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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Foots in Raw Linseed Oil (Gravimetric Method)[1]

This standard is issued under the fixed designation D 1966; 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 method covers the determination of foots in raw linseed oil by the gravimetric method. The procedure is commonly known in Europe as the PAT foots method.

1.2 *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:*

D 1193 Specification for Reagent Water[2]

3. Terminology

3.1 *Definition:*

3.1.1 *foots*—those solid impurities that precipitate from raw linseed oil during storage and which then settle to the bottom or" foot" of a storage tank.

3.2 *Definition of Term Specific to This Standard:*

3.2.1 *foots*—in this method, the material that is precipitated from the oil by phosphoric acid and which is insoluble in acetone under the specific conditions of the test.

4. Summary of Test Method

4.1 The oil to be tested is refined with 85 % phosphoric acid. The precipitated material is settled by centrifuging and is washed free of oil with acetone. It is dried and weighed to determine the foots content gravimetrically.

5. Apparatus

5.1 *Fritted Glass Filtering Crucibles*, having a medium porosity (10 to 15 μm) and a capacity of 30 mL.

5.2 *Agitator*—The agitator shall consist of a horizontal shaft suitably supported and fitted with clamps or a clamping device for holding pear-shaped centrifuge tubes. The tubes shall be held in such fashion that when the shaft rotates, the tubes will be tipped end over end, thus allowing the liquid content of the tube to mix as it flows from one end of tube to the other. The shaft shall be rotated mechanically by any means desired that allows one to choose either of two fixed speeds; a slow speed of 16 ± 2 rpm and a fast speed of 32 ± 2 rpm.

5.3 *Centrifuge Tube*, pear-shaped conforming to dimensions given in Fig. 1, and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct and the mouth shall be constricted in shape for closure with a stopper.

5.4 *Centrifuge*—Capable of whirling two or more filled tubes at a speed that can be controlled to give a relative centrifugal force of between 500 to 800 *g* at the tip of the tubes. Calculate the speed of the rotating head as follows:

$$\text{rpm} = 265\sqrt{\text{rcf}/d} \quad (1)$$

where:

rcf = relative centrifugal force, *g*, and
d = diameter of swing, in., measured between tips of opposite tubes when in rotating position.

5.4.1 If *d* is given in millimetres, use the following equation:

$$\text{rpm} = 1346\sqrt{\text{rcf}/d} \quad (2)$$

NOTE 1—A list of applicable rotational speeds is given in Table 1.

5.5 *Pipet*, 1-mL measuring type graduated in 0.01-mL subdivisions.

5.6 *Desiccator*, containing an efficient desiccant. Anhydrous calcium sulfate is satisfactory.

6. Reagents

6.1 *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.[3] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications, and is the direct responsibility of D01.32 on Drying Oils.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *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 *Analar 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.

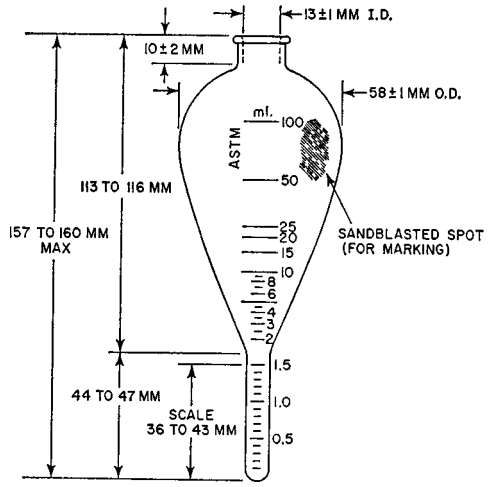

FIG. 1 Pear-Shaped Centrifuge Tube

TABLE 1 Rotational Speeds Applicable for Centrifuges of Various Diameters of Swing

Diameter in. (mm)	rpm at 500 rcf	rpm at 800 rcf
12(305)	1710	2160
13(330)	1650	2080
14(356)	1590	2000
15(381)	1530	1930
16(407)	1480	1870
17(432)	1440	1820
18(458)	1400	1770
19(483)	1360	1720
20(508)	1330	1680
21(533)	1300	1640
22(560)	1270	1600
23(584)	1240	1560
24(610)	1210	1530

6.3 *Acetone*.

