BRITISH STANDARD

BS 5666-3: 1991

Methods of analysis of

Wood preservatives and treated timber —

Part 3: Quantitative analysis of preservatives and treated timber containing copper/chromium/arsenic formulations

Confirmed November 2008

NOTE It is essential that this Part is read in conjunction with Part 1 "Guide to sampling and preparation of wood preservatives and treated timber for analysis".

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Wood Preservation Standards Policy Committee (WPC/-) to Technical Committee WPC/11, upon which the following bodies were represented:

Association of Consulting Scientists
British Drier Manufacturers' Association
British Wood Preserving and Damp-proofing Association
Department of the Environment (Building Research Establishment)
Ministry of Defence

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Contents

Foreword

This Part of BS 5666 has been prepared under the direction of the Wood Preservation Standards Policy Committee.

This Part of BS 5666 was first published in 1979. This edition introduces technical changes but it does not reflect a full review or revision of this Part of the standard, which will be undertaken in due course. This edition supersedes BS 5666-3:1979 which is withdrawn.

CAUTION. Attention is drawn to the Health and Safety at Work etc. Act 1974, and the need for ensuring that the methods of test specified in this standard are carried out with suitable precautions.

The procedures described in this standard are intended to be carried out by qualified chemists or other suitably trained and/or supervised personnel. Normal safety precautions should be observed throughout the use of the methods.

Attention is drawn to the general safety precautions mentioned in clause **3** of BS 5666-1:1987.

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 8, 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 5666 details procedures for the determination of copper, chromium and arsenic in preservatives and in treated timber containing those water-borne preservative compositions which consist essentially of copper, chromium and arsenic compounds. The wood sample may be in the form of sawdust, wood flour, or thin sections. There is no inter-elemental interference during the determinations and the procedures are specific for arsenic even in the presence of phosphorus compounds.

The procedure for the analysis of preservatives has been primarily designed for solutions complying with the requirements of BS 4072; for samples of other compositions it may be necessary to make adjustments. It is difficult to make recommendations as to the quantity of sample to be taken in the case of treated wood because the predominant factor is the quantity of preservative within the wood rather than the mass of the wood itself. The digestion procedure has been designed to deal with wood samples up to 8 g in mass, but it may be necessary to adjust the concentration of the resulting solution before a satisfactory analysis can be carried out.

Two methods are described, the first involving atomic absorption spectrometry and the second colorimetry.

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

2 Method 1. Atomic absorption spectrometric method

2.1 Principle

Copper, chromium and arsenic compounds are leached quantitatively from the wood sample with a mixture of dilute sulphuric acid and hydrogen peroxide solution. Preservatives are treated with the same mixture. The resulting solutions, after addition of sodium sulphate solution, are analysed using atomic absorption spectrometry[1)].

2.2 Reagents

Reagents shall be of recognized analytical reagent grade, and water complying with the requirements of grade 3 of BS 3978 shall be used throughout.

WARNING NOTE. Sulphuric acid (ρ_{20} = 1.84 g/mL) and hydrogen peroxide solution (100 volumes) are corrosive and cause burns. Care should be taken to avoid their contact with eyes and skin.

2.2.1 *Sulphuric acid solution,* $c(H_2SO_4)$ = 2.5 mol/L. Cautiously add, with stirring and cooling, 280 mL of sulphuric acid (ρ_{20} = 1.84 g/mL) to 1 600 mL of water. Cool and dilute to 2 litres with water.

2.2.2 *Hydrogen peroxide solution,* $c(H_2O_2)$, 300 g/L (100 volumes).

2.2.3 *Sodium sulphate solution,* $c(Na_2SO_4)$ = 30 g/L. Dissolve 30 g of anhydrous sodium sulphate in water and dilute to 1 litre with water.

2.2.4 *Sulphuric acid solution,* $c(H_2SO_4)$ = 0.5 mol/L/*sodium sulphate 3 g/L solution*. Dilute 200 mL of sulphuric acid solution (**2.2.1**) plus 100 mL of 30 g/L sodium sulphate solution (**2.2.3**) to 1 litre with water and mix.

