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Potassium sulphate for industrial use — Determination of potassium content — Flame emission spectrophotometric method

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FOREWORD

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Potassium sulphate for industrial use — Determination of potassium content — Flame emission spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a flame emission spectrophotometric method for the determination of the potassium content of potassium sulphate for industrial use.[1)]

2 PRINCIPLE

Dissolution of a test portion taken from the laboratory sample, previously ground and sifted.

Acidification to pH 1 after suitable dilution and atomization of the resultant solution in the burner of a flame spectrophotometer.

Determination of the potassium content by measurement of the intensity of the radiation emitted at 766 nm, compared with the intensity obtained for standard potassium chloride solutions acidified in the same way.

3 REAGENTS

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

This water shall, moreover, satisfy the following test :

The spectrophotometer having been adjusted so that a zero reading (minimum of the scale) is obtained for water (3.1) and about 100 (maximum of the scale) for a standard solution containing 10 mg/l of potassium, the reading obtained for the water, evaporated in a sodium-free vessel (platinum, silica, etc.) until it is reduced to one fiftieth of of its volume shall not exceed 10.

3.2 Sulphuric acid, approximately N solution, practically free from potassium. This solution, diluted ten times, should not give a spectrophotometer reading of more than 2, under the conditions stated in 3.1.

3.3 Standard potassium chloride solution, corresponding to 4,000 g of potassium per litre.

Weigh, to the nearest 0,001 g, 7,627 g of potassium chloride (KCl) previously dried at 400 °C and cooled in a desiccator. Dissolve it in the water (3.1), dilute to 1 000 ml in a one-mark volumetric flask and mix.

1 ml of this standard solution contains 4 mg of K.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Filter paper, of a sufficiently fine texture to retain all matter likely to choke the opening of the atomizer or the spectrophotometer burner, and which does not itself release troublesome fragments.

4.2 Flame spectrophotometer, fitted with an atomizer-burner constructed so as to excite emission of the potassium line at 766 nm.

NOTE — In the presence of other cations than potassium, and especially in the presence of sodium ions, the intensity of the potassium radiation is increased. In order to render this interference from other cations negligible, it is advisable to use as "cold" a flame as possible such as a butane-air complete combustion flame.

The sensitivity of the apparatus should be adjustable within wide limits, for example, for concentrations of potassium of 10 to 100 mg/l, at the bottom of the scale. In addition, after bringing the spectrophotometer into a state of normal operation for 30 to 60 min, the readings should be reproducible and stable 20 to 40 s after the start of atomization.

5 PROCEDURE

5.1 Preparation of test sample

Grind the laboratory sample until it passes completely through a 500 μm nominal mesh sieve.[2)]

NOTE — The moisture content of the laboratory sample may vary appreciably as a result of grinding and sifting. It is advisable to determine the moisture content of the ground and sifted product (test sample) before determining its potassium content so as to be able to relate it to the untreated product (laboratory sample), the moisture content of which should also be determined.

1) See also the same determination in
— ISO 2485, *Gravimetric method as potassium tetraphenylborate.*
— ISO 2486, *Sodium tetraphenylborate volumetric method.*

2) See Table 1, ISO/R 565, *Woven wire cloth and perforated plates in test sieves — Nominal sizes of apertures.*