



Hydrochloric acid for industrial use — Determination of oxidizing or reducing substances content — Titrimetric method

Acide chlorhydrique à usage industriel — Dosage des matières oxydantes ou des matières réductrices — Méthode titrimétrique

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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 908 was developed by Technical Committee ISO/TC 47, *Chemistry*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 908-1968, which 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, F. R.	New Zealand	USSR
Hungary	Poland	Yugoslavia

No member body had expressed disapproval of the document.

Hydrochloric acid for industrial use — Determination of oxidizing or reducing substances content — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the content of oxidizing or reducing substances in hydrochloric acid for industrial use.

The method is applicable to products having oxidizing substances contents, expressed as chlorine (Cl), or reducing substances contents, expressed as sulphur dioxide (SO₂), equal to or greater than 0,001 % (m/m).

2 Principle

Detection of the presence of either oxidizing or reducing substances by a preliminary qualitative test. Iodometric determination directly (in the case of oxidizing substances) or indirectly (in the case of reducing substances).

3 Reagents

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

3.1 Potassium iodide, crystals.

3.2 Hydrochloric acid, $\rho \approx 1,19$ g/ml, about 38 % (m/m) solution, with oxidizing substances content, expressed as chlorine (Cl), or reducing substances content, expressed as sulphur dioxide (SO₂), lower than 0,000 2 % (m/m).

3.3 Potassium iodide, 100 g/l solution.

3.4 Sodium thiosulphate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}^{(1)}$.

3.5 Iodine, approximately 12,70 g/l solution, containing at least 25 g of potassium iodide per litre.

3.6 Starch solution, freshly prepared.

Triturate 1,0 g of soluble starch with 5 ml of water, and, while stirring, pour the mixture into 100 ml of boiling water (if necessary, boil for a few minutes); then cool.

This solution can be kept for 2 weeks only.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Weighing bottle, capacity approximately 60 ml, provided with a ground glass stopper.

4.2 Conical flasks, capacity 500 ml, provided with ground glass stoppers.

5 Procedure

5.1 Preliminary test

Place about 20 ml of the test sample in a 100 ml conical flask, add 50 ml of water, one crystal of the potassium iodide (3.1) and 0,5 ml of the starch solution (3.6) and stir.

If a blue colour appears, follow the procedure specified in 5.4. If no colour appears, follow the procedure specified in 5.5.

5.2 Test portion

Take from the weighing bottle (4.1), filled with some of the test sample, a test portion of approximately 50 g, weighing by difference to the nearest 0,01 g.

1) Hitherto expressed as "0,1 N standard volumetric solution".

5.3 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same reagents as used for the determination, but replacing the test portion (5.2) by an equivalent quantity of the hydrochloric acid solution (3.2).

5.4 Determination of oxidizing substances

Transfer the test portion (5.2) to one of the conical flasks (4.2) containing 100 ml of water. Stopper the flask and cool to ambient temperature.

Add 10,0 ml of the potassium iodide solution (3.3) to the conical flask. Stopper the flask, shake it and allow it to stand for 2 min in the dark and then add 1 ml of the starch solution (3.6).

Titrate the liberated iodine with the sodium thiosulphate solution (3.4), until the blue colour disappears.

5.5 Determination of reducing substances

Transfer the test portion (5.2), with cooling, to one of the conical flasks (4.2) containing 100 ml of water and an exactly measured volume of the iodine solution (3.5), for example 10,0 ml. Stopper and shake the flask.

Titrate the excess of iodine with the sodium thiosulphate solution (3.4). When the colour of the solution becomes pale yellow, add 1 ml of the starch solution (3.6) and continue the titration until the blue colour disappears.

6 Expression of results

6.1 Oxidizing substances

The oxidizing substances content, expressed as a percentage by mass of chlorine (Cl), is given by the formula

$$\frac{(V_1 - V_0) \times 0,003\,55 \times 100}{m} = \frac{0,355 (V_1 - V_0)}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used for the blank test (5.3);

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used for the determination (5.4);

m is the mass, in grams, of the test portion (5.2);

0,003 55 is the mass, in grams, of chlorine corresponding to 1 ml of sodium thiosulphate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$.

6.2 Reducing substances

The reducing substances content, expressed as a percentage by mass of sulphur dioxide (SO_2), is given by the formula

$$\frac{(V_0 - V_2) \times 0,003\,203 \times 100}{m} = \frac{0,3203 (V_0 - V_2)}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used for the blank test (5.3);

V_2 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used for the determination (5.5);

m is the mass, in grams, of the test portion (5.2);

0,003 203 is the mass, in grams, of sulphur dioxide corresponding to 1 ml of sodium thiosulphate solution $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction factor has to be applied.

7 Test report

The test report shall include the following particulars :

- an identification of the sample;
- 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 909 — Determination of iron content — 1,10-Phenanthroline spectrophotometric method.
- ISO 2762 — Determination of soluble sulphates — Turbidimetric method.
- ISO 5785 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

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