BRITISH STANDARD

BS 5634:1978

Methods of test for

Potassium hydroxide

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Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Fatty Acid Distillers
British Tar Industry Association
Chemical Industries Association*
Department of Health and Social Security
Department of Industry, Laboratory of the Government Chemist
Fertiliser Manufacturers' Association Ltd.
Hydrocarbon Solvents Association
Ministry of Agriculture Fisheries and Food
Ministry of Defence*
National Sulphuric Acid Association
Paintmakers Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association*
Society for Analytical Chemistry
Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Asbestos Cement Manufacturers Association
British Man-made Fibres Federation
British Textile Employers' Association
Fabric Care Research Association
Society of Glass Technology

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board on 29 December 1978

The following BSI references relate to the work on this standard:
Committee reference CIC/22
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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Foreword

This British Standard, prepared under the direction of the Chemicals Standards Committee, comprises methods of test for potassium hydroxide in solid form and in aqueous solution. It provides a full range of tests for use in conjunction with BS 5633, which specifies potassium hydroxide for use in alkaline cells. Not all of these tests may be necessary when testing potassium hydroxide for other industrial uses (in such cases the methods may be used selectively).

The opportunity has been taken, where appropriate, to incorporate substantially the technical content of International Standards from the series of methods of test for potassium hydroxide for industrial use which has been prepared, with the active participation of the United Kingdom, by Sub-committee 5 of Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO). The relationship between the appropriate British Standard methods and the International Standards is shown in the table below.

The method of ISO 2466 has been modified by the introduction of two sample solutions, one a solution of the sample corresponding to solution A in ISO 2466 and the other an acidified solution of the sample, designated solution B. A third solution, designated C, is a blank for the acid used in the preparation of solution B. If it is necessary to determine all the trace metals, the use of solution B results in considerable saving of time.

The procedure used for the determination of arsenic, which is the same as that described in BS 4404, is technically similar to the method given in ISO 2590.

Atomic absorption methods are described for the determinations of calcium, zinc and lead. The flame emission photometric method used for the determination of sodium is similar in principle to that described in ISO 1550 but, in accordance with United Kingdom comments not accepted by the ISO committee, does not restrict the fuel for the atomizer-burner of the flame spectrophotometer to a butane/air mixture.

Other International Standards in the series of methods of test for potassium hydroxide for industrial use will be published as addenda to this British Standard, subject to their approval by the United Kingdom.

There are at present no ISO methods for the determination of the contents of nitrate, cyanide, zinc, copper, lead and nickel in potassium hydroxide.

BS 5634: Preparation/ Determination of	Corresponding International Standard
sample solutions	ISO 2466, "*Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations*"
total hydroxide content	ISO 990, "*Potassium hydroxide for industrial use — Method of assay*"
carbonate content	ISO 2900, "*Potassium hydroxide for industrial use — Determination of carbon dioxide content — Titrimetric method*"
chloride content	ISO 3177, "*Potassium hydroxide for industrial use — Determination of chlorides content — Photometric method*"
content of sulphur compounds	ISO 3194, "*Potassium hydroxide for industrial use — Determination of sulphur compounds — Method by reduction and titrimetry*"
silica content	ISO 995, "*Potassium hydroxide for industrial use — Determination of silica content — Reduced molybdosilicate photometric method*"

BS 5634: Preparation/ Determination of	Corresponding International Standard
calcium content	ISO 3698, "*Potassium hydroxide for industrial use — Determination of calcium and magnesium contents — Flame atomic absorption method*"
iron content	ISO 994, "*Potassium hydroxide for industrial use — Determination of iron content — 1,10-Phenanthroline photometric method*"

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 26, 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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Section 1. General

1 Scope

This British Standard specifies methods for sampling and testing potassium hydroxide in solid form and in aqueous solution.

2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

3 Preparation of sample solution

3.1 Principle. Three solutions are prepared, solution A which is a solution of the sample in water, solution B which is an acidified solution of the sample in water and solution C which is a blank for the acid used in preparing solution B.

NOTE The use of these bulk sample solutions serves to reduce sampling errors. In the case of solution B, acidification ensures dissolution of metallic impurities and allows more uniform sampling for the determination of metallic impurities.

3.2 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

3.2.1 *Hydrochloric acid,* approximately 5N solution.

3.2.2 *Methyl orange indicator solution,* 0.4 g/l, prepared as described in BS 4123.

3.3 Apparatus. Ordinary laboratory apparatus is required.

3.4 Procedure

3.4.1 *Preliminary treatment of sample.* Protect the laboratory sample from the atmosphere and handle it in such a way that no appreciable absorption of moisture or carbon dioxide or reaction with silica can occur.

The maximum particle size of solid test portions shall not exceed about 6 mm. If crushing is necessary, carry it out as quickly as possible, in as inert an atmosphere as possible, preferably in an unventilated glove box containing evenly-distributed shallow dishes of phosphorus (V) oxide and similar dishes of potassium hydroxide pellets to remove water vapour and carbon dioxide, respectively. These absorbents should be left in the glove box for at least 1 h before it is used.

3.4.2 *Preparation of solution A.* Weigh, accurately, 100 ± 1 g of the solid sample or 200 ± 2 g of the liquid sample into an alkali-resistant container fitted with a lid. If the sample is solid, dissolve it in 250 ml of freshly boiled and cooled water in an alkali-resistant beaker and cool. Transfer the solution, or the original sample if liquid, to a 500 ml alkali-resistant graduated cylinder, dilute to the mark with water, mix and transfer immediately to an airtight, alkali-resistant plastics bottle. Label as "Solution A".

3.4.3 *Preparation of solution B.* Weigh 10.0 ± 0.1 g of the solid sample, or 20.0 ± 0.2 g of the liquid sample, into a 250 ml beaker. Add 50 ml of water, swirl to dissolve or mix, and titrate with the hydrochloric acid solution (**3.2.1**) using the methyl orange solution (**3.2.2**) as indicator. Let the titration be X ml. Discard the solution.

Weigh, accurately, 100 ± 1 g of the solid sample or 200 ± 2 g of the liquid sample into a 1 000 ml beaker. Add 200 ml of water and swirl to dissolve or mix. From a measuring cylinder cautiously add (10 X + 5) ml of the hydrochloric acid solution (**3.2.1**) with continuous stirring. Cool the contents of the beaker to ambient temperature, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Label as "Solution B".

3.4.4 *Preparation of solution C.* Evaporate (10 X + 5) ml (see **3.4.3**) of the hydrochloric acid solution (**3.2.1**) to 5 ml in a 1 000 ml beaker, taking care to avoid contamination. Transfer the residual solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Label as "Solution C".

Section 2. Methods of test

4 Determination of total hydroxide content

4.1 Field of application. The method is applicable to all commercial products, both solid and liquid.

4.2 Principle. The total alkalinity content is determined by titration with hydrochloric acid solution using methyl orange as indicator and a correction is made for the carbonate content, determined by the method of test specified in clause **5**, to give total hydroxide content.

4.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978, freshly boiled to free it from carbon dioxide, shall be used throughout.

4.3.1 *Hydrochloric acid,* 1N standard volumetric solution.

4.3.2 *Methyl orange indicator solution,* 0.4 g/l, prepared as described in BS 4123.

4.4 Apparatus. Ordinary laboratory apparatus and the following are required.

4.4.1 *Burette,* 50 ml capacity, complying with the requirements for class A of BS 846.

4.5 Procedure. Weigh, to the nearest 0.01 g, 25 g of the solid sample or 50 g of the liquid sample. Dissolve in or mix with 200 ml of water in a 400 ml beaker. Cool and transfer to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix. Pipette 50.0 ml of this solution into a 500 ml conical flask. Add 80 ml of water and five drops of the methyl orange solution (**4.3.2**) and titrate with the hydrochloric solution (**4.3.1**) until the colour changes from yellow to orange.

4.6 Expression of results. The total hydroxide content, expressed as the percentage by mass of potassium hydroxide (KOH), is given by the following formula:

$$\frac{56.1 \times V}{m} - 0.812A$$

where

V is the volume of the hydrochloric acid solution (**4.3.1**) used in the determination of total alkali (in ml)

m is the mass of the test portion (in g)

A is the carbonate content of the sample, expressed as the percentage by mass of potassium carbonate (K_2CO_3) (see clause **5**)

5 Determination of carbonate content

5.1 Field of application. The method is applicable to products having carbonate contents, expressed as potassium carbonate (K_2CO_3), greater than 0.02 % *m*/*m*.

5.2 Principle. Carbon dioxide is released by acidifying and heating the test solution. The released carbon dioxide is entrained in a current of nitrogen or air and absorbed in standard barium hydroxide solution. The excess barium hydroxide is titrated with a standard volumetric solution of hydrochloric acid using thymolphthalein as indicator.

5.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

5.3.1 *Hydrochloric acid,* approximately 5N solution.

5.3.2 *Hydrochloric acid,* 0.1N standard volumetric solution.

5.3.3 *Barium hydroxide,* approximately 0.1N solution.

Weigh 15 ± 0.1 g of barium hydroxide octahydrate, $[Ba(OH)_2.8H_2O]$, dissolve in water, dilute to 1 000 ml and mix. Allow precipitated barium carbonate to settle out before use.

5.3.4 *Methyl orange indicator solution,* 0.4 g/l, prepared as described in BS 4123.

5.3.5 *Thymolphthalein indicator solution,* 2 g/l, prepared as described in BS 4123.

5.3.6 *Nitrogen or air,* free from carbon dioxide.

5.4 Apparatus. Ordinary laboratory apparatus and the following are required.

5.4.1 *Apparatus for the release, absorption and titration of carbon dioxide.* A typical apparatus, shown in Figure 1, comprises

A flask, 500 ml capacity

B dropping funnel, 100 ml capacity

C_1 and C_2 Drechsel bottles, containing 20 % *m*/*m* sodium hydroxide solution

C_3 Drechsel bottle, containing saturated barium hydroxide solution

D spiral condenser

E absorption vessel

F absorption coil, made from glass tubing of diameter 7 mm to 8 mm and consisting of five or six turns of diameter 55 mm to 60 mm

G burette, 50 ml capacity, complying with the requirements for class A of BS 846

H stopcock

J inlet for nitrogen or air (free from carbon dioxide)

K outlet for nitrogen or air (free from carbon dioxide)

L mercury lute fitted with guard tube containing activated charcoal

5.5 Procedure

5.5.1 *Test portion.* Measure out a volume of the solution A (**3.4.2**) containing not more than 25 g of potassium hydroxide or 150 mg of potassium carbonate.

5.5.2 *Blank test.* Subsequent to the determination, carry out a blank test following the procedure specified in **5.5.3** but omitting the test portion.

