



Designation: D4294 – 21

Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry¹

This standard is issued under the fixed designation D4294; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of total sulfur in petroleum and petroleum products that are single-phase and either liquid at ambient conditions, liquefiable with moderate heat, or soluble in hydrocarbon solvents. These materials can include diesel fuel, jet fuel, kerosine, other distillate oil, naphtha, residual oil, lubricating base oil, hydraulic oil, crude oil, unleaded gasoline, gasoline-ethanol blends, biodiesel (see [Note 2](#)), and similar petroleum products.

NOTE 1—Oxygenated fuels with ethanol or methanol contents exceeding the limits given in [Table 1](#) can be dealt with using this test method, but the precision and bias statements do not apply (see [Appendix X3](#)).

NOTE 2—For samples with high oxygen contents ($>3\%$ by weight) sample dilution as described in [1.3](#) or matrix matching must be performed to assure accurate results.

1.2 Interlaboratory studies on precision revealed the scope to be 17 mg/kg to 4.6 % by mass. An estimate of this test method's pooled limit of quantitation (PLOQ) is 16.0 mg/kg as calculated by the procedures in Practice [D6259](#). However, because instrumentation covered by this test method can vary in sensitivity, the applicability of the test method at sulfur concentrations below approximately 20 mg/kg must be determined on an individual basis. An estimate of the limit of detection is three times the reproducibility standard deviation, and an estimate of the limit of quantitation² is ten times the reproducibility standard deviation.

1.3 Samples containing more than 4.6 % by mass sulfur can be diluted to bring the sulfur concentration of the diluted material within the scope of this test method. Samples that are diluted can have higher errors than indicated in Section [17](#) than non-diluted samples.

1.4 Volatile samples (such as high vapor pressure gasolines or light hydrocarbons) may not meet the stated precision because of selective loss of light materials during the analysis.

1.5 A fundamental assumption in this test method is that the standard and sample matrices are well matched, or that the matrix differences are accounted for (see [6.2](#)). Matrix mismatch can be caused by C/H ratio differences between samples and standards (see Section [6](#)) or by the presence of other heteroatoms.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:³

[D4057](#) Practice for Manual Sampling of Petroleum and Petroleum Products

[D4175](#) Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

[D4177](#) Practice for Automatic Sampling of Petroleum and Petroleum Products

[D6259](#) Practice for Determination of a Pooled Limit of Quantitation for a Test Method

[D6299](#) Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

[D7343](#) Practice for Optimization, Sample Handling,

¹ This test method is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.03](#) on Elemental Analysis.

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² *Analytical Chemistry*, Vol 55, 1983, pp. 2210-2218.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard