



Designation: D874 – 07

British Standard 4716



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Standard Test Method for Sulfated Ash from Lubricating Oils and Additives¹

This standard is issued under the fixed designation D874; 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.

1. Scope*

1.1 This test method covers the determination of the sulfated ash from unused lubricating oils containing additives and from additive concentrates used in compounding. These additives usually contain one or more of the following metals: barium, calcium, magnesium, zinc, potassium, sodium, and tin. The elements sulfur, phosphorus, and chlorine can also be present in combined form.

1.2 Application of this test method to sulfated ash levels below 0.02 mass % is restricted to oils containing ashless additives. The lower limit of the test method is 0.005 mass % sulfated ash.

NOTE 1—This test method is not intended for the analysis of used engine oils or oils containing lead. Neither is it recommended for the analysis of nonadditive lubricating oils, for which Test Method D482 can be used.

NOTE 2—There is evidence that magnesium does not react the same as other alkali metals in this test. If magnesium additives are present, the data is interpreted with caution.

NOTE 3—There is evidence that samples containing molybdenum can give low results because molybdenum compounds are not fully recovered at the temperature of ashing.

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

1.4 *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.*

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures. DOI: 10.1520/D0874-07.

2. Referenced Documents

2.1 ASTM Standards:²

D482 Test Method for Ash from Petroleum Products

D1193 Specification for Reagent Water

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

3. Terminology

3.1 Definitions:

3.1.1 *sulfated ash*—the residue remaining after the sample has been carbonized, and the residue subsequently treated with sulfuric acid and heated to constant weight.

4. Summary of Test Method

4.1 The sample is ignited and burned until only ash and carbon remain. After cooling, the residue is treated with sulfuric acid and heated at 775°C until oxidation of carbon is complete. The ash is then cooled, re-treated with sulfuric acid, and heated at 775°C to constant weight.

5. Significance and Use

5.1 The sulfated ash can be used to indicate the concentration of known metal-containing additives in new oils. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulfates and tin (stannic) and zinc to their oxides (Note 5). Sulfur and chlorine do not interfere, but when phosphorus is present with metals, it remains partially or wholly in the sulfated ash as metal phosphates.

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.

*A Summary of Changes section appears at the end of this standard.