Then wash the impinger walls and stem with the 8 N HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing *both permanganate impingers combined.* Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse. Finally, pour the 25 ml of 8 N HCl rinse carefully into the container with the 200 ml of water. Mark the height of the fluid level on the outside of the container in order to determine if leakage occurs during transport.

* * * * *

10.4 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference-see 40 CFR 60.17) Class 6 (or better). Daily before use, the field balance must measure the weight within ±0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.5 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures, and conduct the multipoint calibration before use.

* * * * *

### Method 30A—Determination of Total Vapor Phase Mercury Emissions From Stationary Sources (Instrumental Analyzer Procedure)

* * * * *

8.1 Selection of Sampling Sites and Sampling Points * * *

* * * * *

### Method 30B—Determination of Total Vapor Phase Mercury Emissions From Coal-Fired Combustion Sources Using Carbon Sorbent Traps

* * * * *

8.1 Selection of Sampling Sites and Sampling Points * * *

* * * * *

8.3.3.8 Sample Handling, Preservation, Storage, and Transport. While the performance criteria of this approach provides for verification of appropriate sample handling, it is still important that the user consider, determine and plan for suitable sample preservation, storage, transport, and holding times for these measurements. Therefore, procedures in ASTM D6911–15 ''Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis'' (incorporated by reference-see 40 CFR 60.17) shall be followed for all samples, where appropriate. To avoid Hg contamination of the samples, special attention should be paid to cleanliness during transport, field handling, sampling, recovery, and laboratory analysis, as well as during preparation of the sorbent cartridges. Collection and analysis of blank samples (*e.g.,* reagent, sorbent, field, etc.) is useful in verifying the absence or source of contaminant Hg.

* * * * *

■ 17. In appendix B to part 60:

■ a. Add the entry ''Performance Specification 16—Specifications and Test Procedures for Predictive Emission Monitoring Systems in Stationary Sources'' at the end of the table of contents for appendix B to part 60.

■ b. Add a sentence to the end of section 8.1(2)(i) in Performance Specification 1.

■ c. Revise sections 3.11, 6.1.1, 6.1.2, 16.3.2, and section 18.0 in Performance Specification 2.

■ d. Revise section 13.2 in Performance Specification 3.

■ e. Revise sections 8.3, 8.3.1, and 13.3 in Performance Specification 4A.

■ f. Revise sections 12.1 and 13.1 in Performance Specification 11.

■ g. Revise section 9.1.2 in Performance Specification 15.

■ h. Add reserved sections 14.0 and 15.0 in Performance Specification 15.

■ i. Revise the introductory text of section 12.2.3 in Performance Specification 16.

■ j. Revise table 16–1 in Performance Specification 16.

The revisions and additions read as follows:

## Appendix B to Part 60—Performance Specifications

* * * * *

### Performance Specification 1—Specifications and Test Procedures for Continuous Opacity Monitoring Systems in Stationary Sources

* * * * *

8.1 * * *

(2) * * *

(i) * * * Alternatively, you may select a measurement location specified in paragraph 8.1(2)(ii) or 8.1(2)(iii).

* * * * *

### Performance Specification 2—Specifications and Test Procedures for $SO_2$ and $NO_X$ Continuous Emission Monitoring Systems in Stationary Sources

* * * * *

3.11 *Span Value* means the calibration portion of the measurement range as specified in the applicable regulation or other requirement. If the span is not specified in the applicable regulation or other requirement, then it must be a value approximately equivalent to two times the emission standard. For spans less than 500 ppm, the span value may either be rounded upward to the next highest multiple of 10 ppm, or to the next highest multiple of 100 ppm such that the equivalent emission concentration is not less than 30 percent of the selected span value.

* * * * *

6.1.1 Data Recorder. The portion of the CEMS that provides a record of analyzer output. The data recorder may record other pertinent data such as effluent flow rates, various instrument temperatures or abnormal CEMS operation. The data recorder output range must include the full range of expected concentration values in the gas stream to be sampled including zero and span values.