2.2.5 *Standard solution* (1 mL μ 500 μg of copper, 1 000 μg of chromium and 1 000 μg of arsenic). Dissolve 0.9825 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in a little water and transfer the solution to a 500 mL one-mark volumetric flask. Dissolve 1.4135 g of potassium dichromate ($K_2Cr_2O_7$) in a little water, add 50 mL of sulphuric acid solution (**2.2.1**) and 10 mL of hydrogen peroxide solution (**2.2.2**), boil until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer the mixture to the one-mark volumetric flask containing the copper sulphate solution. Dissolve 0.6600 g of arsenic trioxide (As_2O_3) by boiling it in a solution containing 50 mL of sulphuric acid solution (**2.2.2**) and 75 mL of water. Continue boiling until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer it to the one-mark volumetric flask. Add 50 mL of sodium sulphate solution (**2.2.3**) to the volumetric flask, dilute to the mark with water and mix.

2.3 Apparatus

2.3.1 *Volumetric glassware*, complying with the requirements for class A quality in accordance with BS 700-2 (type 2), BS 846, BS 1583 or BS 1792, as appropriate.

2.3.2 *Atomic absorption spectrometer*, together with suitable sources of resonance radiation for copper, chromium and arsenic, e.g. hollow-cathode lamps.

[1)] For reference, see Williams, A.I., *Analyst*, 1970, 95, 670.

2.4 Procedure

2.4.1 *Instruments settings and operation*

The instrument settings and operating conditions for the determination of copper, chromium and arsenic shall be as recommended in the instrument users' manual. Copper is determined in a fuel-lean air/acetylene flame at 324.8 nm, chromium in a fuel-rich air/acetylene flame or a fuel-lean nitrous oxide/acetylene flame at 357.9 nm or 429.0 nm, and arsenic in an argon/hydrogen or nitrous oxide/acetylene flame at 193.7 nm or 197.2 nm.

2.4.2 *Safety precautions*

It is essential that particular care be exercised when igniting the argon/hydrogen flame and that the instructions given in the instrument users' manual be followed precisely.

2.4.3 *Preparation of calibration solutions*

Transfer portions of 0.5, 1, 2, 3, 4, 5, 6 and 7 mL of the standard solution (**2.2.5**) to a series of 100 mL one-mark volumetric flasks, dilute to the mark with the sulphuric acid/sodium sulphate solution (**2.2.4**) and mix.

1 mL portions of the solutions so constituted contain 2.5, 5, 10, 15, 20, 25, 30 and 35 μg, respectively, of copper and 5, 10, 20, 30, 40, 50, 60 and 70μg, respectively, of chromium and of arsenic.

NOTE The range of the set of calibration solutions is given as a guide; being a function of the instrumental sensitivity, it can be varied. If varied, it is for the analyst to adjust the concentration of the material to be analysed in the test solution for presentation to the instrument in order to obtain optimum conditions, after checking that the possible interferences remain corrected and that others do not appear.

2.4.4 *Analysis of preservatives*

Transfer by pipette a suitable quantity[2)] of preservative to a 250 mL conical flask, add 40 mL of sulphuric acid solution (**2.2.1**) and, with caution, 8 mL of hydrogen peroxide solution (**2.2.2**). Boil the contents of the flask until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool the flask to room temperature and transfer its contents to a 200 mL one-mark volumetric flask. Add 20 mL of sodium sulphate solution (**2.2.3**), dilute to the mark with water and mix to give the test solution.

Using the operating conditions suitable for the instrument, aspirate the sulphuric acid/sodium sulphate solution (**2.2.4**) to obtain the blank absorbance, followed by a suitable range of calibration solutions and then the test solution(s). Check the calibration solutions after the last test solution has been run.

NOTE If a number of samples are to be analysed it may be advisable to check the instrument stability by bracketing each sample with appropriate standard solutions.

Plot calibration graphs of μg/mL of copper, chromium, and arsenic against absorbance. Determine the contents of copper, chromium, and arsenic in the test solutions by comparing the absorbance readings with the calibration graphs.

2.4.5 *Analysis of treated wood*

2.4.5.1 *Preparation of timber sample for analysis*

Prepare the sample for analysis by converting the treated timber into a form suitable for extraction (i.e. shavings or sawdust) as described in clause **5** of BS 5666-1:1987.

