BRITISH STANDARD

BS 3903:1977
Incorporating Amendment No. 1

Methods of test for

Sulphuric acid, oleum and liquid sulphur trioxide

UDC 661.25:546.226 – 325:543

Cooperating organizations

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

Association of Fatty Acid Distillers
Chemical Industries Association*
Chemical Society — Analytical Division
Department of Health and Social Security
Department of Industry
Department of Industry — Laboratory of the Government Chemist
Fertilizer Manufacturers' Association Ltd
Ministry of Agriculture, Fisheries and Food
National Sulphuric Acid Association*
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association*
Society for Analytical Chemistry

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:

British Textile Employers' Association

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Amendments issued since publication

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2529	April 1978	Indicated by a sideline in the margin

Contents

Foreword

This British Standard has been prepared under the authority of the Chemical Standards Committee and includes methods of test for sulphuric acid, oleums and liquid sulphur trioxide of commercial quality and intended for general industrial purposes.

The standard, first published in 1965, has been revised in the light of current requirements. Where possible, methods adopted by the International Organization for Standardization (ISO) have been used and the situation in this respect is summarized as tabulated opposite.

This revision also differs from the 1965 publication in the following respects.

a) Methods for the determination of density and temperature rise are omitted following the decision that only the titrimetric method for the determination of sulphuric acid content should be used for referee purposes.

b) The methods for the determination of copper and aluminium contents, which are seldom used, are omitted.

c) In accordance with current practice, alternative methods for the determination of iron and lead contents by atomic absorption spectrophotometry are included. The original methods in the 1965 publication are, however, retained and have undergone no significant change in the course of the revision.

WARNING. Concentrated sulphuric acid, oleum and sulphur trioxide are extremely corrosive and poisonous and should be used with all necessary precautions. Protective clothing, including goggles and gloves, should be worn at all times when handling these materials.

Determination	Relationship to ISO method of test
Residue on heating	Heating temperature is identical with that used in ISO/R 913
Sulphur dioxide content	Conforms to the method specified in ISO 3423 amended to incorporate comments made by the United Kingdom during its development
Arsenic content	Conforms to the method specified in ISO 2590
Ammoniacal nitrogen content	Conforms to the method specified in ISO 2899
Nitrogen oxides content	Conforms to the method specified in ISO 2363
Chloride content	Conforms to the method specified in ISO 2877
Iron content (spectrophotometric method)	Is similar in principle to the method specified in ISO/R 915
Lead content (spectrophotometric method)	Conforms to the method specified in ISO 2717

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 and ii, pages 1 to 24, 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.

Section 1. General

1 Scope

This British Standard specifies methods of test for commercial sulphuric acid, oleum and liquid sulpur trioxide. Section 2 covers tests for acids containing up to 100 % of H_2 SO_4. Section 3 describes methods of test for oleums and liquid sulphur trioxide and includes a method for their dilution prior to analysis by the methods specified in section 2, clauses **5** to **9**.

2 References

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

Section 2. Methods of test for acids containing up to 100 % of H_2SO_4

3 Determination of sulphuric acid content

NOTE This method differs from that specified in ISO/R 910 which was disapproved for technical reasons by the United Kingdom.

3.1 Principle. A weighed quantity of the sample is diluted with water and titrated directly with standard volumetric sodium hydroxide solution using screened methyl orange as indicator.

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

3.2.1 *Sodium hydroxide*, N standard volumetric solution.

3.2.2 *Screened methyl orange indicator solution*, 1 g/l, prepared as described in BS 4123.

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

3.3.1 *Lunge-Rey pipette*, complying with the requirements of BS 2058.

3.3.2 *Bulb burette*, 105 ml nominal capacity, complying with the requirements of BS 846, having a bulb capacity of 80 ml and a scale range from 80 ml to 105 ml with 0.05 ml subdivisions.

3.4 Procedure. By means of the Lunge-Rey pipette (**3.3.1**), transfer a quantity of sample equivalent to 4.5 g to 5 g of 100 % sulphuric acid, weighed to the nearest 1 mg, to a 400 ml conical beaker already containing 50 ml of water. Add a few drops of the screened methyl orange indicator solution (**3.2.2**) and titrate with the N sodium hydroxide solution (**3.2.1**), contained in the bulb burette (**3.3.2**), until the first appearance of a green tint.

3.5 Expression of results. Sulphuric acid content, expressed as a percentage by mass of H_2SO_4, is given by the formula:

$$\frac{4.904 \times V}{M}$$

where

V is the volume of N sodium hydroxide solution used (in ml)

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

4 Determination of residue on heating

NOTE This method uses the heating temperature specified in ISO/R 913.

4.1 Field of application. The method is applicable to samples in which the residue on heating is equal to or greater than 5 mg/kg.

4.2 Principle. The sample is evaporated and the residue is heated at 800 ± 50 °C.

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

4.3.1 *Platinum basin*, flat bottom, capacity about 100 ml.

4.3.2 *Muffle furnace*, capable of being controlled at 800 ± 50 °C.

4.3.3 ***Desiccator***

4.4 Procedure. Heat the platinum basin (**4.3.1**) in the muffle furnace (**4.3.2**) controlled at 800 ± 50 °C. Cool in the desiccator (**4.3.3**) and weigh to the nearest 0,1 mg. Transfer to the basin approximately 50 g of sample weighed to the nearest 0.01 g.

Evaporate the acid carefully on a sand bath inside a fume cupboard and heat the basin containing the residue to dryness. Transfer the basin containing the residue to the muffle furnace controlled at 800 ± 50 °C and heat for about 15 min. Remove the basin from the furnace, place in a desiccator, cool and weigh to the nearest 0.1 mg. Repeat the operations of heating, cooling and weighing until the difference between successive weighings does not exceed 0.5 mg.

4.5 Expression of results. The residue on heating, expressed as milligrams per kilogram, is given by the formula:

$$\frac{M_1 \times 10^6}{M_2}$$

where

M_1 is the mass of the residue (in g)

M_2 is the mass of the test portion (in g)

5 Determination of sulphur dioxide content

NOTE This method conforms to that specified in ISO 3423 as amended to incorporate the comments made by the United Kingdom during its development.

5.1 Field of application. The method is applicable to products with sulphur dioxide content equal to or greater than 2 mg/kg.

5.2 Principle. Sulphur dioxide is removed from the sample by means of a current of nitrogen and absorbed into a known volume of iodine solution. The excess iodine is titrated against standard volumetric sodium thiosulphate solution.

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

5.3.1 *Hydrazinium sulphate*

5.3.2 *Iodine,* approximately 0.1N, 0.05N or 0.01N solution, as required (see **5.5.3**). Prepare this solution at the time of use.

5.3.3 *Sodium thiosulphate*, 0.1N, 0.05N or 0.01N standard volumetric solution, as required (see **5.5.4**). Prepare this solution at the time of use.

5.3.4 *Starch*, 5 g/l solution.

5.3.5 *Nitrogen*, pure, containing less than 0.001 % of oxygen.

It is recommended that the reduction valve from the nitrogen cylinder be connected to a gas-washing bottle containing a 150 g/l solution of titanium (III) chloride.

5.3.6 *Crushed ice*, prepared from water complying with the requirements of BS 3978.

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

5.4.1 *Glass apparatus*, with ground glass joints, as shown in Figure 1, comprising the following:

a) 3-necked flask (A), 500 ml or 250 ml capacity;

b) cylindrical dropping funnel (B) fitting into one of the two side-necks of the flask;

c) tube, fitted with a stopcock (C), fitting into the central neck of the flask and terminating in a fritted disc at a level of about 10 mm above the bottom of the flask;

d) three gas-washing bottles (D_1), (D_2), (D_3), Dreschel type, 100 ml or 125 ml capacity, with the inlet tubes terminating in fritted discs complying with the requirements of BS 1752 (grade no. 4, pore diameter 5 µm to 15 µm) to disperse the gas.

NOTE The corresponding ISO method uses fritted discs of grade P16, pore diameter 4 µm to 16 µm.

5.4.2 *Glass flask*, with a ground glass stopper.

5.4.3 *Flowmeter or bubble-counter*, for use in the range 5 l/h to 50 l/h.

5.5 Procedure

5.5.1 *Test portion.* Fill the flask (**5.4.2**) with the test sample and take, weighing by difference to the nearest 0.05 g, a test portion of approximately 200 g.

If the density of the sample exceeds 1.70 g/ml, slowly pour the test portion, cooling so that the temperature remains below 10 °C, onto sufficient of the crushed ice (**5.3.6**) to give a solution of density approximately 1.70 g/ml.

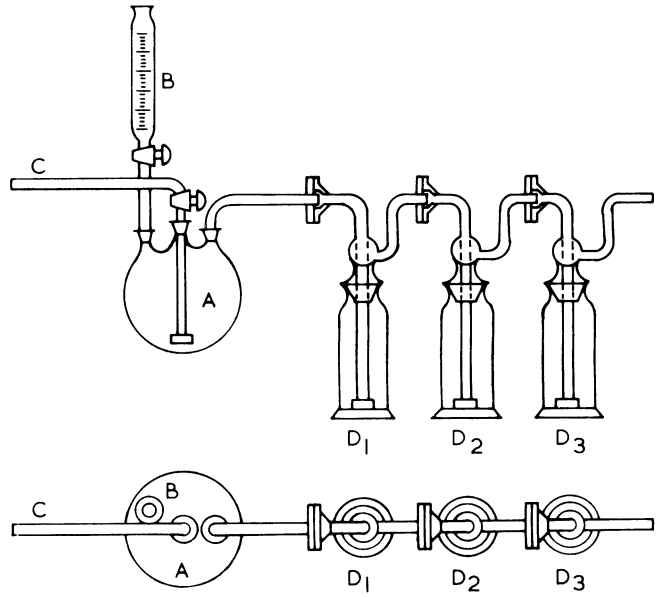

Figure 1 — Apparatus for determination of sulphur dioxide content

5.5.2 *Blank test.* At the same time as the determination, carry out a blank test following the same procedure and using the same quantities of all reagents as used in the determination.

5.5.3 *Preparation of the apparatus.* Place 50 ml of the appropriate iodine solution (**5.3.2**) in the first gas-washing bottle (D_1). Divide 25 ml of the corresponding standard volumetric sodium thiosulphate solution (**5.3.3**) between the second and third gas-washing bottles (D_2, D_3), if necessary adding a little water to ensure sufficient scrubbing of the gas.