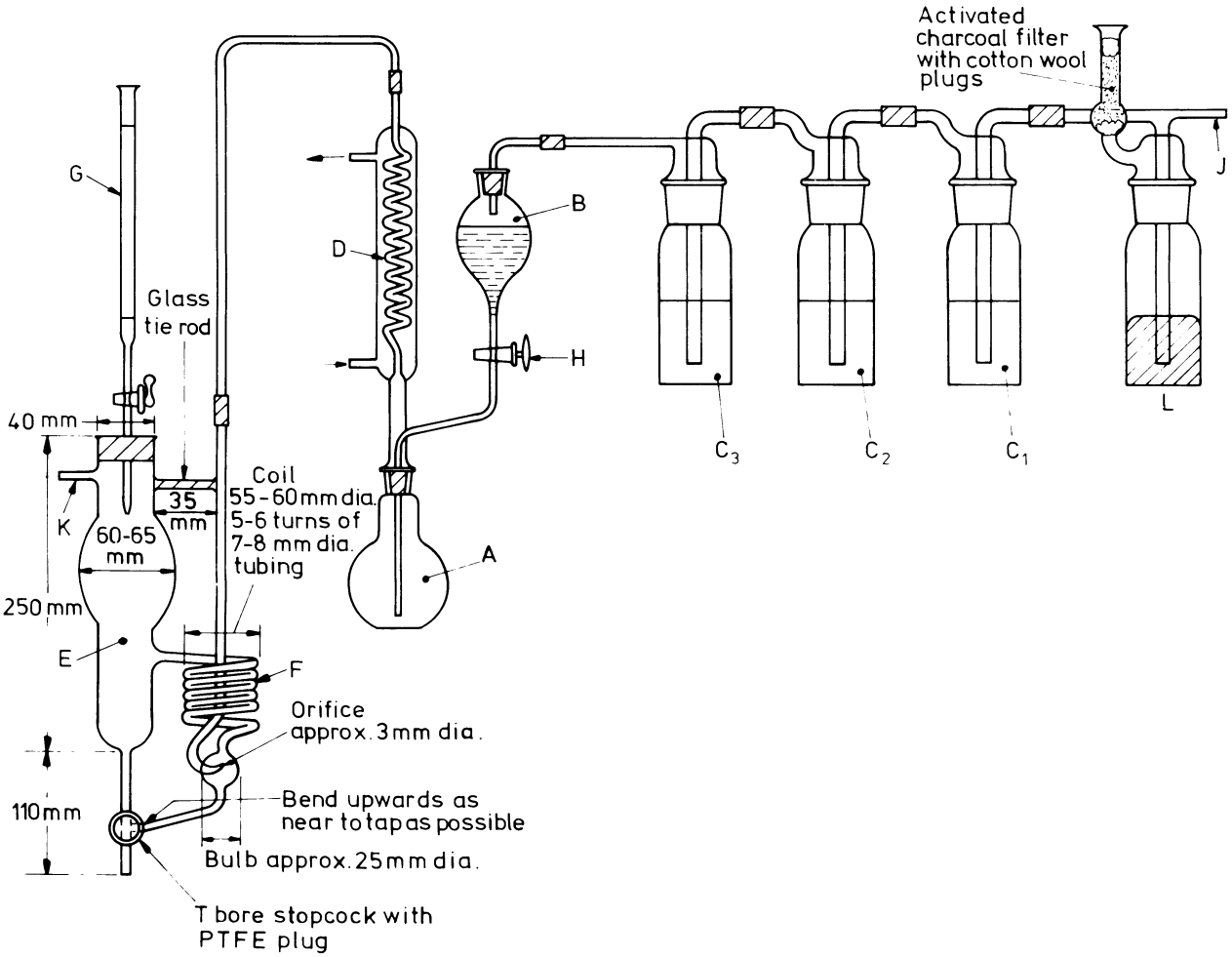

Figure 1 — Typical apparatus for the determination of carbonate content

5.5.3 *Determination.* Pass the nitrogen or the air (**5.3.6**) through the apparatus at a rate of about 5 bubbles per second for 10 min. Stop the current of gas and introduce the test portions and reagents into the apparatus as follows:

a) into the dropping funnel (B) about 100 ml of the hydrochloric acid solution (**5.3.1**);

b) into the flask (A) the test portion (**5.5.1**), diluted to 150 ml with water, and three drops of the methyl orange solution (**5.3.4**);

c) into the absorption vessel (E) 50.00 ml of the barium hydroxide solution (**5.3.3**) and 10 drops of the thymolphthalein solution (**5.3.5**).

Close the apparatus, start the flow of water into the jacket of the condenser (D) and carefully allow the acid in the dropping funnel (B) to run into the flask (A). Check that the solution is now acid.

Pass a current of the gas (**5.3.6**), controlled to 2 bubbles per second for 10 min and then, without interrupting the flow, heat the contents of the flask (A) just to boiling and maintain at this temperature for 20 min. Stop heating, increase the gas flow to 5 bubbles per second and titrate the excess of the barium hydroxide solution contained in the vessel (E) with the standard volumetric hydrochloric acid solution (**5.3.2**), using the burette (G), until the blue colour of the indicator just changes to colourless.

5.6 Expression of results. The carbonate content, expressed as a percentage by mass of potassium carbonate (K_2CO_3), is given by the following formula:

$$(V_0 - V_1) \times \frac{500}{V_2} \times \frac{100}{m_0} \times 0.0069 = \frac{345\,(V_0 - V_1)}{V_2 \times m_0}$$

where

V_0 is the volume of the standard volumetric hydrochloric acid solution (**5.3.2**) used for the blank (in ml)

V_1 is the volume of the standard volumetric hydrochloric acid solution (**5.3.2**) used for the determination (in ml)

V_2 is the aliquot portion of solution A taken for the test (in ml)

m_0 is the mass of sample used in the preparation of solution A (in g)

6 Determination of sodium content

6.1 Field of application. The method is applicable to products having sodium contents, expressed as sodium hydroxide (NaOH), in the range 0.2 % to 2 % *m*/*m* for liquid products and 0.4 % to 4 % *m*/*m* for solid products.

6.2 Principle. The sample solution is neutralized with hydrochloric acid and acidified by the addition of a small excess. The sodium content is then determined using either a flame photometer with a sodium filter or a flame spectrophotometer set at a wavelength of about 589 nm.

6.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

6.3.1 *Hydrochloric acid,* approximately 1N solution.

6.3.2 *Methyl orange indicator solution,* 0.4 g/l, prepared as described in BS 4123.

6.3.3 *Potassium hydrogen carbonate,* 89 g/l solution. Store in a plastics container.

6.3.4 *Sodium chloride,* standard solution, corresponding to 2 000 g of sodium hydroxide per litre.

Dissolve 2.925 g of sodium chloride, previously dried at a temperature of 500 °C, in water and dilute to the mark in a 1 000 ml one-mark volumetric flask. Mix.

1 ml of this solution contains the equivalent of 2 mg of NaOH.

6.4 Apparatus. Ordinary laboratory apparatus and the following are required.

6.4.1 *Flame photometer,* fitted with a sodium filter, with appropriate gas supplies, or

6.4.2 *Flame spectrophotometer,* with appropriate gas supplies.

6.5 Procedure

6.5.1 *Test portion.* Using a pipette, transfer 10.0 ml of the solution A (**3.4.2**) into a 100 ml one-mark volumetric flask.

6.5.2 *Preparation of standard matching solutions.* Into a series of six 250 ml beakers, each containing 10.0 ml of the potassium hydrogen carbonate solution (**6.3.3**), add to each 18.9 ml of the hydrochloric acid solution (**6.3.1**) and then measure accurately, from a 10 ml burette, the volumes of the standard sodium chloride solution (**6.3.4**) shown in the following table:

Standard sodium chloride solution (6.3.4)	Equivalent sodium hydroxide (NaOH) content
ml	mg
0[a]	0
2.0	4.0
4.0	8.0
6.0	12.0
8.0	16.0
10.0	20.0

[a] Blank standard matching solution.

Treat the contents of each beaker as follows.

Heat to boiling and cool. Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

6.5.3 *Adjustment of the apparatus.* Switch on the current a sufficient time in advance to ensure stabilization. Turn on the gas supplies and ignite the burner. If the flame photometer (**6.4.1**) is used, insert the sodium filter. If the flame spectrophotometer (**6.4.2**) is used, set the monochromator to a wavelength of about 589 nm. Aspirate water into the flame and set the instrument to zero reading. Aspirate the most concentrated standard matching solution (**6.5.2**) into the flame and adjust the instrument sensitivity to give a suitable response. In the case of the flame spectrophotometer it may be necessary to offset the burner to reduce the sensitivity.

6.5.4 *Photometric measurements.* Aspirate the series of standard matching solutions (**6.5.2**) into the flame and measure the response for each. Spray water through the burner after each measurement. Take care to keep the aspiration rate constant throughout this series of measurements.

6.5.5 *Preparation of the calibration graph.* Plot a graph having, for example, the number of equivalent milligrams of sodium hydroxide contained in 1 000 ml of the standard matching solutions as abscissae and the corresponding values of the measured response, less the measured value for the blank standard matching solution, as ordinates.

NOTE It is essential that the calibration graph obtained is rectilinear.

6.5.6 ***Determination***

6.5.6.1 *Preparation of the test solution.* Dilute the test portion in the 100 ml one-mark volumetric flask (**6.5.1**) to the mark with water and mix.

Using a pipette, transfer 25.0 ml of this solution into a 150 ml conical flask and titrate with the hydrochloric acid solution (**6.3.1**) using the methyl orange solution (**6.3.2**) as indicator. Note the volume of the hydrochloric acid solution used in the titration, X ml, and discard the titrated solution.

Transfer a further 25.0 ml of the solution from the 100 ml one-mark volumetric flask into a 1 000 ml one-mark volumetric flask, add $(10.0 + X)$ ml of the hydrochloric acid solution (**6.3.1**), dilute to the mark with water and mix.

6.5.6.2 *Photometric measurements.* Immediately after making the photometric measurements on the standard matching solutions (**6.5.4**), determine in a similar manner the response from the test solution (**6.5.6.1**). Subtract from it the value for the blank standard matching solution (**6.5.2**) and, using this corrected value, read from the calibration graph the sodium content of the test solution (**6.5.6.1**) expressed as milligrams of sodium hydroxide.

6.6 Expression of results. The sodium content, expressed as a percentage by mass of sodium hydroxide (NaOH), is given by the following formula:

$$\frac{m_1}{1000} \times \frac{500}{2.5 \times m_0} \times 100 = \frac{20\,m_1}{m_0}$$

where

m_1 is the mass of sodium, expressed as sodium hydroxide, in the test solution (**6.5.6.1**) (in mg)

m_0 is the mass of sample in 500 ml of the solution A (**3.4.2**) (in g)

7 Determination of chloride content

7.1 Field of application. The method is applicable to products having chloride contents, expressed as potassium chloride (KCl), in the range 4 mg/kg to 100 mg/kg.

7.2 Principle. Thiocyanate (SCN) ions, derived from a solution of mercury (II) thiocyanate, are displaced by the chloride (Cl) ions contained in the test solution when the two solutions are mixed. The displaced (SCN) ions are reacted with a solution of iron (III) nitrate to form red coloured iron (III) thiocyanate.

The intensity of the developed colour is measured photometrically at a wavelength of about 450 nm.

7.3 Reagents. The reagents used shall be of recognized analytical quality. Only double-distilled water, or water of equivalent purity, shall be used throughout. The preparation and storage of the reagents, as well as the sampling and determination, shall take place in an atmosphere free from chlorine and hydrochloric acid.

7.3.1 *Nitric acid, ρ* approximately 1.42 g/ml, about 70 % *m/m* solution or approximately 16N solution, having a chloride (Cl) content not exceeding 0.5 mg/kg.

7.3.2 *Iron (III) nitrate,* solution corresponding to 8 g of iron per litre. Pour 80 ml of water into a 500 ml conical flask and add 4.0 g of pure (not less than 99.5 %) iron wire. Cautiously add 80 ml of the nitric acid solution (**7.3.1**). Heat and then boil in a ventilated fume cupboard until the reaction is complete and nitrous fumes have been completely eliminated. Decolorize the solution by adding a few drops of 30 % *m/m* hydrogen peroxide solution and boil again for a few minutes. Cool, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

7.3.3 *Mercury (II) thiocyanate,* 0.5 g/l solution. Weigh, to the nearest 0.001 g, 0.100 g of mercury (II) thiocyanate [$Hg(SCN)_2$] and dissolve in 180 ml of water at a temperature of 50 °C, while stirring. Cool, filter, collecting the filtrate in a 200 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

7.3.4 *Sodium chloride,* standard solution corresponding to 0.100 g of chloride per litre. Weigh, to the nearest 0.001 g, 0.165 g of sodium chloride, previously dried at a temperature of 500 °C for 1 h and then cooled, dissolve in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 0.1 mg of Cl.

7.3.5 *Sodium chloride,* standard solution corresponding to 10 mg of chloride per litre. Pipette 20.0 ml of the standard sodium chloride solution (**7.3.4**) into a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

1 ml of this standard solution contains 0.01 mg of Cl.

7.3.6 *Phenolphthalein indicator solution,* 10 g/l, prepared as described in BS 4123.

7.4 Apparatus. Ordinary laboratory apparatus, carefully rinsed with the nitric acid solution (**7.3.1**) and double-distilled water, and the following are required.

7.4.1 *Spectrophotometer,* or

7.4.2 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of about 450 nm.

7.4.3 *Optical cells,* 4 cm optical path length.

7.5 Procedure

7.5.1 *Test portion.* Weigh, in an alkali-resistant container fitted with a lid, to the nearest 0.01 g, 20 g of the solid sample or 40 g of the liquid sample.

7.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **7.5.4.2**, but omitting the test solution and the quantity of the nitric acid solution (**7.3.1**) necessary for its neutralization.

7.5.3 ***Preparation of the calibration graph***

7.5.3.1 *Preparation of the standard matching solutions.* Into a series of five 50 ml one-mark volumetric flasks, introduce the volumes of the standard sodium chloride solution (**7.3.5**) shown in the following table:

Standard sodium chloride solution (7.3.5)	**Corresponding mass of chloride (Cl)**
ml	mg
0[a]	0
1.0	0.010
2.5	0.025
5.0	0.050
7.5	0.075

[a] Compensation solution.

7.5.3.2 *Colour development.* To each of the standard matching solutions (**7.5.3.1**) add, in the order shown, the following reagents:

a) 5 ml of the nitric acid solution (**7.3.1**);

b) 5 ml of the iron (III) nitrate solution (**7.3.2**);

c) 20 ml of the mercury (II) thiocyanate solution (**7.3.3**).

