



Standard Guide for Calibration Requirements for Elemental Analysis of Petroleum Products and Lubricants¹

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1. Scope*

1.1 This guide covers different ways by which the test methods used for elemental analysis of petroleum product and lubricant samples are calibrated before the sample analysis.

1.2 Uniform practice for test method calibration is beneficial in standardizing the procedures, and obtaining consistent results across different laboratories.

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

1.4 This guide is not a substitute for a thorough understanding of the actual test method to be used, caveats it contains, and additional instrument preparation that may be required.

1.5 The user should not expand the scope of the test methods to materials or concentrations outside the scope of the test methods being used.

1.6 This guide should also be applicable to sample preparation of non-petroleum based bio-fuels for elemental analysis. Work is underway on these aspects in Subcommittee D02.03. As more information becomes available, it will be added to this standard.

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

1.8 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This guide 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. Referenced Documents

2.1 ASTM Standards:²

- D129 Test Method for Sulfur in Petroleum Products (General High Pressure Decomposition Device Method)
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- 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
- D892 Test Method for Foaming Characteristics of Lubricating Oils
- 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 IR Detection
- D1839 Test Method for Amyl Nitrate in Diesel Fuels
- D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
- D2784 Test Method for Sulfur in Liquefied Petroleum Gases (Oxy-Hydrogen Burner or Lamp)
- D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry

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
- D3831** Test Method for Manganese in Gasoline By Atomic Absorption Spectroscopy
- D4045** Test Method for Sulfur in Petroleum Products by Hydrogenolysis and Rateometric Colorimetry
- D4046** Test Method for Alkyl Nitrate in Diesel Fuels by Spectrophotometry
- D4047** Test Method for Phosphorus in Lubricating Oils and Additives by Quinoline Phosphomolybdate Method
- D4294** Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
- D4307** Practice for Preparation of Liquid Blends for Use as Analytical Standards
- 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 Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
- 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 Methods 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 Petroleum and Petroleum Products by Boat-Inlet Chemiluminescence
- D5800** Test Method for Evaporation Loss of Lubricating Oils by the Noack Method
- 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
- D6792** Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories
- D6920** Test Method for Total Sulfur in Naphthas, Distillates, Reformulated Gasolines, Diesels, Biodiesels, and Motor Fuels by Oxidative Combustion and Electrochemical Detection

- 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 Light Hydrocarbons, Motor Fuels, and Oils by Online 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
- D7455** Practice for Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis
- 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
- E77** Test Method for Inspection and Verification of Thermometers
- E319** Practice for the Evaluation of Single-Pan Mechanical Balances
- E898** Test Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances
- 2.2 *NIST Standard*.⁴
- NIST Special Publication 260-136** Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements
- 2.3 *ISO Standard*.⁵
- ISO Guide 30** Terms and definitions used in connection with reference materials

3. Terminology

3.1 Definitions:

3.1.1 *calibration standard, n*—material with a certified value for a relevant property, issued by or traceable to a national organization such as NIST, and whose properties are known with sufficient accuracy to permit its use to evaluate the same property of another sample. **D6792**

3.1.2 *certified reference material, CRM, n*—reference material one or more of whose property values are certified by a technically valid procedure, accompanied by a traceable certificate or other documentation which is issued by a certifying body. **ISO Guide 30**

3.1.3 *check standard, n*—material having an assigned (known) value (reference value) used to determine the accuracy of the measurement system or instrument. This standard is not used to calibrate the measurement instrument or system. **D7171**

3.1.4 *reference material, RM, n*—material with accepted reference value(s), accompanied by an uncertainty at a stated level of confidence for desired properties, which may be used for calibration or quality control purposes in the laboratory.

3.1.4.1 *Discussion*—Sometimes these may be prepared “in-house” provided the reference values are established using standard primary procedures with known precision uncertainties. See below for further discussion. **D6792**

3.1.5 *traceability, n*—property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties. **D6792**

4. Summary of Guide

4.1 This guide covers procedures used for calibrating instruments or methods for the analysis of petroleum products or

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

⁵ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.