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# INTERNATIONAL STANDARD ISO 3200

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## Sodium and potassium silicates for industrial use – Determination of sulphates content – Barium sulphate gravimetric method

*Silicates de sodium et de potassium à usage industriel — Dosage des sulfates — Méthode gravimétrique à l'état de sulfate de baryum*

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## FOREWORD

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International Standard ISO 3200 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in October 1973.

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

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- Switzerland
- Thailand
- Turkey
- United Kingdom
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- Yugoslavia

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

No Member Body expressed disapproval of the document.

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# Sodium and potassium silicates for industrial use — Determination of sulphates content — Barium sulphate gravimetric method

## 1 SCOPE

This International Standard specifies a barium sulphate gravimetric method for the determination of the sulphates content of sodium and potassium silicates for industrial use.

## 2 FIELD OF APPLICATION

The method is applicable to the analysis of products having sulphates contents either above 0,1 % or above 0,25 % (*m/m*) according to whether the product is readily or sparingly soluble in water.

## 3 PRINCIPLE

Dissolution of a test portion, either directly or after alkaline fusion, depending on whether the product is readily or sparingly soluble in water respectively. Separation of the insoluble matter. Precipitation, while boiling, of the sulphate ions in the form of barium sulphate, in an acid medium. Filtration, washing and calcination of the precipitate. Weighing of the barium sulphate after treatment with hydrofluoric acid in order to remove any silica which may be present.

## 4 REAGENTS

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

**4.1 Potassium sodium carbonate** ($KNaCO_3$), having a maximum sulphate content of 0,005 % (*m/m*).

**4.2 Hydrochloric acid**, approximately 2 N solution.

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

**4.4 Hydrofluoric acid**, $\rho$ approximately 1,15 g/ml, about 40 % (*m/m*) solution.

**4.5 Potassium sodium carbonate**, 10 g/l solution, prepared from 4.1.

**4.6 Barium chloride**, approximately 1 N solution.

Dissolve 122 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**4.7 Silver nitrate**, 5 g/l solution in nitric acid.

Dissolve 0,5 g of silver nitrate in a little water or take 32 ml of approximately 0,1 N silver nitrate solution. Add 10 ml of nitric acid, $\rho$ approximately 1,40 g/ml, dilute to 100 ml and mix.

**4.8 Methyl orange**, 0,5 g/l solution.

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Electric oven**, capable of being controlled at 110 ± 2 °C.

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

**5.3 Desiccator**, containing either molecular sieves, silica gel or phosphorus(V) oxide.

**5.4 Platinum crucible**, fitted with lid. Upper diameter approximately 30 mm and height approximately 30 mm.

**5.5 Platinum dish**, capacity approximately 80 ml.

## 6 PROCEDURE

### 6.1 Test portion

Weigh, to the nearest 0,01 g, a quantity of test sample equal respectively to :

- 10 g of liquid products;
- 5 g of solid products readily soluble in water;
- 2 g of solid products sparingly soluble in water.

### 6.2 Preparation of the test solution

#### 6.2.1 *Liquid products or solid products readily soluble in water*

Place the test portion (6.1) in a 1 000 ml beaker, dilute it or dissolve it in 200 ml of water and allow to stand for 1 h. If the resultant solution is clear, dilute it to 400 ml and proceed as specified in 6.3.

If the resultant solution is turbid, filter it and then wash the filter five times with the potassium sodium carbonate solution (4.5), heated to approximately 70 °C, collecting the filtrate and washings in a second 1 000 ml beaker. Dilute to 400 ml and then proceed as specified in 6.3.

### 6.2.2 *Solid products sparingly soluble in water*

Place the test portion (6.1) in the platinum dish (5.5) and mix it with 10 g of the potassium sodium carbonate (4.1). Heat until the mixture fuses, allow to cool and dissolve in 200 ml of hot water, transferring the solution quantitatively to a 400 ml beaker. After allowing to stand for 1 h, filter the solution, wash the filter five times with the potassium sodium carbonate solution (4.5), heated to approximately 70 °C, collecting the filtrate and washings in a 1 000 ml beaker. Then dilute to 350 ml and proceed as specified in 6.3.

### 6.3 Determination

Add 5 drops of the methyl orange solution (4.8) to the test solution (6.2), neutralize it with the hydrochloric acid solution (4.2) and add an excess of 25 ml of this acid. Boil for 2 min, turn off the heat and add very slowly, while stirring, 50 ml of the barium chloride solution (4.6).

Rinse down the internal walls of the beaker with about 20 ml of water, *without stirring*, so as to maintain a layer of water on the liquid surface and thus prevent solids climbing up the walls during heating. Cover the beaker with a watch glass and allow to stand for 1 h on a boiling water bath and then for 24 h at room temperature. Filter the precipitate through an ash-free fine grade filter paper having a sufficiently small pore dimension to retain the whole of the precipitate (pore diameter : 0,4 to 1 $\mu$m). Wash the filter paper and precipitate with boiling water until 10 ml of the liquid passing through the filter remains clear for 5 min after the addition of 10 ml of the silver nitrate solution (4.7). If chloride ion is detected, the precipitate should be washed a further two or three times with small quantities of water until the chloride ion has completely disappeared from the wash water. Place the filter paper and precipitate carefully in the crucible (5.4), previously heated to red heat, cooled in the desiccator (5.3) and weighed to the nearest 0,1 mg. Place the crucible and its contents in the oven (5.1), controlled at 110 ± 2 °C, until completely dry.

Char the filter paper in air, at a low temperature, then calcine at red heat for 15 min using either a butane or acetylene gas burner or the electric furnace (5.2), controlled at 800 ± 25 °C.

After cooling, add a few drops of the sulphuric acid solution (4.3) and 5 ml of the hydrofluoric acid solution (4.4). Evaporate to dryness on an air bath or hot-plate, then calcine again for 15 min under the same conditions as before.

Allow to cool in the desiccator (5.3) to room temperature and weigh to the nearest 0,1 mg.

NOTE — The filtration of the barium sulphate precipitate can be accelerated by first lining the filter with ash-free cellulose powder.

## 7 EXPRESSION OF RESULTS

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

$$m_1 \times 0{,}411\,57 \times \frac{100}{m_0} = \frac{41{,}157\, m_1}{m_0}$$

where

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

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

0,411 57 is the conversion factor from $BaSO_4$ to $SO_4$.

## 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 regarded as optional.