BRITISH STANDARD

BS 3993:1966

Specification for

Hydrochloric acid commercial Types 1 and 2

Confirmed January 2011

Co-operating organizations

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

Board of Trade
British Iron and Steel Federation
Chemical Industries Association*
Fertilizer Manufacturers' Association
Gas Council*
Institute of Vitreous Enamellers
Institution of Gas Engineers
Ministry of Defence, Army Department*
Ministry of Health
National Sulphuric Acid Association*
Royal Institute of Public Health & Hygiene

The Government department and industrial 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:

Royal Institute of Chemistry

This British Standard, having been approved by the Chemicals Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 7 March 1966

The following BSI references relate to the work on this standard:
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Contents

Foreword

This standard makes reference to the following British Standards:

BS 506, *Methanol.*

BS 612, *Nessler cylinders.*

BS 846, *Burettes and bulb burettes.*

BS 976, *Density composition tables for aqueous solutions of hydrochloric acid.*

BS 1647, *pH scale.*

BS 1752, *Laboratory sintered or fritted filters.*

BS 1792, *One-mark volumetric flasks.*

BS 3218, *Test tubes and boiling tubes.*

BS 3591, *Industrial methylated spirits.*

The preparation of this British Standard was authorized by the Chemicals Industry Standards Committee in order to provide specifications for hydrochloric acid.

The importance of the impurities that may be present in hydrochloric acid depends to some extent on the use to which the material is to be put. This standard specifics those impurities that are commonly of importance to users who may, however, wish to supplement them with requirements in respect of such matters as extraneous odour and organic impurities.

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 16 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This British Standard specifies two types of hydrochloric acid.

2 Description

The material shall consist essentially of an aqueous solution of hydrogen chloride, HCl. It shall be clear, ranging in colour from colourless to pale yellow.

3 Requirements

The material shall comply with the requirements as shown in the following table when tested by the appropriate method given in Column 4:

	Type 1	Type 2	Method of test in Appendix
Relative density[a] at 20/20 °C	**1.137 to 1.142 or 1.157 to 1.162 or 1.177 to 1.182**		—
Residue on evaporation: per cent by weight, maximum	0.1	[b]	A
Sulphated residue on ignition: per cent by weight, maximum	0.05	0.2	B
Total sulphate (as H_2SO_4): per cent by weight, maximum	0.05		C
		0.6	D
Iron: parts per million by weight, maximum	5	50	E
Arsenic: parts per million by weight, maximum	1	2	F
Lead: parts per million by weight, maximum	1	2	G
Copper: parts per million by weight, maximum	1	2	H
Mercury: parts per million by weight, maximum	5	[b]	J
Oxidizing substances (as Cl_2): parts per million by weight, maximum	50	100	K
Reducing substances (as SO_2): parts per million by weight, maximum	50	100	K

NOTE The HCl content of a sample of Type 1 acid may be obtained by reference to BS 976, "*Density composition tables for aqueous solutions of hydrochloric acid*".
Because of the dissolved solids it may contain, the HCl content of a sample of Type 2 acid may be obtained only approximately in this way.
[a] Formerly "specific gravity" : "relative density" is the term adopted for this concept, with water as the reference substance, by the International Organization for Standardization (ISO).
[b] Not specified.

4 Sampling and size of sample

For the purpose of examination in accordance with this specification a representative sample of the material measuring not less than 2 litres shall be taken from the bulk. The sample shall be placed in a clean dry air-tight glass-stoppered bottle of such a size that it is nearly filled by the sample. When it is necessary to seal the container, care shall be taken to avoid the risk of contaminating the contents in any way.

5 Packaging and marking

The material shall be supplied in suitable sound, clean, moisture-resistant containers which shall be marked to indicate clearly the full identity of the contents. The marking shall include the number of this British Standard, viz. BS 3993, Type 1, or BS 3993, Type 2.

NOTE The mark BS 3993 on or in relation to the product is a claim by the manufacturer that it complies with the requirements of the standard.

The British Standards Institution is the owner of a registered certification trade mark. This is shown below, enclosed in the words "Approved to British Standard". This mark can be used only by manufacturers licensed under the certification mark scheme operated by the BSI. The presence of this mark on or in relation to a product is an assurance that the goods have been produced to comply with the requirements of the British Standard under a system of supervision, control and testing operated during manufacture and including periodical inspection at the manufacturer's works in accordance with the certification mark scheme of the BSI.

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Appendix A Method for the determination of residue on evaporation

A.1 Apparatus

Platinum dish, approx. 2 in (50 mm) diameter.

A.2 Procedure

Transfer 100 ml of the sample in successive small portions to the weighed platinum dish and evaporate to dryness on a boiling-water bath in a fume cupboard. Dry the dish and residue for 30 minutes in an oven at a temperature of 105 ± 1 °C. Cool in a desiccator and weigh immediately.

A.3 Calculation

Residue on evaporation, per cent by weight

$$= \frac{W_1}{d}$$

where d = relative density, of the sample at room temperature

and W_1 = weight, in grammes, of residue found.

Appendix B Method for the determination of sulphated residue on ignition

B.1 Apparatus

Platinum dish, approx. 2 in (50 mm) diameter.

B.2 Reagent

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

Sulphuric acid, concentrated, d = 1.84.

B.3 Procedure

Transfer 250 ml (for Type 1 acid) or 50 ml (for Type 2 acid) of the sample in successive small portions to the weighed platinum dish and evaporate to dryness on a boiling-water bath in a fume cupboard. Cool and cover the residue with a few drops of concentrated sulphuric acid. Heat the dish on a sand-bath or hot plate until no further fumes are evolved and finally ignite to constant weight at 850 ± 50 °C.

B.4 Calculation

Sulphated residue on ignition, per cent by weight

$$= \frac{W_2 \times 100}{d \times V_1}$$

where d = relative density of the sample at room temperature,

V_1 = volume, in millilitres, of sample taken

and W_2 = weight, in grammes, of residue found.

