BRITISH STANDARD

BS 7319-9: 1990

Analysis of sodium chloride for industrial use —

Part 9: Method for determination of mercury content

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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

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Committee references CIC/22, FAC/23
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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, but did not include a method for mercury.

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.

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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.

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1 Scope

This Part of BS 7319 describes a cold vapour atomic absorption spectrometric method for the determination of total mercury in sodium chloride.

The method is applicable to products having mercury contents greater than 0.02 mg of mercury per kilogram of 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 a mixture of water, sodium chlorate and hydrochloric acid;

b) the conversion of all forms of mercury to ionic mercury (II) by the chlorine generated;

c) the reduction of the excess of oxidant by hydroxylammonium chloride;

d) the reduction of the mercury (II) to atomic mercury by tin (II) chloride;

e) the entrainment of the mercury in a stream of gas and passage of the gas containing the mercury vapour through a measuring cell;

f) the measurement of the absorbance at a wavelength of approximately 253.7 nm using an atomic absorption spectrometer fitted with a low-pressure mercury vapour lamp or a mercury hollow cathode lamp.

3 Reagents

3.1 *General.* Unless otherwise stated, use only reagents of recognized analytical grade having the lowest possible mercury content and only water complying with grade 3 of BS 3978.

Store all the reagent solutions in glass bottles.

3.2 *Sodium chloride*, with a mercury content of lower than 0.02 mg/kg.

3.3 *Hydrochloric acid solution*, containing 220 g/L HCl, approximately, (azeotropic mixture)

Dilute hydrochloric acid solution, ρ = 1.19 g/mL, (440 g/L HCl approximately) with an equal volume of water. Add to each litre, 5 mL of sulphuric acid, ρ = 184 g/mL and distil.

3.4 *Sodium chlorate solution*, 100 g/L solution.

Dissolve 100 g of sodium chlorate, $NaClO_3$, in 1 000 mL of water.

3.5 *Potassium dichromate solution*, 4 g/L solution.

Dissolve 4 g of potassium dichromate, $K_2Cr_2O_7$, in 500 mL of water. Add 500 mL of nitric acid, ρ = 1.40 g/mL and mix.

3.6 *Tin (II) chloride solution.* Dissolve 25 g of tin (II) chloride dihydrate, $SnCl_2.2H_2O$, in 50 mL of warm hydrochloric acid solution, ρ = 1.19 g/mL. Allow to cool and add 1 g to 2 g of metallic tin. Dilute to 250 mL with water and mix.

Prepare this solution freshly just before use.

Pass a gentle stream of nitrogen through the solution for 30 min, in order to remove any mercury, before use.

NOTE Avoid oxidation of solid tin (II) chloride by air.

3.7 *Hydroxylammonium chloride solution.* Dissolve 100 g of hydroxylammonium chloride, $NH_2OH.HCl$, in 1 000 mL water.

3.8 *Mercury stock solution*, corresponding to 1 000 mg/L mercury.

Weigh, to the nearest 0.001 g, 1.354 g of mercury (II) chloride, $HgCl_2$, into a 250 mL beaker. Add 50 mL of the hydrochloric acid solution (**3.3**) and 50 mL of the potassium dichromate solution (**3.5**). Transfer the solution quantitatively to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix.

Store this solution in a cool, dark place and renew after 2 months.

3.9 *Mercury, standard solution*, corresponding to 1 mg/L Hg.

Prepare this solution on the day of use by successive dilution of the stock solution (**3.8**). The final solution shall contain 50 mL of potassium dichromate solution (**3.5**) per litre of water.

3.10 *Charcoal*

3.11 *Drying agent*, e.g. anhydrous calcium sulphate (3 mm to 5 mm, for desiccant use)[1)].

3.12 *Air or nitrogen.* Use air or nitrogen containing no mercury or other components absorbing radiation at a wave length of approximately 253.7 nm. As an additional precaution, introduce a charcoal filter before the aeration flask (see Figure 1).

