

§ 80.47

40 CFR Ch. I (7–1–19 Edition)

Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence, approved November 1, 2012 (“ASTM D5453”).

(xi) ASTM D5599–00 (Reapproved 2010), Standard Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection, approved October 1, 2010 (“ASTM D5599”).

(xii) ASTM D5769–10, Standard Test Method for Determination of Benzene, Toluene, and Total Aromatics in Finished Gasolines by Gas Chromatography/Mass Spectrometry, approved May 1, 2010 (“ASTM D5769”).

(xiii) ASTM D6550–10, Standard Test Method for Determination of Olefin Content of Gasolines by Supercritical-Fluid Chromatography, approved October 1, 2010 (“ASTM D6550”).

(xiv) ASTM D6667–10, Standard Test Method for Determination of Total Volatile Sulfur in Gaseous Hydrocarbons and Liquefied Petroleum Gases by Ultraviolet Fluorescence, approved October 1, 2010 (“ASTM D6667”).

(xv) ASTM D6920–13, Standard Test Method for Total Sulfur in Naphthas, Distillates, Reformulated Gasolines, Diesels, Biodiesels, and Motor Fuels by Oxidative Combustion and Electrochemical Detection, approved September 15, 2013 (“ASTM D6920”).

(xvi) ASTM D7039–13, Standard Test Method for Sulfur in Gasoline, Diesel Fuel, Jet Fuel, Kerosine, Biodiesel, Biodiesel Blends, and Gasoline-Ethanol Blends by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry, approved September 15, 2013 (“ASTM D7039”).

(2) [Reserved]

[59 FR 7813, Feb. 16, 1994, as amended at 59 FR 36961, July 20, 1994; 61 FR 58306, Nov. 13, 1996; 63 FR 63793, Nov. 17, 1998; 65 FR 6822, Feb. 10, 2000; 65 FR 53189, Sept. 1, 2000; 66 FR 17263, Mar. 29, 2001; 67 FR 8737, Feb. 26, 2002; 67 FR 40181, June 12, 2002; 68 FR 56781, Oct. 2, 2003; 68 FR 57819, Oct. 7, 2003; 71 FR 16499, Apr. 3, 2006; 73 FR 74355, Dec. 8, 2008; 74 FR 6233, Feb. 6, 2009; 76 FR 65385, Oct. 21, 2011; 79 FR 23632, Apr. 28, 2014; 80 FR 9090, Feb. 19, 2015]

§ 80.47 Performance-based Analytical Test Method Approach.

All sample handling, testing procedures, and tests must be conducted using good laboratory practices.

(a) *Definitions.* As used in this subpart D:

(1) *Performance-based Analytical Test Method Approach* means a measurement system based upon established performance criteria for accuracy and precision with use of analytical test methods. As used in this subpart, this is a measurement system used by laboratories to demonstrate that a particular analytical test method is acceptable for demonstrating compliance.

(2) *Accuracy* means the closeness of agreement between an observed value from a single test measurement and an accepted reference value.

(3) *Precision* means the degree of agreement in a set of measurements performed on the same property of identical test material.

(4) *Absolute fuel parameter* means a fuel parameter for which a gravimetric standard is practical to construct and use. Sulfur content of gasoline, butane, or diesel fuel are examples of an absolute fuel parameter.

(5) *Gravimetric standard* means a test material made by adding a carefully weighed quantity of the analyte to a measured quantity of another substance known not to contain any of the analyte, resulting in a solution with an accurately known concentrate of the analyte.

(6) *Consensus named fuels* are homogeneous quantities of fuel that have been analyzed by a number of different laboratories (by sending around small samples). The average concentration of some parameter of interest across all of the different laboratories is then used as the “consensus name” for that material.

(7) *Locally-named reference materials* are gasoline or diesel fuels that are usually from the regular production of the facility where they are used in laboratory quality control efforts and have been analyzed using the designated method (either by the facility’s lab or by a reference lab) to obtain an estimate of their concentration.

(8) *Method-defined fuel parameter* means a fuel parameter for which an

EPA-prescribed primary test method or designated method defines the regulatory standard. Examples of method-defined fuel parameters include olefin content in gasoline, Reid vapor pressure (RVP) of gasoline, distillation parameters of gasoline, benzene content of gasoline, aromatic content of gasoline and diesel fuel, and oxygen/oxygenates content of gasoline.

(9) *Reference installations* are designated test method installations that are used to qualify the accuracy of other method-defined parameter instruments. Reference installations of the designated test method will be used to evaluate the accuracy of other method-defined alternative test methods and to establish correlation equations if necessary.

(10) *Correlation equation* is a correction equation as determined by the use of ASTM D6708. This standard practice determines whether the comparison between the alternative test method and the designated test method is a null result. If the comparison is not null, then the standard practice provides for a correlation equation that predicts designated test method results from the applicable method-defined alternative test method.