6.1.2 The CEMS design should also allow the determination of calibration drift at the zero and span values. If this is not possible or practical, the design must allow these determinations to be conducted at a low-level value (zero to 20 percent of the span value) and at a value between 50 and 100 percent of the span value. In special cases, the Administrator may approve a single-point calibration drift determination.

* * * * *

16.3.2 For diluent CEMS:

RA≡d; ≤0.7 percent $O_2$ or $CO_2$, as applicable.

**Note:** Waiver of the relative accuracy test in favor of the alternative RA procedure does not preclude the requirements to complete the CD tests nor any other requirements specified in an applicable subpart for reporting CEMS data and performing CEMS drift checks or audits.

* * * * *

*18.0 Tables, Diagrams, Flowcharts, and Validation Data*

TABLE 2–1—t-VALUES

| n [a] | $t_{0.975}$ | n [a] | $t_{0.975}$ | n [a] | $t_{0.975}$ |
|---|---|---|---|---|---|
| 2 | 12.706 | 7 | 2.447 | 12 | 2.201 |
| 3 | 4.303 | 8 | 2.365 | 13 | 2.179 |
| 4 | 3.182 | 9 | 2.306 | 14 | 2.160 |
| 5 | 2.776 | 10 | 2.262 | 15 | 2.145 |
| 6 | 2.571 | 11 | 2.228 | 16 | 2.131 |

[a] The values in this table are already corrected for n − 1 degrees of freedom. Use n equal to the number of individual values.

TABLE 2–2—MEASUREMENT RANGE

| Measurement point | Pollutant monitor | Diluent monitor for | |
|---|---|---|---|
| | | $CO_2$ | $O_2$ |
| 1 | 20–30% of span value | 5–8% by volume | 4–6% by volume. |
| 2 | 50–60% of span value | 10–14% by volume | 8–12% by volume. |

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**Figure 2-1. Calibration Drift Determination**

| | Day | Date and time | Calibration value (C) | Monitor value (M) | Difference (C-M) | Percent of span value (C-M)/span value × 100 |
|---|---|---|---|---|---|---|
| Low-level | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| High-level | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |

**FIGURE 2-2. RELATIVE ACCURACY DETERMINATION.**

| Run No. | Date and time | $SO_2$ | | | $NO_x$[b] | | | $CO_2$ or $O_2$[a] | | $SO_2$[a] | | | $NO_x$[a] | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | RM | CEMS | Diff | RM | CEMS | Diff | RM | CEMS | RM | CEMS | Diff | RM | CEMS | Diff |
| | | ppm[c] | | | ppm[c] | | | %[c] | %[c] | mass/GCV | | | mass/GCV | | |
| 1 | | | | | | | | | | | | | | | |
| 2 | | | | | | | | | | | | | | | |
| 3 | | | | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | | | | |
| 10 | | | | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | | | | |
| Average | | | | | | | | | | | | | | | |
| Confidence Interval | | | | | | | | | | | | | | | |
| Accuracy | | | | | | | | | | | | | | | |

[a] For Steam generators.
[b] Average of three samples.
[c] Make sure that RM and CEMS data are on a consistent basis, either wet or dry.

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* * * * *

**Performance Specification 3—Specifications and Test Procedures for $O_2$ and $CO_2$ Continuous Emission Monitoring Systems in Stationary Sources**

* * * * *

13.2 CEMS Relative Accuracy Performance Specification. The RA of the CEMS must be no greater than 20.0 percent of the mean value of the reference method (RM) data when calculated using equation 3–1. The results are also acceptable if the result of Equation 3–2 is less than or equal to 1.0 percent $O_2$ (or $CO_2$).

$$RA = \frac{\left[\left|\bar{d}\right| + \left|CC\right|\right]}{\overline{RM}} \times 100$$ Eq. 3-1

Where:

$\left|\bar{d}\right|$ = Absolute value of the mean of the differences (from Equation 2-3 of Performance Specification 2).

|CC| = Absolute value of the confidence coefficient (from Equation 2-5 of Performance Specification 2).

$\overline{RM}$ = Average Reference Method value.

$RA = \overline{RM} - \overline{CEMS}$ Eq. 3-2

$\overline{RM}$ = Average Reference Method value.

