BRITISH STANDARD

BS 5962:1980

Specifications for

Naphthalene (including test methods)

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Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

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

The organizations marked with an asterisk in the above list were directly represented on the Technical Committee entrusted with the preparation of this British Standard.

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board and comes into effect on 30 September 1980

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

Foreword

This British Standard was prepared under the direction of the Chemicals Standards Committee in order to provide specifications and methods of test for naphthalene for industrial purposes.

In the drafting of this standard, account has been taken of specifications used by the Ministry of Defence.

By agreement with the Standardization of Tar Products Tests Committee (STPTC), some of the methods of testing and descriptions of apparatus contained in the appendices have been reproduced with editorial modifications from the publication "Standard methods of testing tar and its products" (6th edition 1967)[1].

NOTE The following definitions have been approved by the STPTC and have been adopted for the purposes of this British Standard:

accuracy, for statistical purposes, is the closeness of the test results to the true value. Variations due to sampling errors are included and these are often responsible for most of the difference between the test result and the true value. No estimates of the accuracy of the test methods are given.

precision is the closeness of agreement among test results obtained under any definite test conditions. Thus, a method may be precise without being accurate. Repeatability and reproducibility are aspects of precision under two such defined sets of conditions. (See also BS 4306.)

repeatability is a measure of the precision of test results obtained by one operator using one set of apparatus. Repeatability (r) is defined as the difference between duplicate results on the same sample that would be equalled or exceeded, in the long run, in only one case in twenty.

reproducibility is a measure of the precision of test results obtained by different operators using different sets of the same apparatus. Reproducibility (R) is defined as the difference between a single result by one operator at one laboratory and a single result by another operator at another laboratory that would be equalled or exceeded, in the long run, in only one case in twenty.

A number of industries, e.g. the petroleum industry, use methods of test that are also applicable to naphthalene and, wherever appropriate, these methods have been adopted.

For routine purposes, methods of test alternative to those given in this British Standard may be used provided that they give results of sufficient accuracy. However, in the event of doubt or dispute, only the specified methods of test should be used.

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 12, an inside back cover and a back cover.

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

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[1] These methods are intended to be carried out in accordance with the general principles and apparatus requirements laid down in the STPTC publication mentioned.

1 Scope

This British Standard specifies requirements and methods of test for three grades of naphthalene for industrial purposes. It is applicable to materials that consist essentially of naphthalene, $C_{10}H_8$.

NOTE For the purposes of this British Standard, the word "essentially" is understood to mean "at least 94.6 %". See the footnotes to Table 1, Table 2 and Table 3, which give the percentages by mass of naphthalene corresponding to the specified corrected wet crystallizing points.

2 References

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

3 Requirements

The material shall comply with the requirements specified in Table 1, Table 2 or Table 3, as appropriate.

NOTE Solid naphthalene is available in a variety of physical forms such as flake, balls, eggs, etc. Users of this standard are advised to specify the form in which the material is to be supplied.

4 Sampling

A representative sample of not less than 1 000 g shall be taken from the bulk of the material to provide three separate laboratory samples.

Recommended methods of sampling are given in Parts 1, 3 and 4 of BS 5309.

Table 1 — Naphthalene, grade 1 (designated BS 5962-1)

Characteristic	Requirement	Test method
Appearance (colour)	White or faintly yellow and free from foreign matter or visible impurities	Visual inspection
Physical state	Solid or liquid	Visual inspection
Matter non-volatile at 250 °C	Not greater than 0.1 % by mass	Appendix A
Water content	Not greater than 0.2 % by mass	BS 4385
Corrected wet crystallizing point	Not lower than 79.6 °C[a]	Appendix B
Total sulphur content	Not greater than 0.3 % by mass	Appendix E
Ash (residue after ignition)	Not greater than 0.1 % by mass	Appendix C
Sulphuric acid test (acid wash test)	Not deeper than the standard colour solution	Appendix D

[a] Corresponding to 98.75 % (*m*/*m*) of naphthalene.

Table 2 — Naphthalene, grade 2 (designated BS 5962-2)

Characteristic	Requirement	Test method
Physical state	Solid or liquid	Visual inspection
Matter non-volatile at 250 °C	Not greater than 0.3 % by mass	Appendix A
Water content	Not greater than 1 % by mass	BS 4385
Corrected wet crystallizing point	Not lower than 78.5 °C[a] and not higher than 79.6 °C	Appendix B
Total sulphur content	Not greater than 1 % by mass	Appendix E
Ash (residue after ignition)	Not greater than 0.1 % by mass	Appendix C

[a] Corresponding to 96.65 % (*m*/*m*) of naphthalene.

