

CONFIRMED
DECEMBER 2007

Methods for

Analysis and testing of coal and coke —

Part 10: Arsenic in coal and coke

UDC [662.66 + 662.74]:543.847:546.19

Cooperating organizations

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

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 British Carbonization Research Association*
 British Cast Iron Research Association
 British Ironfounders' Association
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 Central Electricity Generating Board*
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 Solid Smokeless Fuels Federation
 Independent experts

This British Standard, having been prepared under the direction of the Solid Fuel Standards Committee, was published under the authority of the Executive Board on 29 April 1977

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First published March 1942
 First revision December 1960
 Second revision April 1977

The following BSI references relate to the work on this standard:
 Committee references SFC/45 and SFC/45/8
 Draft for comment 73/42191DC

ISBN 0 580 09373 5

Amendments issued since publication

Amd. No.	Date of issue	Comments

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Foreword

This British Standard is Part 10 of a series issued under the same number, BS 1016, dealing with methods for the analysis and testing of coal and coke; the other Parts are as follows.

- *Part 1: Total moisture of coal;*
- *Part 2: Total moisture of coke;*
- *Part 3: Proximate analysis of coal;*
- *Part 4: Moisture, volatile matter and ash in the analysis sample of coke;*
- *Part 5: Gross calorific value of coal and coke;*
- *Part 6: Ultimate analysis of coal;*
- *Part 7: Ultimate analysis of coke;*
- *Part 8: Chlorine in coal and coke;*
- *Part 9: Phosphorus in coal and coke;*
- *Part 11: Forms of sulphur in coal;*
- *Part 12: Caking and swelling properties in coal;*
- *Part 13: Tests special to coke;*
- *Part 14: Analysis of coal ash and coke ash;*
- *Part 15: Fusibility of coal ash and coke ash;*
- *Part 16: Reporting of results.*

The methods given in BS 1016 are specified for the analysis of coal or of coke or, in some instances, for the analysis of both coal and coke. It is expected that the methods given in this Part would also be applicable to the determination of arsenic in manufactured smokeless fuels (other than coke) made by a conventional process, although no systematic survey has been made.

The revision of Part 10 has been undertaken to complete the introduction of metric units and to adopt a uniform presentation. No technical changes have been made to the procedures retained from the previous edition of this standard, but the “paper stain” method has now been omitted.

The wet oxidation procedure and the subsequent procedure for the colorimetric determination of the arsenic are technically equivalent to those specified in ISO/R 601; however, the dry oxidation procedure differs from that in the ISO standard in the nature of the oxidants and in the maximum temperature reached.

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 6, an inside back cover and a back cover.

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

1 Scope

This Part of BS 1016 describes two methods for determining the amount of arsenic in coal and coke.

2 References

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

3 Definitions

For the purposes of this Part of BS 1016 the following definitions apply.

3.1

coke

the solid residue of the distillation of coal at high temperature (above 800 °C)

3.2

repeatability

the maximum acceptable difference between duplicate determinations carried out at different times in the same laboratory on the same analysis sample by the same operator using the same apparatus

3.3

reproducibility

the maximum acceptable difference between the mean or duplicate determinations carried out in one laboratory and the mean of duplicate determinations carried out in any other laboratory on representative samples taken from the same gross sample after the last stage of the reduction process

4 Introduction

Two procedures are given for taking the sample into solution, after which the same procedure is followed in each case for the determination of the arsenic.

5 Sample

The coal or coke used for the determination of arsenic shall be the analysis sample having a top size not greater than 0.2 mm (ground to pass a 212 µm test sieve complying with the requirements of BS 410), taken and prepared according to BS 1017-1 or BS 1017-2 as appropriate. The sample shall be thoroughly mixed, preferably by mechanical means, immediately before the determination.

