

Oxygen and Oxygenate Content in Gasoline Spreadsheet Example Key for Requirements at 40 CFR 80.47(f) and 80.47(l)

Compliance Division
Office of Transportation and Air Quality
U.S. Environmental Protection Agency
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Introduction: On December 4, 2020, EPA promulgated regulatory streamlining requirements (see 85 FR 78412).

Beginning January 1, 2021, for oxygenate content in gasoline, a test facility must self-qualify a voluntary consensus-based standard body (VCSB) alternative test method to show that it has met the precision requirements codified in the regulations at §1090.1365(b). A test facility must also self-qualify that it has met the qualification criteria for accuracy by conducting an ASTM D6708 assessment as codified in the regulations at §1090.1365(d). The regulations also specify criteria for the designated EPA referee test method reference installation, ASTM D5599, used to qualify accuracy of method defined test method used to measure method defined fuel parameter at §1090.1365(d).

If your test facility was utilizing the designated EPA referee test method, ASTM D5599, (IBR §1090.95(c)(24)), the regulations provide for an exemption in meeting these precision and accuracy self-qualification requirements. It is important to note, the on-going statistical quality control requirements at §1090.1375 apply to all methods, including the EPA referee test method.

The following spreadsheet template key applies to any party self-qualifying in meeting the PBMS requirements at §1090.1365(b) and §1090.1365(d). This guidance deals only with the self-qualification of analytical test methods at a testing facility for measuring oxygenate content in gasoline.

The discussions of the applicable regulations in this document are not verbatim. The discussions of the applicable regulations in this document are not verbatim. The reader is encouraged to read and become familiar with the applicable regulations of §1090.1360, §1090.1365, §1090.1370 and §1090.1375¹. These instructions are intended to help a test facility self-qualify a VCSB alternative analytical test method for the measurement of oxygen and oxygen content in gasoline.

Applicable Dates: These requirements for method qualification under §1090.1365 became effective on January 1, 2021.

Note: Please see below for instructions on use of this spreadsheet example key along with its associated spreadsheet example for oxygenate content in gasoline which is provided by the Agency for determining compliance. See Part I for precision criteria of §1090.1365(b). See part II for the ASTM D6708-19a accuracy assessment requirements of §1090.1365(d). See Part III for reference installation requirement of §1090.1370 along with other PBMS information in Part III. We encourage parties to use this spreadsheet example key and its associated spreadsheet example for oxygenate content in gasoline as an affirmative defense in meeting the PBMS requirements at §1090.1360, §1090.1365, §1090.1370 and §1090.1375.

¹ See: <https://www.govinfo.gov/content/pkg/FR-2020-12-04/pdf/2020-23164.pdf>

Part I - Precision demonstration for oxygenate content in gasoline.

Precision Criteria, §1090.1365(b) - the maximum allowable standard deviation computed from the results of a minimum of 20 tests made over 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 D5599-18 (IBR 1090.95(c)(24)). You may make up to 4 separate measurements in a 24-hour period, as long as the interval between measurements is at least 4 hours. Example: A gasoline sample containing 3 percent by mass oxygenates: Maximum allowable standard deviation of 20 tests $\leq 0.3 * (0.13 * 3 \text{ percent by mass}^{0.83}) = 0.097 \text{ percent by mass}$.¹