Table 3 — Naphthalene, grade 3 (designated BS 5962-3)

Characteristic	Requirement	Test method
Physical state	Solid or liquid	Visual inspection
Matter non-volatile at 250 °C	Not greater than 0.3 % by mass	Appendix A
Water content	Not greater than 1 % by mass	BS 4385
Corrected wet crystallizing point	Not lower than 77.5 °C[a] and not higher than 78.5 °C	Appendix B
Total sulphur content	Not greater than 1 % by mass	Appendix E
Ash (residue after ignition)	Not greater than 0.1 % by mass	Appendix C

[a] Corresponding to 94.64 % (*m*/*m*) of naphthalene.

Appendix A Determination of non-volatile matter

A.1 Principle

Melting and heating a test portion on a heating block for a specified time; cooling and weighing to determine the loss of mass.

A.2 Apparatus

Ordinary laboratory apparatus is required, together with the following.

A.2.1 *Electrically heated metal block*[2], capable of being maintained at 250 ± 5 °C for 1 h and conforming to the dimensions given in Figure 1.

A.2.2 *Petri dish*, complying with the requirements of BS 611, of heat-resisting glass and of the following dimensions:

Height 16 ± 1 mm, diameter 63 ± 1 mm.

A.2.3 *Thermometer*, complying with the requirements of type GP 360C-100 of BS 1704.

A.3 Procedure

A.3.1 *Test sample*. Melt the laboratory sample by immersing the laboratory sample container in a hot water bath. Stir thoroughly. Pour a little more than 5 g of the laboratory sample into a porcelain dish and reseal the laboratory sample container. Allow the test sample in the dish to cool in a dry atmosphere and then crush the solidified material.

A.3.2 *Method*. Weigh, to the nearest 0.01 g, approximately 5 g of the well-mixed test sample into the Petri dish (**A.2.2**), preconditioned at 100 °C to 130 °C for 1 h, allowed to cool in a desiccator and weighed to the nearest 0.1 mg, and place the Petri dish in the well of the heating block (**A.2.1**), maintained at 250 ± 5 °C.

Maintain the block and test portion at this temperature for not less than 30 min and not more than 35 min, remove, cool the dish and contents in a desiccator and then weigh to the nearest 0.1 mg.

A.4 Expression of results

The matter non-volatile at 250 °C, expressed as a percentage by mass, is given by the expression:

$$\frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where

m_0 is the mass of the Petri dish (**A.2.2**) (in g)

m_1 is the mass of the Petri dish and test portion (in g)

m_2 is the mass of the Petri dish and residue (in g)

A.5 Precision (see foreword)

% (m/m) **Non-volatile matter**	**Repeatability,** r	**Reproducibility,** R
0.05	0.025	0.045
0.10	0.035	0.06
0.50	0.07	0.14
1.00	0.10	0.20
1.50	0.13	0.24
2.00	0.15	0.28
3.00	0.18	0.34

Appendix B Determination of crystallizing point

B.0 Introduction

The difference between the crystallizing points of dry naphthalene and of naphthalene containing a small quantity of water is appreciable. The depression of crystallizing point reaches a maximum and remains constant at 0.85 °C when at least 2 % by mass of water is present.

The depression of the crystallizing point is also affected by the presence of phenols. If more than 1 % of phenols is present, the figure of 0.85 °C is invalid.

B.1 Definitions

B.1.1 *uncorrected wet crystallizing point*. The crystallizing point when water has been added in excess of 2 % by mass.

B.1.2 *corrected wet crystallizing point*. The crystallizing point when the correction of 0.85 °C has been added to the uncorrected wet crystallizing point. This corresponds to the crystallizing point of the material on the dry basis.

B.2 Principle

Melting and cooling of a test portion and plotting of a cooling curve.

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[2] Information on supplies of suitable blocks may be obtained on application to the Secretary, Standardization of Tar Products Test Committee, British Carbonization Research Association, Wingerworth, Chesterfield, Derbyshire, S42 6JS.

B.3 Apparatus

B.3.1 *Crystallizing point apparatus*, as illustrated in Figure 2, comprising a glass test tube 150 mm × 25 mm nominal size placed inside a 200 mm × 38 mm test tube which has been cut down to 160 mm × 38 mm. The latter tube is so flanged that it may be supported centrally by a metal cover plate in a 1 000 ml tall-form beaker filled with water to within 20 mm of the top. The wider tube is weighted with lead shot or similar material and the inner tube is closed by means of a cork which carries a glass stirrer and the thermometer (**B.3.2**) through its centre. The stirrer has a loop of outside diameter 18 mm, to surround the thermometer. The thermometer is so fixed in the cork that the bottom of the bulb is about 15 mm from the bottom of the inner tube. A thermometer for the water bath passes through a hole in the cover plate and is held by a rubber ring.

B.3.2 *Thermometer*, complying with the requirements of type A 100C/total of BS 593.

B.3.3 *Pipette*, 1 ml graduated, complying with the requirements of class B of BS 700.

