Designation: D7151 – 15 (Reapproved 2023)

Standard Test Method for Determination of Elements in Insulating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[1]

This standard is issued under the fixed designation D7151; 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 describes the determination of metals and contaminants in insulating oils by inductively coupled plasma atomic emission spectrometry (ICP-AES). The specific elements are listed in Table 1. This test method is similar to Test Method D5185, but differs in methodology, which results in the greater sensitivity required for insulating oil applications.

1.2 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 several micrometers.[2]

1.3 This test method determines the dissolved metals (which can originate from overheating or arcing, or both) and a portion of the particulate metals (which generally originate from a wear mechanism). While this ICP method detects nearly all particles less than several micrometers, the response of larger particles decreases with increasing particle size because larger particles are less likely to make it through the nebulizer and into the sample excitation zone.

1.4 This test method includes an option for filtering the oil sample for those users who wish to separately determine dissolved metals and particulate metals (and hence, total metals).

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

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

D923 Practices for Sampling Electrical Insulating Liquids

D1744 Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent (Withdrawn 2016)[4]

D2864 Terminology Relating to Electrical Insulating Liquids and Gases

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions for general terms are found in Terminology D2864.

3.2 *Definitions of Terms Specific to This Standard:*

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

Current edition approved Dec. 1, 2023. Published January 2024. Originally approved in 2005. Last previous edition approved in 2015 as D7151 – 15. DOI: 10.1520/D7151-15R23.

[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.

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

TABLE 1 Elements Determined and Suggested Wavelengths[A]

Element	Wavelength, nm
Aluminum	308.22, 396.15, 309.27
Cadmium	226.50. 214.44
Cobalt	228.62 231.16
Copper	324.75
Iron	259.94, 238.20
Lead	220.35
Nickel	231.60, 227.02, 221.65
Scandium	361.38
Silicon	288.16, 251.61
Silver	328.07
Sodium	589.59
Tin	189.99, 242.95
Tungsten	239.71
Yttrium	371.03
Zinc	206.20, 202.55, 213.86, 334.58, 481.05

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

3.2.1 *Babington-type nebulizer, n*—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.

3.2.2 *inductively-coupled plasma (ICP), n*—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.

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

3.2.4 *profiling, n*—a technique that determines the wavelength for which the signal intensity measured for a particular analyte is a maximum.

3.2.5 *wear metal, n*—an element introduced into the oil by wear of oil-wetted parts.

3.2.6 *dissolved metal, n*—a metallic element in the oil which will pass a 0.45 µm filter.

4. Summary of Test Method

4.1 A weighed portion of a thoroughly homogenized insulating oil is diluted by weight with kerosine or other suitable solvent. Standards are prepared in the same manner. An internal standard may be added to the solutions to compensate for variations in test specimen introduction efficiency. The solutions may be introduced to the ICP instrument by a peristaltic pump. If free aspiration is used, an internal standard shall be used. By comparing emission intensities of elements in the test specimen with emission intensities measured with the standards, the concentrations of elements in the test specimen are calculated.

5. Significance and Use

5.1 This test method covers the rapid determination of 12 elements in insulating oils, and it provides rapid screening of used oils for indications of wear. Test times approximate several minutes per test specimen, and detectability is in the 10 µg/kg through 100 µg/kg range.

5.2 This test method can be used to monitor equipment condition and help to define when corrective action is needed. It can also be used to detect contamination such as from silicone fluids (via Silicon) or from dirt (via Silicon and Aluminum).

5.3 This test method can be used to indicate the efficiency of reclaiming used insulating oil.

6. Interferences

6.1 *Spectral*—Check all spectral interferences expected from the elements listed in Table 1. Follow the ICP manufacturer's operating guide to develop and apply correction factors to compensate for the interferences. To apply interference corrections, all concentrations must be within the previously established linear response range of each element listed in Table 1.

6.1.1 Spectral interferences can usually be avoided by judicious choice of analytical wavelengths. When spectral interferences cannot be avoided, the necessary corrections shall be made using the computer software supplied by the ICP manufacturer or the empirical method given in Test Method C1109 or by Boumans.[5]

6.1.2 Interference correction factors can be negative if off-peak background correction is employed for an element. A negative correction factor can result when an interfering line is encountered at the background correction wavelength rather than at the peak wavelength.

6.2 *Viscosity Effects*—Differences in the viscosities of the test specimen solutions and standard solutions can cause differences in the uptake rates. These differences can adversely affect the accuracy of the analysis. The effects can be reduced by using a peristaltic pump to deliver solutions to the nebulizer or by the use of internal standardization, or both.

6.3 *Particulates*—The use of an internal standard will reveal when particulates cause flow problems. Use of a Babington-type high-solids nebulizer helps to minimize plugging of the nebulizer. Also, the specimen introduction system can limit the transport of particulates, and the plasma can incompletely atomize larger particulates, thereby causing low results.

6.4 *Solvent Moisture*—Excessive moisture (>30 mg/kg) in the kerosine used for dilution can cause poor recovery of some elements. This can be minimized by checking each lot of kerosine for moisture content using Test Method D1744 and by analyzing all diluted standards and test specimens within 24 h of preparation.

7. Apparatus

7.1 *Balance,* top loading, with automatic tare, capable of weighing to 0.001 g, capacity of at least 150 g. A balance with a capacity of at least 250 g is required if preparing the Internal Standard according to 10.2.

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

[5] Boumans, P. W .J. M., "Corrections for Spectral Interferences in Optical Emission Spectroscopy with Special Reference to the RF Inductively Coupled Plasma," *Spectrochimica Acta*, 1976, Vol 318, pp. 147-152.