BRITISH STANDARD

BS 7319-10: 1990

Analysis of sodium chloride for industrial use —

Part 10: Method for determination of pH and total alkalinity

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/22, upon which the following bodies were represented:

British Association for Chemical Specialities
Chemical Industries' Association
Man-made Fibres Producers' Committee
Soap and Detergent Industry Association
Textile Research Council (FRCA)

The following bodies were also represented in the drafting of the standard, through Technical Committee FAC/23:

AFRC Institute of Food Research
Creamery Proprietors' Association
Department of Trade and Industry (Laboratory of the Government Chemist)
Food and Drink Federation
Milk Marketing Board for Northern Ireland
Royal Association of British Dairy Farmers
Salt Manufacturers' Association

This British Standard, having been prepared under the direction of the Chemicals Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 30 September 1990

The following BSI references relate to the work on this standard:
Committee references CIC/22, FAC/23
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Contents

Foreword

BS 7319 has been prepared under the direction of the Chemicals Standards Policy Committee, at the request of Technical Committee FAC/23, Salt, primarily to provide appropriate methods for determination of vacuum salt for food use as specified in BS 998:1990. The methods for determination were previously published as appendices to BS 998:1969.

A list of the Parts of BS 7319 is given in Part 1.

This Part of BS 7319 is based upon a method developed on behalf of the European Committee for the Study of Salt. It supersedes Appendix F of BS 998:1969 which is withdrawn.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, 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

This Part of BS 7319 specifies a potentiometric method for the measurement of the pH of a sodium chloride solution, of concentration 100 g/L, and for the determination of total alkalinity.

The method is applicable to products of total alkalinity content, expressed as Na_2CO_3, of lower than 1 000 mg/kg.

NOTE The titles of the publications referred to in this Part of this British Standard are listed on the inside back cover.

2 Principle

The principles of this Part of BS 7319 are as follows:

a) the measurement, at 20 °C, of the pH of a sodium chloride solution of concentration 100 g/L in water, using a pH meter fitted with a glass electrode and a calomel reference electrode;

b) after the measurement of pH, the addition of a known excess volume of standard hydrochloric acid;

c) the removal of carbon dioxide by boiling;

d) checking that the pH of the solution is less than 3;

e) titrating the excess acid with a standard sodium hydroxide solution to pH 5, using a pH meter.

3 Reagents

3.1 *General.* Use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978 freed from carbon dioxide by boiling and cooling to room temperature, or by bubbling through with nitrogen or with carbon dioxide-free air for 10 min.

3.2 *Hydrochloric acid*, standard volumetric solution c(HCl) = 0.1 mol/L exactly.

3.3 *Sodium hydroxide/barium chloride solution*, standard volumetric solution, c(NaOH) = 0.1 mol/L exactly, free of carbonates.

Add to each litre of a solution of sodium hydroxide, c(NaOH) = 0.11 mol/L approximately, contained in an aspirator fitted with a guard tube to absorb atmospheric carbon dioxide, 10 mL of a solution containing 100 g/L $BaCl_2.2H_2O$. Mix the solution to react and leave it to stand until the precipitate of $BaCO_3$ has settled at the bottom of the aspirator. Adjust the concentration to 0.1 moL/L NaOH exactly.

3.4 *Sodium tetraborate buffer solution.* Dissolve 3.81 ± 0.01 g of sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$) in water, transfer the solution quantitatively to a 1 000 mL one-mark volumetric flask, dilute it to the mark with water and mix well. Protect this solution from carbon dioxide and prepare a fresh solution each month.

The pH of this buffer solution at 20 °C is 9.22.

3.5 *Potassium hydrogen phthalate buffer solution.* Dissolve 10.21 ± 0.01 g of potassium hydrogen phthalate ($KHC_8O_4H_4$) in water, transfer the solution quantitatively to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix well. Keep this solution in a well-sealed container and protect it from carbon dioxide.

The pH of this buffer solution at 20 °C is 4.00.

3.6 *Methyl red solution.* Dissolve 1 g of methyl red in 1 L of 95 % (*V*/*V*) ethanol.

