International Standard 6245

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Petroleum products — Determination of ash

Produits pétroliers — Détermination des cendres

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6245 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in July 1980.

It has been approved by the member bodies of the following countries :

Australia	Hungary	Portugal
Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Ireland	Spain
Canada	Israel	Sweden
Chile	Italy	Switzerland
China	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	Norway	USA
France	Peru	USSR
Germany, F.R.	Poland	Venezuela

No member body expressed disapproval of the document.

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Petroleum products — Determination of ash

1 Scope and field of application

This International Standard specifies a method for the determination of the ash in the range 0,001 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 added ash-forming additives, including certain phosphorus compounds.

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, *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 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.

3 Apparatus

3.1 Evaporating dish or **crucible,** of capacity 90 to 120 ml, of platinum, silica or porcelain.

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

3.3 Cooling container, not containing a desiccating agent.

4 Sampling

Take samples in accordance with established practice. Before transferring the portion of the sample to be ashed to the evaporating dish or crucible, take particular care to ensure that the test portion taken is truly representative of the larger sample. Vigorous shaking may be necessary.

5 Procedure

5.1 Heat the evaporating dish or crucible (3.1) at 700 to 800 °C for 10 min or more. Cool to room temperature in the cooling container (3.3), and weigh to the nearest 0,1 mg.

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

5.2 The mass of test portion to be taken will depend upon the ash level of the material. Weigh into the dish or crucible (3.1), to the nearest 0,1 %, a test portion of sufficient mass (up to a maximum of 100 g) to give up to 20 mg of ash. 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 of a suitable test portion container. Heat the dish or crucible and test portion until the contents can be ignited with a flame. 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.

NOTE — 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 to 2 ml of 99 % propan-2-ol before heating. If this is not satisfactory, add 10 ml of an equivolume mixture of toluene and propan-2-ol and mix thoroughly. Place several strips of ashless filter paper in the mixture and heat; when the paper begins to burn, the greater part of the water will have been removed.

5.3 Heat the residue in the muffle furnace (3.2) at 775 ± 25 °C until all carbonaceous material has disappeared. Cool the dish to room temperature in the cooling container, and weigh to the nearest 0,1 mg.

5.4 Reheat the dish at 775 ± 25 °C for 20 to 30 min, cool in the cooling container, and reweigh. Repeat the heating and weighing operations until the results of consecutive weighings do not differ by more than 0,5 mg.

6 Expression of results

6.1 Method of calculation

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

$$\frac{m_1}{m_0} \times 100$$

where

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

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

6.2 Precision

See the annex.

7 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard or to a national standard;

c) the mass of the test portion taken;

d) the result of the test to two significant figures as the ash, stating the mass of sample taken (see 6.1);

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

f) the date of the test.

Annex

Precision of the method

(Does not form an integral part of the Standard.)

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

A.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, %	Repeatability
0,001 to 0,079	0,003
0,080 to 0,180	0,007

A.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories 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, %	Reproducibility
0,001 to 0,079	0,005
0,080 to 0,180	0,024