INTERNATIONAL STANDARD 5143

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Sodium carbonate for industrial use — Determination of sulphur compounds content — Method by reduction and titrimetry

Carbonate de sodium à usage industriel — Dosage des composés soufrés — Méthode par réduction et titrimétrie

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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 5143 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1976.

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

Austria	India	Spain
Belgium	Israel	Switzerland
Brazil	Korea, Rep. of	Thailand
Chile	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
France	Poland	Yugoslavia
Germany	Romania	
Hungary	South Africa, Rep. of	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Italy
U.S.S.R.

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

Printed in Switzerland

Sodium carbonate for industrial use — Determination of sulphur compounds content — Method by reduction and titrimetry

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method, by reduction and titrimetry, for the determination of sulphur compounds content of sodium carbonate for industrial use.

The method is applicable to products having a sulphur compounds content, expressed as sulphate (SO_4^{2-}), equal to or greater than 5 mg/kg.

2 REFERENCE

ISO 739, *Sodium carbonate for industrial use — Preparation and storage of test samples.*

3 PRINCIPLE

Progressive neutralization, with evolution of carbon dioxide, by adding slowly a mixture of hydriodic acid and hypophosphorous acid. Reduction of sulphur compounds to hydrogen sulphide by heating with this reducing solution.

Distillation of hydrogen sulphide, entrainment in a current of oxygen-free nitrogen and absorption in a mixture of acetone and sodium hydroxide solution.

Titration of the sulphide with a standard volumetric mercury(II) nitrate solution in the presence of dithizone as indicator.

4 REAGENTS

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

4.1 Acetone.

4.2 Nitrogen, free from oxygen.

4.4 Sodium hydroxide, approximately 10 N solution.

4.4 Sodium hydroxide, approximately 1 N solution.

4.5 Reducing solution.

Place the flask (F) of the apparatus (5.3), swirling after each addition :

- 50 ml of phosphinic (hypophosphorous) acid (H_3PO_2) solution, ρ approximately 1,21 g/ml, about 50 % (*m/m*) solution;
- 100 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution;
- 120 ml of hydriodic acid solution, ρ approximately 1,97 g/ml, about 67 % (*m/m*) solution.

Assemble the reflux condenser (G), flask (F) and inlet tube (H). Then, while passing a slow stream of the nitrogen (4.2), boil under reflux for 4 h.

Cool to ambient temperature, under a flow of the nitrogen, and store the reagent away from direct sunlight in an amber glass bottle, previously purged with the nitrogen, fitted with a ground glass stopper.

4.6 Sodium sulphate, 0,001 M (= 0,002 N) standard reference solution.

Weigh, to the nearest 0,001 g, 0,142 0 g of anhydrous sodium sulphate, previously dried at 110 °C and cooled in a desiccator. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this solution corresponds to 96 μg of SO_4.

4.7 Mercury(II) nitrate, 0,05 M (= 0,1 N) standard volumetric solution.

Weigh 10,85 ± 0,01 g of mercury(II) oxide (HgO), place in a beaker of suitable capacity (100 ml for example) and dissolve in 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution. Dilute the solution, transfer it quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.8 Mercury(II) nitrate, 0,001 M (= 0,002 N) standard volumetric solution.

Place 20,00 ml of the standard volumetric mercury(II) nitrate solution (4.7) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution just before use.

NOTE — The concentration of the solutions (4.7 and 4.8) prepared as described above are sufficiently exact, taking into consideration the small amounts of sulphur compounds to be determined. Standardization is therefore unnecessary.

In most laboratories, an exactly 0,05 M (= 0,1 N) standard volumetric mercury(II) nitrate solution will be available, this solution being commonly used for the mercurimetric determination of chlorides.