



Designation: D2622 – 26

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

This standard is issued under the fixed designation D2622; 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, kerosene, other distillate oil, naphtha, residual oil, lubricating base oil, hydraulic oil, crude oil, unleaded gasoline, gasoline-ethanol blends, and biodiesel.

1.2 The range of this test method is between the PLOQ value (calculated by procedures consistent with Practice [D6259](#)) of 3 mg/kg total sulfur and the highest level sample in the round robin, 4.6 % by weight total sulfur.

NOTE 1—Instrumentation covered by this test method can vary in sensitivity. The applicability of the test method at sulfur concentrations below 3 mg/kg may be determined on an individual basis for WDXRF instruments capable of measuring lower levels, but precision in this test method does not apply.

1.2.1 The values of the limit of quantitation (LOQ) and method precision for a specific laboratory's instrument depends on instrument source power (low or high power), sample type, and the practices established by the laboratory to perform the method.

1.3 Samples containing more than 4.6 % by mass sulfur should 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 [15](#) 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 [13.2](#)). Matrix mis-

match can be caused by C/H ratio differences between samples and standards or by the presence of other interfering heteroatoms or species (see [Table 1](#)).

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:²

- [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](#)
- [D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry](#)
- [D4927 Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy](#)
- [D6259 Practice for Determination of a Pooled Limit of Quantitation for a Test Method](#)

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 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. 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

TABLE 1 Concentrations of Interfering Species

Species	Mass % Tolerated
Phosphorus	0.3
Zinc	0.6
Barium	0.8
Lead	0.9
Calcium	1
Chlorine	3
Oxygen	2.8
FAME (see Note 16)	25
Ethanol (see Note 16)	8.6
Methanol (see Note 16)	6

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

[D7343 Practice for Optimization, Sample Handling, Calibration, and Validation of X-ray Fluorescence Spectrometry Methods for Elemental Analysis of Petroleum Products and Lubricants](#)

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology [D4175](#).

4. Summary of Test Method

4.1 The sample is placed in the X-ray beam, and the peak intensity of the sulfur K-L_{2,3} (K α) line at 0.5373 nm is measured. The background intensity, measured at a recommended wavelength of 0.5190 nm (0.5437 nm for a Rh target tube) is subtracted from the peak intensity. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the concentration of sulfur in milligrams per kilogram (mg/kg) or mass percent (see Section [13](#)).

5. Significance and Use

5.1 This test method provides rapid and precise measurement of total sulfur in petroleum and petroleum products with a minimum of sample preparation. A typical analysis time is 1 min to 2 min per sample.

5.2 The quality of many petroleum products is related to the amount of sulfur present. Knowledge of sulfur concentration is necessary for processing purposes. There are also regulations promulgated in federal, state, and local agencies that restrict the amount of sulfur present in some fuels.

5.3 This test method provides a means of determining whether the sulfur content of petroleum or a petroleum product meets specification or regulatory limits.

5.4 When this test method is applied to petroleum materials with matrices significantly different from the white oil calibration materials specified in this test method, the cautions and recommendations in Section [6](#) should be observed when interpreting results.

NOTE 2—The equipment specified for Test Method D2622 tends to be more expensive than that required for alternative test methods, such as

Test Method [D4294](#). Consult the Index to ASTM Standards for alternative test methods.

6. Interferences

6.1 When the elemental composition (excluding sulfur) of samples differs significantly from the standards, errors in the sulfur determination can result. For example, differences in the carbon-hydrogen ratio of sample and calibration standards introduce errors in the determination. Some other interferences and action levels are listed in [Table 1](#). If a sample is known from its history or another analysis to contain any of the species listed in [Table 1](#) at or above the values listed there, that sample should be diluted with blank sulfur solvent to reduce the interferent concentration below the value to mitigate the effect of this interference.

NOTE 3—The concentrations of the first seven substances in [Table 1](#) were determined by the calculation of the sum of the mass absorption coefficients times mass fraction of each element present. This calculation was made for dilutions of representative samples containing approximately 3 % of interfering substances and 0.5 % sulfur. Refer to [Note 16](#) for additional information regarding FAME, ethanol, and methanol.

6.2 Fuels containing large quantities of FAME, ethanol, or methanol (see [Table 1](#)) have a high oxygen content leading to significant absorption of sulfur K α radiation and low sulfur results. Such fuels can, however, be analyzed using this test method provided either that correction factors are applied to the results (when calibrating with white oils) or that the calibration standards are prepared to match the matrix of the sample. See [12.5](#).

6.3 In general, petroleum materials with compositions that vary from white oils as specified in [10.1](#) can be analyzed with standards made from base materials that are of the same or similar composition. Thus a gasoline may be simulated by mixing *isooctane* and toluene in a ratio that approximates the expected aromatic content of the samples to be analyzed. Standards made from this simulated gasoline can produce results that are more accurate than results obtained using white oil standards.

6.4 Test Method [D4927](#) is the recommended test method for the determination of sulfur >100 mg/kg in lubricating oils and lubricating oil additives because method [D4927](#) implements inter-element correction factors. Test Method D2622 is not suitable because it does not encompass the measurement of the additional elements present in lubricating oils and their additives making matrix correction impossible.

7. Apparatus

7.1 *Wavelength Dispersive X-Ray Fluorescence Spectrometer (WDXRF)*, equipped for X-ray detection in the wavelength range from about 0.52 nm to about 0.55 nm (specifically at 0.537 nm). For optimum sensitivity to sulfur, the instrument should be equipped with the following items:

7.1.1 *Optical Path*, vendor specified, helium preferred, ambient air or nitrogen are inferior.

7.1.2 *Pulse-Height Analyzer*, or other means of energy discrimination.

7.1.3 *Detector*, for the detection of X-rays with wavelengths in the range of interest (from about 0.52 nm to about 0.55 nm).