Divide the prepared sample into two unequal portions, (0.5 g and 5 g respectively). Determine the moisture content on the 0.5 g portion according to the procedure described in clause **6** of BS 5666-1:1987. Reserve the 5 g sample for the analysis (**2.4.5.2**).

2.4.5.2 *Extraction and preparation of test solution*

Weigh about 5 g of the prepared test sample (**2.4.5.1**) to the nearest 0.1 g into a 250 mL conical flask, add 50 mL of the sulphuric acid solution (**2.2.1**) and, with caution, 10 mL of the hydrogen peroxide solution (**2.2.2**). Heat at 75 °C in a water bath for 30 min with occasional swirling to mix the contents of the flask. Filter the solution through a filter paper[3)] into a second conical flask and thoroughly wash the solids on the filter paper with a maximum of 100 mL of water.

Boil the filtrate and washings until evolution of oxygen ceases and all the hydrogen peroxide is decomposed. Cool to room temperature and transfer the solution to a 250 mL one-mark volumetric flask, add 25 mL of the sodium sulphate solution (**2.2.3**) and make up to the mark with water to give the test solution.

2.4.5.3 *Measurement*

Using the operating conditions suitable for the instrument, aspirate the sulphuric acid/sodium sulphate solution (**2.2.4**) to obtain the blank absorbance, followed by a suitable range of calibration solutions and then the test solution(s). Check the calibration solutions after the last test solution has been run (see note in **2.4.4**). Plot calibration graphs of μg/mL of copper, chromium and arsenic against absorbance. Determine the contents of copper, chromium and arsenic in the test solutions by comparing the absorbance readings with the calibration graphs.

[2)] For 30 g/L solutions of preservatives complying with BS 4072-1, transfer 2 mL of the solution.

[3)] A Whatman No. 44 filter paper has been found to be suitable.

NOTE This procedure is suitable for samples in the mass range from 3 g to 8 g. For smaller samples use 100, 50 or 25 mL one-mark volumetric flasks, as appropriate, and correspondingly smaller volumes of reagents.

2.4.6 ***Calculations***

2.4.6.1 *The concentration of metals in a preservative*

The concentration, in g/100 mL, of copper or chromium or arsenic is given by:

$$\frac{C}{50V}$$

where:

C is the concentration of the appropriate metal in μg/mL of the test solution;

V is the volume in mL of the quantity of preservative taken (**2.4.4**).

To express the results in terms of copper sulphate (as $CuSO_4.5H_2O$), of potassium dichromate (as $K_2Cr_2O_7$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentrations of the respective metals by the following factors:

C_{copper} by 3.93

$C_{chromium}$ by 2.83 for the potassium salt
or 2.87 for the sodium salt

$C_{arsenic}$ by 1.77

2.4.6.2 *The percentages of metals in treated wood*

The percentage by mass of copper or chromium or arsenic in the dry wood is given by:

$$\frac{C\,(100+h)}{4\,000\,m}$$

where

C is the concentration of the appropriate metal in μg/mL of the test solution (**2.4.5**);

h is the moisture content in % (m/m) of the prepared timber sample (**2.4.5.1**);

m is the mass of the prepared timber sample in g.

To express the results in terms of copper sulphate (as $CuSO_4.5H_2O$), of potassium dichromate (as $K_2Cr_2O_7$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentration of the appropriate metal by the corresponding factor given in **2.4.6.1**.

2.4.6.3 *The dry salt retentions in treated wood*

The analysis result may also be expressed as kg/m^3 if the density of the actual sample of timber is known; it is not satisfactory to take an average density figure for the species of timber concerned. Adjustments for the moisture content (see Part 1 of this standard) of the timber are also necessary if the density figure does not apply to the dry material.

The retention of the toxic ingredient of the oven-dry wood (expressed in kg/m^3) is given by:

$$\frac{T\times D}{100}$$

where

T is the percentage by mass of toxic ingredient;

D is the oven-dry density of the wood (kg/m^3).

3 Method 2. Colorimetric method

3.1 Principle

Copper, chromium and arsenic compounds are leached quantitatively from the wood sample with a mixture of dilute sulphuric acid and hydrogen peroxide solution. Preservatives are treated with the same mixture. Copper and chromium, in portions of the resulting solution, are allowed to react with zinc dibenzyldithiocarbamate and diphenylcarbazide respectively[4]. The arsenic is converted to arsine, and reacted with silver diethyldithiocarbamate. The concentrations of the resulting coloured complexes are measured spectrometrically.