Dilute the contents of each flask to the mark, mix and allow to stand for 30 min to allow for colour development.

7.5.3.3 *Photometric measurements.* Carry out the photometric measurements, using either the spectrophotometer (**7.4.1**) at the wavelength of maximum absorption (about 450 nm), or the photoelectric absorptiometer (**7.4.2**), fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

7.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**7.5.3.1**) from those of the standard matching solutions (**7.5.3.1**). Plot a graph having, for example, the chloride (Cl) content, expressed in milligrams per 50 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

7.5.4 ***Determination***

7.5.4.1 *Preparation of the test solution.* Treat the test portion (**7.5.1**) as follows. Dissolve in or mix with water and allow to cool to room temperature. Transfer this solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

7.5.4.2 *Colour development.* Transfer 10.0 ml of the test solution (**7.5.4.1**) to a 50 ml one-mark volumetric flask. Add three drops of the phenolphthalein solution (**7.3.6**) and neutralize by adding slowly, with stirring and thorough cooling under cold running water, the nitric acid solution (**7.3.1**). Follow the procedure specified in **7.5.3.2**.

7.5.4.3 *Photometric measurement.* Carry out the photometric measurement on the test solution (**7.5.4.2**), and on the blank test solution (**7.5.2**) following the procedure specified in **7.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the test solution (**7.5.4.1**) and modifying the calculation accordingly.

7.6 Expression of results. By means of the calibration graph (**7.5.3.4**), determine the quantity of chloride corresponding to the value of the photometric measurement.

The chloride content, expressed as milligrams of potassium chloride (KCl) per kilogram, is given by the following formula:

$$(m_1 - m_2) \times \frac{100}{10} \times \frac{1000}{m_0} \times \frac{74.6}{35.5}$$

$$= \frac{(m_1 - m_2)}{m_0} \times 2.10 \times 10^4$$

where

m_1 is the mass of Cl found in the test solution (in mg)

m_2 is the mass of Cl found in the blank test solution (in mg)

m_0 is the mass of the sample in the test portion (**7.5.1**) (in g)

Express the result to the nearest whole number.

8 Determination of content of sulphur compounds

8.1 Field of application. The method is applicable to products having contents of sulphur compounds, expressed as potassium sulphate (K_2SO_4), in the range 10 mg/kg to 100 mg/kg.

8.2 Principle. The sulphur compounds are reduced to hydrogen sulphide by heating with a mixture of hydriodic acid and hypophosphorous acid. The hydrogen sulphide, entrained in the current of oxygen-free nitrogen, is absorbed in a mixture of sodium hydroxide and acetone and titrated with standard volumetric mercury (II) nitrate solution, using dithizone as indicator.

8.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

8.3.1 *Acetone*

8.3.2 *Nitrogen,* oxygen-free.

8.3.3 *Sodium hydroxide,* approximately 1N solution.

8.3.4 *Potassium permanganate and mercury (II) chloride solution.* Dissolve in turn, in 100 ml of water, 2 g of potassium permanganate ($KMnO_4$) and 7 g of mercury (II) chloride ($HgCl_2$) and filter.

8.3.5 *Pyrogallol solution.* Dissolve 15 g of pyrogallol in 25 ml of water and add, with cooling, 150 ml of 30 % *m*/*m* potassium hydroxide solution.

8.3.6 *Wash solution.* Dissolve 10 g of sodium dihydrogen orthophosphate monohydrate ($NaH_2PO_4.H_2O$) in 100 ml of water.

8.3.7 *Reducing solution.* Into the 500 ml three-necked flask K (see Figure 3) of the apparatus (**8.4.2**), transfer, shaking after each addition, the following reagents:

a) 50 ml of hypophosphorous acid (H_3PO_2) solution, ρ approximately 1.21 g/ml, about 50 % *m*/*m* solution;

b) 100 ml of hydrochloric acid solution, ρ approximately 1.18 g/ml, about 36 % *m*/*m* solution;

c) 120 ml of hydriodic acid solution, ρ approximately 1.97 g/ml, about 67 % *m*/*m* solution.

Pass the nitrogen (**8.3.2**) through the mixture and reflux under a water condenser for 4 h.

Cool to room temperature under the nitrogen (**8.3.2**) and store the reagent away from direct sunlight in a bottle previously flushed with the nitrogen (**8.3.2**) and fitted with a ground glass stopper.

8.3.8 *Mercury (II) nitrate,* 0.1N standard volumetric solution. Dissolve 10.85 ± 0.01 g of mercury (II) oxide (HgO) in 10 ml of nitric acid solution, ρ approximately 1.42 g/ml, about 70 % *m*/*m* solution, or approximately 16N and dilute to the mark with water in a 1 000 ml one-mark volumetric flask. Mix.

NOTE The strength of this solution is sufficiently exact for the small quantities of sulphur compounds to be determined and standardization is therefore unnecessary.
In most laboratories, however, a precisely standardized 0.1N standard volumetric solution, commonly used for the mercurimetric determination of chlorides, is available.

8.3.9 *Mercury (II) nitrate,* 0.001N standard volumetric solution. Dilute 10.00 ml of the standard volumetric mercury (II) nitrate solution (**8.3.8**) to the mark in a 1 000 ml one-mark volumetric flask. Mix.

Prepare this solution immediately before use.

8.3.10 *Dithizone,* 1 g/l solution in acetone (**8.3.1**).

8.4 Apparatus. Ordinary laboratory apparatus and the following are required.

8.4.1 *Apparatus for reduction and distillation.* A typical apparatus, shown in Figure 2, in which all the components are fitted together with ground glass joints, comprises

A Drechsel bottle, capacity 50 ml, for purification of the nitrogen, containing the potassium permanganate and mercury (II) chloride solution (**8.3.4**)

B Drechsel bottle, capacity 50 ml, for purification of the nitrogen, containing the pyrogallol solution (**8.3.5**)

C conical flask, capacity 100 ml, for the reduction reaction

D reflux condenser

E Drechsel bottles, capacity 50 ml, containing the wash solution (**8.3.6**) which absorbs the acid vapours entrained by the distillation, with the exception of the hydrogen sulphide.

F absorption vessel, internal diameter 25 mm and height 100 mm, for the absorption and titration of the hydrogen sulphide

G dropping funnel, capacity 50 ml

H microburette, 10 ml capacity, graduated in 0.01 ml divisions

J mercury lute, fitted with a guard tube containing activated charcoal

1, 2, 3, 4 stopcocks

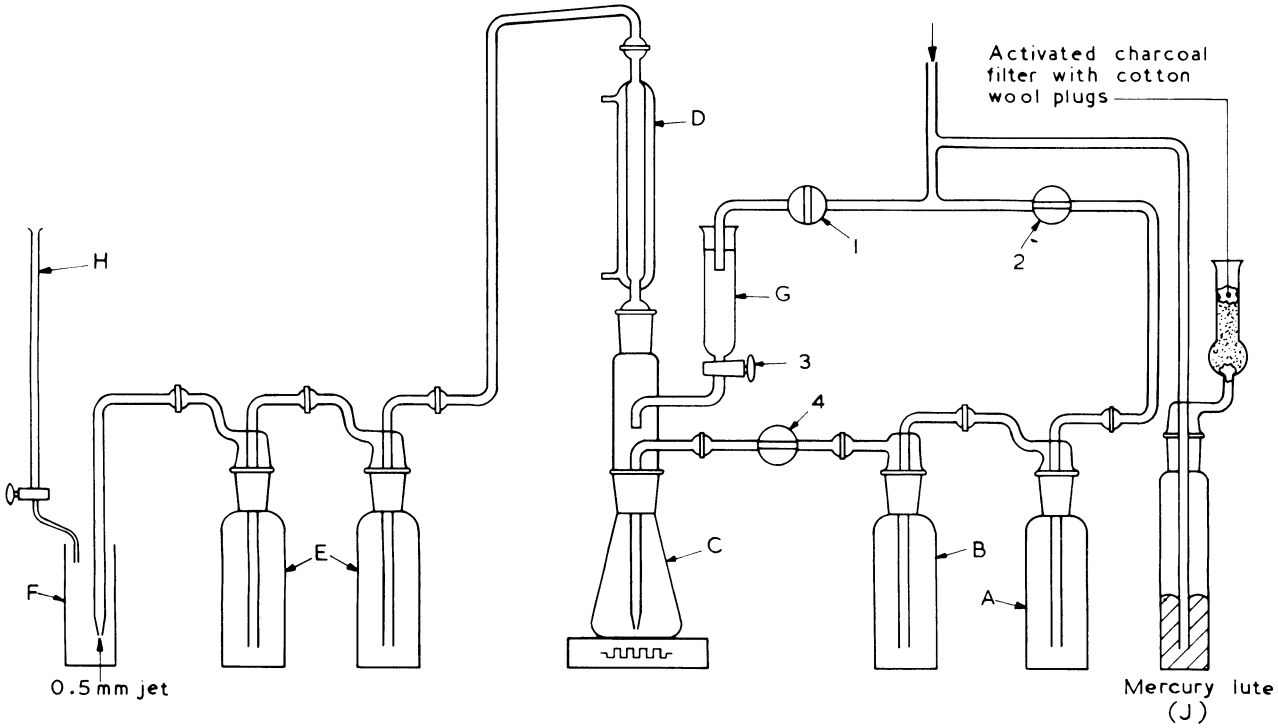

Figure 2 — Typical apparatus for the determination of content of sulphur compounds

8.4.2 *Apparatus for the preparation of the reducing solution (***8.3.7***).* A typical apparatus, shown in Figure 3, comprises

K flask, capacity 500 ml, three-necked

L reflux condenser

M nitrogen supply tube

8.5 Procedure

8.5.1 *Test portion.* Weigh, to the nearest 0.01 g, into the 100 ml conical flask (C), fitted with a stopper, a mass of the test sample, solid or liquid, not exceeding 5 g or 10 g respectively.

8.5.2 *Blank test.* After the determination, carry out a blank test in the same apparatus following the same procedure and using the same quantities of all the reagents as in the determination (**8.5.3**).

8.5.3 *Determination.* Introduce the reagents into the apparatus as follows:

a) into the Drechsel bottle A, 25 ml of the potassium permanganate and mercury (II) chloride solution (**8.3.4**);

b) into the Drechsel bottle B, 25 ml of the pyrogallol solution (**8.3.5**);

c) into each of the Drechsel bottles E, 25 ml of the wash solution (**8.3.6**);

d) into the absorption vessel F, 5 ml of the sodium hydroxide solution (**8.3.3**), 5 ml of the acetone (**8.3.1**) and 0.1 ml of the dithizone solution (**8.3.10**);

e) into the conical flask C containing the test portion (**8.5.1**), a few glass beads and, in the case of a solid sample, 5 ml of water;

f) into the dropping funnel G, 50 ml of the reducing solution (**8.3.7**);

g) into the microburette H, the standard volumetric mercury (II) nitrate solution (**8.3.9**).

Connect up the apparatus and pass the nitrogen (**8.3.2**) through the apparatus via stopcocks 2 and 4 at a speed of about 2 bubbles per second. After 5 min, run the mercury (II) nitrate solution (**8.3.9**) drop by drop into the absorption vessel F until the indicator colour changes from yellow to red.

Open the stopcocks 1 and 3 to allow the reducing solution (**8.3.7**) to run into the conical flask C, until a few millimetres of liquid are left in the dropping funnel G. Then close the stopcocks 1 and 3.

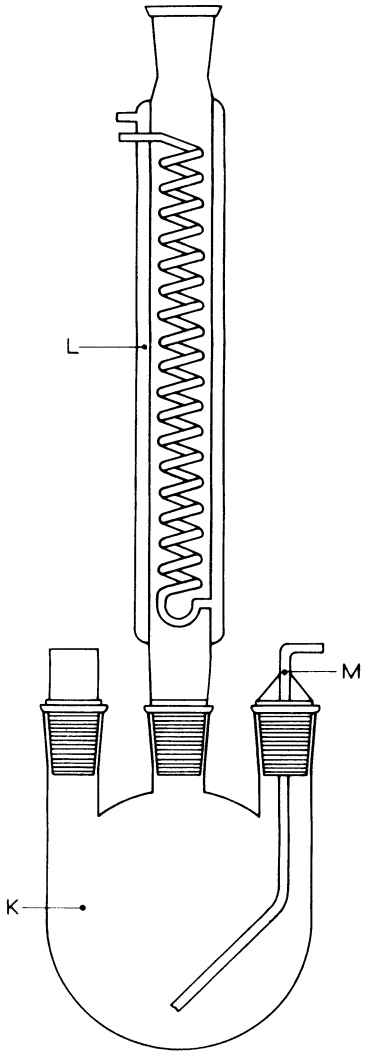

Figure 3 — Typical apparatus for the preparation of the reducing solution (8.3.7) used in the determination of content of sulphur compounds

Start the flow of water into the jacket of the condenser D and, while continuing the flow of nitrogen, boil the solution gently in the conical flask C for not less than 30 min. The presence of hydrogen sulphide is shown by the colour change of the indicator from red to yellow in the absorption vessel F.

