

Designation: D7544 - 09

Standard Specification for Pyrolysis Liquid Biofuel¹

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1. Scope

1.1 This specification covers a pyrolysis liquid biofuel produced from biomass intended for use in industrial burners equipped to handle these types of fuels. This type of biofuel is not intended for use in residential heaters, small commercial boilers, engines, or marine applications.

NOTE 1—For information on the significance of the physical, chemical, and performance properties identified in this specification, see Appendix X1.

1.2 This specification is for use in contracts for the purchase of pyrolysis liquid biofuel and for guidance of consumers of this type of fuel.

1.3 Nothing in this specification should preclude observance of national or local regulations, which may be more restrictive.

NOTE 2—The generation and dissipation of static electricity may create problems in the handling of pyrolysis liquid biofuel. For more information on the subject, see Guide D4865.

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

1.4.1 *Exception*—BTU units are included for information only in 3.5.1.

1.5 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.

2. Referenced Documents

2.1 ASTM Standards:²

- D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- D97 Test Method for Pour Point of Petroleum Products

- D240 Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
- D396 Specification for Fuel Oils
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D482 Test Method for Ash from Petroleum Products
- D4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter
- 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
- D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

- D6469 Guide for Microbial Contamination in Fuels and Fuel Systems
- E70 Test Method for pH of Aqueous Solutions With the Glass Electrodeobd 88eb6e43/astm-d7544-09

E203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.2 *bulk fuel*, *n*—fuel in the storage facility in quantities over 190 L.

3.3 *char*, *n*—fine carbonaceous powder that is separated from the vapors of biomass during pyrolysis.

3.3.1 *Discussion*—Pyrolysis liquid biofuel contains uniformly suspended char.

3.4 *fuel degradation products*, *n*—those materials that are formed in fuel during extended storage or exposure to high temperatures.

3.4.1 *Discussion*—During storage, reactive organic compounds in pyrolysis liquid can act together to form larger molecules (fuel degradation products), which can become insoluble or increase the fuel viscosity, or both.

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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.



TABLE 1 Detailed Requirements for Pyrolysis Liquid Biofuels

Property	Test Method	Specification	Units
Gross Heat of Combustion	D240	15 min	MJ/kg
Water Content	E203	30 max	mass %
Pyrolysis Solids Content	Annex A1	2.5 max	mass %
Kinematic Viscosity at 40°C	D445 ^A	125 max	mm²/s
Density at 20°C	D4052	1.1–1.3	kg/dm ³
Sulfur Content	D4294	0.05 max	mass %
Ash Content	D482	0.25 max	mass %
pH	E70	Report	
Flash Point	D93, Procedure B	45 min	°C
Pour Point	D97	–9 max	°C

^A Without filtering

3.5 *industrial burner*, *n*—device which produces heat for industrial use through the combustion of liquid fuels.

3.5.1 *Discussion*—Industrial burners are typically designed for one of two applications: (1) industrial furnaces—integral components of manufacturing processes that provide direct heating; for example, in aggregate, cement, lime, or phosphate kilns; coke ovens; or blast, smelting, melting, refining, or drying ovens and (2) industrial boilers—large indirect heating units which transfer thermal energy to water or other fluids or gases for use in heating in industrial settings, power generation and in manufacturing processes. These boilers can be classified as utility/large industrial boilers with a heat input greater than 105 GJ/h (100 × 106 BTU/h) or small industrial boilers with a heat input of between 10.5 to 105 GJ/h (10 to 100 × 106 BTU/h).

3.6 *long-term storage*—storage of fuel for longer than 3 months after it is received by the user.

3.7 *medium-term storage*—storage of fuel for up to 3 months after it is received by the user.

3.7.1 *Discussion*—It is recommended that fuel be consumed within 6 months of receipt.

3.8 *pyrolysis*, *n*—chemical decomposition of organic materials by heating in the absence of oxygen.

3.9 *pyrolysis liquid biofuel*, *n*—liquid product from the pyrolysis of biomass.

3.9.1 *Discussion*—Pyrolysis liquid biofuel is comprised of a complex mixture of the decomposition products of lignocellulosic biomass including highly oxygenated organic compounds. It is produced from the pyrolysis of biomass, followed by the rapid condensation of its vapors.

3.10 *pyrolysis solids*, *n*—solid particles contained within the pyrolysis liquid biofuel.

3.10.1 *Discussion*—Pyrolysis solids is comprised of ash and char.

4. General Requirements

4.1 The pyrolysis liquid biofuel specified in this specification shall remain uniform in medium-term storage and not separate by gravity into layers.

NOTE 3—Long-term storage or equipment down time can necessitate circulation of pyrolysis liquid biofuel in-tank to prevent such separation. The buyer and seller should agree on any requirements for long-term storage. If minor separation occurs during medium-term storage, mild agitation or product circulation should reverse such separation.

5. Detailed Requirements

5.1 The pyrolysis liquid biofuel specified shall conform to the detailed requirements shown in Table 1.

5.2 The properties selected for limitation are those that are believed to be of the greatest significance in obtaining acceptable performance of the burner.