3.2 Reagents

Reagents shall be of recognized analytical reagent grade, and water complying with the requirements of grade 3 of BS 3978 shall be used throughout.

WARNING NOTE. Sulphuric acid (ρ_{20} = 1.84 g/mL) and hydrogen peroxide solution (100 volumes) are corrosive and cause burns. Care should be taken to avoid their contact with eyes and skin.

3.2.1 *Sulphuric acid solution, c*(H_2SO_4) = 2.5 mol/L. Cautiously add, with stirring and cooling, 140 mL of sulphuric acid (ρ_{20} = 1.84 g/mL) to 800 mL of water. Cool and dilute to one litre with water.

3.2.2 *Hydrogen peroxide solution, c*(H_2O_2), 300 g/L (100 volumes).

3.2.3 *Sulphuric acid solution, c*(H_2SO_4) = 0.5 mol/L. Cautiously add, with stirring, 28 mL of sulphuric acid (ρ_{20} = 1.84 g/mL) to 900 mL of water. Cool and dilute to one litre with water.

[4] For reference, see Williams, A.1., *Analyst*, 1972, 97, 104.

3.2.4 *Zinc dibenzyldithiocarbamate solution*, 1 g/L. Dissolve 0.5 g of zinc dibenzyldithiocarbamate $[[(C_6H_5.CH_2)_2NCS.S]_2Zn]$ in 500 mL of carbon tetrachloride (CCl_4).

3.2.5 *Standard solution* A(1 mL ≡ 500 μg of copper, 1 000 μg of chromium and 1 000 μg of arsenic). Dissolve 0.9825 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in a little water and transfer the solution to a 500 mL one-mark volumetric flask. Dissolve 1.4135 g of potassium dichromate ($K_2Cr_2O_7$) in a little water, add 50 mL of sulphuric acid solution (**3.2.1**) and 10 mL of hydrogen peroxide solution (**3.2.2**), boil until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer the mixture to the one-mark volumetric flask containing the copper sulphate solution. Dissolve 0.6600 g of arsenic trioxide (As_2O_3) by boiling it in a solution containing 50 mL of sulphuric acid solution (**3.2.1**), 10 mL of hydrogen peroxide solution (**3.2.2**) and 75 mL of water. Continue boiling until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, cool and transfer it to the volumetric flask. Dilute to the mark with water and mix.

3.2.6 *Standard copper solution* (1 mL ≡ 2 μg of copper). Transfer by pipette 10 mL of standard solution A (**3.2.5**) to a 250 mL one-mark volumetric flask, make up to the mark with water and mix. Transfer by pipette 10 mL of this solution to a 100 mL one-mark volumetric flask, add 20 mL of sulphuric acid solution (**3.2.1**), make up to the mark with water and mix.

3.2.7 *Standard chromium solution* (1 mL ≡ 100 μg of chromium). Transfer by pipette 10 mL of standard solution A to a 100 mL one-mark volumetric flask, dilute to the mark with water and mix.

3.2.8 *Standard arsenic solution* (1 mL ≡ 1 μg of arsenic). Transfer by pipette 10 mL of standard solution A to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix. Transfer by pipette 10 mL of this solution to a 100 mL one-mark volumetric flask, dilute to the mark with water and mix.

3.2.9 *Potassium permanganate solution,* $c(KMnO_4)$ = 0.02 mol/L. Dissolve 0.32 g of potassium permanganate in water and dilute to 100 mL with water.

3.2.10 *Sodium azide solution*, 50 g/L. Dissolve 5 g of sodium azide (NaN_3) in water and dilute to 100 mL with water.

WARNING NOTE. Prepare the solution in a fume cupboard as sodium azide liberates poisonous fumes in contact with water or acids. Prevent contact with skin and eyes.

3.2.11 *1,5-diphenylcarbazide solution*, 10 g/L. Dissolve 0.5 g of 1,5-diphenylcarbazide $[(C_6H_5NH.NH)_2CO]$ in 40 mL of acetone $[(CH_3)_2CO]$ containing 3 drops of sulphuric acid solution (**3.2.1**), and dilute to 50 mL with acetone. It is essential that this solution be freshly prepared.