$\overline{CEMS}$ = Average CEMS value.

* * * * *

**Performance Specification 4A—Specifications and Test Procedures for Carbon Monoxide Continuous Emission Monitoring Systems in Stationary Sources**

* * * * *

8.3 Response Time Test Procedure. The response time test applies to all types of CEMS, but will generally have significance only for extractive systems. The entire system is checked with this procedure including applicable sample extraction and transport, sample conditioning, gas analyses, and data recording.

8.3.1 Introduce zero gas into the system. When the system output has stabilized (no change greater than 1 percent of full scale for 30 sec), introduce an upscale calibration gas and wait for a stable value. Record the time (upscale response time) required to reach 95 percent of the final stable value. Next, reintroduce the zero gas and wait for a stable reading before recording the response time (downscale response time). Repeat the entire procedure until you have three sets of data to determine the mean upscale and downscale response times. The slower or longer of the two means is the system response time.

* * * * *

13.3 Response Time. The CEMS response time shall not exceed 240 seconds to achieve 95 percent of the final stable value.

* * * * *

**Performance Specification 11—Specifications and Test Procedures for Particulate Matter Continuous Emission Monitoring Systems at Stationary Sources**

* * * * *

12.1 How do I calculate upscale drift and zero drift? You must determine the difference in your PM CEMS output readings from the established reference values (zero and upscale check values) after a stated period of operation during which you performed no unscheduled maintenance, repair or adjustment.

(1) Calculate the upscale drift (UD) using Equation 11–1:

$$UD = \frac{|R_{CEM} - R_U|}{R_r} \times 100$$ Eq. 11-1

Where:

UD = The upscale (high-level) drift of your PM CEMS in percent,

$R_{CEM}$ = The measured PM CEMS response to the upscale reference standard,

$R_U$ = The pre-established numerical value of the upscale reference standard, and

$R_r$ = The response range of the analyzer.

(2) Calculate the zero drift (ZD) using Equation 11–2:

$$ZD = \frac{|R_{CEM} - R_L|}{R_r} \times 100 \qquad \text{Eq. 11-2}$$

Where:

ZD = The zero (low-level) drift of your PM CEMS in percent,

$R_{CEM}$ = The measured PM CEMS response to the zero reference standard,

$R_L$ = The pre-established numerical value of the zero reference standard, and

$R_r$ = The response range of the analyzer.

(3) Summarize the results on a data sheet similar to that shown in Table 2 (see section 17).

* * * * *

13.1 What is the 7-day drift check performance specification? Your daily PM CEMS internal drift checks must demonstrate that the average daily drift of your PM CEMS does not deviate from the value of the reference light, optical filter, Beta attenuation signal, or other technology-suitable reference standard by more than 2 percent of the response range. If your CEMS includes diluent and/or auxiliary monitors (for temperature, pressure, and/or moisture) that are employed as a necessary part of this performance specification, you must determine the calibration drift separately for each ancillary monitor in terms of its respective output (see the appropriate performance specification for the diluent CEMS specification). None of the calibration drifts may exceed their individual specification.

* * * * *

**Performance Specification 15—Performance Specification for Extractive FTIR Continuous Emissions Monitor Systems in Stationary Sources**

* * * * *

9.1.2 Test Procedure. Spike the audit sample using the analyte spike procedure in section 11. The audit sample is measured directly by the FTIR system (undiluted) and then spiked into the effluent at a known dilution ratio. Measure a series of spiked and unspiked samples using the same procedures as those used to analyze the stack gas. Analyze the results using sections 12.1 and 12.2. The measured concentration of each analyte must be within ±5 percent of the expected concentration (plus the uncertainty), *i.e.,* the calculated correction factor must be within 0.93 and 1.07 for an audit with an analyte uncertainty of ±2 percent.

* * * * *

*14.0 Pollution Prevention [Reserved]*

*15.0 Waste Management [Reserved]*

* * * * *

**Performance Specification 16—Specifications and Test Procedures for Predictive Emission Monitoring Systems in Stationary Sources**

* * * * *

*12.0 Calculations and Data Analysis*

* * * * *

12.2.3 Confidence Coefficient. Calculate the confidence coefficient using Equation 16–3 and Table 16–1 for n − 1 degrees of freedom.