(11) *Statistical quality control (SQC)* means a planned system of activities whose purpose is to provide a level of quality that meets the needs of compliance with the standards of this part. This subpart prescribes specific SQC requirements for both absolute and method driven fuel parameters for both voluntary and non-voluntary consensus-based standards bodies.

(12) *Voluntary consensus-based standards body (VCSB)* means a domestic or international organization that plans, develops, establishes, or coordinates voluntary consensus standards using agreed-upon procedures and which possesses the attributes of openness, balance of interest, due process, and consensus, as explained in OMB Circular A-119 and the National Technology Transfer and Advancement Act of 1995, P.L. 104-113, sec. 12(d).

(13) *Non-voluntary consensus-based standards body (non-VCSB)* means a domestic or international regulated party that has developed a proprietary ana-

lytical test method that has not been adopted by a VCSB organization.

(b) *Precision and accuracy criteria for approval for the absolute fuel parameter of gasoline sulfur*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline sulfur standard at §§ 80.195 and 80.1603, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 1.5 times the repeatability “r” divided by 2.77, where “r” equals the ASTM repeatability of ASTM D7039 (Example: A 10ppm sulfur gasoline sample: Maximum allowable standard deviation of 20 tests  $\leq 1.5 \times (1.73 \text{ ppm} / 2.77) = 0.94 \text{ ppm}$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) *Accuracy*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline sulfur standard at §§ 80.195 and 80.1603:

(i) The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the range of 1–10 ppm, say 10 ppm, shall not differ from the accepted reference value (ARV) of the standard by more than 0.70 ppm sulfur;

(ii) The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the range of 10–20 ppm, say 20 ppm, shall not differ from the ARV of the standard by more than 1.02 ppm sulfur; and

(iii) In applying the tests of paragraphs (b)(2)(i) and (ii) of this section,

**§ 80.47****40 CFR Ch. I (7–1–19 Edition)**

individual test results shall be compensated for any known chemical interferences using good laboratory practices.

(3) The test method specified at § 80.46(a)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraphs (b)(1) and (2) of this section.

(c) *Precision and accuracy criteria for approval of the absolute fuel parameter of sulfur in butane*—(1) *Precision*. Beginning January 1, 2016, for butane subject to the butane sulfur standard at §§ 80.82, 80.195, 80.340(b) and 80.1603, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available butane must be less than or equal to 1.5 times the repeatability (r) divided by 2.77, where “r” equals the ASTM repeatability of ASTM D6667 (Example: A 10 ppm sulfur butane sample: Maximum allowable standard deviation of 20 tests  $\leq 1.5 * (1.15 \text{ ppm} / 2.77) = 0.62 \text{ ppm}$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) *Accuracy*. Beginning January 1, 2016, for butane subject to the butane sulfur standard at §§ 80.82, 80.195, 80.340(b) and 80.1603—

(i) The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the range of 1–10 ppm, say 10 ppm, shall not differ from the accepted reference value (ARV) of the standard by more than 0.47 ppm sulfur;

(ii) The arithmetic average of a continuous series of at least 10 tests performed using good laboratory practices on a commercially available gravimetric sulfur standard in the

range of 10–20 ppm, say 20 ppm, shall not differ from the accepted reference value (ARV) of the standard by more than 0.94 ppm sulfur; and

(iii) In applying the tests of paragraphs (c)(2)(i) and (ii) of this section, individual test results shall be compensated for any known chemical interferences using good laboratory practices.

(3) The test method specified at § 80.46(a)(2) and in use prior to October 28, 2013 is exempt from the requirements of paragraphs (c)(1) and (2) of this section.

(d) *Precision criteria for approval of the method defined fuel parameter of olefins in gasoline*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline standards of this part, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.3 times the reproducibility (R), where “R” equals the ASTM reproducibility of ASTM D1319 (Example: A gasoline containing 9 Vol% olefins: Maximum allowable standard deviation of 20 tests  $\leq 0.3 * (3.06 \text{ Vol}\%) = 0.92 \text{ Vol}\%$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(b)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (d)(1) of this section.

(e) *Precision criteria for approval of the method defined fuel parameter of aromatics in gasoline*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle

gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline standards of this part, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.3 times the reproducibility (R), where "R" equals the ASTM reproducibility of ASTM D1319 (Example: A gasoline containing 32Vol% aromatics: Maximum allowable standard deviation of 20 tests  $\leq 0.3 \times (3.7 \text{ Vol}\%) = 1.11 \text{ Vol}\%$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(f)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (e)(1) of this section.

(f) *Precision criteria for approval of the method defined fuel parameter of oxygen and oxygenate content in gasoline*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline standards of this part, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.3 times the reproducibility (R), where "R" equals the ASTM reproducibility of ASTM D5599 (Example: A gasoline containing 3Mass% total oxygen: Maximum allowable standard deviation of 20 tests  $\leq 0.3 \times (0.32 \text{ Mass}\%) = 0.10 \text{ Mass}\%$ ). The 20 results must be a

series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(g)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (f)(1) of this section.

