



Designation: D 1954 – 86 (Reapproved 1995)^{ε1}

Standard Test Method for Foots in Raw Linseed Oil (Volumetric Method)¹

This standard is issued under the fixed designation D 1954; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε¹ NOTE—Unit of measurement statement and Keywords were added editorially in May 1995.

1. Scope

1.1 This test method covers the determination of foots in raw linseed oil.

NOTE 1—Test Method D 1966, is a more reproducible method and can be used in place of Test Method D 1954 upon mutual agreement between the purchaser and the seller.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1966 Test Method for Foots in Raw Linseed Oil (Gravimetric Method)³

E 1 Specification for ASTM Thermometers⁴

3. Terminology

3.1 Definitions:

3.1.1 *foots*—a term that originally was used to describe those solid impurities that precipitate from raw linseed oil during storage and then settle to the bottom or “foot” of a storage tank.

3.2 Definitions of Terms Specific to This Standard:

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 Subcommittee D01.32 on Drying Oils.

Current edition approved March 27, 1986. Published May 1986. Originally published as D 1954 – 61. Last previous edition D 1954 – 62 (1984).

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

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

3.2.1 *foots*—a material insoluble in a mixture of equal parts of acetone and the oil under test, and insoluble in calcium chloride solution, under specific conditions of the test.

4. Summary of Test Method

4.1 The oil to be tested is treated by mixing under defined conditions with an acid-calcium chloride solution and acetone. This mixture is allowed to settle in a graduated tube and the foots content is determined volumetrically.

5. Significance and Use

5.1 This test method provides an empirical measure of the amount of solid impurities present in raw linseed oil. The impurities are usually suspended in the oil. Addition of the reagents stratifies the impurities in a layer that is measured volumetrically. After addition of the reagents, a period of time is provided to allow the impurities to stratify. The test method is empirical since the volume of the impurity stratum will increase over a period of time due to the hydration of the impurities.

5.2 After heating a sample of the oil to 65°C, it is divided into two parts. One part is cooled to about 25°C and the other to 0°C before the test is run. The test results run on the two samples are known as the heated foots and chilled foots respectively.

6. Apparatus

6.1 *Beakers*, 150-mL, made of chemically resistant glass.

6.2 *Thermometer*—An ASTM Partial Immersion Thermometer having a range from – 20 to + 150°C and conforming to the requirements for Thermometer 1C as prescribed in Specification E 1.

6.3 *Sample Bottles*, 60-mL (2-oz).

6.4 *Graduated Cylinders*, 10 and 25-mL.

6.5 *Graduated Tube*—A buret or color comparison tube, having an internal diameter of 10 to 15 mm, and a capacity of not less than 70 mL. The graduations in 0.1-mL should extend at least from 10 to 50 mL above the bottom of the tube.

6.6 *Water Bath*, capable of being maintained at 25 ± 0.5°C.



7. Reagents

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.⁵ 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.

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

7.3 *Acetone*. (**Precaution**—See 8.1)

7.4 *Acid-Calcium Chloride Solution*—Dissolve 600 g of calcium chloride (CaCl_2) or 800 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in a mixture of 80 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) (**Precaution**—see 8.2) and 500 mL of water. Dilute to 1 L and mix. Filter if not clear.

8. Hazards

8.1 *Acetone* is a flammable liquid. Harmful if inhaled, use only with adequate ventilation. Avoid prolonged contact with skin or any contact with the eyes. See supplier's Material Safety Data Sheet.

8.2 *Hydrochloric Acid*—Concentrated hydrochloric acid is corrosive and may cause burns to the skin and eyes; the vapor is irritating to mucous membranes. Avoid contact with skin and eyes. Wash clothing before reuse. See supplier's Material Safety Data Sheet.

9. Preparation of Oil

9.1 Place 75 g of the oil in a 150-mL beaker, heat to 65°C, and maintain at $65 \pm 1^\circ\text{C}$ for 10 min. At the end of the 10-min period, divide it into two equal portions by pouring one half into a clean, dry, 60-mL (2-oz) bottle. Stopper the 2-oz bottle and cool immediately as described in 10.2.

⁵ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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10. Procedure

10.1 *Determination of Heated Foots*—Allow the oil in the beaker (Section 9) to cool to a temperature of 20 to 27°C and maintain at this temperature for 30 min. Measure 25 mL of the prepared oil into a graduate and at the same time measure out 25 mL of acetone and 10 mL of the acid- CaCl_2 solution. All materials should be at 20 to 27°C. Pour the oil into the graduated tube. Rinse the graduate used for oil measurement with the acetone. Transfer the acetone rinses and the acid- CaCl_2 solution into the graduated tube. Stopper the tube. Mix for 1 min by completely inverting and reinverting the tube ten to twelve times. Clamp the tube in a vertical position and allow to stand 24 h at $25 \pm 0.5^\circ\text{C}$. Determine the volume of the stratum lying between the clear CaCl_2 solution and the clear acetone and oil mixture to 0.1 mL.

10.2 *Determination of Chilled Foots*—Place the 60-mL (2-oz) bottle in an ice water bath (0°C) for 2 h. Then place it in a constant-temperature bath at $25 \pm 0.5^\circ\text{C}$ for 30 min. Continue as described in 10.1.

11. Calculation

11.1 Multiply the volume of the intermediate strata (10.1 and 10.2) by four to obtain the percent of heated and chilled foots.

12. Report

12.1 Report the results to the first decimal place.

13. Precision

13.1 The precision of the heated foots and chilled foots test methods has never been formally adopted from a statistical analysis of historical cooperative tests by ASTM members. A 1961 examination of ASTM results published in 1927 shows:

- (1) Heated foots: 66 % coefficient of variation between observers, and
- (2) Chilled foots: 35 % coefficient of variation between observers.

13.2 These very wide coefficients were calculated from single observations by seven observers on each of six samples of oil. Values reported ranged from 0.0 to 0.2 % heated foots and 0.4 to 2.6 chilled foots in the cleanest sample, to 3.3 to 17.2 % heated foots and 9.6 to 20.0 % chilled foots in the dirtiest sample of oil.

14. Keywords

14.1 foots; linseed oil; linseed oil