Appendix C Method for the determination of total sulphate content

(For Type 1 acid only)

C.1 Outline of method

The sulphate present is determined nephelometrically using a standard solution of sulphuric acid for reference purposes.

C.2 Apparatus

a) *Microburette*, 5 ml complying with BS 846[1)].

b) *Two similar test tubes*, 125 × 16 mm, complying with BS 3218[2)].

[1)] BS 846, "*Burettes and bulb burettes*".
[2)] BS 3218, "*Test tubes and boiling tubes*".

C.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Sodium carbonate*, approximately N solution.

b) *Hydrochloric acid*, approximately N solution.

c) Sulphuric acid, 0.002N solution.

d) *Sulphate seeding reagent*. Dissolve 2 g of barium chloride in 75 ml of water, add 20 ml of 95 per cent (v/v) ethanol[3)] and 5 ml of the 0.002N sulphuric acid solution. This reagent should be prepared fresh daily.

C.4 Procedure

Measure 2.0 ml of the sample into a small porcelain or silica basin, add 0.2 ml of the N sodium carbonate solution and evaporate to dryness at 120 °C. Dissolve the residue in 10 ml of hot water and add 1 ml of the N hydrochloric acid solution. Filter if necessary, allow to cool to room temperature, add 1 ml of the sulphate seeding reagent, transfer to one of the two similar test tubes and dilute with water to 20 ml. Prepare a blank test in the second test tube from 0.2 ml of the N sodium carbonate solution, 1.2 ml of the N hydrochloric acid solution and 1 ml of the sulphate seeding reagent in 15 ml of water. Match the turbidity of the sample solution with the 0.002N sulphuric acid solution added from the microburette and carrying out the final match at equal height of liquid in the two tubes. (The matching may conveniently be made using the method depicted in Figure 2.)

If more than 5 ml of 0.002N sulphuric acid solution is required to match the turbidity of the sample solution, repeat the test using a smaller volume of the sample.

C.5 Calculation

Total sulphate content, calculated as sulphuric acid, H_2SO_4,

$$\text{Parts per million} = \frac{98 \times T_1}{d \times V_2}$$

where d = relative density of the sample at room temperature,

T_1 = volume, in millilitres, of 0.002N sulphuric acid solution used

and V_2 = volume, in millilitres, of sample taken.

Appendix D Method for the determination of total sulphate content

(For Type 2 acid only)

D.1 Outline of method

The sulphate present is precipitated as barium sulphate and determined gravimetrically.

D.2 Apparatus

Sintered glass or porous porcelain crucible, of porosity grade No. 4[4)].

D.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Sodium chloride.*

b) *Hydrochloric acid*, concentrated, d = 1.18.

c) *Barium chloride*, 10 per cent (w/v) solution.

D.4 Procedure

Transfer a suitable quantity of the sample (10 ml to 100 ml of sample according to the sulphate content) to a 300 ml squat-form beaker, add 1 g of sodium chloride and evaporate just to dryness. Moisten the residue with 1 ml of concentrated hydrochloric acid and add 200 ml of boiling water. Filter, if necessary.

[3)] Ethanol may be replaced by methanol complying with BS 506, "*Methanol*".
[4)] BS 1752, *"Laboratory sintered or fritted filters"*.

To the boiling solution add dropwise 10 ml of the barium chloride solution with constant stirring. Cover the beaker and allow to stand in a warm place for four hours.

Filter through the tared sintered glass or porous porcelain crucible, wash the precipitate with hot water until the washings are free from chloride, dry at 150 °C. Allow to cool in a desiccator and weigh.

At the same time carry out a blank test on the reagents alone.

D.5 Calculation

Total sulphate content, calculated as sulphuric acid, H_2SO_4,

$$\text{per cent by weight} = \frac{42.02 \times W_3}{V_3 \times d}$$

where d = relative density of the sample at room temperature,

V_3 = volume, in millilitres, of sample taken

and W_3 = weight, in grammes, of the precipitate.

Appendix E Method for the determination of iron content

E.1 Outline of method

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

E.2 Apparatus

a) *Photoelectric absorptiometer or Spectrophotometer* with 4 cm cells.

Alternatively Nessler cylinders, complying with BS 612[5].

b) *Twelve one-mark volumetric flasks*[6], 100 ml capacity.

c) *One-mark volumetric flask*[6], 250 ml capacity.

E.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Hydrochloric acid,* concentrated, d = 1.18.

b) *Hydrochloric acid,* approximately N solution.

c) *Hydroxyammonium chloride,* 10 per cent (w/v) solution.

d) *Ammonium acetate,* 20 per cent (w/v) solution.

e) 2,2′-*bipyridyl*, 0.1 per cent (w/v) solution. Dissolve 0.1 g of the reagent in 50 ml of water containing 2 ml of N hydrochloric acid and dilute to 100 ml.

f) *Standard iron solution*. Dissolve 7.022 g of ammonium ferrous sulphate in a mixture of 600 ml of water and 350 ml of concentrated sulphuric acid, d = 1.84. Dilute to 1 000 ml with water and further dilute 10 ml of the solution so obtained to 1 000 ml with water. 1 ml of the resulting solution contains 10 µg of iron.

E.4 Procedure

a) *Preparation of colour standards.* Into eleven of the 100 ml one-mark volumetric flasks, each containing 50 ml of water and 2 ml of the N hydrochloric acid solution, transfer amounts of the standard iron solution, containing from 0 to 100 µg of iron increasing by stages of 10 µg and treat each solution in the following manner:

Add 2 ml of the hydroxyammonium chloride solution and allow to stand for one minute. Add 10 ml of the ammonium acetate solution and 3 ml of the 2,2′-bipyridyl solution. Dilute the contents of each flask to 100 ml and thoroughly mix.

These standards are used directly for visual comparison. If an instrument is to be used measure the optical density of each solution at a wavelength of 515 mµ and prepare a calibration chart.

