



Designation: D 1952 – 86 (Reapproved 1995)^{ε1}

Standard Test Method for Quantitative Determination of Break in Drying Oils¹

This standard is issued under the fixed designation D 1952; 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—Keywords were added editorially in May 1995.

1. Scope

1.1 This test method covers the quantitative determination of break in drying oils. This test method is applicable to raw and refined drying or fatty oils that have not been substantially polymerized, oxidized, or chemically modified.

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 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:

E 1 Specification for ASTM Thermometers²

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *break*—in drying oils, that material rendered insoluble when an oil is heated to polymerization or varnish-making temperatures.

4. Summary of Test Method

4.1 The oil under test is treated with hydrochloric acid heated to 290°C, allowed to cool, diluted with carbon tetrachloride, and the resulting insoluble break is separated and weighed.

5. Significance and Use

5.1 This test method provides a quantitative determination of the amount of non-oil material that will become insoluble when the oil is heated. Break is not significant except in oils that are to be heated, as in manufacture of varnishes and alkyd resins. This test method is not applicable to polymerized, oxidized, or chemically modified oils. The break is rendered

insoluble in the oil by the addition of hydrochloric acid and heating to a specified temperature. Since the temperature must be specified, the results are empirical.

6. Apparatus

6.1 *Beaker*, 100-mL capacity, tall-form.

6.2 *Thermometer*—An ASTM Open Flash Thermometer having a range from –6 to +400°C and conforming to the requirements for Thermometer 11C as prescribed in Specification E 1.

6.3 *Filtering Crucible*, porcelain bitumen-type, with top diameter of 44 mm and depth of 25 mm prepared by matting with medium fiber asbestos (**Precaution**—See 8.1), drying to constant weight (within 0.1 mg) at 105°C, and cooling in a desiccator.

7. Reagents and Materials

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.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.

7.2 *Carbon Tetrachloride* (CCl₄) (**Precaution**—See 8.3).

7.3 *Filter Aid*—Diatomaceous silica⁴ dried at 105°C and stored in a desiccator.

7.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl) (**Precaution**—See 8.2).

8. Hazards

8.1 *Asbestos*—Inhalation of asbestos fiber or dust has been found to be a cause of asbestosis and lung cancer. Handle dry

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² *Annual Book of ASTM Standards*, Vol 14.03.

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

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



asbestos in a suitable hood. Prewetting of asbestos with water or solvent can help reduce the concentration of airborne particles. Use of a particulate filter face mask is recommended. See supplier's Material Safety Data Sheet for proper handling procedures.

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.

8.3 *Carbon Tetrachloride* is a very hazardous liquid. It is absorbed by the skin. Its vapor is hazardous through inhalation. It is an irritant to skin and eyes; avoid breathing (TLV-10 PPM). It causes liver and kidney damage and has cumulative effects. Use with adequate ventilation (in a hood) and wear rubber gloves. See supplier's Material Safety Data Sheet.

9. Procedure

9.1 Weigh, to 0.1 g, 25 g of the well-mixed sample into the beaker on an analytical balance. Add 0.15 mL of HCl from a measuring pipet and stir thoroughly with the thermometer. Suspend the thermometer in the center of the mixture so that the bulb is completely immersed in the liquid but not touching the bottom of the beaker. Apply heat (**Warning**, Note 1) so that the temperature rises to 290°C in 3 to 3.5 min. After heating to 290°C, withdraw the flame.

NOTE 1—**Warning:** Do not stir the specimen after heating has begun.

9.2 Cool to room temperature; then add, while stirring, 50 mL of CCl₄ followed by 0.2 g of diatomaceous filter aid weighed to 0.1 mg. Allow to stand for 1 h, stirring at 15-min intervals.

9.3 Filter, with the aid of vacuum, through the porcelain crucible previously prepared and weighed to 0.1 mg. Wash the residue with four 15-mL portions of CCl₄, using a policeman to remove any traces of solids from the beaker, and adding each portion as soon as the previous one has drained.

9.4 Dry the crucible and its contents at 105°C to constant weight (within 0.1 mg), allowing it to cool in a desiccator prior to weighing.

10. Calculation

10.1 Calculate the percent of break, *B*, as follows:

$$B, \% = [(A - C - W)/S] \times 100 \quad (1)$$

where:

A = total weight of dried crucible and its contents, g,

C = filter aid added, g

W = weight of crucible and asbestos mat, g, and

S = specimen weight, g.

11. Report

11.1 Report the results to the second decimal place.

12. Precision and Bias

12.1 *Precision*—Cooperative results indicate that the reproducibility of this test method is approximately 0.02 % relative.

12.2 *Bias*—Bias has not been determined.

13. Keywords

13.1 break; drying oils; drying oils

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