BRITISH STANDARD

BS 5666-2: 1980

Methods of analysis of

Wood preservatives and treated timber —

Part 2: Qualitative analysis

NOTE It is essential that this Part is read in conjunction with Part 1 "General considerations and sampling and preparation of materials for analysis".

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, 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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Contents

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1 Scope and field of application

This Part of this British Standard details procedures for the detection of preservative materials commonly found in organic solvent type and water-borne type preservative solutions and in preservative-treated wood. The complex nature of the tar oil type preservatives precludes the use of a specific qualitative test for this group, although some indication of their presence may be provided by odour and appearance characteristics.

The procedures described in this Part will frequently be applied before carrying out quantitative analyses using methods described in other Parts of this standard. Two types of qualitative test are described, those carried out directly on timber sections and those requiring prior preparation of an extract from the timber sample. Some preservative materials can be detected by either type of method, others by only one of them. In addition to the detection of preservative materials, the spray tests described may be used to estimate the penetration of the preservatives.

Although these tests have been validated for the types of preservative in current use, the introduction of new materials could give rise to difficulties and analysts should be alert to this possibility.

2 References

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

3 Preliminary inspection of the sample

3.1 General. Much information can be gained from a careful inspection of the sample. Aged or partially decayed samples may have a misleading appearance and should be examined thoroughly.

3.2 Visual examination. A wide variety of colouring materials may be present in preservative formulations, so colour can only be considered as a guide.

Note the colour of the sample; the following inferences may be drawn.

a) *Green colouration.* The presence of copper naphthenate (see **3.3**), copper/chromium/arsenic, or fluorine/chromium/arsenic preservative should be suspected. Copper naphthenate gives a bright blue-green coloration, copper/chromium/arsenic salts give a dark green or brown coloration. Fluorine/chromium/arsenic salts give a yellow-green coloration.

b) *Brown colouration.* This may indicate the presence of creosote or occasionally of zinc naphthenate. Care should be taken to differentiate between a brown colouration and general staining or accumulated dirt.

c) *Natural wood colour.* With the exception of zinc naphthenate, the materials mentioned in items a) and b) are almost certainly absent.

3.3 Odour. The sense of smell varies considerably with the individual, but it is generally possible to detect the presence of certain preservatives by their odour.

Smell the sample and, if appropriate, compare its odour with that of dilute solutions of appropriate preservative.

Creosote, naphthenic acids (present as the copper or zinc compounds) and tri-*n*-butyltin oxide all have characteristic odours. In addition, a "solvent smell" may indicate treatment with an organic solvent type preservative.

3.4 Differentiation of heartwood and sapwood in *Pinus* species. This is sometimes useful in the interpretation of the qualitative tests, for instance some specifications require the whole of the sapwood zone to be penetrated by preservative solution following treatment. The differentiation of the heartwood and sapwood of most *Pinus* species can be achieved by a simple colour change reaction, details of which are given in Appendix A.

4 Qualitative tests on timber sections

4.1 Principle. Copper, chromium, boron, fluoride, tin, zinc and pentachlorophenol are detected by spray or brush application of an appropriate chromogenic material to the timber surface.

NOTE 1 Preservative solutions may also be tested by absorbing them on to an ashless or chromatographic grade filter paper.
NOTE 2 The tests described are not always ideal but are the best available at the present time. In particular, it should be noted that the presence of other compounds may interfere with or mask the development of the distinguishing colour (positive response) and that treated wood may give a negative response because the level of preservative is below the detection limit of the reagent.

4.2 Reagents. All reagents shall be of recognized analytical reagent quality and water complying with the requirements of BS 3978 shall be used throughout.

Ethanol (absolute) complying with the requirements of BS 507 or industrial methylated spirits[1)] 99 % (*V*/*V*) (74 degrees O.P.) complying with the requirements of BS 3591 shall be used to make up the reagents where ethanol is specified.

4.2.1 *Dithio-oxamide solution,* 5 g/l. Dissolve 0.5 g of dithio-oxamide in 100 ml of ethanol.

NOTE Dithio-oxamide is also known by the name "rubeanic acid"; the relevant test involving this reagent (**4.4.2.1**) is referred to as the "Rubeanic acid test".

