INTERNATIONAL STANDARD 5142

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Sodium sulphate for industrial use — Determination of sodium sulphate content — Gravimetric method

Sulfate de sodium à usage industriel — Détermination de la teneur en sulfate de sodium — Méthode gravimétrique

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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 5142 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1976.

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

Austria	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Switzerland
Chile	Korea, Rep. of	Thailand
Czechoslovakia	Mexico	Turkey
France	Netherlands	United Kingdom
Germany	Philippines	Yugoslavia
Hungary	Poland	
India	Romania	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Sodium sulphate for industrial use — Determination of sodium sulphate content — Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the sodium sulphate content of sodium sulphate for industrial use.

The method is applicable to products in which not all impurities are known or determined, that is to say in cases where the calculation and gravimetric methods specified in ISO 3237 cannot be applied.

2 REFERENCES

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

ISO 3236, *Sodium sulphate for industrial use — Determination of chlorides content — Mercurimetric method.*

ISO 3237, *Sodium sulphate for industrial use — Determination of sulphates content — Calculation method and barium sulphate gravimetric method.*

ISO 3240, *Sodium sulphate for industrial use — Determination of acidity or alkalinity.*

3 PRINCIPLE

Dissolution of a test portion, removal of insoluble matter, iron, aluminium and calcium from the solution, and evaporation to dryness, in the presence of sulphuric acid.

Heating and weighing of the total sodium sulphate.

Calculation of the sodium sulphate content by subtraction of the sodium sulphate arising from any sodium chloride and sodium carbonate which may be present in the test portion.

4 REAGENTS

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

4.1 Sulphuric acid, approximately 2 N solution.

4.2 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

4.3 Nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution.

4.4 Ammonia solution, ρ approximately 0,91 g/ml, about 25 % (*m/m*) or approximately 13 N solution.

4.5 *di*-**Ammonium oxalate,** 40 g/l solution.

4.6 *di*-**Ammonium oxalate,** 1 g/l washing solution.

4.7 Ammonium carbonate, solution saturated at room temperature.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Silica dish, of capacity approximately 100 ml.

5.2 Radiant heater, 200 W, or **infra-red lamp.**

5.3 Electric furnace, capable of being controlled at 650 ± 20 °C.

6 PROCEDURE

6.1 Test portion

6.1.1 *Anhydrous sodium sulphate*

Weigh, to the nearest 0,001 g, about 5 g of the test sample.

6.1.2 *Sodium sulphate decahydrate*

Weigh, to the nearest 0,001 g, about 10 g of the test sample.

6.2 Preparation of the test solution

Place the test portion (6.1) in a 500 ml beaker. Add 100 ml of hot water, 5 ml of the hydrochloric acid solution (4.2) and a few drops of the nitric acid solution (4.3). Boil the mixture for 10 min in order to dissolve the soluble salts, and add a slight excess of the ammonia solution (4.4), and 10 ml of the *di*-ammonium oxalate solution (4.5). Boil until the smell of ammonia has almost completely disappeared, cover the beaker with a watch-glass and allow to stand for 15 min.

Filter the contents of the beaker, collecting the filtrate in a 250 ml one-mark volumetric flask. Wash the beaker with the *di*-ammonium oxalate solution (4.6), as cold as possible, and filter the washings into the flask until it is filled almost to the mark. Remove the flask, collect the remainder of the filtrate in a clean beaker and ensure that this residual filtrate is free from sulphate as shown by the absence of opalescence when adding a small portion to a solution of barium chloride and dilute hydrochloric acid solution.

Cool the solution in the one-mark volumetric flask to about 20 °C, dilute to the mark and mix.

6.3 Determination

Heat the silica dish (5.1) for 15 min in the furnace (5.3), controlled at 650 ± 20 °C, allow it to cool to ambient temperature in a desiccator and weigh it to the nearest 0,000 2 g. Place 50,0 ml of the test solution (6.2) in the tared dish.

Carry out the following procedure, performing all the heating operations in a well-ventilated fume cupboard.

Place the dish on an electric hot-plate and evaporate the solution, without boiling, to a pasty consistency by heating simultaneously with the electric hot-plate and either the radiant heater or the infra-red lamp (5.2).

Place a pipe-clay triangle between the dish and the hot-plate and continue heating until the solution has evaporated to dryness. Transfer the dish onto an asbestos/wire gauze mounted on a tripod and heat by means of a gas burner, increasing the temperature progressively until ammonium salts or sulphur trioxide fumes cease to be evolved. Do not overheat. Remove the asbestos/wire gauze and pass the gas flame quickly and progressively over the external surface of the dish, mounted on the tripod, to remove the last traces of volatile material. Transfer the basin to the furnace, controlled at 650 ± 20 °C, and heat for 15 min.

Remove the dish from the furnace, allow it to cool and moisten the residue with about 2 ml of the sulphuric acid solution (4.1). Again evaporate to dryness and heat, using the procedure specified in the third paragraph ("Place a pipe-clay triangle . . .").

Allow to cool, and moisten the residue with a small quantity of the ammonium carbonate solution (4.7). Again evaporate to dryness and heat using the procedure specified in the third paragraph.

Transfer the dish to a desiccator, allow to cool to ambient temperature and weigh to the nearest 0,000 2 g.

7 EXPRESSION OF RESULTS

7.1 Acid sample

If the acidity of the products is attributed to the presence of sodium hydrogensulphate ($NaHSO_4$), the sodium sulphate (Na_2SO_4) content, expressed as a percentage by mass, is given by the formula

$$m_1 \times \frac{250}{50} \times \frac{100}{m_0} - \left(C \times 2{,}003\,2 + D \times \frac{142}{98} \right)$$

$$= \frac{500\,m_1}{m_0} - (2{,}003\,2\,C + 1{,}449\,0\,D)$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the sodium sulphate obtained at the end of the determination;

C is the chloride content, expressed as a percentage by mass of chlorine (Cl), determined by the method specified in ISO 3236;

D is the acidity, expressed as a percentage by mass of sulphuric acid (H_2SO_4), determined by the method specified in ISO 3240;

2,0032 is the conversion factor of Cl to Na_2SO_4;

$\frac{142}{98}$ is the conversion factor to Na_2SO of the H_2SO_4 produced by the reaction

$$2\,NaHSO_4 \rightarrow Na_2SO_4 + H_2SO_4$$

7.2 Alkaline sample

The sodium sulphate (Na_2SO_4) content, expressed as a percentage by mass, is given by the formula

$$m_1 \times \frac{250}{50} \times \frac{100}{m_0} - (C \times 2{,}003\,2 + D \times 1{,}340\,1)$$

$$= \frac{500\,m_1}{m_0} - (2{,}003\,2\,C + 1{,}340\,1\,D)$$

where

m_0, m_1 and C are as defined in 7.1 :

D is the alkalinity, expressed as a percentage by mass of sodium carbonate (Na_2CO_3), determined by the method specified in ISO 3240;

1,340 1 is the conversion factor of Na_2CO_3 to Na_2SO_4.

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.

8 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

ISO PUBLICATIONS RELATING TO SODIUM SULPHATE FOR INDUSTRIAL USE

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.

ISO 5142 — Determination of sodium sulphate content — Gravimetric method.

ISO 5994 — Determination of calcium content — Flame atomic absorption method.

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