(g) *Precision criteria for approval of the method defined fuel parameter of Reid Vapor Pressure (RVP) in gasoline*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline standards of this part and volatility standards at § 80.27, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.3 times the reproducibility (R), where "R" equals the ASTM reproducibility of ASTM D5191 (Example: A gasoline having a RVP of 6.8psi: Maximum allowable standard deviation of 20 tests withdrawn from a 250 milliliter container  $\leq 0.3 \times (0.40 \text{ psi}) = 0.12 \text{ psi}$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(c)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (g)(1) of this section.

(h) *Precision criteria for approval of the method defined fuel parameter of gasoline distillation*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline

standards of this part, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.3 times the reproducibility (R), where “R” equals the ASTM reproducibility in Table 10, Groups 2, 3 and 4 (Automated) of ASTM D86–07 for the initial boiling point, E10, E50, E90 and final boiling point. (Example: A gasoline having an initial boiling point of 26 °C and a final boiling point of 215 °C: Maximum allowable standard deviation of 20 tests for initial boiling point  $\leq 0.3 \cdot (8.5 \text{ °C}) = 2.55 \text{ °C}$ , maximum allowable standard deviation of 20 tests for E10  $\leq 0.3 \cdot (3.0 + 2.64 \cdot S_c) \text{ °C}$ , maximum allowable standard deviation of 20 tests for E50  $\leq 0.3 \cdot (2.9 + 3.97 \cdot S_c) \text{ °C}$ , maximum allowable standard deviation of 20 tests for E90  $\leq 0.3 \cdot (2.0 + 2.53 \cdot S_c) \text{ °C}$ , and maximum allowable standard deviation of 20 tests for final boiling point  $\leq 0.3 \cdot (10.5 \text{ °C}) = 3.15 \text{ °C}$ ), where  $S_c$  is the average slope (or rate of change) of the gasoline distillation curve as calculated in accordance with section 13.2 of ASTM D86–07. The 20 results must be a series of tests with a sequential record of analysis and no omissions. Note that the precision criteria described in this paragraph (h)(1) differ from what is specified in ASTM D86–12. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(d)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (h)(1) of this section.

(i) *Precision criteria for approval of the method defined fuel parameter of benzene in gasoline*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle gasoline, gasoline blendstock, and gasoline fuel additives subject to the gasoline standards of this part and MSAT2

standards at §§ 80.41, 80.101, 80.1230, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available gasoline must be less than or equal to 0.15 times the reproducibility (R), where “R” equals the ASTM reproducibility of ASTM D3606 (Example: A gasoline having a 1Vol% benzene: Maximum allowable standard deviation of 20 tests  $\leq 0.15 \cdot (0.18 \text{ Vol}\%) = 0.027 \text{ Vol}\%$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.46(e)(1) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (i)(1) of this section.

(j) *Precision criteria for approval of the method defined fuel parameter of aromatics in diesel*—(1) *Precision*. Beginning January 1, 2016, for motor vehicle diesel fuel subject to the motor vehicle diesel standards at § 80.520, the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 20 days (tests may be arranged into no fewer than five batches of four or fewer tests each, with only one such batch allowed per day over the minimum of 20 days) on samples using good laboratory practices taken from a single homogeneous commercially available diesel fuel must be less than or equal to 0.3 times the reproducibility (R), where “R” equals the ASTM reproducibility of ASTM D1319 (Example: A diesel fuel containing 35 Vol% aromatics: maximum allowable standard deviation of 20 tests  $\leq 0.3 \cdot (3.3 \text{ Vol}\%) = 0.99 \text{ Vol}\%$ ). The 20 results must be a series of tests with a sequential record of analysis and no omissions. A laboratory facility may exclude a given sample or test result

only if the exclusion is for a valid reason under good laboratory practices and it maintains records regarding the sample and test results and the reason for excluding them.

(2) The test method specified at § 80.2(z) and in use prior to October 28, 2013 is exempt from the requirements of paragraph (j)(1) of this section.

(k) *Criteria for designated test method reference installations used to qualify the accuracy of other method-defined parameter instruments.* (1) Beginning January 1, 2016, for a single laboratory test facility qualifying a method defined alternative test method, the reference installation of the method-defined fuel parameter for the applicable designated test method must have precision equal to 0.3 times the reproducibility (R) of the method-defined fuel parameter's designated test method, where "R" is the reproducibility of the designated test method.

(i) For olefins in gasoline, see paragraph (d)(1) of this section.

(ii) For aromatics in gasoline, see paragraph (e)(1) of this section.

(iii) For oxygen and oxygenate content of gasoline, see paragraph (f)(1) of this section.

(iv) For Reid Vapor Pressure (RVP) of gasoline, see paragraph (g)(1) of this section.

(v) For gasoline distillation, see paragraph (h)(1) of this section.

(vi) For benzene in gasoline, see paragraph (i)(1) of this section.

(vii) For aromatics in diesel fuel, see paragraph (j)(1) of this section.