Stop heating and, still maintaining the flow of nitrogen, titrate the sulphide in the absorption vessel F with the standard volumetric mercury (II) nitrate solution (**8.3.9**) contained in the microburette H, until the indicator colour changes from yellow to red.

8.6 Expression of results. The content of sulphur compounds, expressed as milligrams of potassium sulphate (K_2SO_4) per kilogram, is given by the following formula:

$$(V_1 - V_0) \times \frac{1}{1000} \times \frac{1000}{m} \times 87 = \frac{(V_1 - V_0) \times 87}{m}$$

where

V_1 is the volume of the standard volumetric mercury (II) nitrate solution (**8.3.9**) used for the determination (in ml)

V_0 is the volume of the standard volumetric mercury (II) nitrate solution (**8.3.9**) used for the blank test (in ml)

m is the mass of the test portion (**8.5.1**) (in g)

9 Determination of nitrate content

9.1 Field of application. The method is applicable to products having nitrate contents, expressed as potassium nitrate (KNO_3), in the range 3 mg/kg to 30 mg/kg for liquid products and 6 mg/kg to 60 mg/kg for solid products.

9.2 Principle. The nitrate in the neutralized sample is reduced to nitrite with cadmium sponge at pH 9.6 and the resulting nitrite is determined colorimetrically as the diazonium compound of sulphanilic acid and *N*-2-aminoethyl-1-naphthylamine at a wavelength of 550 nm.

9.3 Reagents. All reagents shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

9.3.1 *Ammonia/ammonium chloride buffer solution pH 9.6.* Adjust the pH of a 38 g/l ammonium chloride solution to 9.6 by the addition of concentrated ammonia solution, ρ 0.91 g/ml, using a pH meter.

9.3.2 *Cadmium sponge.* Prepare by immersing zinc rods in 200 g/l cadmium sulphate solution. Wash the precipitated cadmium with 0.1N hydrochloric acid solution and then several times with water. Store under water.

9.3.3 *Colour reagent.* Prepare immediately before use by mixing one volume of aqueous *N*-2-aminoethyl-1-naphthylamine hydrochloride solution, 1.3 g/l, with one volume of aqueous sulphanilic acid solution, 0.85 g/l, and adding three volumes of glacial acetic acid.

9.3.4 *Potassium nitrate, standard solution,* corresponding to 100 mg of potassium nitrate per litre. Dissolve 0.100 g of potassium nitrate in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 100 μg of KNO_3.

9.3.5 *Sulphuric acid,* 20 % *v*/*v* solution.

9.3.6 *Phenolphthalein indicator solution,* 10 g/l, prepared as described in BS 4123.

9.4 Apparatus. Ordinary laboratory apparatus and the following are required.

9.4.1 *Spectrophotometer,* or

9.4.2 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of about 550 nm.

9.4.3 *Optical cells,* 1 cm optical path length.

9.4.4 *pH meter*

9.4.5 *Mechanical shaker*

9.5 Procedure

9.5.1 *Test portion.* Transfer 40.0 ml of the solution A (**3.4.2**) into a 100 ml one-mark volumetric flask.

9.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **9.5.4.2** but using 60 ml of water instead of the test solution (**9.5.4.1**).

9.5.3 ***Preparation of the calibration graph***

9.5.3.1 *Preparation of the standard matching solutions.* Into a series of six 100 ml one-mark volumetric flasks, introduce the volumes of the standard potassium mitrate solution (**9.3.4**) shown in the following table:

Standard potassium nitrate solution (9.3.4)	**Corresponding mass of potassium nitrate (KNO_3)**
ml	µg
0[a]	0
1.0	100
2.0	200
3.0	300
4.0	400
5.0	500

[a] Compensation solution.

9.5.3.2 *Colour development.* Treat each of the standard matching solutions as follows.

Add 5 ml of the ammonia/ammonium chloride buffer solution (**9.3.1**) and 2 g of the moist cadmium sponge (**9.3.2**) and dilute to about 80 ml with water. Shake vigorously by means of the mechanical shaker (**9.4.5**) for 5 min, dilute to the mark with water and mix.

Filter off approximately 50 ml of solution and, using a pipette, transfer 20.0 ml of the filtrate into a 50 ml one-mark volumetric flask. Add 5 ml of the freshly prepared colour reagent (**9.3.3**), dilute to the mark with water and mix. Allow to stand in subdued light at a temperature of 20 ± 5 °C for 10 min.

9.5.3.3 *Photometric measurements.* Carry out the photometric measurements using either the spectrophotometer (**9.4.1**) at the wavelength of maximum absorption (about 550 nm) or the photometric absorptiometer (**9.4.2**), fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

9.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**9.5.3.1**) from those of the standard matching solutions (**9.5.3.1**). Plot a graph having, for example, the potassium nitrate content, expressed in micrograms of potassium nitrate added originally to the 100 ml one-mark volumetric flask, as abscissae and the corresponding values of absorbance as ordinates.

9.5.4 ***Determination***

9.5.4.1 *Preparation of the test solution.* Measure 40.0 ml of the solution A (**3.4.2**) into a 250 ml beaker and titrate with the sulphuric acid solution (**9.3.5**) using the phenolphthalein solution (**9.3.6**) as indicator. Record the volume of the sulphuric acid solution used for the titration (*X* ml) and discard the titrated solution.

To the test portion in the 100 ml one-mark volumetric flask (**9.5.1**), add slowly *X* ml of the sulphuric acid solution (**9.3.6**), swirl and allow to cool to room temperature.

9.5.4.2 *Colour development.* Using the neutralized test solution in the 100 ml one-mark volumetric flask (**9.5.4.1**), follow the procedure specified in **9.5.3.2**.

9.5.4.3 *Photometric measurement.* Carry out the photometric measurement on the test solution (**9.5.4.2**) and on the blank test solution (**9.5.2**) following the procedure specified in **9.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution A (**3.4.2**) as test portion (**9.5.1**) and modifying the calculation accordingly.

9.6 Expression of results. By means of the calibration graph (**9.5.3.4**) determine the quantity of potassium nitrate (KNO_3) corresponding to the value of the photometric measurement.

The nitrate content, expressed as milligrams of potassium nitrate (KNO_3) per kilogram, is given by the following formula:

$$\frac{m_1 - m_2}{1000} \times \frac{100}{8\,m_0} \times 1000 = \frac{m_1 - m_2}{m_0} \times 12.5$$

where

m_1 is the mass of potassium nitrate in the test solution (in µg)

m_2 is the mass of potassium nitrate in the blank test solution (in μg)

m_0 is the mass of the test sample used to prepare the solution A (in g)

10 Determination of cyanide content

10.1 Field of application. This method is applicable to products having cyanide contents, expressed as potassium cyanide (KCN), in the range 0.5 mg/kg to 5 mg/kg for liquid products and 1 mg/kg to 10 mg/kg for solid products.

10.2 Principle. A test portion is carefully neutralized with hydrochloric acid solution. Slight excesses of both acid and bromine water are added to convert any cyanide present to cyanogen bromide. Excess bromine is removed and *p*-phenylenediamine is added to give a characteristic colour on reaction with cyanogen bromide. The colour is measured spectrophotometrically at a wavelength of approximately 515 nm.

10.3 Reagents. The reagents used shall be of recognized analytical quality and all solutions shall be freshly prepared. Water complying with the requirements of BS 3978 shall be used throughout.

10.3.1 *Hydrochloric acid,* approximately 2N solution.

10.3.2 *Potassium hydroxide,* 200 g/l solution.

10.3.3 *Bromine water,* saturated.

10.3.4 *Arsenic trioxide,* 20 g/l solution.
Dissolve 2.0 g of arsenic trioxide in 100 ml of water by boiling under reflux.

10.3.5 *Mixed amines solution.* Prepare solutions of *p*-phenylenediamine dihydrochloride (solution 1) and pyridine (solution 2) as follows.

Solution 1. Dissolve 0.1 g of *p*-phenylenediamine dihydrochloride in 25 ml of 0.1N hydrochloric acid solution contained in a 50 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Solution 2. Mix 540 ml of pyridine, 360 ml of water and 90 ml of hydrochloric acid, approximately 36 % *m*/*m* solution.

Mix one volume of the solution 1 with three volumes of the solution 2. This mixture comprises the mixed amine solution.

10.3.6 *Potassium cyanide,* standard solution corresponding to 0.1 g of potassium cyanide per litre. Dissolve 0.100 g of potassium cyanide in water containing 5 ml of 1N sodium hydroxide solution. Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0.1 mg of KCN.

10.3.7 *Potassium cyanide,* standard solution corresponding to 2.5 mg of potassium cyanide per litre. Dilute 2.5 ml of the standard potassium cyanide solution (**10.3.6**) to the mark in a 100 ml one-mark volumetric flask and mix.

1 ml of this solution contains 2.5 μg of KCN.

10.3.8 *Phenolphthalein indicator solution.* 10 g/l, prepared as described in BS 4123.

10.4 Apparatus. Ordinary laboratory apparatus and the following are required.

10.4.1 *Spectrophotometer,* or

10.4.2 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of about 500 nm.

10.4.3 *Optical cells,* 4 cm optical path length.

10.4.4 *Water bath,* capable of being maintained at a temperature of 20 ± 1 °C.

10.5 Procedure

10.5.1 *Test portion.* Measure 5.0 ml of the solution A (**3.4.2**) into a 50 ml one-mark volumetric flask.

10.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as described in **10.5.4.2** but using 20 ml of water, instead of the test solution (**10.5.4.1**), in a 50 ml one-mark volumetric flask.

10.5.3 ***Preparation of the calibration graph***

10.5.3.1 *Preparation of the standard matching solutions.* Using a 50 ml burette measure 5.0 ml of the potassium hydroxide solution (**10.3.2**) into a 100 ml conical flask. Add 10 ml of water and one drop of the phenolphthalein solution (**10.3.8**) and titrate to the end point with the hydrochloric acid solution (**10.3.1**). Note the volume of the hydrochloric acid used (*X* ml) and discard the titrated solution.

Into each of a series of five 50 ml one-mark volumetric flasks, introduce 5.0 ml of the potassium hydroxide solution (**10.3.2**) and measure accurately, from a 10 ml burette, the volumes of the standard cyanide solution (**10.3.7**) shown in the following table:

Standard cyanide solution (10.3.7)	Corresponding mass of potassium cyanide (KCN)
ml	μg
0[a]	0
1.0	2.5
2.0	5.0
3.0	7.5
4.0	10.0

[a] Compensation solution.

Treat the contents of each flask as follows.

Add sufficient water from a burette to make the volume up to 15 ml. Carefully neutralize the solution by adding slowly *X* ml of the hydrochloric acid solution (**10.3.1**), ensuring that the temperature of the solution does not exceed 25 °C during the neutralization.

10.5.3.2 *Colour development.* Treat the contents of each flask (**10.5.3.1**) as follows.

Add 5 ml of the hydrochloric acid solution (**10.3.1**), followed immediately by 2 ml of the bromine water (**10.3.3**). Stopper immediately and mix.

Allow to stand for 2 min at room temperature, add 2 ml of the arsenic trioxide solution (**10.3.4**) and shake to ensure removal of excess bromine from the solution and the vapour phases. Immediately add 14 ml of the mixed amines solution (**10.3.5**), dilute to the mark and mix. Immediately place the flask in the water bath (**10.4.4**) maintained at a temperature of 20 ± 1 °C.

10.5.3.3 *Photometric measurements.* Carry out the photometric measurements within 30 ± 2.5 min of the time of final mixing in **10.5.3.2**, using either the spectrophotometer (**10.4.1**) at the wavelength of maximum absorption (about 515 nm), or the photometric absorptiometer (**10.4.2**) fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

10.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**10.5.3.1**) from those of the standard matching solutions (**10.5.3.1**). Plot a graph having, for example, the potassium cyanide content, expressed in micrograms of potassium cyanide per 50 ml of standard matching solution, as abscissae and the corresponding values of absorbance plotted as ordinates.

