Designation: D8351 – 22

Standard Test Method for Determination of Trace Elements in Biodiesel and Biodiesel Blends by Microwave Plasma Atomic Emission Spectrometry (MP-AES)[1]

This standard is issued under the fixed designation D8351; 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 elements in biodiesel and biodiesel blends by microwave plasma atomic emission spectrometry (MP-AES). The specific elements within the scope of this method are calcium (Ca), magnesium (Mg), phosphorus (P), potassium (K), and sodium (Na).

1.2 This test method conforms to Practice D6300, subection 8.4.5, valid test result range, and subsection 8.4.6, working range specifications. The valid test result range and working range are recent additions to Practice D6300, and a graphical representation using example values is shown in Appendix X2, Test Method Operating Range.

1.3 Method working range:

high expected concentration limit = highest ILS sample mean
low expected concentration limit = lowest ILS sample mean if:
lowest ILS sample mean – $R_{\text{lowest ILS sample mean}} > 0$; otherwise it is determined by solving for X using the following equation:
$X - R_X$ = coarsest resolution, determined by $0.5^{*}\sigma_{r\ \text{lowest ILS sample mean}}$

Biodiesel and Biodiesel Blends	
Element	Method Working Range (expected mg/kg)
Calcium	0.24 to 15.01
Magnesium	0.12 to 11.55
Phosphorus	1.69 to 14.24
Potassium	0.49 to 13.98
Sodium	0.90 to 14.30

1.4 This test method uses organic elemental standards 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 can cause results to appear low.

1.5 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. Alternate wavelengths noted in Appendix X1 may be used in the rare case of spectral interference.

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:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards
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

[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.

2.2 *ISO Standards:*[3]
ISO 8573-1 Compressed air – Part 1: Contaminants and purity classes

3. Terminology

3.1 *Definitions:*

3.1.1 *biodiesel, n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

3.1.2 *biodiesel blend (BXX), n*—a homogeneous mixture of hydrocarbon oils and mono-alkyl esters of long chain fatty acids.

3.1.2.1 *Discussion*—In the abbreviation BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.2.2 *Discussion*—Diesel fuel, fuel oil, and non-aviation turbine oil are examples of hydrocarbon oils.

3.1.3 *biodiesel fuel, n*—synonym for biodiesel.

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

3.1.5 *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.

3.1.6 *quality control sample, n*—for use in quality assurance program to determine and monitor the precision and stability of a measurement system; a stable and homogenous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system.

3.1.6.1 *Discussion*—This material should be properly stored to ensure sample integrity, and is available in sufficient quantity for repeated long term testing.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *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 biodiesel blank.

3.2.3 *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.

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

3.2.5 *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.

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

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

3.2.8 *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.

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

4. Summary of Test Method

4.1 For determination of elements (Ca, Mg, K, Na, and P) in biodiesel and biodiesel blends, samples are introduced through the nebulizer and spray chamber into the microwave plasma atomic emission spectrometer. An internal standard (IS) is incorporated and the emission intensity of each element is divided by the emission intensity of the IS to produce an adjusted intensity for each element. Calibration standards are prepared by diluting organo-elemental standards in narrow cut kerosene solvent and measured with the IS in the same way as the samples. Quantitative results are obtained by comparing the intensity ratios measured from the samples to the intensity ratios obtained from the calibration standards.

5. Significance and Use

5.1 This test method covers the determination of five elements (Ca, Mg, K, Na, and P) in biodiesel and biodiesel blends.

5.2 The presence of metals and metalloids in engine fuels can influence the performance of engines and contribute to shortening the lifetime of the equipment. In addition, some elements act as catalyst poison contributing to increases in the amount of unwanted gases and particulate matter emitted by vehicles.

6. Interferences

6.1 *Spectral*—Check all spectral interferences expected from the elements listed in Table 1 using manufacturer's operating guide to develop and apply correction factors to compensate for the interferences. To apply interference

TABLE 1 Element Wavelengths

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

Element	Wavelength (nm)	Standards Used in each Curve (mg/kg)
Calcium	396.847	Biodiesel Blank, 0.1, 1, 5, 10
Magnesium	285.213	Biodiesel Blank, 0.1, 1, 5 10, 20
Phosphorus	213.618	Biodiesel Blank, 1, 5, 10, 20
Potassium	766.491	Biodiesel Blank, 0.1, 1, 5, 10, 20
Sodium	588.995	Biodiesel Blank, 0.1, 1, 5, 10, 20
Yttrium (IS)	371.029	20 mg/kg (dilution to 10 mg/kg during Tee assembly aspiration with the sample specimen.)

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