



ASTM INTERNATIONAL
Manual

Analysis of Biofuels - A Laboratory Resource

R.A. Kishore Nadkarni



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Analysis of Biofuels: A Laboratory Resource

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Foreword

This publication, *Analysis of Biofuels: A Laboratory Resource*, was sponsored by Committee D02 on Petroleum Products, Lubricants, and Liquid Fuels. This is Manual 77 in ASTM International's manual series.

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ASTM Standards and Other Standards Quoted in the Text

#	Analysis	ASTM Research Report # RR-D02-
ASTM D56	Flash Point by Tag Closed Cup Tester	NA
ASTM D86	Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure	1621; 1694
ASTM D92	Flash and Fire Points by Cleveland Open Cup Tester	1009
ASTM D93	Flash Point by Pensky-Martens Closed Cup Tester	1683
ASTM D95	Water in Petroleum Products and Bituminous Materials by Distillation	NA
ASTM D97	Pour Point of Petroleum Products	1499
ASTM D128	Analysis of Lubricating Grease	NA
ASTM D129	Sulfur in Petroleum Products (General High Pressure Decomposition Device Method)	1278
ASTM D130	Corrosiveness to Copper from Petroleum Products by Copper Strip Test	1703
ASTM D156	Saybolt Color of Petroleum Products (Saybolt Chromometer Method)	NA
ASTM D189	Conradson Carbon Residue of Petroleum Products	1227
ASTM D240	Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter	38
ASTM D3228	Total Nitrogen in Lubricating Oils and Fuel Oils by Modified Kjeldahl Method	NA
ASTM D381	Gum Content in Fuels by Jet Evaporation	1466
ASTM D396	Specification for Fuel Oils	
ASTM D445	Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)	1498
ASTM D473	Sediment in Crude Oils and Fuel Oils by the Extraction Method	NA
ASTM D4628	Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry	1207
ASTM D482	Ash from Petroleum Products	NA
ASTM D4927	Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy	1259
ASTM D524	Ramsbottom Carbon Residue of Petroleum Products	1228
ASTM D525	Oxidation Stability of Gasoline (Induction Period Method)	NA
ASTM D613	Cetane Number of Diesel Fuel Oil	
ASTM D664	Acid Number of Petroleum Products by Potentiometric Titration	1727
ASTM D874	Sulfated Ash from Lubricating Oils and Additives	1597
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ASTM D975	Specification for Diesel Fuel Oils	
ASTM D976	Calculated Cetane Index of Distillate Fuels	NA
ASTM D1078	Distillation Range of Volatile Organic Liquids	NA
ASTM D1160	Distillation of Petroleum Products at Reduced Pressure	1206; 1766
ASTM D1266	Sulfur in Petroleum Products (Lamp Method)	NA
ASTM D1298	Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method	1387
ASTM D1310	Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus	NA
ASTM D1319	Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption	1361
ASTM D1500	ASTM Color of Petroleum Products (ASTM Color Scale)	1234