6.4 *Filter Aid*, diatomaceous silica.[4]

6.5 *Phosphoric Acid* (85 %) (H_3PO_4).

7. Hazard

7.1 This standard may involve the use of hazardous materials, operations, and equipment. It is the responsibility of whoever uses this standard to establish appropriate safety practices to determine the applicability of regulatory limitations prior to use.

8. Preparation of Sample

8.1 Allow the sample to come to room temperature (25± 1°C) and shake or mix thoroughly. Be sure that all sediment has been thoroughly dispersed. If the oil is damp, dry by heating at 100°C under vacuum or by sparging with dry carbon dioxide or nitrogen at 100°C, cool the oil to 25°C, and proceed with test.

9. Procedure

9.1 Weigh into the centrifuge tube 50.0 ± 0.01 g of the sample and then add 0.5 ± 0.05 mL of phosphoric acid (85 %) with a pipet.

9.2 Stopper the tube and tilt it so that the acid runs out of the tip and into the oil. Shake vigorously for a few seconds. Repeat the tilting and shaking twice more.

9.3 Place the tube on the agitator and mix for 5 min at such a speed that the acid disperses completely throughout the oil and the tip of the tube empties of oil at each revolution. A speed of 16 r/m is adequate when using the above agitator. Adjust the speed of the agitator so that intimate mixing without separation takes place; 32 rpm is satisfactory. Mix at this rate for 25 min.

9.4 Place the tube in the centrifuge and spin for 1 h with a relative centrifugal force of at least 500 *g* at the tip or until the deposit stays in position as a compact mass when the tube is inverted. The temperature should be maintained at approximately 25°C. This may be accomplished by admitting air to the centrifuge casing.

9.5 Decant or siphon the supernatant oil as completely as possible into a clean centrifuge tube and allow time for drainage. If the foots layer is liquid, extra care must be taken to remove the oil without disturbing the foots layer. A modified siphon can be used to advantage.

9.6 Add 25 mL of acetone to the precipitate in the first tube and mix until the gummy material is dispersed. Use a wire to loosen them from the tip of the tube if necessary. Dilute to 100 mL with more acetone and shake.

9.7 Prepare fritted glass crucibles as follows: Add 0.3 to 0.6 g of diatomaceous silica to the empty crucible. With experience this can roughly be measured on the tip of a spatula. Slurry with approximately 15 mL of acetone. Remove the acetone by applying a vacuum to the filter. Dry the crucible in an oven at 100 ± 5°C for 1 h or to constant weight (0.01 mg). Cool for 1 h in a desiccator and weigh to the fourth decimal place. Store prepared crucibles in a desiccator until they are to be used.

9.8 Filter the mixture prepared in accordance with 9.6 through a weighed, fritted glass crucible. Use a moderate vacuum and always maintain some acetone in the crucible. Thoroughly wash the centrifuge tube and precipitate on the filter with four, 15-mL portions of acetone. Since oil tends to creep up the sides of the crucible care must be exercised. A wash bottle containing acetone should be used to ensure thorough washing of the centrifuge tube and crucible. After the crucible is exhausted of acetone, dry it at 100°C, cool to room temperature in the desiccator, and weigh to nearest 0.1 mg.

NOTE 2—The fritted glass filters must be cleaned periodically with cleaning solution. It is well to test the filtration rate of each crucible with pure acetone and then to discard any that cannot be cleaned to give satisfactory rates.

9.9 Treat the supernatant oil obtained in 9.5 with a second portion of phosphoric acid in accordance with 9.1 to 9.8.

10. Calculation

10.1 Calculate the percent of gravimetric foots as follows:

$$\text{Gravimetric foots, \%} = 2(A + B) \tag{3}$$

where:

A = weight of sediment from 50 g of original oil on first phosphoric treatment, g, and

[4] Hyflo Super-Cel, produced by Johns-Manville Corp. has been found satisfactory for this purpose.

B = weight of sediment from 50 g of supernatant oil on second phosphoric treatment, g.

11. Report

11.1 Report the results to the second decimal place.

12. Precision

12.1 At the 95 % confidence level, assays run in the samelaboratory should not differ by more than 0.017 %. Assays run in two different laboratories, each of which is the mean of two determinations, should not differ by more than 0.026 %.

13. Keywords

13.1 foots; linseed oil