6 Wet oxidation method

6.1 Principle. The carbonaceous material is removed from a known mass of the sample and the arsenic is taken into solution by repeated oxidation with nitric acid in the presence of sulphuric acid. The arsenic in solution is reduced to the trivalent state and evolved as arsine by the action of zinc, the arsine being absorbed and oxidized to arsenic acid in a dilute iodine solution. Treatment with ammonium molybdate solution and reduction with hydrazinium sulphate produces a molybdenum blue coloration, of which the absorbance is proportional to the amount of arsenic present in the sample. The absorbance is measured by means of a suitable instrument and the arsenic content determined by reference to a calibration graph.

6.2 Special reagents. All reagents, unless otherwise specified, shall be of analytical reagent quality or AsT grade¹⁾ (arsenic test grade), as appropriate. Distilled or deionized water complying with the requirements of BS 3978 shall be used throughout.

6.2.1 Hydrochloric acid, $d = 1.18$.

6.2.2 Nitric acid, $d = 1.42$ (AsT grade).

6.2.3 Sulphuric acid, $d = 1.84$ (AsT grade).

6.2.4 Sulphuric acid, 7N approximately. Carefully add 10 ml of the sulphuric acid (see **6.2.3**) to 50 ml of water.

6.2.5 Sulphuric acid, 5.0N. Carefully add 14 ml of the sulphuric acid (see **6.2.3**) to 80 ml of water, cool and dilute to 100 ml. Standardize the solution against sodium carbonate and adjust to 5.0N.

6.2.6 Potassium iodide solution. Dissolve 15 g of potassium iodide (AsT grade) in 100 ml of water. Prepare a fresh solution each day.

6.2.7 Tin (II) chloride solution. Dissolve 40 g of tin (II) chloride dihydrate, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, in 100 ml of the hydrochloric acid (see **6.2.1**).

6.2.8 Iodine solution (stock solution, approximately 0.02N). Dissolve 2.54 g of iodine in 25 ml of water containing 8 g of potassium iodide (AsT grade). Dilute to 1 litre and store in a dark glass bottle.

6.2.9 Iodine solution, approximately 0.001N. Prepare a fresh solution daily by dilution of a suitable volume of the stock iodine solution (see **6.2.8**) with distilled water.

¹⁾ AsT grades complying with the British Pharmacopoeia are suitable.

