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Hydrochloric acid for industrial use — Determination of soluble sulphates — Turbidimetric method

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FOREWORD

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International Standard ISO 2762 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in April 1972.

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

Austria
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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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Hydrochloric acid for industrial use — Determination of soluble sulphates — Turbidimetric method

1 SCOPE

This International Standard specifies a turbidimetric method for the determination of soluble sulphates in hydrochloric acid for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products containing less than 0,1 % (*m/m*) of soluble sulphates, expressed as SO_4.[1]

3 PRINCIPLE

Evaporation of a test portion to dryness and solution of the residue in hydrochloric acid.

Measurement of the turbidity obtained by precipitation, under well-defined conditions, of barium sulphate.

4 REAGENTS

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

4.1 **Barium chloride dihydrate**, of uniform particle size between 0,50 and 1,25 mm, standardized by screening.

It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

4.2 **Sodium carbonate**, approximately N solution.

4.3 **Hydrochloric acid**, approximately N solution.

4.4 **Sulphuric acid standard solution**, corresponding to 0,100 g of SO_4 per litre.

Transfer 20,80 ml of 0,1 N standard volumetric sulphuric acid solution 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 SO_4.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 **Spectrophotometer**, or

5.2 **Photoelectric absorptiometer**, fitted with filters, giving only a negligible transmission below 450 nm and above 550 nm.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, a quantity of the test sample containing 0,5 to 4 mg of SO_4.

If the test sample is turbid, first pass it through a dry filter, collecting the filtrate in a dry receiver, discarding the first portions of the filtrate.

NOTE — In this case any sulphates contained in the insoluble matter are not determined.

6.2 Blank test

At the same time as the determination, carry out a blank test, following the same procedure and using the same quantities of all the reagents used for the determination, but replacing the test portion by 5 ml of the sulphuric acid standard solution (4.4), corresponding to 0,5 mg of SO_4, added to allow operation in the linear part of the calibration curve.

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[1] For products having an SO_4 content greater than 0,1 % (*m/m*) see ISO/R 906, *Hydrochloric acid for industrial use — Determination of sulphate content — Barium sulphate gravimetric method.*

6.3 Preparation of the calibration curve

6.3.1 *Preparation of the standard matching solutions*

Into each of a series of nine 50 ml one-mark volumetric flasks, place the volumes of the sulphuric acid standard solution (4.4) shown in the following table :

Sulphuric acid standard solution (4.4)	Corresponding mass of SO_4
ml	mg
0 *	0
5,0	0,5
10,0	1,0
15,0	1,5
20,0	2,0
25,0	2,5
30,0	3,0
35,0	3,5
40,0	4,0

* Compensation solution.

To each flask, add 2 ml of the sodium carbonate solution (4.2) and 5 ml of the hydrochloric acid solution (4.3), stir, dilute to the mark and mix.

6.3.2 *Turbidimetric reaction*

Transfer 25,0 ml of each solution except the first, to separate dry 100 ml beakers, each containing 0,15 g of barium chloride (4.1). Stir by hand for 1 min at the rate of 2 revolutions per second.

Allow to stand for 15 min at 20 ± 2 °C.

NOTE — Stagger the tests in order to adhere to the contact times indicated.

6.3.3 *Turbidimetric measurement*

Transfer a sufficient quantity of the compensation solution (6.3.1) to a cell of suitable pathlength and use this solution to adjust the spectrophotometer (5.1) or the photoelectric absorptiometer (5.2) to the optical zero.

Stir by hand and transfer the other solutions (6.3.2) to cells of the same pathlength and carry out the measurements at a wavelength of about 470 nm.

6.3.4 *Preparation of the calibration curve*

Plot a graph with the SO_4 content, in milligrams, of the total quantity (50 ml) of the standard matching solution as abscissae, and the corresponding values of measurements as ordinates. It should be noted that the calibration curve is linear only above 0,5 mg of SO_4.

6.4 Determination

6.4.1 *Preparation of the test solution*

Transfer the test portion (6.1) to an evaporating dish of suitable capacity and add 2 ml of the sodium carbonate solution (4.2). Evaporate to dryness on a boiling water bath and dissolve the residue in 5 ml of the hydrochloric acid solution (4.3) and 20 ml of water. Heat for a few minutes on the boiling water bath, allow to cool, then transfer quantitatively to a 50 ml one-mark volumetric flask, dilute to the mark and mix.

6.4.2 *Turbidimetric reaction*

Take 25,0 ml of the test solution (6.4.1) and transfer this rapidly to a dry 100 ml beaker containing 0,15 g of barium chloride (4.1). Stir by hand for 1 min at the rate of 2 revolutions per second. The barium chloride should then be in complete solution. Leave undisturbed for 15 min at 20 ± 2 °C.

6.4.3 *Turbidimetric measurement*

Transfer a sufficient quantity of the test solution (6.4.1) to a cell of pathlength corresponding to that used for the preparation of the calibration curve (6.3.1), and use this solution as compensation solution for adjusting the spectrophotometer (5.1) or the photoelectric absorptiometer (5.2) to the optical zero.

Stir the solution (6.4.2) by hand, transfer it to a cell of the same pathlength and conduct the turbidimetric measurement by the method described in 6.3.3.

NOTE — If the reaction leads to turbidity beyond the limit of the calibration curve, dilute an aliquot of the test solution (6.4.1) to 50 ml and conduct the determination on 25 ml of this dilute solution. In this case, use this latter solution as compensation solution and take account of this additional dilution in calculating the result.

7 EXPRESSION OF RESULTS

By means of the calibration curve (6.3.4), determine the quantity of sulphate (SO_4) corresponding to the values of the turbidimetric measurements.

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

$$(m_1 - m_2) \times \frac{1}{1\,000} \times \frac{100}{m_0} = \frac{(m_1 - m_2)}{10\, m_0}$$

where

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

m_1 is the mass, in milligrams, of sulphate found in the total quantity of the test solution (6.4.1);

m_2 is the mass, in milligrams, of sulphate found in the total quantity of the blank test solution (6.2), after deducting the 0,5 mg of sulphate added (see 6.2).

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.