(2) The reference installation of the method-defined fuel parameter for the applicable designated test method must be shown to stay within the middle 50% of the distribution of an industry or commercially available monthly inter-laboratory crosscheck program for 3 out of 5 successive exchanges for at least a period of five months using good laboratory practices. Specifically, compute the difference between the instrument's average measurement of the fuel closest to the applicable fuel standard (or to the average value for the fuel parameter in the complex model) and the mean for that fuel obtained by all of the non-outlier labs in the monthly inter-laboratory crosscheck program. Standardize this

difference by expressing it in standard deviation units. These standardized inter-laboratory crosscheck differences should be placed in a moving average with a minimum span of five months. The instrument's moving average in standard deviation units cannot be outside the central 50% of the distribution of all laboratories that participated in the inter-laboratory crosscheck program.

(3) The reference installation of the method-defined fuel parameter for the applicable designated test method must be shown to be in statistical quality control as specified in ASTM D6299 for a minimum period of five months using good laboratory practices. The system is still considered to be in statistical quality control and the five month time period will not re-start if—

(i) Regular maintenance and/or recalibration conducted during the five months in SQC qualification time period is considered as part of in-control normal operation, and/or;

(ii) If an assignable cause for 'out of control' is found, mitigated, and the system is brought back in statistical quality control during the five month time period that the reference installation is attempting to meet the five month in-statistical-control requirement, the five month time period does not re-start and the system is still considered to be 'in-control'.

(4) For a voluntary consensus standards body, such as ASTM, or for a commercially available industry crosscheck program, the summary statistics (mean and standard error = standard deviation/square root [number of results]) from the VCSB or commercially available inter-laboratory crosscheck program (ILCP) data may be used as is without imposing the reference installations requirements of this section, provided that the number of non-outlying results is greater than 16 for both the designated and alternative test methods. The determination of ARV of check standards as specified in ASTM D6299, clause 6.2.2.1 and Note 7 shall be followed for the inter-laboratory crosscheck program. The use of VCSB or commercially available ILCP data as described above is deemed suitable for an ASTM D6708 assessment of VCSB alternative test methods.

(1) *Qualification criteria for Voluntary Consensus Standard Based (VCSB) Method-Defined Parameter Test Methods and Non-voluntary Consensus Standard Based (non-VCSB) Absolute Fuel Parameter of Sulfur in Gasoline and Butane.* (1)(i) Beginning January 1, 2016, the test facility or VCSB include full test method documentation by the Voluntary Consensus Standard Based (VCSB) organization, including a description of the technology and/or instrumentation that makes the method functional.

(ii) For the Non-voluntary Consensus Standard Based (non-VCSB) Absolute Fuel Parameter of Sulfur in Gasoline and Butane, the test facility include full test method documentation, including a description of the technology and/or instrumentation that makes the method functional.

(2)(i) The test facility or VCSB include information reported in the test method that demonstrates the test method meets the applicable precision information for the method-defined fuel parameter as described in this section.

(ii) For the Non-VCSB absolute fuel parameter of sulfur in gasoline and butane, the test facility include information reported in the test method that demonstrates the applicable accuracy criteria as described in § 80.47(b)(2) for gasoline and § 80.47(c)(2) for butane.

(3) The test facility or VCSB include information reported in the test method that demonstrates the test method has been evaluated using ASTM D6708 and whether the comparison is a “null” result or whether a correlation equation needs to be applied that predicts designated test method results from the applicable method-defined alternative test method.

(4) The test methods specified at §§ 80.2(w) and 80.46(a)(1), (a)(2), (b)(1), (c)(1), (d)(1), (e)(1), (f)(1), and (g)(1) and in use by a test facility prior to October 28, 2013 are exempt from the requirements of paragraphs (1)(1) through (3) of this section.

(m) *Qualification criteria for Non-Voluntary Consensus Standard Based (non-VCSB) Method-Defined Parameter Test Methods.* For a non-VCSB method to be approved, the following information must be submitted to the Administrator by each test facility for each

method that it wishes to have approved.

(1) Beginning January 1, 2016, full and thorough test method documentation, including a description of the technology and/or instrumentation that makes the method functional so a person lacking experience with the test instrument would be able to replicate its results.

(2) Information reported in the test method that demonstrates the test method meets the applicable precision information using good laboratory practices for the method-defined fuel parameter as described in this section.

(3) Both the candidate method-defined Non-VCSB test method and its respective designated test method must be tested on a range of consensus named fuels or locally-named reference materials that are typical of those analyzed by the facility in practice using good laboratory practices and must meet the data requirements for variability as required in ASTM D6708.

(4) The facility using the candidate method-defined non-VCSB test method must statistically establish through application of ASTM D6708 that the candidate method measures the same aspect of samples as applicable to its respective designated test method using good laboratory practices.

