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Sulphuric acid for industrial use — Determination of chlorides content — Potentiometric method

AMENDMENT 1

Amendment 1 to International Standard ISO 2877 was drawn up by Technical Committee ISO/TC 47, *Chemistry*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

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Annex : The results given in the example were obtained using a platinum electrode. As it was subsequently agreed to use a silver electrode (see 5.1.4), the example should be related to the results obtained using a silver electrode, even if the analytical result is essentially the same. Hence, replace the table by the following :

Volume of silver nitrate solution V	**Potential** E	$\Delta_1 E$	$\Delta_2 E$
ml	mV		
4,8 4,9 5,0 5,1 5,2	125 134 159 275 324	9 25 116 49	+ 16 + 91 − 67
$V_{EQ} = 5,1 + 0,1 \times \frac{91}{91 + 67} = 5,157$			

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Descriptors : sulphuric acid, chemical analysis, determination of content, chlorides, potentiometric analysis.

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Sulphuric acid for industrial use — Determination of chlorides content — Potentiometric method

Acide sulfurique à usage industriel — Dosage des chlorures — Méthode potentiométrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 2877 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in September 1972.

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

Austria
Belgium
Czechoslovakia
Egypt. Arab Rep. of
France
Germany
Hungary
India
Ireland
Israel
Italy
Mexico
Netherlands
New Zealand
Poland
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South Africa, Rep. of
Sweden
Switzerland
Thailand
Turkey
United Kingdom
U.S.S.R.

No Member Body expressed disapproval of the document.

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Sulphuric acid for industrial use — Determination of chlorides content — Potentiometric method

1 SCOPE

This International Standard specifies a potentiometric method for the determination of the chlorides content of sulphuric acid for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having chlorides contents equal to or greater than 0,000 3 % (*m/m*), expressed as hydrochloric acid.

3 PRINCIPLE

Potentiometric titration of the chloride (Cl^-) ions with silver nitrate solution in a sulphuric acid medium, using a silver measurement electrode and a calomel reference electrode.

4 REAGENTS

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

4.1 Sulphuric acid, ρ approximately 1,30 g/ml, about 40 % (*m/m*) solution.

Carefully add about 29 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to 70 ml of water and, after cooling, dilute to 100 ml and mix.

4.2 Silver nitrate, approximately 0,1 N solution.

Dissolve 8,5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution in a brown glass bottle.

4.3 Silver nitrate, approximately 0,01 N solution.

Take 50 ml of the silver nitrate solution (4.2), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

4.4 Silver nitrate, approximately 0,004 N solution.

Take 20 ml of the silver nitrate solution (4.2), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

4.5 Potassium chloride, 0,1 N standard reference solution.

Weigh, to the nearest 0,000 1 g, 3,727 6 g of potassium chloride, previously dried for 1 h at 130 °C and cooled in a desiccator. Dissolve in a little water, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.6 Potassium chloride, 0,01 N standard reference solution.

Take 50,0 ml of the standard reference potassium chloride solution (4.5), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.7 Potassium chloride, 0,004 N standard reference solution.

Take 20,0 ml of the standard reference potassium chloride solution (4.5), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Potentiometric titration apparatus, comprising :

5.1.1 Potentiometer, sensitivity 2 mV, covering the range − 500 to + 500 mV.

5.1.2 Calomel electrode, fitted with a safety reservoir, filled with saturated potassium chloride solution.

5.1.3 Bridge, containing sulphuric acid, ρ approximately 1,48 g/ml, connected to the calomel electrode (5.1.2).

5.1.4 Silver electrode.

5.2 Magnetic stirrer, with a polytetrafluorethylene (PTFE)-coated rod.

5.3 Burette, with fine-pointed tip, graduated in 0,05 ml divisions.

6 PROCEDURE

Select the reagent solutions and test portion according to the expected chlorides content, as indicated in the following table.

Expected chlorides content, expressed as a percentage by mass of HCl	Silver nitrate solution	Standard reference potassium chloride solution	Test portion
Above 0,000 3 % up to and including 0,01 %	0,004 N (4.4)	0,004 N (4.7)	10 to 30 g, weighed to the nearest 0,01 g
Above 0,01 % up to and including 0,1 %	0,01 N (4.3)	0,01 N (4.6)	1 to 10 g, weighed to the nearest 0,001 g
Above 0,1 %	0,1 N (4.2)	0,1 N (4.5)	1 to 3 g, weighed to the nearest 0,001 g

6.1 Calibration of the silver nitrate solution.

Take 5,00 ml and 10,00 ml respectively of the appropriate standard reference potassium chloride solution and place in two low-form beakers of convenient capacity (for example 100 ml). Carry out the following titration on the contents of each beaker.