B.4 Procedure

B.4.1 Place about 40 g of the laboratory sample in a conical flask, cork loosely and warm in a water bath until just molten. Warm the inner tube of the crystallizing point apparatus in the water bath, and pipette 0.5 ml of water into the tube. Thoroughly mix the contents of the flask and pour about 20 g into the inner tube. Insert the cork carrying the thermometer and stirrer.

If the test portion has begun to crystallize, heat it gently until it regains complete fluidity and then cool it rapidly to determine the approximate crystallizing point. Warm the tube in the water bath at about 5 °C above this point so that the crystals, except for a trace necessary for seeding, melt.

Replace the tube in its jacket with the water in the beaker maintained at 6 °C to 8 °C below the expected crystallizing point. Stir the contents of the tube continuously and with sufficient vigour to ensure thorough mixture of the sample and water. Record thermometer readings at 30 s intervals when the temperature has fallen to 2 °C above the expected crystallizing point. The uncorrected wet crystallizing point corresponds to the highest of the first five consecutive readings, corrected for thermometer error, during which the temperature remains constant to within 0.05 °C.

If supercooling occurs, as shown by a rise in temperature, observe the constant temperature after the rise. A temperature rise of 1 °C is the maximum allowable. If five consecutive readings within 0.05 °C are not obtained, record six readings commencing with the point at which the maximum temperature is first attained. Plot the complete cooling curve of temperature against time and draw a straight line to lie evenly between the first and second and between the fifth and sixth points mentioned above. Extend this line to meet the section of the cooling curve before the inflexion. Record the temperature reading, adjusted for thermometer error, corresponding to the point of intersection as the uncorrected wet crystallizing point, and then add 0.85 °C to the result to give the corrected wet crystallizing point.

B.5 Precision (see foreword)

The following values are applicable to results on "as received" samples and to uncorrected results on wet samples.

Approximate range of crystallizing point	Repeatability, r	Reproducibility, R
78 °C to 80 °C	0.08 °C	0.15 °C

Appendix C Determination of ash

C.1 Principle

Ignition of a test portion; weighing of the residue.

C.2 Apparatus

Ordinary laboratory apparatus is required, together with the following.

C.2.1 *Crucible*, squat form, of silica or a suitable grade of laboratory porcelain, capacity about 30 ml, complying with the requirements of BS 4244.

C.2.2 *Furnace* capable of being maintained at 650 ± 5 °C.

C.3 Procedure

Heat the crucible for 1 h in the furnace (**C.2.2**), maintained at 650 ± 5 °C, cool in a desiccator and weigh to the nearest 0.0002 g. Weigh, to the nearest 0.002 g, approximately 2 g of the test sample, which should be as finely divided as possible. Heat the crucible and contents in a fume cupboard over a low Bunsen flame, gently at first to remove volatile matter, and then more strongly to complete the incineration. Place the crucible and contents in the furnace (**C.2.2**), maintained at 650 ± 5 °C, for 1 h.

Remove, allow to cool in a desiccator, and weigh to the nearest 0.0002 g. Repeat the operations of heating in the furnace, cooling and weighing until two successive weighings do not differ by more than 0.0001 g.

C.4 Expression of results

The ash, expressed as a percentage by mass, is given by the expression:

$$\frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where

m_0 is the mass of crucible empty (in g)

m_1 is the mass of crucible and test portion (in g)

m_2 is the mass of crucible and residue (in g)

Appendix D Sulphuric acid test

D.1 Principle

Comparison of the depth of colour produced by a test portion in sulphuric acid against a standard colour solution.

D.2 Reagents

Use only recognized analytical grade reagents and water complying with the requirements of BS 3978 or of equivalent purity.

D.2.1 *Stock solutions.* Prepare three stock solutions by dissolving the following salts, each in 1 000 ml of 1 % (*m*/*m*) hydrochloric acid solution:

Red	119.0 g of cobalt (II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$)
Yellow	45.1 g of iron (III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)
Blue	62.4 g of copper (11) sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$)

D.2.2 *Standard colour solution.* Prepare a colour standard solution by mixing the stock solutions (**D.2.1**) in the quantities given below in a Nessler cylinder, 50 ml capacity, complying with the requirements of BS 612:

Red	Yellow	Blue	Water
ml	ml	ml	ml
3.6	0.7	1.5	6.2

D.2.3 *Sulphuric acid,* 98/99 % (*m*/*m*), water white, and free from nitrogen compounds.

D.3 Apparatus

D.3.1 *Two Nessler cylinders*, 50 ml capacity complying with the requirements of BS 612.

D.3.2 *Water bath,* capable of being maintained at 80 ± 1 °C.

D.4 Procedure

Heat 10 ml of the sulphuric acid (**D.2.3**) to 80 ± 1 °C in one of the Nessler cylinders (**D.3.1**) immersed in the water bath (**D.3.2**) maintained at that temperature. Add 2.0 ± 0.1 g of the finely powdered test sample to the acid in the tube, keep it immersed in the water bath and shake it gently for 2 min. Remove the cylinder from the bath, place it in a suitable rack and immediately compare the colour produced with that of the standard colour solution (**D.2.2**), with the eye level with the centre of the liquid columns.