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#	Analysis	ASTM Research Report # RR-D02-
ASTM D1541	Total Iodine Value of Drying Oils and Their Derivatives (Withdrawn 2006)	NA
ASTM D1552	Sulfur in Petroleum Products by High Temperature Combustion and IR Detection	1231
ASTM D1613	Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products	1041
ASTM D1688	Copper in Water	NA
ASTM D1796	Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)	NA
ASTM D1959	Iodine Value of Drying Oils and Fatty Acids (Withdrawn 2006)	NA
ASTM D1982	Titer of Fatty Acids	NA
ASTM D2500	Cloud Point of Petroleum Products	NA
ASTM D2622	Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry	1622
ASTM D2624	Electrical Conductivity of Aviation and Distillate Fuels	1161
ASTM D2709	Water and Sediment in Middle Distillate Fuels by Centrifuge	1308
ASTM D2887	Boiling Range Distribution of Petroleum Fractions by Gas Chromatography	1406
ASTM D2896	Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration	1237
ASTM D3120	Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry	1546; 1547
ASTM D3227	(Thiol Mercaptan) Sulfur in Gasoline, Kerosine, Aviation Turbine, and Distillate Fuels (Potentiometric Method)	NA
ASTM D3231	Phosphorus in Gasoline	NA
ASTM D3237	Lead in Gasoline by Atomic Absorption Spectroscopy	1376
ASTM D3242	Acidity in Aviation Turbine Fuel	1010
ASTM D3339	Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration	NA
ASTM D3341	Lead in Gasoline—Iodine Monochloride Method	NA
ASTM D3828	Flash Point by Small Scale Closed Cup Tester	NA
ASTM D4045	Sulfur in Petroleum Products by Hydrogenolysis and Rateometric Colorimetry	1405
ASTM D4052	Density, Relative Density, and API Gravity of Liquids by Digital Density Meter	1734
ASTM D4294	Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry	1635
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ASTM D4530	Determination of Carbon Residue (Micro Method)	1589
ASTM D4539	Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT)	NA
ASTM D4629	Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection	1129; 1527
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ASTM D4739	Base Number Determination by Potentiometric Hydrochloric Acid Titration	1217; 1638
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ASTM D4814	Specification for Automotive Spark-Ignition Engine Fuel	
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ASTM D5059	Lead in Gasoline by X-ray Spectroscopy	1283
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ASTM D5441	Analysis of Methyl <i>Tert</i> -Butyl Ether (MTBE) by Gas Chromatography	1306
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ASTM D5622	Determination of Total Oxygen in Gasoline and Methanol Fuels by Reductive Pyrolysis	1338
ASTM D5623	Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection	1335
ASTM D5762	Nitrogen in Petroleum and Petroleum Products by Boat-Inlet Chemiluminescence	1370; 1507
ASTM D5771	Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)	1373; 1508; 1524; 1740
ASTM D5772	Cloud Point of Petroleum Products (Linear Cooling Rate Method)	1373; 1509; 1524
ASTM D5773	Cloud Point of Petroleum Products (Constant Cooling Rate Method)	1373; 1510; 1524
ASTM D5797	Specification for Fuel Methanol (M70-M85) for Automotive Spark-Ignition Engines	
ASTM D5798	Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines	
ASTM D5845	Determination of MTBE, ETBE, TAME, DIPE, Methanol, Ethanol, and <i>Tert</i> -Butanol in Gasoline by Infrared Spectroscopy	1374
ASTM D5846	Universal Oxidation Test for Hydraulic and Turbine Oils Using the Universal Oxidation Test Apparatus	NA
ASTM D5864	Determining Aerobic Aquatic Biodegradation of Lubricants or Their Components	1584
ASTM D5949	Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)	1312; 1499
ASTM D5950	Pour Point of Petroleum Products (Automatic Tilt Method)	1312; 1499; 1740
ASTM D5983	Specification for Methyl Tertiary-Butyl Ether (MTBE) for Downstream Blending for Use in Automotive Spark-Ignition Engine Fuel	
ASTM D5985	Pour Point of Petroleum Products (Rotational Method)	1312
ASTM D6006	Guide for Assessing Biodegradability of Hydraulic Fluids	
ASTM D6046	Classification of Hydraulic Fluids for Environmental Impact	
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ASTM D6304	Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration	1436
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ASTM D6384	Terminology Relating to Biodegradability and Ecotoxicity of Lubricants	
ASTM D6423 ^a	Determination of pH of Denatured Fuel Ethanol and Ethanol Fuel Blends	NA
ASTM D6450	Flash Point by Continuously Closed Cup (CCCFP) Tester	1464
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ASTM D6749	Pour Point of Petroleum Products (Automatic Air Pressure Method)	1499
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ASTM D6866 ^a	Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis	NA
ASTM D6890	Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber	1602
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ASTM D7319 ^a	Determination of Existent and Potential Sulfate and Inorganic Chloride in Fuel Ethanol and Butanol by Direct Injection Suppressed Ion Chromatography	1614
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ASTM D7344	Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Mini Method)	1455; 1621
ASTM D7345	Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure (Micro Distillation Method)	1621
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ASTM D7373	Predicting Biodegradability of Lubricants Using a Bio-kinetic Model	
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ASTM D7467	Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)	
ASTM D7501	Determination of Fuel Filter Blocking Potential of Biodiesel (B100) Blend Stock by Cold Soak Filtration Test (CSFT)	1649; 1672
ASTM D7544	Specification for Pyrolysis Liquid Biofuel	
ASTM D7545	Oxidation Stability of Middle Distillate Fuels—Rapid Small Scale Oxidation Test (RSSOT)	NA
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ASTM D7579^a	Pyrolysis Solid Content in Pyrolysis Liquids by Filtration of Solids in Methanol	1664
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ASTM D7717	Practice for Preparing Volumetric Blends of Denatured Fuel Ethanol and Gasoline Blendstocks for Laboratory Analysis	
ASTM D7754	Determination of Trace Oxygenates in Automotive Spark-Ignition Engine Fuel by Multidimensional Gas Chromatography	NA
ASTM D7757	Silicon in Gasoline and Related Products by Monochromatic Wavelength Dispersive X-Ray Fluorescence Spectrometry	1735
ASTM D7794	Blending Mid-Level Ethanol Fuel Blends for Flexible-Fuel Vehicles with Automotive Spark-Ignition Engines	
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ASTM D7796	Ethyl Tert-Butyl Ether (ETBE) by Gas Chromatography	NA
ASTM D7797^a	Determination of the Fatty Acid Methyl Esters Content of Aviation Turbine Fuel Using Flow Analysis by Fourier Transform Infrared Spectroscopy—Rapid Screening Method	NA
ASTM D7798	Boiling Range Distribution of Petroleum Distillates with Final Boiling Points up to 538°C by Ultra Fast Gas Chromatography (UF-GC)	NA
ASTM D7806^a	Determination of the Fatty Acid Methyl Ester (FAME) Content of a Blend of Biodiesel and Petroleum-Based Diesel Fuel Oil Using Mid-Infrared Spectroscopy	NA
ASTM D7861^a	Determination of the Fatty Acid Methyl Esters (FAME) in Diesel Fuel by Linear Variable Filter (LVF) Array Based Mid-Infrared Spectroscopy	1624; 1795
ASTM D7862	Specification for Butanol for Blending with Gasoline for Use as Automotive Spark-Ignition Engine Fuel	
ASTM D7875^a	Determination of Butanol and Acetone Content of Butanol for Blending with Gasoline by Gas Chromatography	NA
ASTM D7920^a	Determination of Fuel Methanol (M99) and Fuel Methanol Blends (M10 to M99) by Gas Chromatography	NA
ASTM D7923	Water in Ethanol and Hydrocarbon Blends by Karl Fischer Titration	
ASTM D7963^a	Determination of Contamination Level of Fatty Acid Methyl Esters in Middle Distillate and Residual Fuels Using Flow Analysis by Fourier Transform Infrared Spectroscopy—Rapid Screening Method	NA
ASTM E70	pH of Aqueous Solutions with the Glass Electrode	NA
ASTM E203	Water Using Volumetric Karl Fischer Titration	NA
ASTM E870^a	Analysis of Wood Fuels	NA
ASTM E871^a	Moisture Analysis of Particulate Wood Fuels	NA
ASTM E872^a	Volatile Matter in the Analysis of Particulate Wood Fuels	NA
ASTM E873^a	Bulk Density of Densified Particulate Biomass Fuels	NA

