



Designation: D7455 – 19

Standard Practice for Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis¹

This standard is issued under the fixed designation D7455; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This practice covers different means by which petroleum product and lubricant samples may be prepared before the measurement of their elemental content using different analytical techniques.

1.2 This practice includes only the basic steps for generally encountered sample types. Anything out of the ordinary may require special procedures. See individual test methods for instructions to handle such situations.

1.3 This practice is not a substitute for a thorough understanding of the actual test method to be used, caveats the test method contains, and additional sample preparation that may be required.

1.4 The user should not expand the scope of the test methods to materials or concentrations outside the scope of the test methods being used without thoroughly understanding the implications of such deviations.

1.5 This practice may also be applicable to sample preparation of non-petroleum based bio-fuels for elemental analysis. Currently, work is ongoing in ASTM Subcommittee D02.03; as information becomes available, it will be added to this standard.

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 Recom-*

mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 *ASTM Standards:*²

[C1234 Practice for Preparation of Oils and Oily Waste Samples by High-Pressure, High-Temperature Digestion for Trace Element Determinations](#)

[D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure](#)

[D129 Test Method for Sulfur in Petroleum Products \(General High Pressure Decomposition Device Method\)](#)

[D482 Test Method for Ash from Petroleum Products](#)

[D808 Test Method for Chlorine in New and Used Petroleum Products \(High Pressure Decomposition Device Method\)](#)

[D874 Test Method for Sulfated Ash from Lubricating Oils and Additives](#)

[D1018 Test Method for Hydrogen In Petroleum Fractions](#)

[D1091 Test Methods for Phosphorus in Lubricating Oils and Additives](#)

[D1266 Test Method for Sulfur in Petroleum Products \(Lamp Method\)](#)

[D1318 Test Method for Sodium in Residual Fuel Oil \(Flame Photometric Method\)](#)

[D1548 Test Method for Vanadium in Heavy Fuel Oil¹ \(Withdrawn 1997\)³](#)

[D1552 Test Method for Sulfur in Petroleum Products by High Temperature Combustion and Infrared \(IR\) Detection or Thermal Conductivity Detection \(TCD\)](#)

[D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry](#)

[D2784 Standard Test Method for Sulfur in Liquefied Petroleum Gases \(Oxy-Hydrogen Burner or Lamp\) \(Withdrawn 2016\)³](#)

[D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry](#)

¹ This practice 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 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

- D3228** Test Method for Total Nitrogen in Lubricating Oils and Fuel Oils by Modified Kjeldahl Method
- D3230** Test Method for Salts in Crude Oil (Electrometric Method)
- D3231** Test Method for Phosphorus in Gasoline
- D3237** Test Method for Lead in Gasoline by Atomic Absorption Spectroscopy
- D3246** Test Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry
- D3340** Test Method for Lithium and Sodium in Lubricating Greases by Flame Photometer (Withdrawn 2013)³
- D3341** Test Method for Lead in Gasoline—Iodine Monochloride Method
- D3348** Test Method for Rapid Field Test for Trace Lead in Unleaded Gasoline (Colorimetric Method)
- D3605** Test Method for Trace Metals in Gas Turbine Fuels by Atomic Absorption and Flame Emission Spectroscopy
- D3701** Test Method for Hydrogen Content of Aviation Turbine Fuels by Low Resolution Nuclear Magnetic Resonance Spectrometry
- D3831** Test Method for Manganese in Gasoline By Atomic Absorption Spectroscopy
- D4045** Test Method for Sulfur in Petroleum Products by Hydrogenolysis and Rateometric Colorimetry
- D4047** Test Method for Phosphorus in Lubricating Oils and Additives by Quinoline Phosphomolybdate Method
- D4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D4177** Practice for Automatic Sampling of Petroleum and Petroleum Products
- D4294** Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
- D4628** Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry
- D4629** Test Method for Trace Nitrogen in Liquid Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
- D4808** Test Methods for Hydrogen Content of Light Distillates, Middle Distillates, Gas Oils, and Residua by Low-Resolution Nuclear Magnetic Resonance Spectroscopy
- D4927** Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy
- D4929** Test Method for Determination of Organic Chloride Content in Crude Oil
- D4951** Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
- D5056** Test Method for Trace Metals in Petroleum Coke by Atomic Absorption
- D5059** Test Methods for Lead in Gasoline by X-Ray Spectroscopy
- D5184** Test Methods for Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry, and Atomic Absorption Spectrometry
- D5185** Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D5291** Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants
- D5384** Test Methods for Chlorine in Used Petroleum Products (Field Test Kit Method)
- D5453** Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
- D5600** Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D5622** Test Methods for Determination of Total Oxygen in Gasoline and Methanol Fuels by Reductive Pyrolysis
- D5708** Test Methods for Determination of Nickel, Vanadium, and Iron in Crude Oils and Residual Fuels by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry
- D5762** Test Method for Nitrogen in Liquid Hydrocarbons, Petroleum and Petroleum Products by Boat-Inlet Chemiluminescence
- D5863** Test Methods for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption Spectrometry
- D6334** Test Method for Sulfur in Gasoline by Wavelength Dispersive X-Ray Fluorescence
- D6443** Test Method for Determination of Calcium, Chlorine, Copper, Magnesium, Phosphorus, Sulfur, and Zinc in Unused Lubricating Oils and Additives by Wavelength Dispersive X-ray Fluorescence Spectrometry (Mathematical Correction Procedure)
- D6445** Test Method for Sulfur in Gasoline by Energy-Dispersive X-ray Fluorescence Spectrometry (Withdrawn 2009)³
- D6470** Test Method for Salt in Crude Oils (Potentiometric Method)
- D6481** Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy
- D6595** Test Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry
- D6667** Test Method for Determination of Total Volatile Sulfur in Gaseous Hydrocarbons and Liquefied Petroleum Gases by Ultraviolet Fluorescence
- D6728** Test Method for Determination of Contaminants in Gas Turbine and Diesel Engine Fuel by Rotating Disc Electrode Atomic Emission Spectrometry
- D6732** Test Method for Determination of Copper in Jet Fuels by Graphite Furnace Atomic Absorption Spectrometry