Appendix E Determination of total sulphur content

E.1 Principle

Reduction of the sulphur compounds, by means of Raney nickel, to nickel sulphide, liberation of hydrogen sulphide with hydrochloric acid, absorption in sodium hydroxide solution and titration with standardized mercury (II) acetate solution.

E.2 Interferences

Certain oxygenated sulphur compounds are not completely reduced under the conditions specified and some olefins interfere. This method is applicable to sulphur contents up to 10 000 mg/kg [1 % (*m*/*m*)].

E.3 Reagents and materials

Use only recognized analytical grade reagents and, unless otherwise specified, water complying with the requirements of BS 3978 or of equivalent purity.

E.3.1 *Acetone*

E.3.2 *Nitrogen,* compressed gas.

E.3.3 *Raney nickel alloy*, powder, aluminium 50 % (*m*/*m*), nickel 50 % (*m*/*m*).

E.3.4 *Propan-2-ol*, sulphur free.

E.3.5 *Cleaning solution.* Dissolve 5 g of potassium dichromate in 5 ml of water and add the solution to 100 ml of concentrated nitric acid.

CAUTION. This solution is strongly corrosive and a powerful oxidizing agent. It should be used only with good ventilation and an eye shield should be worn. Rinse any splashes or spillage with a copious supply of water.

E.3.6 *Hydrochloric acid*, 5M reagent solution. For samples containing less than 5 mg/kg of sulphur, use microanalytical reagent grade acid.

E.3.7 *Potassium hydroxide*, 40 g/litre reagent solution is ethanediol.

E.3.8 *Sodium hydroxide,* 2.5M reagent solution. Dissolve 40 g of sodium hydroxide pellets in water in a 500 ml stoppered conical flask marked at the 400 ml level, which has been previously cleaned with the cleaning solution (**E.3.5**), well rinsed with water and the stopper and cone lubricated with silicone grease, swirl gently to assist dissolution, allow to cool and dilute to the 400 ml mark with water. If the reagent is stored in a bottle, the stopper should be lubricated with silicone grease.

E.3.9 *Sodium hydroxide,* M reagent solution. Prepare as in **E.3.8** using 16 g of sodium hydroxide pellets. If the reagent is stored in a bottle, the stopper should be lubricated with silicone grease.

E.3.10 *Mercury (II) acetate*, standard solutions.

E.3.10.1 *Stock solution.* Dissolve 3.375 g of mercury (II) oxide, previously dried at 100 °C, in 200 ml of water and 10 ml of glacial acetic acid (17M) in a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Standardize the solution by transferring 50.0 ml **(CAUTION. Do not use mouth suction)** to a 250 ml conical flask, add 2 ml of concentrated nitric acid (16M) and 2 ml of iron (III) sulphate indicator solution. Prepare this solution by dissolving 35 g of iron (III) sulphate in water, add sufficient concentrated nitric acid to change the colour from red-brown to yellow and dilute to 250 ml with water. Titrate with 0.1M ammonium thiocyanate solution to a permanent reddish-yellow end point. Calculate the factor from the formula:

$$T = V \times \frac{0.1083}{3.375}$$

where

V is the volume of ammonium thiocyanate used (in ml).

E.3.10.2 *Working solution.* Transfer 5.0 ml of the stock solution to a 250 ml one-mark volumetric flask (caution) and dilute to the mark with water.

1 ml of this solution is equivalent to 0.02 µg of sulphur.

E.3.11 *Dithizone*, 1 g/litre indicator solution in acetone. Prepare fresh each day or every third day if stored in a refrigerator.

NOTE When experience has been gained with the amount of indicator required, a few particles of the solid indicator may be added to the absorption tube. This procedure overcomes the problem of the instability of the indicator solution.

E.4 Apparatus

All ground glass cones and sockets shall comply with the requirements of BS 570. PVC or similar sulphur-free plastics tubing shall be used to connect the source of nitrogen. Rubber shall in no circumstances be used.

Ordinary laboratory apparatus is required, together with the following.

E.4.1 *Reduction apparatus* (see Figure 3), which shall comprise the following.

a) *reduction flask,* (A) *100 ml,* with three short necks. One neck shall be upright with a 14/23 or 19/26 ground glass conical socket. At least one of the other necks shall be set at such an angle that its axis meets the surface of the flask at its lowest point; these necks shall have either a 10/19 or a 14/23 ground glass conical socket;

b) *glass delivery tube,* (G), with a 10/19 or 14/23 ground glass cone to suit the flask being used. The tube shall be drawn down to a hole 1 mm diameter and be of such a length that, when inserted in an inclined socket, the tip is not more than 5 mm from the bottom of the flask.

c) *dropping funnel*, (B), 20 ml capacity, with a ground glass cone to suit the top and a ground glass cone on the stem to fit the flask in use. If the funnel is to be inserted in an inclined socket, the stem shall be so bent that the axis of the funnel is vertical.

d) *adapter*, (C), right angled with a 10/19 ground glass cone.

e) *Liebig condenser,* (D), effective length 150 mm, with 14/23 or 19/26 ground glass cone and socket. A 140 mm Liebig condenser complying with the requirements of BS 3787 is also suitable.

f) *delivery tube*, (E), glass 6 ± 0.5 mm o.d., bent at an angle of 110° with a small expansion chamber as in the figure and with a ground glass cone to fit the condenser (D). The delivery end shall be drawn down to a hole 1.0 mm in diameter.

