

# Sulfur in Butane Spreadsheet

## Example Key for Requirements at 40 CFR 80.47(c)(1) and 80.47(c)(2)

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

January 2024

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Introduction: On December 4, 2020, EPA promulgated regulatory streamlining requirements (see 85 FR 78412).

One of the provisions of this final rule, explained in §1090.1365, requires that each test facility must meet specified accuracy and precision criteria with their chosen alternative test method for sulfur butane. Evidence of having met these requirements for test methods must be demonstrated by the test facility via self-qualification before using the method to make measurements for demonstrating compliance.

If your test facility is utilizing the EPA referee test method, ASTM D6667, 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 example key applies to any party self-qualifying in meeting the PBMS requirements at §1090.1365. 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 at §1090.1360, §1090.1365 and §1090.1375. These instructions are intended to help a test facility self-qualify an alternative test method for the measurement of sulfur in butane.

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 sulfur in butane which is provided by the Agency for determining compliance with the accuracy and precision criteria of §1090.1365. 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 PBMS requirements at §1090.1365.

Part I - Precision demonstration instructions for sulfur in butane that is blended into gasoline subject to the 30 ppm sulfur average and 80 ppm sulfur cap standard.

Precision Criteria – (§1090.1365(b)) provides the precision criteria for sulfur in butane where the precision demonstration covers a minimum of a minimum of 20 tests over 20 days (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.). See Table 1 to paragraph (b)(3) of §1090.1365 for specific precision criteria for sulfur in butane. The 20 results must be a series of tests with a sequential record of analysis and no emissions.<sup>1</sup>

- 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 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.<sup>2</sup>

Part II - Accuracy demonstration instructions for sulfur in butane that is blended into gasoline subject to the 30 ppm sulfur average and 80 ppm sulfur cap standard.

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<sup>1</sup> 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.

<sup>2</sup> 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.

Accuracy Criterion (§1090.1365(c)(3)(ii)) – The arithmetic average of a continuous series of at least 10 tests performed on a commercially available gravimetric sulfur standard in the range of 1 to 10 ppm sulfur shall not differ from the accepted reference value of that standard by more than 0.04678725 ppm sulfur.<sup>3</sup>

- 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 to the nearest whole parts per million (ppm), see §1090.1350(c).
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 is 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 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). 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). 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). This accuracy criteria must be met 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 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 (i.e., for the 1 to 10 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

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<sup>3</sup> Individual test results shall be compensated for any known chemical interferences.

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.<sup>5</sup>

Part III - Additional information for voluntary consensus-based standards body Test Method Self-Qualification

- 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 for accuracy and precision criteria specified under §1090.1365 are met (Please see Part I 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 this part.
- C. §1090.1375. Quality Assurance procedures for sulfur 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 are met separately for each instrument at the test facility.
- D. §1090.1345. See this section of the regulations for sample retention requirements that apply to your specific test facility.
- E. §1090.1200. 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 title §1090.1365 and §1090.1375 for five years.

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<sup>5</sup> 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.