Instructions for Using EPA Spreadsheet to Submit Solvent Yellow 124 Test Method Precision and Accuracy Information

Transportation and Regional Programs Division
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While we have made every effort to ensure the accuracy of the discussion in these instructions, the obligations of the regulated community are determined by statutes, regulations, or other legally binding requirements. In the event of a conflict between the discussion in this document and any statute or regulation, this document would not be controlling.

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 these instructions and its applicability to a particular situation. EPA may adopt approaches on a case-by-case basis that differ from those described in these instructions.

Mention of trade names or commercial products does not constitute endorsement or recommendation for their use.

The sampling, sample preparation, and testing methods qualified for use in accordance with the requirements of section 40 CFR § 80.582 may involve the use of hazardous materials, operations and equipment. These instructions do not address the associated safety problems which may exist. It is the responsibility of the user of the procedures specified in these instructions to establish appropriate safety and health practices prior to their use. It is also the responsibility of the user to dispose of any byproducts which might result from conducting these procedures in a manner consistent with applicable safety and health requirements.

This is a living document and may be revised periodically without public notice. EPA welcomes public input on this document at any time.

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 its chosen test method for the fuel marker solvent yellow 124 in heating oil and non-road locomotive and marine diesel fuels. 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. The following instructions are for use by those parties that need to qualify a facility/test method for measuring the concentration of this marker in heating oil or non-road locomotive and marine diesel fuel pursuant to 40 CFR 80.510.[1]

EPA has provided a spreadsheet template for recording and evaluating the test data required for establishing the test method's precision and accuracy. Use of the template will, we hope, help an applicant to structure and submit the needed information. It will also facilitate EPA's timely review and approval of test methods. Part I below explains the use of the template, while Part II covers submission of the resulting structured information to EPA.

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

I. Precision demonstration for solvent yellow 124 test method.

Precision Criterion[2] - a standard deviation less than 0.10 milligrams per liter, 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 meeting applicable industry consensus and federal regulatory specifications with fuel marker solvent yellow 124 content in the range of 0.10 to 8 milligrams per liter. The 20 results must be a series of tests with a sequential record of analyses and no omissions.[3]

A. In the workbook entitled "Fuel Marker Solvent Yellow 124 Template", locate the worksheet entitled, "SY124 Precision". Enter data in the light shaded green areas of the worksheet. For an example of the "SY124 Precision" worksheet with data completely entered, please see the worksheet entitled, "EX – SY214 Precision".

Notes:

1. Test results must be reported in milligrams per liter (mg/l) to the number of significant digits specified in the method description or, if no such

[1] In certain areas of the country, solvent yellow 124 has to be used as a fuel marker in heating oil distillate fuel indefinitely beginning June 1, 2007 and, from June 1, 2010 through May 31, 2012, in locomotive and marine distillate fuel.

[2] § 80.582(b)(1).

[3] 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.

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 sample identification number for each test result.

B. After entering the data into the light shaded green area of the "SY 124 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 "SY 124 Precision" worksheet, the standard deviation of the data set (located in cell B18), and an indication as to whether the SY124 precision criterion is met will be displayed by the worksheet. The indication of "PASSED" or "FAILED" is located in cell B17 in the worksheet, after the question, "Is SY124 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.[4]

II. Accuracy demonstration for solvent yellow 124 test method.

Accuracy Criteria -

1. The arithmetic average of a continuous series of at least 10 tests performed on a commercially available marker solvent yellow 124 standard in the range of 0.10 to 1 milligrams per liter solvent yellow 124 shall not differ from the accepted reference value of that standard by more than 0.05 milligrams per liter.[5,6]

2. The arithmetic average of a continuous series of at least 10 tests performed on a marker solvent yellow 124 standard in the range of 4 to 10 milligrams per liter solvent yellow 124 shall not differ from the accepted reference value of that standard by more than 0.05 milligrams per liter.[7,8]

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[4] Additional rows may be inserted to accommodate the extra data points. If these rows are added in the middle of the data set (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.

[5] § 80.582(b)(2)(i).

[6] As explained in § 80.582(b)(2)(iii), individual test results shall be compensated for any known chemical interferences.

[7] § 80.582(b)(2)(ii).

[8] As explained in § 80.582(b)(2)(iii), individual test results shall be compensated for any known chemical interferences.

A. Locate the worksheet entitled, “SY124 Accuracy”. Enter data in the light shaded green areas of the worksheet. For an example of the “SY124 Accuracy” worksheet with data completely entered, please see the worksheet entitled, “EX - SY124 Accuracy”.