* * * * *

*17.0 Tables, Diagrams, Flowcharts, and Validation Data*

TABLE 16–1—t-VALUES FOR ONE-SIDED, 97.5 PERCENT CONFIDENCE INTERVALS FOR SELECTED SAMPLE SIZES *

| n − 1 * | t-value | n − 1 | t-value |
|---|---|---|---|
| 1 | 12.706 | 15 | 2.131 |
| 2 | 4.303 | 16 | 2.120 |
| 3 | 3.182 | 17 | 2.110 |
| 4 | 2.776 | 18 | 2.101 |
| 5 | 2.571 | 19 | 2.093 |
| 6 | 2.447 | 20 | 2.086 |
| 7 | 2.365 | 21 | 2.080 |
| 8 | 2.306 | 22 | 2.074 |
| 9 | 2.262 | 23 | 2.069 |
| 10 | 2.228 | 24 | 2.064 |
| 11 | 2.201 | 25 | 2.060 |
| 12 | 2.179 | 26 | 2.056 |
| 13 | 2.160 | 27 | 2.052 |
| 14 | 2.145 | >28 | t-Table |

* The value n is the number of RM runs; n − 1 equals the degrees of freedom.

* * * * *

■ 18. Revise section 12.0 paragraphs (3) and (4) in Procedure 2 of appendix F to part 60 to read as follows:

**Appendix F to Part 60—Quality Assurance Procedures**

* * * * *

**Procedure 2—Quality Assurance Requirements for Particulate Matter Continuous Emission Monitoring Systems at Stationary Sources**

* * * * *

*12.0 What calculations and data analysis must I perform for my PM CEMS?*

* * * * *

(3) How do I calculate daily upscale and zero drift? You must calculate the upscale drift using Equation 2–2 and the zero drift using Equation 2–3:

$$UD = \frac{|R_{CEM} - R_U|}{R_r} \times 100 \qquad \text{Eq. 2-2}$$

Where:

UD = The upscale drift of your PM CEMS, in percent,

$R_{CEM}$ = Your PM CEMS response to the upscale check value,

$R_U$ = The upscale check value, and

$R_r$ = The response range of the analyzer.

$$ZD = \frac{|R_{CEM} - R_L|}{R_r} \times 100 \qquad \text{Eq. 2-3}$$

Where:

ZD = The zero (low-level) drift of your PM CEMS, in percent,

$R_{CEM}$ = Your PM CEMS response of the zero check value,

$R_L$ = The zero check value, and

$R_r$ = The response range of the analyzer.

(4) How do I calculate SVA accuracy? You must use Equation 2–4 to calculate the accuracy, in percent, for each of the three SVA tests or the daily sample volume check:

$$\text{SVA Accuracy} = \frac{|V_M - V_R|}{V_R} \times 100 \qquad \text{Eq. 2-4}$$

Where:

SVA Accuracy = The SVA accuracy at each audit point, in percent,

$V_M$ = Sample gas volume determined/reported by your PM CEMS (*e.g.,* dscm), and

$V_R$ = Sample gas volume measured by the independent calibrated reference device (*e.g.,* dscm) for the SVA or the reference value for the daily sample volume check.

**Note:** Before calculating SVA accuracy, you must correct the sample gas volumes measured by your PM CEMS and the independent calibrated reference device to the same basis of temperature, pressure, and moisture content. You must document all data and calculations.