[5] BS 612, "*Nessler cylinders*".
[6] BS 1792, "*One-mark volumetric flasks*".

b) *Determination.* Measure 50 ml of the sample into a silica basin and evaporate to dryness on a water bath. Dissolve the residue in 2 ml of the concentrated hydrochloric acid and 5 ml of water. Evaporate again to dryness on the water bath and redissolve in 2 ml of concentrated hydrochloric acid and 25 ml of water.

Cool the solution, transfer to the 250 ml one-mark volumetric flask and dilute with water to the mark. Pipette 50 ml (for Type 1 acid) or 10 ml (for Type 2 acid) of this solution into a 100 ml one-mark volumetric flask, add 2 ml of the hydroxyammonium chloride solution and allow to stand for one minute. Add 10 ml of the ammonium acetate solution, mix, and add 3 ml of the 2,2′-bipyridyl solution. Dilute to 100 ml with water and mix thoroughly.

At the same time carry out a blank test on the reagents alone.

Measure the optical density of the solution at a wavelength of 515 mμ and read the amount of iron present from the calibration chart [see a) above].

Alternatively compare the colour of the solution with the series of prepared colour standards in matched Nessler cylinders, noting the iron content of the standard that most nearly matches the test solution.

E.5 Calculation

Iron content, calculated as Fe, parts per million by weight

$$= \frac{5.0 \times W_4}{V_4 \times d}$$

where d = relative density of the sample at room temperature,

V_4 = volume, in millilitres of the aliquot taken

and W_4 = weight, in microgrammes, of iron found.

Appendix F Method for the determination of arsenic content

F.1 Outline of method

The arsenic present is reduced to arsine which, in contact with mercuric bromide paper, gives a coloured stain ranging from yellow to orange or brown. The colour is compared with a series of stains prepared from solutions containing known amounts of arsenic. The method may be used in the range 1 μg to 10 μg of arsenic.

F.2 Apparatus

The assembly depicted diagrammatically in Figure 2 and consisting of:

a) *Conical flask,* 250 ml capacity, of borosilicate glass, to which are connected, by means of ground joints, b) and c) in series.

b) *Tube for the removal of hydrogen sulphide* with an internal diameter of 12 mm and a height of 70 mm above a bulb of 20 mm diameter. In the bottom of the bulb is placed a thin layer of the dry glass wool, mixed, to the extent of one third, with lead acetate cotton wool pellets, followed by another light layer of glass wool, and lastly strips of damp lead acetate paper.

c) *Glass tube,* with an internal diameter of 3 mm and 120 mm in length with a slight constriction at the bottom end. In this tube is placed a mercuric bromide paper [see reagent g) below].

d) *Burette,* Class A, 10 ml capacity, complying with BS 846[7].

F.3 Reagents

The reagents used shall be of a recognized analytical reagent quality selected for arsenic testing. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Lead acetate cotton wool pellets.* Soak pellets of absorbent cotton wool of a diameter of 5–6 mm in a 5 per cent (w/v) neutral solution of lead acetate, then drain and lightly press.

b) *Lead acetate paper.* Immerse strips of filter paper[8] 8 × 50 mm in a 1 per cent (w/v) neutral solution of lead acetate, drain, and press them lightly between two or three sheets of filter paper.

[7] BS 846, "*Burettes and bulb burettes*".
[8] A Whatman No. 40 or Green's 401 paper is suitable.

c) *Acid sodium chloride solution.* Mix one volume of concentrated sulphuric acid (d = 1.84) with four volumes of water, then dissolve 100 g of sodium chloride in 1 000 ml of the dilute sulphuric acid thus obtained.

d) *Ammonium ferric sulphate solution.* Dissolve in water 84 g of ammonium ferric sulphate $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$; add 10 ml of the acid sodium chloride solution and dilute to 1 000 ml with water.

e) *Stannous chloride solution.* Dissolve 22.6 g of stannous chloride, $SnCl_2.2H_2O$, in 56 ml of the acid sodium chloride solution and dilute to 1 000 ml with water. Keep the solution in a bottle of amber coloured glass in the presence of a few pieces of pure tin.

f) *Zinc,* granulated, in pieces of 4 mm to 5 mm diameter. The zinc weighed for the experiment should be washed at the time of use with the acid sodium chloride solution and then with water.

g) *Mercuric bromide paper.* Immerse sheets of consistent, fine grained filter paper[9] (weighing 8–12 mg/cm^2), for one hour in a 5 per cent (w/v) ethanolic[10] solution of mercuric bromide. Then dry the well drained and pressed paper in a desiccator over anhydrous calcium chloride.

Cut, from the dried sheets, strips exactly 2.5 × 120 mm. Keep these in the dark in a ground-glass stoppered container. Handle the papers only with tweezers and take care not to crease them.

(Unsensitized strips, 2.5 × 120 mm, are available commercially.)

h) *Potassium chlorate.*

j) *Sulphuric acid,* concentrated, d = 1.84.

k) *Standard arsenic solution.* Dissolve 0.132 g of arsenous oxide in 20 ml of 35 per cent (w/v) sodium hydroxide solution. Dilute with about 100 ml of water, add slowly 10 ml of concentrated sulphuric acid (d = 1.84) and dilute to 1 000 ml with water. Further dilute 10 ml of this solution to 1 000 ml. 1 ml of the diluted solution contains 1 µg of arsenic.

F.4 Procedure

a) *Preparation of series of standard stains.* Into the 250 ml conical flask a), add from a micro-burette 1 ml of standard arsenic solution, followed by 50 ml of water, 30 ml of the acid sodium chloride solution, 10 ml of the ammonium ferric sulphate solution and 20 ml of the stannous chloride solution.

Heat to boiling, cool immediately to about 20 °C by placing the flask into cold water. Then place in the flask 10 g of the granulated zinc and connect quickly to the absorption tubes b) and c). (See Figure 2.)

Immerse the flask in water at 20 °C to 25 °C, allow the reaction to proceed for one hour, and then withdraw the mercuric bromide paper. Preserve it in darkness in a sealed container. In this way the colour obtained may be preserved for several months.

This standard stain corresponds to 1 µg of arsenic.

