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

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

## 1 SCOPE AND FIELD OF APPLICATION

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

## 2 PRINCIPLE

Dissolution of a test portion.

Precipitation of the sulphate ions as barium sulphate in boiling acid medium.

Filtration, washing and ignition of the precipitate. Weighing of the barium sulphate.

## 3 REAGENTS

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

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

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

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

**3.3 Sodium hydroxide,** 100 g/l solution.

**3.4 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.

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

## 4 APPARATUS

Ordinary laboratory apparatus and

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

## 5 PROCEDURE

### 5.1 Test portion

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

### 5.2 Preparation of the test solution

Place the test portion (5.1) in a 400 ml beaker and add approximately 200 ml of cold water. Cover the beaker with a watch glass, heat and keep boiling gently for 15 min. Cool to 20 °C, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water at 20 °C and mix. This is solution A.

Filter a sufficient quantity of solution A through a dry filter paper, discarding the first portions of the filtrate and collecting the remainder in a dry receiver.

### 5.3 Determination

Transfer 25,0 ml of the filtrate to a 400 ml beaker. Add a few drops of the methyl orange solution (3.5). If the colour is yellow, add hydrochloric acid solution (3.1) drop by drop until the colour changes to pink.

If the colour is pink, add sodium hydroxide solution (3.3) drop by drop until the colour changes to yellow, followed by hydrochloric acid solution (3.1) until the colour again changes to pink.

Then add (in either case) 5 ± 0,5 ml of the hydrochloric acid solution (3.1). Dilute to about 300 ml with water. Bring to the boil and, while reducing the rate of boiling but without interrupting it, add 10 ± 0,5 ml of the barium chloride solution (3.4) drop by drop and keep boiling for about a further 1 min, stirring with a glass rod.

Cover the beaker with a watch glass and allow to stand for 4 h at a temperature of about 80 °C.

Then decant the supernatant liquid through a close-textured ashless filter paper. Wash the precipitate three times with boiling water, by decantation. Collect the precipitate on the filter and continue the washing with boiling water until chlorides disappear from the filtrate (test with silver nitrate solution). Place the residue and filter paper in a tared porcelain crucible and dry in an oven. Ignite the paper in air, then ignite it for 2 h in the furnace (4.1) controlled at 800 ± 25 °C.

If the ignited precipitate has a greyish appearance, indicating the presence of graphitic carbon, moisten it with 1 drop of the sulphuric acid solution (3.2), replace it in the furnace and ignite it again at 800 ± 25 °C for 15 min.

NOTE — Alternatively the filtration of the barium sulphate precipitate can be carried out on a borosilicate glass filter crucible, of porosity grade P 10 or P 16 (i.e. maximum pore size between 4 and 16 μm). Drying at 450 °C to constant mass will then be sufficient.

Allow to cool in a desiccator and weigh to the nearest 0,000 2 g.

Ignition for 2 h is generally sufficient; to confirm this, carry out a further ignition for 1 h, allow to cool and weigh. The two masses should not differ by more than 0,000 2 g.

## 6 EXPRESSION OF RESULTS

### 6.1 Method of calculation and formula

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

$$m_1 \times 0{,}411\,53 \times \frac{1\,000}{25} \times \frac{100}{m_0} = \frac{m_1 \times 41\,153}{25\,m_0} = \frac{1\,646\,m_1}{m_0}$$

where

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

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

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

### 6.2 Repeatability and reproducibility

The statistical information given below was obtained from analyses carried out in eleven laboratories, two operators in each case, each operator carrying out two determinations.

| | | Sample | | | Global value |
|---|---|---|---|---|---|
| | | A | B | C | |
| Mean (% $SO_4$) | | 43,9 | 44,6 | 44,8 | — |
| Standard deviation for | repeatability ($\sigma_r$) | 0,21 | 0,14 | 0,15 | 0,17 |
| | reproducibility ($\sigma_R$) | 0,33 | 0,16 | 0,17 | 0,25 |

## 7 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.