4.2.2 *Sodium acetate solution,* 50 g/l. Dissolve 5 g of sodium acetate trihydrate in 100 ml of water.

4.2.3 *Diethylammonium diethyldithiocarbamate solution,* 5 g/l. Dissolve 0.5 g of diethylammonium diethyldithiocarbamate in 100 ml of acetone.

4.2.4 *1,5-diphenylcarbazide solution,* 5 g/l. Continuously add, with stirring and cooling, 50 ml of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/ml) to 450 ml of water and dilute to 1 litre with ethanol. Dissolve 0.5 g of 1,5-diphenylcarbazide in 100 ml of this solution.

4.2.5 *Curcumin solution.* Continuously add, with stirring and cooling, 100 ml of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/ml) to 800 ml of ethanol and dilute to 1 litre with ethanol. Dissolve 0.25 g of curcumin and 10 g of salicylic acid in 100 ml of this solution.

4.2.6 *Zirconyl chloride solution,* 10 g/l. Dissolve 1 g of zirconyl chloride octahydrate in 100 ml of water.

4.2.7 *Sodium alizarinsulphonate solution,* 4 g/l. Dissolve 1 g of sodium alizarinsulphonate in 20 ml of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/ml) and dilute to 250 ml with water.

4.2.8 *Catechol violet (pyrocatechol sulphonphthalein) solution,* 5 g/l. Dissolve 0.1 g of catechol violet in 100 ml of ethanol.

4.2.9 *Bromopyrogallol red (dibromopyrogallol sulphonphthalein) solution,* 2 g/l. Dissolve 0.2 g of bromopyrogallol red in 100 ml of ethanol.

4.2.10 *Dithizone solution,* 1 g/l. Dissolve 0.1 g of dithizone in 100 ml of toluene.

4.2.11 *Triammonium citrate solution,* 24 g/l. Dissolve 2.4 g of *tri*ammonium citrate in 100 ml of water.

4.2.12 *4-Aminophenazone solution,* 2 g/l. Dissolve 0.2 g of 4-aminophenazone in 100 ml of water.

4.2.13 *Potassium ferricyanide [potassium hexacyanoferrate (III)] solution,* 100 g/l. Dissolve 10 g of potassium ferricyanide in 100 ml of water.

4.3 Apparatus

4.3.1 *Spray equipment,* capable of producing a fine even spray. Both gas-powered aerosol units and small hand-powered units have been found to be suitable.

4.3.2 *Soft paint brush,* 5 mm to 50 mm wide, depending on the size of the timber section to be tested.

4.3.3 *Source of infra-red or broad spectrum ultraviolet radiation.*

4.4 Procedure

4.4.1 *General.* Apply the reagent to the sample of wood by brushing, spraying or by dipping the sample in the reagent solution.

NOTE Spraying has the advantage of ready control of the quantity of reagent applied and this method is referred to, for simplicity, in the procedures given in **4.4.2** to **4.4.8**.

Prepare the sample to give a clean surface and remove any loose sawdust. Exercise care so as not to remove superficial preservative treatments and to avoid smearing the preservative from treated to untreated areas, particularly when examining freshly treated timber.

If paint or other finishes have been applied to the timber, the whole of the surface coating should, if possible, be removed before sampling. If this is not practical, i.e. in the case of a superficial treatment where removal of the film may remove all traces of preservative, it should be borne in mind when interpreting the results that copper and zinc are common components in many finishes.

In testing, avoid an excess of reagent as this may mask the result.

CAUTION. It is advisable to wear protective goggles and work under a fume hood when carrying out the spray tests.

4.4.2 *Copper/chromium/arsenic (CCA) preservatives or copper naphthenate preservatives: detection of the copper component*

4.4.2.1 *Rubeanic acid test.* Spray the sample successively with the dithio-oxamide solution (**4.2.1**) and the sodium acetate solution (**4.2.2**).

The development of a dark green colour indicates the presence of copper; a pale yellow colour indicates that if copper is present the amount is below the limit of detection.