4 Apparatus

4.1 *Ordinary laboratory apparatus*

4.2 *pH meter*, fitted with a glass measuring electrode and a calomel reference electrode, of sensitivity 0.05 pH units.

4.3 *Mechanical or magnetic stirrer*

4.4 *Burette*, capacity 10 mL, graduated in 0.01 mL.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.1 g, a test portion of approximately 25 g.

5.2 Preparation of the test solution

Introduce the test portion (see **5.1**) stirring slowly, with a mechanical or magnetic stirrer (**4.3**), into a 400 mL beaker containing 250 mL of water.

Continue stirring until dissolution is complete.

5.3 Calibration of the pH meter

Calibrate the pH meter (**4.2**) at 20 ± 1 °C using the buffer solutions (**3.4** and **3.5**). Rinse the electrodes well with water to remove all traces of the buffer solutions.

5.4 Measurement of pH

Introduce into the beaker the two electrodes of the pH meter and carry out the measurement at a controlled temperature of 20 ± 1 °C and with constant stirring, under the same conditions as the calibration (see **5.3**).

5.5 Determination of total alkalinity

Remove the two electrodes and the stirrer (**4.3**) and add 10.0 mL of the standard hydrochloric acid (**3.2**). Boil for 2 min and cool to 20 ± 1 °C.

Replace the two electrodes of the pH meter in the beaker. If the pH is not below 3.00 add a known further excess of the standard hydrochloric acid (**3.2**) and boil again. Introduce the stirrer (**4.3**) and titrate the excess acid with the standard sodium hydroxide/barium chloride solution (**3.3**) to pH 5.

Keep this solution for the blank test (see **5.6**).

NOTE If the determination of the pH is not to be carried out, replace the pH meter with five drops of methyl red solution (**3.6**) and titrate with a colour change from red to orange at the end point.

It is important to check that initially the pH was lower than 3, using indicator paper.

5.6 Blank test

Remove the two electrodes and the stirrer (**4.3**) from the solution obtained after the titration (see **5.5**), and add the same volume of standard hydrochloric acid used in the determination (see **5.5**). Boil for 2 min, cool to 20 ± 1 °C and back-titrate under the same conditions as **5.5**.

6 Expression of results

6.1 pH

Express the results of this measurement in pH units, to the nearest 0.1 pH unit.

6.2 Total alkalinity, expressed as Na_2CO_3

Calculate the total alkalinity, expressed as milligrams of Na_2CO_3 per kilogram of sodium chloride on a moisture free basis, using the following expression:

$$\left(\frac{0.53}{\mathrm{m}}\right)(\mathrm{V}_0 - V_1) \times 10^4 \times \left\{\frac{100}{(100 - H)}\right\}$$

where

V_0 is the volume of the standard sodium hydroxide/barium chloride solution (**3.3**) used for the blank test (see **5.6**) (in mL);

V_1 is the volume of the standard sodium hydroxide/barium chloride solution (**3.3**) used for the determination (see **5.5**) (in mL);

m is the mass of the test portion (see **5.1**) (in g);

H is the moisture content determined in accordance with BS 7319-2 [in % (*m*/*m*)].

Express the results to the nearest milligram per kilogram.

7 Precision

Analyses carried out on two samples gave the statistical results shown in Table 1 and Table 2, each laboratory having given two results obtained by the same operator on two test samples.

Table 1 — Results of pH analysis

Sample	Number of laboratories	Mean	Repeatability σ_r	Reproducibility σ_R
Vacuum salt	16	8.90	0.04	0.33
Sea salt	16	8.85	0.05	0.32

Table 2 — Results of alkalinity analysis

Sample	Number of laboratories	Mean	Repeatability σ_r	Reproducibility σ_R
Vacuum salt	13	100	8	11
Sea salt	13	210	7	15

Publication(s) referred to

BS 998, *Specification for vacuum salt for food use*[1)].
BS 3978, *Specification for water for laboratory use.*
BS 7319, *Analysis of sodium chloride for industrial use.*
BS 7319-1, *Method for determination of sodium chloride content.*

[1)] Referred to in the foreword only.

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