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Petroleum products — Determination of sulfated ash in lubricating oils and additives

Produits pétroliers — Détermination des cendres sulfatées dans les huiles lubrifiantes et dans les additifs

Reference number
ISO 3987:2010(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3987 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 3987:1994), which has been technically revised.

Petroleum products — Determination of sulfated ash in lubricating oils and additives

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard describes a procedure for the determination of the mass percentage of 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.

Application of this procedure to sulfated ash levels below 0,02 % (*m/m*) is restricted to oils containing ashless additives. The lower limit of applicability of the procedure is 0,005 % (*m/m*) sulfated ash.

NOTE 1 For the purposes of this International Standard, the terms % (*m/m*) and % (*V/V*) are used to represent the mass fraction and volume fraction of a material, respectively.

This International Standard is not intended for the analysis of used engine oils containing lead, nor is it recommended for the analysis of non-additive lubricating oils, for which ISO 6245 [1] is suitable.

NOTE 2 There is evidence that magnesium does not react in the same manner as alkali metals in this procedure. If magnesium additives are present, it is advisable to interpret the data with caution.

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

The sulfated ash may be used to indicate the concentration of known metal-containing additives in new lubricating oils. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulfates, and tin (IV) and zinc to their oxides.

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

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.

NOTE 5 Fatty acid methyl esters (FAME) conforming to EN 14213 [2] and EN 14214 [3], when tested using this International Standard, were shown to meet its precision.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
sulfated ash
residue remaining after the lubricating oil sample has been carbonized, and the residue subsequently treated with sulfuric acid and heated to constant mass

4 Principle

The sample of unused lubricating oil 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, retreated with sulfuric acid, and heated at 775 °C to constant mass. The mass percentage of sulfated ash obtained is then calculated.

5 Reagents

For the analysis described in this International Standard, use only reagents of recognized analytical reagent grade and water complying with the requirements of grade 3 of ISO 3696:1987.

5.1 Low-ash mineral oil, white oil having a sulfated ash content (determined as follows) lower than the limit capable of being determined by this International Standard.

Determine the sulfated ash of the oil by the procedure given in Clause 8, but using 100 g of white oil, weighed to the nearest 0,5 g, in a 120 ml to 150 ml platinum dish. Deduct the sulfuric acid blank as described in 8.11.

5.2 Sulfuric acid (H_2SO_4), concentrated, 98 % minimum purity.

CAUTION — Sulfuric acid is highly corrosive, a strong oxidizer, and has a high heat of hydration. Protective clothing, including gloves and face mask, should be worn during operations involving this acid.

5.3 Sulfuric acid (1 + 1), prepared by slowly adding one volume of the concentrated acid (5.2) to one volume of water.

CAUTION — Mixing sulfuric acid into water generates considerable heat. When necessary, cool the solution before adding more acid. Do not allow the solution to boil. Never add the water to the acid.

5.4 Propan-2-ol, 99 % minimum purity.

CAUTION — Propan-2-ol is flammable, and can be explosive when evaporated to dryness.

5.5 **Toluene**, 99 % minimum purity.

CAUTION — Toluene is flammable and toxic.

6 Apparatus

6.1 **Evaporating dish or crucible**, made of porcelain, fused silica or platinum, of 50 ml to 100 ml capacity. For samples yielding less than 0,2 % (m/m) sulfated ash, use a platinum evaporating dish or crucible of 120 ml to 150 ml capacity. Do not use a platinum vessel if the sample is known to contain elements, such as phosphorus, which are injurious to platinum.

6.2 **Electric muffle furnace**, capable of maintaining a temperature of 775 °C ± 25 °C and preferably having apertures at the front and rear to allow a slow natural draught of air to pass through the furnace.

6.3 **Balance**, capable of weighing to 0,1 mg.

6.4 **Cooling container**, without desiccant.

6.5 **Filter paper**, 0,01 % (m/m) ash maximum.

7 Samples and sampling

Samples shall be taken in accordance with ISO 3170, ISO 3171 or an equivalent national standard. The sample shall be thoroughly mixed before removal of the laboratory test portion.

8 Procedure

8.1 Select an evaporating dish or crucible (6.1) of suitable size according to the quantity of sample necessary (see 8.3).

8.2 Heat the evaporating dish or crucible in the furnace (6.2) at 775 °C for at least 10 min. Cool to room temperature in a suitable container (6.4) and weigh to the nearest 0,1 mg.

8.3 Weigh, to the nearest 0,1 mg, into the dish, a quantity, m_1, of the sample to be tested, given by Equation (1) as follows.

$$m_1 = \frac{10}{m_0} \tag{1}$$

where

m_0 is the expected sulfated ash, expressed as a percentage mass fraction;

m_1 is the mass of test portion, expressed in grams.

Do not take a test portion in excess of 80 g. In the case of lubricating oil additives yielding a sulfated ash of 2,00 % (m/m) or more, dilute the weighed test portion with approximately ten times its mass of low-ash mineral oil (5.1).