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ASTM E1064	Water in Organic Liquids by Coulometric Karl Fischer Titration	NA
ASTM E1126^a	Terminology Relating to Biomass Fuels (Withdrawn 2003)	
ASTM E1192	Guide for Conducting Acute Toxicity Tests on Aqueous Ambient Samples and Effluents with Fishes, Macroinvertebrates, and Amphibians	
ASTM E1279	Biodegradation by a Shake-Flask Die-Away Method	
ASTM E1295	Guide for Conducting Three-Brood, Renewal Toxicity Tests with <i>Ceriodaphnia dubia</i>	
ASTM E1358^a	Determination of Moisture Content of Particulate Wood Fuels Using a Microwave Oven	NA
ASTM E1534^a	Determination of Ash Content of Particulate Wood Fuels	NA
ASTM E1625	Determining Biodegradability of Organic Chemicals in Semi-Continuous Activated Sludge (SCAS)	NA
ASTM E1690^a	Determination of Ethanol Extractives in Biomass	NA
ASTM E1705^a	Terminology Relating to Biotechnology	
ASTM E1720	Determining Ready, Ultimate, Biodegradability of Organic Chemicals in a Sealed Vessel CO ₂ Production Test	NA
ASTM E1721^a	Determination of Acid-Insoluble Residue in Biomass	NA
ASTM E1755^a	Ash in Biomass	NA
ASTM E1756^a	Determination of Total Solids in Biomass	NA
ASTM E1757^a	Preparation of Biomass for Compositional Analysis	
ASTM E1758^a	Determination of Carbohydrates in Biomass by High Performance Liquid Chromatography	NA
ASTM E1798	Assessing Treatability or Biodegradability, or Both, of Organic Chemicals in Porous Pots	
ASTM E1821^a	Determination of Carbohydrates in Biomass by Gas Chromatography	NA
ASTM E2170	Determining Anaerobic Biodegradation Potential of Organic Chemicals Under Methanogenic Conditions	

