

Sampling and test for sodium carbonate for industrial use —

Part 5: Determination of sulphate content

[ISO title: Sodium carbonate for industrial use — Determination of sulphur compounds content — Method by reduction and titrimetry]

NOTE It is recommended that this Part be read in conjunction with the information in the “*General introduction*”, published separately as BS 6070-0.

UDC 661.833.622.546.33'264:543.24.062:543.226

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board and comes into effect on 27 February 1981

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The following BSI references relate to the work on this standard:
Committee reference CIC/22
Draft for comment 76/52715 DC

ISBN 0 580 12004 X

Amendments issued since publication

Amd. No.	Date of issue	Comments

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National foreword

This Part of BS 6070 is identical with ISO 5143 “*Sodium carbonate for industrial use — Determination of sulphur compounds content — Method by reduction and titrimetry*” published in 1977 by the International Organization for Standardization (ISO).

The National appendix to the Part of this standard describes a procedure to remove mercury from waste solutions from the analytical procedures described herein. This procedure is technically equivalent to that described in ISO 5790 “*Inorganic chemical products for industrial use — General method for determination of chloride content — Mercurimetric method*” (which has not been implemented as a British Standard).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “British Standard”.

Cross-reference. There is no British Standard corresponding directly to ISO 739 to which reference is made in 6.1. Technically equivalent information is given in BS 6070-0.

NOTE BS 3674 “*Specification for sodium carbonate (technical grades)*”, gives details for preparing a suitable test portion complying with the requirements of 6.1.

The standards listed in the Annex are intended for information only. Corresponding British Standards are listed in BS 6070-0.

Additional information

Water. Water complying with the requirements of clause 4 is specified in BS 3978 “*Water for laboratory use*”.

Hydrochloric acid. ρ approximately 1.18 g/ml, about 36 % (m/m) solution, which is the corresponding reagent available in the UK, is suitable for use in place of the solution specified in 4.5.

WARNING NOTES

1. *Mercury (II) nitrate*, specified in 4.7, is toxic (with danger of cumulative effects) and is irritant, particularly in the form of dust. Avoid breathing dust. Avoid contact with skin and eyes. Avoid pollution of waste water with mercury from waste solutions using, for example, the procedure for removal of mercury described in the National appendix to this Part of this standard.

2. *Hydriodic acid solution*, specified in 4.5, gives off harmful vapour, is toxic, corrosive and irritant. Avoid breathing vapour. Prevent contact with eyes and skin.

NOTE *Textual error.* In the first line of 4.5 “Place the flask” should be read as “Place in the flask”. A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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.

4.3 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 hydrochloric 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 $10,85 \pm 0,01$ 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.

4.9 (Phenylazo)thioformic acid, 2-phenylhydrazide (dithizone), 0,5 g/l solution in the acetone (4.1).

Renew this solution after 2 weeks.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Burette (E), of capacity 5 ml, graduated in 0,01 ml.

5.2 Apparatus for reduction and distillation, of which all the components are fitted together by means of ground glass joints. A typical apparatus is shown in Figure 1 and comprises

5.2.1 Conical flask (A) of capacity 100 ml.

5.2.2 Reflux condenser (B)

5.2.3 Absorption vessel (C), of height 100 mm and internal diameter 25 mm.

5.2.4 Dropping funnel (D), of capacity 50 ml.