Designation: D7303 – 23

Standard Test Method for Determination of Metals in Lubricating Greases by Inductively Coupled Plasma Atomic Emission Spectrometry[1]

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 to 600), antimony (10 to 2300), barium (50 to 800), calcium (20 to 50 000), iron (10 to 360), lithium (300 to 3200), magnesium (30 to 10 000), molybdenum (50 to 22 000), phosphorus (50 to 2000), silicon (10 to 15 000), sodium (30 to 1500), sulfur (1600 to 28 000), and zinc (300 to 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 3.

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 development of the technique behind this test method is documented by Fox.[3]

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

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 Sections 8 and 10.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D1193 Specification for Reagent Water

D3340 Test Method for Lithium and Sodium in Lubricating Greases by Flame Photometer (Withdrawn 2013)[5]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

D4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma

[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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1608. Contact ASTM Customer Service at service@astm.org.

[3] Fox, B. S.,“Elemental Analysis of Lubricating Grease by Inductively Coupled Plasm Atomic Emission Spectrometry (ICP-AES),” *J. ASTM International*, Vol 2, No. 8, 2005, pp. 12966.

[4] 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.

[5] The last approved version of this historical standard is referenced on www.astm.org.

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

Atomic Emission Spectrometry

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

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, 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

3. Terminology

3.1 *Definitions:*

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

3.1.2 Refer to terminology identified in Test Method D5185 for spectroscopy terms used in this standard.

4. Summary of Test Method

4.1 A weighed portion of the grease sample is weighed and subjected to alternate means of sample dissolution which may include sulfated ashing in a muffle furnace or by closed vessel microwave digestion in acid. Ultimately these diluted acid solutions are analyzed using ICP-AES. Aqueous calibration standards are used. 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 those measured with the calibration standards, the concentrations of elements in the test specimen can be calculated.

4.2 Additional information on using inductively coupled plasma-atomic emission spectrometry can be found in Practice D7260.

5. Significance and Use

5.1 Lubricating greases are used in almost all bearings used in any machinery. Lubricating grease is composed of ~90 % additized oil and soap or other thickening agent. There are over a dozen metallic elements present in greases, either blended as additives for performance enhancements or as thickeners, or in used greases present as contaminants and wear metals. Determining their concentrations can be an important aspect of grease manufacture. The metal content can also indicate the amount of thickeners in the grease. Additionally, a reliable analysis technique can also assist in the process of trouble shooting problems with new and used grease in the field.

5.2 Although widely used in other sectors of the oil industry for metal analysis, ICP-AES based Test Methods D4951 or D5185 cannot be used for analyzing greases because of their insolubility in organic solvents used in these test methods. Hence, grease samples need to be brought into aqueous solution by acid decomposition before ICP-AES measurements.

5.3 Test Method D3340 has been used to determine lithium and sodium content of lubricating greases using flame photometry. This technique is no longer widely used. This new test method provides a test method for multi-element analysis of grease samples. This is the first D02 standard available for simultaneous multi-element analysis of lubricating greases.

6. Interferences

6.1 *Spectral*—Spectral interferences can usually be avoided by judicious choice of analytical wavelengths. There are no known spectral interferences between elements covered by this test method when using the spectral lines listed in Table 1. However, if spectral interferences exist because of other interfering elements or selection of other spectral lines, correct for the interferences using the technique described in Test Method D5185.

6.1.1 Follow the instrument manufacturer's operating guide to develop and apply correction factors to compensate for the interferences.

6.2 *Chemical*—If the grease sample contains refractory additives such as silicon or molybdenum, it is possible that some of these elements may remain undissolved in the residue, and may result in lower recoveries.

6.2.1 If HF is used for dissolution of grease residues, elements such as silicon may be lost as SiF_6. Residual HF can also attack the ICP sample introduction system. HF can be passivated by adding dilute boric acid to the acid solution.

6.2.2 If the dry ashing in sample preparation step is used, elements such as sulfur will be volatilized during combustion.

7. Apparatus

7.1 *Analytical Balance,* capable of weighing to 0.001 g or 0.0001 g, capacity of 150 g.

7.2 *Inductively Coupled Plasma Atomic Emission Spectrometer*—Either a sequential or simultaneous spectrometer is suitable, if equipped with a quartz ICP torch and RF generator to form and sustain the plasma. Suggested wavelengths for the determination of elements in dissolved grease solutions are given in Table 1.

TABLE 1 Suggested Wavelengths[A,B] for Elements Determined in Grease Samples

Element	Wavelength, nm
Aluminum	167.038, 308.22, 396.15, 309.27
Antimony	206.83, 217.58, 231.15
Barium	223.53, 233.527, 455.40, 493.41
Calcium	315.88, 317.93, 364.4, 396.85, 422.67
Iron	238.20, 259.94
Lithium	670.78, 610.36, 460.29
Magnesium	279.08, 279.55, 280.278, 285.21
Molybdenum	135.387, 202.03, 281.62
Phosphorus	177.51, 178.29, 213.62, 214.91, 253.40
Silicon	288.16, 251.618
Sodium	589.595
Sulfur	182.04, 180.73, 182.63
Zinc	202.55, 206.20, 213.86, 334.58, 481.05

[A] These wavelengths are only suggested and do not represent all possible choices.

[B] Wavelengths for boron, phosphorus, and sulfur below 190 nm require that a vacuum or inert gas purge optical path be used.