BRITISH STANDARD

BS 7319-11:
1990

Analysis of sodium chloride for industrial use —

Part 11: Method for determination of iron content

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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Amendments issued since publication

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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 It supersedes Appendix K of BS 998:1969 which is withdrawn.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, 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 photometric method, using 1,10-phenanthroline, for the determination of iron in sodium chloride.

The method is applicable to products having iron contents equal to or greater than 1 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 reduction of trivalent iron by hydroxylammonium chloride; and the formation of a coloured complex between divalent iron and 1,10-phenanthroline in a buffered medium;

b) the photometric measurement of the coloured complex at a wavelength of approximately 510 nm.

3 Reagents

3.1 *General.* Use only reagents of recognized analytical grade and only water complying with grade 3 of BS 3978.

3.2 *Hydrochloric acid*, $\rho = 1.19$ g/mL, 37 % (*m*/*m*) solution approximately.

3.3 *Acetic acid glacial*, $\rho = 1.05$ g/mL, 99 % to 100 % (*m*/*m*) solution.

3.4 *Hydroxylammonium chloride solution.* Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in 1 L of water.

3.5 *1,10-phenanthroline solution.* Dissolve 2.5 g of 1,10-phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$) in 1 L of water.

NOTE This compound may be replaced by 2.1 g of 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$).

3.6 *Sodium acetate solution.* Dissolve 500 g sodium acetate trihydrate ($CH_3COONa.3H_2O$) in water and dilute to 1 L.

3.7 *Buffer solution.* Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) (**3.6**) in approximately 500 mL of water. Add 240 mL of the acetic acid (**3.3**) to the solution. Transfer quantitatively to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix well. This solution has a pH of 4.9.

3.8 *Iron stock solution*, corresponding to 0.20 g/L Fe.

Dissolve 1.4043 g of ammonium iron (II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$], weighed to the nearest 0.0001 g, in 200 mL of water. Add 20 mL of sulphuric acid solution (ρ = about 1.84 g/mL) and cool to ambient temperature. Transfer to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix well.

1 mL of this solution contains 200 μg of Fe.

3.9 *Iron standard solution*, corresponding to 0.010 g/L Fe.

Transfer 25.0 mL of the standard iron solution (**3.8**) to a 500 mL one-mark volumetric flask, dilute to the mark with water and mix well.

1 mL of this solution contains 10 μg of Fe.

Prepare this solution at the time of use.

4 Apparatus

4.1 *Ordinary laboratory equipment*

4.2 *Spectrometer, or photometer*, fitted with filters which provide a transmission maximum between 500 nm and 520 nm.

4.3 *pH meter equipped with a glass electrode and a calomel reference electrode, or pH indicator paper*, covering the pH range 3.5 to 4.2 and indicating in 0.2 pH unit intervals.

5 Procedure

CAUTION. Ensure that no trace of iron is introduced during the analysis by the reagents, the water or the apparatus, especially the apparatus used in the preparation of the initial sample.

5.1 General

Wash all new glassware used in the determination as follows:

a) with brush and detergent if the sides are greasy;

b) with concentrated nitric acid, followed by several rinses with water.

5.2 Test portion

Weigh, to the nearest 0.01 g, a test portion of approximately 10 g.

5.3 Blank test

At the same time as the determination carry out a blank test following the same procedure and using the same quantities of all the reagents used for the determination but omitting the test portion.

5.4 Preparation of the calibration curve

5.4.1 *Preparation of standard colorimetric solutions*

Add the volumes of the standard iron solution (**3.9**) given in Table 1 to a series of five 100 mL one-mark volumetric flasks.

Dilute the solution in each flask to approximately 50 mL with water, and then add the following:

a) 0.5 mL of the hydrochloric acid solution (**3.2**);

b) 5 mL of the hydroxylammonium chloride solution (**3.4**);

c) 5 mL of the 1,10-phenanthroline solution (**3.5**);

d) 25 mL of the buffer solution (**3.7**).

Dilute to the mark with water, mix well and allow the solution to stand for 10 min.

Table 1 — Mass of iron in standard solutions

Standard iron solution (3.9)	Corresponding mass of iron
mL	μg
0[a]	—
2.5	25
5.0	50
10.0	100
15.0	150

[a] Zero standard.

