BS 7319-12:
1990

Incorporating
Amendment No. 1

Analysis of sodium chloride for industrial use —

Part 12: Methods for determination of anti-caking additives content of salt for food use

Confirmed
December 2011

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (FAC/-) to Technical Committee FAC/23, upon which the following bodies were represented:

AFRC Institute of Food Research
Chemical Industries' Association
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 reference 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.

This Part of BS 7319 has been produced under the direction of the Food and Agriculture Standards Policy Committee. It is based upon a method developed on behalf of the European Committee for the Study of Salt. It supersedes Appendix Q of BS 998:1969 which is withdrawn.

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

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 two methods for the determination of water-soluble hexacyanoferrate (II) (anti-caking additives) in salt for food use.

The two methods are applicable to products having levels of hexacyanoferrate (II), as follows.

a) *Direct method*. For 2.5 mg to 40 mg of hexacyanoferrate (II) per kilogram of salt.

b) *Filtration method*. For 0.25 mg to 4 mg of hexacyanoferrate (II) per kilogram of salt.

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) Hexacyanoferrate (II) with iron (II), in acid solution forms the complex iron (II) hexacyanoferrate (II) which, in the presence of iron (III), oxidizes immediately to Prussian blue. The absorbance of this complex is determined at the wavelength of maximum absorbance, (approximately 700 nm).

b) For the lower levels of potassium hexacyanoferrate (II) the Prussian blue is filtered on a membrane filter. After re-dissolving it in potassium hydroxide solution, the Prussian blue is reformed in a much reduced volume.

NOTE 1 Hexacyanoferrate (III) is determined simultaneously.

NOTE 2 The intensity of the colour depends on the amount of alkali metal ions present in the solution.

3 Reagents

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

3.2 *Sulphuric acid*, standard volumetric solution, $c(\frac{1}{2}H_2SO_4)$ = 1.0 mol/L exactly.

3.3 *Potassium hydroxide*, standard volumetric solution, $c(KOH)$ = 0.05 mol/L exactly.

3.4 *Sodium chloride*, hexacyanoferrate-free

3.5 *Iron (II)/Iron (III) solution*. Dissolve in water, in a 1 000 mL one-mark volumetric flask, 200 g of ammonium iron (II) sulphate hexahydrate, $(NH_4)_2SO_4.FeSO_4.6H_2O$, and 25 g of hydrated ammonium iron (III) sulphate, $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$. Add 100 mL of the sulphuric acid solution (**3.2**).

Dilute to the mark with water and mix well. Filter the solution and store in a dark bottle.

3.6 *Potassium hexacyanoferrate (II) stock solution*, containing 1 mg/mL of potassium hexacyanoferrate. Dissolve in water, in a 1 000 mL one-mark volumetric flask 1.000 g of potassium hexacyanoferrate (II) trihydrate, $K_4Fe(CN)_6.3H_2O$.

Add 5 mL of the potassium hydroxide solution (**3.3**). Dilute to the mark with water and mix well. Store this stock solution in a dark bottle for a maximum of 1 month.

3.7 *Potassium hexacyanoferrate (II) standard solution*, containing 50 μg/mL of $K_4Fe(CN)_6.3H_2O$.

Take 50.0 mL of the stock solution (**3.6**) and transfer to a 1 000 mL one-mark volumetric flask. Add 5 mL of the potassium hydroxide solution (**3.3**). Dilute to the mark with water and mix well.

Prepare this solution at the time of use.

4 Apparatus

4.1 *Ordinary laboratory apparatus*

4.2 *Spectrometer, or photometer*, fitted with filters having a maximum transmission between 690 nm and 710 nm.

4.3 *Membrane filter*, of approximately 50 mm diameter and pore size maximum of 0.3 μm.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.1 g, a test portion of approximately 100 g.

5.2 Blank test

At the same time as the determination carry out a blank test, following the same procedure and using the same quantities of reagents and the same quantity of the sodium chloride (**3.4**), but omitting the test portion.

5.3 Preparation of the calibration curve

5.3.1 *Preparation of standard colorimetric solutions*

Into a series of five 100 mL one-mark volumetric flasks, introduce 10.0 g of the sodium chloride (**3.4**) and the volumes of the potassium hexacyanoferrate (II) solution (**3.7**) as shown in Table 1.

Dilute the solutions to approximately 50 mL with water, swirl until the crystals are dissolved and then add 10.0 mL of the potassium hydroxide solution (**3.3**).