4.4.2.2 *Diethylammonium diethyldithiocarbamate test.* Spray the sample with the diethylammonium diethyldithiocarbamate solution (**4.2.3**).

The development of a dark green colour indicates the presence of copper; a pale yellow colour indicates that if copper is present the amount is below the limit of detection.

NOTE This reagent is particularly useful on hardwoods.

[1] It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230) as amended by The Alcoholic Liquors (Amendment of Units and Methods of Measurement) Regulations 1979 (S.I. 1979, No. 1149). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

4.4.3 *Fluorine/chromium/arsenic (FCA) preservatives: detection of the chromium component.* Spray the sample with the 1,5-diphenylcarbazide solution (**4.2.4**).

The development of a deep violet colour indicates the presence of hexavalent chromium; a yellow colour indicates that if hexavalent chromium is present the amount is below the limit of detection.

NOTE This method will produce satisfactory results only on timbers examined within four weeks of treatment. This reagent also gives a colour with copper-containing materials. For the detection of chromium in copper/chromium/arsenic (CCA) or copper/chromium (CC) preservatives, see **5.2**.

4.4.4 *Borates or organoboron preservatives: detection of boron.* Spray the sample with the curcumin solution (**4.2.5**).

The development of a red colour indicates the presence of boron; a pale yellow colour indicates that if boron is present the amount is below the limit of detection.

4.4.5 *Fluoride and bifluoride preservatives: detection of fluoride.* Add 10 ml of the zirconyl chloride solution (**4.2.6**) to 10 ml of the sodium alizarinsulphonate solution (**4.2.7**). Spray the sample with this mixture. It is essential to examine the sample within a few seconds of spraying.

The development of a bright yellow colour indicates the presence of fluoride; a red colour indicates that if fluoride is present the amount is below the limit of detection.

4.4.6 ***Tri-n-butyltin oxide preservatives: detection of tin compounds***

4.4.6.1 *Catechol violet test.* Spray the sample with the catechol violet solution (**4.2.8**), irradiate with infra-red or broad spectrum ultraviolet radiation for at least 1 h and respray with the catechol violet solution.

The development of a blue colour indicates the presence of tin; a pale yellow colour indicates that if tin is present the amount is below the limit of detection.

4.4.6.2 *Bromopyrogallol red test.* Spray the sample with the bromopyrogallol red solution (**4.2.9**). Irradiate with infra-red or broad spectrum ultraviolet radiation for at least 1 h and respray with the bromopyrogallol red solution.

The development of a blue colour indicates the presence of tin; a red colour indicates that if tin is present the amount is below the limit of detection.

4.4.7 *Zinc naphthenate preservatives: detection of zinc.* Spray the sample with the dithizone solution (**4.2.10**).

The development of a bright pink colour indicates the presence of zinc; a pale green colour indicates that if zinc is present the amount is below the limit of detection. A pale pink colour is sometimes obtained in the absence of zinc naphthenate preservative.

4.4.8 *Detection of pentachlorophenol.* Spray the sample with the *tri*ammonium citrate solution (**4.2.11**). Wait 10 min and successively spray the sample with the 4-aminophenazone solution (**4.2.12**) and the potassium ferricyanide solution (**4.2.13**).

The development of a green colour indicates the presence of pentachlorophenol; a yellow colour indicates that if pentachlorophenol is present the amount is below the limit of detection.

5 Qualitative tests on extracts from treated timber and preservative solutions

5.1 Thin layer chromatography (TLC) for the detection of organic and organometallic preservatives

5.1.1 *Principle.* Pentachlorophenol, pentachlorophenyl laurate, γ-hexachlorocyclohexane, dieldrin, 2-phenylphenol, 1-chloronaphthalene, polychloronaphthalene, zinc naphthenate, copper naphthenate and tri-*n*-butyltin oxide are extracted from timber samples with chloroform. The anti-stain agent sodium pentachlorophenoxide is extracted with a 5 % (*V*/*V*) solution of acetic acid in methanol. The preservatives present in the extract or in preservative solutions are identified using thin layer chromatography.

5.1.2 *Reagents.* All reagents shall be of recognized analytical reagent quality and water complying with the requirements of BS 3978 shall be used throughout.