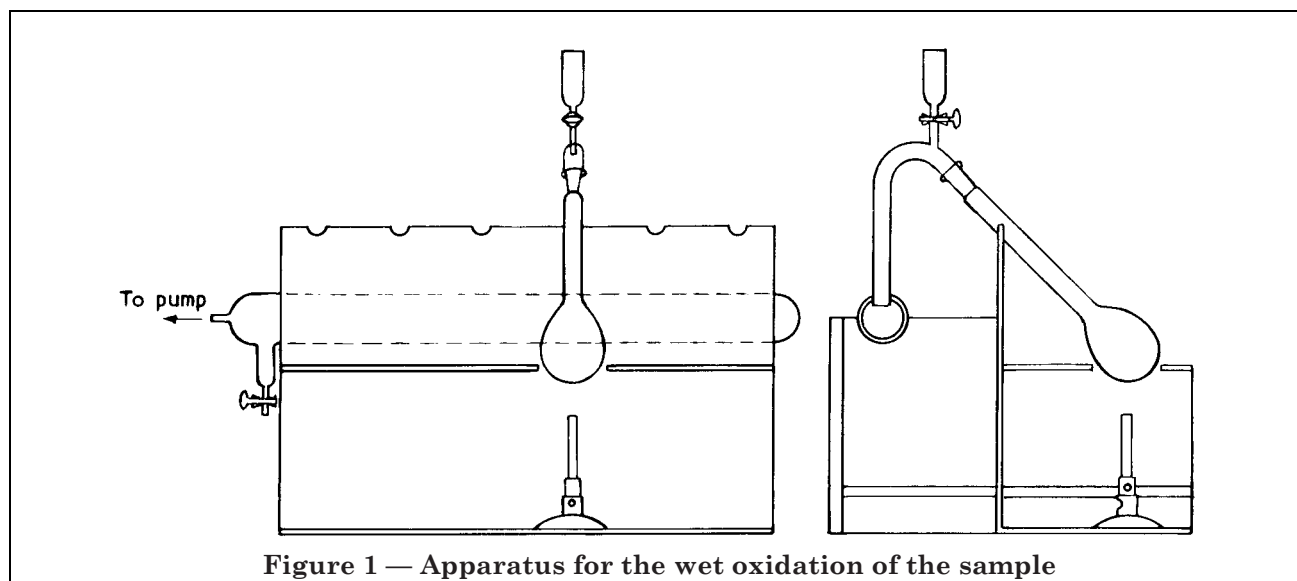


Figure 1 — Apparatus for the wet oxidation of the sample

6.2.10 Lead acetate solution, saturated. Add approximately 4.5 g of lead acetate trihydrate, $(\text{CH}_3\text{COO})_2\text{Pb}\cdot 3\text{H}_2\text{O}$, to 10 ml of water and shake well. Prepare a fresh solution daily.

6.2.11 Zinc, granulated, arsenic content less than 0.02 mg/kg.

6.2.12 Ammonium molybdate solution. Dissolve 10 g of ammonium molybdate tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$, in 1 litre of the sulphuric acid (see 6.2.5).

6.2.13 Hydrazinium sulphate solution. Dissolve 0.15 g of hydrazinium sulphate in 100 ml of water.

6.2.14 Arsenic solution. Dissolve exactly 0.1000 g of arsenic (II) oxide, previously dried at 110 °C for 2 h, in 50 ml of water containing 0.5 ml of a 700 g/l aqueous solution of sodium hydroxide. Add 2.0 ml of the sulphuric acid 5.0N (see 6.2.5) and dilute to 100 ml with water. This solution contains 1 mg/ml of As_2O_3 .

6.3 Special apparatus

6.3.1 Oxidation apparatus. A suitable apparatus, constructed of borosilicate glass, is shown in Figure 1. It consists of the following items.

6.3.1.1 Flask. A Kjeldahl flask of 300 ml capacity fitted with a 24/29 ground glass socket complying with the requirements of BS 572.

6.3.1.2 Fume duct. A fume duct of approximately 28 mm outside diameter, fitted with a dropping funnel of at least 15 ml capacity and a 24/29 ground glass cone complying with the requirements of BS 572. The fume duct may be of one piece or assembled from separate units by means of ground glass joints.

6.3.1.3 Fume extractor. A glass tube of approximately 40 mm diameter, sealed at one end and drawn out at the other end to form a connection to the water pump. The tube is fitted with a drain stopcock and a series of lipped holes to accommodate a number of fume ducts.

6.3.1.4 Digestion rack. A rack to hold several Kjeldahl flasks at an angle of 45° over bunsen burners, with a holder for the fume extractor.

6.3.1.5 Glass water jet pump or other suction device.

6.3.2 Arsenic evolution apparatus. A suitable apparatus is shown in Figure 2. It consists of the following items.

6.3.2.1 Evolution flask (A). A 80 ml conical flask fitted with a 19/26 ground glass socket complying with the requirements of BS 572.

6.3.2.2 Delivery tube (B). A thick-walled glass tube of 4 mm internal diameter, fitted with a 19/26 ground glass cone at one end and a 7/16 ground glass cone at the other, both complying with the requirements of BS 572. Glass hooks are fitted just above the 7/16 cone.

6.3.2.3 Delivery tube extension (C). A thick-walled glass tube of the shape and dimensions shown in Figure 2, fitted with a 7/16 ground glass socket complying with the requirements of BS 572 and glass hooks.