Introduce the rod of the magnetic stirrer (5.2) into the beaker and place the beaker in a container of convenient capacity (basin of about 20 cm diameter, for example); surround the beaker with crushed ice, to the level of the liquid, so as to equalize the temperature conditions around the beaker. Place the container and beaker on the magnetic stirrer (5.2) and set the stirrer in motion.

Place a thermometer in the beaker and control the temperature at between 10 and 20 °C during the titration by the occasional addition of crushed ice to the container.

Add to the beaker, in small portions, 50 ± 2 ml of the sulphuric acid solution (4.1), previously cooled to between 10 and 20 °C.

Immerse the silver electrode (5.1.4) and the free end of the bridge (5.1.3) in the solution, connect the electrodes to the potentiometer (5.1.1) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add the silver nitrate solution having the same normality as that of the standard reference potassium chloride solution used, from the burette (5.3), in 1 ml portions. After each addition, await the stabilization of the potential.

Note the volumes added and the corresponding values of the potential in the first two columns of a table.

When approaching the end point, continue the addition of the silver nitrate solution, in portions of 0,2 ml for the 0,004 N solution, 0,1 ml for the 0,01 N solution or 0,05 ml for the 0,1 N solution.

In a third column of the table, note the successive increments ($\Delta_1 E$) of the potential E. In a fourth column, note the differences ($\Delta_2 E$), positive or negative, between the potential increments ($\Delta_1 E$).

The end of the titration corresponds to the addition of the 0,2 ml, 0,1 ml or 0,05 ml portion of the silver nitrate solution which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume (V_{EQ}) of the silver nitrate solution corresponding to the end of the reaction, use the formula :

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 is the volume, in millilitres, of the silver nitrate solution immediately lower than the volume which gives the maximum increment of $\Delta_1 E$;

b is the last value of $\Delta_2 E$ which is positive;

B is the sum of the absolute values of the final positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (see example in annex);

V_1 is the volume, in millilitres, of the final portion of silver nitrate solution added (0,2, 0,1 or 0,05, as appropriate).

6.2 Determination

6.2.1 *Test portion*

Weigh, to the precision indicated in the table, a mass of test sample between 1 and 30 g, depending on the expected chlorides content, into a dry low-form beaker of convenient capacity (for example 100 ml).

6.2.2 *Titration*

Introduce a well-dried magnetic stirring rod (5.2) into the beaker and place the beaker in a container of convenient capacity (basin of about 20 cm diameter, for example); surround the beaker with crushed ice to the level of the liquid. Add water to the container, so as to equalize the temperature conditions around the beaker.

Place the container and beaker on the magnetic stirrer (5.2) and set the stirrer in motion.

Place a thermometer in the beaker and control the temperature at between 10 and 20 °C during the whole of the period of preparation of the test solution and titration, by the occasional addition of crushed ice to the container.

Add to the beaker, in small portions, 50 ± 2 ml of the sulphuric acid (4.1), previously cooled to between 10 and 20 °C.

Immerse the silver electrode (5.1.4) and the free end of the bridge (5.1.3) in the beaker, connect the electrodes to the potentiometer (5.1.1) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add the silver nitrate solution from the burette (5.3) in 1 ml portions. After each addition, await the stabilization of the potential.

To obtain the end point of the titration, follow the instructions given in 6.1 taking account, in the calculation, of the additions of the silver nitrate solution being in portions of 0,2 ml, 0,1 ml or 0,05 ml, according to the concentration of the solutions used.

7 EXPRESSION OF RESULTS

The chlorides content, expressed as a percentage by mass of hydrochloric acid (HCl), is given by the formula

$$\frac{m_1 \times 5 \times (V_4 - 2V_3 + V_2)}{m_0 \times (V_2 - V_3)} \times 100$$

where

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

m_1 is the mass, in grams, of hydrochloric acid corresponding to 1 ml of the silver nitrate solution (= 0,000 146 for 0,004 N solutions, = 0,000 365 for 0,01 N solutions, and = 0,003 65 for 0,1 N solutions);

V_2 is the value, in millilitres, of V_{EQ} corresponding to the titration of 10 ml of the standard reference potassium chloride solution;

V_3 is the value, in millilitres, of V_{EQ} corresponding to the titration of 5 ml of the standard reference potassium chloride solution;

V_4 is the value, in millilitres, of V_{EQ} corresponding to the determination;

5 is the difference, in millilitres, between the two volumes of standard reference potassium chloride solution used for the calibration.

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.

ANNEX

EXAMPLE

Volume of silver nitrate solution V	Potential E	$\Delta_1 E$	$\Delta_2 E$
ml	mV		
4,8	436	17	
5,0	453	30	+ 13
5,2	483	24	− 6
5,4	507	12	− 12
5,6	519		

$$V_{EQ} = 5{,}0 + 0{,}2 \times \frac{13}{13 + 6} = 5{,}134$$