BRITISH STANDARD

BS 7319-6:
1990

Analysis of sodium chloride for industrial use —

Part 6: Method for determination of cadmium 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 (FCRA)

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 all 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.

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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 describes a flame atomic absorption spectrometric (AAS) method for the determination of total cadmium in sodium chloride.

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 mineralization of cadmium and dissolution of the sample in nitric acid solution;

b) the complexation of the cadmium and certain other metals by ammonium pyrrolidine-1-carbodithioate, and extraction of the metal complexes into chloroform and back-extraction into nitric acid;

c) aspiration of the nitric acid solution into an acetylene-air flame;

d) measurement of the absorbance at a wavelength of about 228.8 nm, using an atomic absorption spectrometer fitted with a cadmium hollow-cathode lamp.

3 Reagents

3.1 *General.* Unless otherwise stated, use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978.

3.2 *Nitric acid,* $\rho = 1.40$ g/mL, 945 g/L HNO_3 approximately.

3.3 *Chloroform,* $\rho = 1.481$ g/mL.

3.4 *Ammonium pyrrolidine-1-carbodithioate (APCD) solution,* special reagent for AAS.

Dissolve 20 g AAS grade APCD in 1 L of water and filter.

Prepare this solution daily.

3.5 *Ammonia solution,* $\rho = 0.9$ g/mL.

3.6 *Diammonium hydrogen citrate solution,* 113 g/L solution.

Dissolve 124 g diammonium hydrogen citrate in a 500 mL one-mark volumetric flask, dilute to the mark with water and mix.

Purify this solution as follows.

Shake 100 mL of the solution with 10 mL of the APCD solution (**3.4**) and extract three times respectively with 10 mL, 5 ml and 5 mL of the chloroform (**3.3**).

Back-extract the combined organic extracts into nitric acid, as follows. After the phase separation following each extraction, run the organic layer into a previously dried 100 mL separating funnel (**4.5**) containing 1.0 mL nitric acid (**3.2**).

a) Repeat the extraction and back extraction procedure until, when aspirating the nitric extracts into the acetylene-air flame, the absorbance measured at approximately 228.8 nm is lower than 10 times the absorbance of 20 mL of chloroform extracted into a mixture of 1.0 mL nitric acid (**3.2**) and 9.0 mL water.

b) Transfer 100 mL of the purified solution into a 200 mL one-mark volumetric flask, add 20 mL ammonia solution (**3.5**), dilute to the mark with water and mix.

3.7 *Cadmium, stock solution,* corresponding to 1 000 mg/L.

Weigh, to the nearest 0.0001 g, 1.000 g of cadmium metal strip or granules (99.99 %) and dissolve them in 10 mL of the nitric acid (**3.2**). Transfer the solution quantitatively to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix.

NOTE 1 1 mL of this solution contains 1 mg of lead.

NOTE 2 A commercial stock solution for atomic absorption spectrometry of the same strength may also be used if available.

3.8 *Cadmium, standard solution,* corresponding to 5 mg/L.

Transfer 5.0 mL of the stock solution (**3.7**) to a 1 000 mL one-mark volumetric flask, add 1.0 mL of the nitric acid (**3.2**), and then dilute to the mark with water and mix.

4 Apparatus

4.1 *Ordinary laboratory apparatus*

4.2 *Atomic absorption spectrometer,* fitted with an acetylene-air burner and scale expansion facility.

NOTE In order to reduce the interferences caused by non-atomic absorption the use of a background correction device is recommended.

4.3 *Cadmium hollow-cathode lamp*

4.4 *Separating funnels of* 500 mL *capacity.*

4.5 *Separating funnels of* 100 mL *capacity.*

5 Procedure

5.1 General

NOTE Ensure that no traces of cadmium are introduced during the analysis, taking care to avoid any contamination by traces of cadmium during the sampling process.

Wash all the glassware to be employed for this determination as follows, rinsing very carefully with water after each operation:

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

b) with diluted nitric acid (1 : 2).

5.2 Test portion

Weigh, to the nearest 1 g, a test portion of 250 g.

5.3 Test solution

Transfer the test portion (see **5.2**) into a 2 000 mL beaker, add 850 mL of water and stir to dissolve.

5.4 Blank test

Transfer 850 mL water into a 2 000 mL beaker to act as a blank solution.

5.5 Mineralization

To each 2 000 mL beaker (see **5.3** and **5.4**) add 10.0 mL of the nitric acid (**3.2**) and stir.

Add some glass beads, heat to the boil and boil gently for 30 min.

Ensure that the total volume never falls below 800 mL, adding more water if necessary. (The same volume to both beakers.)

Allow the solution to cool and transfer it quantitatively to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix.

5.6 Determination

5.6.1 *Preparation of the standard matching solutions*

5.6.1.1 *Preparation of the standard matching solution for the test solution*

Transfer into each of four 500 mL separating funnels (**4.4**) 200 mL of the mineralized test solution (see **5.5**) and the volumes of the standard cadmium solution (**3.8**) indicated in Table 1.