6.3.2.4 Absorption tube (D). A glass tube, 15 mm internal diameter by approximately 150 mm long, fitted with a 14/15 socket and having a corresponding ground glass stopper complying with the requirements of BS 572. The lower 80 mm of the tube is drawn out to approximately 11 mm internal diameter and the end sealed.

6.3.2.5 Helix (E). A helix, 80 mm long and of 5 mm pitch, of glass or inert plastics material²⁾ of circular section. The helix has an outside diameter such that it fits the lower half of the absorption tube freely but not loosely and an internal diameter slightly larger than the narrow end of the delivery tube. A correctly fitted helix allows gas bubbles to follow a spiral course freely and without bypass.

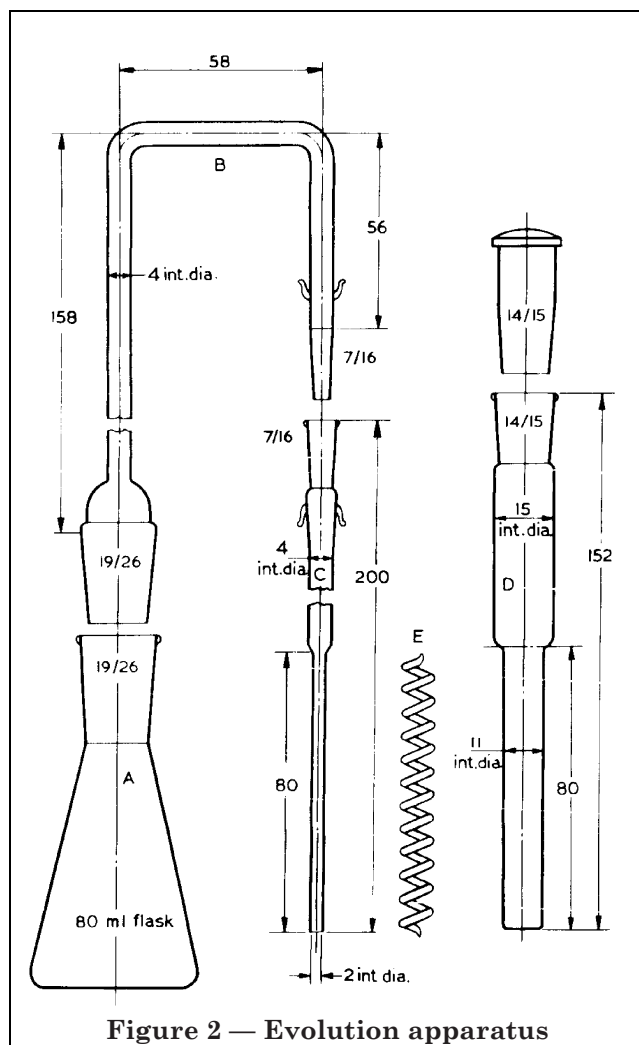


Figure 2 — Evolution apparatus

6.3.2.6 Springs. Small metal springs to ensure a gas-tight joint between the delivery tube and its extension.

6.3.3 Other apparatus. The following are also required.

6.3.3.1 Pipettes. 1 ml, 5 ml and 10 ml pipettes, class A, complying with the requirements of BS 1583.

6.3.3.2 Burettes. Two 5 ml burettes, graduated to 0.02 ml, complying with the requirements of BS 846.

6.3.3.3 Spectrophotometer. An absorptiometer of the required sensitivity³⁾.

6.4 Procedure

6.4.1 Digestion of sample. Weigh accurately about 1 g of the coal or coke and transfer to the clean, dry Kjeldahl flask. Assemble the apparatus, as shown in Figure 1, in a well ventilated fume cupboard. Add 7 ml of the sulphuric acid (see 6.2.3) and 3.5 ml of the nitric acid by means of the dropping funnel, rotating the flask so as to wash down any sample remaining in the neck.

