

ECA Marine Fuel Test Method Spreadsheet Example Key

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

For ECA marine fuel at the 1,000 ppm sulfur level, if your test facility has chosen to use the designated test method, ASTM D 2622 (IBR §1090.95(c)(8)), it is not necessary for your test facility to self-qualify this test method 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.

§1090.1365 describes the accuracy and precision criteria for the applicable standard above. The following guidance applies to any party self-qualifying an alternative test method at their testing facility for ECA marine fuel. This information deals only with the initial self-qualification of test methods at a testing facility for measuring sulfur in ECA marine 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 a VCSB test method for the measurement of sulfur in ECA marine fuel.

Applicable Dates: These requirements for method self-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 ECA marine fuel. 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 ECA marine fuel. We encourage parties to use this spreadsheet to structure their development of the information and data needed for test method self-qualification at their test facility. Please see Part III 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 ECA marine fuel subject to the 1,000 ppm sulfur standard.

Precision Criteria (§1090.1365(b)) - a standard deviation less than 20.1 ppm, computed from the results of a minimum of 20 repeat tests made over 20 days on samples taken from a single homogeneous commercially available ECA marine fuel with a sulfur content in the range of 900-1,000 ppm. 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.¹

- A. In the workbook entitled “ECA Marine Fuel Sulfur Test Spreadsheet Example”, locate the worksheet entitled, “1,000 ppm S Precision”. Enter data in the light shaded green areas of the worksheet. For an example of the “1,000 ppm S Precision” worksheet with data completely entered, please see the worksheet entitled, “EX – 1,000 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 “1,000 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 “1,000 ppm S Precision” worksheet, the standard deviation of the data set (located in cell B15), and an indication as to whether the 1,000 ppm sulfur precision criterion are met will be determined by the worksheet. The indication of “PASSED” or “FAILED” is in cell B16 in the worksheet, after the question, “Is 1,000 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 37), the equations that analyze the data will be automatically adjusted. If difficulties are encountered in doing this, please call for help.

Part II - Accuracy demonstration instructions for ECA marine fuel subject to the 1,000 ppm sulfur standard.

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 900-1,000 ppm sulfur shall not differ from the accepted reference value of that standard by more than 15.075 ppm sulfur.⁴

- A. Locate the worksheet entitled, "1,000 ppm S Accuracy". Enter data in the light shaded green areas of the worksheet. For an example of the "1,000 ppm S Accuracy" worksheet with data completely entered, please see the worksheet entitled, "EX - 1,000 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 the 900-1,000 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 the entered data. Once all data are entered into the "1,000 ppm S Accuracy" worksheet, this worksheet will calculate the arithmetic average for the 900-1,000 ppm sulfur data set (located in cell B20). 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 B24 for the 900-1,000 ppm accuracy demonstration). This worksheet will indicate whether the 1,000 ppm sulfur accuracy criteria are met for the candidate test method by saying "PASSED" or "FAILED" in the cell after the questions, "Is 900-1,000 ppm Sulfur Accuracy Criterion Met?" (located in cell B19). This accuracy criteria must be met for the test method to be considered to have met the 1,000 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 900 to 1,000 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 - Additional information for voluntary consensus-based standards body Test Method Self Qualification

⁴⁴ Individual test results shall be compensated for any known chemical interferences.

⁵⁵ 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.

- 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 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. §1090.1375. 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 §1090.1365 and §1090.1375 for five years.

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