E.4.2 *Absorption tube*, glass boiling tube, 200 mm × 30 mm, complying with the requirements of BS 3218.

E.4.3 *Burette,* 10 ml, complying with the requirements of class A of BS 846.

E.4.4 *Pipettes,* one-mark, 2 ml, 5 ml, 10 ml and 25 ml, complying with the requirements of class A of BS 1583.

E.4.5 *Volumetric flask,* one-mark, 100 ml complying with the requirements of class A of BS 1792.

E.4.6 *Drechsel bottle,* 250 ml, complying with the requirements of BS 2461.

E.4.7 *Measuring cylinder,* 10 ml, complying with the requirements of BS 604.

E.4.8 *Venting device,* a glass T-piece of sufficient length to reach to within 20 mm of the bottom of a glass cylinder containing a column of water 250 mm in height.

E.4.9 *Thermometer,* complying with the requirements of GP 105C/0.5/100 of BS 1704.

E.4.10 *Heating mantle*

E.5 Procedure

Stringent precautions shall be taken to avoid sulphur contamination from the atmosphere, apparatus, reagents or other sources. Care shall also be taken not to allow sodium hydroxide solutions or apparatus "wet" with this reagent to be exposed to the laboratory atmosphere.

E.5.1 *Preparation of apparatus.* Clean new apparatus with the cleaning solution (**E.3.5**) (see caution) and rinse it first with water and then with the acetone (**E.3.1**).

Dry it in an electric oven which has not been contaminated by previous use with sulphur of sulphur-containing compounds. The apparatus is self-scouring in constant use and it shall not be cleaned between determinations otherwise than by rinsing the flask (A), the delivery tube (E), the absorption tube and the thermometer with water. When not in constant use, it shall be cleaned, between determinations, with water.

E.5.2 *Preparation of the Raney nickel.* Using a cone of glazed paper transfer 0.5 ± 0.05 g of the Raney nickel (**E.3.3**) to the reduction flask A (**E.4.1**) and add 10 ml of the sodium hydroxide solution (**E.3.8**) using the measuring cylinder (**E.4.7**). Heat to a temperature of between 75 °C and 80 °C.

CAUTION. It is essential that care be taken at this stage because the reaction is vigorous.

NOTE Encrustations around the stoppers and necks of sodium hydroxide bottles contain sufficient quantities of sulphur to affect test results. Such encrustations should be removed without allowing material to fall into the bottle. Before using solution from the bottle, pour a little to waste. Replace the stoppers promptly.

When the reaction subsides, swirl the flask in order to detach the nickel adhering to the wall. Allow to stand for 10 min and then decant the supernatant liquid. Wash down the neck of the flask through which the nickel was introduced with 10 ml to 15 ml of water and swirl the water so as to disturb the nickel residue vigorously without entrainment of air. With the minimum of delay, for settling, decant the water as quickly as possible but without delaying to remove the last drops. Wash a further three times in the same way and follow with a wash with 10 ml of the propan-2-ol (**E.3.4**). Decant most of the propan-2-ol leaving sufficient to cover the nickel and then add a further 10 ml of the propan-2-ol.

E.5.4 *Preparation of the test sample.* Melt the laboratory sample, mix thoroughly and transfer the appropriate mass, as indicated in Table 4, to a tared weighing bottle. Allow to cool, weigh to the nearest 0.001 g and dissolve in the propan-2-ol (**E.3.4**). Transfer the solution quantitatively to the volumetric flask (**E.4.5**) and dilute to the mark with the propan-2-ol.

Table 4 — Test portion required for determination of sulphur content

Expected sulphur content	Mass of sample required	Volume of test solution
mg/kg	g	ml
20 to 100	5.5 to 6.0	25
100 to 200	5.5 to 6.0	5
500 to 1 000	0.6 to 0.7	25
1 000 to 2 000	0.7 to 0.8	10
2 000 to 5 000	0.7 to 0.8	5
5 000 to 10 000	0.7 to 0.8	2

E.5.5 *Determination.* Slightly grease all the joints of the reduction apparatus (**E.4.1**) with silicone grease and assemble it as in Figure 3 but omitting the dropping funnel. Transfer 50 ml of a mixture of equal volumes of the acetone (**E.3.1**) and the sodium hydroxide solution (**E.3.9**) (see note to **E.5.2**) to the absorption tube (**E.4.2**) and add five drops (0.3 ml) of the indicator solution (**E.3.11**) or a few particles of the solid. (See note to **E.3.11**.)