After the initial reaction has subsided (see note 1), heat the flask carefully so that the reaction proceeds smoothly and without frothing. Continue heating the flask under the same conditions until only fumes of sulphuric acid are evolved. Add 0.2 ml to 0.4 ml of the nitric acid to the dropping funnel and run the acid drop by drop into the flask.

Heat for 2 min to 3 min until no more dense brown fumes are evolved. Add, in a similar manner, a further 0.2 ml to 0.4 ml of the nitric acid and heat again until brown fumes cease to be evolved. Continue such additions of nitric acid, heating in the same way after each addition, until all visible carbonaceous matter has been oxidized, rotating the flask periodically to wash down any carbonaceous matter adhering to the sides of the flask. After about 1½ h to 2 h the reaction mixture should be a pale greenish-yellow colour (see note 2) with no visible carbonaceous matter present.

Heat the flask more strongly until white fumes appear and allow to fume for 5 min (see note 3). Cool the flask to approximately room temperature, remove the dropping funnel and fume duct assembly and add a few glass beads to the contents of the flask.

Add cautiously 10 ml of water, heat until white fumes appear and then allow to fume gently for 10 min. Cool the flask until the evolution of the white fumes ceases, add 0.2 ml of the nitric acid, reheat the flask and allow to fume for a further 10 min.

To ensure complete oxidation, cool the flask to approximately room temperature and repeat the procedure described in the preceding paragraph.

²⁾ Unplasticized polyvinyl chloride is suitable.

³⁾ A Unicam SP 600 spectrophotometer or similar instruments is suitable.

Cool the flask to approximately room temperature, add 10 ml of water, heat to fuming and allow to fume for 20 min. Finally, add 10 ml of water, heat to fuming, allow to fume for 10 min and cool.

Add 10 ml of water and transfer the solution to the evolution flask. Wash the Kjeldahl flask with water, adding the washings to the evolution flask. The combined volume of the washings and original solution should be about 35 ml.

NOTE 1 If violent frothing occurs on addition of the mixed acids, apply a damp cloth to the neck of the flask, and heat intermittently as the froth subsides.

NOTE 2 For the first 15 min of heating a coal sample, the reaction mixture is a tarry mass; subsequently it changes in colour from black to dark reddish-brown, to amber and finally to pale greenish-yellow. The time taken to reach this stage should not be more than 2 h. The carbonaceous matter remaining at this stage should be removed by continued treatment.

If a black liquid still remains after 45 min heating, either:

a) too low a temperature has been used and the nitric acid has not reacted with the sample; raise the temperature to distil off excess nitric acid, then continue the normal oxidation as described; or

b) too high a temperature has been used, nitric acid being distilled off without reacting; cool, add further nitric acid and heat the mixture as described.

Coke samples should also react to produce a clear greenish-yellow solution within 2 h, but certain exceptional samples may take longer.

NOTE 3 If the colour reverts to amber or deep red, add a further 0.2 ml to 0.4 ml of the nitric acid, heat to fuming and allow to fume for 5 min.

6.4.2 Evolution, colour development and measurement. Add to the solution in the evolution flask (see note) 2.0 ml of the potassium iodide solution and 0.5 ml of the tin (II) chloride solution, mixing well after each addition, and allow to stand for 15 min at room temperature.

NOTE If the sample is expected to contain more than 16 µg of As₂O₃ per gram, take an aliquot of the wet oxidized coal or coke solution containing not more than that amount.

Pipette 5 ml of the 0.001N iodine solution (see 6.2.9) into the absorption tube containing the helix. Fit a cotton wool plug into each end of the delivery tube, moistening the plug in the 19/26 cone with the lead acetate solution. Connect the delivery tube to its extension, moisten the joint with water and secure by means of the hooks and springs. Insert the delivery tube into the absorption tube, add 5 g of the zinc to the evolution flask and complete the assembly of the apparatus as quickly as possible.