Expected sulphur dioxide content	**Strength of iodine solution**
mg/kg	N (approximate)
2 to 30	0.01
30 to 150	0.05
> 150	0.1

NOTE The two bottles (D_2) and (D_3) are intended to retain any entrained iodine.

Place the bottle (D_1) in a cooling bath and ensure that during all subsequent operations the temperature of its contents does not rise above 10 °C and that the bottle is not exposed to a bright light.

If the sample contains nitrite or nitrate ions, place an excess of the hydrazinium sulphate solution (**5.3.1**) in the flask (A) in relation to the nitrite and nitrate contents in the test portion. Connect up the apparatus (**5.4.1**) and displace the air by means of a rapid current of the nitrogen (**5.3.5**).

5.5.4 *Determination.* Run the test portion (**5.5.1**) into the flask (A) through the dropping funnel (B) and pass a current of the nitrogen (**5.3.5**) at a rate, measured by the flowmeter or bubble-counter (**5.4.3**), of about 20 l/h for 3 h. At the end of this time there should still be an excess of iodine in the gas-washing bottle (D_1).

Disconnect the three gas-washing bottles (D_1), (D_2) and (D_3) and transfer their contents quantitatively into a beaker. Rinse the bottles with a few millilitres of water, collecting the washings in the beaker. Titrate the excess of iodine with the appropriate standard volumetric sodium thiosulphate solution (**5.3.3**) in the presence of the starch solution (**5.3.4**).

5.6 Expression of results. The sulphur dioxide content, expressed as milligrams of sulphur dioxide (SO_2) per kilogram, is given by the formula:

$$\frac{32 \times T \times 1000}{M}[(50.00 - 25.00 - V_1) - (50.00 - 25.00 - V_0)] = \frac{32\ 000 \times T(V_0 - V_1)}{M}$$

where

V_0 is the volume of the standard volumetric sodium thiosulphate solution (**5.3.3**) used for the blank test (in ml)

V_1 is the volume of the standard volumetric sodium thiosulphate solution (**5.3.3**) used for the determination (in ml)

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

T is the exact normality of the sodium thiosulphate solution used (**5.3.3**)

32 **5.6.1** is the mass of sulphur dioxide (SO_2) corresponding to 1 ml of exactly 1N standard volumetric sodium thiosulphate solution (in mg)

6 Determination of arsenic content

NOTE This method conforms to that specified in ISO 2590, which corresponds to BS 4404.

6.1 Field of application. The method is applicable to the determination of arsenic in the range 1 mg to 10 mg of arsenic per kilogram.

6.2 Principle. The arsenic in the sample is reduced with zinc, the arsine evolved is absorbed in a solution of silver diethyldithiocarbamate in pyridine and the absorbance of the complex is measured spectrophotometrically. A test solution is prepared and tested by the method described in BS 4404.

6.3 Reagents and apparatus. As described in BS 4404.

6.4 Procedure

6.4.1 *Preparation of test solution.* Transfer 7 ml of the concentrated hydrochloric acid [**5.2**(1) of BS 4404] into a 50 ml graduated cylinder and add 25 ml of water. Add from a Lunge-Rey pipette (see BS 2058) a volume of sample equivalent to 1 ± 0.1 g of 100 % sulphuric acid and dilute to 50 ml with water.

6.4.2 *Arsine evolution.* Transfer the contents of the graduated cylinder to the 100 ml conical flask [**4.1**(1) of BS 4404]. To the contents of the conical flask add the potassium iodide and stannous chloride solutions as described in **6.1.1** of BS 4404 and proceed as for that method.

6.4.3 *Blank test.* At the same time as the determination, carry out a blank test following the same procedure as **6.4.1** but add 1 g of the sulphuric acid [**5.2**(2) of BS 4404:1968] instead of the test portion.

6.5 Expression of results. Obtain the corrected arsenic content M_1 from the calibration graph (**6.1** of BS 4404:1968). The arsenic content of the sample, expressed as milligrams of arsenic per kilogram of sample, is given by the following formula:

$$\frac{M_1}{M_2}$$

where

M_1 is the mass of arsenic found in the sample (in μg)

M_2 is the mass of the test portion (in g)

7 Determination of ammoniacal nitrogen content

NOTE This method conforms to that specified in ISO 2899.

7.1 Field of application. The method is applicable to the determination of ammoniacal nitrogen content in the range 1 mg to 5 mg of nitrogen per kilogram.

7.2 Principle. The ammonia present in the sample is distilled in the presence of an excess of sodium hydroxide and the distillate is collected in excess acid. The excess acid is neutralized and a coloured complex is formed by treatment with sodium hydroxide, phenol and sodium hypochlorite in the presence of acetone. The indophenol obtained is determined spectrophotometrically at a wavelength of approximately 630 nm.

7.3 Reagents. The reagents used shall be of a recognized analytical quality. Water complying with the requirements of BS 3978, and of which the ammoniacal nitrogen content is negligible, shall be used throughout.

7.3.1 *Ammonia-free ice*, prepared from water (**7.3**).

7.3.2 *Sodium hydroxide*, 350 g/l solution, previously boiled for 20 min to remove traces of ammoniacal nitrogen. Make up to original volume.

7.3.3 *Sulphuric acid*, 0.1N standard volumetric solution.

7.3.4 *Sodium hydroxide*, N standard volumetric solution.

7.3.5 *Sodium hydroxide*, 0.1N standard volumetric solution.

7.3.6 *Acetone*

7.3.7 *Sodium phenoxide reagent solution*. Dissolve 62.5 g of phenol in 18.5 ml of the acetone (**7.3.6**), dilute to 100 ml with 95 % (*v*/*v*) ethanol[1)] and mix.

Store this solution in the dark, preferably in a refrigerator.

Dissolve 27 g of sodium hydroxide in 100 ml of water.

When required, prepare the reagent by mixing 20 ml of each solution and diluting to 100 ml with water.

7.3.8 *Sodium hypochlorite reagent solution*, 10 g/l available chlorine. Dilute with water a stock sodium hypochlorite solution containing preferably 100 g/l to 140 g/l of available chlorine (do not use a sodium hypochlorite solution containing less than 80 g/l of available chlorine). Standardize this solution weekly by the method specified in BS 4426.

Keep this solution in a cool place away from direct sunlight. The solution remains usable for about four weeks.

7.3.9 *Ammonium chloride stock solution*, corresponding to 1 g of ammoniacal nitrogen per litre. Dissolve in a little water 3.819 g of ammonium chloride, previously dried at 100 °C and cooled in a desiccator, transfer to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

1 ml of this solution contains 1 mg of ammoniacal nitrogen. Replace the solution at least once a month.

[1)] Ethanol may be replaced for this purpose by industrial methylated spirits, 66 degrees O.P., complying with the requirements of 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.

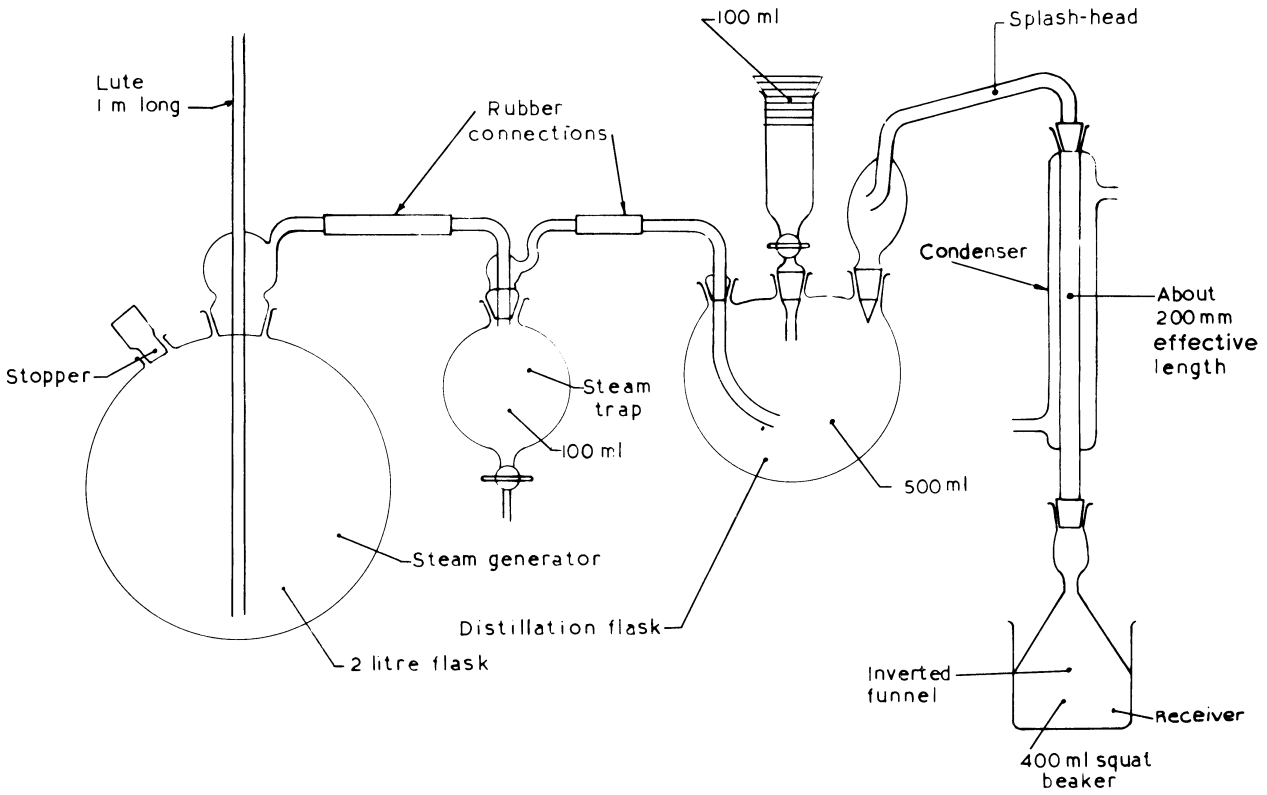

Figure 2 — Apparatus for determination of ammoniacal nitrogen content

7.3.10 *Ammonium chloride solution*, corresponding to 0.1 g of ammoniacal nitrogen per litre. Transfer 50 ml of the ammonium chloride stock solution (**7.3.9**) to a 500 ml one-mark volumetric flask and dilute to the mark with water.

