



Designation: D6443 – 24

Standard Test Method for Determination of Calcium, Chlorine, Copper, Magnesium, Phosphorus, Sulfur, and Zinc in Unused Lubricating Oils and Additives by Wavelength Dispersive X-ray Fluorescence Spectrometry (Mathematical Correction Procedure)¹

This standard is issued under the fixed designation D6443; 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 covers the determination of calcium, chlorine, copper, magnesium, phosphorus, sulfur, and zinc in unused lubricating oils, additives, and additive packages by wavelength dispersive X-ray fluorescence spectrometry. Matrix effects are handled with mathematical corrections.

1.2 For each element, the upper limit of the concentration range covered by this test method is defined by the highest concentration listed in [Table 1](#). Samples containing higher concentrations can be analyzed following dilution.

1.3 For each element, the lower limit of the concentration range covered by this test method can be estimated by the limit of detection (LOD)² (see also 40 CFR 136 Appendix B) or limit of quantification (LOQ),² both of which can be estimated from S_r , the repeatability standard deviation. LOD and LOQ values, determined from results obtained in the interlaboratory study on precision, are listed in [Table 2](#).

1.3.1 LOD and LOQ are not intrinsic constants of this test method. LOD and LOQ depend upon the precision attainable by a laboratory when using this test method.

1.4 This test method uses regression software to determine calibration parameters, which can include influence coefficients (that is, interelement effect coefficients) (Guide [E1361](#)), herein referenced as alphas. Alphas can also be determined from theory using relevant software.

1.5 Setup of this test method is intended for persons trained in the practice of X-ray spectrometry. Following setup, this test method can be used routinely.

1.6 The values stated in either SI units or angstrom ($\text{\AA}$) units are to be regarded separately as standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:³

- [D1552](#) Test Method for Sulfur in Petroleum Products by High Temperature Combustion and Infrared (IR) Detection or Thermal Conductivity Detection (TCD)
- [D4057](#) Practice for Manual Sampling of Petroleum and Petroleum Products
- [D4175](#) Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- [D4177](#) Practice for Automatic 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
- [D4951](#) Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry

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.

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² *Analytical Chemistry*, Vol 55, pp. 2210–2218.

³ 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