
INTERNATIONAL STANDARD



3196

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Sodium hydroxide for industrial use — Determination of carbonates content — Titrimetric method

Hydroxyde de sodium à usage industriel — Dosage des carbonates — Méthode titrimétrique

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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 3196 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1973.

It has been approved by the Member Bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Israel	Switzerland
Chile	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia

No Member Body expressed disapproval of the document.

Sodium hydroxide for industrial use – Determination of carbonates content – Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the carbonates content of sodium hydroxide for industrial use.

The method is applicable to products having carbonates contents, expressed as CO_2 , greater than 0,01 % (m/m) :

Three cases, selected by a preliminary test, are envisaged :

1.1 Sodium hydroxide containing neither sulphides nor chlorates.

1.2 Sodium hydroxide containing sulphides.

1.3 Sodium hydroxide containing chlorates.

2 REFERENCE

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

3 PRELIMINARY TEST

3.1 Principle

Boiling of an acidified test sample, containing methyl orange and with a lead acetate paper strip in the vapour. The presence of sulphides produces blackening of the paper and the presence of chlorates decolorizes the methyl orange.

3.2 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity, free from carbon dioxide.

3.2.1 Hydrochloric acid, approximately 6 N solution.

3.2.2 Methyl orange, 0,5 g/l solution.

3.2.3 Lead acetate paper, cut into strips about 15 mm X 80 mm.

3.3 Procedure

Place about 20 g of the test sample in a 300 ml conical flask. Add 150 ml of water and 3 drops of the methyl orange solution (3.2.2). Neutralize with the hydrochloric acid solution (3.2.1) and add an excess of 5 ml of this acid.

Fix a lead acetate paper strip (3.2.3) inside the neck of the conical flask, curling it over the outside of the flask.

Boil the solution for 5 min and select the procedure to be followed according to the following table.

Appearance		Case number	Procedure
solution	paper		
red	white	1.1	Clause 4
red	blackened	1.2	Clause 5
colourless	white	1.3	Clause 6

4 SODIUM HYDROXIDE CONTAINING NEITHER SULPHIDES NOR CHLORATES

4.1 Principle

Liberation of carbon dioxide by acidification and heating. Entrainment in a flow of gas and absorption in an excess of barium hydroxide solution. Titration of the excess barium hydroxide with a standard volumetric hydrochloric acid solution, in the presence of thymolphthalein as indicator.

4.2 Reagents

Reagents listed in 3.2, and

4.2.1 Nitrogen, or air, free from carbon dioxide.

4.2.2 Barium hydroxide approximately 0,1 N solution.

Weigh about 15,0 g of barium hydroxide octahydrate $[\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}]$, dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Allow precipitated barium carbonate to settle out before use.

4.2.3 Hydrochloric acid, 0,1 N standard volumetric solution.