

TABLE IV-D-1.—DESIGNATED AND ALTERNATIVE SULFUR TEST METHODS ALLOWED UNDER THE HIGHWAY DIESEL PROGRAM

Sulfur test method	500 ppm	15 ppm
ASTM D 2622-98 as modified, Standard Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry.	Designated	Alternative.
ASTM D 3120-96, Standard Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry.	Alternative.
ASTM D 4294, Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy-Dispersive X-ray Fluorescence Spectrometry.	Alternative.	
ASTM D 5453-00, Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence.	Alternative	Alternative.
ASTM D 6428-99, Test Method for Total Sulfur in Liquid Aromatic Hydrocarbons and Their Derivatives by Oxidative Combustion and Electrochemical Detection.	Alternative	Designated.

Under the performance-based approach, a given test method would be approved for use in a specific laboratory by meeting certain precision and accuracy criteria specified in the regulations. The method would be approved for use by that laboratory as long as appropriate quality control procedures were followed. Properly selected precision and accuracy values potentially would allow multiple methods and multiple commercially available instruments to be approved, thus providing greater flexibility in method and instrument selection while also encouraging the development and use of better methods and instrumentation in the future. Under this approach, there would be no designated sulfur test method as specified under previous regulations.

Since any test method that meets the specified performance criteria may qualify, this type of approach does not conflict with the "National Technology Transfer and Advancement Act of 1995" (NTTAA), section 12(d) of Public Law 104-113, and the Office of Management and Budget (OMB) Circular A-119. Both of these documents are designed to encourage the adoption of standards developed by "voluntary consensus bodies" and to reduce reliance on government-unique standards where such consensus standards would suffice. Under the performance criteria approach proposed today, methods developed by consensus bodies as well as methods not yet approved by a consensus body would qualify for approval provided they met the specified performance criteria as well as the recordkeeping and reporting requirements for quality control purposes.

i. How Can a Given Method Be Approved?

Under the proposed performance criteria approach, a given test method would be approved for use under today's program by meeting certain precision and accuracy criteria. Approval would apply on a laboratory/facility-specific basis. If a company chose to employ more than one laboratory for fuel sulfur testing purposes, then each laboratory would have to separately seek approval for each method it intends to use. Likewise, if a laboratory chose to use more than one sulfur test method, then each method would have to be approved separately. Separate approval would not be necessary for individual operators or laboratory instruments within a given laboratory facility.

The specific precision and accuracy criteria that we are proposing were derived from existing sulfur test methods that are either required or allowed under the highway diesel fuel sulfur program. The first criterion, precision, refers to the consistency of a set of measurements and is used to determine how closely analytical results can be duplicated based on repeat measurements of the same material under prescribed conditions. To demonstrate the precision of a given sulfur test method under the performance-based approach, a laboratory facility would perform 20 repeat tests over 20 days on samples taken from a homogeneous supply of a commercially available diesel fuel. We request comment on an alternative number of days over which these 20 repeat tests should be conducted. Using the test results³²⁸ of ASTM D 3120 for

³²⁸ Sulfur Repeatability of Diesel by Method at 15 ppm, ASTM Report on Low Level Sulfur Determination in Gasoline and Diesel Interlaboratory Study—A Status Report, June 2002.

diesel fuel subject to the 15 ppm sulfur standard, the precision would have to be less than 0.72 ppm.³²⁹ Similarly, using the test results of ASTM D 2622 for diesel fuel subject to the 500 ppm sulfur standard, the precision would have to be less than 9.68 ppm.

The second criterion, accuracy, refers to the closeness of agreement between a measured or calculated value and the actual or specified value. To demonstrate the accuracy of a given test method under the performance-based approach, a laboratory facility would be required to perform 10 repeat tests on a standard sample, the mean of which for diesel fuel subject to the 15 ppm sulfur standard could not deviate from the Accepted Reference Value (ARV) of the standard by more than 0.54 ppm and for diesel fuel subject to the 500 ppm sulfur standard could not deviate from the ARV of the standard by more than 7.26 ppm.³³⁰ These tests would be performed using commercially available gravimetric sulfur standards. Ten tests would be required using each of two different sulfur standards—one in the range of 1–10 ppm sulfur and the other in the range of 10–20 ppm sulfur for 15 ppm fuel and one in the range of 100–200 ppm sulfur and the other in the

³²⁹ 0.72 ppm is equal to 1.5 times the standard deviation of ASTM D 3120, where the standard deviation is equal to the repeatability of ASTM D 3120 (1.33) divided by 2.77. 9.68 ppm is equal to 1.5 times the standard deviation of ASTM D 2622, where the standard deviation is equal to the repeatability of ASTM D 2622 (26.81) divided by 2.77. Since the conditions of the precision qualification test admit more sources of variability than the conditions under which ASTM repeatability is determined (longer time span, different operators, environmental conditions, etc.) the repeatability standard deviation derived from the round robin was multiplied by what we believe to be a reasonable adjustment factor, 1.5, to compensate for the difference in conditions.

³³⁰ 0.54 and 7.26 are equal to 0.75 times the precision values of 0.72 for 15 ppm sulfur diesel and 9.68 for 500 ppm sulfur diesel, respectively.