Other Standards

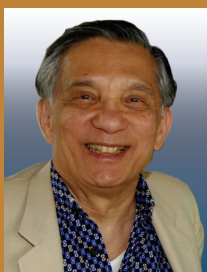
EN 14078 ^a	Determination of Fatty Acid Methyl Ester (FAME) Content in Middle Distillate Fuels by Infrared Spectroscopy	NA
EN 14103 ^a	Determination of Ester and Linolenic Acid Methyl Ester Contents in FAMES	NA
EN 14105 ^a	Determination of Free and Total Glycerol and Mono-, Di-, and Triglyceride Contents of FAMES	NA
EN 14110 ^a	Determination of Methanol Content of FAMES	NA
EN 14112 ^a	Determination of Oxidation Stability (Accelerated Oxidation Test) of FAMES	NA
EN 14538 ^a	Determination of Ca, K, Mg, and Na Content of FAME by Optical Emission Spectral Method with Inductively Coupled Plasma	NA
EN 15751 ^a	Determination of Oxidation Stability of FAME Fuel and Blends with Diesel Fuel by Accelerated Oxidation Method	NA
EN 15779 ^a	Determination of Polyunsaturated Fatty Acid Methyl Esters (FAMES) by Gas Chromatography	NA
IP 599 ^a	Determination of Fatty Acid Methyl Esters (FAME) in Aviation Turbine Fuel	NA
ISO 13032 ^a	Determination of Low concentration of Sulfur in Automotive Fuels Using Energy Dispersive X-Ray Fluorescence Spectrometric Method	NA

UOP Methods

UOP389	Trace Metals in Organics by ICP-OES	NA
UOP391	Trace Metals in Petroleum Products or Organics by AAS	NA

Note: Rows in bold type are ASTM biofuels specifications; NA = not available.

^aTest method standards specifically developed for the analysis of biofuels.



Dr. R.A. Kishore Nadkarni received his Ph.D. in analytical chemistry at the University of Bombay. Since then he has worked as a research associate at the University of Kentucky, manager of the Materials Science Center Analytical Facility at Cornell University, and analytical leader in the ExxonMobil Company. In his last position he was responsible for technical quality management of the Paramins Division's global plant laboratories.

He has authored more than 140 technical publications including 21 new ASTM standards in the area of analytical chemistry and quality management. He is a member of the American Chemical Society and ASTM International. He is very active in ASTM and ISO in the petroleum products and lubricant field, holding the position of immediate past chairman of ISO/TC28, chairman of ASTM's D02.03 on Elemental Analysis, vice-chairman of D02.92 on Proficiency Test Programs, D02.94 on quality Assurance and Statistics.

Dr. Nadkarni has received the Award of Appreciation (1991) and Awards for Excellence (1998, 1999, and 2013) from ASTM's D02 Committee for his contribution to the oil industry, the Award of Merit (2005) and the George Dyroff Award of Honorary D02 membership (2006), and the Sydney D. Andrews D02 Scroll of Achievement Award (2009).

He is the author or editor of STP 1109, Modern Instrumental Methods of Elemental Analysis of Petroleum Products and Lubricants (1991); STP 1468, Elemental Analysis of Fuels and Lubricants (2005); Manual 44, Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants (2007); Manual 61, Guide to ASTM Test Methods for the Analysis of Coal and Coke (2008); Monograph 9, Spectroscopic Analysis of Petroleum Products and Lubricants (2011); Monograph 10, Elemental Analysis of Fossil Fuels and Related Materials (2014) and Monograph 11, Sulfur: Chemistry and Analysis of Fossil Fuel Products.

Review:

Analysis of Biofuels: A Laboratory Resource by R.A. Kishore Nadkarni presents an authoritative and essential review of biofuels technology, a vitally important technology area of increasing importance. Topical areas that are expertly covered include: product specifications and an up-to-date overview of test methods for physical and chemical analysis, environmental analysis, and bioenergy from biomass. In addition, ASTM proficiency testing programs for biofuels are detailed. This book assuredly will be an invaluable working reference for practitioners in the fuels technology area.

—George E. Totten, Ph.D., Portland State University, Portland, OR, USA

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