Add, in the order given, 5 mL of the sulphuric acid solution (**3.2**) and 5 mL of the iron (II)/iron (III) solution (**3.5**). Mix the solution after each addition. Dilute to the mark with water and mix well.

Table 1 — Mass of potassium hexacyanoferrate in standard solutions

Volume of the standard solution (3.7)	Corresponding mass of $K_4Fe(CN)_6.3H_2O$
mL	μg
0[a]	0
2	100
5	250
10	500
15	750

[a] Zero standard.

5.3.2 *Photometric measurements*

Adjust the apparatus to zero absorbance with respect to water prior to each measurement. Measure the absorbance of each solution, using either the spectrometer at the wavelength of maximum absorbance (approximately 700 nm) or the photometer (**4.1**), fitted with the filters specified. Deduct the absorbance of the zero standard from the absorbance obtained for each standard colorimetric solution (see **5.3.1**).

5.3.3 *Plotting the calibration curve*

Plot a graph having, for example, the masses of $K_4Fe(CN)_6.3H_2O$ contained in 100 mL of the standard colorimetric solutions (see **5.3.1**), expressed in micrograms, as abscissae, and the corresponding values of absorbance as ordinates. Prepare a new calibration curve whenever a new stock solution (**3.6**) is prepared.

5.4 Determination

5.4.1 *Preparation of the test solution*

5.4.1.1 *Direct method* (see clause **1**)

Add the test portion (see **5.1**) to a 500 mL one-mark volumetric flask. Dissolve in water, dilute to the mark with water and mix well. Take 50.0 mL of this solution and place in a 100 mL one-mark volumetric flask. Add 10 mL of the potassium hydroxide solution (**3.3**). Follow the procedure described in **5.4.2**.

5.4.1.2 *Filtration method*

Transfer the test portion (see **5.1**) to a 600 mL beaker, dissolve in 450 mL of water and add 10 mL of the sulphuric acid solution (**3.2**) and 25 mL of the iron (II)/iron (III) solution (**3.5**). Mix the solution after each addition. Allow it to stand for 15 min.

Filter the precipitate under vacuum on the membrane filter (**4.3**). Wash twice with 15 mL portions of water. Place the filter in 10 mL of the potassium hydroxide solution (**3.3**) and dissolve the precipitate.

Transfer the solution quantitatively to a 100 mL one-mark volumetric flask and add to it 10.0 g of the sodium chloride (**3.4**). Dilute the solution to approximately 60 mL with water, swirl until the crystals are dissolved.

Follow the procedure described in **5.4.2**.

5.4.2 *Determination*

Add, in the order given, 5 mL of the sulphuric acid solution (**3.2**) and 5 mL of the iron (II)/iron (III) solution (**3.5**). Mix after each addition. Dilute to the mark with water and mix well.

Allow the solution to stand for 15 min.

Adjust the apparatus to zero absorbance with respect to water prior to each measurement.

Measure the absorbance of the solution, using either the spectrometer set at the wavelength of maximum absorbance (approximately 700 nm) or the photometer (**4.2**) fitted with the filters specified.

6 Expression of results

Using the calibration curve (see **5.3.3**), determine the masses, in micrograms, of potassium hexacyanoferrate in the test solution and in the blank solution corresponding to the absorbance of the test solution.

Calculate the water-soluble hexacyanoferrate (II) content, expressed as milligrams per kilogram of hexacyanoferrate $Fe(CN)_6$ on a moisture free basis, using one of the following expressions, as appropriate.

a) *Direct method*

$$\left(\frac{m_1 - m_2}{m_0}\right) \times \frac{500}{50} \times \left\{\frac{100}{(100 - H)}\right\} \times \frac{211.96}{422.39}$$

b) *Filtration method*

$$\left(\frac{m_1 - m_2}{m_0}\right) \times \left\{\frac{100}{(100 - H)}\right\} \times \left(\frac{211.96}{422.39}\right)$$

where

m_1 is the mass of potassium hexacyanoferrate found in the test solution (in μg) (see **5.4.2**);

m_2 is the mass of potassium hexacyanoferrate found in the blank test solution (**5.2**) (in μg);

m_0 is the mass of the test portion (in μg);

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

7 Precision

Comparative analyses on two samples carried out by 11 laboratories, have given the following statistical information.

Mean (mg/kg)	3.09
Standard deviation for repeatability (r)	0.083
Standard deviation for reproducibility (R)	0.536

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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