If the amount of sulfated ash found differs from the expected amount by more than a factor of two, repeat the analysis using a mass of test portion which takes into account the result of the first analysis.

8.4 Heat the dish or crucible and sample carefully over a gas burner 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.

If the test portion contains sufficient water to cause foaming and loss of material from the dish, discard the test portion, and, to an additional test portion, add 1 ml to 2 ml of propan-2-ol (5.4) before heating. If this is not satisfactory, add 10 ml of a mixture of equal volumes of toluene (5.5) and propan-2-ol (5.4) and mix thoroughly. Place several strips of ashless filter paper (6.5) in the mixture and heat; when the paper begins to burn, the greater part of the water will have been removed.

8.5 Allow the dish to cool to room temperature, then completely moisten the residue by the addition, drop by drop, of the concentrated sulfuric acid (5.2). Carefully heat the dish at a low temperature on a hot plate or over a gas burner, avoiding spattering, and continue heating until fumes are no longer evolved.

8.6 Place the dish in the furnace (6.2) controlled at 775 °C and continue heating at that temperature until oxidation of the carbon is complete or almost complete.

8.7 Allow the dish to cool to room temperature. Add three drops of water and ten drops of the sulfuric acid solution (5.3). Move the dish so as to moisten the entire residue. Heat the dish again as specified in 8.5.

8.8 Replace the dish in the furnace (6.2) controlled at 775 °C and maintain at that temperature for 30 min. Cool the dish to room temperature in the cooling container (6.4).

If zinc dialkyl or alkaryl dithiophosphates and blends containing these additives give a residue which is partially black at this stage, repeat the operations specified in 8.7 and 8.8 until a white residue is obtained.

8.9 Using the balance (6.3), weigh the dish and residue to the nearest 0,1 mg.

8.10 Repeat the procedure given in 8.8 and 8.9 until two successive weighings differ by no more than 1,0 mg.

8.11 For samples expected to contain 0,02 % (*m*/*m*) or less of sulfated ash, determine a sulfuric acid blank by adding 1 ml of the concentrated sulfuric acid (5.2) to a tared platinum dish or crucible, heating until fumes are no longer evolved, and then heating in the furnace (6.2) at 775 °C for 30 min.

Cool the dish or crucible to room temperature in the cooling container (6.4) and weigh to the nearest 0,1 mg. If any ash is found in the sulfuric acid, an adjustment to the mass of sulfated ash obtained is made by subtracting the mass of ash contributed by the sulfuric acid from the total mass of sulfated ash for the test portion.

Determine the mass of ash contributed by the sulfuric acid by multiplying the mass of ash found for the 1 ml blank by the total volume of sulfuric acid used. Use the corrected mass as the value, m_2, in calculating the sulfated ash result (see Clause 9).

9 Calculation

Calculate the sulfated ash, A, as a percentage (mass fraction) of the original sample in accordance with Equation (2).

$$A = 100 \frac{m_2}{m_1} \quad (2)$$

where

m_1 is the mass of test portion, expressed in grams;

m_2 is the mass of sulfated ash, expressed in grams.

10 Expression of results

Report the result to the nearest 0,001 % (*m*/*m*) for samples with sulfated ash contents below 0,02 % (*m*/*m*), and to the nearest 0,01 % (*m*/*m*) for those having higher levels.

11 Precision

The precision of this International Standard was established by statistical examination of interlaboratory results on test programmes that were run in 1975 and 1981.

Table 1 gives examples of specimen precision values using the formulae in 11.1 and 11.2.

Table 1 — Specimen precision values

Sulfated ash % (*m*/*m*)	**Precision** % (*m*/*m*)	
	Repeatability *r*	**Reproducibility** *R*
0,005	0,000 5	0,002 1
0,010	0,000 9	0,003 8
0,050	0,003 7	0,014 8
0,100	0,006 6	0,026 7
0,50	0,036	0,084
1,00	0,060	0,142
5,00	0,201	0,475
10,00	0,337	0,799
20,00	0,567	1,343
25,00	0,671	1,588

11.1 Repeatability, *r*

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, and in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

$$r = 0{,}047X^{0{,}85} \quad \text{for } 0{,}005\ \%\ (m/m) \leqslant A \leqslant 0{,}100\ \%\ (m/m)$$

$$r = 0{,}060X^{0{,}75} \quad \text{for } 0{,}11\ \%\ (m/m) \leqslant A \leqslant 25{,}0\ \%\ (m/m)$$

where X is the average of the results being compared, expressed as a percentage (mass fraction).

11.2 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would, in the long run, and in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

$$R = 0{,}047X^{0{,}85} \quad \text{for } 0{,}005\ \%\ (m/m) \leqslant A \leqslant 0{,}100\ \%\ (m/m)$$

$$R = 0{,}060X^{0{,}75} \quad \text{for } 0{,}11\ \%\ (m/m) \leqslant A \leqslant 25{,}0\ \%\ (m/m)$$

where X is the average of the results being compared, expressed as a percentage (mass fraction).