10.5.4 ***Determination***

10.5.4.1 *Preparation of the test solution.* Measure accurately 5.0 ml of the solution A (**3.4.2**) into a 100 ml conical flask and titrate with the hydrochloric acid solution (**10.3.1**) as described in the first paragraph of **10.5.3.1**. Let the value of the titration be *Y* ml of the hydrochloric acid solution (**10.3.1**).

Into the one-mark volumetric flask containing the test portion (**10.5.1**), add 10.0 ml of water. Carefully neutralize this solution by slowly adding *Y* ml of the hydrochloric acid solution (**10.3.1**), ensuring that the temperature of the solution does not exceed 25 °C during neutralization.

10.5.4.2 *Colour development.* Using the test solution (**10.5.4.1**) in the 50 ml one-mark volumetric flask, follow the procedure specified in **10.5.3.2**.

10.5.4.3 *Photometric measurement.* Carry out the photometric measurement on the test solution (**10.5.4.2**) and on the blank test solution (**10.5.2**) following the procedure specified in **10.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution A (**3.4.2**) as test portion (**10.5.1**) and modifying the preparation of the test solution (**10.5.4.1**) and the calculation accordingly.

10.6 Expression of results. By means of the calibration graph (**10.5.3.4**), determine the quantity of potassium cyanide (KCN) corresponding to the value of the photometric measurement.

The cyanide content, expressed as milligrams of potassium cyanide (KCN) per kilogram, is given by the following formula:

$$\frac{m_1 - m_2}{1000} \times \frac{500}{m_0 \times 5} \times 1000 = \frac{m_1 - m_2}{m_0} \times 100$$

where

m_1 is the mass of potassium cyanide found in the test solution (in μg)

m_2 is the mass of potassium cyanide found in the blank test solution (in μg)

m_0 is the mass of the test sample used to prepare the solution A (**3.4.2**) (in g)

11 Determination of silica content

11.1 Field of application. The method is applicable to products having silica contents, expressed as silica (SiO_2), in the range 10 mg/kg to 100 mg/kg for liquid products and 20 mg/kg to 200 mg/kg for solid products.

11.2 Principle. The yellow oxidized silico-molybdic complex is formed, at pH 1.1 ± 0.2, in the presence of boric acid to suppress interference by fluoride.

This complex is selectively reduced with a mixture of 4-amino-3-hydroxynaphthalene-1-sulphonic acid, sodium metabisulphite and sodium sulphite, in the presence of oxalic acid and in a strongly acid medium in order to suppress interference by phosphates.

The blue complex is spectrophotometrically measured at a wavelength, preferably of about 795 nm, but failing this, of about 680 nm.

11.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout. All reagents shall be stored in polyethylene bottles.

11.3.1 *Sulphuric acid,* approximately 9N solution.

11.3.2 *Hydrochloric acid,* approximately 2N solution.

11.3.3 *Boric acid,* saturated solution (about 48 g/l).

11.3.4 *Oxalic acid,* 100 g/l solution.

11.3.5 *Sodium molybdate dihydrate,* ($Na_2MoO_4.2H_2O$), 140 g/l solution.

Dissolve 35 g of this product in 200 ml of water at about 50 °C in a polyethylene beaker. Cool to room temperature, transfer to a 250 ml one-mark volumetric flask, dilute to the mark, mix and transfer to a polyethylene bottle.

If necessary, filter the solution before use.

11.3.6 ***Reducing solution***

11.3.6.1 Dissolve 7 g of anhydrous sodium sulphite in 50 ml of water. Add 1.5 g of 4-amino-3-hydroxynaph-thalene-1-sulphonic acid and dissolve by trituration.

11.3.6.2 Dissolve 90 g of anhydrous sodium metabisulphite in 900 ml of water.

11.3.6.3 Mix the solutions (**11.3.6.1** and **11.3.6.2**) and dilute to 1 000 ml. Filter if necessary and store the solution in a refrigerator. Renew every 14 days.

11.3.7 *Potassium chloride,* 60 g/l solution.

11.3.8 *Silica,* standard solution, corresponding to 0.500 g/l of silica.

In a platinum crucible, weigh to the nearest 0.001 g, either:

a) 0.500 g of silica (SiO_2) produced by calcining pure silicic acid (H_2SiO_3) at 1 000 °C to constant mass and cooling in a dessicator;

or

b) 0.500 g of finely ground pure quartz, which has been previously calcined for 1 h at 1 000 °C and cooled in a desiccator.

Add 5 g of anhydrous sodium carbonate to the crucible. Mix well, preferably with a platinum spatula, and fuse carefully. Allow to cool, add warm water, heat moderately until the contents of the crucible are completely dissolved, cool, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer the solution immediately to a polyethylene bottle.

1 ml of this standard solution contains the equivalent of 0.500 mg of SiO_2.

11.3.9 *Silica,* standard solution, corresponding to 10 mg of silica per litre. Transfer 20.0 ml of the standard silica solution (**11.3.8**) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0.010 mg of SiO_2.

Prepare this standard solution at the time of use.

11.3.10 *Phenolphthalein indicator solution,* 10 g/l, prepared as described in BS 4123.

11.4 Apparatus. Ordinary laboratory apparatus and the following are required.

11.4.1 *Spectrophotometer,* or

11.4.2 *Photoelectric absorptiometer,* fitted with suitable filters with a maximum transmission at a wavelength of about 795 nm.

NOTE If such filters are not available, operate at a wavelength of about 680 nm.

11.4.3 *Optical cells,* 2 cm optical path length (for use at 795 nm) or 4 cm optical path length (for use at 680 nm).

11.5 Procedure

11.5.1 ***Test portion***

11.5.1.1 *Preparation.* Weigh, to the nearest 0.01 g, in a 100 ml polyethylene beaker, 2.6 ± 0.1 g of the solution A (**3.4.2**). During this operation, prevent the solution from coming into contact with glass.

11.5.1.2 *Determination of the mass of* 10.0 ml *of the solution A.* Determine, to the nearest 0.01 g, the mass of 10.0 ml of the solution A (**3.4.2**), measured by means of a pipette or a burette, to enable the mass of the test portion (**11.5.1.1**) to be converted to a volume when the results are calculated.

11.5.2 *Blank test.* At the same time as the determination (**11.5.4**), carry out a blank test following the same procedure and using the same quantities of all the reagents as in the determination (**11.5.4**), but replacing the test portion (**11.5.1.1**) by 10.0 ml of the potassium chloride solution (**11.3.7**).

11.5.3 ***Preparation of the calibration graph***

11.5.3.1 *Preparation of the standard matching solutions.* Into each of a series of four 100 ml polyethylene beakers, introduce the volumes of the standard silica solution (**11.3.9**) shown in the following table.

Standard silica solution (11.3.9)	**Corresponding mass of silica (SiO_2)**
ml	mg
0[a]	0
2.0	0.02
5.0	0.05
10.0	0.10

[a] Compensation solution.

11.5.3.2 *Colour development.* Treat the contents of each beaker (**11.5.3.1**) as follows.

Add 10.0 ml of the potassium chloride solution (**11.3.7**), dilute to 25 ml and then add, mixing after each addition, 8.0 ml of the hydrochloric acid solution (**11.3.2**), 20 ml of the boric acid solution (**11.3.3**) and 10 ml of the sodium molybdate solution (**11.3.5**). The pH of the solution should now be about 1.1. Wait for 10 min and then add, stirring after each addition, 5 ml of the oxalic acid solution (**11.3.4**) and 20 ml of the sulphuric acid solution (**11.3.1**). Allow to stand for 2 min, then add 2 ml of the reducing solution (**11.3.6**). Transfer to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

11.5.3.3 *Photometric measurements.* After at least 10 min, but not more than 40 min, carry out the photometric measurements using the spectrophotometer (**11.4.1**), at the wavelength of maximum absorption (about 795 nm), or the photoelectric absorptiometer (**11.4.1**), fitted with appropriate filters, after having adjusted the instrument to zero absorbance against water.

11.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**11.5.3.1**) from those of the standard matching solutions (**11.5.3.1**). Plot a graph having, for example, the silica content, expressed in milligrams of silica per 100 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

11.5.4 ***Determination***

11.5.4.1 *Colour development.* Add one drop of the phenolphthalein indicator solution (**11.3.10**) to the test portion (**11.5.1.1**) and neutralize with the hydrochloric acid solution (**11.3.2**). Then add, swirling after each addition, 8.0 ml of the hydrochloric acid solution (**11.3.2**), 20 ml of the boric acid solution (**11.3.3**) and 10 ml of the sodium molybdate solution (**11.3.5**). Transfer to a 100 ml one-mark volumetric flask, dilute to the mark and mix. Wait for 10 min then add, swirling after each addition, 5 ml of the oxalic acid solution (**11.3.4**) and 20 ml of the sulphuric acid solution (**11.3.1**). Allow to stand for 2 min, then add 2 ml of the reducing solution (**11.3.6**), dilute to the mark and mix.

11.5.4.2 *Spectrophotometric measurement.* After at least 10 min, but not more than 40 min, carry out the photometric measurement on the test solution (**11.5.4.1**) and on the blank test solution (**11.5.2**) following the procedure described in **11.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution A (**3.4.2**) as the test portion (**11.5.1**) and modifying the calculation accordingly.

11.6 Expression of results. By means of the calibration graph (**11.5.3.4**), determine the mass of silica corresponding to the value of the photometric measurements.

The silica content, expressed as milligrams of silica (SiO_2) per kilogram, is given by the following formula:

$$(m_1 - m_2) \times \frac{m_4}{10\,m_3} \times 500 \times \frac{1000}{m_0}$$

$$= \frac{m_4\,(m_1 - m_2)}{m_0 m_3} \times 5 \times 10^4$$

where

m_1 is the mass of silica found in the determination (in mg)

m_2 is the mass of silica found in the blank test solution (in mg)

m_3 is the mass of the test portion (**11.5.1**) (in g)

m_4 is the mass of 10.0 ml of the solution A (**3.4.2**) (in g)

m_0 is the mass of the test sample used to prepare solution A (**3.4.2**) (in g)

12 Determination of calcium content

12.1 Field of application. This method is applicable to products having calcium contents, expressed as calcium oxide (CaO) as follows.

a) *Using the air/acetylene flame.* For solid products containing more than 4 mg/kg and for liquid products containing more than 2 mg/kg.

b) *Using the nitrous oxide/acetylene flame.* For solid products containing more than 1 mg/kg and for liquid products containing more than 0.5 mg/kg.

12.2 Principle. An acidified solution of the sample is analyzed by atomic absorption spectrophotometry using the calcium line at a wavelength of about 422.7 nm and either a nitrous oxide/acetylene flame or an air/acetylene flame. In the latter case, lanthanum chloride solution is added to the test and standard solutions to suppress certain interferences.

12.3 Reagents. The reagents used shall be of recognized analytical quality. Water double-distilled in apparatus constructed from borosilicate glass, or of equivalent purity, shall be used throughout.

12.3.1 *Hydrochloric acid,* approximately 5N solution.

12.3.2 *Lanthanum chloride solution,* equivalent to 5 g/l of lanthanum.

NOTE This solution is not required when the nitrous oxide/acetylene flame is used.

Prepare the lanthanum chloride solution by either of the procedures given below.

12.3.2.1 Dissolve 5.9 g of lanthanum oxide (La_2O_3) in 100 ml of the hydrochloric acid solution (**12.3.1**). Dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

12.3.2.2 Dissolve 13.4 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

This solution (**12.3.2**) shall satisfy the following test. 20.0 ml of the solution, diluted to the mark in a 100 ml one-mark volumetric flask, shall give an absorbance for calcium not greater than that obtained for a solution containing 3 µg of calcium per 100 ml when tested in accordance with **12.5.2.2** and **12.5.2.3**. In addition, calculate the actual quantity of calcium corresponding to the measured absorbance so that account may be taken of these values in checking the purity of the potassium hydroxide (**12.3.3**).

12.3.3 *Potassium hydroxide.* Preferably use a product having a calcium content, expressed as calcium oxide (CaO), not greater than 4 mg/kg. Check this by the following procedure.

From the calibration graph (**12.5.2.4**), note the absorbance obtained with the standard matching solution number 0. (**12.5.2.1**) in establishing the graph. The calcium value corresponding to this absorbance shall not exceed 2.5 µg plus the quantity of calcium found in the check test on the lanthanum chloride solution (**12.3.2**), in the case where this is used.