3.2.12 *Silver diethyldithiocarbamate solution*, 5 g/L. Dissolve 1.0 g of silver diethyldithiocarbamate $[(C_2H_5)_2N.CS.SAg]$ in 200 mL of pyridine (C_5H_5N). Store in a dark glass bottle, preferably in the dark as the solution is not very stable, and renew the solution after two months.

3.2.13 *Potassium iodide solution*, 150 g/L. Dissolve 15 g of potassium iodide (KI) in water and dilute to 100 mL with water.

3.2.14 *Tin (II) chloride solution*, 336 g/L. Dissolve 40 g of tin (II) chloride dihydrate ($Sn\ Cl_2.2H_2O$) in 100 mL of hydrochloric acid solution (ρ_{20} = 1.18 g/mL).

3.2.15 *Zinc*, 20–30 mesh or granulated; arsenic-free.

3.2.16 *Hydrochloric acid solution*, (HCl) (ρ_{20} = 1.18 g/mL); arsenic-free.

3.2.17 *Lead (II) acetate solution*, 100 g/L. Dissolve 10 g of lead (II) acetate trihydrate $[(CH_3COO)_2Pb.3H_2O]$ in water and dilute to 100 mL with water.

3.3 Apparatus

3.3.1 *Volumetric glassware*, complying with the requirements for class A quality in accordance with BS 700-2 (Type 2), BS 846, BS 1583 or BS 1792, as appropriate.

3.3.2 *Spectrometer or photoelectric absorptiometer*, suitable for the measurement of absorption at wavelengths of 435 nm and 540 nm.

3.3.3 *Apparatus for arsenic determination,* as detailed in BS 4404 (see Figure 1).

3.4 Procedure

3.4.1 *Analysis of preservatives*

3.4.1.1 *General*

Transfer by pipette a suitable quantity[5)] of preservative to a 250 mL conical flask, add 50 mL of the sulphuric acid solution (**3.2.1**) and, with caution, 10 mL of hydrogen peroxide solution (**3.2.2**). Boil the contents of the flask until evolution of oxygen ceases and all the hydrogen peroxide is decomposed, add 100 mL of water, cool the flask to room temperature and transfer the contents to a 250 mL one-mark volumetric flask. Dilute to the mark with water and mix to give the test solution.

3.4.1.2 *Analysis for copper*

Transfer, by pipette, 1 mL of the test solution to a 250 mL separating funnel, dilute to 100 mL with sulphuric acid solution (**3.2.3**) and swirl to mix. Add, by pipette, 10 mL of the zinc dibenzyldithiocarbamate solution (**3.2.4**) and shake the separating funnel for 90 s. Allow the phases to separate. Run off the carbon tetrachloride layer through a dry 70 mm filter paper[6], discarding the first runnings, into a suitable cell. Measure the absorbance of the yellow complex against a reagent blank, prepared in a similar way, at a wavelength of 435 nm. Determine the copper content of the solution, by comparing the absorbance value with a calibration graph (see **3.4.3.1**).

3.4.1.3 *Analysis for chromium*

Transfer, by pipette, 1 mL of the test solution to a 25 mL beaker and gently evaporate to a small volume, but do not evaporate off the sulphuric acid. Allow the solution to cool, then add 6 mL of water, 4 mL of the sulphuric acid solution (**3.2.3**), and 0.5 mL of the potassium permanganate solution (**3.2.9**). Heat on a steam bath for 20 min, adding potassium permanganate solution dropwise as necessary, to maintain a slight excess. Then carefully add sodium azide solution (**3.2.10**) to the hot solution at the rate of about 1 drop every 10 s, swirling after each addition. Continue until the solution is clear. It is important to avoid an excess of sodium azide in the solution. Remove the beaker immediately from the steam bath and cool it to room temperature. Transfer the solution to a 100 mL one-mark volumetric flask containing 15 mL of the sulphuric acid solution (**3.2.3**) and 60 mL of water. Swirl to mix. Add 2 mL of the 1,5-diphenylcarbazide solution (**3.2.11**), dilute to the mark with water and mix. Measure the absorbance of the violet-red complex against a reagent blank, prepared in a similar way, in suitable cells at a wavelength of 540 nm. Determine the chromium content of the solution, by comparing the absorbance value with a calibration graph (see **3.4.3.2**).