Using one of the pipettes (**E.4.4**) **(CAUTION. Do not use mouth suction)** transfer the appropriate volume of the test solution (Table 4) to the reduction flask (**E.4.1**). Insert the dropping funnel containing 10 ml of the hydrochloric acid solution (**E.3.6**). Connect the top of the dropping funnel and the delivery tube to the source of the nitrogen (**E.3.2**) through the Drechsel bottle (**E.4.6**) containing 75 ml of the potassium hydroxide solution (**E.3.7**). (See Figure 3.) Using the burette (**E.4.3**), add just sufficient of the mercury (II) acetate working solution (**E.3.10.2**) to the contents of the absorption tube (absorption solution) to change the colour from yellow to pale pink. Note the final burette reading as the beginning of the titration. Pass nitrogen into the apparatus at a rate of two to three bubbles per second as observed in the Drechsel bottle; the rate may be conveniently regulated by means of a screw clip.

Heat the flask with the heating mantle (**E.4.10**) at such a rate that the contents boil gently in about 10 min. Maintain the heating for a further 30 min at such a rate that small bubbles rise copiously from the nickel and gentle refluxing occurs.

Increase the input to the heating mantle slightly and allow the hydrochloric acid to drip slowly (10 ml in 5 min to 10 min) into the flask. Vigorous generation of hydrogen will occur, but little or no hydrogen sulphide will be evolved until about half the hydrochloric acid has entered the flask. Titrate the absorption solution to a pink colour. As hydrogen sulphide is evolved and absorbed, and the colour of the absorbing solution reverts to yellow, add the mercury (II) acetate solution in order to restore the pink colour.

After the addition of the acid, open the stopcock of the funnel occasionally to sweep forward any hydrogen sulphide that may have collected below it. When the evolution of hydrogen sulphide has almost ceased, increase the nitrogen flow rate to about five bubbles per second to improve the transfer of hydrogen sulphide to the absorption tube.

When the evolution of hydrogen sulphide has apparently ceased, turn off the nitrogen temporarily and cool the flask by reducing the heat input and blowing a little air on to it or by applying a damp cloth. The reduction in pressure will cause the absorption solution to rise up the delivery tube. Restore the nitrogen flow before the absorption solution reaches the bend above the cone. Repeat this operation at about 2 min intervals until no more hydrogen sulphide is washed down, as shown by the persistence of the pink colour. If any liquid enters the flask, abandon the test.

Boil the contents of the flask vigorously and continue the titration until the pink colour remains permanent. Note the burette reading as the completion of the titration.

E.5.4 *Determination of the blank.* Determine a blank value on the reagents, omitting the test portion. Once a day is normally sufficient, but the blank shall always be redetermined if there is any change in the reagents, apparatus or the laboratory atmosphere that could conceivably affect the blank value. If the value of the blank exceeds 0.6 ml, the cause should be established and rectified before proceeding with the estimation.

E.6 Expression of results

The total sulphur content, expressed in milligrams per kilogram, is given by the expression:

$$\frac{2000\ T\ (V_2 - V_1)}{V_0 \times m}$$

where

V_0 is the volume of test solution used (in ml)

V_1 is the volume of mercury (II) acetate solution used for the blank (in ml)