After evolution has taken place for 1 h, disconnect the absorption tube and add, by means of the burettes, 0.5 ml of the ammonium molybdate solution and 0.2 ml of the hydrazinium sulphate solution, inserting the ground glass stopper and shaking well after each addition. Remove the helix without rinsing and suspend the absorption tube in boiling water in a suitable water bath for 10 min. Cool to room temperature, transfer the solution to a 10 mm cell and measure the absorbance of the solution using the spectrophotometer at a wavelength of 835 µm against water set at zero absorbance.

6.4.3 Determination of blank. Carry out a blank determination at the same time and under the same conditions as the actual determination but omitting the sample.

6.5 Preparation of calibration graph.

Pipette 1 ml of the arsenic solution into a 100 ml graduated flask, dilute to 100 ml with water and mix (1 ml = 10 µg As₂O₃).

Take 0.0, 0.1, 0.2, 0.3, .. 1.6 ml portions of this solution, place each of them in an evolution flask and add 35 ml of the dilute sulphuric acid solution (see 6.2.4). Carry out the evolution procedure and measurement of the colour produced as described in 6.4. Deduct the absorbance for 0.0 µg of As₂O₃ from the absorbance of each of the remaining solutions and construct a calibration graph of absorbance against arsenic (II) oxide content in µg.

6.6 Calculation of result

$$As_2O_3 = \frac{m_2 - m_3}{m_1} \times 10^{-4}$$

where

m_1 is the mass of the sample taken (in g)

m_2 is the mass of arsenic (II) oxide equivalent to the absorbance of the test solution (in µg)

m_3 is the mass of arsenic (II) oxide equivalent to the absorbance of the blank solution (in µg)

As_2O_3 is the percentage of arsenic (II) oxide in the analysis sample.

7 Dry oxidation method

7.1 Principle. The carbonaceous material is removed from a known mass of the sample by heating in a stream of oxygen in the presence of potassium permanganate and magnesium oxide. The residue is extracted with sulphuric acid and the arsenic in solution is determined by the same procedures as those for wet oxidation.

7.2 Special reagents. All reagents, unless otherwise specified, shall be of analytical reagent quality or AsT grade⁴⁾ (arsenic test grade) as appropriate. Distilled or deionized water complying with the requirements of BS 3978 shall be used throughout.

7.2.1 Magnesium oxide.

7.2.2 Potassium permanganate⁵⁾.

7.2.3 Sulphuric acid, $d = 1.84$.

7.2.4 Sulphuric acid, 7N approximately. Carefully add 10 ml of the sulphuric acid (see 7.2.3) to 50 ml of water.

7.2.5 Sulphuric acid, 5.0N. Carefully add 14 ml of the sulphuric acid (see 7.2.3) to 80 ml of water, cool and dilute to 100 ml. Standardize the solution against sodium carbonate and adjust to 5.0N.

7.2.6 Potassium iodide solution. Dissolve 15 g of potassium iodide (AsT grade) in 100 ml of water. Prepare a fresh solution each day.

7.2.7 Tin (II) chloride solution. Dissolve 40 g of tin (II) chloride dihydrate, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, in 100 ml of the hydrochloric acid (see 6.2.1).

7.2.8 Iodine solution (stock solution, approximately 0.02N). Dissolve 2.54 g of iodine in 25 ml of water containing 8 g of potassium iodide (AsT grade). Dilute to 1 litre and store in a dark glass bottle.

7.2.9 Iodine solution, approximately 0.001N. Prepare a fresh solution daily by dilution of a suitable volume of the stock iodine solution (see 7.2.8) with distilled water.

7.2.10 Lead acetate solution, saturated. Add approximately 4.5 g of lead acetate trihydrate, $(\text{CH}_3\text{COO})_2\text{Pb} \cdot 3\text{H}_2\text{O}$, to 10 ml of water and shake well. Prepare a fresh solution daily.