12.3.4 *Potassium chloride,* 66 g/l acid solution. Place 25.0 g of the potassium hydroxide (**12.3.3**) in a 400 ml polyethylene beaker. Dissolve, with cooling, in 100 ml of water. Acidify by adding, while stirring, 100 ml of the hydrochloric acid solution (**12.3.1**). Transfer quantitatively to a 500 ml conical flask. Boil for 5 min, cool and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

12.3.5 *Calcium,* standard solution, corresponding to 0.100 g of calcium per litre. Weigh, to the nearest 0.0001 g, 0.2497 g of calcium carbonate, previously dried at a temperature of 250 °C and cooled in a desiccator. Place in a 250 ml beaker and dissolve in 25 ml of the hydrochloric acid solution (**12.3.1**). Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0.1 mg of Ca.

12.3.6 *Calcium,* standard solution, corresponding to 10 mg of calcium per litre. Transfer 20.0 ml of the standard calcium solution (**12.3.5**) into a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this solution immediately before use. 1 ml of this standard solution contains 10 µg of Ca.

12.4 Apparatus. Ordinary laboratory apparatus and the following are required.

NOTE It is essential that all glassware and reagent bottles be made either from borosilicate glass or from glass which will not, during use, give contamination with calcium.

12.4.1 *Atomic absorption spectrophotometer,* fitted with a burner fed either with acetylene and nitrous oxide or acetylene and air.

NOTE It is essential, if the instrument is calibrated in percentage absorption, that all readings be converted to absorbance before use.

12.4.2 *Hollow cathode lamp for calcium*

12.5 Procedure. Two procedures are described, a simple procedure (**12.5.2**) using a calibration graph and a more laborious method (**12.5.3**) using the technique of standard additions. The latter overcomes the effects of certain interferences and shall always be used unless it has been demonstrated that the former is suitable for a particular source of test sample.

12.5.1 *Test portion.* Transfer 10.0 ml of the solution B (**3.4.3**) to a 100 ml one-mark volumetric flask.

12.5.2 ***Procedure using calibration graphs***

12.5.2.1 *Preparation of the standard matching solutions.* Into each of a series of four 100 ml one-mark volumetric flasks, place 20 ml of the acid potassium chloride solution (**12.3.4**). If air/acetylene is to be used, add 20 ml of the lanthanum chloride solution (**12.3.2**).

Then add accurately by means of a 10 ml burette the volumes of the standard calcium solution (**12.3.6**) indicated in the following table.

Standard matching solution number	Standard calcium solution (12.3.6)	Corresponding mass of calcium (Ca)
	ml	µg
0[a]	0	0
1	1.0	10
2	2.0	20
3	3.0	30

[a] Blank standard matching solution.

Dilute to the mark with water and mix.

12.5.2.2 *Adjustment of the apparatus.* Fit the hollow cathode calcium lamp (**12.4.2**) to the apparatus (**12.4.1**) and switch on the current a sufficient time in advance to ensure stabilization. Adjust the instrument to give maximum absorbance at a wavelength of about 422.7 nm. Set the lamp input current, slit aperture, gas pressures and the instrument sensitivity according to the recommended values.

12.5.2.3 *Spectrophotometric measurements.* Aspirate the series of standard matching solutions (**12.5.2.1**) into the flame and measure the absorbance for each. Spray water through the burner after each measurement. Take care to keep the aspiration rate constant throughout this series of measurements.

12.5.2.4 *Preparation of the calibration graph.* Plot a graph having, for example, the number of micrograms of calcium contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the measured absorbances, less the measured value for the blank standard matching solution, as ordinates.

It is essential that the calibration graph obtained is rectilinear.

12.5.2.5 *Determination*

12.5.2.5.1 *Preparation of the test solution.* Dilute the test portion (**12.5.1**) in the 100 ml one-mark volumetric flask to the mark with water and mix, having first added 20.0 ml of the lanthanum chloride solution (**12.3.2**) if using the air/acetylene mixture (**12.1**) for the determination.

12.5.2.5.2 *Blank test.* Pipette 10.0 ml of the solution C (**3.4.4**) into a 100 ml one-mark volumetric flask and treat in exactly the same way as specified in **12.5.2.5.1**.

12.5.2.5.3 *Spectrophotometric measurements.* Following the procedure specified in **12.5.2.2** and **12.5.2.3**, determine, in order, the absorbances for the following solutions:

a) the four standard matching solutions (**12.5.2.1**);

b) the test solution (**12.5.2.5.1**);

c) the blank test solution (**12.5.2.5.2**);

d) the standard matching solution the absorbance of which is closest to that of the test solution (**12.5.2.5.1**).

12.5.3 ***Procedure using standard additions***

12.5.3.1 *Preparation of the test solutions with additions.* Into each of a series of four 100 ml one-mark volumetric flasks, pipette 10.0 ml of the solution B (**3.4.3**). To the first, second and third flasks add, respectively, by means of a 10 ml burette, 1.0 ml, 2.0 ml and 3.0 ml of the standard calcium solution (**12.3.6**). If air/acetylene is to be used, add to all four flasks 20.0 ml of the lanthanum chloride solution (**12.3.2**). Dilute to the mark with water and mix.

12.5.3.2 *Preparation of the test blank solutions with additions.* Into each of a series of four 100 ml one-mark volumetric flasks, pipette 10.0 ml of the solution C (**3.4.4**) and treat in exactly the same way as the test solutions (**12.5.3.1**).

12.5.3.3 *Spectrophotometric measurements.* Following the procedure specified in **12.5.2.2** and **12.5.2.3**, determine the absorbances for the following solutions:

a) the four test solutions with additions;

b) the four test blank solutions with additions.

12.6 Expression of results

12.6.1 *Procedure using calibration graphs.* The calcium content, expressed as milligrams of calcium oxide (CaO) per kilogram, is given by the following formula:

$$m_1 \times \frac{1}{1000} \times \frac{A_1 - A_0}{A_2 - A_3} \times \frac{1000}{10} \times \frac{1000}{m_0} \times \frac{56}{40}$$

$$= \frac{140\, m_1\, (A_1 - A_0)}{m_0\, (A_2 - A_3)}$$

where

A_0 is the absorbance for the blank test solution (**12.5.2.5.2**)

A_1 is the absorbance for the test solution (**12.5.2.5.1**)

A_2 is the mean of the absorbances for the standard matching solutions closest to that of the test solution, measured before and after the test solution

A_3 is the absorbance of the blank standard matching solution (**12.5.2.1**)

m_0 is the mass of the test portion used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of calcium contained in the standard matching solution closest to the test solution (in μg)

12.6.2 *Procedure using standard additions.* Plot a graph having, for example, the number of micrograms of calcium added to the test solutions as abscissae and the corresponding values of the measured absorbances as ordinates, ensuring that the ordinate axis is placed in the centre of the page.

Extrapolate the graph to zero absorbance and read off, from the point of intersection of the graph with the abscissa, the number of micrograms of calcium in the test solution containing no added calcium.

Repeat the procedure with the absorbances obtained on the test blank solutions. Read off the number of micrograms of calcium in the test blank solution containing no added calcium.

The calcium content, expressed as milligrams of calcium oxide (CaO) per kilogram, is given by the following formula:

$$(m_2 - m_3) \times \frac{1000}{10} \times \frac{1.4}{m_0} = \frac{m_2 - m_3}{m_0} \times 140$$

where

m_0 is the mass of the test portion used in the preparation of the solution B (**3.4.3**) (in g)

m_2 is the mass of calcium in the test solution containing no added calcium (in µg)

m_3 is the mass of calcium in the blank test solution containing no added calcium (in µg)

13 Determination of iron content

13.1 Field of application. The method is applicable to products having iron contents, expressed as Fe, in the range 1 mg/kg to 15 mg/kg for liquid products and 2 mg/kg to 30 mg/kg for solid products.

13.2 Principle. The trivalent iron is reduced by hydroxylammonium chloride, and is reacted to form the bivalent iron/1,10-phenanthroline complex in a buffered system. The iron is determined photometrically at a wavelength of about 510 nm.

13.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

13.3.1 *Hydrochloric acid solution.* ρ approximately 1.18 g/ml, about 36 % *m*/*m* solution or approximately 11N.

13.3.2 *Ammonia solution.* ρ approximately 0.91 g/ml, about 25 % *m*/*m* solution or approximately 13N, with a maximum iron content of 0.2 mg/kg.

13.3.3 *Hydroxylammonium chloride* ($NH_2OH.HCl$), 10 g/l solution.

13.3.4 *Buffer solution,* pH 4.9. Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in about 500 ml of water. Add 240 ml of glacial acetic acid (ρ approximately 1.05 g/ml, 99 % *m*/*m* to 100 % *m*/*m* solution or approximately 17.4N) to the solution and dilute to 1 litre.

13.3.5 *1,10-Phenanthroline hydrochloride monohydrate* ($C_{12}H_8N_2.HCl.H_2O$), 2.5 g/l solution.

This reagent may be replaced by a solution of 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$).

13.3.6 *Iron,* standard solution, corresponding to 0.200 g of iron per litre.

Dissolve 1.404 g of ammonium iron (II) sulphate hexahydrate ($(NH_4)_2Fe(SO_4)_2.6H_2O$, in 200 ml of water. Add 20 ml of sulphuric acid, ρ approximately 1.84 g/ml, cool to room temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 0.200 mg of Fe.

13.3.7 *Iron,* standard solution, corresponding to 0.010 g of iron per litre.

Transfer 25.0 ml of the standard iron solution (**13.3.6**) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 0.010 mg of Fe.

13.4 Apparatus. Ordinary laboratory apparatus and the following are required.

13.4.1 *Spectrophotometer,* or

13.4.2 *Photoelectric absorptiometer* fitted with filters providing maximum transmission in the wavelength range 500 nm to 520 nm.

13.4.3 *Optical cells,* 4 cm optical path length.

13.5 Procedure

13.5.1 *Test portion.* Measure 50 ml of the solution B (**3.4.3**) into a 100 ml one-mark volumetric flask.

13.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **13.5.4.1** but using 50 ml of the solution C (**3.4.4**) instead of the test portion (**13.5.1**), in a 100 ml one-mark volumetric flask.

13.5.3 ***Preparation of the calibration graph***

13.5.3.1 *Preparation of the standard matching solutions.* Into each of a series of five 100 ml one-mark volumetric flasks, introduce the volumes of the standard iron solution (**13.3.7**) shown in the following table.

Standard iron solution (13.3.7)	Corresponding mass of iron (Fe)
ml	mg
0[a]	0
2.5	0.025
5.0	0.050
10.0	0.100
15.0	0.150

[a] Compensation solution.

13.5.3.2 *Colour development.* Treat the contents of each flask (**13.5.3.1**) as follows.

Add 0.5 ml of the hydrochloric acid solution (**13.3.1**) and the amount of water necessary to make up the volume to about 50 ml. Then add 5 ml of the hydroxylammonium chloride solution (**13.3.3**), 5 ml of the 1,10-phenanthroline hydrochloride solution (**13.3.5**) and 25 ml of the buffer solution (**13.3.4**). Dilute to the mark, mix and allow to stand for 10 min.

13.5.3.3 *Photometric measurements.* Carry out the photometric measurements using either the spectrophotometer, at the wavelength of maximum absorption (about 510 nm), or the photoelectric absorptiometer, fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

13.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**13.5.3.1**) from those of the standard matching solutions (**13.5.3.1**). Plot a graph having, for example, the iron content, expressed in milligrams per 100 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

13.5.4 ***Determination***

13.5.4.1 *Colour development.* Treat the test portion (**13.5.1**) in the 100 ml one-mark volumetric flask as follows.

Add 0.5 ml of the hydrochloric acid solution and 5 ml of the hydroxylammonium chloride solution (**13.3.3**), 5 ml of the 1,10-phenanthroline hydrochloride solution (**13.3.5**) and 25 ml of the buffer solution (**13.3.4**). Dilute to the mark, mix and allow to stand for 10 min.

13.5.4.2 *Photometric measurement.* Carry out the photometric measurement on the test solution (**13.5.4.1**) and on the blank test solution (**13.5.2**) following the procedure specified in **13.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution B (**3.4.3**) as the test portion (**13.5.1**) and modifying the calculation accordingly.

13.6 Expression of results. By reference to the calibration graph (**13.5.3.4**), determine the quantity of Fe corresponding to the value of the absorbance measured.

The iron content, expressed as milligrams of iron (Fe) per kilogram is given by the following formula:

$$(m_1 - m_2) \times \frac{1000}{m_0} \times \frac{1000}{50} = \frac{(m_1 - m_2)}{m_0} \times 2 \times 10^4$$

where

m_0 is the mass of the sample used to prepare solution B (**3.4.3**) (in g)

m_1 is the mass of iron found in the test solution (in mg)

m_2 is the mass of iron found in the blank test solution (in mg)

14 Determination of zinc content

14.1 Field of application. The method is applicable to products having zinc contents, expressed as Zn, in the range 1 mg/kg to 12 mg/kg for liquid products or 2 mg/kg to 25 mg/kg for solid products.