1 ml of this solution contains 0.1 mg of ammoniacal nitrogen. Replace the solution at least once every 15 days.

7.3.11 *Ammonium chloride solution*, corresponding to 0.001 g of ammoniacal nitrogen per litre. Transfer 10.0 ml of the ammonium chloride solution (**7.3.10**) to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

1 ml of this solution contains 1 µg of ammoniacal nitrogen. Prepare this solution immediately before it is required for use.

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

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

7.4.1 *Distillation apparatus*, consisting of the following items (see Figure 2 for an example of a typical apparatus).

7.4.1.1 *Steam generator*, 2 litre capacity, round-bottom glass flask fitted with a lute 1 m long and an outlet.

7.4.1.2 *Steam trap*, consisting of a dropping funnel of 100 ml capacity.

7.4.1.3 *Distillation flask,* 500 ml capacity, fitted with a tube to allow steam to flow in from the steam generator and a dropping funnel of 100 ml, fitted with a PTFE tap and an airtight stopper, into which is placed the test portion.

7.4.1.4 *Straight condenser,* about 200 mm effective length, fitted with a tapered extension dipping into the receiver (**7.4.1.5**).

7.4.1.5 *Receiver,* 400 ml capacity beaker and inverted glass funnel.

7.4.2 *pH meter,* with glass electrode, complying with the requirements of BS 3145 and BS 2586.

7.4.3 *Spectrophotometer,* or

7.4.4 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of approximately 620 nm.

7.4.5 *Optical cells,* 1 cm.

7.4.6 *Weighing pipette*

7.4.7 *One-mark volumetric flasks,* 50 ml capacity, complying with the requirements of BS 1792.

7.5 Procedure

7.5.1 *Preparation of colour standards.* Into a series of six of the 50 ml one-mark volumetric flasks (**7.4.7**), transfer the amounts of the standard ammonium chloride solution (**7.3.11**) indicated below.

Standard ammonium chloride solution (7.3.11)	**Corresponding mass of ammoniacal nitrogen**
ml	µg
0	0
5	5
10	10
15	15
20	20
25	25

Treat each solution as follows.

Dilute, if necessary, to 25 ml with water, add 0.3 ml of the acetone (**7.3.6**) and mix. By means of rapid delivery pipettes add 10 ml of the sodium phenoxide solution (**7.3.7**) followed immediately by 5 ml of the sodium hypochlorite solution (**7.3.8**). Dilute to the mark with water and mix. Allow to stand for 60 ± 5 min at ambient temperature away from direct sunlight. Carry out the additions rapidly, one flask after the other, staggering the tests in order to allow each solution to stand for a similar time.

Measure the absorbance of each solution at the wavelength of maximum absorption (approximately 620 nm[2)]) against water in the reference cell; correct for the blank and prepare a calibration graph.

7.5.2 *Determination.* Place 30 ml of the 0.1N standard volumetric sulphuric acid solution (**7.3.3**) in the receiver (**7.4.1.5**).

Fill the weighing pipette (**7.4.6**) with some of the test sample and take, weighing by difference to the nearest 10 mg, a test portion of about 50 g.

Transfer the test portion, via the dropping funnel (see **7.4.1.3**), onto a little of the crushed ice (**7.3.1**) contained in the distillation flask (**7.4.1.3**). Rinse the walls of the funnel with water into the flask. The final volume of solution should be about 100 ml.

Add several drops of the phenolphthalein solution (**7.3.12**) and connect the flask to the distillation apparatus (**7.4.1**).

Neutralize the test solution, stirring and cooling, with the sodium hydroxide solution (**7.3.2**), added through the dropping funnel.

Add a further 50 ml of the sodium hydroxide solution (**7.3.2**) and the quantity of water necessary to attain a volume of about 300 ml. During these additions ensure that several drops of solution remain above the stopcock as a seal.

Replace the stopper in the funnel. Warm the flask to the commencement of boiling and distil in a current of steam, controlled to obtain a drop-by-drop rate of delivery into the receiver, until a volume of about 150 ml has collected. Thoroughly wash the end of the condenser, collecting the wash water in the receiver. Adjust the pH of the solution, using the pH meter (**7.4.2**), to between 6 and 7, by adding, first, the N sodium hydroxide solution (**7.3.4**), and then the 0.1N sodium hydroxide solution (**7.3.5**).

Quantitatively transfer the solution to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

[2)] For use with a photoelectric absorptiometer an Ilford 607 orange filter has been found suitable.

Transfer 25.0 ml of the test solution to one of the 50 ml one-mark volumetric flasks (**7.4.7**). Then, swirling throughout each addition, add 0.3 ml of the acetone (**7.3.6**) and, by means of rapid delivery pipettes, 10 ml of the sodium phenoxide solution (**7.3.7**) and 5 ml of the sodium hypochlorite solution (**7.3.8**).

Dilute to the mark, mix and allow to stand, shielded from light, for 60 ± 5 min at room temperature.

NOTE If the presence of copper is suspected, the addition of 1 ml of a 0.05M solution of *di*sodium ethylenediamine-*NNN′N′*-tetra-acetate dihydrate before the sodium phenoxide solution will prevent interference.

7.5.3 *Blank test.* At the same time as the determination, carry out a blank test following the same procedure and using the same quantities of ice and all reagents used in the determination.

7.5.4 *Spectrophotometric measurements.* Measure the absorbances of the solutions at the wavelength used for the calibration, using water in the reference cell. Read off the amount of ammonia present from the calibration graph (see **7.5.1**).

7.6 Expression of results. Ammoniacal nitrogen content, expressed as milligrams of nitrogen (N) per kilogram, is given by the formula:

$$\frac{M_1 - M_2}{M_3} \times 10$$

where

M_1 is the mass of ammoniacal nitrogen found in the test solution (in μg)

M_2 is the mass of ammoniacal nitrogen found in the blank solution (in μg)

M_3 is the mass of the test portion (in g)

8 Determination of nitrogen oxides content

NOTE This method conforms to that specified in ISO 2363.

8.1 Field of application. The method is applicable to the determination of contents of nitrogen oxides, expressed as nitrogen, equal to or greater than 0.2 mg/kg.

8.2 Principle. Nitrous nitrogen is oxidized to nitric nitrogen by potassium permanganate and reacted with 2,4-xylenol to form a nitrate derivative, which is distilled and absorbed in sodium hydroxide solution. The yellow coloured nitrophenol so formed is determined spectrophotometrically at a wavelength of approximately 445 nm.

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

8.3.1 *Mercury (II) acetate*

8.3.2 *Sulphuric acid,* concentrated, ρ = 1.84 g/ml (36N), free from nitrogen oxides.

To ensure complete elimination of nitrogen oxides, cautiously add 80 ml of the concentrated sulphuric acid to about 20 ml of water and heat until white fumes are liberated. Cool and repeat the dilution and heating twice.

8.3.3 *2,4-xylenol,* 10 g/l solution in acetic acid. Dissolve 1 g of 2,4-xylenol in glacial acetic acid (17M), and dilute to 100 ml with the same acid. Prepare the solution immediately before use.

8.3.4 *Potassium permanganate,* approximately 0.1N solution.

8.3.5 *Sodium hydroxide,* approximately 2N solution.

8.3.6 *Hydrogen peroxide,* 1 g/l solution.

8.3.7 *Potassium nitrate stock solution,* corresponding to 0.500 g of nitrogen (N) per litre. Weigh, to the nearest 0.001 g, 3.609 g of potassium nitrate, previously dried at 120 °C and cooled in a desiccator. Transfer to a 1 000 ml one-mark volumetric flask, dissolve in a little water, dilute to the mark and mix.

1 ml of this standard solution contains 500 μg of nitrogen.

8.3.8 *Potassium nitrate standard solution,* corresponding to 0.050 g of nitrogen (N) per litre. Transfer 50 ml of the potassium nitrate stock solution (**8.3.7**) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 50 μg of nitrogen.

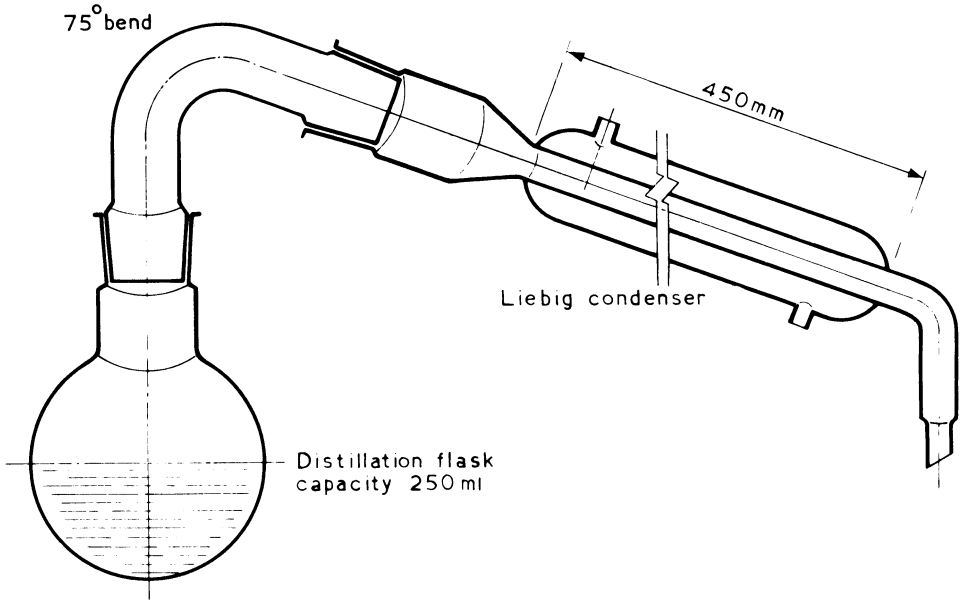

Figure 3 — Distillation apparatus for determination of nitrogen oxides content

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

8.4.1 *Weighing pipette,* approximately 50 ml capacity, with ground glass stoppers.

8.4.2 *Water bath,* capable of being controlled at 35 ± 1 °C.