Ethanol (absolute) complying with the requirements of BS 507 or methylated spirits[2)] 99 % (*V*/*V*) (74 degrees O.P.) complying with the requirements of BS 3591 shall be used to make up the reagents where ethanol is specified.

5.1.2.1 *Silver nitrate solution,* 2.5 g/l. Dissolve 0.25 g of silver nitrate in 100 ml of an aqueous solution containing 66 % (*V*/*V*) acetone.

[2)] It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230) as amended by The Alcoholic Liquors (Amendment of Units and Methods of Measurement) Regulations 1979 (S.I. 1979, No. 1149). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

5.1.2.2 *Catechol violet solution,* 0.5 g/l. Dissolve 0.05 g of catechol violet in 100 ml of ethanol.

5.1.2.3 *Chrome azurol S solution.* Dissolve 0.5 g of chrome azurol S and 5 g of sodium acetate in 100 ml of water.

5.1.2.4 *Dithizone solution,* 0.1 g/l. Dissolve 0.01 g of dithizone in 100 ml of chloroform.

5.1.2.5 *Developing solvent 1.* Add 66 ml of ethyl acetate to 33 ml of glacial acetic acid and mix.

5.1.2.6 *Developing solvent 2.* Thoroughly mix 75 ml of cyclohexane, 15 ml of acetone and 10 ml of liquid paraffin (colourless).

5.1.2.7 *Acetone solution,* 80 % (*V*/*V*). Dilute 80 ml of acetone to 100 ml with water.

5.1.2.8 *Acetic acid solution,* 5 % (*V*/*V*) *in methanol.* Dilute 5 ml of glacial acetic acid to 100 ml with methanol.

5.1.2.9 *Standard solutions.* For each preservative, except for sodium pentachlorophenoxide, prepare a solution by dissolving 0.5 g of the active ingredient in 100 ml of chloroform; dissolve sodium pentachlorophenoxide in the acetic acid solution (**5.1.2.8**).

5.1.3 ***Apparatus***

5.1.3.1 *Chromatographic tanks.* Lined with chromatography paper[3)].

5.1.3.2 *Pre-coated chromatography plates,* free from fluorescent indicator. Cellulose (0.1 mm layer thickness) and silica gel (0.25 mm layer thickness) are required. Plates 200 mm long are suitable.

5.1.3.3 *Disposable micropipettes,* e.g. of 1 pl and 5 µl capacity.

5.1.3.4 *Spray units,* capable of delivering a very fine light spray.

5.1.3.5 *Source of broad spectrum ultraviolet radiation.*

5.1.4 *Procedure, lower sensitivity method.* Where possible, use sapwood areas of the wood sample.

5.1.4.1 *Extraction technique.* Remove the surface of the sample with a chisel, rasp, or microtome and place the shavings in a 50 ml beaker. Normally, sufficient shavings should be prepared to half fill the beaker. Saturate the shavings with chloroform, cover the beaker with a watch glass and heat on a hot plate at a temperature of 50 °C for 10 min.

It is essential that the shavings remain saturated with chloroform during the extraction but the final volume of the extract should be kept to a minimum to ensure maximum sensitivity. Allow the extract to cool, then apply to the TLC plates as described in **5.1.4.2**.

If the presence of the anti-stain agent, sodium pentachlorophenoxide, is suspected, extract with the acetic acid/methanol solution (**5.1.2.8**) and examine the extract as described for the detection of pentachlorophenol (see **5.1.4.4**). If both extractants are used, carry out the chloroform extraction first, to enable differentiation between free pentachlorophenol soluble in chloroform and sodium pentachlorophenoxide extracted into the acetic acid/methanol solution.

5.1.4.2 *Preparation of plates.* Prepare the silica gel and the cellulose plates as follows. With a soft pencil, lightly rule lines 20 mm from each end of the 200 mm TLC plate. Make small pencilled crosses, spaced at least 10 mm apart, along one line on each plate (the base line). Label each cross lightly with the identification number of the standard solution or sample that will be applied to it. Spot the timber extracts or preservative solutions on to each plate at their labelled stations, an equal volume of the timber extract being applied to each plate using the micropipettes (**5.1.3.3**). Similarly, spot suitable standard solutions (**5.1.2.9**) to their labelled stations. Dry the plates in a gentle current of air.