(5) If the use of ASTM D6708 reveals that the candidate method-defined non-VCSB test method has sample-specific biases due to matrix effects that cannot be determined as random the method is disqualified. If however, it is determined that the candidate method-defined non-VCSB test method can be qualified on a narrow circumscribed range of fuels while still meeting the data requirements for variability as required in ASTM D6708 (see paragraph (m)(3) of this section), then the types of fuels on which the qualification was achieved and for which the method is to be approved must be specified in the candidate method-defined non-VCSB test method description. If there is any restriction on the scope of fuels for which the candidate method-defined non-VCSB test method is to be qualified, the applicant must include a discussion of how the facility plans to screen sample for conformity to the scope. If the candidate method-defined

test method is found to have minimal matrix effects, a statement to this effect must be included by the applicant in its application.

(6) The candidate method-defined non-VCSB test method precision qualification must be conducted in the form of "between methods reproducibility" (Rcm) of the candidate method and applicable designated test method as recommended in ASTM D6708, where the Rcm must be equal to or less than 70 percent of the published reproducibility of the applicable designated test method using good laboratory practices.

(7) The applicant of the candidate method-defined non-VCSB test method must demonstrate through the use of ASTM D6708 whether a correlation to applicable designated test method is necessary. If it is determined through the use of this practice that the candidate method-defined non-VCSB test method requires a correlation equation in order to predict designated test method results, then this correlation equation must be applied to the candidate instruments output to obtain measurement results for regulatory purposes using good laboratory practices.

(8) Any additional information requested by the Administrator and necessary to render a decision as to approval of the test method.

(9) Samples used for precision and accuracy determination must be retained for 90 days.

(10) Within 90 days of the receipt of materials required to be submitted under paragraphs (m)(1) through (9) of this section, the Administrator shall determine whether the test method is approved under this section.

(11) If the Administrator denies approval of the test method, within 90 days of receipt of all materials required to be submitted in paragraphs (m)(1) through (9) of this section, the Administrator will notify the applicant of the reasons for not approving the method. If the Administrator does not notify the applicant within 90 days of receipt of the application, then the test method shall be deemed approved.

(12) The Administrator may revoke approval of a test method under this section for cause, including, but not

limited to, a determination by the Administrator that the approved test method has proved to be inadequate in practice.

(13) An independent third-party scientific review and written report and verification of the information provided pursuant to paragraphs (m)(1) through (9) of this section. The report and verification shall be based upon a site visit and review of relevant documents and shall separately identify each item required by paragraphs (m)(1) through (9) of this section, describe how the independent third-party evaluated the accuracy of the information provided, state whether the independent third-party agrees with the information provided, and identify any exceptions between the independent third-party's findings and the information provided.

(i) The information required under this section must be conducted by an independent third party who is a professional chemist and statistician, or who is a chemical engineer, with the following qualifications:

(A) For a refiner, importer, oxygenate producer, and oxygenate blender, the independent third party must have at least a bachelor's degree in chemistry and statistics, or at least a bachelor's degree in chemical engineering, from an accredited college in the United States, or the independent third party must be a subject matter expert with equivalent knowledge and qualification, with professional work experience in the petroleum or oxygenate field, especially with a demonstrated good working knowledge of ASTM D6708 and ASTM D6299.

(B) [Reserved]

(ii) To be considered an independent third-party under this paragraph (m)(13):

(A) The third-party shall not be employed by the refiner, importer, oxygenate producer, or oxygenate blender, or any subsidiary or employee of the refiner, import facility, oxygenate producing facility, or oxygenate blender.

(B) The third party shall be free from any interest in the refiner's, importer's, oxygenate producer's, or oxygenate blender's business.

(C) The refiner, importer, oxygenate producer, or oxygenate blender shall be



free from any interest in the third-party's business.

(D) Use of a third-party that is debarred, suspended, or proposed for debarment pursuant to the Government-wide Debarment and Suspension regulations, 40 CFR part 32, or the Debarment, Suspension and Ineligibility provisions of the Federal Acquisition Regulations, 48 CFR part 9 subpart 9.4, shall be deemed in noncompliance with the requirements of this section.

(iii) The independent third-party shall retain all records pertaining to the verification required under this section for a period of five years from the date of creation and shall deliver such records to the Administrator upon request.

(iv) The independent third party must provide EPA documentation of his or her qualifications as described in this paragraph (m) as part of the scientific review.

(14) If the Administrator finds that an individual test facility has provided false or inaccurate information under this section, upon notice from the Administrator the approval shall be void ab initio.

(n) *Accuracy and Precision Statistical Quality Control (SQC) Requirements for the Absolute Fuel Parameters.* Beginning January 1, 2016, a test shall not be considered a test using an approved test method unless the following quality control procedures are performed separately for each instrument used to make measurements:

(1)(i) *Accuracy SQC.* Every facility shall conduct tests on every instrument with a commercially available gravimetric reference material, or check standard as defined in ASTM D6299 at least three times a year using good laboratory practices. The facility must pre-treat and assess results from the check standard testing after at least 15 testing occasions as described in section 8.2 of this standard practice. The facility must construct "MR" and "I" charts with control lines as described in section 8.4 and appropriate Annex sections of this standard practice. In circumstances where the absolute difference between the mean of multiple back-to-back tests of the standard reference material and the accepted reference value of the standard

reference material is greater than 0.75 times the published reproducibility of the test method, the cause of such difference must be investigated by the facility. Records of the standard reference materials measurements as well as any investigations into any exceedance of these criteria must be kept for a period of five years.

