

INTERNATIONAL STANDARD**906**

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Hydrochloric acid for industrial use — Determination of sulphate content — Barium sulphate gravimetric method*Acide chlorhydrique à usage industriel — Dosage des sulfates — Méthode gravimétrique à l'état de sulfate de baryum*

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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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 906-1968 and found it technically suitable for transformation. International Standard ISO 906 therefore replaces ISO Recommendation R 906-1968, to which it is technically identical.

ISO Recommendation R 906 had been approved by the member bodies of the following countries :

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

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

Hydrochloric acid for industrial use — Determination of sulphate content — Barium sulphate gravimetric method

1 SCOPE

This International Standard specifies a barium sulphate gravimetric method for the determination of the sulphate content of hydrochloric acid for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having a sulphate (SO_4) content equal to or greater than 0,1 % (*m/m*).

3 PRINCIPLE

Precipitation of the sulphate in a test portion as barium sulphate in dilute hydrochloric acid medium. Separation of the precipitate, ignition at $800 \pm 25^\circ\text{C}$ and weighing.

4 REAGENTS

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

4.2 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) or approximately 36 N solution.

4.3 Ammonium hydroxide, ρ approximately 0,91 g/ml, about 25 % (*m/m*) or approximately 15 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 nitric solution.

Dissolve 0,5 g of silver nitrate in a little water, add 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, dilute to 100 ml with water and mix.

4.6 Methyl orange, 0,5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Weighing bottle, of capacity about 10 ml, with ground glass stopper.

5.2 Platinum crucible, of upper diameter approximately 30 mm and depth approximately 30 mm, with lid.

5.3 Electric oven, capable of being controlled at $110 \pm 2^\circ\text{C}$.

5.4 Electric furnace, capable of being controlled at $800 \pm 25^\circ\text{C}$.

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 0,001 g.

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

6.2 Determination

Dilute the solution to about 150 ml, add a few drops of the methyl orange solution (4.6) and neutralize with the ammonium hydroxide solution (4.3). Then acidify by adding 3 ml of the hydrochloric acid solution (4.1). Bring to the boil, stirring constantly and add, drop by drop, 25 ml of the barium chloride solution (4.4). (The addition should take about 90 s.)

Keep boiling for 2 min, stirring constantly. Place on a boiling water bath and leave for 2 h, remove from the bath and leave to stand for about 16 h. Filter on an ashless slow-speed filter paper (pore diameter between 0,4 and $1\ \mu\text{m}$ approximately) of diameter approximately 90 mm.

Wash the precipitate on the filter paper with boiling water until 10 ml of the filtrate remain clear for 5 min after the addition of 10 ml of the silver nitrate solution (4.5).

Place the filter paper and its contents in the platinum crucible (5.2), previously weighed to the nearest 0,000 1 g after igniting in the furnace (5.4), controlled at 800 ± 25 °C, and cooling in a desiccator to ambient temperature.

Place the crucible and its contents in the oven (5.3), controlled at 110 ± 2 °C, until complete desiccation. Then ignite in the furnace (5.4), carefully at first to char the filter paper, ensuring that the paper does not burst into flames, then at 800 ± 25 °C for 15 min. Allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 1 g.

If, however, the ignited precipitate has a greyish appearance, indicating the presence of graphitic carbon, moisten it with 1 drop of the sulphuric acid solution (4.2), place again in the furnace and again ignite at 800 ± 25 °C for 15 min, allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 1 g.

7 EXPRESSION OF RESULTS

The sulphate content, expressed a percentage by mass of SO_4 , is given by the formula

$$\frac{m_1 \times 0,4115 \times 100}{m_0} = \frac{41,15 \times m_1}{m_0}$$

where

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

m_1 is the mass, in grams, of the barium sulphate precipitate obtained;

0,4115 is the conversion factor from BaSO_4 to SO_4 .

8 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used,
- the results and the method of expression used,
- any unusual features noted during the determination,
- any operation not included in this International Standard or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO HYDROCHLORIC ACID FOR INDUSTRIAL USE

ISO 904 – Determination of total acidity – Titrimetric method.

ISO 905 – Evaluation of hydrochloric acid concentration by measurement of density.

ISO 906 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 907 – Determination of sulphated ash – Gravimetric method.

ISO 908 – Determination of oxidizing or reducing substances – Titrimetric method.

ISO/R 909 – Determination of iron content – 2,2'-Bipyridyl spectrophotometric method.

ISO 2762 – Determination of soluble sulphates – Turbidimetric method.

ISO 5785 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.