Designation: D5185 – 13$^{\varepsilon 1}$

Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[1]

This standard is issued under the fixed designation D5185; 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.

ε[1] NOTE—Table 4 was editorially corrected in April 2014.

INTRODUCTION

Costs associated with maintenance due to engine and machine wear can be significant. Therefore, diagnostic methods for determining the condition of engines and other machinery can be important. This test method is intended to quantify, for the purpose of equipment monitoring, the concentration of metals in used lubricating oils. Although the precision statement was determined by analyzing a variety of used oils this test method can, in principle, be used for the analysis of unused oils to provide more complete elemental composition data than Test Methods D4628, D4927, or D4951.

1. Scope*

1.1 This test method covers the determination of additive elements, wear metals, and contaminants in used and unused lubricating oils and base oils by inductively coupled plasma atomic emission spectrometry (ICP-AES). The specific elements are listed in Table 1.

1.2 This test method covers the determination of selected elements, listed in Table 1, in re-refined and virgin base oils.

1.3 For analysis of any element using wavelengths below 190 nm, a vacuum or inert-gas optical path is required. The determination of sodium and potassium is not possible on some instruments having a limited spectral range.

1.4 This test method uses oil-soluble metals for calibration and does not purport to quantitatively determine insoluble particulates. Analytical results are particle size dependent, and low results are obtained for particles larger than a few micrometers.[2]

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

1.6 For elements other than calcium, sulfur, and zinc, the low limits listed in Table 2 and Table 3 were estimated to be ten times the repeatability standard deviation. For calcium, sulfur, and zinc, the low limits represent the lowest concentrations tested in the interlaboratory study.

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

1.8 *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 6.1, 8.2, and 8.4.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C1109 Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy

D1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

[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] Eisentraut, K. J., Newman, R. W., Saba, C. S., Kauffman, R. E., and Rhine, W. E., *Analytical Chemistry*, Vol 56, 1984.

[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**

TABLE 1 Elements Determined and Suggested Wavelengths[A]

Element	Wavelength, nm
Aluminum	308.22, 396.15, 309.27
Barium	233.53, 455.40, 493.41
Boron	249.77
Calcium	315.89, 317.93, 364.44, 422.67
Chromium	205.55, 267.72
Copper	324.75
Iron	259.94, 238.20
Lead	220.35
Magnesium	279.08, 279.55, 285.21
Manganese	257.61, 293.31, 293.93
Molybdenum	202.03, 281.62
Nickel	231.60, 227.02, 221.65
Phosphorus	177.51, 178.29, 213.62, 214.91, 253.40
Potassium	766.49
Sodium	589.59
Silicon	288.16, 251.61
Silver	328.07
Sulfur	180.73, 182.04, 182.62
Tin	189.99, 242.95
Titanium	337.28, 350.50, 334.94
Vanadium	292.40, 309.31, 310.23, 311.07
Zinc	202.55, 206.20, 213.86, 334.58, 481.05

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

TABLE 2 Repeatability

Element	Range, mg/kg	Repeatability, μg/g[A]
Aluminum	6–40	$0.71\ X^{0.41}$
Barium	0.5–4	$0.24\ X^{0.66}$
Boron	4–30	$0.26\ X$
Calcium	40–9000	$0.0020\ X^{1.4}$
Chromium	1–40	$0.17\ X^{0.75}$
Copper	2–160	$0.12\ X^{0.91}$
Iron	2–140	$0.13\ X^{0.80}$
Lead	10–160	$1.6\ X^{0.32}$
Magnesium	5–1700	$0.16\ X^{0.86}$
Manganese	5–700	$0.010\ X^{1.3}$
Molybdenum	5–200	$0.29\ X^{0.70}$
Nickel	5–40	$0.52\ X^{0.49}$
Phosphorus	10–1000	$1.3\ X^{0.58}$
Potassium	40–1200	$3.8\ X^{0.33}$
Silicon	8–50	$1.3\ X^{0.26}$
Silver	0.5–50	$0.15\ X^{0.83}$
Sodium	7–70	$0.49\ X^{0.66}$
Sulfur	900–6000	$0.49\ X^{0.81}$
Tin	10–40	$2.4\ X^{0.17}$
Titanium	5–40	$0.54\ X^{0.37}$
Vanadium	1–50	$0.061\ X$
Zinc	60–1600	$0.15\ X^{0.88}$

[A] where: X = mean concentration, μg/g.

TABLE 3 Reproducibility

Element	Range, mg/kg	Reproducibility, μg/g[A]
Aluminum	6–40	$3.8\ X^{0.26}$
Barium	0.5–4	$0.59\ X^{0.92}$
Boron	4–30	$13\ X^{0.01}$
Calcium	40–9000	$0.015\ X^{1.3}$
Chromium	1–40	$0.81\ X^{0.61}$
Copper	2–160	$0.24\ X$
Iron	2–140	$0.52\ X^{0.80}$
Lead	10–160	$3.0\ X^{0.36}$
Magnesium	5–1700	$0.72\ X^{0.77}$
Manganese	5–700	$0.13\ X^{1.2}$
Molybdenum	5–200	$0.64\ X^{0.71}$
Nickel	5–40	$1.5\ X^{0.50}$
Phosphorus	10–1000	$4.3\ X^{0.50}$
Potassium	40–1200	$6.6\ X^{0.29}$
Silicon	8–50	$2.9\ X^{0.39}$
Silver	0.5–50	$0.35\ X$
Sodium	7–70	$1.1\ X^{0.71}$
Sulfur	900–6000	$1.2\ X^{0.75}$
Tin	10–40	$2.1\ X^{0.62}$
Titanium	5–40	$2.5\ X^{0.47}$
Vanadium	1–50	$0.28\ X^{1.1}$
Zinc	60–1600	$0.083\ X^{1.1}$

[A] where: X = mean concentration, μg/g.

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

D7260 Practice for Optimization, Calibration, and Validation of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Elemental Analysis of Petroleum Products and Lubricants

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *emission spectroscopy*—refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *additive element*—a constituent of a chemical compound that improves the performance of a lubricating oil.

3.2.2 *analyte*—an element whose concentration is being determined.

3.2.3 *Babington-type nebulizer*—a device that generates an aerosol by flowing a liquid over a surface that contains an orifice from which gas flows at a high velocity.

3.2.4 *calibration*—the process by which the relationship between signal intensity and elemental concentration is determined for a specific element analysis.

3.2.5 *calibration curve*—the plot of signal intensity versus elemental concentration using data obtained by making measurements with standards.

3.2.6 *contaminant*—a foreign substance, generally undesirable, introduced into a lubricating oil.

3.2.7 *detection limit*—the concentration of an analyte that results in a signal intensity that is some multiple (typically two) times the standard deviation of the background intensity at the measurement wavelength.

3.2.8 *inductively-coupled plasma (ICP)*—a high-temperature discharge generated by flowing an ionizable gas through a magnetic field induced by a load coil that surrounds the tubes carrying the gas.

3.2.9 *linear response range*—the elemental concentration range over which the calibration curve is a straight line, within the precision of the test method.