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Petroleum products — Determination of sulfated ash in lubricating oils and additives and fatty acid methyl esters

Produits pétroliers — Détermination des cendres sulfatées dans les huiles lubrifiantes et additifs et esters méthyliques d'acides gras

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

This fourth edition cancels and replaces the third edition (ISO 3987:2010), which has been technically revised. It also incorporates the Technical Corrigendum ISO 3987:2010/Cor 1:2011.

The main changes are as follows:

- the addition of fatty acid methyl esters (FAME) in the scope and title;
- modification of the precision paragraphs based on ISO 4259-1:2017/Amd 1:2019.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Petroleum products — Determination of sulfated ash in lubricating oils and additives and fatty acid methyl esters

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

1 Scope

This document 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. This document is also applicable to fatty acid methyl esters (FAME).

This test method is applicable to products having sulphated ash contents in the range mass fraction of 0,005 % to 25,00 %. Application of this procedure to sulfated ash levels below a mass fraction of 0,02 % is restricted to oil products containing ashless additives.

This document is not intended for the analysis of used engine oils containing lead, nor for the analysis of non-additive lubricating oils, for which ISO 6245 is suitable.

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *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.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

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

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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 document, use only reagents of recognized analytical reagent grade and water complying with the requirements of grade 3 specified in ISO 3696.

5.1 Low ash mineral oil, white oil having a sulfated ash content lower than the limit that can be determined by this document.

The sulfated ash of the mineral oil shall be determined by the procedure given in Clause 8, but use 100 g of mineral 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, 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.

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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 a mass fraction of 0,2 % 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, a mass fraction of 0,01 % ash maximum.

7 Samples and sampling

Samples shall be taken in accordance with ISO 3170 or ISO 3171. 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 the 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 Formula (1) as follows:

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

where

m_0 is the expected sulfated ash, expressed as a mass fraction in per cent;

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

Do not take a test portion in excess of 80 g. If the lubricating oil additives yield a sulfated ash with a mass fraction of 2,00 % or more, dilute the weighed test portion with approximately 10 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 so that the sample continues to burn at a uniform and moderate rate. When the 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 adding, drop by drop, 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 10 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.

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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 a mass fraction of 0,02 % 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, to calculate the sulfated ash result (see Clause 9).

For samples containing metals and additives, more details are given in Annex A.

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9 Calculation

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

$$A = 100 \frac{m_2}{m_1} \tag{2}$$

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 mass fraction of 0,001 % for samples with sulfated ash contents equal and below the mass fraction of 0,02 %, and to the nearest mass fraction of 0,01 % for those with higher levels.

11 Precision

11.1 General

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

Fatty acid methyl esters conforming to EN 14213[1] and EN 14214, when tested using this document, were shown to meet its precision. Details are available in the interlaboratory test report.[5]

Table 1 gives examples of specimen precision values using Formula (3) to (6).

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[1] Withdrawn.

Table 1 — Specimen precision values

Sulfated ash %	Precision %	
	Repeatability r	Reproducibility R
0,005	0,000 5	0,002 1
0,010	0,000 9	0,003 8
0,05	0,003 7	0,014 8
0,10	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

11.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using Formula (3), for a mass fraction of 0,005 % $\leqslant A \leqslant$ mass fraction of 0,10 %:

$$r = 0{,}047X^{0{,}85} \qquad (3)$$

and Formula (4) for a mass fraction of 0,10 % $< A \leqslant$ mass fraction of 25,00 %:

$$r = 0{,}060X^{0{,}75} \qquad (4)$$

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

11.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using Formula (5) for a mass fraction of 0,005 % $\leqslant A \leqslant$ mass fraction of 0,10 %:

$$R = 0{,}189X^{0{,}85} \qquad (5)$$

and Formula (6) for a mass fraction of 0,10 % $< A \leqslant$ mass fraction of 25,00 %:

$$R = 0{,}142X^{0{,}75} \qquad (6)$$

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

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12 Test report

The test report shall contain at least the following information:

a) reference to this document, i.e. ISO 3987:2024;

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 10);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) any unusual features observed;

f) the date of the test.

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Annex A
(informative)

Information on the potential presence of some metals and additives

This annex gives information about this procedure when some metals and additives are present in the sample.

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.

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.

Since zinc sulfate slowly decomposes to its oxide at the ignition temperature specified in this procedure, samples containing zinc can 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.

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