(ii) The expanded uncertainty of the accepted reference value of consensus named fuels shall have the following accuracy qualification criterion: Accuracy qualification criterion = square root  $[(0.75R)^2 + (0.75R)^2/L]$ , where L = the number of single results obtained from different labs used to calculate the consensus ARV.

(2)(i) *Precision SQC.* Every facility shall conduct tests on every instrument with a quality control material as defined in paragraph 3.2.8 in ASTM D6299 either once per week or once per every 20 production tests, whichever is more frequent. The facility must construct and maintain an "I" chart as described in section 8 and section A1.5.1 and a "MR" chart as described in section A1.5.4. Any violations of control limit(s) should be investigated by personnel of the facility and records kept for a period of five years.

(ii) *Validation of New QC Material.* When a test facility is making a transition from one batch of QC material to the next batch of QC material, the facility will either construct an "I" chart as described in section 8.7 and section A1.5.1 of ASTM D6299, or follow the "Q-Procedure" in Annex 1.9 of ASTM D6299. In following the Q-Procedure, if the plot of results from the "old" and "new" QC materials on its respective chart shows no special-cause signals, then the result of the "new" QC material will be considered valid.

(iii) For test facilities opting to use the Q-procedure, the first run on the new QC batch should be validated by either an overlap in-control result of the old batch, or by a single execution of an accompanying standard reference material. The new QC material result would be considered validated if the single result of the standard reference material is within the established site precision (R') of the Accepted Reference Value of the standard reference

material, as determined by ASTM D6792.

(iv) [Reserved]

(v) These records must be kept by the facility for a period of five years.

(o) *Accuracy and Precision Statistical Quality Control (SQC) Requirements for the Voluntary Consensus Standard Based (VCSB) Method-Defined Fuel Parameters.* Beginning January 1, 2016, a test shall not be considered a test using an approved test method unless the following quality control procedures are performed separately for each instrument used to make measurements:

(1)(i) *Accuracy SQC.* Every facility shall conduct tests of every instrument with a commercially available check standard as defined in ASTM D6299 at least three times a year using good laboratory practices. The check standard must be an ordinary fuel with levels of the fuel parameter of interest close to either the applicable regulatory standard or the average level of use for the facility. For facilities using a VCSB designated method defined test method, the Accepted Reference Value of the check standard must be determined by the respective designated test method for the fuel parameter following the guidelines of ASTM D6299. Facilities using a VCSB alternative method defined test method must use the Accepted Reference Value of the check standard as determined in a VCSB Inter Laboratory Crosscheck Program (ILCP) or a commercially available ILCP following the guidelines of ASTM D6299. If the Accepted Reference Value is not provided in the ILCP, accuracy must be assessed based upon the respective EPA-designated test method using appropriate production samples. The facility must pre-treat and assess results from the check standard testing after at least 15 testing occasions as described in section 8.2 of this standard practice. The facility must construct “MR” and “I” charts with control lines as described in section 8.4 and appropriate Annex sections of this standard practice. In circumstances where the absolute difference between the mean of multiple back-to-back tests of the standard reference material and the accepted reference value of the standard reference material is greater than 0.75 times the published reproducibility of

the test method, the cause of such difference must be investigated by the facility. Participation in a VCSB ILCP at least three times a year satisfies this Accuracy SQC requirement (Examples of ILCP: ASTM Reformulated Gasoline ILCP or ASTM motor gasoline ILCP). Records of the standard reference materials measurements as well as any investigations into any exceedance of these criteria must be kept for a period of five years.

(ii) The expanded uncertainty of the accepted reference value of consensus named fuels shall have the following accuracy qualification criterion: Accuracy qualification criterion = square root  $[(0.75R)^2 + (0.75R)^2/L]$ , where L = the number of single results obtained from different labs used to calculate the consensus ARV.

(2)(i) *Precision SQC.* Every facility shall conduct tests of every instrument with a quality control material as defined in paragraph 3.2.8 in ASTM D6299 either once per week or once per every 20 production tests, whichever is more frequent. The facility must construct and maintain an “I” chart as described in section 8 and section A1.5.1 and a “MR” chart as described in section A1.5.4. Any violations of control limit(s) should be investigated by personnel of the facility and records kept for a period of five years.

(ii) *Validation of New QC Material.* When a test facility is making a transition from one batch of QC material to the next batch of QC material, the facility will either construct an “I” chart as described in section 8.7 and section A1.5.1 of ASTM D6299, or follow the “Q-Procedure” in Annex 1.9 of ASTM D6299. In following the Q-Procedure if the plot of results from the “old” and “new” QC materials on its respective chart shows no special-cause signals, then the result of the “new” QC material will be considered valid.

