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## Sulphur for industrial use — Determination of chloride content — Photometric method

*Soufre à usage industriel — Dosage des chlorures — Méthode photométrique*

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## FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5793 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Austria
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Yugoslavia

No member body expressed disapproval of the document.

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

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# Sulphur for industrial use — Determination of chloride content — Photometric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of the chloride content of sulphur for industrial use.

The method is applicable to products having chloride contents, expressed as chlorine (Cl), greater than 2 mg/kg.

## 2 PRINCIPLE

Extraction of chlorides by boiling a test portion in aqueous 2-propanol.

Quantitative displacement of the thiocyanate ($SCN^-$) ions of mercury(II) thiocyanate by the chloride ($Cl^-$) ions contained in an aliquot portion of the extraction solution. Reaction of the $SCN^-$ ions with iron(III) nitrate to form iron(III) thiocyanate (red).

Photometric measurement of the colour at a wavelength of about 450 nm.

## 3 REACTIONS

$$2Cl^- + Hg(SCN)_2 \rightarrow HgCl_2 + 2SCN^-$$

$$3SCN^- + Fe(NO_3)_3 \rightarrow 3NO_3^- + Fe(SCN)_3$$

## 4 REAGENTS

Prepare and store the reagents in an atmosphere free from chlorine and hydrochloric acid.

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

**4.1 2-Propanol** (isopropanol).

**4.2 Nitric acid,** $\rho$ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N solution, having a chloride content, expressed as chlorine (Cl), not exceeding 0,5 mg/kg.

**4.3 Iron(III) nitrate** solution corresponding to 8 g of Fe per litre.

Pour 80 ml of water into a 500 ml conical flask, and add 4,0 g of iron wire (99,5 % minimum purity). Cautiously add 80 ml of the nitric acid solution (4.2). Heat the flask and contents in a ventilated fume cupboard, gently at first, and then bring to the boil. Continue boiling until the reaction is complete and no more nitrous fumes are evolved. Decolorize the solution by adding a few drops of hydrogen peroxide solution (30 % *m/m*), boil again for a few minutes and allow to cool. Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

**4.4 Mercury(II) thiocyanate,** 0,5 g/l solution.

Weigh, to the nearest 0,001 g, 0,100 g of mercury(II) thiocyanate [$Hg(SCN)_2$] and dissolve it, with stirring, in 180 ml of water at 50 °C.

Filter the solution into a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

**4.5 Sodium chloride,** standard solution corresponding to 0,100 g of Cl per litre.

Weigh, to the nearest 0,001 g, 0,165 g of sodium chloride, previously dried at 500 °C for 1 h and cooled in a desiccator. Dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,1 mg of Cl.

**4.6 Sodium chloride,** standard solution corresponding to 10 mg of Cl per litre.

Transfer 20,0 ml of the standard sodium chloride solution (4.5) to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,01 mg of Cl.

Prepare this solution at the time of use.

## 5 APPARATUS

Ordinary laboratory apparatus, carefully rinsed first with the nitric acid solution (4.2) and then with doubly distilled water, and

**5.1 Spectrophotometer**, or

**5.2 Photometer**, fitted with filters providing maximum transmission at about 450 nm.

## 6 PROCEDURE

Carry out the calibration and determination in an atmosphere free from chlorine and hydrochloric acid.

### 6.1 Test portion

Grind the test sample sufficiently for it to pass through a sieve of 250 µm aperture size (see ISO 565).

Weigh, to the nearest 0,1 g, between 20 and 100 g of the ground test sample.

### 6.2 Preparation of the calibration graph

**6.2.1** *Preparation of the standard colorimetric solutions* for photometric measurements in cells of optical path length 4 or 5 cm

Into a series of six 50 ml one-mark volumetric flasks, place the volumes of the standard sodium chloride solution (4.6) shown in the following table.

| Standard sodium chloride solution (4.6) | Corresponding mass of chlorine (Cl) |
|---|---|
| ml | mg |
| 0* | 0 |
| 1,0 | 0,010 |
| 2,5 | 0,025 |
| 5,0 | 0,050 |
| 7,5 | 0,075 |
| 10,0 | 0,100 |

* Compensation solution.