14.2 Principle. The zinc content of an acidified solution of the sample is determined by atomic absorption spectrophotometry.

14.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

14.3.1 *Hydrochloric acid,* approximately 5N solution.

14.3.2 *Potassium chloride,* 66 g/l solution.

14.3.3 *Zinc,* standard solution corresponding to 0.20 g of zinc per litre. Dissolve 0.20 g of pure zinc in a mixture of 5 ml of hydrochloric acid (ρ approximately 1.18 g/ml) and 10 ml of water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 0.2 mg of Zn.

14.3.4 *Zinc,* standard solution corresponding to 2.0 mg of zinc per litre. Pipette 10.0 ml of the standard zinc solution (**14.3.3**) into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Prepare immediately before use.

1 ml of this solution contains 2.0 µg of Zn.

14.4 Apparatus. Ordinary laboratory apparatus and the following are required.

14.4.1 *Atomic absorption spectrophotometer,* fitted with suitably regulated supplies of air and acetylene and background corrector.

14.4.2 *Zinc hollow cathode lamp*

14.5 Procedure

14.5.1 *Test portion.* Transfer 10.0 ml of the solution B (**3.4.3**) into a 100 ml one-mark volumetric flask.

14.5.2 *Preparation of standard matching zinc solutions.* Into each of a series of five 100 ml one-mark volumetric flasks, measure accurately the volumes of the standard zinc solution (**14.3.4**) shown in the following table.

Standard zinc solution (14.3.4)	**Corresponding mass of zinc (Zn)**
ml	µg
0[a]	0
2.5	5.0
5.0	10.0
10.0	20.0
15.0	30.0

[a] Blank standard matching solution.

Treat the contents of each flask as follows.

Dilute to between 55 ml and 60 ml with water. Add 2 ml of the hydrochloric acid solution (**14.3.1**) followed by 20 ml of the potassium chloride solution (**14.3.2**). Dilute to the mark and mix.

14.5.3 *Adjustment of the apparatus.* Fit the zinc hollow cathode lamp (**14.4.2**) to the spectrophotometer (**14.4.1**) and switch on the current a sufficient time in advance to ensure stabilization. Adjust the wavelength to give maximum absorbance at a wavelength of about 214.0 nm. Set the lamp input current, slit aperture, air and acetylene pressures and the instrument sensitivity according to the recommended values. Switch on the background corrector and adjust, allowing it to warm up for the recommended time.

14.5.4 *Spectrophotometric measurements.* Aspirate the series of standard matching solutions (**14.5.2**) into the flame and measure the absorbance for each. Spray water through the burner after each measurement. Take care to keep the aspiration rate constant throughout this series of measurements.

14.5.5 *Preparation of the calibration graph.* Plot a graph having, for example the number of micrograms of zinc contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the measured absorbances, less the measured value for the blank standard matching solution, as ordinates.

NOTE It is essential that the calibration graph obtained is rectilinear.

14.5.6 *Determination*

14.5.6.1 *Preparation of the test solution.* To the test portion in the 100 ml one-mark volumetric flask (**14.5.1**) add 2 ml of the hydrochloric acid solution (**14.3.1**), dilute to the mark and mix.

14.5.6.2 *Blank test.* Measure 10 ml of the solution C (**3.4.4**) into a 100 ml one-mark volumetric flask and treat in exactly the same say as specified in **14.5.6.1**.

14.5.6.3 *Spectrophotometric measurements.* Following the procedure specified in **14.5.3** and **14.5.4**, determine, in order, the absorbances for the following solutions:

a) the five standard matching solutions (**14.5.2**);

b) the test solution (**14.5.6.1**);

c) the blank test solution (**14.5.6.2**);

d) the standard matching solution, the absorbance of which is closest to that of the test solution.

14.6 Expression of results. The zinc content, expressed as milligrams of zinc (Zn) per kilogram, is given by the following formula:

$$m_1 \times \frac{1}{1000} \times \frac{A_1 - A_0}{A_2 - A_3} \times \frac{1000}{10} \times \frac{1000}{m_0}$$

$$= \frac{100\, m_1\, (A_1 - A_0)}{m_0\, (A_2 - A_3)}$$

where

A_0 is the absorbance for the blank test solution (**14.5.6.2**)

A_1 is the absorbance for the test solution (**14.5.6.1**)

A_2 is the mean of the absorbances for the standard matching solutions closest to that of the test solution, measured before and after the test solution

A_3 is the absorbance of the blank standard matching solution (**14.5.2**)

m_0 is the mass of the test portion used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of zinc contained in the standard matching solution closest to the test solution (in µg)

15 Determination of copper content

15.1 Field of application. This method is applicable to products having copper contents, expressed as Cu, in the range 0.5 mg/kg to 6 mg/kg.

15.2 Principle. The copper present is reduced with ascorbic acid and a violet coloured complex is formed by addition of 2,2′-biquinolyl. This complex is extracted with amyl alcohol and its colour measured spectrophotometrically.

15.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

15.3.1 *Sodium sulphate,* anhydrous.

15.3.2 *Hydrochloric acid solution,* ρ approximately 1.18 g/ml, approximately 36 % *m*/*m* solution or approximately 11N.

15.3.3 *Amyl alcohol*

15.3.4 *(+)-Tartaric acid,* 500 g/l solution.

15.3.5 *Sodium hydroxide,* 200 g/l solution.

15.3.6 *L-Ascorbic acid,* 100 g/l solution, freshly prepared.

15.3.7 *2,2′ Biquinolyl,* 0.5 g/l solution. Dissolve 0.25 g of 2,2′ biquinolyl in the amyl alcohol (**15.3.3**) and dilute with more of the amyl alcohol to 500 ml.

15.3.8 *Bromine water,* saturated solution.

15.3.9 *Copper,* standard solution corresponding to 0.1 g of copper per litre. Dissolve 0.3928 g of copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$) in water in a 1 000 ml one-mark volumetric flask, add 25 ml of approximately 6N sulphuric acid solution, dilute to the mark and mix.

1 ml of this standard solution contains 0.100 mg of Cu.

15.3.10 *Copper,* standard solution corresponding to 0.01 g of copper per litre. Dilute 10.0 ml of the standard copper solution (**15.3.9**) to the mark in a 100 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 10 µg of Cu.

15.3.11 *Narrow range indicator papers,* covering the range pH 5.5 to 7.0, as specified in BS 1647.

15.4 Apparatus. Ordinary laboratory apparatus and the following are required.

15.4.1 *Spectrophotometer,* or

15.4.2 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of about 545 nm.

15.4.3 *Optical cells,* 4 cm optical path length.

15.5 Procedure

15.5.1 *Test portion.* Measure into a 400 ml beaker 80.0 ml of the solution B (**3.4.3**) prepared from the liquid product or 160 ml of the solution B (**3.4.3**) prepared from the solid product.

15.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **15.5.4.2** but using a volume of the solution C (**3.4.4**) identical to the volume of the solution B (**3.4.3**) specified in **15.5.1**.

15.5.3 ***Preparation of the calibration graph***

15.5.3.1 *Preparation of standard matching solutions.* Into each of a series of six 500 ml stoppered separating funnels, introduce the volumes of the standard copper solution (**15.3.10**) shown in the following table:

Standard copper solution (15.3.10)	**Corresponding mass of copper (Cu)**
ml	µg
0[a]	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

[a] Compensation solution.

15.5.3.2 *Colour development.* Treat the contents of each funnel (**15.5.3.1**) as follows.

Dilute with water to approximately 400 ml, then add 2 ml of the tartaric acid solution (**15.3.4**). Adjust the pH of the solution to about 6.0 by addition of the sodium hydroxide solution (**15.3.5**) using the narrow range indicator paper (**15.3.11**) externally. Add 2 ml of the ascorbic acid solution (**15.3.6**), shake so as to mix thoroughly and allow to stand for 5 min. Add 10 ml of the 2,2′-biquinolyl solution (**15.3.7**) and shake well for about 2 min. Extract the copper complex with two 20 ml portions of the amyl alcohol (**15.3.3**), and transfer the extracts to a 100 ml beaker. Add about 2 g of the anhydrous sodium sulphate (**15.3.1**) to the combined extracts and stir thoroughly to remove traces of water. Filter the dried extract into a 50 ml one-mark volumetric flask, wash the residual sodium sulphate twice with 2 ml portions of the amyl alcohol (**15.3.3**). Transfer the washings to the flask, dilute to the mark with the amyl alcohol (**15.3.3**) and mix.

15.5.3.3 *Photometric measurements.* Carry out the photometric measurements either with the spectrophotometer (**15.4.1**) at the wavelength of maximum absorption (about 545 nm), or with the photoelectric absorptiometer (**15.4.2**), fitted with suitable filters, after having adjusted the instrument to zero absorbance against the amyl alcohol (**15.3.3**).

15.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**15.5.3.1**) from those of the standard matching solutions (**15.5.3.1**). Plot a graph having, for example, the copper content, expressed in micrograms per 50 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

15.5.4 ***Determination***

15.5.4.1 *Preparation of the test solution.* To the test portion in the beaker (**15.5.1**), add 5 ml of the hydrochloric acid solution (**15.3.2**) and 10 ml of the bromine water (**15.3.8**). Boil the solution until free from bromine and allow to cool. Transfer the contents of the beaker quantitatively to a 500 ml separating funnel fitted with a stopper.

15.5.4.2 *Colour development.* Treat the test solution in the separating funnel (**15.5.4.1**) as specified in **15.5.3.2**.

15.5.4.3 *Photometric measurement.* Carry out the photometric measurement on the test solution (**15.5.4.2**) and on the blank test solution (**15.5.2**) following the procedure described in **15.5.3.3**, after having adjusted the instrument to zero absorbance against the amyl alcohol (**15.3.3**).

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution B (**3.4.3**) as the test portion (**15.5.1**) and modifying the calculation accordingly.

15.6 Expression of results. By means of the calibration graph (**15.5.3.4**), determine the quantity of copper (Cu) corresponding to the value of the photometric measurement.

The copper content, expressed as milligrams of copper (Cu) per kilogram, is given by the following formula:

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_0 \times V} \times 1000 = \frac{(m_1 - m_2)}{m_0 \times V} \times 1000$$

where

m_0 is the mass of sample used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of copper in the test solution (in µg)

m_2 is the mass of copper in the blank test solution (in µg)

V is the volume of the solution B used in the determination (in ml)

16 Determination of lead content

16.1 Field of application. This method is applicable to products having lead contents, expressed as Pb, in the range 0.5 mg/kg to 7.5 mg/kg.

16.2 Principle. Lead, as the dipyrrolidine dithiocarbamate complex, is extracted from a neutralized solution of the sample at pH 4 with 4-methylpentan-2-one. Lead standard solutions are prepared by extracting aqueous lead solutions in the same way.

The 4-methylpentan-2-one extracts are then analysed by atomic absorption spectrophotometry using an air/acetylene flame and lead lamp at a wavelength of about 283.3 nm.

16.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

16.3.1 *Ammonium dipyrrolidine dithiocarbamate,* 10 g/l solution in water. Dissolve 1 g in 100 ml of water. Adjust to pH 5 with 5N hydrochloric acid solution or dilute ammonia solution as necessary. Extract twice with 5 ml portions of 4-methylpentan-2-one, rejecting the upper solvent layers.

16.3.2 *Potassium chloride,* 66 g/l solution.

16.3.3 *4-methylpentan-2-one (methyl isobutyl ketone),* water washed.

16.3.4 *Hydrochloric acid,* approximately 5N solution.

16.3.5 *Sodium hydroxide,* approximately 1N solution.

16.3.6 *Lead,* standard solution corresponding to 1 g/l of lead. Dissolve 1.598 g of lead nitrate, $Pb(NO_3)_2$, in water, add 5 ml of nitric acid, approximately 16N solution, and dilute to the mark in a 1 000 ml one-mark volumetric flask. Mix.

1 ml of this standard solution contains 1.000 mg of Pb.

16.3.7 *Lead,* standard solution corresponding to 0.01 g of lead per litre. Pipette 10.0 ml of the standard lead solution (**16.3.6**) into a 1 000 ml one-mark volumetric flask, add 5 ml of nitric acid, approximately 16N solution and dilute to the mark. Mix. Prepare freshly as required.

1 ml of this standard solution contains 10 µg of Pb.

16.3.8 *Lead,* standard solution corresponding to 0.001 g/l of lead. Pipette 100 ml of the standard lead solution (**16.3.7**) into a 1 000 ml one-mark volumetric flask. Add 5 ml of nitric acid, approximately 16N solution, and dilute to the mark. Prepare freshly as required.

