



Standard Test Method for Low Sulfur in Automotive Fuels by Energy-Dispersive X-ray Fluorescence Spectrometry Using a Low-Background Proportional Counter¹

This standard is issued under the fixed designation D7212; 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 the total sulfur content of automotive fuels with a concentration range from 7 mg/kg to 50 mg/kg.

1.1.1 The pooled limit of quantitation of this test method as obtained by statistical analysis of interlaboratory test results is 7 mg/kg sulfur.

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

1.3 *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

D4045 Test Method for Sulfur in Petroleum Products by Hydrogenolysis and Rateometric Colorimetry

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

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 June 15, 2013. Published August 2013. Originally approved in 2006. Last previous edition approved in 2007 as D7212 – 07. DOI: 10.1520/D7212-13.

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.

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

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 *Definitions of Terms Specific to This Standard:*

3.1.1 *low background proportional counter, n*—an X-ray proportional counter that can suppress the noise generated when incident X rays are absorbed near the wall with resulting incomplete charge collection.

3.1.1.1 *Discussion*—An electrode shield close to the wall detects incomplete charge collection and associated electronic detection circuitry rejects those events. In comparison to conventional proportional counters, this gives lower spectral background and a lower limit of detection.

4. Summary of Test Method

4.1 The sample is placed in the beam emitted from an X-ray source with titanium target and primary filtration so that excitation is by essentially monochromatic radiation of 4.51 keV and virtually no background at 2.3 keV. A low background proportional counter measures the intensity of the fluorescent sulfur K series intensity and argon K series intensity (from residual air) and the accumulated counts are compared with counts from previously prepared calibration standards to obtain the sulfur concentration in mg/kg. If chlorine is expected to be present in some samples then other regions of the spectrum must be measured to provide compensation for spectral overlap. One group of calibration standards is required to span the concentration range from 0 to 150 mg/kg sulfur.

NOTE 1—Operation of analyzers using X-ray tubes is to be conducted in accordance with the manufacturer's safety instructions and federal, state, and local regulations governing the use of ionizing radiation.

4.2 Practice **D7343** should be consulted regarding standard operating protocols in XRF analysis.

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