

Guidance and Form Instructions to Parties Submitting Diesel Sulfur Accuracy and Precision Information for Approval of Test Methods by Facility

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

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Introduction: On May, 2004, EPA promulgated new requirements for non-road diesel fuel. One of the provisions of this final rule, explained in Subpart I of 40 CFR Part 80, requires that each test facility must meet specified accuracy and precision criteria with their chosen test method for sulfur in on-road or non-road diesel fuel additives and/or diesel fuel. Evidence of having met these requirements must be submitted to and approved by the Agency 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 diesel sulfur accuracy and precision criteria, or may be required to meet both of these accuracy and precision criteria. For diesel fuel at the 500 ppm sulfur level, if your test facility has chosen to use the designated test method, ASTM D 2622-03, or any of the following alternative test methods, ASTM D 4294-03, ASTM D 5453-03a, or ASTM D 3120-03a with correlation to the designated test method, it is not necessary for your test facility to qualify these methods for precision or accuracy.

40 CFR § 80.520(a)(1) explains the 15 ppm sulfur standard for motor vehicle diesel fuel and fuel additives. 40CFR § 80.510(b)(1)(i) and § 80.510(c) explains the 15 ppm sulfur standard for Non-road Locomotive Marine(NRLM) diesel fuel and fuel additives. 40 CFR § 80.520(c) explains the 500 ppm sulfur standard for motor vehicle diesel fuel. 40 CFR § 80.510(a) explains the 500 ppm sulfur standard for NRLM diesel fuel. 40 CFR § 80.584 explains the actual accuracy and precision criteria for the applicable standards above. 40 CFR § 80.585 explains both the requirement that the criteria must be met on a facility-specific basis and a description of the process by which methods may be approved. The following guidance applies to any party applying to EPA for such approval of their test method at their testing facility. This guidance deals only with the approval of 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 of Subpart I of 40 CFR Part 80. These instructions are intended to help a test facility qualify a method for the measurement of sulfur in diesel motor fuel or diesel motor fuel additives, as well as, to help facilitate the timely review and approval of each test facility's method.

Applicable Dates: These requirements for method qualification under § 80.585 become effective on December 27, 2004.

Note: Please see Part I for instructions on use of a spreadsheet template provided by the Agency for determining compliance with the accuracy and precision criteria of § 80.584. We encourage parties to use this spreadsheet to structure their development of the information and data needed for a method qualification application and to submit it to the Agency in order to expedite the review and approval process. Please see Part II for submission instructions.

Part I - Instructions for use of spreadsheet for evaluating method precision and accuracy.

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

Precision Criteria, § 80.584(a)(1) - a standard deviation less than 0.72 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 diesel fuel with a sulfur content in the range of 5-15 ppm. The 20 results must be a series of tests with a sequential record of analyses and no omissions.¹

- A. In the workbook entitled "Sulfur Test Method Template", 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 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 B18), 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 located in cell B17 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

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 motor vehicle diesel fuel and diesel fuel additives subject to the 15 ppm sulfur 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 0.54 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 0.54 ppm sulfur.⁴

- 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".

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 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 "15 ppm S precision" worksheet, for your convenience, this information is automatically referenced from the "15 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 "15 ppm S Accuracy" worksheet, this worksheet will calculate the arithmetic average for both the 1-10 ppm sulfur data set (located in cell B20) and the 10-20 ppm sulfur data set (located in cell I20). 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 1-10 ppm accuracy demonstration & located in cell I24 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 1-10 ppm Sulfur Accuracy Criterion Met?" (located in cell B19) and "Is 10-20 ppm Sulfur Accuracy Criterion Met?" (located in cell I19). Both of these accuracy criteria must be met in order 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

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

⁴ Individual test results shall be compensated for any known chemical interferences.

on the concentration of the gravimetric standard in cell E23 and cell L23 (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

III. Precision demonstration for motor vehicle diesel fuel subject to the 500 ppm sulfur standard.

Precision Criterion, § 80.584(a)(2) - a standard deviation less than 9.68 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 diesel fuel (SHCADF) with a sulfur content in the range of 200-500 ppm. The 20 results must be a series of tests with a sequential record of analyses and no omissions.⁶

- A. Locate the worksheet entitled, "500 ppm S Precision". Follow the instructions for Section I above, but enter the data for the 500ppm precision demonstration rather than that for the 15ppm 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.

IV. Accuracy demonstration for motor vehicle diesel fuel subject to the 500 ppm sulfur 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 100-200 ppm sulfur shall not differ from the accepted reference value of that standard by more than 7.26 ppm sulfur.⁷
 2. The arithmetic average of a continuous series of at least 10 tests performed on a CAGSS in the range of 400-500 ppm sulfur shall not differ from the accepted reference value of that standard by more than 7.26 ppm sulfur.⁸
- A. Locate the worksheet entitled, "500 ppm S Accuracy". Follow the instructions for section II above, but enter the data for the 500ppm accuracy demonstration rather than that for the 15ppm 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".

⁷ Individual test results shall be compensated for any known chemical interferences.

⁸ Individual test results shall be compensated for any known chemical interferences.