(iii) For test facilities opting to use the Q-procedure, the first run on the new QC batch should be validated by either an overlap in-control result of the old batch, or by a single execution of an accompanying standard reference material. The new QC material result would be considered validated if the single result of the standard reference material is within the established site

precision (R') of the Accepted Reference Value of the standard reference material, as determined by ASTM D6792.

(iv) [Reserved]

(v) These records must be kept by the facility for a period of five years.

(p) *Accuracy and Precision Statistical Quality Control (SQC) Requirements for the Non-Voluntary Consensus Standard Based (Non-VCSB) Method-Defined Fuel Parameters*. Beginning January 1, 2016, a test shall not be considered a test using an approved test method unless the following quality control procedures are performed separately for each instrument used to make measurements:

(1)(i) *Accuracy SQC for Non-VCSB Method-Defined test methods with minimal matrix effects*. Every facility shall conduct tests on every instrument with a commercially available check standard as defined in the ASTM D6299 at least three times a year using good laboratory practices. The check standard must be an ordinary fuel with levels of the fuel parameter of interest close to either the applicable regulatory standard or the average level of use for the facility. Facilities using a Non-VCSB alternative method defined test method must use the Accepted Reference Value of the check standard as determined in either a VCSB Inter Laboratory Crosscheck Program (ILCP) or a commercially available ILCP following the guidelines of ASTM D6299. If the Accepted Reference Value is not provided in the ILCP, accuracy must be assessed based upon the respective EPA designated test method using appropriate production samples. The facility must pre-treat and assess results from the check standard testing after at least 15 testing occasions as described in section 8.2 of this standard practice. The facility must construct "MR" and "I" charts with control lines as described in section 8.4 and appropriate Annex sections of this standard practice. In circumstances where the absolute difference between the mean of multiple back-to-back tests of the standard reference material and the accepted reference value of the standard reference material is greater than 0.75 times the published reproducibility of the fuel parameter's respective des-

ignated test method, the cause of such difference must be investigated by the facility. Records of the standard reference materials measurements as well as any investigations into any exceedance of these criteria must be kept for a period of five years.

(ii) The expanded uncertainty of the accepted reference value of consensus named fuels shall have the following accuracy qualification criterion: Accuracy qualification criterion = square root  $[(0.75R)^2 + (0.75R)^2/L]$ , where L = the number of single results obtained from different labs used to calculate the consensus ARV.

(2)(i) *Accuracy SQC for Non-VCSB Method-Defined test methods with high sensitivity to matrix effects*. Every facility shall conduct tests on every instrument with a production fuel on at least a quarterly basis using good laboratory practices. The production fuel must be representative of the production fuels that are routinely analyzed by the facility. The Accepted Reference Value of the production fuel must be determined by the respective reference installation of the designated test method for the fuel parameter following the guidelines of ASTM D6299. The facility must pre-treat and assess results from the check standard testing after at least 15 testing occasions as described in section 8.2 of this standard practice. The facility must construct "MR" and "I" charts with control lines as described in section 8.4 and appropriate Annex sections of this standard practice. In circumstances where the absolute difference between the mean of multiple back-to-back tests of the standard reference material and the accepted reference value of the standard reference material is greater than 0.75 times the published reproducibility of the test method must be investigated by the facility. Documentation on the identity of the reference installation and its control status must be maintained on the premises of the method-defined alternative test method. Records of the standard reference materials measurements as well as any investigations into any exceedances of this criterion must be kept for a period of five years.

(ii) Each facility is required to send every 20th production batch of gasoline

or diesel fuel to EPA's laboratory, along with the facility's measurement result used to certify the batch using the respective method-defined non-VCSB test method. The EPA retains the right to return such sample on a blind basis for a required reanalysis on the respective method-defined non-VCSB test method within 180 days upon receipt of such sample.

(3)(i) *Precision SQC*. Every facility shall conduct tests on every instrument with a quality control material as defined in paragraph 3.2.8 in ASTM D6299 either once per week or once per every 20 production tests, whichever is more frequent. The facility must construct and maintain an "I" chart as described in section 8 and section A1.5.1 and a "MR" chart as described in section A1.5.4. Any violations of control limit(s) should be investigated by personnel of the facility and records kept for a period of five years.

(ii) *Validation of New QC Material*. When a test facility is making a transition from one batch of QC material to the next batch of QC material, the facility will either construct an "I" chart as described in section 8.7 and section A1.5.1 of ASTM D6299, or follow the "Q-Procedure" in Annex 1.9 of ASTM D6299. In following the Q-Procedure, if the plot of results from the "old" and "new" QC materials on its respective chart shows no special-cause signals, then the result of the "new" QC material will be considered valid.

(iii) For test facilities opting to use the Q-procedure, the first run on the new QC batch should be validated by either an overlap in-control result of the old batch, or by a single execution of an accompanying standard reference material. The new QC material result would be considered validated if the single result of the standard reference material is within the established site precision (R') of the Accepted Reference Value of the standard reference material, as determined by ASTM D6792.