Repeat the above procedure using, instead of 1 ml respectively 2, 4, 6, 8 and 10 ml of the standard arsenic solution to prepare standard stains corresponding respectively to 2, 4, 6, 8 and 10 µg of arsenic.

b) *Determination.* Measure 5.0 ml of the sample into a 250 ml beaker. Add a few crystals of potassium chlorate, 5 ml of concentrated sulphuric acid. Evaporate the solution on a sand bath until white fumes are evolved. Cool and transfer the solution carefully to the 250 ml conical flask a), using 10 ml of water; add 30 ml of the acid sodium chloride solution, 10 ml of the ammonium ferric sulphate solution and 20 ml of the stannous chloride solution.

Heat to boiling, cool immediately to about 20 °C by immersing the flask in cold water. Then place in the flask 10 g of the granulated zinc and connect quickly to the absorption tubes b) and c) (see Figure 2).

Immerse the flask in water at 20 °C to 25 °C, allow the reaction to proceed for one hour, and then withdraw the mercuric bromide paper. Compare the colour obtained with the series of standard stains prepared from the same batch of reagents and estimate the amount of arsenic present.

If the colour is more intense than any in the range of standard stains, repeat the test using a smaller volume of sample.

[9] A Whatman No. 130 paper is suitable.

[10] Ethanol may be replaced 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 Regulation, 1952 (S.I. 1952, No. 2230).

F.5 Calculation

Arsenic content, calculated as As, parts per million by weight

$$= \frac{W_5}{V_5 \times d}$$

where d = relative density of the sample at room temperature,

V_5 = volume, in millilitres, of sample used in the determination

and W_5 = weight, in microgrammes, of arsenic corresponding to the standard stain most closely matching the colour produced in the determination.

Appendix G Method for the determination of lead content

G.1 Outline of method

The lead present is extracted with dithizone solution and determined spectrophotometrically. The method may be used in the range 10 μg to 100 μg of lead.

G.2 Apparatus

All glassware shall be of borosilicate glass or other glass free from lead. Reagent bottles for aqueous solutions shall be of borosilicate glass or polythene[11)].

a) *Photoelectric absorptiometer* or *spectrophotometer* with 4 cm cells.

b) *Twelve one-mark volumetric flasks*[12)], 50 ml capacity.

G.3 Reagents

The reagents used shall be of a recognized analytical reagent quality selected for lead testing and all solutions shall be freshly prepared. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Chloroform,* redistilled.

b) *Ammonium citrate,* 10 per cent (w/v) solution.

c) *Potassium cyanide,* 5 per cent (w/v) solution.

d) *Hydroxyammonium chloride,* 10 per cent (w/v) solution.

e) *Ammonia,* approximately 5N solution.

NOTE It is essential that this solution be freshly prepared if a large blank correction is to be avoided, since dilute ammonia dissolves lead from glass much more readily than a strong solution.

f) *Dithizone solution.* Dissolve 1 g of reagent in 75 ml of chloroform and filter into a 250 ml separating funnel. Shake with four successive 100 ml portions of approximately 0.2N ammonia solution. Reject the chloroform layer. Filter the combined orange coloured extracts into a litre beaker and precipitate the dithizone by rendering the solution slightly acid with saturated SO_2 water. After settling, filter off the product on a sintered glass crucible and wash free from acid with water. Dry the well drained precipitate over concentrated sulphuric acid (d = 1.84) in vacuo for 3 to 4 days, in the dark. Crush the dried solid rapidly and lightly and transfer immediately to a small amber coloured bottle. The purified solid is stable for at least six months when stored in the dark.

Prepare a 0.0025 per cent (w/v) solution of dithizone in redistilled chloroform and transfer it to a dry stoppered amber coloured bottle.

g) *Extraction solution,* 20 ml of 5 per cent (w/v) potassium cyanide solution, and 10 ml of ammonia solution (d = 0.88) in 1 000 ml of water.

h) *Standard lead solution.* Dissolve 1.6 g of lead nitrate in water, add 1 ml of concentrated nitric acid (d = 1.42) and dilute to 1 000 ml. When required for use, take 10.0 ml of this solution, add 1 ml of concentrated nitric acid (d = 1.42) and dilute to 1 000 ml. 1 ml of the diluted solution contains 10 μg of lead.

j) *Narrow range indicator papers,* to include the pH range 8.5 to 10.0[13)].

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11) It should be noted that the name "polythene" is equivalent to the name "polyethylene".
12) BS 1792, "*One-mark volumetric flasks*".
13) Defined as in BS 1647, "*pH scale*".

G.4 Procedure

Dithizonates are particularly sensitive to ultra-violet light and shall be shielded from direct sunlight or from fluorescent lighting in the laboratory.

a) *Preparation of colour standards.* Into eleven 100 ml separating funnels, each containing 10 ml of water and 0.5 ml of concentrated hydrochloric acid, transfer known amounts of the standard lead solution, containing 0 to 100 µg of lead increasing by stages of 10 µg and treat each solution in the following manner:

Add 1 ml of the hydroxyammonium chloride solution and 10 ml of the ammonium citrate solution and adjust the pH of the solution to between 8.5 and 10.0 by addition of the ammonia solution, using the narrow range indicator papers externally. Add 2 ml of the potassium cyanide solution and extract with 5 ml portions of the dithizone solution until the green colour is unchanged. Combine the extracts and remove the excess dithizone by extracting with the minimum quantity of the extraction solution in 5 ml portions until the colour of the chloroform solution is a clear pink. Filter the chloroform solution through a dry acid-washed filter paper[14)] into a 50 ml one-mark volumetric flask and dilute to the mark with chloroform.