- A. In the workbook entitled "VCSB oxygen content gas test method sprdsht example", locate the worksheet entitled, "Oxygenate Precision Demonstrate". Enter Precision demonstration data in the light shaded green areas of the worksheet.
 1. Test results should be entered in spreadsheet to at least the nearest thousandth mass percent for each calibrated oxygenate (0.001 mass percent).
 2. The date and time of each test measurement must be reported.
 3. Please include the laboratory sample test identification number for each test result.
- B. After entering the data into the light shaded green area of the "Oxygenate Precision Demonstration" worksheet, go to the "File" menu at the top of the screen and select "Save" to save your data. Once all the data are entered into the "Oxygenate Precision Demonstration" worksheet, the standard deviation of the data set (located in cell B16), and an indication as to whether the Oxygenate precision criterion are met will be determined by the worksheet. The indication of "PASSED" or "FAILED" is located in the worksheet in cell B15, after the question, "Is Oxygenate Content Precision Criterion Met?". If the worksheet is missing required data, an indication of "REQUIRED DATA MISSING" will appear after this question. There is a QC data entry check for each test result in column E (i.e., if data is entered in a test result cell, an indication of "OK" will appear next to that cell, but if no data is entered in a test result cell, an indication of "DATA REQUIRED IN CELL #" will appear next to that cell). Note: If the applicant wishes to include more than the 20 minimum tests, please report the additional data by adding rows to the spreadsheet.²

¹ A laboratory 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 Additional rows may be inserted to accommodate the extra data points. If these rows are added in the middle (say around row 25), the equations that analyze the data will be automatically adjusted. If difficulties are encountered in doing this, please call for help.

Part II. ASTM D6708-19a Accuracy demonstration for oxygenate content in gasoline (§1090.1365(d)).

- A. In the workbook entitled “Spreadsheet Example VCSB oxygenates and oxygen content gasoline test method”, locate the worksheet entitled, “D6708 Assessment Accuracy”. Enter applicable information as discussed below in the D6708 Assessment Accuracy worksheet.
- B. Include information reported in the test method documentation to the user of the voluntary consensus standard body (VCSB) organization test method, including a description of the technology and/or instrumentation that makes the method functional.
- C. Include information reported in the test method that demonstrates the test facility is using a VCSB test method ASTM D6708-19a assessment (§1090.1365(d), IBR 1090.95(c)(33)). Indicate by typing “Yes” in cell “B15”,
- D. Include the correlation equation via an ASTM D6708-19a accuracy assessment to utilize for reporting purposes for the fuel parameter in cell “B16”. If the ASTM D6708 assessment between the candidate VCSB alternative test method and the VCSB EPA referee test method results in a “null” comparison, then type the word “null” in cell “B16” indicating the ASTM D6708 assessment has determined the alternative test method provides equivalent results to its respective EPA referee test method, ASTM D5599-18 (IBR 1090.95(c)(24))².

Part III – Additional PBMS Information.

- A. §1090 CFR 1365. For test methods that are approved by a voluntary consensus-based standards body (VCSB) organization, such as the American Society for Testing and Materials (ASTM) or International Standards Organization (ISO), each individual test facility must demonstrate through self-qualification that the applicable criteria for accuracy and precision specified under §1090.1365 are met (Please see Part I and Part II of this spreadsheet example key). The self-qualification of the test method is limited to the single test facility that performed the testing for accuracy and precision.
- B. §1090.1360(5)(i). Testing you performed to qualify alternative procedures under 40 CFR part 80.47 continues to be valid for making the demonstrations required in part 1090.
- C. §1090 CFR 1370. Reference installation requirements at §1090.1370 are used for grounding the EPA referee test method, ASTM D5599-18 (IBR 1090.95(c)(24)), for use in the ASTM D6708 (IBR 1090.95(c)(33)), accuracy assessment with the candidate alternative test method.
- D. §1090 CFR 1375. Quality Assurance procedures for oxygenate content in gasoline measurement instrumentation. A test method shall not be considered a test using an approved test method unless the quality control procedures specified at 1090.1375 for both precision and accuracy are met separately for each instrument at the test facility.
- E. §1090.1345. See this section of the regulations for sample retention requirements that apply to your specific test facility.
- F. §1090 CFR 1200. Record retention requirements for approved test methods. Each individual test facility must retain records related to the establishment of accuracy and precision values, all test method documentation, and any quality control test and analysis under §1090.1365, §1090.1370 and §1090.1375 for five years.

² ASTM D5599–18, Standard Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection, approved June 1, 2018 (“ASTM D5599”); IBR approved for §§ 1090.1360(d) and 1090.1365(b).

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