



# Standard Specification for Pyrolysis Liquid Biofuel<sup>1</sup>

This standard is issued under the fixed designation D7544; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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:<sup>2</sup>

[D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester](#)

[D97 Test Method for Pour Point of Petroleum Products](#)

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- [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 Electrode](#)
- [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.

**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 <sup>A</sup>	125 max	mm <sup>2</sup> /s
Density at 20°C	D4052	1.1–1.3	kg/dm <sup>3</sup>
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

<sup>A</sup> 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

**TABLE 2 Typical Sampling Procedures for Containers**

Type of Container	Procedure
Storage tanks that are well-mixed by circulation or agitation Tank cars, tank trucks, or ship tanks	Automatic or Manual Pipeline Sampling 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.<sup>3</sup>

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 ± 3°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.<sup>4</sup> 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.)*

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.

<sup>3</sup> 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](http://www.vtt.fi/inf/pdf/publications/2001/P450.pdf)

<sup>4</sup> *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.

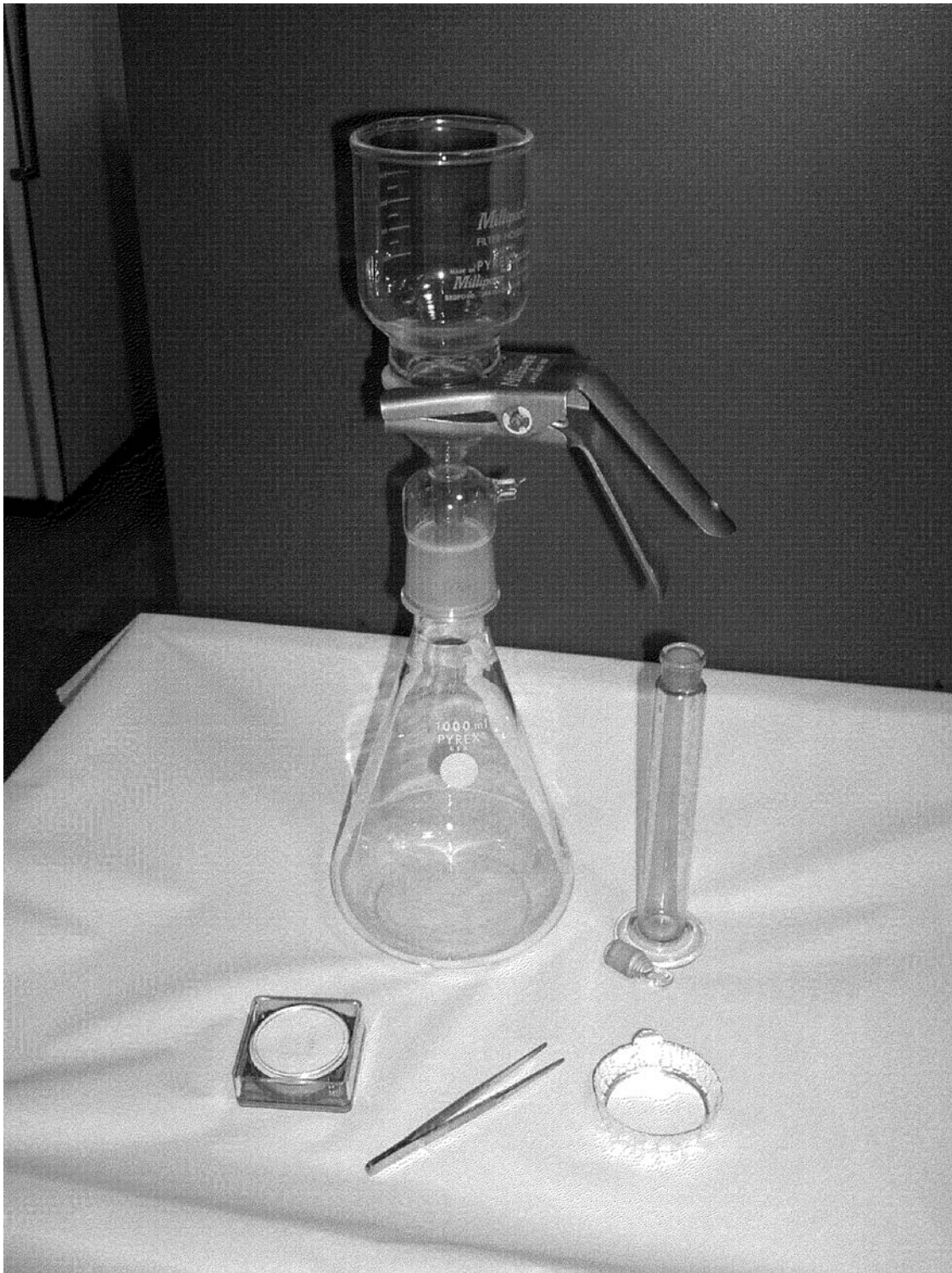


FIG. A1.1 Paper Filtration Apparatus

## A1.7 Procedure

A1.7.1 Dry a clean filter membrane for 15 min in a desiccator, place in a labeled aluminum weighing dish, and weigh to the nearest 0.1 mg.

A1.7.2 Place a 400 mL beaker on the balance, and tare it.

A1.7.3 Vigorously shake the sample by hand for a minimum of 15 s.

A1.7.4 Using a disposable pipette, weigh approximately 15 g of pyrolysis liquid sample into the beaker. Record the weight to the nearest 0.1 mg.

A1.7.5 Add approximately 100 to 200 mL MeOH-DCM solution (1:1) into the beaker and stir the mixture vigorously to dissolve the pyrolysis liquid in the solvent.