8.4.3 *Distillation apparatus,* with conical ground glass joints, as shown in Figure 3.

8.4.4 *Spectrophotometer,* or

8.4.5 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of approximately 445 nm.

8.4.6 *Optical cells,* 4 cm.

8.4.7 *One-mark volumetric flasks,* 100 ml capacity, complying with the requirements of BS 1792.

8.4.8 *Stoppered graduated measuring cylinders,* 100 ml capacity, complying with the requirements of BS 604.

8.5 Procedure

8.5.1 *Preparation of colour standards.* Into a series of six one-mark volumetric flasks (**8.4.7**), transfer amounts of the standard potassium nitrate solution (**8.3.8**) indicated below.

Standard potassium nitrate solution (8.3.8)	**Corresponding mass of nitrogen**
ml	µg
0	0
4	200
8	400
12	600
16	800
20	1 000

Dilute to the mark with water, mix and treat each diluted solution as follows.

Place the distillation flask (see Figure 3) in a bath of water and ice; introduce 10 ml of one of the diluted standard potassium nitrate solutions and 0.200 g of mercury (II) acetate[3)] (**8.3.1**) into the flask and then add very slowly, in small portions and with stirring, so that the temperature remains all the time below 35 °C, 20 ml of the sulphuric acid (**8.3.2**). Withdraw the flask from the cooling bath, add 1 ml of the 2,4-xylenol solution (**8.3.3**), stir and place the flask in the water bath (**8.4.2**), controlled at 35 ± 1 °C. After 30 min, add to the flask the quantity of water necessary to bring the volume to approximately 120 ml.

Connect the flask to the distillation apparatus, heat to boiling and collect, over a period of approximately 15 min, 60 ml of distillate in one of the 100 ml graduated cylinders (**8.4.8**) containing 10 ml of the 2N sodium hydroxide solution (**8.3.5**).

When a volume of approximately 60 ml has been collected, stop the circulation of condenser water and distil a few more millilitres.

Cool the cylinder containing the distillate to room temperature, dilute to 100 ml, stopper and mix.

The distillates collected and diluted to 100 ml will have nitrogen (N) contents from 0 to 100 μg, increasing by steps of 20 μg.

Measure the absorbance of each solution against water in the reference cell at a wavelength of approximately 445 nm, and prepare a calibration graph.

8.5.2 *Determination.* Fill the weighing pipette (**8.4.1**) with the sample and take a test portion of approximately 20 g (or a lesser quantity for materials of high nitrogen oxide contents) containing not more than 100 μg of nitrogen, weighing by difference to the nearest 0.1 g (M_3). Adjust the mass to approximately 20 g, if necessary, by the addition of the sulphuric acid solution (**8.3.2**).

If sulphuric acid solution with a concentration greater than 75 % (*m*/*m*) is to be tested, add the test portion to crushed ice in the distillation flask, calculating the quantity of ice so that the resulting concentration of the solution is approximately 75 % (*m*/*m*). If, however, sulphuric acid with a concentration below 75 % (*m*/*m*) is to be tested, introduce the test portion into the distillation flask (A) and add the quantity of the sulphuric acid solution (**8.3.2**) necessary to bring the final concentration of the solution to approximately 75 % (*m*/*m*). During these operations, keep the temperature of the acid mixture below 35 °C.

Add to the distillation flask containing the test portion 0.200 g of the mercury (II) acetate (**8.3.1**) and the quantity of the potassium permanganate solution (**8.3.4**) necessary to give a stable pink coloration for a few minutes. Decolorize the solution by adding a few drops of the hydrogen peroxide solution (**8.3.6**), add 1 ml of the 2,4-xylenol solution (**8.3.3**), stir and place the flask in the water bath (**8.4.2**) controlled at 35 ± 1 °C.

After 30 min, dilute the contents of the flask with water to approximately 100 ml. Connect the flask to the distillation apparatus (**8.4.3**), heat to boiling and collect, over a period of approximately 15 min, 60 ml of distillate in one of the 100 ml graduated measuring cylinders (**8.4.8**) containing 10 ml of the 2N sodium hydroxide solution (**8.3.5**). When a volume of approximately 60 ml has been collected, stop the circulation of condenser water and distil a few more millilitres. Cool the cylinder containing the distillate to room temperature, dilute to the 100 ml mark, stopper and mix.

8.5.3 *Blank test.* At the same time as the determination, carry out a blank test using the reagents alone.

8.5.4 *Photometric measurements.* Measure the absorbance of the test solution and the blank solution at a wavelength of approximately 445 nm against water in the reference cell and read the amount of nitrogen present from the calibration graph (see **8.5.1**).

8.6 Expression of results. Nitrogen oxides, expressed as milligrams of nitrogen (N) per kilogram, are given by the formula:

$$\frac{M_1 - M_2}{M_3}$$

where

M_1 is the mass of nitrogen found in the test solution (in μg)

M_2 is the mass of nitrogen found in the blank (in μg)

M_3 is the mass of the test portion (in g)

[3)] The addition of mercury (II) acetate has been found necessary to avoid interference from chlorides.

9 Determination of chloride content

NOTE This method conforms to that specified in ISO 2877.

9.1 Field of application. This method is applicable to the determination of chloride content, expressed as hydrochloric acid, equal to or greater than 3 mg/kg.

9.2 Principle. The chloride ions present are titrated potentiometrically with standard volumetric silver nitrate solution in sulphuric acid medium, using silver and calomel electrodes.

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

9.3.1 *Sulphuric acid,* ρ approximately 1.30 g/ml, about 40 % (*m*/*m*) solution.

Carefully add, with stirring, about 29 ml of sulphuric acid, ρ approximately 1.84 g/ml, about 96 % (*m*/*m*) solution, to 70 ml of water, cool, dilute to 100 ml with water and mix.

9.3.2 *Silver nitrate,* approximately 0.1N solution.

Dissolve 8.5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution in a brown glass bottle.

9.3.3 *Silver nitrate,* approximately 0.01N solution.

Take 50 ml of the silver nitrate solution (**9.2**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

9.3.4 *Silver nitrate,* approximately 0.004N solution. Take 20 ml of the silver nitrate solution (**9.3.2**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

9.3.5 *Potassium chloride,* 0.1N standard reference solution.

Weigh, to the nearest 0.0001 g, 3.7276 g of potassium chloride, previously dried for 1 h at 130 °C and cooled in a desiccator. Dissolve in a little water, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

9.3.6 *Potassium chloride,* 0.01N standard reference solution.

Take 50.0 ml of the standard reference potassium chloride solution (**9.3.5**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

9.3.7 *Potassium chloride,* 0.004N standard reference solution.

Place 20.0 ml of the standard reference potassium chloride solution (**9.3.5**) in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

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

9.4.1 *Potentiometric titration apparatus,* as shown in Figure 4, comprising the items listed in **9.4.1.1** to **9.4.1.4**.

9.4.1.1 *Potentiometer,* sensitivity 2 mV, operating in the range – 500 mV to + 500 mV.

9.4.1.2 *Calomel electrode,* fitted with a safety reservoir, filled with saturated potassium chloride solution.

9.4.1.3 *Bridge,* containing sulphuric acid solution, ρ approximately 1.48 g/ml, connected to the calomel electrode (**9.4.1.2**).

9.4.1.4 *Silver electrode*

9.4.2 *Magnetic stirrer,* with a polytetrafluoroethylene (PTFE) coated rod.

9.4.3 *Burette,* 10 ml, with fine-pointed tip, graduated in 0.05 ml divisions and complying with the requirements of BS 646.

9.5 Procedure. Select the reagent solutions and test portion according to the expected chloride content, as indicated in Table 1.

9.5.1 *Calibration of the silver nitrate solution.* Take 5.0 ml and 10.0 ml respectively of the appropriate standard reference potassium chloride solution and place in two low-form beakers of convenient capacity (e.g. 100 ml). Carry out the following titration on the contents of each beaker.

Introduce a well-dried magnetic stirring rod (**9.4.2**) into the beaker and place the beaker in a container of convenient capacity (e.g. a basin of about 200 mm diameter). Surround the beaker with crushed ice to the level of the liquid. Add water to the container so as to equalize the temperature conditions around the beaker.

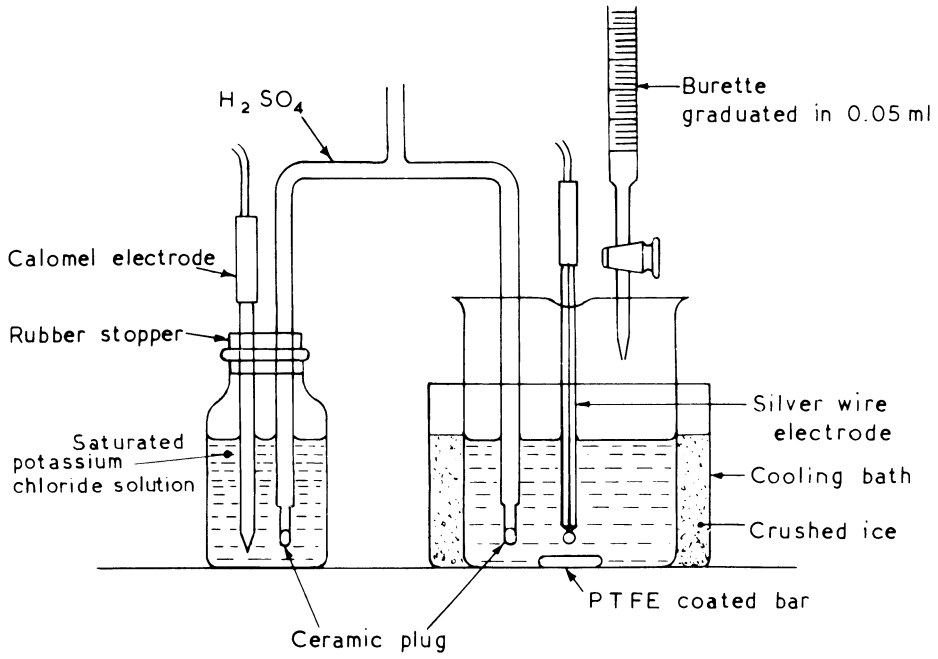

Figure 4 — Apparatus for use in determination of chloride content

Table 1 — Selection of reagent solutions and test portion

Expected chloride content, expressed as HCl	Silver nitrate solution concentration	Standard reference potassium chloride solution	Mass of test portion
mg/kg			
Above 3, up to and including 100	0.004N (**9.3.4**)	0.004N (**9.3.7**)	10 g to 30 g, weighed to the nearest 0.01 g
Above 100, up to and including 1 000	0.01N (**9.3.3**)	0.01N (**9.3.6**)	1 g to 10 g, weighed to the nearest 0.001 g
Above 1 000	0.1N (**9.3.2**)	0.1N (**9.3.5**)	1 g to 3 g, weighed to the nearest 0.001 g

Place the container and beaker on the magnetic stirrer (**9.4.2**) and set the stirrer in motion.

