# INTERNATIONAL STANDARD

# ISO 6245

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# Petroleum products — Determination of ash

*Produits pétroliers — Détermination de la teneur en cendres*

Reference number
ISO 6245:2001(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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6245 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 6245:1993), which has been technically revised.

# Petroleum products — Determination of ash

**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 specifies a method for the determination of the ash content of petroleum products, such as distillate and residual fuel oils, crude petroleum, lubricating oils, waxes and other petroleum products, in which any ash-forming constituents present are normally considered to be undesirable impurities or contaminants. Ash can result from oil-soluble or water-soluble metallic compounds or from extraneous solids such as dirt and rust.

The method is applicable to products having ash contents in the range 0,001 % ($m/m$) to 0,180 % ($m/m$), but does not apply to products which contain ash-forming additives, including certain phosphorus compounds. The analysis of unused or used lubricating oils containing additives, used engine crankcase oils, lubricating oils containing lead, or certain non-hydrocarbon diesel fuels, should be determined using ISO 3987[1], which includes a step to produce sulfated ash of higher melting point.

NOTE 1 For the purposes of this International Standard, the term "% ($m/m$)" is used to represent the mass fraction of a material.

NOTE 2 With certain types of samples, the test results may not account quantitatively for all the metal compounds capable of forming an ash. Some unused lubricating oils and distillate fuel oils contain metal inorganic additives of low molecular mass (e.g. sodium, potassium), where the metal oxide may sublime below the temperatures specified in this International Standard.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*

ISO 5272:1979, *Toluene for industrial use — Specifications*

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[1] ISO 3987:1994, *Petroleum products — Lubricating oils and additives — Determination of sulfated ash*.

## 3 Principle

A test portion contained in a suitable vessel is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 775 °C, allowed to cool and weighed.

## 4 Reagents and materials

### 4.1 Propan-2-ol

Minimum purity 99 %.

### 4.2 Methylbenzene (toluene)

Conforming to the requirements of ISO 5272, grade B.

### 4.3 Filter paper

Ashless.

## 5 Apparatus

### 5.1 Evaporating vessel

A dish or crucible of capacity 50 ml to 150 ml, made of platinum, silica or porcelain.

NOTE Alumina vessels may be used where appropriate. The use of vessels that are not made of platinum may contaminate the ash.

### 5.2 Muffle furnace

An electric furnace capable of maintaining a temperature of 775 °C ± 25 °C. A furnace with manual temperature control and suitable apertures at the front and rear to permit a slow natural draught of air, or a programmable furnace controlling both temperature and air flow, is suitable.

### 5.3 Cooling vessel

A closed vessel of adequate capacity, not containing a desiccating agent.

### 5.4 Analytical balance

Capable of weighing to the nearest 0,1 mg.

### 5.5 Mixer

Of non-aerating, high-speed shear type.

### 5.6 Burner

A Meeker or Bunsen gas burner, or an equivalent.

## 6 Sampling

Unless otherwise specified, samples shall be obtained in accordance with the procedures described in ISO 3170 or ISO 3171.

## 7 Sample preparation

### 7.1 General

Before transferring the test portion to be ashed to the evaporating vessel (5.1), ensure that the sample in its container is homogeneous by one of the procedures described in 7.2 or 7.3.

### 7.2 Mobile samples

Mix mobile samples to distribute particulate material, such as catalyst fines, rust or adventitious particulates, throughout the sample evenly. Increasing mixing intensity is used until visual homogeneity is observed. Mixing by manual or mechanical shaking for 10 min is the lowest intensity mixing procedure, followed by repeated and longer mixing times. When it is evident that repeated or lengthy shaking is not achieving homogeneity, use the mixer (5.5) for the minimum time to make the sample homogeneous. If the sample cannot be satisfactorily homogenized, discard and obtain a new sample.

### 7.3 Viscous or solid samples

Heat samples of viscous, semi-solid and solid materials at room temperature until they are sufficiently mobile to mix by the procedures given in 7.2 (see the note below). Take great care if the heating required is likely to be above 90 °C, as any water in the sample may boil causing spattering or foaming. Wear appropriate safety goggles and gloves. Use an oven or hot-water bath to heat the sample container containing the sample. As a first mixing procedure, stirring with a rod that reaches the bottom of the container is the lowest intensity first mixing step.