A1.7.6 Mount the filter on a dry holder and apply a vacuum. Mount and securely clamp the filter funnel to the filter holder.

A1.7.7 Use methanol to wash the filter paper to properly seal the latter to the bottom of the funnel.

A1.7.8 Filter the solution through the vacuum filter system on 1 μm filter paper. Quickly but carefully pour the solution into the center of the funnel. Thoroughly wash the beaker with MeOH-DCM solution and return the contents to the funnel. Wash the filtrand with methanol until the filtrate runs clear.

A1.7.9 Remove the filter funnel from the filter holder and ensure the edges of the filter funnel are clean of any pyrolysis solids. If required, rinse any pyrolysis solids on the filter funnel onto the filter paper with methanol, ensuring no pyrolysis solids are lost.

A1.7.10 Release the vacuum, and using the smooth-tip forceps transfer the filter membrane and filtrand to its original weighing dish, and dry in the oven at  $105 \pm 3^\circ\text{C}$  for 30 min.

A1.7.11 Remove the weighing dish and filter membrane and cool to room temperature in a desiccator for a minimum of 1 h.

A1.7.12 Weigh the filter membrane and weighing dish and record the stabilized weight to the nearest 0.1 mg.

## A1.8 Calculation

A1.8.1 Calculate the pyrolysis solids content of the pyrolysis liquid sample in accordance with Eq A1.1.

$$\text{pyrolysis solids (wt \%)} = \frac{PS_1}{PL} \times 100\% \quad (\text{A1.1})$$

pyrolysis solids = pyrolysis solids content, wt%,  
 $PS_1$  = pyrolysis solids retained on 1 μm filter paper (g), and  
 $PL$  = pyrolysis liquid sample taken for analysis (g).

## A1.9 Report

A1.9.1 Report the pyrolysis oil solids content to two significant figures.

## A1.10 Precision and Bias <sup>5</sup>

A1.10.1 This interim precision statement represents replicate analyses performed in two laboratories over ten successive days by the same analyst on the same day on the same instrument.

A1.10.2 *Repeatability*—The difference between test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material in a short amount of time, would in the long run, in the normal and correct operation of this test method, exceed 0.1303X wt% only in one case in twenty.

