International Standard 5993

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Sodium hydroxide for industrial use — Determination of mercury content — Flameless atomic absorption spectrometric method

Hydroxyde de sodium à usage industriel — Dosage du mercure — Méthode par spectrométrie d'absorption atomique sans flamme

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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 5993 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 :

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

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Sodium hydroxide for industrial use — Determination of mercury content — Flameless atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flameless atomic absorption spectrometric 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 0,02 mg/kg.

NOTE — The alternative dithizone photometric method for the determination of mercury content, specified in ISO 5992, is intended for use in laboratories where flameless atomic absorption equipment is not 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 5992, *Sodium hydroxide for industrial use — Determination of mercury content — Dithizone photometric 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. Reduction of the mercury(II) ions to mercury by tin(II) chloride. Entrainment of the mercury in air or nitrogen and passage of the gaseous mixture through a measuring cell.

Measurement of the absorption at a wavelength of 253,7 nm, using an atomic absorption spectrometer.

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 Nitrogen, in a cylinder.

4.2 Sulphuric acid, approximately 490 g/l solution.

4.3 Potassium permanganate, 40 g/l solution.

4.4 Hydroxylammonium chloride ($NH_2OH.HCl$), 100 g/l solution.

4.5 Tin(II) chloride, 100 g/l solution in hydrochloric acid.

Dissolve 25 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 50 ml of hot hydrochloric acid solution, ϱ approximately 1,19 g/ml. Transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark with water and mix. Transfer this solution to a glass flask and purge it by bubbling the nitrogen (4.1) through for 5 min, then add a few particles of granular metallic tin to assist stabilization. Discard when turbidity appears.

4.6 Iodine, 2,5 g/l solution.

Dissolve 2,5 g of iodine and 30 g of potassium iodide in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

4.7 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, ϱ approximately 1,19 g/ml 38 % (m/m) solution. 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 cool, dark place and renew after 2 months.

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

4.8 Mercury, standard solutions corresponding to 1 and 0,1 mg of Hg per litre.

Prepare these solutions on the day of use by successive dilution of the standard solution (4.7) with an approximately 11 g/l hydrochloric acid solution. These solutions should be freshly prepared as their concentration can vary because of loss of mercury through evaporation or adsorption by the flask.

1 ml of each of these standard solutions respectively contains 1 µg and 0,1 µg of Hg.

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