



Designation: D4628 – 23

Standard Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation D4628; 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 is applicable for the determination of mass percent barium from 0.005 % to 1.0 %, calcium and magnesium from 0.002 % to 0.3 %, and zinc from 0.002 % to 0.2 % in lubricating oils.

1.2 Higher concentrations can be determined by appropriate dilution. Lower concentrations of metals such as barium, calcium, magnesium, and zinc at about 10 ppm level can also be determined by this test method. Use of this test method for the determination at these lower concentrations should be by agreement between the buyer and the seller.

1.3 Lubricating oils that contain viscosity index improvers may give low results when calibrations are performed using standards that do not contain viscosity index improvers.

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

1.5 *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.* Specific warning statements are given in 4.1, 7.3, and 9.1.

1.6 *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.*

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. Referenced Documents

2.1 *ASTM Standards*:²

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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

3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

4. Summary of Test Method

4.1 A sample is weighed and base oil is added to 0.25 g ± 0.01 g total mass. Fifty millilitres of a kerosene solution, containing potassium as an ionization suppressant, are added, and the sample and oil are dissolved. (**Warning**—Hazardous. Potentially toxic and explosive.) Standards are similarly prepared, always adding oil if necessary to yield a total mass of 0.25 g. These solutions are burned in the flame of an atomic absorption spectrophotometer. An acetylene/nitrous oxide flame is used. (**Warning**—Combustible. Vapor harmful.)

5. Significance and Use

5.1 Some oils are formulated with metal-containing additives that act as detergents, antioxidants, antiwear agents, etc. Some of these additives contain one or more of these metals: barium, calcium, zinc, and magnesium. This test method provides a means of determining the concentration of these metals that gives an indication of the additive content in these oils.

5.2 Several additive metals and their compounds are added to the lubricating oils to give beneficial performance. (See Table 1.)

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