A1.10.3 *Reproducibility*—At this time, no interlaboratory precision data have been obtained to allow a calculation for reproducibility.

A1.10.4 *Bias*—No information can be presented on the bias of this procedure for measuring pyrolysis solids content because no accepted reference value is available.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1664.

## APPENDICES

### (Nonmandatory Information)

#### X1. SIGNIFICANCE OF ASTM SPECIFIED PROPERTIES FOR PYROLYSIS LIQUID BIOFUEL

##### X1.1 Heat of Combustion

X1.1.1 Heat of combustion is a measure of the energy content of pyrolysis liquid. As a reference, pyrolysis liquid has approximately half the heat of combustion on a volumetric basis compared to #2, D396 fuel oil. The heat of combustion is an important basis for quantifying its monetary value and for equipment selection or design, or both.

##### X1.2 Water Content

X1.2.1 Higher water content leads to lower overall viscosity and heat of combustion of pyrolysis liquid.

X1.2.2 Water content reduces the flame temperature of pyrolysis liquid, which contributes to lower nitrogen dioxide emissions during combustion.

X1.2.3 Water content higher than the limit, can cause phase separation, leading to a non-homogenous mixture.

##### X1.3 Char Content

X1.3.1 Pyrolysis liquid biofuel contains uniformly suspended char. Pyrolysis liquid can be produced to various char concentrations. Increasing char content may also increase the ash content, viscosity and pour point of pyrolysis liquid biofuel and affect the pyrolysis liquid biofuel handling, atomization and storage stability in a negative manner.

##### X1.4 Viscosity

X1.4.1 The viscosity of a pyrolysis liquid is a measure of its resistance to flow. It is of major importance so that adequate

preheating facilities can be provided to permit the pyrolysis liquid to be pumped to the burner and to provide good atomization. However, it is equally important that the maximum viscosity under the existing conditions be such that the pyrolysis liquid can be pumped satisfactorily from the storage tank to the preheater.

### **X1.5 Density**

X1.5.1 Density alone is of little significance as an indication of the burning characteristics of pyrolysis liquid. However, when used in conjunction with other properties, it is of value in mass-volume relationships.

### **X1.6 Sulfur Content**

X1.6.1 Knowledge of the sulfur content is important for evaluating the Sulfur Oxides emissions from combusting pyrolysis liquid.

### **X1.7 Ash Content**

X1.7.1 Ash is the amount of noncombustible material in pyrolysis liquid biofuel. Ash-forming materials may be present in pyrolysis liquid biofuel in two forms, solid particles or water soluble metallic compounds, or both. Depending on the size and type (sand, char, etc.) of the solid ash particles, these particles can contribute to wear of burner pumps and valves,

and decrease fuel efficiency by fouling heat exchange surfaces. The soluble metallic compounds have little or no effect on wear or plugging, but they can contain elements that produce corrosion and deposits on boiler heating surfaces. Excessive amounts of ash also may necessitate particulate collection equipment for compliance with national or local air emission regulations.

### **X1.8 pH**

X1.8.1 Due to its low pH, materials which are in contact with pyrolysis liquid shall be selected for suitability and corrosion resistance.

### **X1.9 Flash Point**

X1.9.1 The flash point of a fuel is an indication of the maximum temperature at which it can be stored and handled without serious fire hazard.

### **X1.10 Pour Point**

X1.10.1 The pour point of pyrolysis liquid is an indication of the lowest temperature at which the pyrolysis liquid is capable of flowing under very low forces. The pour point is prescribed in accordance with the conditions of storage and use. Higher pour point fuels are permissible where heated storage and adequate piping facilities are provided.

## **X2. LONG-TERM STORAGE OF PYROLYSIS LIQUID**

### **X2.1 Scope**

X2.1.1 This appendix provides guidance for consumers of pyrolysis liquid who may wish to store quantities of the fuel for extended periods.

X2.1.2 Pyrolysis liquid has adequate stability properties to withstand normal storage and use without formation of troublesome amounts of insoluble degradation products.

