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Non-ionic surface active agents — Determination of sulphated ash — Gravimetric method

Agents de surface non ioniques — Détermination du taux de cendres sulfatées — Méthode gravimétrique

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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.

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

Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Italy	Spain
Canada	Japan	Switzerland
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Poland	U.S.A.
Hungary	Portugal	

No member body expressed disapproval of the document.

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Non-ionic surface active agents — Determination of sulphated ash — Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the sulphated ash of non-ionic surface active agents in general.

2 REFERENCE

ISO 607, *Surface active agents — Detergents — Methods of sample division.*[1)]

3 PRINCIPLE

Calcination of a test portion in the presence of sulphuric acid solution and weighing of the ash in the form of sulphate.

4 REAGENTS

During the analysis, use only reagents of analytical grade and only distilled water or water of at least equivalent purity.

4.1 Ammonium nitrate (NH_4NO_3).

4.2 Sulphuric acid, approximately 6 N solution, free from ash.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible, capacity 100 ml.

5.2 Furnace, capable of being controlled at 775 ± 25 °C.

5.3 Gas burner.

6 SAMPLING

The laboratory sample of the non-ionic surface active agent shall be prepared and stored according to the instructions given in ISO 607.

7 PROCEDURE

7.1 Treatment of the laboratory sample

Mix the laboratory sample; if necessary, gently melt the product without overheating to make homogenization possible.

NOTE — This test sample obtained shall then be used only for this determination.

7.2 Test portion

Before weighing the test portion, heat the crucible (5.1) at 775 ± 25 °C for 10 min and, after cooling, place it in a desiccator until it reaches ambient temperature and weigh it to the nearest 0,1 mg.

Place about 30 g, weighed to the nearest 0,1 mg, of the laboratory sample (7.1) in the tared crucible.

7.3 Determination

CAUTION : It is essential to wear safety goggles and to carry out the combustion in a fume cupboard.

Place the crucible containing the test portion (7.2) above a low flame of a gas burner. Warm gently until a small flame played on the product initiates burning at as low a temperature as possible. Then withdraw the burner and allow the combustion to proceed unaided.

NOTE — It is important to allow the combustion to proceed unaided and gently because, if it is accelerated by keeping the burner under the crucible, the rapid burning which will result will give rise to spattering out of the crucible and consequent loss of product.

When combustion has stopped, replace the gas burner under the crucible.

1) In preparation. (Revision of ISO/R 607.)

If the spontaneous combustion recommences, withdraw the burner again and allow to burn until extinction and so on for as long as spontaneous combustion is produced by replacing the burner under the crucible.

NOTE – In practice, the re-ignition can occur three or four times and this step in the procedure can last for 30 to 60 min.

After having obtained a carbonaceous residue, proceed to the calcination proper.

Allow the crucible and its contents to cool, add several crystals (about 0,1 g) of the ammonium nitrate (4.1) and thoroughly wet the residue with 0,5 ml of the sulphuric acid solution (4.2).

Place the crucible on the flame, gradually increase the temperature to dull red and maintain at this temperature until the carbon has burnt completely.

Allow to cool and if necessary add some of the ammonium nitrate and some of the sulphuric acid solution so as to avoid attack of the platinum crucible by sulphides.

Again heat the crucible on the flame to dull red, then introduce it into the furnace (5.2), controlled at 775 ± 25 °C, and leave for 20 to 30 min. Cool the crucible slightly, place it in a desiccator until ambient temperature is reached and then weigh it to the nearest 0,1 mg.

Replace the crucible in the furnace, controlled at 775 ± 25 °C, and leave for 20 min. Allow to cool as before and re-weigh.

Repeat the operations of heating in the furnace, cooling and weighing until two successive weighings do not differ by more than 1 mg.

NOTE – If the required agreement between two successive weighings is not achieved after the sequence of operations has been repeated three times, add several drops of the sulphuric acid solution (4.2) before continuing.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

The sulphated ash is given, as a percentage by mass, by the formula

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

where

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

m_1 is the mass, in grams, of the sulphated ash.

8.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not exceed 0,1 % for sulphated ash levels of about 1 %.

9 TEST REPORT

The test report shall include the following particulars :

a) all information necessary for the complete identification of the sample;

b) the method used;

c) the results obtained and the form in which they are expressed;

d) the test conditions;

e) any operation not included in this International Standard or regarded as optional, as well as any incidents which may have affected the results.