6. Sampling

6.1 Review all intended test methods prior to sampling to understand the importance and effects of sampling technique, proper containers, and special handling required for each test method. See Table 2.

6.2 As indicated in 4.1, during medium-term storage, pyrolysis liquid biofuel shall remain uniform and not separate into layers. Note, however, that separation may occur during long-term storage. Therefore, samples should be well mixed when transferring from the primary sampling process or container, or both, to another container or analytical apparatus, or both. Sampling from an active circulation loop or a well mixed or agitated tank is preferred. Refer to Practice D5854 for more guidance on mixing and handling samples.

6.3 Sample Size—A minimum of 1 L is recommended.

7. Test Methods

7.1 The requirements enumerated in this specification shall be determined in accordance with the following test methods:

- 7.1.1 Gross Heat of Combustion—Test Method D240.
- 7.1.2 Water Content—Test Method E203.
- 7.1.3 Pyrolysis Solids Content—See Annex A1.
- 7.1.4 Kinematic Viscosity—Test Method D445.
- 7.1.5 Density—Test Method D4052.
- 7.1.6 Sulfur—Test Method D4294.
- 7.1.7 Ash Content—Test Method D482.
- 7.1.8 pH—Test Method E70.
- 7.1.9 Flash Point—Test Method D93.
- 7.1.10 Pour Point—Test Method D97.

8. Keywords

8.1 pyrolysis liquid biofuel; bio-oil; pyrolysis oil; pyoil; py-oil; bio-crude-oil; bio-fuel-oil; wood liquids; burner fuel; renewable energy; alternative energy; fuel oils; furnace oils

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TABLE 2 Typical Sampling Procedures for Containers

 Type of Container
 Procedure

 Storage tanks that are well-mixed by circulation or agitation
 Automatic or Manual Pipeline Sampling

 Tank cars, tank trucks, or ship tanks
 Dipper Sampling or All-Level Sampling

ANNEX

(Mandatory Information)

A1. TEST METHOD FOR PYROLYSIS SOLIDS CONTENT IN PYROLYSIS LIQUIDS BY FILTRATION OF SOLIDS IN METHANOL

A1.1 Scope

A1.1.1 This test method describes a filtration procedure for determining the pyrolysis solids content of pyrolysis liquid. It is intended for the analysis of pyrolysis liquid with all ranges of pyrolysis solids concentrations.³

A1.1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

A1.1.3 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. Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

A1.1.4 For Referenced Documents, see Section 2.

A1.1.5 For Terminology, see Section 3.

A1.2 Summary of Test Method

A1.2.1 A pyrolysis liquid sample is dissolved in a methanol and dichloromethane solution (1:1), which is then filtered through a vacuum filter system. After filtering, the filtrand is washed with the solvent until the filtrate is clear. The filter is removed, dried and weighed. The pyrolysis solids content is calculated based on the original pyrolysis liquid sample.

A1.3 Significance and Use

A1.3.1 Pyrolysis liquid can be produced to various char concentrations. Increasing pyrolysis solids content can affect the pyrolysis liquid biofuel handling, atomization, and storage stability in a negative manner.

A1.4 Apparatus (see Fig. A1.1)

A1.4.1 Smooth-tip Forceps.

- A1.4.2 Beaker, 400 mL.
- A1.4.3 Glass Stirring Rod.

A1.4.4 *Oven*, explosion-proof, capable of maintaining a temperature of $105 \pm 3^{\circ}$ C.

A1.4.5 Filtering Flask, 1 L.

A1.4.6 Filter Holders, borosilicate glass.

A1.4.7 Filter Membrane, binder free glass microfiber, 1 µm.

A1.4.8 Weighing Dish, aluminum.

A1.4.9 *Balance*, capable of weighing to the nearest 0.0001 g (0.1 mg) with a range of 300 g.

A1.4.10 Vacuum.

A1.5 Reagents and Solvents

A1.5.1 Purity of Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If an industrial grade reagent is used, it shall be filtered using 0.45 µm filter paper prior to use.

A1.5.2 *Ethanol*, reagent grade (**Warning**—Flammable. Toxic. Can be harmful or fatal if ingested or inhaled. Avoid skin contact.)00-99bd88eb6e43/astm-d7544-09

A1.5.3 *Methanol (MeOH)*, reagent grade (**Warning**—Flammable. Toxic. Can be harmful or fatal if ingested or inhaled. Avoid skin contact.)

A1.5.4 *Dichloromethane* (*DCM*), reagent grade (**Warning**—Flammable. Toxic. Can be harmful or fatal if ingested or inhaled. Avoid skin contact.)

A1.5.5 *Filter Paper*, 1 µm pore size, binder free glass microfiber.

A1.6 Sampling

A1.6.1 Obtain a sample using either Practice D4057 or D4177.

A1.6.2 Obtain a representative sample of the pyrolysis liquid from a well-mixed container. The sample should be well mixed to ensure homogeneity.

³ Oasmaa, A., and Peacocke, C., A Guide To Physical Property Characterization Of Biomass-Derived Fast Pyrolysis Liquids, VTT Publications 450, VTT Energy, Technical Research Centre of Finland, ESPOO 2001, www.vtt.fi/inf/pdf/ publications/2001/P450.pdf

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.