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## Sodium chloride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method

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## FOREWORD

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# Sodium chloride for industrial use – Determination of sulphate content – Barium sulphate gravimetric method

## 1 SCOPE

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

## 2 FIELD OF APPLICATION

### 2.1 General case

The method is applicable to the determination of sulphate soluble in water, in sodium chloride for industrial use.

### 2.2 Special case

Determination of sulphate in a principal solution prepared in an acid medium.

NOTE – Whatever the conditions adopted, all related determinations shall be carried out in the same medium, except the determination of chlorides which shall always be carried out in an aqueous solution.

## 3 REFERENCE

ISO 2479, *Sodium chloride for industrial use – Determination of matter insoluble in water or acid and preparation of principal solutions for other determinations.*

## 4 PRINCIPLE

Dissolution of a test portion and separation of the insoluble residue.

Precipitation of the sulphate ions as barium sulphate in a boiling acid medium. Filtration, washing and ignition of the precipitate. Weighing of the barium sulphate.

## 5 REAGENTS

Distilled water or water of equivalent purity shall be used in the test.

**5.1 Hydrochloric acid,** approximately 6 N solution.

Dilute 50 ml of hydrochloric acid, $\rho$ 1,19 g/ml (approximately 38 % (*m/m*) solution, or approximately 12 N), to 100 ml.

**5.2 Sulphuric acid,** $\rho$ 1,84 g/ml (approximately 96 % (*m/m*) solution or approximately 36 N).

**5.3 Barium chloride,** approximately N solution.

Dissolve 122 g of barium chloride dihydrate in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**5.4 Silver nitrate,** 5 g/l nitric solution

Dissolve 0,5 g of silver nitrate in a little water, add 10 ml of nitric acid solution, $\rho$ 1,40 g/ml approximately, and dilute to 100 ml.

**5.5 Nitric acid** $\rho$ 1,40 g/ml (approximately 68 % (*m/m*) solution, or approximately 14 N).

## 6 APPARATUS

Ordinary laboratory apparatus and

**6.1 Electric furnace,** capable of being controlled at 800 ± 25 °C.

**6.2 Desiccator,** containing pumice moistened with sulphuric acid, silica gel, or phosphorus pentoxide.

**6.3 Platinum or porcelain crucibles,** approximately 30 mm diameter at the top, and approximately 30 mm deep.

## 7 PROCEDURE

### 7.1 Test portion

Take 100,0 ml of the principal solution A[1] containing 100 g of the test sample per 1 000 ml.

### 7.2 Determination

Place the test portion (7.1) in a 250 ml beaker.

Add 2,0 ml of the hydrochloric acid solution (5.1), heat to boiling, stirring continuously, and add, drop by drop, 10 ml of the barium chloride solution (5.3). (Time taken for making the addition, approximately 1,5 min.)

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[1] See clause 7.3 of ISO 2479.

Continue boiling for a few minutes, stirring continuously. Leave standing on a boiling water bath for 1 h, or approximately 12 h at ambient temperature.

Filter the precipitate through an ashless filter paper or a filter paper for which the mass of ash is known, and the pore size of which is sufficiently small to hold back all the precipitate (porosity grade P1,6 i.e. pore size index up to 1,6 $\mu$m) and wash with boiling water until 10 ml of the liquid leaving the funnel remains clear for 5 min after adding 10 ml of the silver nitrate solution (5.4). If chloride ions are detected, the precipitate should be washed again with small quantities of water, two or three times, until the complete disappearance of chloride ions from the washing water.

Carefully place the filter paper and contents in the crucible (6.3), previously heated to a red heat, cooled in the desiccator (6.2) and weighed, and place it in an oven at 110 ± 2 °C until completely dry. Ignite the filter paper at low temperature. This can be carried out with a butane gas or acetylene jet, or in the electric furnace (6.1). Keep the crucible at approximately 800 ± 25 °C for 15 min. If the ignited precipitate smells of sulphides (BaS) or has a grey appearance, indicating the presence of carbon black, moisten it with 1 drop of the nitric acid solution (5.5) and 1 drop of the sulphuric acid solution (5.2) and evaporate to dryness on a hot-plate in the crucible, fitted with its lid to avoid loss. Return to the furnace (6.1) and ignite again at 800 ± 25 °C for 15 min.

Cool in the desiccator (6.2) and weigh to the nearest 0,1 mg.

## 8 EXPRESSION OF RESULTS

### 8.1 Method of calculation and formula

The sulphate ($SO_4$) content is given, as a percentage by mass, by the formula :

$$m_1 \times 0{,}411\,53 \times \frac{1\,000}{100} \times \frac{100}{m_0} = \frac{m_1}{m_0} \times 411{,}53$$

where

$m_0$ is the mass, in grams, of the test portion used for preparing principal solution A;

$m_1$ is the mass, in grams, of barium sulphate weighed;

0,411 53 is the conversion factor of $BaSO_4$ to $SO_4$.

### 8.2 Repeatability and reproducibility

Comparative analyses on two samples in nineteen laboralaboratories have given the following statistical results :

| | | Evaporated salt | Marine salt |
|---|---|---|---|
| Mean (percentage by mass) | | 0,009 | 0,117 |
| Standard deviation | for repeatability ($\sigma_r$) | 0,001 5 | 0,003 3 |
| | for reproducibility ($\sigma_R$) | 0,003 8 | 0,008 5 |

## 9 SPECIAL CASE : Determination of sulphate in a principal solution prepared in an acid medium

### 9.1 Principle

Dissolution in an acid medium and separation of the insoluble residue.

Precipitation of sulphate ions in the filtrate in accordance with the general method.

### 9.2 Reagents

See section 5.

### 9.3 Apparatus

See section 6.

### 9.4 Procedure

#### 9.4.1 *Test portion*

Take 100,0 ml of solution B[1] containing 100 g of the test sample per 1 000 ml.

#### 9.4.2 *Determination*

See 7.2.

### 9.5 Expression of results

Use the formula given in section 8, where $m_0$ is the mass, in grams, of the test portion used for preparing principal solution B.

## 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 operations not included in this International Standard, or regarded as optional.

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[1] See clause 9.5.4 in ISO 2479.