Place a thermometer in the beater and control the temperature at between 10 °C and 20 °C during the titration by the occasional addition of crushed ice to the container.

Add to the beaker, in small portions, 50 ± 2 ml of the sulphuric acid solution (**9.3.1**), previously cooled to between 10 °C and 20 °C.

Immerse the silver electrode (**9.4.1.4**) and the free end of the bridge (**9.4.1.3**) in the solution, ensuring that the liquid levels of this solution and of the saturated potassium chloride solution in the safety reservoir of the calomel electrode (**9.4.1.2**) are the same to prevent siphoning. Connect the electrodes to the potentiometer (**9.4.1.1**) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add the silver nitrate solution having the same normality as that of the standard reference potassium chloride solution used, from the burette (**9.4.3**), in 1 ml portions. After each addition, await the stabilization of the potential.

Note the volumes added and the corresponding values of the potential in the first two columns of a suitable table (for example, Table 2).

When approaching the endpoint, continue the addition of the silver nitrate solution, in portions of 0.2 ml for the 0.004N solution, 0.1 ml for the 0.01N solution or 0.05 ml for the 0.1N solution.

In the third column of Table 2 note the successive increments ($\Delta_1 E$) of the potential E. In the fourth column, Note the differences ($\Delta_2 E$), positive or negative, between the potential increments ($\Delta_1 E$).

The end of the titration corresponds to the addition of the 0.2 ml, 0.1 ml or 0.05 ml portion of the silver nitrate solution which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume (V_{EQ}) of the silver nitrate solution corresponding to the end of the reaction, use the formula:

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 is the volume of the silver nitrate solution immediately before the increment which gives the maximum increment of $\Delta_1 E$ (in ml)

b is the last value of $\Delta_2 E$ which is positive

B is the sum of the absolute values of the final positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (see Table 2)

V_1 is the volume (in ml) of the final increment of silver nitrate solution added (0.2, 0.1 or 0.05, as appropriate)

Table 2 — Worked example of the calibration of silver nitrate solution

Volume of silver nitrate solution V	Potential E	$\Delta_1 E$	$\Delta_2 E$
ml	mV	mV	mV
4.8	125		
		9	
4.9	134		+ 16
		25	
5.0	159		+ 91
		116	
5.1	275		– 67
		49	
5.2	324		
$V_{EQ} = 5.0 + 0.1 \times \frac{91}{91 + 67} = 5.057$			

9.5.2 ***Determination***

9.5.2.1 *Test portion.* Weigh, to the precision indicated in Table 1, a mass of test sample between 1 g and 30 g, depending on the expected chloride content, into a dry low-form beaker of convenient capacity (e.g. 100 ml).

9.5.2.2 *Titration.* Introduce a well-dried magnetic stirring rod (**9.4.2**) into the beaker and place the beaker in a container of convenient capacity (e.g. a basin of about 200 mm diameter). Surround the beaker with crushed ice to the level of the liquid. Add water to the container so as to equalize the temperature conditions around the beaker.

Place the container and beaker on the magnetic stirrer (**9.4.2**) and set the stirrer in motion.

Place a thermometer in the beaker and control the temperature at between 10 °C and 20 °C during the whole of the period of preparation of the test solution and titration, by the occasional addition of crushed ice to the container.

Add to the beaker, in small portions, 50 ± 2 ml of the sulphuric acid solution (**9.3.1**), previously cooled to between 10 °C and 20 °C.

Immerse the silver electrode (**9.4.1.4**) and the free end of the bridge (**9.4.1.3**) in the solution, ensuring that the levels of this solution and of the saturated potassium chloride solution in the safety reservoir of the calomel electrode (**9.4.1.2**) are the same to prevent siphoning. Connect the electrodes to the potentiometer (**9.4.1.1**) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add the silver nitrate solution from the burette (**9.4.3**) in 1 ml portions. After each addition, await the stabilization of the potential.

To obtain the endpoint of the titration, follow the appropriate instructions given in **9.5.1**, using the appropriate volume increment, according to the concentration of the solutions used.

9.6 Expression of results. The chloride content, expressed in milligrams of hydrochloric acid (HCl) per kilogram, is given by the formula:

$$\frac{M_1 \times 5 \times (V_4 - 2V_3 + V_2)}{M_0 \times (V_2 - V_3)} \times 10^6$$

where

M_0 is the mass of hydrochloric acid solution (in g) corresponding to 1 ml of the silver nitrate solution (= 0.000 146 for 0.004N solutions, = 0.000 365 for 0.01N solutions, and = 0.003 65 for 0.1N solutions)

M_1 is the mass of the test portion (in g)

V_2 is the value of V_{EQ} corresponding to the titration of 10 ml of the standard reference potassium chloride solution (in ml)

V_3 is the value of V_{EQ} corresponding to the titration of 5 ml of the standard reference potassium chloride solution (in ml)

V_4 is the value of V_{EQ} corresponding to the titration of the test solution (in ml)

5 is the difference between the two volumes of the standard reference potassium chloride solution used for the calibration (in ml)

10 Determination of iron and lead contents by atomic absorption spectroscopy

NOTE There is no corresponding ISO method.

10.1 Principle. A suitable quantity of the sample is evaporated to dryness, the residue is dissolved in dilute nitric acid solution and diluted to 10 ml. This solution is aspirated into the atomic absorption spectrophotometer, which has been standardized for the metal being determined.

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

10.2.1 *Compressed air supply*

10.2.2 *Acetylene*, from cylinder.

10.2.3 *Nitric acid*, 5N reagent solution.

10.2.4 *Iron solution*, standard solution corresponding to 1 g of iron (Fe) per litre. Dissolve 7.022 g of ammonium (II) iron sulphate hexahydrate in 600 ml of water and 65 ml of concentrated nitric acid solution, 70 % *m*/*m* (16N). Dilute to 1 000 ml with water.

1 ml of this solution contains 0.001 g of iron.

10.2.5 *Lead solution*, standard solution corresponding to 1 g of lead (Pb) per litre. Dissolve 1.60 g of lead nitrate in 600 ml of water and 65 ml of concentrated nitric acid solution, 70 % (*m*/*m*) (16N). Dilute to 1 000 ml with water.

1 ml of this solution contains 0.001 g of lead.

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

10.3.1 *Atomic absorption spectrophotometer*, with associated hollow cathode lamps.

10.3.2 *Silica basin,* 100 mm diameter, capacity 100 ml.

10.3.3 *Sand bath,* to accommodate the silica basin.

10.3.4 *One-mark volumetric flasks*, twelve 100 ml and one 10 ml capacity, complying with the requirements of BS 1792.

10.4 Procedure

10.4.1 *Preparation of standard graphs.* Into a series of six of the 100 ml one-mark volumetric flasks (**10.3.4**), transfer respectively the volumes of the standard iron solution (**10.2.4**) indicated below.

Standard iron solution (10.2.4)	**Corresponding mass of iron**
ml	µg/ml
0.0	0
1.0	10
2.0	20
3.0	30
4.0	40
5.0	50

Add 50 ml of the 5N nitric acid (**10.2.3**) to each flask and dilute to the marks with water.

Prepare dilute standard lead solutions covering the range 10 µg to 50 µg of lead per millilitre by diluting the standard lead solution (**10.2.5**) in a similar manner.

Aspirate each of the standards in turn into the atomic absorption spectrophotometer (**10.3.1**) using water to set the zero and the following wavelengths and fuel systems:

Iron	248 nm	Air/acetylene
Lead	217 nm	Air/acetylene

Use the recommended fuel settings to provide the maximum absorbance for the most concentrated standard solution for each metal and then read the absorbance for each of the remaining standards.

Plot a graph of absorbance against micrograms of metal per millilitre for each of the metals.

10.4.2 *Determination.* Transfer a quantity of the sample containing not more than 500 µg of the metal to be determined to the silica basin (**10.3.2**) and evaporate to dryness on the sand bath (**10.3.3**) in a fume cupboard. To the cooled residue add 5 ml of the nitric acid solution (**10.2.3**) and 25 ml of water.

Warm to dissolve and evaporate to dryness. Dissolve the cooled residue in 5 ml of the nitric acid solution (**10.2.3**) and dilute to 10 ml in the 10 ml one-mark volumetric flask (**10.3.4**). Aspirate this solution into the atomic absorption spectrophotometer (**10.3.1**) standardized for the metal being determined as described in **10.4.1**. Read the amount of metal present in the 10 ml dilution in µg/ml from the appropriate standard graph.

10.4.3 *Blank test.* At the same time as the determination, carry out a blank test following the same procedure and using the same quantities of reagents used in the determination.

10.5 Expression of results. The metal content, expressed as milligrams of iron (Fe) or lead (Pb) per kilogram, is given by the formula:

$$\frac{M_1 - M_2}{M_0} \times 10$$

where

M_0 is the mass of the test portion (in g)

M_1 is the mass of metal found in the test solution (in µg/ml)

M_2 is the mass of metal found in the blank test solution (in µg/ml)

11 Determination of iron content

NOTE This method embodies the same principle as that used in ISO/R 915.

11.1 Field of application. The method is applicable to the determination of iron (Fe) in the range 1 mg/kg to 250 mg/kg.

11.2 Principle. The iron present is reduced to the ferrous state and determined spectrophotometrically using 2,2′-bipyridyl.

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

11.3.1 *Hydrochloric acid*, approximately N solution.

11.3.2 *Hydroxylammonium chloride*, 50 g/l solution.

11.3.3 *Ammonium acetate*, 400 g/l solution.

