SLOVENSKI STANDARD
SIST EN 7:1998

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[Not translated]

Determination of ash from petroleum products

Bestimmung der Asche von Mineralölerzeugnissen

Détermination des cendres dans les produits pétroliers

Ta slovenski standard je istoveten z: **EN 7:1974**

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75.080	Naftni proizvodi na splošno	Petroleum products in general

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English version

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Détermination des cendres dans les produits pétroliers

Bestimmung der Asche von Mineralölerzeugnissen

This standard was accepted by CEN as a European Standard on 31 October 1974, whereby the members committed to accord to it the status of a national standard must do so without change. The details of the conditions under which members are so committed are given in the CEN constitution.

Up-to-date lists of the national standards organizations which have adopted this European Standard, and the relevant bibliographical references, may be obtained on application to the CEN Central Secretariat or to the sales offices of any CEN member.

This European Standard exists in three versions (French, English and German) recognized by CEN as equivalent. National versions in other languages rank as translations, and in case of doubt should be checked against one of the recognized versions (French, English, German).

CEN members are the national standards organizations of Austria, Belgium, Denmark, Finland, France, Germany, Ireland, Italy, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: 5 Boulevard de l'Empereur, B—1000 Bruxelles

Brief history

This European Standard was drawn up by CEN/WG 19 'Methods of Test for Petroleum Products', the secretariat of which is held by the British Standards Institution.

This project was accepted into the work programme of WG 19 in February 1965 with the intention of preparing a Unification Document based on ASTM D 482 : IP 4. In April 1969 the working group accepted a request from the Co-ordinating European Council for the Development of Performance Tests for Fuels and Engine Lubricants, for the provision of a series of test methods for use in the specification of European reference fuels and it was agreed that these methods should be prepared in the form of European Standards.

This European Standard is technically identical with ASTM D 482–63 : IP 4/65 except for a restriction of the scope to ash levels between 0.01 % and 0.20 % (*m/m*).

It was adopted by CEN on 31 October 1974 on the strength of its acceptance by the following member countries:

Belgium, Finland, France, Germany, Ireland, Italy, Netherlands, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Determination of ash from petroleum products

1. Scope and field of application

This European Standard describes a method for the determination of the ash, in the range 0.01 % to 0.20 % (*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 (note 1). The method is limited to petroleum products which are free from added ash-forming additives, including certain phosphorus compounds (note 2).

NOTE 1. With certain types of sample the test result 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.

NOTE 2. This method is not intended for the analysis of unused lubricating oils containing additives; for such samples use a corresponding national standard based on CEN 19/U11–1971– 'Test for sulphated ash from lubricating oils and additives'; neither is it intended for the analysis of lubricating oils containing lead nor for used engine crankcase oils.

2. Summary of method

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 ash by heating in a muffle furnace at 775 °C, cooled and weighed.

3. Apparatus

3.1 *Evaporating dish or crucible,* 90 ml to 120 ml capacity 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 so as to allow a slow natural draught of air to pass through.

3.3 *Cooling container,* not containing a desiccating agent.

4. Procedure

4.1 Heat the evaporating dish or crucible to 700 °C to 800 °C for 10 min or more. Cool to room temperature in the cooling container, and weigh to the nearest 0.1 mg.

4.2 The amount of test portion to be taken will depend upon the ash level from the material. Weigh into the dish or crucible a sufficient test portion (up to a maximum of 100 g) to give up to 20 mg of ash. For quantities which require more than one filling of the dish, obtain the mass from the difference between the initial and final masses of a suitable sample container. Weigh the portion to the nearest 0.1 %. Heat the dish or crucible and test portion, until the contents can be ignited with a flame. Maintain at such a temperature that the test portion continues to burn at a uniform and moderate rate, leaving only ash and carbon when the burning ceases.

NOTE 3. If the test portion contains sufficient moisture to cause foaming and loss of material, discard it, and to a second test portion add 1 ml 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.

4.3 Heat the residue in the muffle furnace 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.

4.4 Reheat the dish to 775 °C for 20 min to 30 min, cool in the cooling container, and reweigh. Repeat the heating and weighing until consecutive weighings differ by not more than 0.5 mg.

5. Calculation

Calculate the ash content *(X)* of the test portion as a percentage as follows :

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

where :

m_1 is the mass, in grams, of ash
m_0 is the mass, in grams, of test portion

6. Test report

Report the result to two significant figures as the percentage ash, with reference to this European Standard, stating the mass of the test portion taken.

7. Precision

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

7.1 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 value only in one case in twenty :

0.20 ($\bar{X}$ + 0.01) in the range 0.01 % to 0.20 % (*m/m*) ash

where :

$\bar{X}$ is the average of the two results.

7.2 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 value only in one case in twenty :

0.25 ($\bar{X}$ + 0.01) in the range 0.01 % to 0.20 % (*m/m*) ash

where :

$\bar{X}$ is the average of the two results.