
International Standard



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Sodium hydroxide for industrial use — Determination of mercury content — Dithizone photometric method

Hydroxyde de sodium à usage industriel — Dosage du mercure — Méthode photométrique à la dithizone

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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.

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

Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Israel	Switzerland
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Australia
Italy

Sodium hydroxide for industrial use – Determination of mercury content – Dithizone photometric method

1 Scope and field of application

This International Standard specifies a dithizone photometric method for the determination of the mercury content of sodium hydroxide for industrial use.

The method is applicable to liquid or solid products having mercury (Hg) contents greater than 0,05 mg/kg.

NOTE – The alternative flameless atomic absorption spectrometric method for the determination of mercury content, specified in ISO 5993, is intended for use in laboratories where such equipment is available.

2 References

ISO 3195, *Sodium hydroxide for industrial use – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.*

ISO 5790, *Inorganic chemical products for industrial use – General method for determination of chloride content – Mercurimetric method.*

ISO 5993, *Sodium hydroxide for industrial use – Determination of mercury content – Flameless atomic absorption spectrometric method.*

3 Principle

Oxidation of the mercury contained in a test portion to mercury(II) ions by potassium permanganate in the presence of sulphuric acid.

Reduction of the excess oxidant by hydroxylammonium chloride, suppression of interference by copper, iron and silver with excess hydroxylammonium chloride and Na₂EDTA followed by extraction in chloroform of the complex formed with dithizone in the pH range 0 to 2.

Photometric measurement of the complex in the presence of excess dithizone at a wavelength of about 490 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade having the lowest possible mercury content, and only distilled water or water of equivalent purity.

4.1 Chloroform.

4.2 Sulphuric acid, approximately 490 g/l solution.

4.3 Acetic acid, approximately 360 g/l solution.

Prepare this solution by diluting glacial acetic acid, ρ approximately 1,05 g/ml.

4.4 Disodiummethylenedinitrilotetracetate dihydrate (Na₂EDTA), 7,45 g/l solution.

Dissolve 7,45 g of the Na₂EDTA in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

4.5 Potassium permanganate, 40 g/l solution.

4.6 Hydroxylammoniumchloride (NH₂OH.HCl), 100 g/l solution.

4.7 (1,5-Diphenyl)thiocarbazon (Dithizone), 150 mg/l solution in chloroform.

Prepare this solution using the chloroform (4.1) and store it in a sealed dry, airtight flask made of dark glass at a temperature below 25 °C. Renew after 2 weeks.

4.8 (1,5-Diphenyl)thiocarbazon (dithizone), 3 mg/l solution in chloroform.

Take 5,0 ml of the dithizone solution (4.7), place in a 250 ml one-mark volumetric flask, dilute to the mark with the chloroform (4.1) and mix.

Prepare this solution on the day of use and store away from light and heat.

4.9 Mercury, standard solution corresponding to 1,000 g of Hg per litre.

Dissolve 1,354 g of mercury(II) chloride (HgCl_2) in 25 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, about 38 % (*m/m*). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

Store this solution in a dark, cool place and renew after 2 months.

1 ml of this standard solution contains 1 mg of Hg.

4.10 Mercury, standard solution corresponding to 20 mg of Hg per litre.

Transfer 10,0 ml of the standard mercury solution (4.9) to a 500 ml one-mark volumetric flask, add 10 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, dilute to the mark and mix.

Prepare this solution on the day of use.

1 ml of this standard solution contains 20 μg of Hg.

4.11 Mercury, standard solution corresponding to 1 mg of Hg per litre.

Transfer 10,0 ml of the standard mercury solution (4.10) to a 200 ml one-mark volumetric flask, add 5 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, dilute to the mark and mix.

Prepare this solution on the day of use.

1 ml of this standard solution contains 1 μg of Hg.

NOTE — In order to prevent pollution of waste water, collect solutions containing mercury salts and remove mercury following the instructions given in annex B of ISO 5790.

5 Apparatus

All new glassware or glassware not previously used for this determination, including flasks for reagents and samples, shall be washed as follows and rinsed with water after each operation :

- with brush and soap if the walls are likely to be greasy;
- with nitric acid solution, ρ approximately 1,42 g/ml;
- with a mixture, prepared at the time of washing, consisting of 4 volumes of sulphuric acid, approximately 100 g/l solution, and 1 volume of the potassium permanganate solution (4.5).

Condition the glassware thus washed by carrying out several preliminary determinations of mercury using mercury standard solutions until satisfactory results are obtained, before using it for actual determinations. Thereafter, use such glassware for mercury determination *only*.