* * * * *

## PART 61—NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS

■ 19. The authority citation for part 61 continues to read as follows:

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

■ 20. In § 61.13, revise paragraph (e)(1)(i) to read as follows:

### § 61.13 Emission tests and waiver of emission tests.

* * * * *

(e) * * *

(1) * * *

(i) The source owner, operator, or representative of the tested facility shall obtain an audit sample, if commercially available, from an AASP for each test method used for regulatory compliance purposes. No audit samples are required for the following test methods: Methods 3A and 3C of appendix A–3 of part 60 of this chapter; Methods 6C, 7E, 9, and 10 of appendix A–4 of part 60; Method 18 and 19 of appendix A–6 of part 60; Methods 20, 22, and 25A of appendix A–7 of part 60; Methods 30A and 30B of appendix A–8 of part 60; and Methods 303, 318, 320, and 321 of appendix A of part 63 of this chapter. If multiple sources at a single facility are tested during a compliance test event, only one audit sample is required for each method used during a compliance test. The compliance authority responsible for the compliance test may waive the requirement to include an audit sample if they believe that an audit sample is not necessary. ''Commercially available'' means that two or more independent AASPs have blind audit samples available for purchase. If the source owner, operator, or representative cannot find an audit sample for a specific method, the owner, operator, or representative shall consult the EPA Web site at the following URL, *www.epa.gov/ttn/emc,* to confirm whether there is a source that can supply an audit sample for that method. If the EPA Web site does not list an available audit sample at least 60 days prior to the beginning of the compliance test, the source owner, operator, or representative shall not be required to include an audit sample as part of the quality assurance program for the compliance test. When ordering an audit sample, the source owner, operator, or representative shall give the sample provider an estimate for the concentration of each pollutant that is emitted by the source or the estimated concentration of each pollutant based on the permitted level and the name, address, and phone number of the compliance authority. The source owner, operator, or representative shall report the results for the audit sample along with a summary of the emission test results for the audited pollutant to the compliance authority and shall report the results of the audit sample to the AASP. The source owner, operator, or representative shall make both reports at the same time and in the same manner or shall report to the compliance authority first and then report to the AASP. If the method being audited is a method that allows the samples to be analyzed in the field and the tester plans to analyze the samples in the field, the tester may analyze the audit samples prior to collecting the emission samples provided a representative of the compliance authority is present at the testing site. The tester may request, and the compliance authority may grant, a waiver to the requirement that a representative of the compliance authority must be present at the testing site during the field analysis of an audit sample. The source owner, operator, or representative may report the results of the audit sample to the compliance authority and then report the results of the audit sample to the AASP prior to collecting any emission samples. The test protocol and final test report shall document whether an audit sample was ordered and utilized and the pass/fail results as applicable.

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■ 21. Revise the section heading for section 11.7.3 in Method 107 of appendix B to part 61 to read as follows:

**Appendix B to Part 61—Test Methods**

* * * * *

**Method 107—Determination of Vinyl Chloride Content of In-Process Wastewater Samples, and Vinyl Chloride Content of Polyvinyl Chloride Resin Slurry, Wet Cake, and Latex Samples**

* * * * *

*11.0 Analytical Procedure*

* * * * *

11.7.3 Dispersion Resin Slurry and Latex Samples. * * *

* * * * *

## PART 63—NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS FOR SOURCE CATEGORIES

■ 22. The authority citation for part 63 continues to read as follows:

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

■ 23. In § 63.7:

■ a. Revise paragraph (c)(2)(iii)(A).

■ b. Add paragraph (g)(2).

The revision and addition read as follows:

### § 63.7 Performance testing requirements.

* * * * *

(c) * * *

(2) * * *

(iii) * * *

(A) The source owner, operator, or representative of the tested facility shall

obtain an audit sample, if commercially available, from an AASP for each test method used for regulatory compliance purposes. No audit samples are required for the following test methods: Methods 3A and 3C of appendix A–3 of part 60 of this chapter; Methods 6C, 7E, 9, and 10 of appendix A–4 of part 60; Methods 18 and 19 of appendix A–6 of part 60; Methods 20, 22, and 25A of appendix A–7 of part 60; Methods 30A and 30B of appendix A–8 of part 60; and Methods 303, 318, 320, and 321 of appendix A of this part. If multiple sources at a single facility are tested during a compliance test event, only one audit sample is required for each method used during a compliance test. The compliance authority responsible for the compliance test may waive the requirement to include an audit sample if they believe that an audit sample is not necessary. ''Commercially available'' means that two or more independent AASPs have blind audit samples available for purchase. If the source owner, operator, or representative cannot find an audit sample for a specific method, the owner, operator, or representative shall consult the EPA Web site at the following URL, *www.epa.gov/ttn/emc,* to confirm whether there is a source that can supply an audit sample for that method. If the EPA Web site does not list an available audit sample at least 60 days prior to the beginning of the compliance test, the source owner, operator, or representative shall not be required to include an audit sample as part of the quality assurance program for the compliance test. When ordering an audit sample, the source owner, operator, or representative shall give the sample provider an estimate for the concentration of each pollutant that is emitted by the source or the estimated concentration of each pollutant based on the permitted level and the name, address, and phone number of the compliance authority. The source owner, operator, or representative shall report the results for the audit sample along with a summary of the emission test results for the audited pollutant to the compliance authority and shall report the results of the audit sample to the AASP. The source owner, operator, or representative shall make both reports at the same time and in the same manner or shall report to the compliance authority first and then report to the AASP. If the method being audited is a method that allows the samples to be analyzed in the field and the tester plans to analyze the samples in the field, the tester may analyze the audit samples prior to collecting the emission samples provided a representative of the compliance authority is present at the testing site. The tester may request, and the compliance authority may grant, a waiver to the requirement that a representative of the compliance authority must be present at the testing site during the field analysis of an audit sample. The source owner, operator, or representative may report the results of the audit sample to the compliance authority and then report the results of the audit sample to the AASP prior to collecting any emission samples. The test protocol and final test report shall document whether an audit sample was ordered and utilized and the pass/fail results as applicable.

* * * * *

(g) * * *

(2) Contents of report (electronic or paper submitted copy). Unless otherwise specified in a relevant standard or test method, or as otherwise approved by the Administrator in writing, the report for a performance test shall include the elements identified in paragraphs (g)(2)(i) through (vi) of this section.

(i) General identification information for the facility including a mailing address, the physical address, the owner or operator or responsible official (where applicable) and his/her email address, and the appropriate Federal Registry System (FRS) number for the facility.

(ii) Purpose of the test including the applicable regulation requiring the test, the pollutant(s) and other parameters being measured, the applicable emission standard, and any process parameter component, and a brief process description.

(iii) Description of the emission unit tested including fuel burned, control devices, and vent characteristics; the appropriate source classification code (SCC); the permitted maximum process rate (where applicable); and the sampling location.

(iv) Description of sampling and analysis procedures used and any modifications to standard procedures, quality assurance procedures and results, record of process operating conditions that demonstrate the applicable test conditions are met, and values for any operating parameters for which limits were being set during the test.

(v) Where a test method requires you record or report, the following shall be included in your report: Record of preparation of standards, record of calibrations, raw data sheets for field sampling, raw data sheets for field and laboratory analyses, chain-of-custody documentation, and example calculations for reported results.

(vi) Identification of the company conducting the performance test including the primary office address, telephone number, and the contact for this test including his/her email address.

* * * * *

■ 24. Revise sections 13.1, 13.4, and 13.4.1 in Method 320 of appendix A to part 63 to read as follows:

## Appendix A to Part 63—Test Methods Pollutant Measurement Methods From Various Waste Media

* * * * *

### Method 320—Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy

* * * * *

*13.0 Method Validation Procedure*

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13.1 Section 6.0 of Method 301 (40 CFR part 63, appendix A), the Analyte Spike procedure, is used with these modifications. The statistical analysis of the results follows section 12.0 of EPA Method 301. Section 3 of this method defines terms that are not defined in Method 301.

* * * * *

13.4 *Statistical Treatment.* The statistical procedure of EPA Method 301 of this appendix, section 12.0 is used to evaluate the bias and precision. For FTIR testing a validation ''run'' is defined as spectra of 24 independent samples, 12 of which are spiked with the analyte(s) and 12 of which are not spiked.

13.4.1 *Bias.* Determine the bias (defined by EPA Method 301 of this appendix, section 12.1.1) using equation 7:

$$B = S_m - CS$$

Where:

B = Bias at spike level.
$S_m$ = Mean concentration of the analyte spiked samples.
CS = Expected concentration of the spiked samples.

* * * * *

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