To each flask, make the following additions in the stated order :

- 5 ml of the nitric acid solution (4.2),
- 5 ml of the iron(III) nitrate solution (4.3), and
- 20 ml of the mercury(II) thiocyanate solution (4.4).

Dilute each solution to the mark, mix and leave for 30 min to allow colour development.

**6.2.2** *Photometric measurements*

Carry out the photometric measurements using either the spectrophotometer (5.1), at the wavelength of maximum absorbance (about 450 nm) or the photometer (5.2), fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

**6.2.3** *Plotting the graph*

Deduct the absorbance of the compensation solution (see 6.2.1) from those of the standard colorimetric solutions (6.2.1).

Plot a graph having, for example, the masses, in milligrams, of chlorine (Cl) contained in 50 ml of the standard colorimetric solutions as abscissae, and the corresponding values of absorbance as ordinates.

### 6.3 Determination

**6.3.1** *Preparation of the test solution*

Place the test portion (6.1) in a 500 ml boiling flask. Add exactly 200 ml of a mixture consisting of 1 volume of the 2-propanol (4.1) and 7 volumes of water.

Fit a reflux condenser and boil the contents of the flask moderately for 30 min. Allow to cool and filter through a dry filter paper of medium texture, discarding the first 100 ml (approximately) of filtrate and collecting the remainder. Do not rinse.

**6.3.2** *Colour development*

Transfer 10,0 ml of the test solution (6.3.1) to a 50 ml one-mark volumetric flask. Add 5 ml of the nitric acid solution (4.2), then 5 ml of the iron(III) nitrate solution (4.3) and 20 ml of the mercury(II) thiocyanate solution (4.4).

Dilute to the mark, mix and leave for 30 min to allow colour development.

**6.3.3** *Photometric measurement*

Carry out the photometric measurement on the test solution (6.3.1) and on the blank test solution (6.4) following the procedure specified in 6.2.2, after having adjusted the instrument to zero absorbance against water.

NOTE — If the absorbance exceeds the maximum plotted value of the calibration graph, repeat the determination using a smaller volume of the test solution and modifying the calculation accordingly.

### 6.4 Blank test

Carry out a blank test, following the same procedure and using the same quantities of all the reagents used for the determination (6.3.2 and 6.3.3), but replacing the 10,0 ml of test solution by 10,0 ml of a mixture consisting of 1 volume of the 2-propanol (4.1) and 7 volumes of water.

## 7 EXPRESSION OF RESULTS

By reference to the calibration graph (6.2.3), determine the quantity of Cl corresponding to the value of the photometric measurement.

The chloride content, expressed as milligrams of chlorine (Cl) per kilogram, is given by the formula

$$(m_1 - m_2) \times \frac{200}{10} \times \frac{1\,000}{m_0}$$

$$= \frac{20\,000\,(m_1 - m_2)}{m_0}$$

where

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

$m_1$ is the mass, in milligrams, of Cl found in the test solution;

$m_2$ is the mass, in milligrams, of Cl found in the blank test solution.

Express the results to the nearest 1 mg/kg.

## 8 TEST REPORT

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or regarded as optional.

## ANNEX

## ISO PUBLICATIONS RELATING TO SULPHUR FOR INDUSTRIAL USE

ISO 2866 — Determination of total carbon content — Titrimetric method.

ISO 3425 — Determination of ash content at 850-900 °C and of residue at 200 °C.

ISO 3426 — Determination of loss in mass at 80 °C.

ISO 3704 — Determination of acidity — Titrimetric method.

ISO 3705 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 5793 — Determination of chloride content — Photometric method.