Measure the optical density of each solution at a wavelength of 520 mµ and prepare a calibration chart.

b) *Determination.* Measure 50 ml of the sample into a silica dish and evaporate almost to dryness. (The amount of sample may be reduced to give a colour intensity suitable for measurement.) Wash the residue into a 100 ml separating funnel with water. Add 1 ml of the hydroxyammonium chloride solution and 10 ml of the ammonium citrate solution and adjust the pH of the solution to between 8.5 and 10.0 by addition of the ammonia solution, using the narrow range indicator papers externally. Add 2 ml of the potassium cyanide solution, and extract with 5 ml portions of dithizone solution until the green colour is unchanged. Combine the extracts and remove the excess dithizone by extracting with the minimum quantity of the extraction solution in 5 ml portions until the colour of the chloroform solution is clear pink. Filter the chloroform solution through a dry acid washed filter paper[14)] into a 50 ml one-mark volumetric flask and dilute to the mark with chloroform.

At the same time carry out a blank test on the reagents alone.

Measure the optical density of the solution at a wavelength of 520 mµ and read the amount of lead present from the calibration chart (see above).

G.5 Calculation

Lead content, calculated as Pb, parts per million by weight

$$= \frac{W_6}{V_6 \times d}$$

where d = relative density of the sample at the room temperature,

V_6 = volume, in millilitres, of sample taken

and W_6 = weight, in microgrammes, of lead found.

Appendix H Method for the determination of copper content

H.1 Outline of method

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 measured spectrophotometrically or visually. The method may be used in the range 10 µg to 100 µg of copper.

H.2 Apparatus

a) *Photoelectric absorptiometer, or Spectrophotometer* with 4 cm cells, Alternatively Nessler cylinders, complying with BS 612[15)].

b) *Seven one-mark volumetric flasks*[16)], 50 ml capacity.

[14)] A Whatman No. 42 paper is suitable.
[15)] BS 612, "*Nessler cylinders*".
[16)] BS 1792, "*One-mark volumetric flasks*".

H.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Sodium sulphate,* anhydrous.

b) *Amyl alcohol.*

c) *Hydrochloric acid,* N solution.

d) *Hydrochloric acid* concentrated, $d = 1.18$.

e) *Tartaric acid,* 50 per cent (w/v) aqueous solution.

f) *Sodium hydroxide,* 20 per cent (w/v) aqueous solution.

g) *Ascorbic acid,* 10 per cent (w/v) aqueous solution, freshly prepared.

h) 2,2′-*biquinolyl,* 0.05 per cent solution. Dissolve 0.25 g of 2,2′-biquinolyl in amyl alcohol and dilute with amyl alcohol to 500 ml.

j) *Standard copper solution.* Dissolve 0.3928 g of copper sulphate pentahydrate in water, add 25 ml of 6N sulphuric acid, dilute to 1 000 ml and further dilute 10 ml of the solution to 100 ml. 1 ml of the diluted solution contains 10 µg of copper.

k) *Narrow range indicator papers,* covering the pH range 5.5–7.0[17)].

H.4 Procedure

a) *Preparation of colour standards,* into six 100 ml beakers, transfer known amounts of the standard copper solution containing 0, 20, 40, 60, 80 and 100 µg of copper and treat each solution in the following manner:

Dilute to approximately 30 ml with water and transfer to a 500 ml stoppered separating funnel. Dilute with water to approximately 400 ml, and add 2 ml of the tartaric acid solution. Adjust the pH of the solution to 6.0 approximately by addition of the sodium hydroxide solution, using a narrow range indicator paper externally. Add 2 ml of the ascorbic acid solution, shake to mix thoroughly and allow to stand for five minutes. Add 10 ml of the 2,2′-biquinolyl solution, shake well for two minutes. Extract the copper complex with two 20 ml portions of amyl alcohol, and transfer the extracts to a 100 ml beaker. Add about 2 g of anhydrous sodium sulphate to the combined extracts and stir thoroughly to remove traces of water. Filter into a 50 ml one-mark volumetric flask; wash the sodium sulphate crystals twice with 2 ml portions of amyl alcohol. Transfer the washings to the flask and dilute the solution to the mark with amyl alcohol.

These standards are used directly for visual comparison. If an instrument is to be used measure the optical density of each solution at a wavelength of 545 mµ and prepare a calibration chart.

b) *Determination.* Measure 20.0 ml (for Type 1 acid) or 10.0 ml (for Type 2 acid) of the sample into a silica dish and evaporate just to dryness. Cool and redissolve the residue in 2 ml of the N hydrochloric acid solution.

Transfer the solution to a 500 ml stoppered separating funnel. Add 1 ml of the concentrated hydrochloric acid, dilute the solution to 400 ml with water, add 2 ml of the tartaric acid solution and adjust the pH to 6.0 approximately by addition of the sodium hydroxide solution, using a narrow range indicator paper externally. Add 2 ml of the ascorbic acid solution, shake to mix thoroughly and allow to stand for five minutes. Add 10 ml of the 2,2′-biquinolyl solution and shake well for two minutes. Extract the copper complex with two 20 ml portions of amyl alcohol, and transfer the extracts to a 100 ml beaker. Add about 2 g of anhydrous sodium sulphate to the combined extracts and stir thoroughly to remove traces of water. Filter into a 50 ml one-mark volumetric flask, wash the sodium sulphate crystals twice with 2 ml portions of amyl alcohol. Transfer the washings to the flask and dilute the solution to the mark with amyl alcohol.

At the same time carry out a blank test on the reagents alone.

Measure the optical density of the solution at a wavelength of 545 mµ and read the amount of copper present from the calibration chart [see a) above].

Alternatively compare the colour of the solution with the series of prepared colour standards in matched Nessler cylinders, noting the copper content of the standard that most nearly matches the test solution.

17) Defined as in BS 1647, "*pH scale*".

H.5 Calculation

Copper content, calculated as Cu, parts per million by weight

$$= \frac{W_7}{V_7 \times d}$$

where d = relative density of the sample at room temperature,

V_7 = volume, in millilitres, of sample taken

and W_7 = weight, in microgrammes, of copper found.

Appendix J Method for the determination of mercury content

J.1 Outline of method

The mercury present is extracted with dithizone solution and determined spectrophotometrically at a wavelength of 605 mμ using a reversion technique. The mercury is then determined from the difference between the optical densities measured before and after the reversion of the mercury dithizone complex.

