Designation: D7579 – 09

Standard Test Method for Pyrolysis Solids Content in Pyrolysis Liquids by Filtration of Solids in Methanol[1]

This standard is issued under the fixed designation D7579; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

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.

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

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. For specific warning statements, see 7.2, 7.3, and 7.4.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 *Other Standards:*[3]

ESPOO 2001 A guide to physical property characterisation of biomass-derived fast pyrolysis liquids

3. Terminology

3.1 *Definitions:*

3.1.1 See also Terminology D4175.

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

3.1.2.1 *Discussion*—Pyrolysis liquid biofuel contains uniformly suspended char at varying concentrations.

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

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

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

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

3.1.5.1 *Discussion*—Pyrolysis solids consists of ash and char.

4. Summary of Test Method

4.1 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.

5. Significance and Use

5.1 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.

6. Apparatus (see Fig. 1)

6.1 *Smooth-tip Forceps*.

6.2 *Beaker*, 400 mL.

6.3 *Glass Stirring Rod*.

6.4 *Oven*, explosion-proof, capable of maintaining a temperature of 105 ± 3°C.

6.5 *Filtering Flask*, 1 L.

6.6 *Filter Holders*, borosilicate glass.

6.7 *Filter Membrane*, binder free glass microfiber, 1 µm.

6.8 *Weighing Dish*, aluminum.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

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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] Available from VTT Energy, New Energy Technologies, Biologinkuja 3–5, P.O. Box 1601, FIN-02044 VTT, Finland. http://www.vtt.fi/inf/pdf/publications/2001/P450.pdf

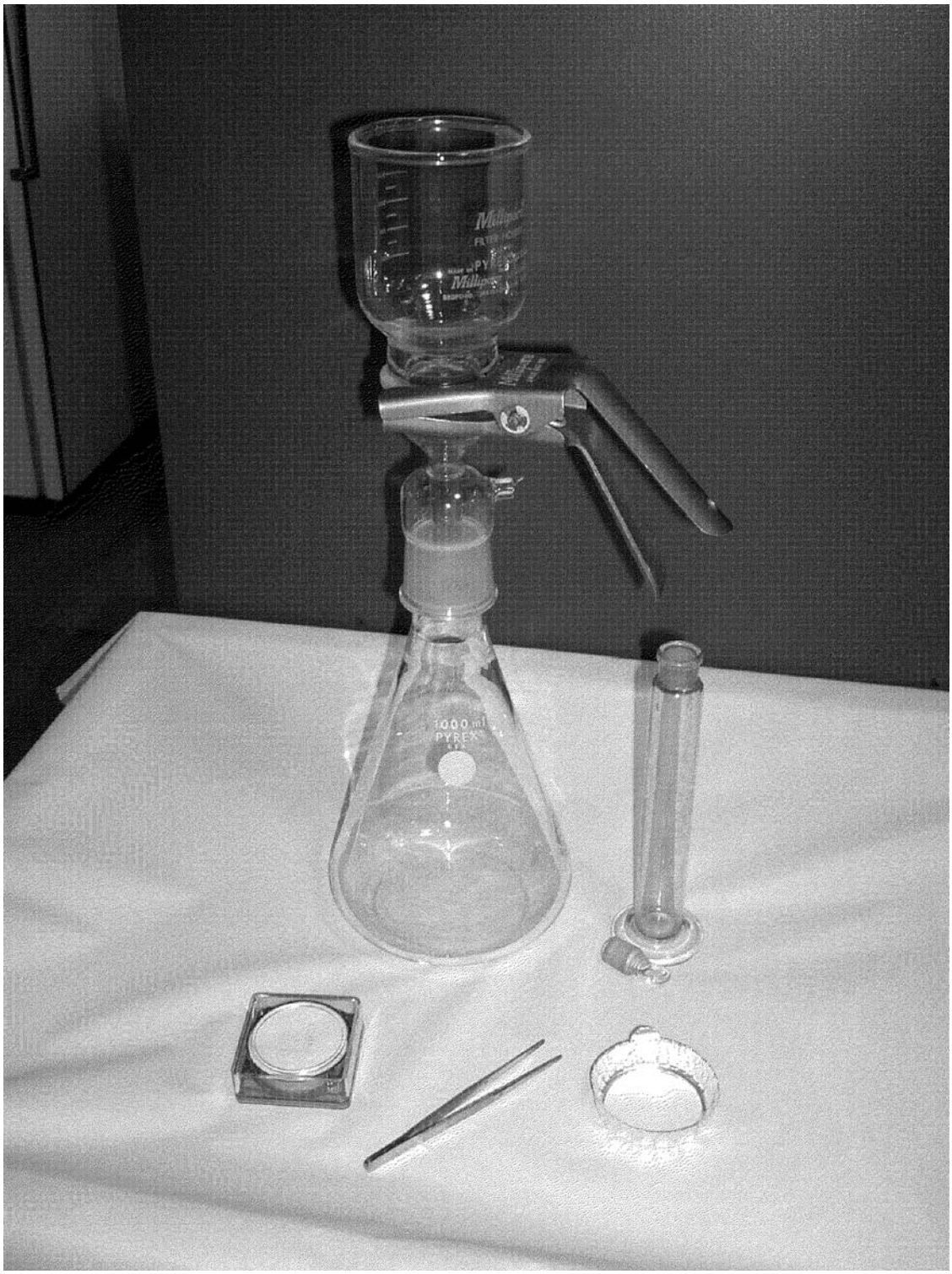

FIG. 1 Paper Filtration Apparatus

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

6.10 *Vacuum*.

7. Reagents and Solvents

7.1 *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.

7.2 *Ethanol*, reagent grade. (**Warning—**Flammable. Toxic. May be harmful or fatal if ingested or inhaled. Avoid skin contact.

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

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

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

8. Sampling

8.1 Obtain a sample using either Practice D4057 or Practice D4177.

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

9. Procedure

9.1 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.

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

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

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

9.5 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.

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

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

9.8 Filter the solution through the vacuum filter system on 1 µm filter paper. Quickly but carefully poor 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.

9.9 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.

9.10 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 ± 3°C for 30 min.

9.11 Remove the weighing dish and filter membrane and cool to room temperature in a dessicator for a minimum of 1 h.

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

10. Calculation

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

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

where:

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).

11. Report

11.1 Report the pyrolysis oil solids content to two significant figures.

12. Precision and Bias [5]

12.1 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.

12.2 *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.

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

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

13. Keywords

13.1 char; filtration; pyrolysis liquids; pyrolysis solids

[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.

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