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ISO RECOMMENDATION R 906

HYDROCHLORIC ACID FOR INDUSTRIAL USE

DETERMINATION OF SULPHATE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 906, *Hydrochloric acid for industrial use – Determination of sulphate content – Barium sulphate gravimetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Based on detailed work on this question carried out by the Technical Committee, a Draft ISO Recommendation was adopted in 1965.

In June 1967, this Draft ISO Recommendation (No. 1176) was circulated to all the ISO Member Bodies. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Iran	South Africa, Rep. of
Belgium	Ireland	Spain
Bulgaria	Israel	Switzerland
Chile	Italy	Thailand
Cuba	Japan	Turkey
Czechoslovakia	Korea, Dem. P. Rep. of	U.A.R.
France	Netherlands	United Kingdom
Germany	New Zealand	U.S.S.R.
Hungary	Poland	Yugoslavia
ICAITI*	Portugal	
India	Romania	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

* Instituto Centroamericano de Investigación y Tecnología Industrial (Costa Rica, Guatemala, Honduras, Nicaragua, El Salvador, Panama).

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HYDROCHLORIC ACID FOR INDUSTRIAL USE

DETERMINATION OF SULPHATE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of sulphate content of hydrochloric acid for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of sulphates in hydrochloric acid for industrial use for SO_4^{2-} contents greater than 0.1 % (m/m).

3. PRINCIPLE

Precipitation of SO_4^{2-} ions as barium sulphate in dilute hydrochloric acid medium.
Filtration of the solution, ignition of the precipitate at 600 to 800 °C and weighing.

4. REAGENTS

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

- 4.1 *Hydrochloric acid*, approximately $d = 1.18$, 37 % (m/m) or 12 N solution, free from sulphate ions.
- 4.2 *Sulphuric acid*, approximately $d = 1.84$, 96 % (m/m) or 36 N solution.
- 4.3 *Ammonium hydroxide*, approximately $d = 0.91$, 25 % (m/m) or 13 N solution.
- 4.4 *Barium chloride, dihydrate* ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), 100 g/l solution.
- 4.5 *Silver nitrate*, 5 g/l solution.
Dissolve 0.5 g of silver nitrate in water and dilute to 100 ml.
- 4.6 *Methyl orange*, 0.5 g/l solution.
Dissolve 0.05 g of methyl orange in water and dilute to 100 ml.

5. APPARATUS

Ordinary laboratory apparatus and

- 5.1 *Weighing bottle*, with ground glass stopper, capacity about 10 ml.
- 5.2 *Platinum crucible*, approximately 30 mm deep with a diameter of 30 mm at the top.

6. PROCEDURE

6.1 Test portion

Fill the weighing bottle (5.1) with the test sample and take a test portion of approximately 5 to 10 g according to the presumed sulphate content, weighing by difference to the nearest 10 mg.

Transfer the test portion to a beaker of suitable capacity (400 ml for example), containing 100 ml of water.

6.2 Determination

Dilute the solution to about 150 ml, add two drops of the methyl orange solution (4.6) and neutralize with the ammonium hydroxide solution (4.3).

Then add 1 ml of the hydrochloric acid solution (4.1), bring to the boil and add drop by drop 20 ml of the barium chloride solution (4.4). (The addition should take about 3 minutes.)

Bring to the boil and keep boiling for 2 minutes; place on a boiling water bath and leave for 2 hours, remove from the bath and leave to stand for about 16 hours.

Filter on an ashless slow-speed filter paper (pore diameter between 0.4 and 1 μm approximately), and wash with boiling water until the filtrate is free from chlorides: the filtrate should remain clear on addition of a few drops of the silver nitrate solution (4.5).

Place the filter paper and its contents in the platinum crucible (5.2), previously weighed after igniting in a furnace at 600 to 800 °C and cooling in a desiccator.

Dry in an oven at about 110 °C, and carefully char the filter paper at a low temperature to ensure that the paper does not burst into flames, then ignite in a furnace at 600 to 800 °C.

Remove the crucible, add a drop of the sulphuric acid solution (4.2) and again ignite. Remove the crucible, place in a desiccator, allow to cool and weigh.

7. EXPRESSION OF RESULTS

Sulphate content, expressed as SO_4 , is given as a percentage, by mass, by the following formula:

$$\frac{A \times 0.4115 \times 100}{E}$$

where

- A is the mass, in grammes, of the barium sulphate precipitate;
- 0.4115 is the conversion factor from barium sulphate to SO_4 ;
- E is the mass, in grammes, of the test portion.