

**Sulfur in Butane Spreadsheet
Example Key for Requirements at
40 CFR 80.47(c)(1) and 80.47(c)(2)**

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

November 2016

DISCLAIMER

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The general description of the self-qualification process provided here may not apply to a particular situation. Interested parties are free to raise questions about the substance of this spreadsheet example key, its associated spreadsheet example, and its applicability to a particular situation. EPA may adopt approaches on a case-by-case basis that differ from those described in this spreadsheet example key.

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

Beginning January 1, 2016, for sulfur blended into butane for use with gasoline at the 30 ppm sulfur average and 80 ppm cap standard for sulfur in gasoline and beginning January 1, 2017, for sulfur blended into butane for use with gasoline at the 10 ppm sulfur average and 80 ppm cap standard, a test facility must self-qualify that it has met the precision and accuracy requirements codified in the regulations at 40 CFR 80.47(c)(1) and 40 CFR 80.47(c)(2). If your test facility was utilizing the designated primary test method, ASTM D6667, codified in the regulations at 40 CFR 80.46(a)(2) prior to October 28, 2013, the regulations provide for an exemption in meeting these precision and accuracy self-qualification requirements.

The following spreadsheet example key applies to any party self-qualifying in meeting the PBATMA requirements at 40 CFR 80.47(c). This guidance deals only with the self-qualification of analytical test methods at a testing facility for measuring sulfur in butane.

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 sulfur in butane.

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 sulfur in butane which is provided by the Agency for determining compliance with the accuracy and precision criteria of § 80.47(c)(1) and of § 80.47(c)(2). We encourage parties to use this spreadsheet example key and its associated spreadsheet example for sulfur in butane as an affirmative defense in meeting the Performance Based Analytical Test Method Approach requirements at 40 CFR 80.47(c)(1) and 40 CFR 80.47(c)(2).

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

I. Precision demonstration for sulfur blended into butane for gasoline subject to the 30 ppm sulfur average and 80 ppm sulfur cap standard, and beginning January 1, 2017, gasoline subject to the 10 ppm sulfur average and 80 ppm sulfur cap standard.

Precision Criteria, § 80.47(c)(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 1.5 times the repeatability "r" divided by 2.77, where "r" equals the ASTM repeatability of ASTM D6667 (Example: A 10 ppm sulfur gasoline sample: Maximum allowable standard deviation of 20 tests $\leq 1.5 \cdot (9.22 \text{ ppm} / 2.77) = 4.99 \text{ ppm}$). 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 sulfur butane test method", locate the worksheet entitled, "10 ppm S Precision". Enter data in the light shaded green areas of the worksheet. For an example of the "10 ppm S Precision" worksheet with data completely entered, please see the worksheet entitled, "EX – 10 ppm S Precision".

Notes:

1. Test results must be reported in parts per million (ppm) 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 "10 ppm S Precision" 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 "10 ppm S Precision" worksheet, the standard deviation of the data set (located in cell B16), and an indication as to whether the 10 ppm sulfur precision criterion are met will be determined by the worksheet. The indication of "PASSED" or "FAILED" is located in cell B15 in the worksheet, after the question, "Is 10 ppm Sulfur 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. Accuracy demonstration for sulfur in butane that is blended into gasoline subject to the 30 ppm sulfur average and 80 ppm sulfur cap standard, and beginning January 1, 2017, gasoline subject to the 10 ppm sulfur average and 80 ppm sulfur cap standard.

- Accuracy Criterion -
1. The arithmetic average of a continuous series of at least 10 tests performed on a commercially available gravimetric sulfur standard (CAGSS) in the range of 1-10 ppm sulfur shall not differ from the accepted reference value of that standard by more than 0.47 ppm sulfur.³
 2. The arithmetic average of a continuous series of at least 10 tests performed on a CAGSS in the range of 10-20 ppm sulfur shall not differ from the accepted reference value of that standard by more than 0.94 ppm sulfur.⁴

- A. Locate the worksheet entitled, "10 ppm S Accuracy". Enter data in the light shaded green areas of the worksheet. For an example of the "10 ppm S Accuracy" worksheet with data completely entered, please see the worksheet entitled, "EX - 10 ppm S Accuracy".

Notes:

1. Test results must be reported in parts per million (ppm) to the number of 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. It is recommended that the date and time of each test measurement be reported.
 3. Please include the laboratory sample test identification number for each test result.
 4. In the appropriate rows, enter the "Vendor Name of Gravimetric Standard", "Lot Identification Number of Gravimetric Standard", and "Accepted Reference Value of the Gravimetric Standard (ppm)" in parts per million for both the 1-10 ppm and 10-20 ppm sulfur gravimetric standards.
 5. Since the Test Method and Laboratory Identification information for this demonstration are the same as the Test Method and Laboratory Identification information in the "10 ppm S precision" worksheet, for your convenience, this information is automatically referenced from the "10 ppm S Precision" worksheet.
- B. After entering the data into the light shaded green area of the worksheet as described above, go to the "File" menu at the top of the screen and select "Save" to save all of the entered data. Once all data are entered into the "10 ppm S Accuracy" worksheet, this worksheet will calculate the arithmetic average for both the 1-10 ppm sulfur data set (located in cell B18) and the 10-20 ppm sulfur data set (located in cell I18). This worksheet will also calculate the difference between the arithmetic average of the data set and the accepted reference value of each respective gravimetric standard (located in cell B22 for the 1-10 ppm accuracy demonstration & located in cell I22 for the 10-20 ppm accuracy demonstration). This worksheet will indicate whether the 10 ppm sulfur accuracy criteria are met for the candidate test method by saying "**PASSED**" or "**FAILED**" in the cell after the questions, "Is 1-10 ppm Sulfur Accuracy Criterion Met?" (located in cell B17) and "Is 10-20 ppm Sulfur Accuracy Criterion Met?" (located in cell I17). Both of these accuracy criteria must be met in order for the test method to be considered to have met the 10 ppm accuracy criteria. If the worksheet is missing required data, an indication of "REQUIRED DATA MISSING" will appear after the applicable question. There is a QC data entry check for each test result in column E and column L (i.e., if data is entered in a test result cell, an indication

³ Individual test results shall be compensated for any known chemical interferences.

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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). There is also a QC data entry check on the concentration of the gravimetric standard in cell E20 and cell L20 (i.e., for either the 1 to 10 ppm sulfur accuracy demonstration or 10 to 20 ppm sulfur accuracy demonstration, if the concentration of gravimetric standard falls within the applicable concentration range, an indication of "OK" will appear in the respective cell, if the concentration of the gravimetric standard falls below the applicable concentration range, an indication of "ARV TOO LOW IN CONCENTRATION" will appear in the respective cell, if the concentration of the gravimetric standard is above the applicable concentration range, an indication of "ARV TOO HIGH IN CONCENTRATION" will appear in the respective cell). Note: If the applicant wishes to include more than the 10 minimum tests, please report the additional data by inserting rows into the spreadsheet.⁵

- C. 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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⁵ Additional rows may be inserted to accommodate the extra data points. If these rows are inserted in the middle of the range (say after row 30) the equations for the average and other functions will be automatically adjusted. Extra rows inserted for one of the two standard levels, while appearing in the range for the other standard level, will not affect the calculations for the level where no data were added.