11.3.4 *2,2′-Bipyridyl, 1 g/l solution.* Dissolve 0.1 g of the reagent in 50 ml of water containing 2 ml of the N hydrochloric acid solution (**11.3.1**) and dilute to 100 ml.

11.3.5 *Iron*, standard solution corresponding to 10 mg of iron per litre. Dissolve 7.022 g of ammonium iron (II) sulphate hexahydrate in a mixture of 600 ml of water and 350 ml of concentrated sulphuric acid, 98 % (*m*/*m*) (35N). Dilute to 1 000 ml with water and further dilute 10 ml of the solution so obtained to 1000 ml with water.

1 ml of the diluted solution contains 10 µg of iron.

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

11.4.1 *Spectrophotometer*, or

11.4.2 *Photoelectric absorptiometer*, fitted with filters providing maximum transmission at a wavelength of approximately 515 nm.

11.4.3 *Optical cells*, 4 cm.

11.4.4 *One-mark volumetric flasks*, 100 ml capacity, complying with the requirements of BS 1792.

11.4.5 *One-mark volumetric flask*, 250 ml capacity, complying with the requirements of BS 1792.

11.5 Procedure

11.5.1 *Preparation of colour standards.* Into a series of six of the 100 ml one-mark volumetric flasks (**11.4.4**), each containing 50 ml of water and 2 ml of the N hydrochloric acid solution (**11.3.1**), transfer the volumes of the standard iron solution (**11.3.5**) indicated below.

Standard iron solution (11.3.5)	**Corresponding mass of iron**
ml	µg
0	0
2	20
4	40
6	60
8	80
10	100

Treat each solution as follows.

Add 4 ml of the hydroxylammonium chloride solution (**11.3.2**) and allow to stand for 1 min. Add 5 ml of the ammonium acetate solution (**11.3.3**) and 3 ml of the 2,2′-bipyridyl solution (**11.3.4**). Dilute the contents of each flask to the mark and mix thoroughly.

Measure the absorbance of each solution against water in one of the 4 cm cells (**11.4.3**) at a wavelength of about 515 nm and prepare a calibration graph having, for example, the iron content in µg/100 ml of the colour standards as abscissae and the corresponding values of absorbance as ordinates.

11.5.2 *Determination.* Weigh, to the nearest 0.1 g, about 10 g of the sample into a silica basin and evaporate just to dryness. Cool and redissolve the residue in 50 ml of the hydrochloric acid solution (**11.3.1**). Transfer to the 250 ml one-mark volumetric flask (**11.4.5**) and dilute with water to the mark. Pipette 10 ml of this solution into one of the 100 ml one-mark volumetric flasks (**11.4.4**), add 4 ml of the hydroxylammonium chloride solution (**11.3.2**) and allow to stand for 1 min. Add 5 ml of the ammonium acetate solution (**11.3.3**), mix and add 3 ml of the 2,2′-bipyridyl solution (**11.3.4**). Dilute to 100 ml with water and mix thoroughly.

11.5.3 *Blank test.* At the same time as the determination, carry out a blank test on the reagents alone.

11.5.4 *Photometric measurements.* Measure the absorbances of the solutions deriving from the test portion and from the blank test at a wavelength of approximately 515 nm against water and read the amounts of iron present from the calibration graph (see **11.5.1**).

11.6 Expression of results. Iron content, expressed as milligrams of iron (Fe) per kilogram of test sample, is given by the formula:

$$\frac{M_1 - M_2}{M_0} \times 25$$

where

M_0 is the mass of the test portion (in g)

M_1 is the mass of iron found in the test portion (in μg)

M_2 is the mass of iron found in the corresponding blank test determination (in μg)

12 Determination of lead content

NOTE This method conforms to that specified in ISO 2717.

12.1 Field of application. The method is applicable to the determination of lead (Pb) content greater than 1 mg/kg.

12.2 Principle. The lead is extracted by evaporating the test portion to dryness and dissolving the residue in hydrochloric acid. This is followed by reduction using hydroxylammonium chloride. Interfering elements are suppressed by the formation of complexes with ammonium citrate and potassium cyanide. The lead is finally extracted in the form of a solution of lead dithizonate in chloroform and determined photometrically.

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

12.3.1 *Chloroform*, redistilled in a borosilicate glass apparatus with ground joints, ρ 1.471 g/ml to 1.474 g/ml.

12.3.2 *Hydrochloric acid*, ρ approximately 1.19 g/ml, about 38 % (*m*/*m*) solution, or approximately 12N.

12.3.3 di*Ammonium hydrogen citrate*, $[(NH_4)_2HC_6H_5O_7]$, 100 g/l solution.

12.3.3.1 *Purification of the solution.* To 100 ml of the solution add ammonia solution, ρ approximately 0.91 g/ml, until the pH reaches a value of between 8.5 and 10, checking with the indicator paper (**12.3.11**). Transfer the solution to a separating funnel, add 10 ml of the dithizone solution (**12.3.7**), and shake vigorously. Allow to separate and withdraw and reject the organic phase. Repeat the extraction, each time with 5 ml of the dithizone solution (**12.3.7**), until the green colour remains. Allow to separate and withdraw and reject the organic phase.

12.3.4 *Potassium cyanide*, 50 g/l solution.

WARNING. EXTREMELY POISONOUS (see **12.5**).

12.3.5 *Hydroxylammonium chloride*, 100 g/l solution.

12.3.6 *Ammonia,* approximately 5N solution.

NOTE Lead contained in glass is soluble in ammonia solution and is dissolved more rapidly by diluted than by concentrated solutions. It is essential, therefore, that a freshly prepared ammonia solution be used to avoid obtaining too high a value in the blank test.

12.3.7 *Dithizone*, (diphenylthiocarbazone), 0.025 g/l solution in chloroform.

12.3.7.1 *Purification of the dithizone.* If dithizone of satisfactory quality is not available (as indicated, for example, by anomalous calibration results) it may be purified as follows.

Dissolve 1 g of dithizone in 75 ml of the chloroform (**12.3.1**). Filter the solution, collecting the filtrate in a 250 ml separating funnel. Add 100 ml of approximately 0.2N ammonia solution and shake vigorously. Withdraw the organic phase, collecting it in another separating funnel, and repeat the same operation a further three times, using 100 ml of approximately 0.2N ammonia solution each time. (The dithizone passes into the alkaline aqueous phase, colouring it orange, while the oxidation products remain in the organic phase, which develops an intense reddish-yellow coloration.)

Discard the organic phase and filter the combined orange-coloured aqueous extracts, collecting the filtrate in a 1 000 ml beaker.

Precipitate the dithizone by slight acidification with a saturated solution of sulphur dioxide. Allow the precipitate to settle, filter through a sintered glass crucible and wash with water until there is no further acid reaction. Dry the precipitate in a desiccator containing concentrated sulphuric acid solution, ρ approximately 1.84 g/ml, under vacuum and in darkness, for a period of 3 days to 4 days.

Grind the solid dry product quickly and transfer immediately to a small dark glass bottle. If stored away from direct sunlight, the dithizone can be used for this determination during a period up to at least 6 months after purification.

12.3.7.2 *Preparation of the solution*. Immediately before use, weigh, to the nearest 1 mg, 25 mg of the purified dithizone (**12.3.7.1**), transfer to a 100 ml one-mark volumetric flask, dissolve in the chloroform (**12.3.1**), dilute to the mark with the same chloroform and mix.

Store the solution in a dry, dark glass, airtight bottle.

12.3.8 *Potassium cyanide*, 1 g/l ammoniacal solution.

WARNING. EXTREMELY POISONOUS (see **12.5**).

Transfer 20 ml of the potassium cyanide solution (**12.3.4**) to a 1 000 ml one-mark volumetric flask. Dilute with water, add 10 ml of ammonia solution, ρ approximately 0.88 g/ml, dilute to the mark and mix.

12.3.9 *Lead,* standard solution corresponding to 1 g of lead (Pb) per litre.

Weigh, to the nearest 0.001 g, 1.600 g of lead nitrate [$Pb(NO_3)_2$], previously dried at 105 °C and cooled in a desiccator, and transfer to a beaker of suitable capacity. Dissolve in a little water and 1 ml of nitric acid solution, ρ approximately 1.40 g/ml. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 mg of lead.

12.3.10 *Lead*, standard solution corresponding to 0.010 g of lead (Pb) per litre.

Transfer 10.00 ml of the standard lead solution (**12.3.9**) to a 1 000 ml one-mark volumetric flask, add 1 ml of nitric acid solution, ρ approximately 1.40 g/ml, dilute to the mark and mix.

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

Prepare this solution immediately before use.

12.3.11 *Narrow range indicator papers*, covering the range from 8.5 to 10, as defined in BS 1647.

12.4 Apparatus

NOTE It is essential that all glassware, including the reagent bottles,
a) be made of borosilicate glass or of another suitable lead-free glass (alternatively, suitable plastics material may be used in this respect);
b) be washed with an approximately 7N solution of nitric acid and rinsed three times with water.

Ordinary laboratory apparatus, other than glassware, and the following are required.

12.4.1 *Weighing pipette,* capacity about 60 ml, with ground glass stoppers.

12.4.2 *Burette,* 25 ml capacity, graduated in 0.05 ml, complying with the requirements of BS 846.

12.4.3 *Spectrophotometer,* or

12.4.4 *Photoelectric absorptiometer,* fitted with filters giving maximum transmission between 500 nm and 540 nm.

12.4.5 *Optical cells,* 1 cm.

12.4.6 *Separating funnels,* 100 ml capacity, fitted with ground glass stoppers.

12.4.7 *One-mark volumetric flask,* 50 ml capacity, complying with the requirements of BS 1792.

12.5 Procedure

WARNING. Potassium cyanide is extremely poisonous and shall only be used with all necessary precautions. In particular, do not add acids to solutions containing potassium cyanide, otherwise hydrogen cyanide will be released.

12.5.1 *Test portion.* Fill the weighing pipette (**12.4.1**) with part of the test sample and, weighing by difference to the nearest 0.02 g, take a test portion of about 50 g. Transfer the test portion to a 250 ml beaker.

12.5.2 *Blank test.* At the same time as the determination, carry out a blank test following the same procedure and preparation of the calibration graph and using the same quantities of all reagents as used in the determination.