J.2 Apparatus

a) *Photoelectric absorptiometer or spectrophotometer* with 1 cm cells.

b) *One-mark volumetric flask*[18)], 500 ml capacity.

c) *One-mark volumetric flask*[18)], 1 000 ml capacity.

J.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Chloroform,* redistilled.

b) *Ammonia, solution, d* = 0.88, free from mercury.

c) *Hydrochloric acid,* concentrated, *d* = 1.18, free from mercury.

d) *Sulphuric acid,* approximately 18N solution.

e) *Sulphuric acid,* approximately N solution.

f) *Potassium permanganate,* saturated solution.

g) *Hydroxyammonium chloride solution.* Dissolve 25 g of hydroxyammonium chloride in about 60 ml of water, add 0.2 ml of phenol red indicator solution, and make alkaline with ammonia solution to the full red colour of the indicator. Cool and extract with a 0.01 per cent (w/v) solution of dithizone in chloroform, using 5 ml portions, until the last extract remains green; then wash the solution free from excess of dithizone by repeated extraction with 10 ml portions of chloroform. Warm the solution until the excess of chloroform has been removed, cool, filter and dilute to 250 ml.

h) *Dithizone solution.* Dissolve 1 g of reagent in 75 ml of chloroform and filter into a 250 ml separating funnel. Shake with four successive 100 ml portions of the ammonia solution. Reject the chloroform layer. Filter the combined orange coloured extracts into a litre beaker and precipitate the dithizone by rendering the solution slightly acid with saturated SO_2 water. After settling, filter off the product on a sintered glass crucible and wash free from acid with water. Dry the well drained precipitate over concentrated sulphuric acid (*d* = 1.84) *in vacuo* for 3 or 4 days, in the dark. Crush the dried solid rapidly and lightly and transfer immediately to a small amber coloured bottle. The purified solid is stable for at least six months when stored in the dark, preferably in a refrigerator.

Prepare a 0.025 per cent (w/v) stock solution of dithizone in redistilled chloroform and transfer it to a dry stoppered amber coloured glass bottle. Store in a cool dark place, preferably in a refrigerator. Dithizone solutions have a limited life and should only be stored for a limited period.

Immediately before use, prepare a 0.0006 per cent (w/v) solution by diluting 24 ml of the stock solution to 100 ml with redistilled chloroform. Check the strength of this solution by measuring the optical density at a wavelength of 605 mμ using 1 cm cells. The reading should be between 0.85 and 1.05. If it is outside these limits, adjust the strength of the solution by addition of chloroform or of stronger dithizone solution as required.

[18)] BS 1792, "*One-mark volumetric flasks*".

j) *Reversion solution.* Dissolve 10.2 g of potassium hydrogen phthalate and 30 g of potassium iodide in water and dilute to 500 ml. Store the solution in an amber coloured glass bottle. Before use, reduce any iodine liberated by adding sodium thiosulphate solution until just colourless.

k) *Standard mercury solution.* Weigh, to the nearest milligramme, 0.4 g to 0.5 g of pure dry mercury into a 100 ml beaker, add 10 ml of water, cover the beaker with a watchglass and gradually add 10 ml of concentrated nitric acid (d = 1.42); warm the mixture until the mercury is completely dissolved. Add 25 ml of the 18N sulphuric acid solution, and evaporate the solution until white fumes of sulphur trioxide are evolved. Cool the solution and dilute it cautiously with 50 ml of water, boil for 1 minute and then cool. Transfer the solution to the 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

When required for use, measure into a 1 000 ml one-mark volumetric flask, the volume of the mercury solution that contains 10 mg of mercury, add 5 ml of the 18N sulphuric acid solution, dilute the solution to the mark with water and mix.

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

l) *Narrow range indicator papers*, covering the pH range 5.5 to 7.0[19].

J.4 Procedure

Dithizonates are particularly sensitive to ultra-violet light and shall be shielded from direct sunlight and from fluorescent light in the laboratory.

a) *Preparation of colour standards*. Into each of nine 250 ml conical flasks measure 10 ml of the mercury-free hydrochloric acid and amounts of the standard mercury solution containing from 0 µg to 40 µg of mercury increasing by stages of 5 µg. Treat each in the following manner:

Dilute to 50 ml with water, and, while keeping the solution cool, neutralize with the ammonia solution to pH 7[19] using the narrow range indicator papers. Add 10 ml of the N sulphuric acid solution, add the saturated potassium permanganate solution dropwise until a pink colour persists for one minute. Add 5 ml of the hydroxyammonium chloride solution, mix and stand until the pink colour is destroyed.

If the solution is not clear, filter through a dry, acid-washed filter paper[20] and wash the paper with water, keeping the total volume below 100 ml.

Transfer to a 250 ml separating funnel and dilute to about 100 ml. Add 2 ml of redistilled chloroform, shake vigorously, allow the layers to separate and reject the chloroform layer. Add 20.0 ml of the 0.0006 per cent (w/v) dithizone solution from a burette, shake for one minute and allow the layers to separate. If the chloroform layer is now orange, add a further 20.0 ml of the 0.0006 per cent (w/v) dithizone solution and extract again. Allow the layers to separate, run off sufficient of the chloroform layer through a dry acid-washed filter paper[20] to fill a 1 cm cell. Transfer the remaining chloroform layer to a second separating funnel containing 20 ml of the reversion solution. Shake for one minute, allow the layers to separate. Run off the lower layer through a dry acid-washed filter paper[20] into another 1 cm cell.

Measure the optical density of both solutions against redistilled chloroform in the reference cell, at a wavelength of 605 mµ.

For each colour standard calculate the optical density difference, by subtracting the reading obtained on the solution from that obtained on the reverted solution. Prepare a calibration chart by plotting the weight, in microgrammes, of mercury against the optical density difference.

