BRITISH STANDARD

**BS 7319-8:
1990**

Analysis of sodium chloride for industrial use —

Part 8: Method for determination of lead 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

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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 It supersedes Appendix N of BS 998:1969 which is withdrawn.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 describes a flame atomic absorption spectrometric (AAS) method for the determination of total lead 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 dissolution of the sample in nitric acid solution;

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

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

d) the measurement of the absorbance at a wavelength of approximately 283.3 nm, using an atomic absorption spectrometer fitted with a lead 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/mL HNO_3 approximately.

3.3 *Chloroform*, $\rho = 1.48$ g/mL.

3.4 *Ammonium pyrrolidine-1-carbodithioate solution*, special reagent for AAS. Dissolve 20 g of 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 of diammonium hydrogen citrate in a 500 mL one-mark volumetric flask, dilute to the mark with water and mix.

Purify this solution as follows.

a) 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**).

b) After the phase separation, following each extraction, back-extract the combined organic extracts into nitric acid by running the organic layer into a previously dried 100 mL separating funnel (**4.5**) containing 1.0 mL of the nitric acid (**3.2**).

c) Repeat the extraction and back-extraction procedure until, when aspirating the nitric acid extracts into the acetylene-air flame, the absorbance measured at approximately 283.3 nm is lower than 10 times the absorbance of 20 mL of chloroform extracted with a mixture of 1.0 mL of the nitric acid (**3.2**) and 9.0 mL of water.

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

3.7 *Lead stock solution*, corresponding to 1 000 mg/L Pb. Weigh, to the nearest 0.0001 g, 1.598 g of lead nitrate, $Pb(NO_3)_2$, grind it and dissolve it in 10 mL of the nitric acid (**3.2**). Transfer the solution quantitively 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 0.001 g of lead.

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

3.8 *Lead standard solution*, corresponding to 10 mg/L Pb. Transfer 10.0 mL of the lead stock solution (**3.5**) to a 1 000 mL one-mark volumetric flask, add 1.0 mL of 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 interferences caused by non-atomic absorption the use of a background correction device recommended.

4.3 *Lead hollow-cathode lamp*

4.4 *Separating funnels, 500 mL.*

4.5 *Separating funnels, 100 mL.*

5 Procedure

5.1 General

NOTE Ensure that no traces of lead are introduced during the analysis, taking care to avoid any contamination during the sampling process and particularly at the grinding stage.

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**) to a 2 000 mL beaker, add 850 mL of water and swirl 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 boiling 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 solutions 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 lead solution (**3.8**) indicated in Table 1.

Complex and extract lead 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 lead standard solution indicated in Table 1.

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

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

5.6.2 *Complexation and extraction of lead*

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

Table 1 — Mass of lead in standard solutions

Standard matching solution reference number	Volume of the standard lead solution (3.8)	Corresponding mass of lead
	mL	μg
1[a]	0	0
2	2.5	25
3	5.0	50
4	10.0	100

[a] Test solutions.

5.6.2.2 Carry out the procedure with this 500 mL separating funnel (see **5.6.2.1**) and the four from **5.6.1.1** in the following way.

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

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

c) Add 10.0 mL of the chloroform (**3.3**) and 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 the nitric acid (**3.2**).

5.6.2.4 Repeat this extraction procedure twice, using 5 mL of the chloroform (**3.2**) 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 cadmium. (See BS 7319-6.)

5.6.3 *Apparatus setting*

Equip the spectrometer (**4.2**) with the lead hollow-cathode lamp (**4.3**). 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 283.3 nm.

5.6.4 *Spectrometric measurement*

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 (see **5.4**) after the complexation and extraction of lead.

c) The three final standard matching solutions for the test solution after the complexation and extraction of lead.

d) The final test solution after **5.6.2** (number 1 from 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 lead 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 lead 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 *Lead content*

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

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

where

A_2 is the absorbance measured for the final test solution;

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

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

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

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.

7 Precision

Analyses carried out on three samples led to the statistical results given 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 Pb/kg sample		
Rock salt	15	0.045	0.0566	0.1299
Vacuum salt	14	0.025	0.0483	0.1566
Sea salt	15	0.808	0.1254	0.4141

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.*
BS 7319-6, *Method for determination of cadmium content.*

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[1] Referred to in the foreword only.

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