

International Standard



5790

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Inorganic chemical products for industrial use — General method for determination of chloride content — Mercurimetric method

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Foreword

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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 5790 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Austria	Germany, F.R.	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Bulgaria	Israel	Switzerland
Chile	Italy	Turkey
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Egypt, Arab Rep. of	Korea, Rep. of	USSR
France	New Zealand	Yugoslavia

No member body expressed disapproval of the document.

Inorganic chemical products for industrial use — General method for determination of chloride content — Mercurimetric method

1 Scope

This International Standard specifies a general mercurimetric method for the determination of chloride content.

2 Field of application

The method is applicable to inorganic chemical products for industrial use. It enables quantities of chloride (Cl^-) exceeding 1 mg in the test portion to be determined with an absolute error of 0,2 to 0,5 mg, according to the concentration of the standard volumetric solution used.

The determination is not affected by the elements or compounds listed in annex A when they are present in quantities up to the specified limits.

In certain cases, this annex contains instructions for the treatment to be applied to reduce or eliminate interferences.

3 Principle

Conversion of Cl^- ions into weakly ionized mercury(II) chloride, using highly ionized standard volumetric mercury(II) nitrate solution. Detection of the end-point by reaction of the excess of mercury(II) nitrate with 1,5-diphenylcarbazone used as an indicator, by the appearance of a violet colour.

4 Reagents

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

4.1 Nitric acid, $\rho \approx 1,40$ g/ml, approximately 68 % (m/m) solution, of which the chloride content, expressed as chlorine (Cl), is not greater than 0,5 mg/kg.

4.2 Nitric acid, approximately 126 g/l solution, prepared by dilution of the nitric acid solution (4.1).

4.3 Sodium hydroxide, approximately 80 g/l solution of which the chloride content, expressed as chlorine (Cl) is not greater than 5 mg/kg.

4.4 Sodium chloride, standard reference solution, $c(\text{NaCl}) = 0,1 \text{ mol/l}^{(1)}$.

Weigh, to the nearest 0,000 1 g, 5,844 2 g of sodium chloride, previously dried for 1 h at approximately 500 °C and cooled in a desiccator, and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.5 Sodium chloride, standard reference solution, $c(\text{NaCl}) = 0,05 \text{ mol/l}^{(2)}$.

Place 100,0 ml of the standard reference solution (4.4) in a 200 ml one-mark volumetric flask, dilute to the mark and mix.

4.6 Sodium chloride, standard reference solution, $c(\text{NaCl}) = 0,02 \text{ mol/l}^{(3)}$.

Place 100,0 ml of the standard reference solution (4.4) in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.7 Standard end-point matching solution.

Prepare this solution at the time of use. Do not use the solution for more than 30 min after preparation.

Place the same volume of water as in the test solution (6.2.1) in a 500 ml conical flask, add 3 drops of the bromophenol blue solution (4.11) and the nitric acid solution (4.2), drop by drop, until the colour changes from blue to yellow. Add an excess of 3 drops of this acid, 0,5 to 1,0 ml of the diphenylcarbazone solution (4.12) and, from the burette (5.1), the minimum volume of the standard volumetric mercury(II) nitrate solution (4.8, 4.9 or 4.10) necessary to change the colour of the solution from yellow to mauve.

1) Previously 0,1 N

2) Previously 0,05 N

3) Previously 0,02 N

4.8 Mercury(II) nitrate, standard volumetric solution, $c[1/2 \text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}] = 0,1 \text{ mol/l}^{(1)}$.

Use this solution to determine quantities greater than 20 mg of chloride (Cl^-) in the test portion.

4.8.1 Preparation of the solution

Weigh $10,85 \pm 0,01 \text{ g}$ of mercury(II) oxide (HgO) and dissolve in 10 ml of the nitric acid solution (4.1). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

NOTE — This solution may also be prepared by dissolution of a mass of 17,13 g of mercury(II) nitrate monohydrate [$\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$] in water acidified with 1 ml of the nitric acid solution (4.1).

Standardize this solution following the procedure specified in 4.8.2 and adjust it, if necessary, to exactly 0,1 mol/l.