[19] Defined as in BS 1647, *"pH scale"*.
[20] A Whatman No. 31 paper is suitable.

b) *Determination.* Measure 10 ml of the sample into a 250 ml conical flask and dilute to 50 ml with water. While keeping the solution cool, neutralize with the ammonia solution to pH 7[21] using the narrow range indicator papers. Add 10 ml of the N sulphuric acid solution, add the saturated potassium permanganate solution dropwise until a pink colour persists for one minute. Add 5 ml of the hydroxyammonium chloride solution mix and stand until the pink colour is destroyed.

If the solution is not clear, filter through a dry acid-washed filter paper[22] and wash the paper with water, keeping the total volume below 100 ml.

Transfer to a 250 ml separating funnel and dilute to about 100 ml. Add 2 ml of redistilled chloroform, shake vigorously, allow the layers to separate and reject the chloroform layer. Add 20.0 ml of the 0.0006 per cent (w/v) dithizone solution from a burette, shake for one minute and allow the layers to separate. If the chloroform layer is now orange, add a further 20.0 ml of the 0.0006 per cent (w/v) dithizone solution and extract again. Allow the layers to separate, run off sufficient of the chloroform layer, through a dry acid-washed filter paper[22], to fill a 1 cm cell. Transfer the remaining chloroform layer to a second separating funnel containing 20 ml of the reversion solution. Shake for one minute, allow the layers to separate. Run off the lower layer through a dry acid-washed filter paper[22] into another 1 cm cell.

Measure the optical density of both solutions against redistilled chloroform in the reference cell at a wavelength of 605 mμ and read the amount of mercury present from the calibration chart [see a) above].

If more than one 20.0 ml portion of the 0.0006 per cent (w/v) dithizone solution was used, multiply the amount of mercury found by the number of extractions.

Calculation

Mercury content, calculated as Hg, parts per million by weight $= \frac{0.1 W_8}{d}$

where d = relative density of the sample at room temperature

and W_8 = weight, in microgrammes, of mercury found.

Appendix K Method for the determination of oxidizing and reducing substances

K.1 Outline of method

Oxidizing or reducing substances present are determined iodometrically using sodium thiosulphate or potassium iodate as appropriate.

K.2 Reagents

The reagents used shall be of analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used throughout.

a) *Hydrochloric acid,* concentrated, d = 1.18.

b) *Cadmium iodide,* 5 per cent (w/v) solution.

c) *Iodine,* 0.01N solution.

d) *Sodium thiosulphate,* 0.01N solution, freshly prepared.

e) *Potassium iodate,* 0.01N solution.

f) *Starch indicator solution,* 0.25 per cent (w/v) aqueous solution of soluble starch.

K.3 Procedure

To 1 800 ml of water in a 2 litre beaker add 1 ml of concentrated hydrochloric acid, 2 ml of the cadmium iodide solution and 20 ml of the starch indicator solution. Titrate with the 0.01N iodine solution until the first trace of a permanent blue colour is seen in the liquid by transverse illumination against a white background.

[21] Defined as in BS 1647, "*pH scale*".
[22] A Whatman No. 31 paper is suitable.

Transfer 900 ml of the prepared solution to a 1 litre beaker, add 100 ml of the sample, mix and allow to stand for two minutes. If, at the end of this period the blue colour remains, titrate with the 0.01N sodium thiosulphate solution until the colour of the solution matches that of the remainder of the prepared solution, used as a blank, in a similar litre beaker.

If the blue colour formed on addition of the sample disappears, titrate instead with the 0.01N potassium iodate solution until the colour of the solution again matches that of the remainder of the blank in a similar 1 litre beaker.

K.4 Calculation

Oxidizing substances, calculated as chlorine, Cl_2,

$$\text{parts per million by weight} = \frac{3.55 \times T_2}{d}$$

Reducing substances, calculated as sulphur dioxide, SO_2,

$$\text{parts per million} = \frac{3.20 \times T_3}{d}$$

where d = relative density of the sample at room temperature,

T_2 = volume, in millilitres, of 0.01N sodium thiosulphate solution used

and T_3 = volume, in millilitres, of 0.01N potassium iodate solution used.

