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Sulfur in diesel fuel (15 ppm sulfur Ultra-low sulfur diesel fuel and 500 ppm sulfur locomotive marine fuel) Spreadsheet Example Key

**Compliance Division
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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 in on-road or non-road diesel fuel additives and/or diesel fuel as well as locomotive marine fuel. 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. There are separate accuracy and precision criteria for diesel fuels and additives subject to the two sulfur standards --15 ppm and 500 ppm.

Depending on your test facility situation, you may be required just to meet the 15 ppm diesel sulfur accuracy and precision criteria, you may choose to meet the 500 ppm locomotive marine fuel accuracy and precision criteria, or may be required to meet both 15 ppm diesel sulfur and 500 ppm locomotive marine fuel accuracy and precision criteria.

If your test facility has chosen to use the EPA referee test method, ASTM D 2622 (IBR §1090.95(c)(8)), it is not necessary for your test facility to qualify these methods for precision or accuracy (see Table 1 to paragraph(d) of §1090.1360). 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 guidance applies to any party self-qualifying a Voluntary Consensus-based Standards Body (VCSB) or Non-VCSB alternative test method at their testing facility for sulfur diesel at the 15 ppm sulfur level and locomotive marine fuel at the 500 ppm sulfur level. This information deals only with the self-qualification of alternative test methods at a testing facility for measuring sulfur in diesel fuel additives or sulfur in diesel fuel.

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 §1090.1375.¹ These instructions are intended to help a test facility self-qualify an alternative test method for the measurement of sulfur in diesel motor fuel or diesel motor fuel additives and sulfur in locomotive marine fuel.

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

Note: Please see Part I for instructions on use of a spreadsheet example provided by the Agency for determining compliance with the precision criteria of §1090.1365(b) for sulfur in diesel fuel at the 15 ppm sulfur standard. Please see Part II for instructions on use of the spreadsheet example for determining compliance with accuracy criteria §1090.1365(c)(3)(ii) for sulfur in diesel fuel at the 15 ppm sulfur standard. Please see Part III of instructions for precision criteria for locomotive marine fuel at §1090.1365(b). Please see Part IV of instructions for accuracy criteria for locomotive marine fuel at §1090.1365(c)(3)(ii). We encourage parties to use this spreadsheet example to structure their development of the information and data needed for test method self-qualification at their test facility.. Please see Part V for additional PBMS requirements under part 1090.

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

Part I. - Precision demonstration instructions for motor vehicle diesel fuel and diesel fuel additives subject to the 15 ppm sulfur standard.

Precision Criteria (§1090.1365(b)) - provides the precision criteria for diesel fuel with a sulfur content in the range of 5-15 ppm, 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).² See Table 1 to paragraph (b)(3) of §1090.1365 for specific precision criteria for 15 ppm sulfur diesel fuel and 500 ppm locomotive marine fuel.

- A. In the workbook entitled “Diesel Sulfur Test Method Spreadsheet Example”, locate the worksheet entitled, “15 ppm S Precision”. Enter data in the light shaded green areas of the worksheet. For an example of the “15 ppm S Precision” worksheet with data completely entered, please see the worksheet entitled, “EX - 15 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 “15 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 “15 ppm S Precision” worksheet, the standard deviation of the data set (located in cell B19), and an indication as to whether the 15 ppm sulfur precision criterion are met will be determined by the worksheet. The indication of “PASSED” or “FAILED” is in cell B18 in the worksheet, after the question, “Is 15 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

Part II - Accuracy demonstration instructions for motor vehicle diesel fuel and diesel fuel additives subject to the 15 ppm sulfur standard.

Accuracy Criterion - 1. §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 (CAGSS) in the range of 10-20 ppm sulfur shall not differ from the accepted reference value of that standard by more than 0.54 ppm sulfur.³

- A. Locate the worksheet entitled, “15 ppm S Accuracy”. Enter data in the light shaded green areas of the worksheet. For an example of the “15 ppm S Accuracy” worksheet with data completely entered, please see the worksheet entitled, “EX - 15 ppm S Accuracy”.

² 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.

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

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. It is recommended that the date and time of each test measurement be reported.
 3. 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 10-20 ppm sulfur gravimetric standards.
- 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 "15 ppm S Accuracy" worksheet, this worksheet will calculate the arithmetic average for the 10-20 ppm sulfur data set (located in cell B21). 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 B25 for the 10-20 ppm accuracy demonstration). This worksheet will indicate whether the 15 ppm sulfur accuracy criteria are met for the candidate test method by saying "PASSED" or "FAILED" in the cell after the questions, "Is 10-20 ppm Sulfur Accuracy Criterion Met?" (located in cell B120). This accuracy criteria must be met for the test method to be considered to have met the 15 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 E24 (i.e., for the 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

Part III - Precision demonstration instructions for locomotive marine fuel subject to the 500-ppm sulfur standard.

Precision Criterion (§1090.1365(b)) - provides the precision criteria for locomotive marine fuel with a sulfur content of in the range of 350 ppm to 500 ppm.⁶

- A. Locate the worksheet entitled, "500 ppm S Precision". Follow the instructions for Section I above as indicated for "15 ppm S Precision", but enter the data for the 500ppm precision demonstration rather than that for the 15ppm precision demonstration. For an example of the "500 ppm S Precision" worksheet with data completely entered, please see the worksheet entitled, "EX - 500 ppm S Precision".

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.

⁶ 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.

Part IV - Accuracy demonstration instructions for motor vehicle diesel fuel subject to the 500 ppm sulfur standard.

Accuracy Criterion - 1. §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 (CAGSS) in the range of 450-500 ppm sulfur shall not differ from the accepted reference value of that standard by more than 8.65 ppm sulfur.⁷

- A. Locate the worksheet entitled, "500 ppm S Accuracy". Follow the instructions for section II above "15 ppm S Accuracy", but enter the data for the 500ppm accuracy demonstration rather than that for the 15ppm accuracy demonstration. For an example of the "500 ppm S Accuracy" worksheet with data completely entered, please see the worksheet entitled, "EX - 500 ppm S Accuracy".

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

- A. §1090.1365. 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 Parts I through Parts IV 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 demonstrations under part 1090.
- C. §1090.1375. 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 precision and accuracy 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 §1090.1365, §1090.1370 and §1090.1375 for a minimum of 5 years.

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⁷⁷ Individual test results shall be compensated for any known chemical interferences.