Table 1 — Mass of cadmium in standard solutions

Standard matching solution reference number	Volume of the standard cadmium solution (3.8)	Corresponding mass of cadmium
	mL	µg
1[a]	0.00	0.0
2	0.50	2.5
3	1.00	5.0
4	2.00	10.0

[a] Test solution.

Complex and extract cadmium as described in **5.6.2.2** to **5.6.2.6**.

5.6.1.2 *Preparation of the standard matching solutions for the blank*

Transfer to four 50 mL one-mark volumetric flasks the volumes of the standard cadmium solution (**3.8**) indicated in Table 1.

To each flask add 5.0 mL of nitric acid (**3.2**), dilute to the mark with water and mix.

Carry out the procedure described in **5.6.4**.

5.6.2 *Complexation and extraction of cadmium*

5.6.2.1 Transfer 200 mL of the mineralized blank solution (see **5.5**) to a 500 mL separating funnel (**4.4**).

5.6.2.2 Carry out the procedure with this 500 mL separatory funnel and the four 500 mL separating funnels from **5.6.1.1** as follows.

a) Add 20.0 mL of the diammonium hydrogen citrate solution (**3.6**).

b) Add 5.0 mL of the APCD solution (**3.4**), shake for 30 s.

c) Add 10.0 mL of the chloroform (**3.3**), shake vigorously for 1 min.

5.6.2.3 After phase separation run the organic layer into a previously dried 100 mL separating funnel (**4.5**) containing 1.0 mL of nitric acid (**3.2**).

5.6.2.4 Repeat this extraction procedure twice, using 5 mL of chloroform (**3.3**) each time and combining the organic phases in the separating funnel (**4.5**).

5.6.2.5 Shake the separating funnel vigorously for 30 s, add 9.0 mL of water and shake vigorously for 1 min.

5.6.2.6 When the layers have separated discard the lower organic layer and transfer the upper aqueous layer to a dry tube.

NOTE The solution thus obtained may also be used for the determination of lead. (See BS 7319-8.)

5.6.3 *Apparatus setting*

Equip the spectrometer (**4.1**) with the cadmium lamp (**4.2**). Set the lamp current, the slit and the pressure of acetylene and air according to the instrument manufacturer's instructions.

Adjust the wavelength to that of the maximum absorption at approximately 228.8 nm.

5.6.4 *Spectrometric measurements*

Aspirate all the final solutions (see **5.6.2**) and the standard matching solutions for the blank (see **5.6.1.2**) into the acetylene-air flame in the following order, and at the same time, determine the absorbance of each.

a) The four standard matching solutions for the blank test solution (see Table 1).

b) The final blank test solution (**5.4**) after the complexation and extraction.

c) The three final standard matching solutions for the test solution after the complexation and extraction of cadium (numbers 2, 3 and 4 from Table 1).

d) The final test solution after the complexation and extraction of cadium (number 1 form Table 1).

Aspirate water after each measurement.

6 Expression of results

6.1 Calculation

6.1.1 *Calibration factors*

6.1.1.1 *Calibration factors for the test solution*

The calibration factors (f_1) of the three standard matching solutions for the test solution (see Table 1) is given by the following formula:

$$f_1 = \frac{m_1}{A_1 - A_2}$$

where

m_1 is the mass of cadmium added to the standard matching solution (see numbers 2, 3 and 4 from Table 1) (in μg);

A_1 is the absorbance obtained for each final standard matching solution;

A_2 is the absorbance of the final test solution.

6.1.1.2 *Calibration factor for the blank test solution*

The calibration factor (f_3) of the three standard matching solutions for the blank test (see **5.6.1.2**) is given by the following formula:

$$f_3 = \frac{m_1}{A_3 - A_4}$$

where

m_1 is the mass of cadmium added to the standard matching solution (see numbers 2, 3 and 4 from Table 1) (in μg);

A_3 is the absorbance obtained for each standard matching solution;

A_4 is the absorbance of the standard matching solution.

6.1.2 *Average calibration factors*

The average calibration factors are the arithmetic mean of each group of the three calibration factors found in **6.1.1.1**.

6.1.3 *Cadmium content*

The cadmium content, expressed in milligrams of cadmium (Cd) per kilogram of sodium chloride on a moisture free basis, is given by the following formula:

$$\frac{1}{m}\left[\left\{(5A_2 \times f_{1m}) - (A_5 \times f_{3m})\right\}\left\{\frac{100}{(100 - H)}\right\}\right]$$

where

A_2 is the absorbance measured for the final test solution;

A_5 is the absorbance measured for the final blank test solution;

f_{1m} is the average calibration factor (see **6.1.2**) for the test solution;

f_{3m} is the average calibration factor (see **6.1.2**) for the blank test solution.

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

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

7 Precision

Analyses carried out on three samples led to the statistical results shown in Table 2, each laboratory having furnished results obtained by the same operator performing two analyses on each sample.

Table 2 — Statistical results of sodium chloride analysis

Sample	Number of laboratories	Mean	Standard deviation for	
			Repeatability σ_r	Reproducibility σ_R
		mg Cd/kg sample		
Rock salt	14	0.002	0.0010	0.0011
Vacuum salt	15	0.001	0.0054	0.0104
Sea salt	15	0.011	0.0022	0.0102

blank

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

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[1] Referred to in the foreword only.

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