

§ 80.582 What are the sampling and testing methods for the fuel marker?

For heating oil and NRLM diesel fuel subject to the fuel marker requirement in § 80.510(d), (e), or (f), the identification of the presence and concentration of the fuel marker in diesel fuel may be determined using the test procedures qualified in accordance with the requirements in this section.

(a) *Sampling and testing for methods for the fuel marker.* The sampling, sample preparation, and testing methods qualified for use in accordance with the requirements of this section may involve the use of hazardous materials, operations and equipment. This section does not address the associated safety problems which may exist. It is the responsibility of the user of the procedures specified in this section 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.

(b) *What are the precision and accuracy criteria for qualification of fuel marker test methods?*—(1) *Precision.* A standard deviation of less than 0.10 milligrams per liter is required, computed from the results of a minimum of 20 repeat tests made over 20 days on samples taken from a homogeneous commercially available diesel fuel which meets the applicable industry consensus and federal regulatory specifications and which contains the fuel marker at a concentration in the range of 0.10 to 8 milligrams per liter. In order to qualify, the 20 results must be a series of tests on the same material and there must be a sequential record of the analysis with no omissions. A laboratory facility 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) *Accuracy.* (i) 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 shall not differ from

the ARV of that standard by more than 0.05 milligrams per liter.

(ii) 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 4 to 10 milligrams per liter shall not differ from the ARV of that standard by more than 0.05 milligrams per liter.

(iii) In applying the tests of paragraphs (b)(2)(i) and (ii) of this section, individual test results shall be compensated for any known chemical interferences.

(c) *What process must a test facility follow in order to qualify a test method for determining the fuel marker content of distillate fuels and how will EPA qualify or decline to qualify a test method?*—(1) *Qualification of test methods approved by voluntary consensus-based standards bodies.* Any standard test method developed by a Voluntary Consensus-Based Standards Body, such as the American Society for Testing and Materials (ASTM) or International Standards Organization (ISO), shall be considered a qualified test method for determining the fuel marker content of distillate fuel provided that it meets the precision and accuracy criteria under paragraph (b) of this section. The qualification of a 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 used to demonstrate precision, following procedures established by the Administrator.

(2) *Qualification of test methods that have not been approved by a voluntary consensus-based standards body.* A test method that has not been approved by a voluntary consensus-based standards body may be qualified upon approval by the Administrator. The following information must be submitted in the application for approval by each test facility, for each test method that it wishes to have approved:

(i) Full test method documentation, including a description of the technology and/or instrumentation that makes the method functional.

(ii) Information demonstrating that the test method meets the accuracy and precision criteria under paragraph (b) of this section, including information on the date and time of each test measurement used to demonstrate precision.

(iii) Samples used for precision and accuracy determination must be retained for 90 days.

(iv) If requested by the Administrator, test results utilizing the method and performed on a sample of commercially available distillate fuel which meets the applicable industry consensus and federal regulatory specifications and which contains the fuel marker.

(v) Any additional information requested by the Administrator and necessary to render a decision as to qualification of the test method.

(vi) The qualification of a test method is limited to the single test facility that performed the testing for accuracy and precision and any other required testing.

(3)(i) Within 90 days of receipt of all materials required to be submitted under paragraph (c)(1) or (c)(2) of this section, the Administrator shall determine whether to qualify the test method under this section. The Administrator shall qualify the test method if all materials required under this section are received and the test method meets the accuracy and precision criteria of paragraph (b) of this section.

(ii) If the Administrator denies approval of the test method, within 90 days of receipt of all materials required to be submitted under this section, 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.

(iii) If the Administrator finds that an individual test facility has provided false or inaccurate information under this section, upon notice from the Administrator, the qualification shall be void *ab initio*.

(iv) The qualification of any test method under this paragraph (c) shall be valid for the duration of the period

during which the fuel marker requirements remain applicable under this subpart.

(d) *Quality control procedures for fuel marker measurement instrumentation.* A test shall not be considered a test using a qualified 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. 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., NW., Room B102, EPA West Building, Washington, DC 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.

(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 0.10 milligrams per liter must be investigated.

(3) Samples of tested 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,

Environmental Protection Agency

§ 80.583

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.

(5) Retain results of quality control testing and retesting of retained samples under paragraph (d)(3) of this section for five years.

[69 FR 39185, June 29, 2004]

§ 80.583 What alternative sampling and testing requirements apply to importers who transport motor vehicle diesel fuel, NRLM diesel fuel, or ECA marine fuel by truck or rail car?

Importers who import diesel fuel subject to the 15 ppm sulfur standard under § 80.510(b) or (c) or 80.520(a) into the United States by truck or by rail car may comply with the following requirements instead of the requirements to sample and test each batch of fuel designated as subject to the 15 ppm sulfur standard under § 80.581 otherwise applicable to importers:

(a) *Terminal testing.* For purposes of determining compliance with the 15 ppm sulfur standard, the importer may use test results for sulfur content testing conducted by the foreign truck-loading or rail car-loading terminal operator for diesel fuel contained in the storage tank from which trucks or rail cars used to transport diesel fuel designated as subject to the 15 ppm sulfur content standard into the United States are loaded, provided the following conditions are met:

(1) The sampling and testing shall be performed after each receipt of diesel fuel into the storage tank, or immediately before each transfer of diesel fuel to the importer's truck or rail car.

(2) The sampling and testing shall be performed according to § 80.580.

(3) At the time of each transfer of diesel fuel to the importer's truck or rail car for import to the U.S., the importer must obtain a copy of the terminal test result that indicates the sulfur content of the truck or rail car

load, or truck or rail car compartment load, as applicable.

(b) *Quality assurance program.* The importer must conduct a quality assurance program, as specified in this paragraph (b), for each truck or rail car loading terminal.

(1) Quality assurance samples must be obtained from the truck-loading or rail car loading terminal and tested by the importer, or by an independent laboratory, and the terminal operator must not know in advance when samples are to be collected.

(2) The sampling and testing must be performed using the methods specified in § 80.580.

(3) The frequency of the quality assurance sampling and testing must be at least one sample for each 50 of an importer's trucks or rail cars that are loaded at a terminal, or one sample per month, whichever is more frequent.

(c) *Party required to conduct quality assurance testing.* The quality assurance program under paragraph (b) of this section shall be conducted by the importer. In the alternative, this testing may be conducted by an independent laboratory that meets the criteria under § 80.65(f)(2)(iii), provided the importer receives copies of all results of tests conducted no later than 21 days after the sample was taken.

(d) *Alternative batch designations.* For purposes of maintaining batch records under §§ 80.592, 80.600, and 80.602, designation of batches under § 80.598, and reporting under §§ 80.593, 80.601, and 80.604:

(1) In lieu of treating each portion of a tank truck compartment delivered to a different facility as a different batch, a truck importer may treat each compartment as a batch, if all the fuel in the compartment is delivered only to retail outlets, wholesale purchaser-consumers or other end users. Where different compartments contain homogeneous product of identical designations, the total volume of those compartments may be treated as a single batch, if the entire volume is delivered only to retail outlets, wholesale purchaser-consumers or other ultimate consumers.

(2) Each portion of a rail car (or rail cars) delivery of a different designation