[1)] For information on the availability of other drying agents, apply to Enquiry Section, BSI, Linford Wood, Milton Keynes MK14 6LE.

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4 Apparatus

NOTE An example of a suitable apparatus is shown in Figure 1. This depicts an open-circuit measuring system and includes **4.1** to **4.9**.

4.1 *Atomic absorption spectrometer*, fitted with a low-pressure mercury vapour lamp or a mercury hollow-cathode lamp.

4.2 *Recorder or integrating read-out*, giving full deflection in less than 1 s.

4.3 *Measuring cell*, minimum optical path length 10 cm, with windows transparent to radiation at 253.7 nm.

4.4 *Aeration flask*, for example a 100 mL gas washing bottle, with a sintered glass or fine jet inlet tube and a mark at 60 mL.

NOTE If several bottles are used, check that identical results are obtained with each.

4.5 *Four-way stopcock*

4.6 *Flow control system*

4.7 *Conical flasks*, 100 mL.

4.8 *Adsorber*, filled with the charcoal (**3.10**), for the removal of mercury vapour from the exhaust gases. Length approximately 100 mm, internal diameter approximately 15 mm.

4.9 *Adsorber*, filled with drying agent (**3.11**). Length approximately 100 mm, internal diameter approximately 15 mm.

5 Procedure

5.1 General

Wash all glassware not previously used for this determination, including flasks used for reagents and samples, as follows rinsing with water after each operation:

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

b) with aqua regia or with nitric acid solution, ρ = 1.42 g/mL.

Before using the glassware thus washed for actual determinations, check it by carrying out several blank tests until satisfactory results are obtained. Thereafter use such glassware for mercury determinations only.

After each use treat the aeration flasks with the potassium dichromate solution (**3.5**), in order to oxidize any traces of tin (II) that they may contain.

Traces of tin (IV) oxide which may adhere to the walls of the aeration flask are removed by rinsing with hydrochloric acid ρ = 1.19 g/L.

All connecting tubes shall be as short as possible, in order to reduce adsorption of mercury.

Keep the aeration flasks full of water when not in use.

5.2 Test portion

Weigh, to the nearest 0.1 g, approximately 10 g of the test sample, transfer it to a 100 mL conical flask and add 30 mL of water.

NOTE The sample solution prepared according to BS 7319-3 should not be used, as in this solution mercury losses will occur. Samples should be mixed as well as possible and the test portion should be taken directly from the solid sample blend.

Carry out the determination described in **5.5**.

5.3 Blank test

Transfer to a 100 mL conical flask 30 mL of water.

Proceed in accordance with **5.5**, using the same quantities of all the reagents as used for the determination.

5.4 Preparation of the standard matching solutions

To a series of six conical flasks (**4.7**) add the same quantity of the sodium chloride (**3.2**) as the test portion (**5.2**), 30 mL of water and the volumes of the standard mercury solution (**3.9**) indicated in Table 1.

Carry out the determination described in **5.5**.

NOTE 1 Because of matrix effects, dependent on the concentration of sodium chloride in the solution during aeration (see **5.5.3**), a quantity of sodium chloride equal to the test portion (see **5.2**) should be taken for the preparation of standard matching solutions.

NOTE 2 Samples of unknown composition should be tested for the presence of matrix effects caused by components present other than sodium chloride, using the method of standard additions.

5.5 Determination

5.5.1 *Mineralization*

Add to each of the conical flasks (see **5.2**, **5.3** and **5.4**) some glass beads and 4.0 mL of the hydrochloric acid solution (**3.3**) and 3.0 mL of the sodium chlorate solution (**3.4**).

Dissolve the sodium chloride, heat to boiling, continue boiling for 5 min and allow to cool to room temperature.

Transfer the solution quantitatively to a 100 mL one-mark volumetric flask, dilute to the mark with water and mix.

Immediately before the measurement described in **5.5.3**, transfer 10.0 mL of this solution to the aeration flask, followed by 3 mL of the hydroxylammonium chloride solution (**3.7**).

