



Standard Test Method for Determination of Metals in Lubricating Greases by Inductively Coupled Plasma Atomic Emission Spectrometry¹

This standard is issued under the fixed designation D7303; 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 a number of metals such as aluminum, antimony, barium, calcium, iron, lithium, magnesium, molybdenum, phosphorus, silicon, sodium, sulfur, and zinc in unused lubricating greases by inductively coupled plasma atomic emission spectrometry (ICP-AES) technique.

1.1.1 The range of applicability for this test method, based on the interlaboratory study conducted in 2005,² is aluminum (10–600), antimony (10–2300), barium (50–800), calcium (20–50 000), iron (10–360), lithium (300–3200), magnesium (30–10 000), molybdenum (50–22 000), phosphorus (50–2000), silicon (10–15 000), sodium (30–1500), sulfur (1600–28 000), and zinc (300–2200), all in mg/kg. Lower levels of elements may be determined by using larger sample weights, and higher levels of elements may be determined by using smaller amounts of sample or by using a larger dilution factor after sample dissolution. However, the test precision in such cases has not been determined, and may be different than the ones given in [Table 1](#).

1.1.2 It may also be possible to determine additional metals such as bismuth, boron, cadmium, chromium, copper, lead, manganese, potassium, titanium, etc. by this technique. However, not enough data is available to specify the precision for these latter determinations. These metals may originate into greases through contamination or as additive elements.

1.1.3 During sample preparation, the grease samples are decomposed with a variety of acid mixture(s). It is beyond the scope of this test method to specify appropriate acid mixtures for all possible combination of metals present in the sample. But if the ash dissolution results in any visible insoluble material, the test method may not be applicable for the type of grease being analyzed, assuming the insoluble material contains some of the analytes of interest.

1.2 Elements present at concentrations above the upper limit of the calibration curves can be determined with additional appropriate dilutions of dissolved samples and with no degradation of precision.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.* Specific warning statements are given in [Sections 8](#) and [10](#).

2. Referenced Documents

2.1 ASTM Standards:³

- [D1193](#) Specification for Reagent Water
- [D3340](#) Test Method for Lithium and Sodium in Lubricating Greases by Flame Photometer
- [D4057](#) Practice for Manual Sampling of Petroleum and Petroleum Products
- [D4951](#) Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
- [D5185](#) Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in 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
- [D6792](#) Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories
- [D7260](#) Practice for Optimization, Calibration, and Validation of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Elemental Analysis of 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.

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² Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1608.

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