1 ml of this standard solution contains 1 µg of Pb.

16.4 Apparatus. Ordinary laboratory apparatus and the following are required.

16.4.1 *Atomic absorption spectrophotometer,* with air/acetylene burner and appropriately regulated air and acetylene supplies.

16.4.2 *Lead hollow cathode lamp*

16.4.3 *pH meter*

16.5 Procedure

16.5.1 *Test portion.* Transfer 10.0 ml of the solution B (**3.4.3**) into a 250 ml beaker.

16.5.2 *Preparation of standard matching solutions.* Into each of a series of four 250 ml beakers measure accurately, by means of a 10 ml burette, the volumes of the standard lead solution (**16.3.8**) shown in the following table.

Standard lead solution (16.3.8)	Corresponding mass of lead (Pb)
ml	µg
0[a]	0
5.0	5.0
10.0	10.0
15.0	15.0

[a] Blank standard matching solution.

Treat the contents of each beaker as follows.

Add 50 ml of water and 20 ml of the potassium chloride solution (**16.3.2**). Using the pH meter (**16.4.3**), adjust the pH to 4.0 by adding the sodium hydroxide solution (**16.3.5**) and then add 5 ml of the ammonium dipyrrolidine dithiocarbamate solution (**16.3.1**). Dilute to 100 ml with water. Readjust the pH to 4.0 and transfer to a 250 ml separating funnel. Extract twice with 5 ml portions of the 4-methylpentan-2-one (**16.3.3**), combining the extracts into a dry 10 ml one-mark volumetric flask and readjusting the pH of the solution between the extractions if necessary. Dilute the contents of the flask to the mark with the 4-methylpentan-2-one (**16.3.3**) and mix.

16.5.3 *Adjustment of the apparatus.* Fit the lead hollow cathode lamp (**16.4.2**) to the spectrophotometer (**16.4.1**) and switch on the current a sufficient time in advance to ensure stabilization. Adjust the instrument to give maximum absorbance at a wavelength of about 283.3 nm. Set the lamp input current, slit aperture, air and acetylene pressures and the instrument sensitivity according to the recommended values.
Aspirate 4-methylpentan-2-one and reset the fuel supply. Check that the flame does not go out when the solution is withdrawn.

16.5.4 *Spectrophotometric measurements.* Aspirate the series of standard matching solutions (**16.5.2**) into the flame and measure the absorbance for each. Spray water saturated with the 4-methylpentan-2-one through the burner after each measurement. Take care to keep the aspiration rate constant throughout this series of measurements.

16.5.5 *Preparation of the calibration graph.* Plot a graph having, for example, the number of micrograms of lead contained in 10 ml of the standard matching solutions as abscissae and the corresponding values of the measured absorbances, less the measured value for the blank standard matching solution, as ordinates.

NOTE It is essential that the calibration graph obtained is rectilinear.

16.5.6 *Determination*

16.5.6.1 *Preparation of the test solution.* To the test portion in the 250 ml beaker (**16.5.1**), add 50 ml of water and, using the pH meter (**16.4.3**), adjust the pH to 4.0 by adding the sodium hydroxide solution (**16.3.5**) and then add 5 ml of the ammonium dipyrrolidine dithiocarbamate solution (**16.3.1**). Dilute to 100 ml with water. Readjust the pH to 4.0 and transfer to a 250 ml separating funnel. Extract twice with 5 ml portions of the 4-methylpentan-2-one (**16.3.3**) combining the extracts into a dry 10 ml one-mark volumetric flask and readjusting the pH of the solution between the extractions if necessary.

Dilute the contents of the flask to the mark with the 4-methylpentan-2-one (**16.3.3**) and mix.

16.5.6.2 *Blank test.* Measure 10.0 ml of the solution C (**3.4.4**) into a 250 ml beaker and treat in exactly the same way as specified in **16.5.6.1**.

16.5.6.3 *Spectrophotometric measurements.* Following the procedure specified in **16.5.3** and **16.5.4**, determine, in order, the absorbances of the following solutions:

a) the four standard matching solutions (**16.5.2**);

b) the test solution (**16.5.6.1**);

c) the blank test solution (**16.5.6.2**);

d) the standard matching solution, the absorbance of which is closest to that of the test solution.

16.6 Expression of results. The lead content, expressed as milligrams of lead (Pb) per kilogram, is given by the following formula:

$$m_1 \times \frac{1}{1000} \times \frac{A_1 - A_0}{A_2 - A_3} \times \frac{1000}{10} \times \frac{1000}{m_0} = \frac{100\, m_1\, (A_1 - A_0)}{m_0\, (A_2 - A_3)}$$

where

A_0 is the absorbance for the blank test solution (**16.5.6.2**)

A_1 is the absorbance for the test solution (**16.5.6.1**)

A_2 is the mean of the absorbances for the standard matching solutions closest to that of the test solution, measured before and after the test solution

A_3 is the absorbance of the blank standard matching solution (**16.5.2**)

m_0 is the mass of the test portion used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of lead contained in the standard matching solution closest to the test solution (in µg)

17 Determination of nickel content

17.1 Field of application. This method is applicable to products having nickel contents, expressed as Ni, in the range 0.5 mg/kg to 6 mg/kg for liquid products and 1.0 mg/kg to 12 mg/kg for solid products.

17.2 Principle. Nickel is determined photometrically at a wavelength of about 480 nm as the red-orange complex formed with furil α-dioxime in ammoniacal solution. Interference from ferric iron is suppressed by the use of tartaric acid added after oxidizing any ferrous iron present to the ferric state with concentrated nitric acid.

17.3 Reagents. The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

17.3.1 *Potassium chloride,* 200 g/l solution.

17.3.2 *Nitric acid,* solution, approximately 1.42 g/l, approximately 70 % (*m*/*m*) solution or approximately 16N solution.

17.3.3 *Hydrochloric acid,* solution, approximately 1.18 g/ml, approximately 36 % (*m*/*m*) solution or approximately 11N solution.

17.3.4 *(+)-Tartaric acid,* 200 g/l solution.

17.3.5 *Ammonia,* approximately 5N solution.

17.3.6 *Furil α-dioxime,* 30 g/l solution in ethanol[1)].

17.3.7 *Nickel,* standard solution corresponding to 0.5 g/l of nickel. Dissolve 3.364 g of ammonium nickel sulphate hexahydrate, $(NH_4)_2SO_4.NiSO_4.6H_2O$, in water in a 1 000 ml one-mark volumetric flask and dilute to the mark with water. Mix.

1 ml of this standard solution contains 0.500 mg of Ni.

17.3.8 *Nickel,* standard solution corresponding to 0.01 g of nickel per litre. Dilute 10.0 ml of the standard nickel solution (**17.3.7**) to the mark in a 500 ml one-mark volumetric flask and mix. Prepare freshly before use.

1 ml of this standard solution contains 10 µg of Ni.

17.3.9 *Litmus paper,* blue or neutral.

17.4 Apparatus. Ordinary laboratory apparatus and the following are required.

17.4.1 *Spectrophotometer,* or

17.4.2 *Photoelectric absorptiometer* fitted with filters providing maximum transmission at a wavelength of about 480 nm.

17.4.3 *Optical cells,* 4 cm optical path length

17.5 Procedure

17.5.1 *Test portion.* Measure into a 250 ml beaker 80 ml of the solution B (**3.4.3**).

17.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **17.5.4.1** but using 80 ml of the solution C (**3.4.4**), instead of the test portion (**17.5.1**), in a 250 ml beaker.

[1)] Ethanol may be replaced for this purpose by industrial methylated spirits, 66 degrees O.P., complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230). It is not permissible to use duty-free ethanol, received under the provisions of the Customs and Excise Act 1952, Section 111, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

17.5.3 ***Preparation of the calibration graph***

17.5.3.1 *Preparation of the standard matching solutions.* Into each of a series of six 100 ml beakers, each containing 55 ml of the potassium chloride solution (**17.3.1**), introduce, by means of a 10 ml burette, the volumes of the standard nickel solution (**17.3.8**) shown in the following table.

Standard nickel solution (17.3.8)	Corresponding mass of nickel (Ni)
ml	µg
0[a]	0
1.0	10
2.0	20
3.0	30
4.0	40
5.0	50

[a] Compensation solution.

17.5.3.2 *Colour development.* Treat the contents of each beaker (**17.5.3.1**) as follows.

Add two drops of the nitric acid solution (**17.3.2**), boil, allow to cool and add 1 ml of the tartaric acid solution (**17.3.4**). Make the solution just alkaline by addition of the ammonia solution (**17.3.5**). Transfer quantitatively to a 100 ml one-mark volumetric flask. Add 0.25 ml of the furil α-dioxime solution (**17.3.6**) by means of a pipette. Dilute to the mark, mix and allow to stand for 5 min.

17.5.3.3 *Photometric measurements.* Carry out the photometric measurements using either the spectrophotometer (**17.4.1**), at the wavelength of maximum absorption (about 480 nm), or with the photoelectric absorptiometer (**17.4.2**), fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

17.5.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**17.5.3.1**) from those of the standard matching solutions (**17.5.3.1**). Plot a graph, having for example, the nickel (Ni) content, expressed in micrograms per 100 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

17.5.4 ***Determination***

17.5.4.1 *Colour development.* Treat the test portion in the 250 ml beaker (**17.5.1**) following the procedure described in **17.5.3.2**.

17.5.4.2 *Photometric measurement.* Carry out the photometric measurement on the test solution (**17.5.4.1**) and on the blank test solution (**17.5.2**) following the procedure specified in **17.5.3.3**, after having adjusted the instrument to zero absorbance against water.

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller volume of the solution B (**3.4.3**) as the test portion (**17.5.1**) and modifying the calculation accordingly.

17.6 Expression of results. By means of the calibration graph (**17.5.3.4**), determine the quantity of nickel (Ni) corresponding to the value of the photometric measurement.

The nickel content, expressed as milligrams of nickel (Ni) per kilogram is given by the following formula:

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_0 \times 80} \times 1000 = \frac{m_1 - m_2}{m_0} \times 12.5$$

where

m_0 is the mass of sample used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of nickel found in the test solution (in µg)

m_2 is the mass of nickel found in the blank test solution (in µg)

18 Determination of arsenic content

18.1 Field of application. This method is applicable to products having arsenic contents, expressed as As, in the range 0.2 mg/kg to 2 mg/kg.

18.2 Principle. Inorganic arsenic is reduced to arsine (AsH_3) by zinc in acid solution. The arsine is passed through a guard tube containing lead acetate wool and then into a bubbler tube containing silver diethyldithiocarbamate dissolved in pyridine. The arsine reacts with the silver salt forming a soluble red complex which is determined spectrophotometrically at a wavelength of about 540 nm.

18.3 Reagent. The reagent used shall be of a recognized analytical quality.

18.3.1 *Sulphuric acid solution, ρ* approximately 1.88 g/ml approximately 98 % *m/m* solution or approximately 36N solution.

18.3.2 *Other reagents.* The reagents specified in BS 4404 are required.

18.4 Apparatus. The apparatus specified in BS 4404 is required.

18.5 Procedure

18.5.1 *Preparation of test solution and blank test solution.* Into a 100 ml beaker measure 50 ml of the solution B (**3.4.3**), for solid products, or 25 ml of solution B (**3.4.3**), for the liquid products. Into a second 100 ml beaker measure either 50 ml of the solution C (**3.4.4**), for solid products, or 25 ml of the solution C (**3.4.4**) for the liquid products. Cautiously add, from a 10 ml burette, 3.5 ml of the sulphuric acid solution (**18.3.1**) to each beaker and mix.

NOTE The above paragraph corresponds to **6.2.1** and **6.2.3** of BS 4404:1968.

18.5.2 *Determination.* Determine the arsenic content by the method specified in BS 4404.

18.6 Expression of results. The arsenic content, expressed as milligrams of arsenic (As) per kilogram, is given by the following formula:

$$\frac{m_1}{1000} \times \frac{1000}{m_0\ V} \times 1000 = \frac{1000\ m_1}{m_0\ V}$$

where

m_0 is the mass of sample used in the preparation of the solution B (**3.4.3**) (in g)

m_1 is the mass of arsenic in the test solution (in μg)

V is the volume of the solution B taken (in ml)

blank

Publications referred to

BS 846, *Burettes and bulb burettes.*

BS 1647, *pH scale.*

BS 1792, *One-mark volumetric flasks.*

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

BS 4123, *Schedule of preferred chemical indicators.*

BS 4404, *Method for the determination of arsenic (silver diethyldithiocarbamate procedure).*

BS 5633, *Specification for potassium hydroxide for use in alkaline cells*[2)].

ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

[2)] Referred to in the foreword only.

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