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Sodium sulphate for industrial use — Determination of acid-insoluble matter

Sulfate de sodium à usage industriel — Détermination des matières insolubles dans l'acide

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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 3235 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in November 1973.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Poland
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Ireland	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Korea, Rep. of	Turkey
Finland	Netherlands	United Kingdom
France	New Zealand	U.S.S.R.

No Member Body expressed disapproval of the document.

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Sodium sulphate for industrial use — Determination of acid-insoluble matter

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of acid-insoluble matter in sodium sulphate for industrial use. The method is applicable to anhydrous and hydrated sodium sulphate.

2 REFERENCES

ISO 3234, *Sodium sulphate for industrial use — Determination of loss in mass at 110 °C.*

ISO . . ., *Chemical products — Sampling.* [1)]

3 PRINCIPLE

Extraction of a test portion with dilute acid, filtration, drying and weighing of the residue.

4 REAGENTS

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

4.1 Hydrochloric acid, approximately 6 N solution.

4.2 Sodium hydroxide, approximately 2 N solution.

4.3 Barium chloride, approximately 1 N solution.

Dissolve 12 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in water, dilute to 100 ml and mix.

4.4 Methyl orange, 0,5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Crucible or **funnel,** of glass or porcelain, with a sintered filter base approximately 30 mm in diameter, of pore size index P 16 (diameter of pores 4 to 16 μm).

5.2 Electric oven, with natural draught, capable of being controlled at 110 ± 2 °C.

6 SAMPLING

For the sampling instructions, including the number of increments to be taken from a batch of given size, use the method specified in ISO . . .

7 PROCEDURE

7.1 Test portion

7.1.1 *Anhydrous sodium sulphate*

Weigh, to the nearest 0,01 g, about 20 g of the test sample.

7.1.2 *Sodium sulphate decahydrate*

Weigh, to the nearest 0,01 g, about 45 g of the test sample.

7.2 Determination

Place the test portion (7.1) in a 1 000 ml beaker and add 700 ml of water and 3 drops of the methyl orange solution (4.4).

— If the colour is yellow add, drop by drop, the hydrochloric acid solution (4.1) until a pink colour is obtained.

— If the colour is pink add, drop by drop, the sodium hydroxide solution (4.2) until a yellow colour is obtained, then add the hydrochloric acid solution until a pink colour is obtained.

Then add 50 ml of the hydrochloric acid solution.

Heat for 10 min just below boiling, with stirring, and then place the beaker, covered with a watch-glass, on a boiling water bath for 30 min. Cool to about 20 °C.

Filter under vacuum through the crucible or funnel (5.1), previously dried for 1 h in the oven (5.2), controlled at 110 ± 2 °C, allowed to cool in a desiccator and weighed to the nearest 0,1 mg. Collect the filtrate and the washings in a 1 000 ml filter flask. Wash with cold water. Verify the absence of sulphates at the end of the washing by a test with the barium chloride solution (4.3) in hydrochloric acid medium.

1) In preparation.

Dry the crucible or the funnel and its contents for 1 h in the oven (5.2), controlled at 110 ± 2 °C, allow to cool in a desiccator and weigh to the nearest 0,1 mg.

8 EXPRESSION OF RESULTS

The content of acid-insoluble matter of the product "as received" is given, as a percentage by mass, by the formula :

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the crucible or filter and its contents;

m_2 is the mass, in grams, of the crucible or funnel.

Express the result to three decimal places.

NOTE – If it is desired to express the result on the basis of the material dried at 110 °C, multiply the result obtained for the product "as received" by the factor

$$\frac{100}{100 - P}$$

where P is the loss in mass at 110 °C, determined by the method specified in ISO 3234.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

ANNEX

This document forms part of a series of International Standards on methods of test for sodium sulphate for industrial use.

The complete list of the International Standards published is as follows :

ISO 3234 – Determination of loss in mass at 110 °C.

ISO 3235 – Determination of acid-insoluble matter.

ISO 3236 – Determination of chlorides content – Mercurimetric method.

ISO 3237 – Determination of sulphates content – Calculation method and barium sulphate gravimetric method.

ISO 3238 – Determination of calcium content – EDTA complexometric method.

ISO 3239 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 3240 – Determination of acidity or alkalinity.

ISO 3241 – Measurement of pH – Potentiometric method.