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## Potassium sulphate for industrial use — Determination of chloride content — Mercurimetric method

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## FOREWORD

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International Standard ISO 2488 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in August 1971.

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

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No Member Body expressed disapproval of the document.



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# Potassium sulphate for industrial use — Determination of chloride content — Mercurimetric method

## 1 SCOPE AND FIELD OF APPLICATION

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

## 2 PRINCIPLE

Dissolution of a test portion.

Addition, in a dilute acid medium, of mercuric salts which, with the chloride ions, form soluble and only slightly ionized mercury(II) chloride.

Indication of the completion of the reaction by the appearance of a violet coloration due to the reaction between the excess of mercury(II) ions and the diphenylcarbazone used as indicator.

## 3 REAGENTS

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

**3.1 Nitric acid,** approximately 0,1 N solution.

Transfer 10 ml of nitric acid, $\rho$ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**3.2 Sodium hydroxide,** approximately N solution.

Dissolve 40 g of sodium hydroxide in water, dilute to 1 000 ml and mix.

**3.3 Mercury(II) nitrate,** approximately 0,1 N standard volumetric solution.

Weigh 10,85 ± 0,01 g of mercury(II) oxide (HgO) and dissolve it in 10 ml of nitric acid, $\rho$ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N, in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

Determine the exact concentration of the resultant mercury(II) nitrate solution, by means of 10 ml of the standard reference solution of potassium chloride (3.4) following the procedure described in 6.3.

**3.4 Potassium chloride,** standard reference solution.

Dry a quantity of potassium chloride at 400 °C and, after cooling in a desiccator, weigh 10,00 g, to the nearest 0,001 g, transfer it to a 1 000 ml one-mark volumetric flask and add water. After dissolution, dilute to the mark and mix.

1 ml of this solution contain 4,755 7 mg of chlorine (Cl).

**3.5 Bromophenol blue,** 0,5 g/l solution in 95 % (*V/V*) ethanol.

**3.6 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 definite colour change.

## 4 APPARATUS

Ordinary laboratory apparatus and

**4.1 Magnetic stirrer,** adjustable speed, with sheathed magnetic bar.

## 5 PREPARATION OF THE TEST SAMPLE

Grind the laboratory sample until it passes completely through a 500 $\mu$m nominal mesh sieve.[1)]

## 6 PROCEDURE

### 6.1 Test portion

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

### 6.2 Preparation of the test solution

Transfer the test portion (6.1) to a 400 ml beaker and add about 200 ml of cold water. Cover the beaker with a watch-glass, heat gently to boiling and continue boiling for 15 min. Cool to 20 °C, and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

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

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

### 6.3 Determination

Transfer 100,0 ml of the filtrate to a 250 ml conical flask. Add 1 drop of the bromophenol blue solution (3.5).

If the colour is blue, add the nitric acid solution (3.1) drop by drop until the colour turns yellow, then add 0,5 ml in excess.

If the colour is yellow, add the sodium hydroxide solution (3.2) drop by drop until the colour turns blue, then the nitric acid solution (3.1) until the colour turns yellow. Then add an excess of 0,5 ml of the nitric acid solution (3.1).

In either case, continue by adding 0,25 ml of the diphenylcarbazone solution (3.6).

Place a magnetic bar in the conical flask. Put this on the magnetic stirrer (4.1), inserting a piece of matt white paper between the container and the stirrer plate. Titrate with the mercury(II) nitrate solution (3.3) until the colour changes from yellow to pale mauve, which immediately follows a transitory pink coloration, this change being that observed in standardizing the mercury(II) nitrate solution (3.3).

## 7 EXPRESSION OF RESULTS

### 7.1 Method of calculation and formula

The chloride content, expressed as chlorine (Cl), is given, as a percentage by mass, by the formula

$$V_2 \times \frac{1\,000}{100} \times \frac{4{,}755\,7}{1\,000} \times \frac{10}{V_1} \times \frac{100}{m} = \frac{47{,}557}{m} \times \frac{V_2}{V_1}$$

where

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

$V_1$ is the volume, in millilitres, of the mercury(II) nitrate solution (3.3) necessary to reach the end-point with 10 ml of the potassium chloride standard reference solution (3.4);

$V_2$ is the volume, in millilitres, of the mercury(II) nitrate solution (3.3) used for the titration.

### 7.2 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 (% Cl) | | 2,5 | 1,8 | 0,9 | — |
| Standard deviation for | repeatability ($\sigma_r$) | 0,015 | 0,03 | 0,02 | 0,025 |
| | reproducibility ($\sigma_R$) | 0,04 | 0,05 | 0,05 | 0,05 |

## 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.