3.4.1.4 *Analysis for arsenic*

Transfer, by pipette, 5 mL of the test solution to a 200 mL one-mark volumetric flask and dilute to the mark with water. Transfer, by pipette, 5 mL of this solution to the 100 mL conical flask (A in Figure 1), make up to 22 mL with water, and add 5 mL of hydrochloric acid solution (**3.2.16**), 2 mL of potassium iodide solution (**3.2.13**), and 8 drops of tin (II) chloride solution (**3.2.14**).

WARNING NOTE. The production of arsine should be carried out in a fume cupboard.

Swirl to mix and allow to stand for 15 min. Impregnate the glass wool or cotton wool in the connecting tube B (see Figure 1) with lead (II) acetate solution (**3.2.17**) and charge the absorption tube C with 4 mL of the silver diethyldithiocarbamate solution (**3.2.12**). Add 5.0 g of zinc (**3.2.15**) to the solution in the 100 mL conical flask and immediately connect the flask to the tube assembly. The evolution of arsine is 99 % complete in 30 min and virtually complete in about 40 min. Check that the volume of solution in the absorption tube is still 4 mL; if necessary, add sufficient pyridine to make up to 4 mL and mix. Measure the absorbance of the violet-red complex against a reagent blank, prepared in a similar way, in suitable cells at a wavelength of 540 nm. Determine the arsenic content of the solution, by comparing the absorbance value with a calibration graph (see **3.4.3.3**).

3.4.2 ***Analysis of treated wood***

3.4.2.1 *Preparation of timber sample for analysis*

Prepare the sample for analysis by converting the treated timber into a form suitable for extraction (i.e. shavings or sawdust) as described in Clause **5** of BS 5666-1:1987.

Divide the prepared sample into two unequal portions, (0.5 g and 5 g respectively). Determine the moisture content on the 0.5 g portion according to the procedure described in Clause **6** of BS 5666-1:1987. Reserve the 5 g sample for the analysis (**3.4.2.2**).

3.4.2.2 *Extraction and preparation of test solution*

Weigh about 5 g of the prepared test sample (**3.4.2.1**) to the nearest 0.1 g into a 250 ml conical flask, add 50 mL of the sulphuric acid solution (**3.2.1**) and, with caution, 10 ml of the hydrogen peroxide solution (**3.2.2**). Heat in a water bath at 75 °C for 30 min with occasional swirling to mix. Filter the solution through a filter paper[7] into a second conical flask and thoroughly wash the solids on the filter paper with a maximum of 100 mL of water. Boil the filtrate and washings until evolution of oxygen ceases and all the hydrogen peroxide is decomposed. Cool to room temperature and transfer to a 250 mL one-mark volumetric flask and make up to the mark with water to give the test solution.

[5] For 30 g/L solutions of preservative complying with BS 4072-1, transfer 3 mL of the solution.

[6] A Whatman No. 1 filter paper has been found to be suitable.

[7] A Whatman No. 44 filter paper has been found to be suitable.

3.4.2.3 *Measurement*

3.4.2.3.1 *Analysis for copper*

Transfer, by pipette, 1 mL of the test solution to a 250 mL separating funnel and proceed as detailed in **3.4.1.2**.

3.4.2.3.2 *Analysis for chromium*

Transfer, by pipette, 1 mL of the test solution to a 25 mL beaker and proceed as detailed in **3.4.1.3**.

3.4.2.3.3 *Analysis for arsenic*

Transfer, by pipette, 0.5 mL of the test solution to the 100 mL conical flask (A in Figure 1) and proceed as detailed in **3.4.1.4**.

3.4.3 ***Preparation of calibration graphs***

3.4.3.1 *Copper calibration*

Transfer, by pipette, portions of 2, 5, 10, 15 and 20 mL of the standard copper solution (**3.2.6**) to a series of 250 mL separating funnels. Proceed for each portion as detailed in **3.4.1.1**. The portions taken contain the equivalent of 4, 10, 20, 30 and 40 μg, respectively, of copper. Prepare the calibration graph by plotting the absorbances against micrograms of copper.

