INTERNATIONAL STANDARD

ISO 6245

Second edition
1993-07-01

Petroleum products — Determination of ash

Produits pétroliers — Détermination des cendres

Reference number
ISO 6245:1993(E)

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.

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.

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

This second edition cancels and replaces the first edition (ISO 6245:1982), of which it constitutes a technical revision.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Petroleum products — Determination of ash

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this 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 in the range 0,001 % (*m/m*) to 0,180 % (*m/m*) from distillate and residual fuel oils, crude oils, lubricating oils, waxes and other petroleum products, in which any ash-forming materials present are normally considered to be undesirable impurities or contaminants. The method is limited to petroleum products which are free from ash-forming additives.

NOTES

1 With certain types of sample, the test results may not account quantitatively for all the metal compounds capable of forming an ash. This is particularly true in the case of distillate oils, for which it is necessary to use a special procedure if it is desired to determine the metal compounds.

2 This method is not intended for the analysis of unused lubricating oils containing additives; for such samples, use ISO 3987:1980, *Petroleum products — Lubricating oils and additives — Determination of sulphated ash*. Neither is it intended for the analysis of lubricating oils containing lead nor for used engine crankcase oils.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

Members of IEC and ISO maintain registers of currently valid International Standards.

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

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

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

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, cooled and weighed.

4 Apparatus

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

4.2 Electric muffle furnace, capable of maintaining a temperature of 775 °C ± 25 °C and preferably having suitable apertures at the front and rear permitting the passage of a slow natural draught of air.

4.3 Cooling container, not containing a desiccating agent.

4.4 Homogenizer, non-aerating, high-speed shear type.

4.5 Balance, single or double pan, sensitivity 0,1 mg.

5 Reagents and materials

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

5.2 Methylbenzene (toluene), conforming to ISO 5272, grade B.

5.3 Filter paper, ashless.

6 Sampling

Samples shall be obtained by ISO 3170, ISO 3171 or an equivalent national standard.

7 Sample preparation

7.1 Before transferring the portion of the sample to be ashed to the evaporating dish or crucible (4.1), take particular care to ensure that the test portion taken is truly representative of the larger sample. Vigorous shaking or the use of the homogenizer (4.4) is necessary.

7.2 Solid/semi-solid samples shall be heated to 5 °C above their pour point before homogenization.

8 Procedure

8.1 Select an evaporating dish or crucible (4.1) of suitable size according to the quantity of sample necessary (see 8.3).

8.2 Heat the evaporating dish or crucible (4.1) in the furnace (4.2) at 700 °C to 800 °C for 10 minutes or more. Cool to room temperature in the cooling container (4.3), 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.

All weighings of the crucible or dish should be performed as soon as it has cooled. If it is necessary for the dish to remain in the cooling container for a longer period, then all subsequent weighings shall be made after allowing the dish and its contents to remain in the container for the same length of time.

8.3 Weigh into the dish or crucible (4.1), to the nearest 0,1 mg, a test portion of sufficient mass (up to a maximum of 100 g) to give up to 20 mg of ash.

NOTE 3 The mass of test portion taken will depend upon the ash level of the material.

For test portion masses which require more than one filling of the dish, obtain the mass from the difference between the initial and final masses (to the nearest 0,1 mg) of a suitable test portion container. Heat the dish or crucible and test portion until the contents can be ignited with a flame. If a platinum vessel is used, do not allow it to come in contact with the reducing part of the Bunsen flame as a loss of mass will occur. Maintain at 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.

If the test portion contains sufficient moisture to cause foaming and loss of material, discard the test portion. To a second test portion, add 1 ml to 2 ml of propan-2-ol (5.1) before heating. If this is not satisfactory, add 10 ml of an equivolume mixture of toluene (5.2) and propan-2-ol (5.1) and mix thoroughly. Place several strips of ashless filter paper (5.3) in the mixture and heat; when the paper begins to burn, the greater part of the water will have been removed. Alternatively, add toluene/propan-2-ol mixture 2 ml to 3 ml at a time, heating between additions until the water has been removed.

8.4 Heat the residue in the muffle furnace (4.2) at 775 °C ± 25 °C until carbonaceous material has disappeared. Cool the dish to room temperature in the cooling container (4.3), and weigh to the nearest 0,1 mg.

8.5 Reheat the dish at 775 °C ± 25 °C for 20 min to 30 min, cool in the cooling container (4.3), 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.

9 Expression of results

Calculate the amount of ash A as a percentage by mass of the test portion from the formula:

$$A = 100\left(\frac{m_1}{m_0}\right)$$

where

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

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

Report the result to the nearest 0,001 % (*m/m*).

10 Precision

The precision of the method, as obtained by statistical examination of interlaboratory test results, is as follows:

10.1 Repeatability

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20:

Ash, % (*m/m*)	Repeatability, % (*m/m*)
0,001 to 0,079	0,003
0,080 to 0,180	0,007

10.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20:

Ash, % (*m/m*)	Reproducibility, % (*m/m*)
0,001 to 0,079	0,005
0,080 to 0,180	0,024

11 Test report

The test report shall contain at least the following information:

a) all details necessary for complete identification of the product tested;

b) a reference to this International Standard;

c) the result of the test (see clause 9);

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

e) the date of the test.

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UDC 665.7:543.822

Descriptors: petroleum products, crude oil, liquid fuels, lubricating oils, oil waxes, tests, ash determination.

Price based on 3 pages