



Standard Test Method for Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry¹

This standard is issued under the fixed designation D7220; 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.

1. Scope

1.1 This test method specifies an energy-dispersive X-ray fluorescence (EDXRF) method for the determination of total sulfur in automotive, No. 2 heating, and jet fuels with a concentration range of 3 to 942 mg/kg.

1.1.1 The pooled limit of quantitation of this test method as obtained by statistical analysis of inter laboratory test results is 3 mg/kg sulfur.

1.1.2 This test method is applicable to gasoline, oxygen enriched gasoline (RFG), diesel, diesel/biodiesel blends containing up to twenty volume percent biodiesel, kerosene, jet fuel, jet fuel/biodiesel blends containing up to five volume percent biodiesel and No. 2 home heating oil.

1.2 A fundamental assumption in this test method is that the standard and sample matrix is well matched. Matrix mismatch can be caused by C/H ratio differences between samples and standards or by the presence of other heteroatoms.

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

1.3.1 The preferred concentration units are mg/kg sulfur.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Jan. 15, 2012. Published March 2012. Originally approved in 2006. Last previous edition approved in 2006 as D7220-06. DOI: 10.1520/D7220-12.

² 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.

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Terminology

3.1 Definitions:

3.1.1 *monochromatic X-radiation, n*—an incident X-ray beam on a sample having a selected photon energy with a narrow energy bandwidth of $\pm 5\%$ relative to the selected energy.

3.1.1.1 *Discussion*—Monochromatic X-ray radiation in EDXRF instrumentation can be obtained by using Bragg optics (at an angle of $\theta = 45 \pm 5^\circ$, in the low energy range). Bragg optics (monochromators) create very intense mono-energetic radiation. A combination of a selected X-ray tube (typically a Pd or Ag anode) with a highly ordered pyrolytic graphite (HOPG) Bragg optic can be used to create monochromatic radiation of the characteristic radiation of the anode material of the X-ray tube. The use of such radiation for sample excitation results in increased sensitivity for the determination of sulfur in petroleum products.

3.2 Abbreviations:

3.2.1 *DBS*—actual mass of Di-*n*-butyl sulfide, g

3.2.2 *Kcps*—kilo-counts per second.

3.2.3 *EDXRF*—Energy dispersive X-ray spectrometry

3.2.4 *PTFE*—Polytetrafluorethylene

3.2.5 *SDBS*—mass % of sulfur in Di-*n*-butyl sulfide, typically 21.91%

3.2.6 *SStd*—mg/kg sulfur in the calibration standard

3.2.7 *SStock*—mg/kg of sulfur in the stock standard

3.2.8 *STK*—actual mass of stock standard, g