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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*



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## Foreword

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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.

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# 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 <sup>[1]</sup> 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 <sup>[2]</sup> and EN 14214 <sup>[3]</sup>, 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<sub>2</sub>SO<sub>4</sub>)**, 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.**