Notes:

1. Test results must be reported in milligrams per liter (mg/l) 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 sample identification number for each test result.
4. In the appropriate rows, enter the “Name of Standard Vendor”, “Standard Lot Identification Number”, and “Accepted Reference Value of the Standard (mg/L)” in milligrams per liter for both the 0.1-1 mg/L and 4-10 mg/L marker solvent yellow 124 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 “SY124 precision” worksheet, for your convenience, this information is automatically referenced from the “SY124 Precision” worksheet.

B. After entering the data into the light shaded green area of the worksheet as described above:

1. Go to the “File” menu at the top of the screen and select “Save” to save all of the entered data.
2. Once all data are entered into the “SY124 Accuracy” worksheet, the worksheet will calculate the arithmetic average for both the 0.1-1 mg/L marker solvent yellow 124 data set (located in cell B20) and the 4-10 mg/L marker solvent yellow 124 (located in cell I20). It will also calculate the difference between the arithmetic average of the data set and the accepted reference value of each standard (located in cell B24 for the 0.1-1 mg/L concentration and in cell I24 for the 4-10 mg/L concentration).
3. The worksheet will indicate whether the marker solvent yellow 124 accuracy criteria are met for the candidate test method by saying “**PASSED**” or “**FAILED**” in the cell after the questions, “Is 0.1-1 mg/L marker solvent yellow 124 Accuracy Criterion Met?” (located in cell B19) and “Is 4-10 mg/L marker solvent yellow 124 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 demonstrated sufficient accuracy.
4. 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 standards in cell E23 and cell L23. Note: If the applicant wishes to include more than the 10 minimum tests, please report the additional data by inserting rows into the spreadsheet.[9]

[9] 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.

PART II - Instructions for submission of Accuracy and Precision Information 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:**

A. For methods that are approved by a voluntary consensus-based organization standards body (VCSB)[10], 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 § 80.582(b) (Please use instructions in Part I of this document). The approval of the test method is limited to the single test facility that performed the testing for accuracy and precision.[11] 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. For methods that are **not** approved by a VCSB[12], 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 and all procedures that are part of the method.[13]
2. Information demonstrating that the test method meets the applicable accuracy and precision criteria of § 80.582(b), including information on the date and time of each test measurement to demonstrate precision (Please use instructions in Part I of this guidance).[14]
3. If requested by EPA, test results from use of the test method to analyze samples of commercially available distillate fuel containing the fuel marker solvent yellow 124 provided by EPA.[15]
4. Any additional information requested by the Administrator and considered necessary for deciding whether to approve the test method.

C. Sample Retention[16]. Samples used for precision and accuracy determination must be retained for 90 days.

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[10] 40 CFR 80.582(c)(1).

[11] Where a facility has more than one functionally identical instrument implementing a single method, only one of the instruments must be qualified (but statistical quality control must be applied to each instrument, as indicated below).

[12] 40 CFR 80.585(c)(2).

[13] 40 CFR 80.585(c)(2)(i).

[14] 40 CFR 80.585(c)(2)(ii).

[15] 40 CFR 80.585(c)(2)(iv).

[16] 40 CFR 80.585(c)(2)(iii).

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.

Please send one paper hard copy of the information described above and a cover letter 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. EPA Approval.[17]

1. Within 90 days of receipt of all materials required to be submitted under 40 CFR 80.582(c)(1) or (2), 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 80.582(c)(1) or (2), the Administrator will notify the applicant of the reasons for not approving the method. If the Administrator does not notify the applicant of disapproval or incomplete information during that time span then the test method shall be deemed approved.

3. If the Administrator finds that and individual test facility has provided false or inaccurate information under 40 CFR 80.582(c), upon notice from the Administrator the approval shall be void ab initio.

4. The qualification of any test method under the requirements of 40 CFR 80.582 shall be valid for the duration of the period during which the fuel marker provisions remain applicable under the Motor Vehicle Diesel Fuel regulations at 40 CFR 80 Subpart I.

[17] 40 CFR 80.582(c)(3).

G. Quality Assurance procedures for solvent yellow 124 measurement instrumentation.[18] A test will not be considered to have been made with an approved test method unless the following quality control procedures are performed separately for each instrument used 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.[19]

2. Follow paragraph 7.3.1 of ASTM D 6299-02 to test 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 0.10 milligrams per liter 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.

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 0.10 milligrams per liter, conduct an investigation into the cause of 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 80.582(d)(5). 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 80.582 for five years.

[18] 40 CFR 80.582(d).

[19] 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.

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