Designation: D7111 – 16 (Reapproved 2021)

Standard Test Method for Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[1]

This standard is issued under the fixed designation D7111; 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 selected elements in middle distillate fuels by inductively coupled plasma atomic emission spectrometry (ICP-AES). The specific elements are listed in Table 1. The concentration range of this test method is approximately 0.1 mg/kg to 2.0 mg/kg. The test method may be used for concentrations outside of this range; however, the precision statements may not be applicable. Middle distillate fuels covered in this test method have all distillation fractions contained within the boiling range of 150 °C to 390 °C. This includes, but is not limited to, diesel fuels and aviation turbine fuels.

1.2 This test method is not intended to analyze insoluble particulates. However, very small particulate matter (smaller than a micrometre) will be carried into the plasma and be included in the quantitative analysis.

1.3 This test method may give a result that is higher than the true value if an analyte is present in the sample in a form which is sufficiently volatile. For example, hexamethyldisiloxane will generate a biased high result for silicon.

1.4 The values stated in SI units are to be regarded as 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.*

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:*[2]

D2880 Specification for Gas Turbine Fuel Oils

D3605 Test Method for Trace Metals in Gas Turbine Fuels by Atomic Absorption and Flame Emission Spectroscopy

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

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

2.2 *Military Standard:*

MIL-DTL-16884 Fuel, Naval Distillate[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration, n*—the determination of the values of the significant parameters by comparison with values indicated by a set of reference standards.

3.1.2 *calibration curve, n*—the graphical or mathematical representation of a relationship between the assigned (known) values of standards and the measured responses from the measurement system.

[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] 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.

[3] Available online at http://quicksearch.dla.mil or http://assistdocs.com

TABLE 1 Elements and Recommended Wavelengths

Element	Wavelengths, nm
Aluminum	308.215, 396.153
Barium	455.403, 493.408
Calcium	393.366
Chromium	267.716, 283.563
Cobalt	228.615, 236.375, 238.892
Copper	324.752
Iron	259.939
Lithium	670.784
Lead	220.353, 224.688, 283.306
Magnesium	279.553
Manganese	257.610
Molybdenum	202.030, 204.597, 281.616
Nickel	221.648, 341.476
Phosphorus	177.495, 178.287, 185.944, 214.914, 213.618
Palladium	340.458, 342.124
Platinum	214.423
Potassium	766.490
Sodium	588.995
Silicon	251.611
Silver	328.068
Strontium	407.771
Tin	283.999, 189.991
Titanium	334.940
Vanadium	310.230
Zinc	213.857

3.1.3 *calibration standard, n*—a standard having an accepted value (reference value) for use in calibrating a measurement instrument or system.

3.1.4 *detection limit, n*—a stated limiting value that designates the lowest concentration that can be determined with confidence and that is specific to the analytical procedure used.

3.1.5 *emission spectroscopy, n*—measurement of the energy spectrum emitted by or from an object under some form of energetic stimulation; for example, light or electrical discharge.

3.1.6 *inductively coupled plasma, n*—a high temperature discharge generated by passing an ionizable gas through a magnetic field induced by a radio frequency coil surrounding the tubes that carry the gas.

3.1.7 *radio frequency, n*—the range of frequencies between 3 kHz and 300 GHz.

3.1.8 *standard, n*—a physical or chemical reference used as a basis for comparison or calibration.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *detection limit, n*—the lowest concentration value for an element that can be determined by ICP analysis and that is calculated by multiplying three times the standard deviation of ten repetitive element analyses of the blank solution.

3.2.2 *internal standard, n*—a chemical standard having an accepted value (and added to the fuel test specimen and calibration standard) to determine the emission intensity ratio of an element to the internal standard.

4. Summary of Test Method

4.1 Calibration standards are prepared by mixing organometallic standard materials in kerosine. An internal standard material is added to the calibration standards and fuel samples. The calibration standards and the fuel samples are aspirated into the ICP-AES instrument. The concentrations of the elements in the fuel are calculated by comparing emission intensity ratios of the fuel and calibration standards to the internal standard.

TABLE 2 Internal Standards, Recommended Wavelengths, and Approximate Use Concentrations

Internal Standard	Wavelength, nm	Concentration, mg/kg
Scandium	361.383	1-2
Yttrium	371.029	1-5

4.2 Consult Practice D7260 regrading the optimum operation of any ICP-AES system.

5. Significance and Use

5.1 Trace elemental analysis is used to indicate the level of contamination of middle distillate fuels. Trace metals in turbine fuels can cause corrosion and deposition on turbine components at elevated temperatures. Some diesel fuels have specification limit requirements for trace metals to guard against engine deposits. Trace level copper in middle distillate aviation turbine fuel can significantly accelerate thermal instability of the fuel, leading to oxidation and production of detrimental insoluble deposits in the engine.

5.2 Gas turbine fuel oil Specification D2880 provides recommended upper limits for five trace metals (calcium, lead, sodium, potassium, and vanadium). Military specification MIL-DTL-16884 for naval distillate fuel sets requirements for maximum concentrations of the same five metals. Both specifications designate Test Method D3605, an atomic absorption/flame emission method, for the quantitative analysis of four of the metals. Test Method D3605 does not cover potassium. This test method provides an alternative to Test Method D3605, covers potassium and a number of additional elements.

5.3 There are several sources of multi-element contamination of naval distillate fuel. Sea water is pumped into the diesel fuel tanks (as ballast) to trim ships. Also, some of the oilers (fuel supply ships) have dirty tanks. Corrosion products come from unlined tanks, piping, pumps, and heat exchangers.

6. Interferences

6.1 Elemental wavelengths listed in Tables 1 and 2 have been found to be free of spectral interferences with all other elements listed in Tables 1 and 2 in the concentration range of this test method.

6.2 If a spectral interference does exist, then selecting an analytical wavelength other than those listed in Table 1 or Table 2 may be used as long as the new wavelength possesses appropriate sensitivity for the scope of the method.

6.3 Alternatively, the ICP spectrometer manufacturer's software may be used to provide corrections to interferences that cannot be avoided by wavelength selection and background correction.

6.4 An empirical method for correcting for spectral interferences is detailed in Test Method D5185, Section 6.1 (*Spectral*).