5.1 Spectrophotometer, or

5.2 Photoelectric absorptiometer, fitted with filters providing maximum transmission at a wavelength of about 490 nm.

6 Sampling

Follow the procedure specified in ISO 3195, observing the additional precautions specified in 6.1 and 6.2.

6.1 Liquid products

As liquid products may contain small particles of metallic mercury, take a laboratory sample of about 1 litre at a place where the turbulence is sufficient to ensure, as far as possible, a representative sample.

Take the test sample immediately from the laboratory sample and oxidize, *at once*, following the procedure specified in 7.1.1 and 7.4.1.1 in order to avoid loss of mercury through evaporation or adsorption.

6.2 Solid products

The sampling of solid products, involves no particular difficulty as such products are obtained by evaporation of sodium hydroxide solutions, which removes any volatile mercury. The only precautionary requirement is the immediate oxidation of the test solution prepared for the determination, in order to avoid loss of mercury.

Take and treat the test portion following the procedure specified in 7.1.2 and 7.4.1.2.

7 Procedure

7.1 Test portion

7.1.1 Liquid samples

Weigh, to the nearest 0,1 g, a polyethylene flask of capacity 100 ml containing 10 ml of the potassium permanganate solution (4.5). Immediately after its preparation (see 6.1), transfer approximately 50 ml of the test sample (6.1) into the flask and again weigh the flask and contents to the nearest 0,1 g. Immediately follow the procedure specified in 7.4.1.1 (see 9.2).

7.1.2 Solid samples

Into a 400 ml beaker, weigh, to the nearest 0,1 g, about 20 g of the test sample. Immediately follow the procedure specified in 7.4.1.2.

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all reagents used for the sampling and the determination, but omitting the test portion.

7.3 Preparation of the calibration graph

7.3.1 Preparation of standard colorimetric solutions relating to measurements carried out with cells of 4 or 5 cm optical path length

Into the stems of series of four 500 ml separating funnels, insert a small, loose, plug of filter paper. To each funnel, add 30 ml of the sulphuric acid solution (4.2) and, using a burette, the volumes of the standard mercury solution (4.11) shown in the following table.

Standard mercury solution (4.11)	Corresponding mass of mercury
ml	µg
0*	0
2,0	2
5,0	5
10,0	10

* Compensation solution.

Treat each of these solutions as follows.

Dilute to approximately 300 ml, and add 1 ml of the hydroxylammonium chloride solution (4.6), 10 ml of the acetic acid solution (4.3) and 10 ml of the Na₂EDTA solution (4.4). Using a safety pipette, add 25,0 ml of the dithizone solution (4.8), shake vigorously for 1 min and allow to separate for 10 min.

Fill one of the photometer cells with the chloroform extract, taking care to avoid entrainment of water particles.

NOTE — The purpose of the filter paper plug is to remove entrained water from the chloroform extract.

7.3.2 Photometric measurements

Carry out the photometric measurements using either the spectrophotometer (5.1) at the wavelength of maximum absorption (about 490 nm) or the photoelectric absorptiometer (5.2) fitted with suitable filters, after having in each case adjusted the apparatus to zero absorbance against the chloroform (4.1). Deduct the absorbance of the compensation solution (7.3.1 — 0 term) from those of the standard colorimetric solutions (7.3.1).

7.3.3 Plotting the graph

Plot a graph having, for example, the masses of mercury, expressed in micrograms contained in the standard colorimetric solutions, as abscissae, and the corresponding values of absorbance as ordinates.

7.4 Determination

7.4.1 Preparation of the test solution

7.4.1.1 Liquid samples

Transfer the test portion (7.1.1) to a 600 ml beaker, add approximately 150 ml of water and allow to cool. Rinse the flask which

contained the test portion, using 25 ml of the sulphuric acid solution (4.2) to which 2 drops of the hydroxylammonium chloride solution (4.6) have been added, in order to dissolve any manganese hydroxide precipitate adhering to the walls of the flask. Pour this rinsing solution slowly and carefully into the 600 ml beaker, stirring continuously.

Then add, slowly and carefully, stirring continuously, a further 150 ml of the sulphuric acid solution (4.2), cover the beaker with a watch-glass and boil for about 10 min.

Allow to cool to room temperature, then add, drop by drop, the hydroxylammonium chloride solution (4.6) until decoloration of the permanganate takes place, and add an excess of 0,5 ml of this solution. Transfer the contents of the beaker quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark, mix and immediately follow the procedure specified in 7.4.2.

