INTERNATIONAL STANDARD 3236

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Sodium sulphate for industrial use — Determination of chlorides content — Mercurimetric method

Sulfate de sodium à usage industriel — Dosage des chlorures — Méthode mercurimétrique

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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 3236 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	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Ireland	Switzerland
Chile	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
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France	New Zealand	

No Member Body expressed disapproval of the document.

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Sodium sulphate for industrial use — Determination of chlorides content — Mercurimetric method

1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable to products of which the chlorides content, expressed as Cl, is greater than 0,005 % (*m/m*).

NOTE — If the analyst can detect easily the indicator colour change using a 0,02 N standard volumetric mercury(II) nitrate solution, the method can, subject to agreement between the interested parties, be extended to products of which the chlorides content is greater than 0,001 % (*m/m*).

2 REFERENCES

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

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

3 PRINCIPLE

Titration of the chloride ions (Cl^-) with a standard volumetric solution of mercury(II) nitrate in the presence of 1,5-diphenylcarbazone as indicator.

4 REAGENTS

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

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

4.2 Nitric acid, approximately 2 N solution.

4.3 Sodium hydroxide, approximately 2 N solution.

4.4 Sodium chloride, 0,1 N standard reference solution.

Dissolve 5,844 3 g of sodium chloride, previously dried for 1 h at 500 °C and allowed to cool in a desiccator, in water in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

4.5 Colour matching solution

Prepare this solution immediately before use.

Place 200 ml of water and 3 drops of the bromophenol blue solution (4.7) in a 500 ml conical flask and add the nitric acid solution (4.2), drop by drop, until the colour changes from blue to yellow. Add an excess of 3 drops of this acid, 0,5 to 1,0 ml of the 1,5-diphenylcarbazone solution (4.8) and the volume of the standard volumetric mercury(II) nitrate solution (4.6), from a burette, necessary to change the colour of the solution from yellow to mauve (about 1 drop).

4.6 Mercury(II) nitrate, 0,1 N standard volumetric solution.

4.6.1 *Preparation of solution*

Weigh 10,85 ± 0,01 g of mercury(II) oxide (HgO) and dissolve in 10 ml of the nitric acid solution (4.1) in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

Standardize this solution by the procedure specified in 4.6.2, and adjust, if necessary, to the exact titre.

NOTE — Analysts who can detect easily the 1,5-diphenylcarbazone colour change may increase the sensitivity of the method by using a 0,02 N standard volumetric solution (2,18 g of HgO in 1 000 ml, standardized against a standard reference solution of sodium chloride containing 1,168 8 g of NaCl in 1 000 ml).

1) In preparation.

4.6.2 *Standardization of solution*

Transfer 40,00 ml of the standard reference sodium chloride solution (4.4), 160 ml of water and 3 drops of the bromophenol blue solution (4.7) to a 500 ml conical flask. Add the nitric acid solution (4.2), drop by drop, until the indicator changes colour from blue to yellow, an excess of 3 drops of this acid and then the same volume of the 1,5-diphenylcarbazone solution (4.8) as in the preparation of the colour matching solution (4.5). Titrate with the mercury(II) nitrate solution (4.6.1) until the mauve tint matches that of the colour matching solution and deduct the volume of the mercury(II) nitrate solution introduced during the preparation of the colour matching solution (about 1 drop).

The volume corresponding to the specified titre is 40,00 ml.

4.7 Bromophenol blue, 1 g/l solution in 95 % (*V/V*) ethanol.

4.8 1,5-Diphenylcarbazone, 5 g/l solution in 95 % (*V/V*) ethanol.

Store this solution in a refrigerator and replace it when it no longer gives a sharp end-point.

5 APPARATUS

Ordinary laboratory apparatus.

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

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

7.2 Determination

7.2.1 *Preparation of the test solution*

Place the test portion (7.1) in a 250 ml conical flask and add about 175 ml of cold water. Heat to boiling and continue to boil gently for 15 min. After cooling to 20 °C, transfer quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix. Filter, through a dry filter paper, a little more than 100 ml of this solution, discarding the first portion of filtrate and collecting the remainder in a dry receiver.

7.2.2 *Titration of the test solution*

Place 100,0 ml of the test solution (7.2.1) in a 500 ml conical flask and add 100 ml of water and 3 drops of the bromophenol blue solution (4.7).

— If the colour is blue add, drop by drop, the nitric acid solution (4.2) until the colour changes to yellow, then add an excess of 0,5 ml of this acid.

— If the colour is yellow add, drop by drop, the sodium hydroxide solution (4.3) until the colour changes to blue, then add the nitric acid solution (4.2) until the colour changes back to yellow and finally add an excess of 0,5 ml of this acid.

Then add the same volume of the 1,5-diphenylcarbazone solution (4.8) as in the preparation of the colour matching solution (4.5).

Titrate with the standard volumetric mercury(II) nitrate solution (4.6) until the mauve tint matches that of the colour matching solution.

8 EXPRESSION OF RESULTS

The chlorides content of the product "as received", expressed as a percentage by mass of chlorine (Cl), is given by the formula

$$(V_0 - V_1) \times 0{,}003\,545 \times \frac{200}{100} \times \frac{100}{m} = \frac{V_0 - V_1}{m} \times 0{,}709$$

where

V_0 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.6) used for the determination;

V_1 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.6) used to prepare the colour matching solution (4.5);

m is the mass, in grams, of the test portion (7.1);

0,003 545 is the mass, in grams, of chloride corresponding to 1 ml of the standard volumetric mercury(II) nitrate solution (4.6).

NOTES

1 If 0,02 N standard volumetric mercury(II) nitrate solution and sodium chloride solution are used, the formula becomes :

$$(V_0 - V_1) \times 0{,}000\,709\,0 \times \frac{200}{100} \times \frac{100}{m} = \frac{V_0 - V_1}{m} \times 0{,}141\,8$$

2 If the chlorides content, expressed as a percentage by mass of Cl, exceeds 1,77 % (*m/m*), which corresponds to 50 ml of the standard volumetric mercury(II) nitrate solution (4.6), it is desirable to take 50 ml of the filtered test solution (7.2.1) for the determination rather than 100 ml, to add 150 ml of water, 3 drops of the bromophenol blue solution (4.7) and then to follow the determination as indicated in 7.2.2, from "If the colour is blue...". In this case, the factor 0,709 in the formula becomes 1,418.

3 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 REPEATABILITY AND REPRODUCIBILITY

Comparative analyses on three samples, carried out in fourteen laboratories using the 0,1 N standard volumetric mercury(II) nitrate solution, have given the following statistical information :

Characteristic		Sample A	Sample B	Sample C
Mean, % (*m/m*)		0,006	0,301	0,342
Standard deviation	of repeatability, σ_r	0,001	0,004	0,004
	of reproducibility, σ_R	0,005	0,012	0,013

10 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.