BRITISH STANDARD

BS 7319-7:
1990

# Analysis of sodium chloride for industrial use —

## Part 7: Method for determination of copper 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 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 It supersedes Appendix L of BS 998:1969 which is withdrawn.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 photometric method, using zinc dibenzyldithiocarbamate for the determination of copper in sodium chloride.

The method is applicable to products having copper contents equal to or greater than 0.01 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 principle of this Part of BS 7319 is the formation of a coloured complex by reaction of the copper with zinc dibenzyldithiocarbamate and photometric measurement at a wavelength of approximately 435 nm.

NOTE By carrying out the determination in acid medium, interferences, especially from iron, manganese, nickel and cobalt, are suppressed or much reduced.

## 3 Reagents

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

**3.2** *Carbon tetrachloride,* redistilled.

**3.3** *Hydrochloric acid,* $c$(HCl) = 2 mol/L.
Dilute 160 mL of hydrochloric acid, $\rho = 1.19$ g/mL, to 1 000 mL with water.

**3.4** *Zinc dibenzyldithiocarbamate,* 0.5 g/L solution in carbon tetrachloride. Dissolve 0.5 g of zinc dibenzyldithiocarbamate in 100 mL of carbon tetrachloride (**3.2**).

**3.5** *Copper stock solution,* 0.100 g of Cu per litre. Weigh, to the nearest 0.0001 g, 0.393 g of copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$) and transfer to a 1 000 mL one-mark volumetric flask. Add approximately 50 mL of water and swirl until the salt has dissolved.

Add 50 mL of the hydrochloric acid solution (**3.3**), dilute to the mark with water, and mix well.

Make this solution freshly each week.

1 mL of this solution contains 100 $\mu$g of Cu.

**3.6** *Copper standard solution,* 0.0010 g of Cu per litre. Introduce 10.0 mL of the stock copper solution (**3.5**) into a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix well.

1 mL of this standard solution contains 1 $\mu$g of Cu.

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 430 nm and 440 nm.

**4.3** *Burette,* 25 mL, graduated in 0.02 mL.

## 5 Procedure

CAUTION. Ensure that no trace of copper 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 soap if the sides are greasy;

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

Use the glassware a few times before carrying out an actual determination. Then keep exclusively for the determination of copper.

### 5.2 Test portion

Weigh, to the nearest 0.1 g, a test portion of approximately 100 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 the standard colorimetric solutions***

Introduce, from the burette (**4.3**), into a series of five 500 mL separating funnels, each containing 25 mL of the hydrochloric acid (**3.3**), the volumes of the copper standard solution (**3.6**) shown in Table 1.

**Table 1 — Mass of copper in standard solutions**

| Standard copper solution (3.6) | Corresponding mass of copper |
|---|---|
| mL | $\mu$g |
| 0[a] | 0 |
| 2.5 | 2.5 |
| 5.0 | 5.0 |
| 7.5 | 7.5 |
| 10.0 | 10.0 |

[a] Zero standard.

NOTE The calibration can be extended, if necessary. The curve is linear up to 50 $\mu$g of Cu.

**5.4.2** ***Formation of the coloured complex***

Dilute the contents of each separating funnel to approximately 250 mL with water, and mix.
Add 25.0 mL of the zinc dibenzyldithiocarbamate solution (**3.4**). Stopper, shake well for 1 min and allow the layers to separate. Run the lower layer through a hydrophobic filter paper into a cell of optical path length 4 cm.

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

**5.4.3** ***Photometric measurements***

Adjust the apparatus to zero absorbance with respect to carbon tetrachloride (**3.2**) prior to each measurement. Measure the absorbance of each solution 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 435 nm) or the photometer fitted with the filters specified. Deduct the absorbance of the zero standard from the absorbance obtained for each colorimetric solution (see **5.4.1**).

NOTE Carry out the measurements immediately after extraction in order to minimize the evaporation of the carbon tetrachloride.

**5.4.4** ***Plotting the calibration curve***

Plot a graph having, for example, the mass of copper contained in 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 (**3.5**) is prepared.

### 5.5 Determination

**5.5.1** ***Preparation of the test solution***

Add the test portion (**5.2**) to a 600 mL beaker, and add 350 mL of cold water and 50.0 mL of the hydrochloric acid (**3.3**). Boil until the sample has completely dissolved. Cool to approximately 20 °C.

Transfer the solution quantitatively to a 500 mL one-mark volumetric flask, dilute to the mark with water and mix well.

NOTE If the final solution is cloudy, filter it.

**5.5.2** ***Formation of the coloured complex***

Introduce an aliquot portion of the test solution (see **5.5.1**) containing 0 to 10 $\mu$g of copper into a 500 mL separating funnel and add 25.0 mL of the zinc dibenzyldithiocarbamate solution (**3.4**). Stopper, shake well for 1 min and allow the layers to separate. Run out the lower layer through a hydrophobic filter paper into a cell of optical path length of 4 cm.

NOTE If cells of 5 cm path length have been used to prepare the calibration curve, then use cells of 5 cm path length for the determination also.

**5.5.3** ***Photometric measurements***

Adjust the apparatus to zero absorbance with respect to carbon tetrachloride (**3.2**) prior to each measurement. Measure the absorbance, 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 435 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.

## 6 Expression of results

Using the calibration curve (**5.4.4**), determine the mass, in micrograms, of copper in the test solution and in the blank solution corresponding to the absorbance of the zero standard.

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

$$\frac{(500m_1)}{(Vm_0)} \times \frac{100}{(100-H)}$$

where

| | |
|---|---|
| $m_0$ | is the mass of the test portion (in g) (**5.2**); |
| $m_1$ | is the mass of copper found in the aliquot portion of the test solution (in $\mu$g) (**5.5.2**); |
| $H$ | is the moisture content determined in accordance with BS 7319-2 [in % ($m/m$)]; |
| $V$ | is the volume, in mL, of the aliquot portion of the test solution (**5.5.2**). |

## 7 Precision

Analyses carried out on three samples have been statistically evaluated as shown in Table 2, each laboratory having obtained results by the same operator on two test portions.

**Table 2 — Statistical results of sodium chloride analysis**

| Sample | Number of laboratories | Mean | Standard deviation for | |
|---|---|---|---|---|
| | | | Repeatability $\sigma_r$ | Reproducibility $\sigma_R$ |
| | | mg Cu/kg sample | | |
| Rock salt | 11 | 0.020 | 0.0018 | 0.0153 |
| Vacuum salt | 13 | 0.030 | 0.0030 | 0.0159 |
| Sea salt (solid) | 13 | 0.054 | 0.0083 | 0.0258 |

*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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