7.4.1.2 Solid samples

Treat the test portion (7.1.2) in the beaker as follows.

Dissolve in 100 ml of water, allow to cool to room temperature, add 5 ml of the potassium permanganate solution (4.5) and, slowly, carefully, and stirring continuously, 85 ml of the sulphuric acid solution (4.2). Cover the beaker with a watch-glass and boil for 10 min.

Allow to cool, carefully add, drop by drop, the hydroxylammonium chloride solution (4.6) until decoloration of the permanganate takes place, and add an excess of 0,25 ml of this solution in order to dissolve any manganese hydroxide precipitate adhering to the walls of the flask. Transfer the contents of the beaker quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark, mix and immediately follow the procedure specified in 7.4.2.

7.4.2 Extraction of the mercury dithizonate

Using a safety pipette, take a volume of the test solution (7.4.1) not exceeding 100 ml and containing not more than 10 µg of mercury.

Transfer this aliquot portion to a 500 ml separating funnel, the stem of which contains a small, loose plug of filter paper. Dilute to approximately 300 ml, and add 1 ml of the hydroxylammonium chloride solution (4.6), 10 ml of the acetic acid solution (4.3) and 10 ml of the Na₂EDTA solution (4.4). Introduce, using a safety pipette, 25,0 ml of the dithizone solution (4.8), shake vigorously for 1 min and allow to separate for 10 min.

Fill one of the photometer cells with the chloroform extract, taking care to avoid entrainment of water particles.

NOTE — See the note at the end of 7.3.1.

7.4.3 Photometric measurement

Carry out the photometric measurements on the test solution (7.4.1) and on the blank test solution (7.2), following the procedure specified in 7.3.2, after having adjusted the instrument to zero absorbance against the chloroform (4.1).

8 Expression of results

By means of the calibration graph (7.3.3), determine the mass of mercury corresponding to the photometric measurement.

The mercury content, expressed as milligrams of mercury (Hg) per kilogram, is given by the formulae :

— for liquid products

$$\frac{(m_1 - m_2)}{1\ 000} \times \frac{500}{V} \times \frac{1\ 000}{m_0}$$

$$= \frac{500 (m_1 - m_2)}{V \times m_0}$$

— for solid products

$$\frac{(m_1 - m_2)}{1\ 000} \times \frac{250}{V} \times \frac{1\ 000}{m_0}$$

$$= \frac{250 (m_1 - m_2)}{V \times m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in micrograms, of mercury found in the aliquot portion of the test solution (7.4.1) used for the determination;

m_2 is the mass, in micrograms, of mercury found in the corresponding aliquot portion of the blank test solution;

V is the volume, in millilitres, of this aliquot portion of the test solution.

9 Notes on procedure

9.1 If it is not possible to take the test sample immediately, proceed as follows.

Record the mass of the laboratory sample. Shake the laboratory sample vigorously and then take the test sample. Empty the laboratory sample container and recover any mercury adhering to the walls by shaking mechanically for 1 h after addition of a mixture of the potassium permanganate solution (4.5) and the sulphuric acid solution (4.2). Determine the mercury content of this mixture and correct the result (clause 8) appropriately.

9.2 If it was not possible to carry out the determination immediately, some mercury may have been adsorbed by the walls of the flask. Under such conditions, apply a correction following the procedure specified in 9.1.

10 Test report

The test report shall include the following particulars :

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a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operations not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

Annex

ISO publications relating to sodium hydroxide for industrial use

- ISO 979 — Method of assay.
- ISO 980 — Determination of carbonates content — Gas-volumetric method.
- ISO 981 — Determination of chloride content — Mercurimetric method.
- ISO 982 — Determination of sulphate content — Barium sulphate gravimetric method.
- ISO 983 — Determination of iron content — 1,10-Phenanthroline photometric method.
- ISO 984 — Determination of silica content — Reduced silicomolybdic complex photometric method.
- ISO 985 — Determination of silica content — Gravimetric method by precipitation of quinoline molybdosilicate.
- ISO 986 — Determination of calcium — EDTA (d/sodium salt) complexometric method.
- ISO 3195 — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.
- ISO 3196 — Determination of carbon dioxide content — Titrimetric method.
- ISO 3197 — Determination of chloride content — Photometric method.
- ISO 3198 — Determination of sulphur compounds — Method by reduction and titrimetry.
- ISO 3697 — Determination of calcium and magnesium contents — Flame atomic absorption method.
- ISO 5992 — Determination of mercury content — Dithizone photometric method.
- ISO 5993 — Determination of mercury content — Flameless atomic absorption spectrometric method.

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