12.5.3 ***Preparation of the calibration graph***

12.5.3.1 *Preparation of the standard matching solutions, for photometric measurements with a 1 cm cell.* Into a series of six of the separating funnels (**12.4.6**) transfer 10 ml of water and add respectively the volumes, measured with the burette (**12.4.2**), of the standard lead solution (**12.3.10**) indicated as follows:

Standard lead solution (12.3.10)	**Corresponding mass of lead**
ml	µg
0	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

Treat each of these solutions in the following manner.

Add 1 ml of the hydroxylammonium chloride solution (**12.3.5**) and 10 ml of the *di*ammonium hydrogen citrate solution (**12.3.3**), and adjust the pH to between 8.5 and 10 by adding the ammonia solution (**12.3.6**) drop by drop, checking with the indicator paper (**12.3.11**). Add 2 ml of the potassium cyanide solution (**12.3.4**) and shake. Add 5 ml of the dithizone solution (**12.3.7**) and extract the lead dithizonate by shaking vigorously for 1 min. Allow to separate and draw off the organic phase, collecting it in one of the 50 ml one-mark volumetric flasks (**12.4.7**). Repeat the extraction with successive 5 ml portions of the dithizone solution (**12.3.7**), until the last portion of the dithizone solution remains green after swirling. Collect all the extracts (organic phase) in the same 50 ml one-mark volumetric flask, dilute to the mark with the chloroform (**12.3.1**) and mix.

Remove the excess of dithizone present in the organic phase by extracting with the minimum number of successive 5 ml portions of the ammoniacal cyanide solution (**12.3.8**) until the aqueous extract is no longer yellow. Then draw off the organic phase, which will have a clear pink colour, and pass it through a dry, "acid-washed" filter paper, collecting the filtrate in a dry vessel.

NOTE Dithizonates are particularly sensitive to ultraviolet light and should, therefore, be protected from sunlight and fluorescent light.

12.5.3.2 *Photometric measurements.* Carry out the photometric measurements using the spectrophotometer (**12.4.3**) at the maximum of the absorption curve (wavelength of about 520 nm) or with the photoelectric absorptiometer (**12.4.4**) fitted with suitable filters; in each case adjust the instruments to zero absorbance against the chloroform (**12.3.1**).

12.5.3.3 *Preparation of the calibration graph.* Plot a graph having, for example, the lead (Pb) contents, expressed in micrograms per 50 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

12.5.4 ***Determination***

12.5.4.1 *Preparation of the test solution.* Place the beaker containing the test portion (**12.5.1**) on a sand bath and evaporate cautiously to dryness in a well-ventilated fume cupboard. Cool, take up with 2 ml of the hydrochloric acid solution (**12.3.2**) and warm moderately to complete the dissolution. Allow to cool, transfer the solution quantitatively to a one-mark volumetric flask of suitable capacity, dilute to the mark and mix.

12.5.4.2 *Extraction of the lead dithizonate.* According to the expected lead content, take an aliquot portion of the test solution (**12.5.4.1**) containing 10 µg to 100 µg of lead and transfer it to a separating funnel of suitable capacity.

Then proceed with the determination as described in **12.5.3.1**, from paragraph 2, for the preparation of the standard matching solutions.

NOTE If the Pb content to be determined is of the order of 1 mg/kg, use the whole of the test solution, without dilution, for the extraction of the lead dithizonate.

12.5.4.3 *Photometric measurements.* Carry out the photometric measurements of the chloroform solutions of lead dithizonate deriving from the test solution and from the blank test, according to the method described in **12.5.3.2**, but after having adjusted the instrument to zero absorbance against the chloroform (**12.3.1**).

12.6 Expression of results. By means of the calibration graph (**12.5.3.3**), determine the quantity of lead corresponding to the values of the photometric measurements.

The lead (Pb) content is given in milligrams per kilogram, by the formula:

$$\frac{M_1 - M_2}{M_0} \times D$$

where

M_0 is the mass of the test portion (in g)

M_1 is the mass of lead found in the aliquot portion of the test solution (in μg)

M_2 is the mass of lead found in a corresponding aliquot portion of the blank test solution (in μg)

D is the ratio of the volume of test solution to the volume of aliquot portion taken for the extraction of the lead dithizonate

12.7 Note on procedure. The chloroform used for the analysis may be recovered. For this purpose, remove the lead from the collected organic phase by shaking with an aqueous hydrochloric acid solution. Then remove the dithizone by shaking with ammonia solution. Repeat this treatment until the chloroform becomes colourless, then wash it by shaking with water and finally distil it in a borosilicate glass apparatus, with ground glass joints, in the presence of a little phosphorus pentoxide. It is recommended that the distilled chloroform be stabilized against the development of acidic impurities by the addition of 1 % of industrial methylated spirits.

Section 3. Methods of test for oleums and liquid sulphur trioxide

NOTE Although the methods of test described in clauses **14** and **15** may be used for oleums or liquid sulphur trioxide, it is emphasized that the method of sampling described in clause **13** is suitable only for oleums. It is advised that the manufacturer be consulted in respect of techniques for sampling liquid sulphur trioxide.

13 Sampling of oleums

NOTE 1 This method is unsuitable for the sampling of liquid sulphur trioxide.
NOTE 2 There is no corresponding ISO method.

13.1 Principle. The sample is obtained by immersing a glass bottle, suspended in a metal cage or holder, into the material to be sampled.

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

13.2.1 *Glass bottle and stopper of suitable size.* For assay samples it is convenient to use a 100 ml capacity narrow-mouthed, glass-stoppered bottle.

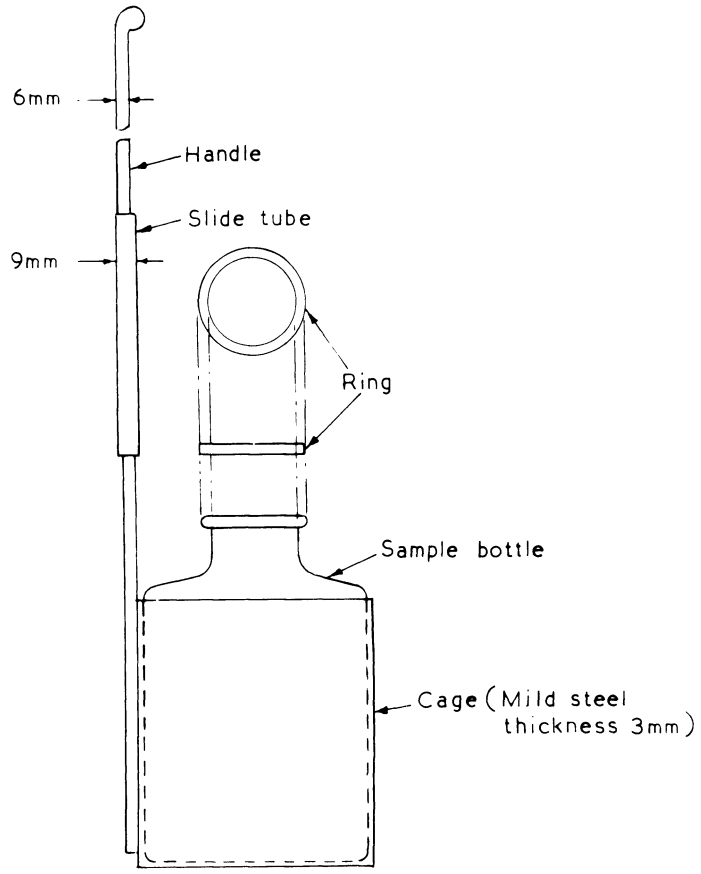

Figure 5 — Sampling cage: sampling of oleums

13.2.2 *Metal sampling cage,* as shown in Figure 5.

The cage is constructed of 3 mm mild steel sheet to fit the bottle closely and is welded to a mild steel rod, which forms the handle, 6 mm in diameter and approximately 1.5 m in length. The handle is fitted with a slide tube 600 mm in length, made of mild steel tubing having an internal diameter of 9 mm to which is welded a mild steel ring. When lowered, the ring fits over the neck of the sample bottle thus retaining the latter in position during the sampling operation.

13.3 Procedure

WARNING. Protective clothing including goggles and gloves shall be worn at all times when handling oleums.

Clean and *thoroughly dry* the sample bottle (**13.2.1**) and sampling cage (**13.2.2**). Place the bottle in the cage and fit the ring into position around the neck of the bottle. Withdraw the stopper from the bottle.

Collect the sample by immersing the cage and bottle in the material to be sampled. Carefully withdraw the sampling assembly, stopper the bottle immediately and allow to drain. Remove the bottle from the sampling cage and carefully wipe the outside of the bottle with filter paper or a clean dry cloth to remove all traces of oleum.

14 Dilution of oleums and liquid sulphur trioxide for analysis

NOTE There is no corresponding ISO method.

14.1 Principle. The oleum or liquid sulphur trioxide is added to a calculated quantity of ice in an enclosed externally cooled system prior to using the diluted solution for analysis by any of the methods described in clauses **5** to **9**.

