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Sodium sulphate for industrial use — Determination of loss in mass at 110 °C

Sulfate de sodium à usage industriel — Détermination de la perte de masse à 110 °C

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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 3234 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 loss in mass at 110 °C

1 SCOPE

This International Standard specifies a method for the determination of the loss in mass at 110 °C of sodium sulphate for industrial use.

2 FIELD OF APPLICATION

The method is applicable to anhydrous sodium sulphate.

2.1 Special case

Sodium sulphate decahydrate (see clause 10).

3 REFERENCE

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

4 PRINCIPLE

Heating of a test portion, spread in a thin layer, to constant mass in an oven the temperature of which is controlled at 110 ± 2 °C.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Weighing bottle, squat form (diameter 50 mm and height 25 mm) with ground glass lid.

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

NOTE — The thermometer used to check this temperature shall be positioned so that its bulb is as close as possible to the area where the test portions are placed.

5.3 Desiccator, containing molecular sieves or phosphorus(V) oxide.

6 SAMPLING

For 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

Weigh, to the nearest 0,001 g, about 20 g of the test sample in the weighing bottle (5.1), previously dried in the oven (5.2) controlled at 110 ± 2 °C, cooled in the desiccator (5.3) and weighed to the nearest 0,001 g.

7.2 Determination

Regulate the temperature of the oven (5.2) at 110 ± 2 °C. Insert the weighing bottle containing the test portion (7.1), with its lid resting on top without closing the bottle, and a watch-glass of diameter slightly greater than that of the weighing bottle. After drying for 2 h, replace the lid by the watch-glass and transfer all to the desiccator (5.3). After cooling, close the weighing bottle with its lid and weigh to the nearest 0,001 g. Repeat the operations of heating, for periods of about 1 h, cooling and weighing until two consecutive weighings do not differ by more than 0,001 g.

8 EXPRESSION OF RESULTS

The loss in mass at 110 °C, expressed as a percentage by mass, is given by the formula

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

where

m_0 is the mass, in grams, of the weighing bottle with its lid;

m_1 is the mass, in grams, of the weighing bottle, with its lid, containing the test portion before drying;

m_2 is the mass, in grams, of the weighing bottle, with its lid, containing the test portion after drying.

1) In preparation.

9 REPEATABILITY AND REPRODUCIBILITY

Comparative analyses on three samples, carried out in fourteen laboratories, have given the following statistical information :

Characteristic		Sample A	Sample B	Sample C
Mean, % (*m/m*)		0,011	0,068	0,261
Standard deviation	of repeatability, σ_r	0,002	0,005	0,006
	of reproducibility, σ_R	0,005	0,015	0,052

10 SPECIAL CASE : Sodium sulphate decahydrate

10.1 Principle

Progressive heating of a test portion, spread in a thin layer, to constant mass by means of a hot-plate and a radiant heater.

NOTE — The temperature shall be raised slowly and it is desirable to heat the test portion from above so as to avoid creeping and loss of the crystals by decrepitation.

10.2 Apparatus

The same apparatus as specified in clause 4 and

10.2.1 Hot-plate, electric.

10.2.2 Radiant heater or **infra-red lamp.**

NOTE — A 200 W silica radiant heater, placed about 10 cm above the test portion, is recommended to obtain the required temperature of 110 ± 2 °C. This method of heating enables a constant mass (to within 0,001 g) to be obtained after 2 h without creeping and loss of the crystals by decrepitation.

10.3 Procedure

10.3.1 *Test portion*

Weigh, to the nearest 0,001 g, about 10 g of the test sample into the weighing bottle (5.1), previously dried in the oven (5.2) controlled at 110 ± 2 °C, cooled in the desiccator (5.3) and weighed to the nearest 0,001 g.

10.3.2 *Determination*

Place a sheet of asbestos on the cold hot-plate (10.2.1) and a silica triangle on the asbestos sheet.

Place the weighing bottle (5.1) containing the test portion (10.3.1) on the triangle and heat gradually, using the hot-plate, until the temperature, as measured by a thermometer the bulb of which is positioned in the vicinity of the surface of the test portion, reaches about 90 °C. Then place the radiant heater or infra-red lamp (10.2.2) above the weighing bottle and gradually raise the temperature to 110 ± 2 °C. Allow to remain at this temperature for 2 h and then proceed as specified in 7.2, from "After drying for 2 h, ...".

10.4 Expression of results

See clause 8.

11 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 Standard 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.