X2.1.3 The following suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the fuel equipment manufacturer or by federal, state or local government regulations. Further guidance in developing a fuel management system could be provided by fuel suppliers.

### **X2.2 Terminology (See Section 3)**

### **X2.3 Fuel Selection**

X2.3.1 The stability properties of pyrolysis liquid are not fully understood and appear to depend on the biomass feedstock sources, the pyrolysis reaction conditions, the pyrolysis solids content of the pyrolysis liquid, whether additives are present and storage conditions.

X2.3.2 The composition and stability properties of pyrolysis liquid produced at specific production plants can be different. A special requirement of the user, such as long-term storage, should be discussed with the supplier.

### **X2.4 Fuel Storage Conditions**

X2.4.1 Store at a minimum temperature of 10°C to maintain adequate fluidity, but not higher than 30°C for long-term storage. High storage temperatures accelerate fuel degradation. Recommended storage temperature is 15 to 20°C.

X2.4.2 Store in containers built of corrosion resistant steel and materials such as AISI 304, AISI 316, PTFE (polytetrafluoroethylene), PP (polypropylene), HDPE (high density polyethylene) and PVC (polyvinylchloride).

X2.4.3 Long-term storage in an agitated or circulated container is recommended to maintain homogeneity.

X2.4.4 Limit exposure to air to prevent oxidation.

### **X2.5 Fuel Additives**

X2.5.1 Fuel additives are not required in pyrolysis liquid biofuel.

X2.5.2 Polar solvent additives, in particular methanol and ethanol, can be used to improve the long-term storage of pyrolysis liquid. For maximum benefits, additives should be added directly following production. The addition of a polar solvent to the pyrolysis liquid can significantly lower the flash point and appropriate precautions shall be taken.

X2.5.3 Hydrocarbon fuel is not miscible in pyrolysis liquid biofuel.

## **X2.6 Tests for Fuel Quality**

X2.6.1 Work has started on a stability index that correlates the long-term storage stability of pyrolysis liquid against accelerated aging tests at increased temperatures.

## **X2.7 Fuel Monitoring**

X2.7.1 Stored fuel should be periodically sampled and its quality assessed. Measurement and comparison of the stored fuel's viscosity by Test Method **D445** can be used to assess fuel degradation.

## **X2.8 Use of Degraded Fuels**

X2.8.1 Fuels that have undergone mild-to-moderate degradation can often be consumed in a normal way, depending on

the fuel system requirements. Filters and other cleanup equipment may require special attention and increased maintenance. Burner nozzle or injector fouling may occur more rapidly.

X2.8.2 If for example, the fuel is too thick for a particular application, mixing in a polar solvent to the pyrolysis liquid prior to use is known to reduce its viscosity and with vigorous mixing will dissolve some insolubles back into the fuel. The addition of a polar solvent to the pyrolysis liquid can significantly lower the flash point and appropriate precautions shall be taken.

## **X3. HAZARDS**

X3.1 The biofuels defined by this specification may have a lower pH and gross heat of combustion than **D396** specification fuels. The materials of construction and equipment sizing of

the industrial burner and its fuel handling system shall be compatible with the properties of the biofuels defined by this specification.

## **X4. MICROBIAL CONTAMINATION**

X4.1 Uncontrolled microbial contamination in fuel systems can cause or contribute to a variety of problems including increased corrosivity, and decreased stability, filterability, and caloric value. Microbial processes in fuel systems can also cause or contribute to system damage.

X4.2 Because the microbes contributing to the aforementioned problems may not be necessarily present in the fuel itself, no microbial quality criterion for fuels is recommended.

However, it is important that personnel responsible for fuel quality understand how uncontrolled microbial contamination can affect fuel quality.

X4.3 Guide **D6469** provides personnel with limited microbiological background an understanding of the symptoms, occurrences, and consequences of chronic microbial contamination. Guide **D6469** also suggests means for detecting and controlling microbial contamination in fuels and fuel systems.

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