

# International Standard

## ISO 3987

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

Fourth edition 2024-09

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

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### ISO 3987:2024(en)

### 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.

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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

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:

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- 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 <u>www.iso.org/members.html</u>.

# 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.

### 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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

### 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 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 <u>Clause 8</u>, 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 <u>8.11</u>.

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.

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**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 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.