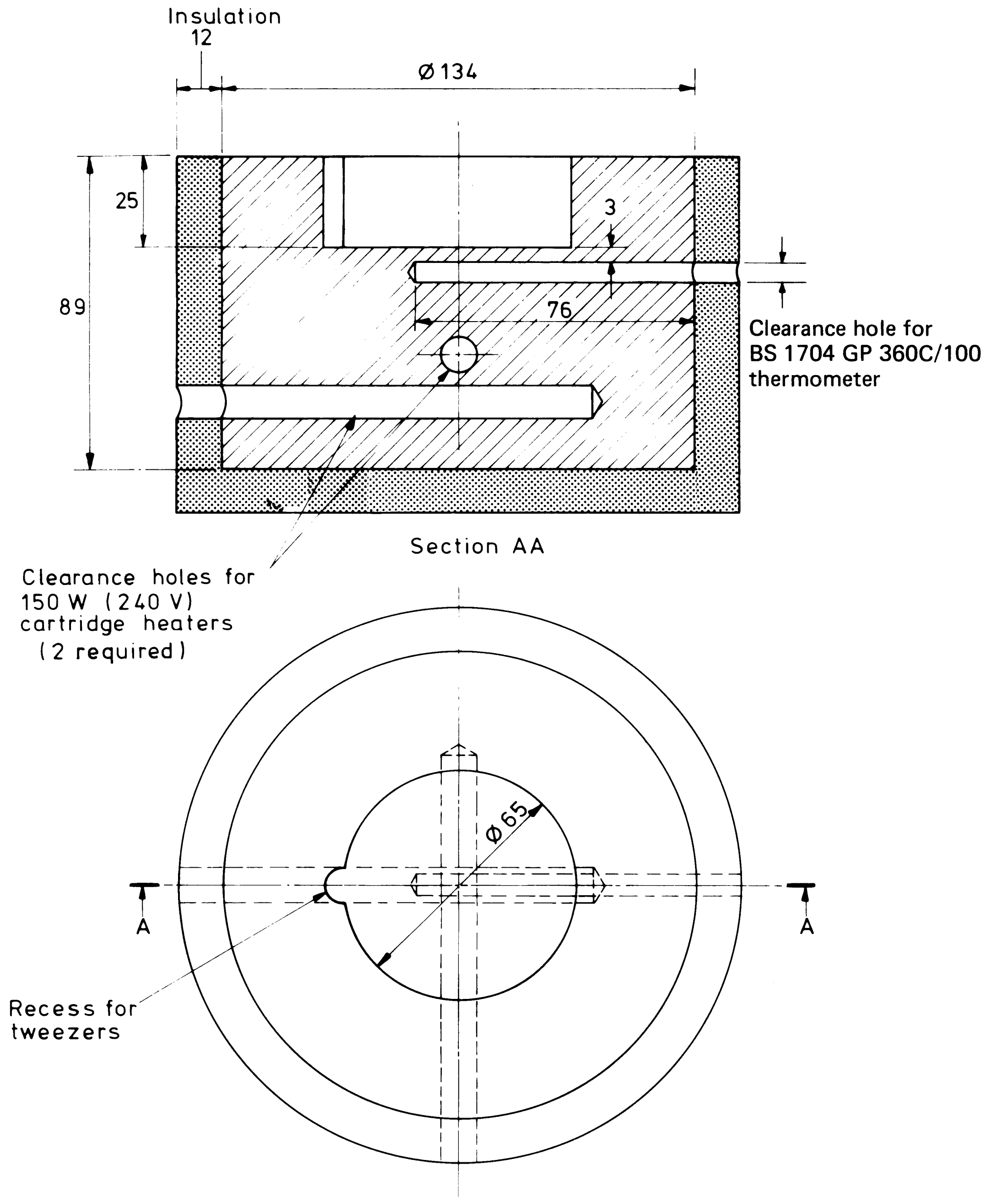
V_2 is the volume of mercury (II) acetate solution used for the titration (in ml)

m is the mass of sample taken (in g)

T is the calibration factor of the mercury (II) acetate solution

E.7 Precision (see foreword)

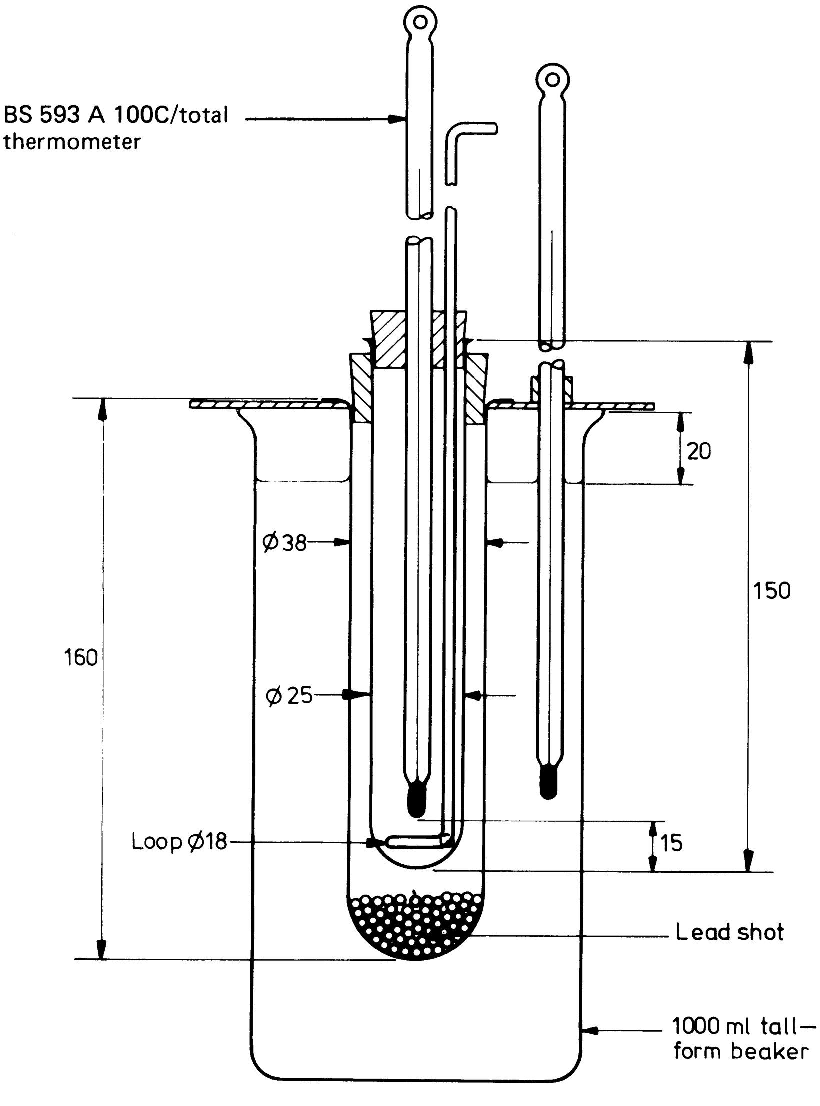
Sulphur level (mg/kg)	1	10	20	40	80	100
Repeatability, *r*	0.2	0.6	1.2	2.3	4.6	5.8
Reproducibility, *R*	0.3	1.8	3.5	7.0	14.0	17.6