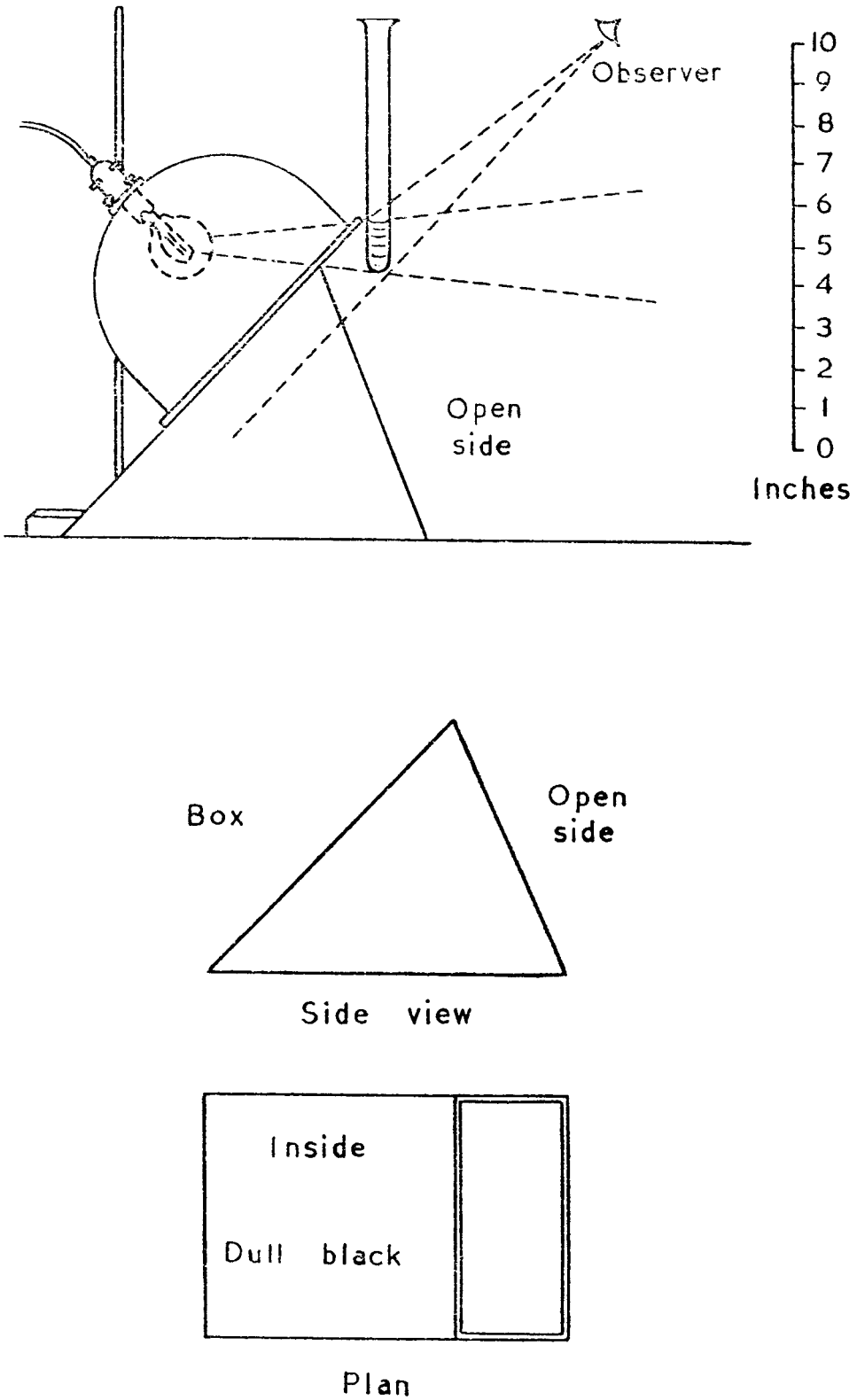

Figure 1 — Convenient form of apparatus for the determination of minute quantities of sulphate (Appendix C)

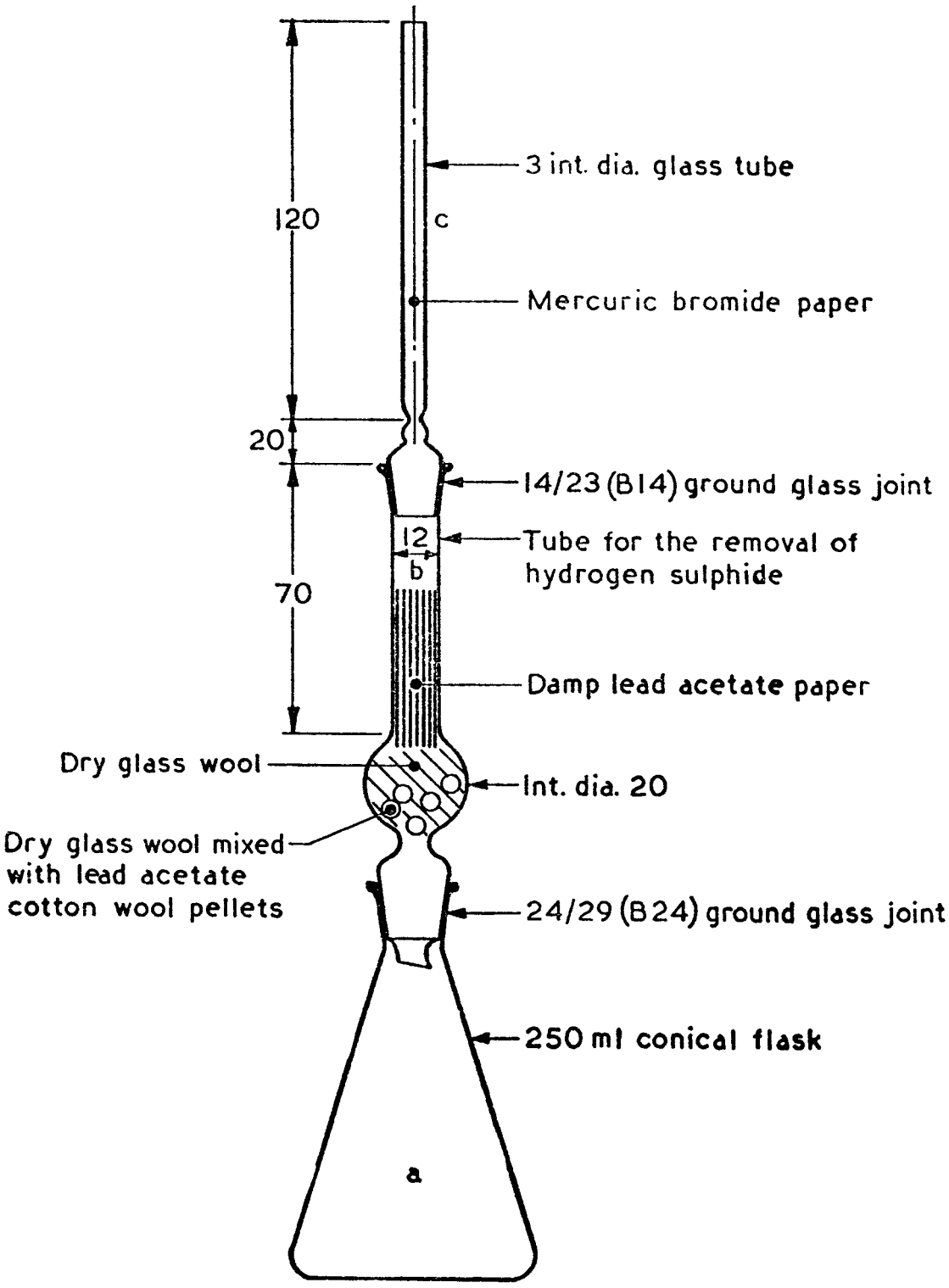

Figure 2 — Apparatus for use in the determination of arsenic
(All dimensions are in millimetres)

blank

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