3.4.3.2 *Chromium calibration*

Transfer, by pipette, portions of 0.1, 0.2, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.3 mL of the standard chromium solution (**3.2.7**) to a series of 25 mL beakers and dilute to 5 mL with water in each case. Add 5 mL of the sulphuric acid solution (**3.2.3**) to each beaker together with 0.5 mL of the potassium permanganate solution (**3.2.9**). Proceed for each portion, from the appropriate point, as detailed in **3.4.1.2**. The portions taken contain the equivalent of 10, 20, 40, 60, 80, 100, 120 and 130 μg, respectively, of chromium. Prepare the calibration graph by plotting the absorbances against micrograms of chromium.

3.4.3.3 *Arsenic calibration*

Transfer, by pipette, portions of 1, 2, 4, 6, 8 and 10 mL of the standard arsenic solution (**3.2.8**) to a series of 100 mL conical flasks (A in Figure 1) and dilute each to 22 mL with water. Proceed for each portion, from the appropriate point, as detailed in **3.4.1.3**. The portions taken contain the equivalent of 1, 2, 4, 6, 8 and 10 μg, respectively, of arsenic. Prepare the calibration graph by plotting the absorbances against micrograms of arsenic.

3.4.4 ***Calculations***

3.4.4.1 *The concentration of metals in a preservative*

The concentration in g/100 mL, of copper or chromium or arsenic is given by:

$\frac{C}{40V}$ for copper

$\frac{C}{40V}$ for chromium

$\frac{C}{5V}$ for arsenic

where:

C is the appropriate metal content (μg) of the solution submitted to the spectrometer;

V is the quantity in mL of the preservative taken for analysis.

To express the results in terms of copper sulphate (as $CuSO_4.5H_2O$), of potassium dichromate (as $K_2Cr_2O_7$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentrations of the respective metals by the following factors:

C_{copper} by 3.93

$C_{chromium}$ by 2.83 for the potassium salt or 2.87 for the sodium salt

$C_{arsenic}$ by 1.77

3.4.4.2 *The percentages of metals in treated wood*

The percentage by mass of copper or chromium or arsenic in the dry wood is given by:

$\frac{C\ (100+h)}{4000\ m}$ for copper

$\frac{C\ (100+h)}{4000\ m}$ for chromium

$\frac{C\ (100+h)}{2000\ m}$ for arsenic

where:

C is the appropriate metal content (μg) of the solution submitted to the spectrophotometer;

h is the moisture content in percentage by mass, of the prepared timber sample (**3.4.2.1**);

m is the mass of the prepared timber sample in g.

To express the results in terms of copper sulphate (as $CuSO_4.5H_2O$), of potassium dichromate (as $K_2Cr_2O_7$), of sodium dichromate dihydrate (as $Na_2Cr_2O_7.2H_2O$) and of arsenic pentoxide (as $As_2O_5.2H_2O$), multiply the concentration of the appropriate metal by the corresponding factor given in **3.4.4.1**.

3.4.4.3 *The dry salt retentions in treated wood*

The analysis result may also be expressed as kg/m^3 if the density of the actual sample of timber is known; it is not satisfactory to take an average density figure for the species of timber concerned. Adjustments for the moisture content (see Part 1 of this standard) of the timber are also necessary if the density figure does not apply to the dry material.

The retention of the toxic ingredient of the oven-dry wood (expressed in kg/m^3) is given by:

$$\frac{T \times D}{100}$$

where:

T is the percentage by mass of toxic ingredient;

D is the oven-dry density of the wood (kg/m^3).

4 Test report

The test report shall include the following particulars:

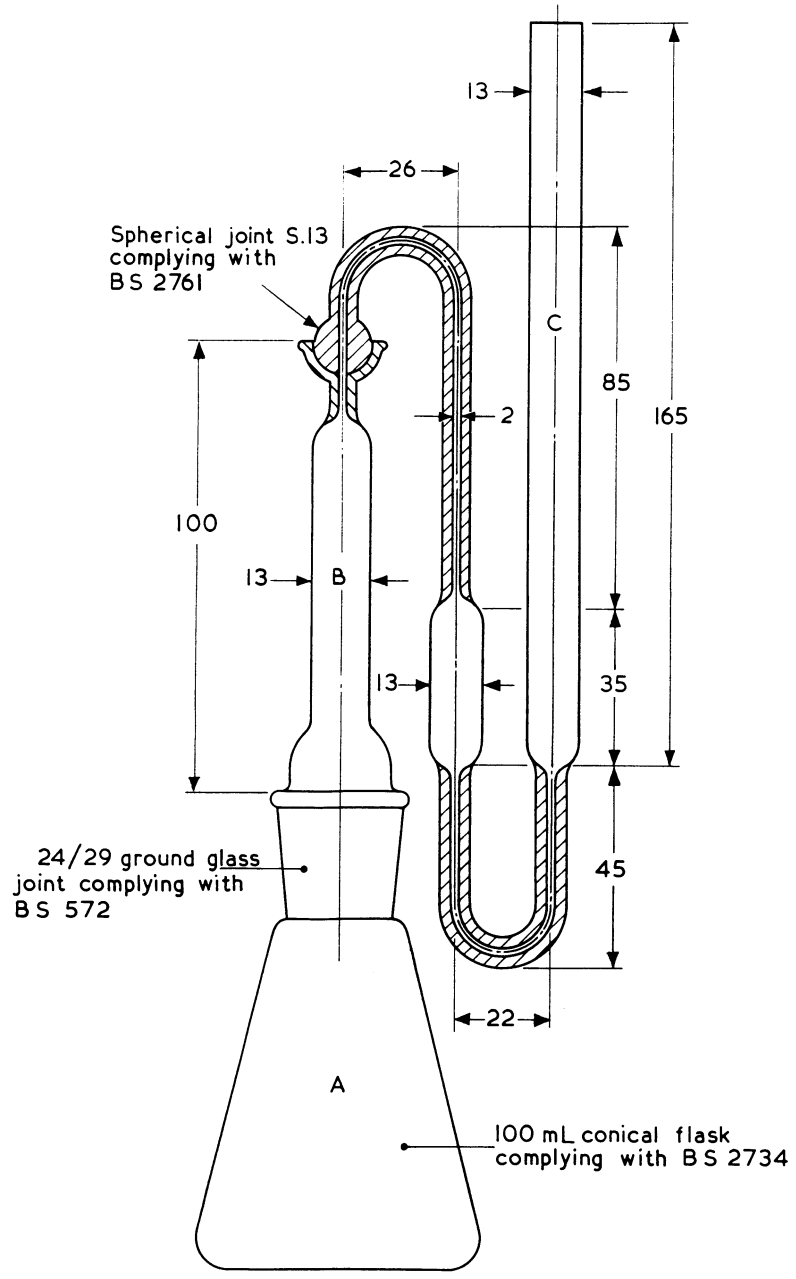
a) full identification of the sample tested and details of its preparation for analysis;

b) a reference to this British Standard and the method used, e.g. BS 5666-3:Method 2;

c) any deviation from the method described, e.g. the use of a different sample mass;

d) the results of the analysis and the method of expression used, e.g. 2.0 % by mass of copper, expressed as $CuSO_4.5H_2O$;

e) any unusual features noted during the analysis.



A = conical flask for evolution of arsine
B = connecting tube to trap hydrogen sulphide
C = absorption tube

All dimensions are in millimetres.

Figure 1 — Apparatus for arsenic determination

Publication(s) referred to

BS 572, *Specification for interchangeable conical ground glass joints.*
BS 700, *Graduated pipettes.*
BS 700-2, *Specification for pipettes for which no waiting time is specified.*
BS 846, *Specification for burettes.*
BS 1583, *Specification for one-mark pipettes.*
BS 1792, *Specification for one-mark volumetric flasks.*
BS 2734, *Specification for boiling flasks (narrow-necked), conical, flat bottom and round bottom.*
BS 2761, *Specification for spherical ground joints.*
BS 3978, *Specification for water for laboratory use.*
BS 4072, *Wood preservation by means of copper/chromium/arsenic compositions.*
BS 4072-1, *Specification for preservatives.*
BS 4404, *Method for the determination of arsenic (silver diethyldithiocarbamate procedure).*
BS 5666, *Methods of analysis of wood preservatives and treated timber.*
BS 5666-1, *Guide to sampling and preparation of wood preservatives and treated timber for analysis.*

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