NOTE A minimum temperature approximately 5 °C above the pour point of the material is normally required to achieve adequate mobility for mixing.

## 8 Procedure

**8.1** Select an evaporating vessel (5.1) of sufficient capacity to accommodate the test portion size according to the expected ash content (see 8.3).

**8.2** Heat the evaporating vessel (5.1) in the muffle furnace (5.2) at 700 °C to 800 °C for at least 10 min. Allow to cool to room temperature in the cooling vessel (5.3), and weigh to the nearest 0,1 mg. Repeat the heating and weighing operations until the results of consecutive weighings do not differ by more than 0,5 mg.

Perform all weighings of the evaporating vessel as soon as it has cooled to ambient temperature in the cooling vessel, or at a constant time interval after removal from the furnace.

**8.3** Select the size of the test portion, up to a maximum of 100 g, from Table 1, to yield up to 20 mg of ash. Weigh the test portion into the evaporation vessel. For the test portion masses which require more than one firing of the vessel in order to achieve sufficient ash for measurement, obtain the mass from the difference between the initial and final masses, to the nearest 0,1 mg, of a suitable container containing homogenized sample.

**8.4** Heat the evaporating vessel and test portion until the contents can be ignited with a flame applied externally near to the surface of the liquid. If a platinum vessel is used, do not allow it to come into contact with the reducing part of the gas flame, as a loss in mass will occur. Maintain a temperature such that the test portion continues to burn at a uniform and moderate rate, leaving only ash and carbon when the burning ceases.

**8.5** Do not leave burning samples unattended. If the test portion contains sufficient moisture to cause foaming or spattering and loss of material, discontinue the test. To a second test portion, add 1 ml to 2 ml of propan-2-ol (4.1) before heating. If this is still not satisfactory, to a third test portion add 10 ml of an equivolume mixture of toluene (4.2) and propan-2-ol (4.1) and mix thoroughly. Place several strips of filter paper (4.3) in the mixture and heat. When the paper begins to burn, the greater part of water will have been removed. Alternatively, add the toluene/propan-2-ol mixture to the test portion in 2 ml to 3 ml increments, heating between additions until the water has been removed.

**Table 1 — Test portion mass**

| **Expected ash** % ($m/m$) | **Test portion mass** g | **Mass of ash** mg |
|---|---|---|
| 0,18 | 10 | 20 |
| 0,10 | 20 | 20 |
| 0,05 | 40 | 20 |
| 0,04 | 50 | 20 |
| 0,02 | 100 | 20 |
| 0,01 | 100 | 10 |
| 0,001 | 100 | 1 |

**8.6** Some samples will require extra heating after the burning has ceased to reduce the residue to carbon and ash only. This occurs when heavy samples, such as residual fuels, form a crust over incompletely burned material. Break the crust with a glass rod, and return any material adhering to the rod to the evaporating vessel by wiping with a strip of filter paper (4.3). Add the filter paper to the crucible. Continue burning the test portion until only carbon and ash remain.

**8.7** The operator shall exercise considerable care if the heavier material in the evaporating vessel exhibits a tendency to foam. Neither the test portion nor the evaporating vessel shall be heated to a red-hot appearance, nor shall the flame reach a height greater than the rim of the dish. Both of these actions lead to superheating of the test portion residue and significant loss of ash.

**8.8** Heat the residue in the muffle furnace (5.2) at 775 °C ± 25 °C until carbonaceous material has disappeared. Allow the evaporating vessel to cool to ambient temperature in the cooling vessel (5.3), and weigh to the nearest 0,1 mg.

**8.9** Reheat the evaporating vessel at 775 °C ± 25 °C for 20 min to 30 min, allow to cool in the cooling vessel to ambient temperature, and weigh to the nearest 0,1 mg. Repeat the heating and weighing operations until the results of consecutive weighings do not differ by more than 0,5 mg.

## 9 Calculation

Calculate the ash content, $A$, as a percentage by mass of the test portion, using the following equation.

$$A = \frac{m_1}{m_0} \times 100\ \%$$

where

$m_1$ is the mass of the ash, expressed in grams;

$m_0$ is the mass of the test portion, expressed in grams.