5.5.2 *Apparatus settings*

The settings of the atomic absorption spectrometer are as follows.

Air or nitrogen flow: 60 L/h

Wavelength: 253.7 nm

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Table 1 — Mass of mercury in standard solutions

Volume of the standard mercury solution (3.9)	Corresponding mass of mercury
mL	μg
0[a]	0
0.5	0.5
1.0	1.0
1.5	1.5
2.0	2.0
3.0	3.0

[a] Zero standard.

Adjust the spectrometer (**4.1**) according to the manufacturer's instructions.

5.5.3 ***Measurement***

Dilute the contents of the aeration flask to the 60 mL mark with water. Add 2 mL of the tin (II) chloride solution (**3.6**) and immediately connect the flask to the apparatus (see Figure 1). Swirl to mix and allow to stand for some minutes. Pass air or nitrogen (**3.12**) through the aeration flask by manipulating the four-way stopcock. Continue the gas flow until no mercury is left in the system then switch off the gas flow and remove the aeration flask.

5.5.4 ***Preparation of the calibration curve***

Deduct the absorbance of the zero standard from the absorbance obtained for the other standards.

Plot a graph having, for example, the mass of mercury contained in the standard matching solutions, expressed in micrograms, as abscissae and the corresponding values of absorbance as ordinates.

6 Expression of results

Using the calibration curve (see **5.5.4**), determine the mass, in micrograms, of mercury in the test solution and in the blank solution corresponding to the absorbance of the zero standard.

Calculate the mercury content, expressed in micrograms of mercury (Hg) per kilogram on a moisture free basis, using the following expression.

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

where

m_1 is the mass of mercury in the test portion (see **5.2**) (in μg);

m_2 is the mass of mercury in the blank solution (see **5.3**) (in μg);

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

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

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 Hg/kg sample		
Rock salt	14	6.2	3.98	24.10
Vacuum salt	12	3.3	2.78	11.42
Sea salt	12	4.6	3.12	15.98

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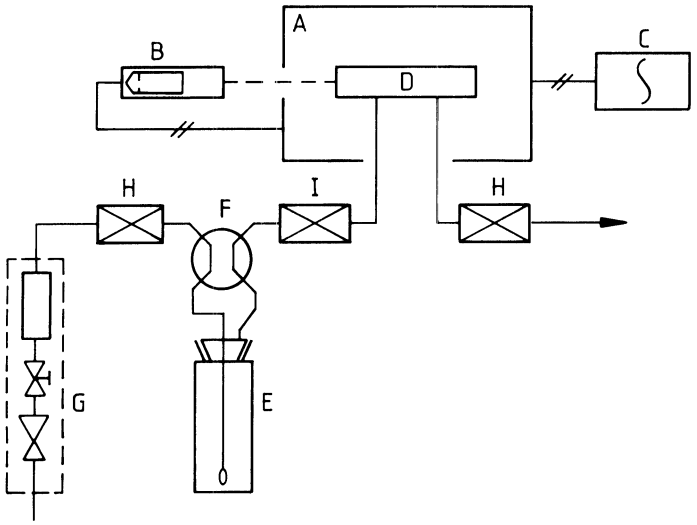

Key

A is the atomic absorption spectrometer
B is the mercury hollow-cathode lamp or low-pressure mercury vapour lamp
C is the recorder or maximum deflection indicator
D is the measuring cell
E is the aeration flask with sintered glass inlet or pointed immersion tube
F is the four-way stopcock
G is the flow control system, e.g. pressure regulator, needle valve and flow meter
H is the adsorption tube with charcoal
I is the adsorption tube with drying agent

Figure 1 — Typical apparatus for determination of mercury by atomic absorption spectrometry

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Publication(s) referred to

BS 998, *Specification for vacuum salt for food use*[2)].
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 7319-3, *Method for determination of matter insoluble in water or in acid.*

[2)] Referred to in the foreword only.

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