- D6920** Test Method for Total Sulfur in Naphthas, Distillates, Reformulated Gasolines, Diesels, Biodiesels, and Motor Fuels by Oxidative Combustion and Electrochemical Detection (Withdrawn 2018)³
- D7039** Test Method for Sulfur in Gasoline, Diesel Fuel, Jet Fuel, Kerosine, Biodiesel, Biodiesel Blends, and Gasoline-Ethanol Blends by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry
- D7040** Test Method for Determination of Low Levels of Phosphorus in ILSAC GF 4 and Similar Grade Engine Oils by Inductively Coupled Plasma Atomic Emission Spectrometry
- D7041** Test Method for Determination of Total Sulfur in Liquid Hydrocarbons and Hydrocarbon-Oxygenate Blends by Gas Chromatography with Flame Photometric Detection
- D7111** Test Method for Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D7171** Test Method for Hydrogen Content of Middle Distillate Petroleum Products by Low-Resolution Pulsed Nuclear Magnetic Resonance Spectroscopy
- D7212** Test Method for Low Sulfur in Automotive Fuels by Energy-Dispersive X-ray Fluorescence Spectrometry Using a Low-Background Proportional Counter
- D7220** Test Method for Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence 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
- D7303** Test Method for Determination of Metals in Lubricating Greases by Inductively Coupled Plasma Atomic Emission Spectrometry
- D7318** Test Method for Existent Inorganic Sulfate in Ethanol by Potentiometric Titration
- D7319** Test Method for Determination of Existent and Potential Sulfate and Inorganic Chloride in Fuel Ethanol and Butanol by Direct Injection Suppressed Ion Chromatography
- D7328** Test Method for Determination of Existent and Potential Inorganic Sulfate and Total Inorganic Chloride in Fuel Ethanol by Ion Chromatography Using Aqueous Sample Injection
- D7343** Practice for Optimization, Sample Handling, Calibration, and Validation of X-ray Fluorescence Spectrometry Methods for Elemental Analysis of Petroleum Products and Lubricants
- D7482** Practice for Sampling, Storage, and Handling of Hydrocarbons for Mercury Analysis
- D7620** Test Method for Determination of Total Sulfur in Liquid Hydrocarbon Based Fuels by Continuous Injection, Air Oxidation and Ultraviolet Fluorescence Detection
- D7622** Test Method for Total Mercury in Crude Oil Using Combustion and Direct Cold Vapor Atomic Absorption Method with Zeeman Background Correction
- D7623** Test Method for Total Mercury in Crude Oil Using Combustion-Gold Amalgamation and Cold Vapor Atomic Absorption Method
- D7691** Test Method for Multielement Analysis of Crude Oils Using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D7740** Practice for Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants
- D7751** Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis
- D7757** Test Method for Silicon in Gasoline and Related Products by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry
- D7876** Practice for Practice for Sample Decomposition Using Microwave Heating (With or Without Prior Ashing) for Atomic Spectroscopic Elemental Determination in Petroleum Products and Lubricants
- D8110** Test Method for Elemental Analysis of Distillate Products by Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
- D8127** Test Method for Coupled Particulate and Elemental Analysis using X-ray Fluorescence (XRF) for In-Service Lubricants
- D8150** Test Method for Determination of Organic Chloride Content in Crude Oil by Distillation Followed by Detection Using Combustion Ion Chromatography

3. Summary of Practice

3.1 This practice covers alternative ways of preparing a petroleum product or lubricant sample for elemental analysis measurements. The means of preparation of samples may vary from no special steps to extensive detailed procedures dependent on the sample matrix and the measurement technique to be used.

3.2 This practice may also be applicable to non-petroleum based biofuels. Work is underway to validate the applicability to these types of materials.

4. Significance and Use

4.1 Crude oil, petroleum, petroleum products, additives, and lubricants are routinely analyzed for their elemental content such as chlorine, nitrogen, phosphorus, sulfur, and various metals using a variety of analytical techniques. Some of these test methods require little to no sample preparation; some others require only simple dilutions; while others require elaborate sample decomposition before the product is analyzed for its elemental content.

4.2 Fairly often it can be shown that the round robin results by a co-operator are all biased with respect to those from other laboratories. Presumably, the failure to follow good laboratory practices and instructions in the test methods can be a causal factor of such errors. A further consequence is an unnecessarily large reproducibility estimate or the data being dropped from the study as an outlier.

4.3 Uniform practice for sample preparation is beneficial in standardizing the procedures and obtaining consistent results across the laboratories.