



Designation: D 1951 – 86 (Reapproved 1995)^{e1}

Standard Test Method for Ash in Drying Oils and Fatty Acids¹

This standard is issued under the fixed designation D 1951; 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.

^{e1} NOTE—Unit of measurement statement and Keywords were added editorially in May 1995.

1. Scope

1.1 This test method covers the determination of the residue or ash resulting from ignition, under prescribed conditions, of all natural and synthetic drying oils and their fatty acids. This test method is not applicable to boiled oils.

NOTE 1—Boiled oils do not give reproducible results. Lead driers are partially reduced by the carbon to give metallic lead and oxides of variable composition, and zinc oxide is relatively volatile at the muffle furnace temperature. Ash determinations on boiled oils consequently are empirical and not highly reproducible.

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 4.

2. Significance and Use

2.1 Most natural and processed oils contain small amounts of ash, but the amount is insignificant. Certain, synthetic drying oils may contain residual catalyst or other materials, thus giving larger amounts of ash. Although the ash and metal content of boiled oils may be specified, the trend with new and improved driers is toward the specification of drying time, allowing the manufacturer to obtain this in any way desired.

2.2 In this test method, ash is determined by igniting the specimen under specified conditions and weighing the residue. Due to the volatility or the reduction of certain metal oxides,

some inaccuracies may result. Wet ashing or extraction methods may give more accurate results.

3. Apparatus

3.1 *Crucible*, porcelain or high-silica glass (Note 2), 50-mL capacity.

NOTE 2—Platinum is not recommended. Boiled oils or oils contaminated with driers containing lead may ruin platinum by alloy formation.

3.2 *Electric Muffle Furnace*.

3.3 *Desiccator*, containing an efficient desiccant. Anhydrous calcium sulfate (CaSO_4), phosphorus pentoxide (P_2O_5) (**Warning**—see 4.1) or concentrated sulfuric acid (**Warning**—see 4.2) (H_2SO_4 , sp gr 1.84) are satisfactory.

NOTE 3—Magnesium Perchlorate and Barium Perchlorate are also efficient desiccators and were previously listed in this section. However because of their explosive danger, and the availability of other safer materials, the recommendation for their use has been discontinued.

3.4 *Oil Sample Bottle*, 120-mL (4-oz).

3.5 *Triangle*, Nichrome or clay.

4. Hazards

4.1 *Phosphorus Pentoxide* is a strong oxidizer and reacts violently with water, reducing agents and organic matter. Causes burns. Avoid contact with skin or eyes, or clothing, or inhalation as dust. Refer to supplier's Material Safety Data Sheet.

4.2 *Sulfuric Acid* is corrosive to skin, eyes and mucous membranes in form of liquid, mist or fumes. It causes severe burns. Take care to prevent the contact of the acid with eyes, skin or on clothing. In making dilute solutions, always add the acid to water with care. See supplier's Material Safety Data Sheet.

5. Procedure

5.1 Ignite the crucible in the muffle furnace at 550 to 650°C. Cool slightly, place in a desiccator for 1 h, and weigh to 0.1 mg.

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.

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D 1951

5.2 Fill a 120-mL (4-oz) sample bottle with the sample and weigh to 0.05 g. Pour about 20 g of the sample from the bottle into the crucible supported on a triangle, using care so that no oil runs down the outside of the crucible or bottle.

5.3 Heat gently by moving a flame on the bottom and sides of the crucible until the oil ignites. Reduce the size of the flame until the heat is just sufficient to keep the sample burning. When the first batch of oil has burned out, add about 20 g more of the sample and continue in the same manner until all of the oil in the 20-mL bottle has been added. Reweigh the sample bottle to obtain the total weight of sample used, which will be somewhat over 100 g.

5.4 Continue heating the crucible until the oil is oxidized to a black char and transfer to a muffle furnace. Heat at 550 to 650°C for 1 h. Remove from the furnace, cool slightly, place in a desiccator, and cool to room temperature. Weigh and repeat heating in the furnace to constant weight (within 0.1 mg).

6. Calculation

6.1 Calculate the ash content, A , of the sample as follows:

$$A, \% = (R/S) \times 100$$

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or,

$$A, \text{ ppm} = (R/S) \times 1\,000\,000$$

where:

R = residue (5.4), g, and

S = sample used, g.

7. Precision and Bias

7.1 Precision and bias were not established at the time this test method was written. An effort is being made to obtain the precision and, if obtainable, it will be published in future revisions. This method has been in use for many years, and its usefulness has been well established.

8. Keywords

8.1 ash; drying oils; fatty acids