Designation: D4951 – 14

Standard Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation D4951; 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 quantitative determination of barium, boron, calcium, copper, magnesium, molybdenum, phosphorus, sulfur, and zinc in unused lubricating oils and additive packages.

1.2 The precision statements are valid for dilutions in which the mass % sample in solvent is held constant in the range of 1 to 5 mass % oil.

1.3 The precision tables define the concentration ranges covered in the interlaboratory study. However, both lower and higher concentrations can be determined by this test method. The low concentration limits are dependent on the sensitivity of the ICP instrument and the dilution factor. The high concentration limits are determined by the product of the maximum concentration defined by the linear calibration curve and the sample dilution factor.

1.4 Sulfur can be determined if the instrument can operate at a wavelength of 180 nm.

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

1.6 *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]

D1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4628 Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption 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

D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

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

3. Summary of Test Method

3.1 A sample portion is weighed and diluted by mass with mixed xylenes or other solvent. An internal standard, which is required, is either weighed separately into the test solution or is previously combined with the dilution solvent. Calibration standards are prepared similarly. The solutions are introduced to the ICP instrument by free aspiration or an optional peristaltic pump. By comparing emission intensities of elements in the test specimen with emission intensities measured with the calibration standards and by applying the appropriate internal standard correction, the concentrations of elements in the sample are calculable.

4. Significance and Use

4.1 This test method usually requires several minutes per sample. This test method covers eight elements and thus provides more elemental composition data than Test Method D4628 or Test Methods D4927. In addition, this test method provides more accurate results than Test Method D5185, which is intended for used lubricating oils and base oils.

[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.

Current edition approved Oct. 1, 2014. Published October 2014. Originally approved in 1989. Last previous edition approved in 2009 as D4951 – 09. DOI: 10.1520/D4951-14.

[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.

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

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