5.4.2 *Photometric measurements*

Adjust the apparatus to zero absorbance with respect to water prior to each measurement. Measure the absorbance of each solution in a cell of optical path length 4 cm (see note) using the spectrometer (**4.2**) set at the wavelength of maximum absorbance (approximately 510 nm), or the photometer fitted with the filters specified. Deduct the absorbance of the zero standard from the absorbance obtained for the other standards.

NOTE Cells of path length 5 cm may be used if 4 cm cells are not available.

5.4.3 *Plotting the calibration curve*

Plot a graph having, for example, the masses of iron contained in 100 mL of the standard colorimetric solutions (see **5.4.1**), expressed in micrograms, as abscissae and the corresponding values of absorbance as ordinates. Prepare a new calibration curve whenever a new stock solution is prepared.

5.5 Determination

5.5.1 *Preparation of the test solution*

Add the test portion (see **5.2**) to a suitable size beaker (for example 400 mL) and add 25 mL of hydrochloric acid solution (**3.2**) and approximately 100 mL of water. Boil until the sample has completely dissolved, and for a further 5 min. Cool to ambient temperature. Transfer the solution quantitatively to a 500 mL one-mark volumetric flask, dilute to the mark with water and mix well.

5.5.2 *Adjustment of pH*

5.5.2.1 *Selection of aliquot portions*

Take two aliquot portions of the test solution (see **5.5.1**) according to the expected iron content, as indicated in Table 2.

Table 2 — Expected iron content

Expected iron content	Volume of aliquot portion
mg/kg Fe	mL
100	50
100 to 250	25
250 to 500	10
500	5

Add the first aliquot portion to a beaker of suitable size (for example 100 mL), and the second to a 100 mL one-mark volumetric flask.

5.5.2.2 *Preliminary pH check*

Dilute the aliquot portion in the beaker to about 50 mL.

Then add the following:

a) 5 mL of the hydroxylammonium chloride solution (**3.4**);

b) 5 mL of the 1,10 phenanthroline solution (**3.5**);

c) 25 mL of the buffer solution (**3.7**).

Check the pH of the solution with the pH meter or paper indicator (**4.3**). The value shall be between 3.5 and 4.2. If the value is not in this range, adjust the pH by adding slowly, stirring after each addition, either the necessary amount of the acetic acid solution (**3.3**) or the necessary amount of the sodium acetate solution (**3.6**). Record the amount of reagent needed to adjust the pH and reject the solution.

5.5.3 *Formation of the coloured complex*

Add the same amounts of all the reagents used for the preliminary test (see **5.5.2.2**) to the aliquot portion in the volumetric flask. Dilute to 100 mL, mix and allow to stand for 10 min.

5.5.4 *Photometric measurements*

Adjust the apparatus to zero absorbance with respect to water prior to each measurement. Measure the absorbance of the test solution (see **5.5.1**) and the blank solution (see **5.3**) in a cell of optical path length 4 cm (see note to **5.4.2**), using the spectrometer (**4.2**) set at the wavelength of maximum absorbance (approximately 510 nm), or the photometer fitted with the filters described in **4.2**. Deduct the absorbance of the blank from the absorbance obtained for the sample.

NOTE If 5 cm cells were used to prepare the calibration graph, use 5 cm cells here also.

6 Expression of results

Using the calibration curve (**5.4.3**) determine the mass, in micrograms, of iron in the test solution and in the blank solution, corresponding to the absorbance of the zero standard.

Calculate the iron content, expressed as milligrams of per kilogram on a moisture free basis, using the following expression.

$$\frac{m_1 - m_2}{m_0} \times \frac{500}{V} \times \frac{100}{(100 - H)}$$

where

m_0 is the mass of the test portion (see **5.2**) (in g);

m_1 is the mass of iron found in the test solution (see **5.5**) (in μg);

m_2 is the mass of iron found in the blank test solution (see **5.3**) (in μg);

V is the volume of the aliquot portion of the test solution (see **5.5.2.1**) (in mL);

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

7 Precision

A collaborative study to determine the precision of this method has not been undertaken.

blank

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*.
BS 7319-2, *Method for determination of moisture content*.

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[1] Referred to in the foreword only.

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