Designation: D8322 – 25

Standard Test Method for Determination of Elements in Residual Fuels and Crude Oils by Microwave Plasma Atomic Emission Spectroscopy (MP-AES)[1]

This standard is issued under the fixed designation D8322; 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 metals and other elements in residual fuel and crude oil by microwave plasma atomic emission spectroscopy (MP-AES). The specific elements within the scope of this method are V, Ni, Ca, Na, Al, Si, Zn, P, and S for residual fuel oil and Fe, V, Ni, Ca, Na, K, and S for crude oils.

1.2 Method working range:
high expected concentration limit = highest ILS sample mean
low expected concentration limit = lowest ILS sample mean if:
(1) lowest ILS sample mean − $R_{\text{lowest ILS sample mean}} > 0$; otherwise it is determined by solving for *X* using the following equation:
(2) $X - R_X$= coarsest resolution, determined by $0.5*\sigma_{\text{r lowest ILS sample mean}}$

Crude Oil:

Element	Method Working Range (expected mg/kg)
Iron	0.70 to 161.02
Vanadium	2.88 to 417.50
Nickel	0.36 to 107.66
Calcium	5.41 to 96.78
Sodium	1.18 to 97.13
Potassium	7.01 to 63.83
Sulfur	1059 to 35194

Residual Fuel Oil:

Element	Method Working Range (expected mg/kg)
Vanadium	3.88 to 370.09
Nickel	1.47 to 96.68
Calcium	4.41 to 102.01
Sodium	2.80 to 112.67
Aluminum	4.13 to 154.12
Silicon	5.99 to 237.56
Zinc	2.75 to 102.46
Sulfur	1314 to 30134

1.3 This test method uses soluble metals in organic solvents for calibration and does not purport to quantitatively determine insoluble particulates. Analytical results are particle size dependent, and particles larger than a few micrometers may cause results to appear low

1.4 Elements present at mass fractions above the upper limit of the calibration curves can be determined with additional appropriate dilutions. Elements shall be measured at the wavelengths presented in Table 1.

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

1.6 *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.*

1.7 *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]

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D1548 Test Method for Vanadium in Heavy Fuel Oil[1] (Withdrawn 1997)[3]

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

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

D5708 Test Methods for Determination of Nickel, Vanadium, and Iron in Crude Oils and Residual Fuels by

[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.

Current edition approved July 1, 2025. Published July 2025. Originally approved in 2020. Last previous edition approved in 2020 as D8322 – 20. DOI: 10.1520/D8322-25.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] 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**

TABLE 1 Element Wavelengths

NOTE 1—These wavelengths shall be used for this method. The precision for this method is based on these wavelengths.

Element	Wavelength, nm	Standards Used in Each Curve (mg/kg)
Iron	259.940	Solvent blank, 0.1, 1.0, 10, 25
Vanadium	311.070	Solvent Blank, 0.1, 1.0, 10, 25, 50
Nickel	341.476	Solvent Blank, 0.1, 1.0, 10, 25, 50
Calcium	396.847	Solvent Blank, 0.1, 1.0, 10
Calcium	616.217	Solvent Blank, 10, 25, 50, 70
Sodium	588.995	Solvent Blank, 0.1, 1.0, 10
Aluminum	396.152	Solvent Blank, 0.1, 1.0, 10, 25, 50
Silicon	288.158	Solvent Blank, 0.1, 1.0, 10, 25
Zinc	213.857	Solvent Blank, 0.1, 1.0, 10
Potassium	766.491	Solvent Blank, 0.1, 1.0, 10, 25, 50, 70
Sulfur	181.972	Solvent Blank, 40, 200, 500, 1000, 2000
Yttrium (IS)	371.029	

Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry

D6021 Test Method for Measurement of Total Hydrogen Sulfide in Residual Fuels by Multiple Headspace Extraction and Sulfur Specific Detection

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

D7372 Guide for Analysis and Interpretation of Proficiency Test Program Results

2.2 *ISO Standard:*[4]

ISO 8573–1 Compressed air – Part 1: Contaminants and purity classes

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration, n*—process by which the relationship between signal intensity and elemental mass fraction is determined for a specific element analysis. **D4175**

3.1.2 *emission spectroscopy, n*—measurement of energy spectrum emitted by or from an object under some form of energetic stimulation; for example, light, electrical discharge, and so forth. **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration curve, n*—plot of signal intensity versus elemental mass fraction using data obtained by making measurements with standards.

3.2.2 *check standards, n*—a second source QC sample prepared in o-xylene (wt/wt), such that the mass fraction is the median of the linear calibration range and 10 % of the highest mass fraction standard (see Practice D6792 for guidance in this area).

3.2.3 *continuing calibration blank (CCB), n*—a blank calibration standard that does not contain any elements of interest, analyzed immediately following each CCV; this solution is the sample blank.

3.2.4 *continuing calibration verification (CCV), n*—a mid-range calibration standard analyzed after every ten sample analyses to verify that the instrument calibration has not drifted.

3.2.5 *diluted stock standard (DSS), n*—a material prepared by gravimetric dilution of stock standard (OSS) to facilitate preparation of working standards.

3.2.6 *high solids 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.

3.2.7 *internal standard (IS), n*—a high purity compound not present in the sample which is added to each solution, calibration blank, working standards, and samples and used to calculate quantitatively the compound of interest.

3.2.8 *microwave plasma (MP), n*—a high temperature microwave plasma created by coupling the magnetic field of the microwave energy into the plasma.

3.2.9 *organometallic stock standard (OSS), n*—a material which is well characterized for the elements of interest, with assigned mass fraction values traceable to the SI.

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

3.2.11 *quality control (QC) sample, n—for use in quality assurance programs to determine and monitor the precision and stability of a measurement system*, a stable and homogeneous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system; the material is properly stored to ensure sample integrity and is available in sufficient quantity for repeated, long term testing.

3.2.12 *residual fuel oil, n*—the heavier oils that remain after the distillate fuel oils and lighter hydrocarbons are distilled away in the refinery process (viscosity at 40 °C between 5.5 mm^2/s and 24.0 mm^2/s, inclusive). **D6021**

3.2.13 *working standard (WS), n*—a material prepared from dilution of the DSS organometallic solution containing the elements of interest for the purpose of quantitative calibration.

4. Summary of Test Method

4.1 *Test Method*—For determination of elements in residual fuels and crude oils, a homogenized sample is diluted in a suitable organic solvent and introduced through the nebulizer and spray chamber into the microwave plasma atomic emission spectrometer. Quantification of elements is achieved by comparing measurements of emission intensity at the appropriate wavelength for each element to measurements of emission intensity made under the same conditions on organic standard reference solutions in the same organic solvent.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.