

**Oxygen and Oxygenate Content in
Gasoline Spreadsheet Example Key
for Requirements at 40 CFR 80.47(f)
and 80.47(1)**

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Office of Transportation and Air Quality
U.S. Environmental Protection Agency
November 2016

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Introduction: On April 28, 2014, EPA promulgated new Performance Based Analytical Test Method Approach (PBATMA) requirements for oxygen and oxygenate content in gasoline (please see the Federal Register 79 FR 23414).

Beginning January 1, 2016, for oxygen and oxygenate content in gasoline, a test facility must self-qualify a Voluntary Consensus Standards Body (VCSB) test method to show that it has met the precision requirements codified in the regulations at 40 CFR 80.47(f). 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 40 CFR 80.47(l). The regulations also specify criteria for the designated test method reference installation used to qualify accuracy of method defined test methods used to measure method defined fuel parameter at 40 CFR 80.47(k). If your test facility was utilizing the designated primary test method, ASTM D5599, codified in the regulations at 40 CFR 80.46(g)(1) prior to October 28, 2013, the regulations provide for an exemption in meeting these precision and accuracy self-qualification requirements.

The following spreadsheet template key applies to any party self-qualifying in meeting the PBATMA requirements at 40 CFR 80.47(f) and 40 CFR 80.47(l). This guidance deals only with the self-qualification of analytical test methods at a testing facility for measuring oxygen and oxygenate content in gasoline.

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 Subpart I of 40 CFR Part 80. These instructions are intended to help a test facility self-qualify a VCSB analytical test method for the measurement of oxygen and oxygen content in gasoline.

Applicable Dates: These requirements for method qualification under § 80.47 become effective on January 1, 2016.

Note: Please see below for instructions on use of this spreadsheet example key along with its associated spreadsheet example for oxygen and oxygen content in gasoline which is provided by the Agency for determining compliance with the precision criteria of § 80.47(f) and the ASTM D6708-13 accuracy assessment requirements of § 80.47(l). We encourage parties to use this spreadsheet example key and its associated spreadsheet example for aromatics in gasoline as an affirmative defense in meeting the Performance Based Analytical Test Method Approach requirements at 40 CFR 80.47(f) and 40 CFR 80.47(l).

Instructions for use of spreadsheet example key for evaluating method precision and accuracy.

I. Precision demonstration for oxygen and oxygen content in gasoline.

Precision Criteria, § 80.47(e)(1) - 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” divided by 2.77, where “R” equals the ASTM reproducibility of ASTM D5599 (Example: A gasoline sample containing 3 percent by mass oxygenates: Maximum allowable standard deviation of 20 tests $\leq 0.3 \times (0,32 \text{ percent by mass} / 2.77) = 0.10$ percent by mass). The 20 results must be a series of tests with a sequential record of analysis and no emissions.¹

A. In the workbook entitled “Spreadsheet example VCSB oxygen content gasoline test method”, locate the worksheet entitled, “Oxygenate Precision Demonstration”. Enter Precision demonstration data in the light shaded green areas of the worksheet.

Notes:

1. Test results must be reported in percent by mass to the number of significant digits specified in the method description or, if no such precision is indicated, to as many digits to the right of the decimal point as appear on the instrument readout up to three.
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 B17), 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 cell B16 in the worksheet, 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.

- II. ASTM D6708-13 Accuracy demonstration for oxygenates and oxygenate content in gasoline.**
- 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 Standards 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 assessment. Indicate by typing “Yes” in cell “B16”,
 - D. Include the correlation equation to utilize for reporting purposes for the fuel parameter in cell “B18”. If the ASTM D6708 assessment between the candidate VCSB alternative test method and the VCSB designated primary method results in a “null” comparison, that type the work “null” in cell “B18” indicating the ASTM D6708 assessment has determine the alternative test method provides equivalent results to its respective designated primary test method.
- III. 40 CFR 80.47(q). 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 title 40 CFR sections 80.47 for five years.

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OMB Control No. 2060-0692
Approval expires: 5/31/2019

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