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An American National Standard
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Standard Test Method for Sulfated Ash from Lubricating Oils and Additives[1]

This standard is issued under the fixed designation D 874; 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 D 482 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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 482 Test Method for Ash from Petroleum Products
D 1193 Specification for Reagent Water
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 6299 Practice for Applying Statistical Quality Assurance 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.

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[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.

[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.**

NOTE 4—Since zinc sulfate slowly decomposes to its oxide at the ignition temperature specified in the test method, samples containing zinc can give variable results unless the zinc sulfate is completely converted to the oxide.

5.2 Because of above inter-element interferences, experimentally obtained sulfated ash values may differ from sulfated ash values calculated from elemental analysis. The formation of such non-sulfated species is dependent on the temperature of ashing, time ashed, and the composition of metal compounds present in oils. Hence, sulfated ash requirement generally should not be used in product specifications without a clear understanding between a buyer and a seller of the unreliability of an ash value as an indicator of the total metallic compound content.[3]

6. Apparatus

6.1 *Evaporating Dish or Crucible;* 50 to 100-mL for samples containing more than 0.02 mass % sulfated ash, or 120 to 150-mL for samples containing less than 0.02 mass % sulfated ash, and made of porcelain, fused silica, or platinum. (**Warning**—Do not use platinum when the sample is likely to contain elements such as phosphorus, which attack platinum under the conditions of the test.)

NOTE 5—For best results on samples containing less than 0.1 mass % sulfated ash, platinum dishes are used. The precision values shown in Section 13 for this type of sample were so obtained.

6.2 *Electric Muffle Furnace*—The furnace shall be capable of maintaining a temperature of 775 ± 25°C and preferably have apertures at the front and rear to allow a slow natural draft of air to pass through the furnace.

6.3 *Balance*, capable of weighing to 0.1 mg.

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, or to other recognized standards for reagent chemicals.[4] 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 defined by Types II or III of Specification D 1193.

7.3 *Low-Ash Mineral Oil*—White oil having a sulfated ash lower than the limit capable of being determined by this test method.

NOTE 6—Determine the sulfated ash of this oil by the procedure given in 9.1-9.11 below using 100 g of white oil weighed to the nearest 0.5 g in a 120 to 150-mL platinum dish. Deduct the sulfuric acid blank as described in 9.11.

7.4 *Sulfuric Acid (relative density 1.84)*—Concentrated sulfuric acid (H_2SO_4). (**Warning**—Poison. Corrosive. Strong oxidizer.)

7.5 *Sulfuric Acid (1 + 1)*—Prepare by slowly adding 1 volume of concentrated sulfuric acid (relative density 1.84) to 1 volume of water with vigorous stirring. (**Warning**—Mixing this acid into water generates considerable heat. When necessary, cool the solution before adding more acid. Do not allow the solution to boil.)

7.6 *Propan-2-ol*. (**Warning**—Flammable, can be explosive when evaporated to or near dryness.)

7.7 *Toluene*. (**Warning**—Flammable, toxic.)

7.8 *Quality Control (QC) Samples*, preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 12.

8. Sampling

8.1 Obtain samples in accordance with the instructions in Practice D 4057 or D 4177. The sample shall be thoroughly mixed before removal of the laboratory test portion.

9. Procedure

9.1 Select the size of the evaporating dish or crucible according to the quantity of sample necessary (see 9.3).

9.2 Heat the evaporating dish or crucible that is used for the test at 775 ± 25°C for a minimum of 10 min. Cool to room temperature in a suitable container and weigh to the nearest 0.1 mg.

NOTE 7—The container in which the dish is cooled does not contain a desiccating agent.

9.3 Weigh into the dish a quantity of sample given as follows:

$$W = 10/a \qquad (1)$$

where:

W = mass of test specimen, g, and

a = expected sulfated ash, mass %.

However, do not take a quantity in excess of 80 g. In the case of lubricating oil additives yielding a sulfated ash of 2 mass % or more, dilute the weighed sample in the dish with approximately 10 times its weight of low-ash mineral oil.

NOTE 8—If the amount of sulfated ash found differs from the expected amount by more than a factor of two, repeat the analysis with a different weight of sample calculated from the first analysis.

9.4 Heat the dish or crucible and sample carefully until the contents can be ignited with a flame. Maintain at such a temperature that the sample continues to burn at a uniform and moderate rate. When burning ceases, continue to heat gently until no further smoke or fumes are evolved.

9.4.1 If the sample contains sufficient moisture to cause foaming and loss of material from the dish, discard the sample and to an additional sample add 1 to 2 mL of 99 % propan-2-ol

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[3] Further discussion of these interferences can be found in Nadkarni, R. A., Ledesma, R. R., and Via, G. H., "Sulfated Ash TM: Limitations of Reliability and Reproducibility," SAE Technical Paper No. 952548, available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001, U.S.A.

[4] *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.