



Standard Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry¹

This standard is issued under the fixed designation D 4951; 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 Department of Defense.

1. Scope

1.1 This test method covers the quantitative determination of barium, boron, calcium, copper, magnesium, 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 the standard. The values given in parentheses are for information only.

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:

D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards³

D 4628 Test Method for Analysis of Barium, Calcium,

Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry³

D 4927 Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy⁴

D 5185 Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements by Inductively Coupled Plasma Atomic Emission Spectrometry⁴

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁵

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 usually requires several minutes per sample. This test method covers eight elements and thus provides more elemental composition data than Test Method D 4628 or Test Method D 4927. In addition, this test method provides more accurate results than Test Method D 5185, which is intended for used lubricating oils and base oils.

4.2 Additive packages are blends of individual additives, which can act as detergents, antioxidants, antiwear agents, etc. Many additives contain one or more elements covered by this test method. Additive package specifications are based, in part, on elemental composition. Lubricating oils are typically blends

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 05.03.

⁵ Annual Book of ASTM Standards, Vol 05.04.