7.2.11 Zinc, granulated, arsenic content less than 0.02 p.p.m. *m/m*.

7.2.12 Ammonium molybdate solution. Dissolve 10 g of ammonium molybdate tetrahydrate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, in 1 litre of the sulphuric acid (see 7.2.5).

7.2.13 Hydrazinium sulphate solution. Dissolve 0.15 g of hydrazinium sulphate in 100 ml of water.

7.2.14 Arsenic solution. Dissolve exactly 0.1000 g of arsenic (II) oxide, previously dried at 110 °C for 2 h, in 50 ml of water containing 0.5 ml of a 700 g/l aqueous solution of sodium hydroxide. Add 2.0 ml of the sulphuric acid (see 7.2.5) and dilute to 100 ml with water. This solution contains 1 mg/ml of As_2O_3 .

7.2.15 Oxalic acid solution. Dissolve 30 g of oxalic acid dihydrate, $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$, in 100 ml of ethanol (95 % *v/v*).

7.2.16 Oxygen.

7.3 Special apparatus

7.3.1 Combustion tube (see Figure 3). A transparent silica tube, approximately 24 mm internal diameter, approximately 29 mm outside diameter by 380 mm long, with a side arm, 4 mm internal diameter by 25 mm long, sealed in at a distance of about 25 mm from one end. The other end of the tube is drawn out to 4 mm internal diameter for a distance of 65 mm.

7.3.2 Heating unit. The combustion tube may be heated by gas or electricity. In either case, the heating unit shall be capable of heating the boat and its contents to a final temperature of 800 °C.

7.3.3 Combustion boat. A silica combustion boat, about 100 mm long by 20 mm wide by 12 mm deep.

7.3.4 Flowmeter. A flowmeter capable of measuring a rate of flow of 700 ml/min of oxygen.

7.3.5 Arsenic evolution apparatus, as described in 6.3.2.

7.3.6 Pipettes, as described in 6.3.3.1.

7.3.7 Burettes, as described in 6.3.3.2.

7.3.8 Spectrophotometer, as described in 6.3.3.3.

7.4 Procedure

7.4.1 Oxidation of sample. Cover the bottom of the boat evenly with between 0.3 g and 0.4 g of the magnesium oxide. Weigh accurately about 1.0 g of the sample and add 1.0 g of a mixture of equal masses of the potassium permanganate and the magnesium oxide. Mix well by grinding in an agate mortar and spread evenly in the boat. Cover the contents of the boat with a further 0.5 g of the magnesium oxide.

If gas heating is used, insert the boat into the combustion tube and close the inlet end with a rubber stopper. Connect the oxygen supply to the side arm and pass the oxygen through the apparatus at a rate of about 700 ml/min. Heat the boat slowly at first (see note) and then more strongly until combustion is complete, usually about 5 min after the sample first becomes incandescent.

⁴⁾ AsT grades complying with the British Pharmacopoeia are suitable.

⁵⁾ It is recommended that this reagent be tested specifically for arsenic content which should not exceed 0.1 p.p.m. *m/m*.

If electrical heating is used raise the temperature of the furnace to 800 °C and insert the boat into that part of the combustion tube which is at a temperature of approximately 650 °C. Close the inlet end with a rubber stopper, connect the oxygen supply to the side arm and pass the oxygen through the apparatus at a rate of about 700 ml/min (see note). After a period of 5 min remove the rubber stopper, push the boat to the centre of the furnace, replace the rubber stopper and continue heating for a further 10 min or until combustion is complete, whichever is the longer.

When combustion is complete, as indicated by a green colour of the contents of the boat, stop the oxygen flow, remove the boat from the combustion tube and allow the boat to cool. Shake the ignited mass from the boat into a 100 ml beaker and rinse the boat with 10 ml to 15 ml of water. Add 2 ml of the sulphuric acid (see 7.2.3) and 4 ml of the oxalic acid, cover the beaker with a watch-glass and boil gently for 10 min.

