International Standard



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Sodium hydroxide for industrial use — Determination of mercury content — Dithizone photometric 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 5992 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in September 1977.

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

Austria Hungary Romania South Africa, Rep. of Belgium India Bulgaria Israel Switzerland Chile Kenya Turkey Czechoslovakia Korea, Rep. of United Kingdom Egypt, Arab Rep. of Netherlands USSR France **Philippines** Yugoslavia Germany, F. R. Poland

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 Disodiumethylenedinitrilotetraacetate dihydrate (Na_2EDTA), 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)thiocarbazone (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)thiocarbazone (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.