~~Designation:D7220–06~~ Designation: D7220 – 12

Standard Test Method for ~~Sulfur in Automotive Fuels by Polarization X-ray Fluorescence Spectrometry~~Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry[1]

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

~~1.1This test method specifies an energy-dispersive X-ray fluorescence (EDXRF) method for the determination of total sulfur in automotive fuels with a concentration range of 6 mg/kg to 50 mg/kg.~~

~~1.1.1The pooled limit of quantitation of this test method as obtained by statistical analysis of inter laboratory test results is 6mg/kg sulfur.~~

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.

~~1.3The values stated in SI units are to be regarded as the standard. The preferred concentration units are mg/kg sulfur.~~

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

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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.*

[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 Feb. 15, 2006. Published March 2006. DOI: 10.1520/D7220-06.~~

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. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

~~2.2 *ISO Standard:*~~

~~ISO 4259Determination and application of precision data in relation to methods of test~~

3. Terminology

3.1 *Definitions:*

3.1.1 ~~*polarization X-ray fluorescence*~~monochromatic X-radiation, *n*~~—typically a polarization EDXRF instrument is used. In difference to direct excitation EDXRF spectrometry, polarization X-ray fluorescence uses polarized radiation for excitation. Combined with Cartesian geometry (of excitation, sample and detection system) this results in a significant improvement of the detection limit compared to direct excitation EDXRF.~~ —an incident X-ray beam on a sample having a selected photon energy with a narrow energy bandwidth of ±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 è = 45 ± 5°, 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

~~3.2.3~~

3.2.4 *PTFE*—Polytetrafluorethylene

~~3.2.4~~

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

~~3.2.5~~

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

~~3.2.6~~

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

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

4. Summary of Test Method

4.1 The sample is placed in the ~~polarized~~monochromatic X-ray beam, and the peak area of the sulfur Kα line at 2.307 keV is measured. The background spectrum, measured with a sulfur free white oil or other matrix matching blank sample (see 8.4) is adapted to the measured spectrum using adjustment regions following the instrument manufacturer's instructions and then subtracted from the measured spectrum. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the concentration of sulfur in mg/kg. (**Warning—**Exposure to excessive quantities of X-radiation is injurious to health. The operator needs to take appropriate actions to avoid exposing any part of their body, not only to primary X-rays, but also to secondary or scattered radiation that might be present. The X-ray spectrometer should be operated in accordance with the regulations governing the use of ionizing radiation.)

5. Significance and Use

5.1 This test method provides measurement of total sulfur in automotive, No. 2 heating, and jet fuels with a minimum of sample preparation. A typical analysis time is ~~200~~180 to ~~300~~360 s per sample.

5.2 The quality of automotive, No. 2 heating, and jet fuel can be 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 fuel.

5.3 If this test method is applied to petroleum materials with matrices significantly different from the calibration materials specified in this test method, the cautions and recommendations in Section 6 should be observed when interpreting the results.

[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.