Allow the solution to cool and transfer it to the evolution flask. Rinse the beaker with water, adding the washings to the flask. Add 6 ml of the sulphuric acid (see 7.2.3) to the flask and dilute to 35 ml with water.

NOTE With samples other than anthracite and coke it is necessary to carry out the initial heating very slowly and to reduce the oxygen flow. In all cases a plume of combustion products emerging from the outlet of the tube and starting to spread out at 60 mm to 80 mm from the end of the tube indicates satisfactory combustion conditions.

7.4.2 Evolution, colour development and measurement. Carry out the evolution procedure, colour development, measurement, blank determination, calibration and calculation of result exactly as described in 6.4.2, 6.4.3, 6.5 and 6.6.

8 Reporting of result

The result, preferably the mean of duplicate determinations, shall be reported to the nearest 0.001 % As_2O_3 .

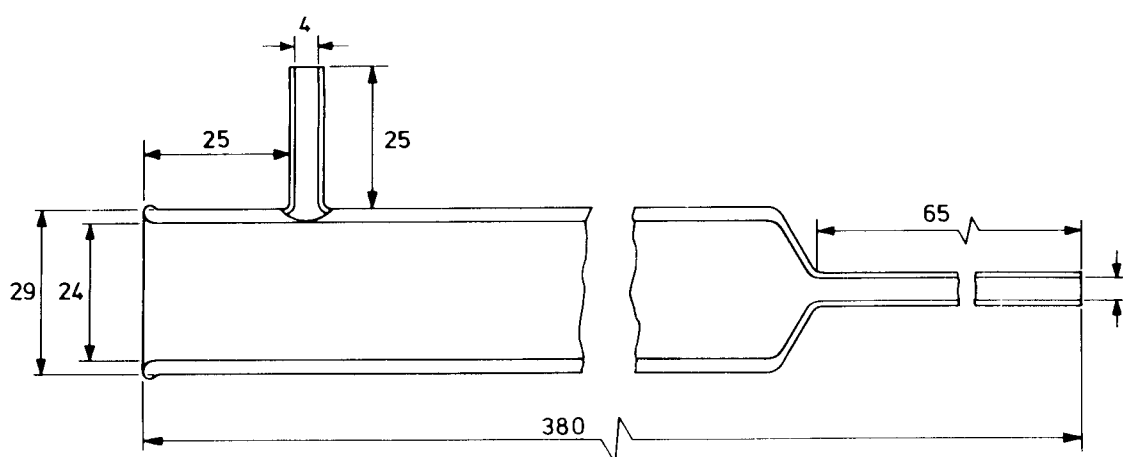
If desired, % As_2O_3 can be converted to % As in the sample by multiplying the % As_2O_3 value (before rounding) by 0.757.

The percentages, either of As_2O_3 or of As, can be expressed as mg/kg (p.p.m.) by multiplying by 10^4 .

9 Precision of the determination

When the methods described in this Part of BS 1016 are operated satisfactorily, the numerical values for repeatability and reproducibility (see clause 3) should not exceed those given below; otherwise, reference should be made to 4.4 in BS 1016-16:1971.

Arsenic (II) oxide content %	Repeatability	Reproducibility
Less than 0.0006	% absolute 0.0001	% absolute 0.0001
Equal to or more than 0.0006	0.00015	0.00015



Dimensions are in millimetres
Figure 3 — Combustion tube

Publications referred to

BS 410, *Test sieves.*

BS 572, *Interchangeable conical ground glass joints.*

BS 846, *Burettes and bulb burettes.*

BS 1016, *Methods for the analysis and testing of coal and coke.*

BS 1016-16, *Reporting of results.*

BS 1017, *The sampling of coal and coke.*

BS 1017-1, *Sampling of coal.*

BS 1017-2, *Sampling of coke.*

BS 1583, *One-mark pipettes.*

BS 3978, *Water for laboratory use.*

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

ISO/R 601, *Determination of arsenic in coal and coke.*

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