All dimensions are in millimetres.

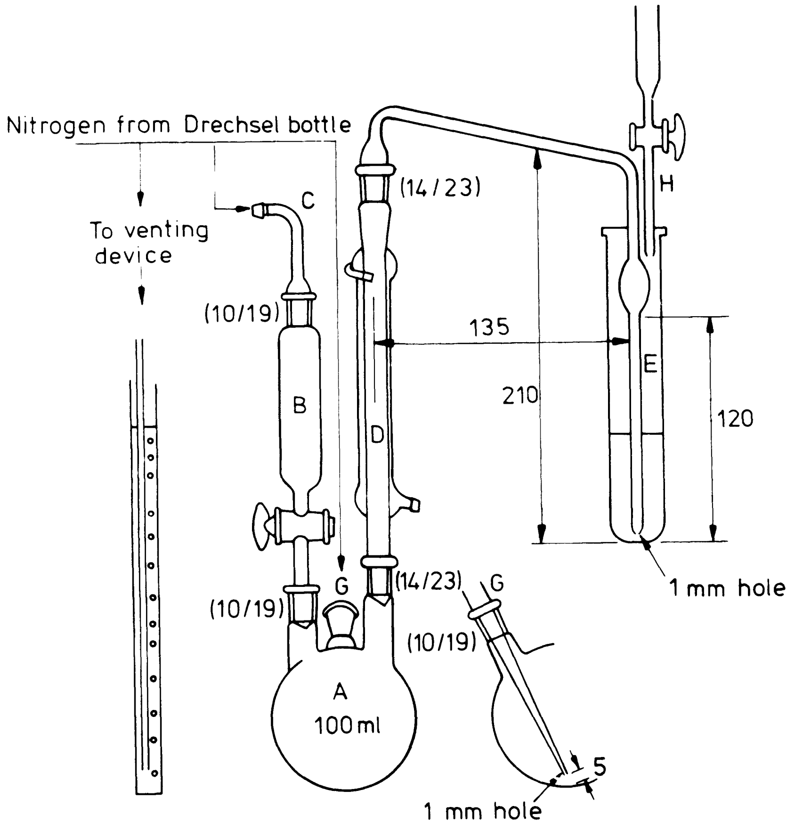
Material: mild steel

Figure 1 — Heating block for non-volatile matter determination



All dimensions are in millimetres.

Figure 2 — Crystallizing-point apparatus



All dimensions are in millimetres.

Figure 3 — Reduction apparatus

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Publications referred to

BS 135, BS 458, BS 805, *Specifications for benzene, xylenes and toluenes.*
BS 479-3, *Aromatic solvent naphtha (90/160).*
BS 570, *Plug-and-socket gas connectors for portable appliances.*
BS 572, *Interchangeable conical ground glass joints.*
BS 593, *Laboratory thermometers.*
BS 604, *Graduated measuring cylinders.*
BS 611, *Petri dishes.*
BS 612, *Nessler cylinders.*
BS 700, *Specification for graduated pipettes (including blowout pipettes).*
BS 756, *Dean and Stark apparatus.*
BS 846, *Burettes and bulb burettes.*
BS 1583, *One-mark pipettes.*
BS 1704, *General purpose thermometers.*
BS 1792, *One-mark volumetric flasks.*
BS 2461, *Gas washing bottles.*
BS 3218, *Test tubes and boiling tubes.*
BS 3787, *Glass condensers with standard joints.*
BS 3978, *Water for laboratory use.*
BS 4244, *Porcelain and silica crucibles.*
BS 4306, *Recommendations for the application of precision data to specifications for petroleum products.*
BS 4385, *Method for determination of water content of petroleum products (Dean and Stark method).*
BS 5309, *Methods for sampling chemical products.*
BS 5309-1, *Introduction and general principles.*
BS 5309-3, *Sampling of liquids.*
BS 5309-4, *Sampling of solids.*

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