4.8.2 Standardization of the solution

Place 40,0 ml of the standard reference sodium chloride solution (4.4), 160 ml of water and 3 drops of the bromophenol blue solution (4.11) in a 500 ml conical flask. Add the nitric acid solution (4.2), drop by drop, until the colour of the indicator changes from blue to yellow and then add an excess of 3 drops of this acid and the volume of the diphenylcarbazone solution (4.12) added for the standard end-point matching solution (4.7). Titrate (see note in 6.2.2) with the mercury(II) nitrate solution (4.8.1) to be standardized until the colour matches the mauve of the standard end-point matching solution and deduct the volume of the mercury(II) nitrate solution (4.8.1) added during the preparation of this standard end-point matching solution (generally 1 drop).

The volume corresponding to exactly 0,1 mol/l is 40,0 ml.

NOTE — Store the residual solution obtained from the titration and treat it as specified in annex B, so as to avoid the pollution of waste water by the mercury.

4.9 Mercury(II) nitrate, standard volumetric solution, $c[1/2 \text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}] = 0,05 \text{ mol/l}^{(2)}$.

Use this solution to determine quantities between 1 and 20 mg of chloride (Cl^-) in the test portion.

4.9.1 Preparation of the solution

Use one of the two following methods :

- dilute 100 ml of the solution (4.8.1) to the mark in a 200 ml one-mark volumetric flask and mix, or
- weigh $5,43 \pm 0,01 \text{ g}$ of mercury(II) oxide (HgO) and dissolve in 10 ml of the nitric acid solution (4.1). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. [See note in

4.8.1 for the use of 8,565 g of mercury(II) nitrate monohydrate.]

Standardize this solution following the procedure specified in 4.9.2 and adjust it, if necessary, to exactly 0,05 mol/l.

4.9.2 Standardization of the solution

Place 10,0 ml of the standard reference sodium chloride solution (4.5), 190 ml of water and 3 drops of the bromophenol blue solution (4.11) in a 500 ml conical flask. Add the nitric acid solution (4.2), drop by drop, until the colour of the indicator changes from blue to yellow and then add an excess of 3 drops of this acid and the volume of the diphenylcarbazone solution (4.12) added for the standard end-point matching solution (4.7). Titrate (see note in 6.2.2) with the mercury(II) nitrate solution (4.9.1) to be standardized until the colour matches the mauve of the standard end-point matching solution and deduct the volume of the mercury(II) nitrate solution (4.9.1) added during the preparation of this standard end-point matching solution (generally 2 drops).

The volume corresponding to exactly 0,05 mol/l is 10,00 ml.

4.10 Mercury(II) nitrate, standard volumetric solution, $c[1/2 \text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}] = 0,02 \text{ mol/l}^{(3)}$.

Analysts who can easily detect the diphenylcarbazone colour change can take advantage of this solution to determine quantities between 1 and 5 mg of chloride (Cl^-) in the test portion.

4.10.1 Preparation of the solution

Use one of the two following methods :

- dilute 100 ml of the solution (4.8.1) to the mark in a 500 ml one-mark volumetric flask and mix, or
- weigh $2,18 \pm 0,01 \text{ g}$ of mercury(II) oxide (HgO) and dissolve in 10 ml of the nitric acid solution (4.1). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. [See note in 4.8.1 for the use of 3,43 g of mercury(II) nitrate monohydrate.]

Standardize this solution following the procedure specified in 4.10.2 and adjust it, if necessary, to exactly 0,02 mol/l.

4.10.2 Standardization of the solution

Place 5,00 ml of the standard reference sodium chloride solution (4.6), 195 ml of water and 3 drops of the bromophenol blue solution (4.11) in a 500 ml conical flask. Add the nitric acid solution (4.2), drop by drop, until the colour changes from blue to yellow and then add an excess of 3 drops of this acid and the volume of the diphenylcarbazone solution (4.12) added for the standard end-point matching solution (4.7). Titrate (see note in 6.2.2) with the mercury(II) nitrate solution to be standardized

1) Previously 0,1 N

2) Previously 0,05 N

3) Previously 0,02 N