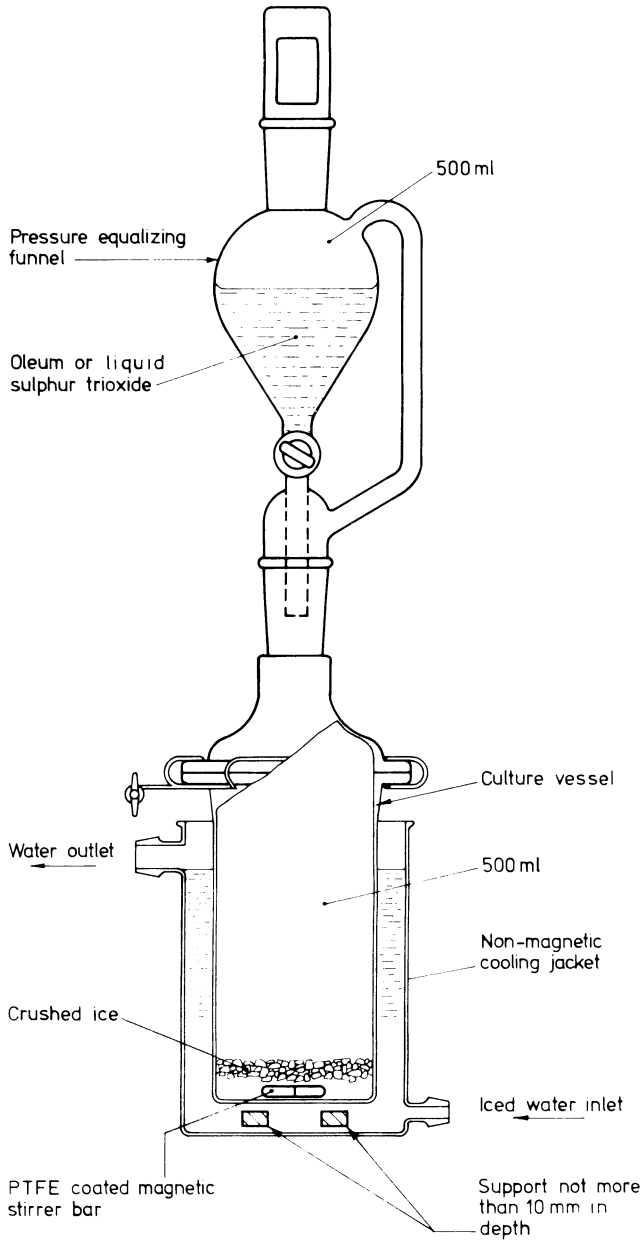

Figure 6 — Dilution apparatus for oleums and liquid sulphur trioxide

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

14.2.1 *Dilution apparatus,* as shown in Figure 6, comprising the items listed in **14.2.1.1** to **14.2.1.4**.

14.2.1.1 *Pressure equalizing funnel,* stoppered, 500 ml capacity.

14.2.1.2 *Culture vessel,* 500 ml capacity, with flat flanged lid and clip.

14.2.1.3 *Non-magnetic cooling jacket,* at least 90 mm in diameter.

14.2.1.4 *Magnetic stirrer and PTFE coated magnetic stirrer bar.*

NOTE The use of glass joints is advocated as a safeguard against uncontrolled pressure build-up.

14.3 Procedure. Place the PTFE coated magnetic stirrer bar (**14.2.1.4**) into the culture vessel (**14.2.1.2**) and add the calculated quantity of crushed ice (prepared from water complying with the requirements of BS 3978) to give 75 % sulphuric acid solution which, together with the oleum or liquid sulphur trioxide under test, will produce the volume of sulphuric acid required for analysis.

Place the funnel (**14.2.1.1**) in position and quickly transfer to it the necessary amount of oleum or liquid sulphur trioxide, determining its mass by weighing the sample before and after transfer.

Immediately assemble the apparatus and place it in position on the magnetic stirrer (**14.2.1.4**). Allow cooling water to pass continuously through the cooling jacket (**14.2.1.3**), to ensure that the temperature does not exceed 35 °C.

Slowly add the oleum or liquid sulphur trioxide sample onto the ice, maintaining stirring throughout.

When the addition is complete, continue to cool the apparatus for a further 10 min.

Remove the cooling jacket, ensure that the lid of the culture vessel is clipped firmly in position and carefully wash through the tubing and funnel by inverting the apparatus.

15 Determination of residue on heating of oleums and liquid sulphur trioxide

NOTE This method uses the heating temperature given in the corresponding method described in ISO/R 913.

15.1 Principle. The sample is evaporated and the residue is heated at 800 ± 50 °C.

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

15.2.1 *Platinum basin,* flat-bottom, capacity about 100 ml.

15.2.2 *Muffle furnace,* capable of being controlled at 800 ± 50 °C.

15.2.3 *Stoppered measuring cylinder,* capacity 100 ml, complying with the requirements of BS 604.

15.2.4 *Desiccator*

15.3 Procedure. Heat the platinum basin (**15.2.1**) in the muffle furnace (**15.2.2**) controlled at 800 ± 50 °C. Cool in the desiccator (**15.2.4**) and weigh to 0.1 mg. Transfer approximately 50 g of the sample to the basin from the measuring cylinder (**15.2.3**), determining the mass of the test portion to 0.01 g by weighing the measuring cylinder before and after transfer.

Evaporate the contents of the basin (**15.2.1**) carefully on a sand bath in a fume cupboard and heat the basin and residue to dryness. Transfer the basin containing the residue to the muffle furnace (**15.2.2**) controlled at 800 ± 50 °C and heat for about 15 min. Remove the basin from the furnace, place in the desiccator, cool and weigh to the nearest 0.1 mg. Repeat the operations of heating, cooling and weighing until the difference between successive weighings does not exceed 0.5 mg.

15.4 Expression of results. The residue on heating, expressed as milligrams per kilogram, is given by the formula:

$$\frac{M_1 \times 10^6}{M_2}$$

where

M_1 is the mass of the residue (in g)

M_2 is the mass of the test portion (in g)

16 Determination of total acidity and calculation of free sulphur trioxide content of oleums

NOTE This method differs from the corresponding method described in ISO/R 910 which was disapproved by the United Kingdom for technical reasons.

16.1 Field of application. The method is applicable to all strengths of oleum up to 70 % free sulphur trioxide.

16.2 Principle. The total acidity is determined by titration of a diluted test portion with sodium hydroxide in the presence of screened methyl orange indicator. The equivalent sulphur trioxide content is obtained by calculation.

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

16.3.1 *Sodium hydroxide*, N standard volumetric solution.

16.3.2 *Screened methyl orange indicator,* 1 g/l solution, prepared as described in BS 4123.

16.3.3 *Ice cubes*

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

16.4.1 *Conical flask,* 50 ml or 25 ml capacity with a 14/23 neck (BS 572) and glass stopper.

16.4.2 *Glass ampoule,* 5 ml capacity, with flat base complying with the requirements of BS 795, type B. Prepare the ampoule for use as follows.

With a diamond pencil, scribe a small circle in the depression at the top of the ampoule, then pierce the glass at this point in order to release the vacuum. A light tap on a fine steel punch or a wire nail is sufficient to effect this.

Soften the stem of the ampoule in a bunsen flame and draw out to a capillary of not more than 1 mm diameter.

Cut off the capillary to a length sufficient to reach near to the bottom of the conical flask (**16.4.1**) when the shoulders of the ampoule rest on the upper rim of the ground neck.

16.4.3 *Bulb burette*, 105 ml nominal capacity, complying with the requirements of BS 846, having a bulb capacity of 80 ml and a scale ranging from 80 ml to 105 ml with 0.05 ml subdivisions.

16.4.4 *Wide-mouth glass bottle*, 500 ml capacity, having a well-fitting rubber stopper.

16.5 Procedure. Transfer sufficient of the sample to fill the conical flask (**16.4.1**), which shall be previously dried, to within 10 mm of the ground glass joint and insert the stopper.

Prepare the ampoule (**16.4.2**) as described and weigh to the nearest 0.1 mg. Remove the stopper from the conical flask (**16.4.1**), immediately insert the capillary of the ampoule in the sample and allow the shoulders of the ampoule to rest on the neck of the flask. This will ensure that the acid does not lose strength while the ampoule is being filled.

With a small bunsen flame, gently warm the ampoule until the flame shows a yellow tinge (from the sodium in the glass). Then remove the flame and allow the ampoule to cool until a quantity of the sample, weighing between 4.0 g and 4.4 g, has been drawn into the ampoule. (A little experience will determine the amount of heating necessary to achieve this.)

Remove the ampoule from the flask, reinsert the stopper and rapidly seal the capillary of the ampoule by inserting the tip in a bunsen flame. Allow to cool, then wash off any acid remaining on the outside of the ampoule with water, carefully wipe dry and weigh to the nearest 0.1 mg.

Transfer the ampoule to the wide-mouth bottle (**16.4.4**) containing about 100 ml water tinted with the screened methyl orange indicator (**16.3.2**) and four ice cubes (**16.3.3**) (about 100 g). Stopper the bottle tightly, wrap in a cloth and shake vigorously until the ampoule is shattered. Continue shaking until any fume in the air space has been completely absorbed.

Remove the stopper, wash any adhering acid back into the bottle and titrate with the sodium hydroxide solution (**16.3.1**) to the neutral grey colour of the indicator. Continue the titration, adding the smallest possible increments, until the indicator just turns green. Record the burette (**16.4.3**) reading to the nearest 0.05 ml.

16.6 Expression of results. Total acidity, expressed as a percentage by mass of sulphuric acid (H_2SO_4), is given by the formula:

$$\frac{4.904V}{M}$$

The free sulphur trioxide content, expressed as a percentage by mass of SO_3, is given by the formula:

$$\frac{21.79V}{M} - 444.4$$

where

V is the volume of the sodium hydroxide solution used (in ml)

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

Publications referred to

BS 572, *Interchangeable conical ground glass joints.*

BS 604, *Graduated measuring cylinders.*

BS 646, *Cartridge fuse-links (rated up to 5 amperes) for a.c. and d.c. service.*

BS 795, *Ampoules.*

BS 846, *Burettes and bulb burettes.*

BS 1647, *pH scale.*

BS 1752, *Laboratory sintered or fritted filters.*

BS 1792, *One-mark volumetric flasks.*

BS 2058, *Lunge-Rey weighing pipette.*

BS 2586, *Glass electrodes for measurement of pH.*

BS 3145, *Laboratory potentiometric pH meters.*

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 4426, *Methods of test for sodium hypochlorite solution.*

ISO/R 910, *Sulphuric acid and oleum for industrial use — Determination of total acidity and calculation of free SO_3 content of oleum — Volumetric method.*

ISO/R 913, *Sulphuric acid and oleum for industrial use — Determination of residue on ignition — Gravimetric method.*

ISO/R 915, *Sulphuric acid and oleum for industrial use — Determination of iron content — 2,2′-bipyridyl spectrophotometric method.*

ISO 2363, *Sulphuric acid and oleums for industrial use — Determination of oxides of nitrogen — 2,4-xylenol spectrophotometric method.*

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

ISO 2717, *Sulphuric acid and oleum for industrial use — Determination of lead content — Dithizone photometric method.*

ISO 2877, *Sulphuric acid for industrial use — Determination of chlorides content — Potentiometric method.*

ISO 2899, *Sulphuric acid and oleums for industrial use — Determination of ammoniacal nitrogen content — Spectrophotometric method.*

ISO 3423, *Sulphuric acid and oleums for industrial use — Determination of sulphur dioxide content — Iodometric method.*

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