PART II - Instructions for submission of Accuracy and Precision data to Agency for Approval

- I. **For each test facility that wishes to have a test method approved, the following information must be submitted to the Agency for approval:**
- A. 40 CFR 80.585(a). For methods that are approved by a voluntary consensus-based organization standards body (VCSB), such as the American Society for Testing and Materials (ASTM) or International Standards Organization (ISO), each individual test facility must submit the information for accuracy and precision criteria specified under § 80.584 (Please use instructions in Part I of this guidance). The approval of the test method is limited to the single test facility that performed the testing for accuracy and precision. The individual facility must submit the accuracy and precision results for each method, including information on the date and time of each test measurement to demonstrate precision, following procedures established by the Administrator.
- B. 40 CFR 80.585(b). For methods that are **not** approved by a VCSB, for such a method to be approved, the following information must be submitted to the Administrator by each test facility for each test method that it wishes to have approved:
1. Full test method documentation, including a description of the technology and/or instrumentation that makes the method functional.
 2. Information demonstrating that the test method meets the applicable accuracy and precision criteria of § 80.584, including information on the date and time of each test measurement to demonstrate precision (Please use instructions in Part I of this guidance).
 3. If requested by EPA, test results from use of the test method to analyze samples of commercially available fuel provided by EPA.
 4. Any additional information requested by the Administrator and considered necessary for deciding whether to approve the test method.
- C. 40 CFR 80.585(c). Sample Retention. Samples used for precision and accuracy determination must be retained for 90 days.
- D. Confidential Business Information Claim. You may claim the information you submit as confidential business information (CBI) by clearly marking your submission. Please be sure to indicate your CBI claim where asked on the spreadsheet and clearly indicate that your submission contains information claimed as CBI in your cover letter. If no such written claim is made, then the information you submit may be made available to the public by EPA without further notice. EPA's regulations regarding CBI are at 40 CFR Part 2.
- E. Please send one paper hard copy of your applicable information described above that includes a cover memo with your signature, along with a 3.5 inch floppy disk or compact disk containing the accuracy and precision data in the spreadsheet described above by overnight mail or courier service to the address below.

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- F. 40 CFR 585(d). EPA Approval.
1. Within 90 days of receipt of all materials required to be submitted under 40 CFR 80.585(a) or (b), the Administrator shall determine whether the test method is approved.
 2. If the Administrator denies approval of the test method, within 90 days of receipt of all materials required to be submitted under 40 CFR 80.585(a) or (b), the Administrator will notify the applicant of the reasons for not approving the method. If the Administrator does not notify the applicant within 90 days of receipt of the application, that the test method is not approved, then the test method shall be deemed approved.
 3. If the Administrator finds that an individual test facility has provided false or inaccurate information under 40 CFR 80.585, upon notice from the Administrator the approval shall be void ab initio.
 4. Non-VCSB test method/test facility approval shall be valid for five years from the date of approval by the Administrator and shall not be extended. If the method is later approved by a VCSB, the approval shall remain valid so long as the conditions of 40 CFR 80.585(a) are met.
- G. 40 CFR 80.585(e). Quality Assurance procedures for sulfur measurement instrumentation. A test method shall not be considered a test using an approved test method unless the following quality control procedures are performed separately for each instrument to make measurements:
1. Follow all mandatory provisions of ASTM D 6299-02 and construct control charts from the mandatory quality control testing prescribed in paragraph 7.1 of the reference method, following guidelines under A 1.5.1 for individual observation charts and A 1.5.2 for moving range charts.⁹
 2. Follow paragraph 7.3.1 of ASTM D 6299-02 to check standards using a reference material at least monthly or following any major change to the laboratory equipment or test procedure. Any deviation from the accepted reference value of a check standard greater than 1.44 ppm (for diesel fuel subject to the 15 ppm sulfur standard) or 19.36 ppm (for diesel fuel subject to the 500 ppm sulfur standard) must be investigated.
 3. Samples of test batches must be retained for 30 days or the period equal to the interval between quality control sample tests, whichever is longer.

⁹ The Director of the Federal Register approved the incorporation by reference of ASTM D 6299-02, Standard Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance, as prescribed in 5 U.S.C. 552(a) and 1 CFR Part 51. Anyone may purchase copies of this standard from the American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19428. Anyone may inspect copies at the U.S. EPA, Air and Radiation Docket and Information Center, 1301 Constitution Ave., N.W., Room B102, EPA West Building, Washington, D.C. 20460 or at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030 or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.

4. Upon discovery of any quality control testing violation of paragraph A 1.5.1.3 or A 1.5.2.1 of ASTM D 6299-02, or any check standard deviation greater than 1.44 ppm (for diesel fuel subject to the 15 ppm sulfur standard) or 19.36 ppm (for diesel fuel subject to the 500 ppm sulfur standard), conduct an investigation into the cause or such violation or deviation and, after restoring method performance to statistical control, retest retained samples from batches originally tested since the last satisfactory quality control material or check standard testing occasion.
- H. 40 CFR 80.586. 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 80.584 and 80.585 for five years.

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