(iv) [Reserved]

(v) These records must be kept by the facility for a period of five years.

(q) *Record retention requirements for the test methods approved under this subpart*. Each individual test facility must retain records related to the establish-

ment of accuracy and precision values, all test method documentation, and any statistical quality control testing and analysis under this section using good laboratory practices for a period for five years.

(r) *Materials incorporated by reference*. The published materials identified in this section are incorporated by reference into this section with the approval of the Director of the Federal Register under 5 U.S.C. 552(a) and 1 CFR part 51. To enforce any edition other than that specified in this section, a document must be published in the FEDERAL REGISTER and the material must be available to the public. All approved materials are available for inspection at the Air and Radiation Docket and Information Center (Air Docket) in the EPA Docket Center (EPA/DC) at Rm. 3334, EPA West Bldg., 1301 Constitution Ave. NW., Washington, DC. The EPA/DC Public Reading Room hours of operation are 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number of the EPA/DC Public Reading Room is (202) 566-1744, and the telephone number for the Air Docket is (202) 566-1742. These approved materials are 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\\_regulations/ibr\\_locations.html](http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html). In addition, these materials are available from the sources listed below.

(1) *ASTM International material*. The following standards are available from ASTM International, 100 Barr Harbor Dr., P.O. Box C700, West Conshohocken, PA 19428-2959, (877) 909-ASTM, or <http://www.astm.org>:

(i) ASTM D86-07, Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure, approved January 15, 2007 ("ASTM D86").

(ii) ASTM D1319-13, Standard Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption, approved May 1, 2013 ("ASTM D1319").

(iii) ASTM D3606-10, Standard Test Method for Determination of Benzene and Toluene in Finished Motor and

## § 80.48

## 40 CFR Ch. I (7–1–19 Edition)

Aviation Gasoline by Gas Chromatography, approved October 1, 2010 (“ASTM D3606”).

(iv) ASTM D5191–13, Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method), approved December 1, 2013 (“ASTM D5191”).

(v) ASTM D5599–00 (Reapproved 2010), Standard Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection, approved October 1, 2010 (“ASTM D5599”).

(vi) ASTM D6299–13, Standard Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance, approved October 1, 2013 (“ASTM D6299”).

(vii) ASTM D6667–10, Standard Test Method for Determination of Total Volatile Sulfur in Gaseous Hydrocarbons and Liquefied Petroleum Gases by Ultraviolet Fluorescence, approved October 1, 2010 (“ASTM D6667”).

(viii) ASTM D6708–13, Standard Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material, approved May 1, 2013 (“ASTM D6708”).

(ix) ASTM D6792–13, Standard Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories, approved May 15, 2013 (“ASTM D6792”).

(x) ASTM D7039–13, Standard Test Method for Sulfur in Gasoline, Diesel Fuel, Jet Fuel, Kerosine, Biodiesel, Biodiesel Blends, and Gasoline-Ethanol Blends by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry, approved September 15, 2013, (“ASTM D7039”).

(2) [Reserved]

[79 FR 23633, Apr. 28, 2014, as amended at 80 FR 9091, Feb. 19, 2015]

### § 80.48 Augmentation of the complex emission model by vehicle testing.

(a) The provisions of this section apply only if a fuel claims emission reduction benefits from fuel parameters that are not included in the complex emission model or complex emission model database, or if the values of fuel parameters included in the complex

emission model set forth in § 80.45 fall outside the range of values for which the complex emission model is deemed valid.

(b) To augment the complex emission model described at § 80.45, the following requirements apply:

(1) The petitioner must obtain prior approval from the Administrator for the design of the test program before beginning the vehicle testing process. To obtain approval, the petitioner must at minimum provide the following information: the fuel parameter to be evaluated for emission effects; the number and description of vehicles to be used in the test fleet, including model year, model name, vehicle identification number (VIN), mileage, emission performance (exhaust THC emission level), technology type, and manufacturer; a description of the methods used to procure and prepare the vehicles; the properties of the fuels to be used in the testing program (as specified at § 80.49); the pollutants and emission categories intended to be evaluated; the precautions used to ensure that the effects of the parameter in question are independent of the effects of other parameters already included in the model; a description of the quality assurance procedures to be used during the test program; the statistical analysis techniques to be used in analyzing the test data, and the identity and location of the organization performing the testing.

(2) Exhaust emissions shall be measured per the requirements of this section and § 80.49 through § 80.62.

(3) The nonexhaust emission model (including evaporative, running loss, and refueling VOC and toxics emissions) shall not be augmented by vehicle testing.

(4) The Agency reserves the right to observe and monitor any testing that is performed pursuant to the requirements of this section.

(5) The Agency reserves the right to evaluate the quality and suitability of data submitted pursuant to the requirements of this section and to reject, re-analyze, or otherwise evaluate such data as is technically warranted.

(6) Upon a showing satisfactory to the Administrator, the Administrator