5.1.4.3 *Detection of organometallic preservatives.* Place the prepared cellulose plates in a chromatographic tank containing 10 mm depth of the developing solvent 1 (**5.1.2.5**) so that the base line of the plate is about 10 mm above the level of the solvent in the tank.

Allow the solvent front to travel about 160 mm, i.e. to the region of the second pencil line. Remove the plate from the tank and dry it in a gentle air stream. Expose the plate to broad spectrum ultraviolet radiation for 30 min.

Divide the plate into three approximately equal portions with two pencil lines parallel to the base line. Spray the lower third of the plate, nearest the base line, with the dithizone solution (**5.1.2.4**) to detect the zinc as a pink streak on a blue-green background. Spray the centre third of the plate with the chrome azurol S solution (**5.1.2.3**) to detect the copper as a blue streak on a pale red background. Spray the upper third of the plate with the catechol violet solution (**5.1.2.2**) to detect the tin as a pale blue spot on a yellow background. The R_F values obtained using this system are given in Table 1 and detection limits are given in Table 2.

[3)] A Whatman No. 1 paper has been found to be suitable.

5.1.4.4 *Detection of other organic preservatives.* Place the prepared silica gel plate in a chromatographic tank containing the developing solvent 2 (**5.1.2.6**) to a depth of 10 mm, so that the base line of the plate is about 10 mm above the level of the solvent in the tank.

Allow the solvent front to travel about 160 mm, to the region of the second pencil line. Remove the plate from the tank and dry it in a gentle stream of air. Expose the plate to broad spectrum ultraviolet radiation for 30 min. At this stage remove the plate from the radiation source and examine it; 2-phenylphenol is indicated by the presence of a purple-brown spot. Spray the plate with the silver nitrate solution (**5.1.2.1**) and expose to filtered ultraviolet radiation with a wavelength of 300 nm to 400 nm for 30 min.

Examine the plate for dark brown spots on a light brown background, and identify the preservatives present by reference to the standards on the plate and to the appropriate R_F values given in Table 1. Table 2 gives the appropriate detection limits for this procedure.

5.1.5 *Procedure when higher sensitivity is required.* The method described in **5.1.4** is adequate for most routine procedures. However, if greater sensitivity is required, the following more elaborate technique shall be applied.

5.1.5.1 *Use of prewashed plates.* Allow the acetone solution (**5.1.2.7**) to traverse the full length of the plate in a chromatographic tank. This decreases the background colouration of the plates by removal of impurities. Prewashing also changes the observed R_F values and typical values found on washed plates are given in Table 1. The detection limits when using washed plates are given in Table 2.

5.1.5.2 *Longer irradiation time.* For the organic preservatives, the sensitivity can be improved by lengthening the second period of exposure to ultraviolet radiation from 30 min to 60 min.

5.1.5.3 *Use of back lighting.* The sensitivities achieved on washed plates can be improved slightly by illuminating from behind.

5.1.5.4 *Use of a Stahl oven.* For very small samples (10 mg to 15 mg), where extraction is difficulty, heating in a Stahl oven is recommended to spot the plate. The principles of operation are shown in Figure 1. All volatile material is transferred to the plate. The method avoids contamination by wood extractives but does not permit detection of copper napthenate or zinc naphthenate.

5.1.6 *Interpretation of results.* Strictly interpreted, the use of a single TLC system cannot be assumed to provide unequivocal evidence that a specific component is present in the formulation under examination. In most cases, however, this will be sufficient. For confirmation of a component's identity, a second TLC system shall be used. This is conveniently achieved by using both the washed and unwashed plate systems described in **5.1.4** and **5.1.5**.

Commercial preparations of pentachlorophenol often contain lower chlorophenols as impurities; these will resolve themselves on the TLC plate as a series of shadow spots directly ahead of the pentachlorophenol spot.

5.2 Specific test for chromium in copper/chromium/arsenic (CCA), fluorine/chromium/arsenic (FCA) and copper/chromium (CC) preservative-treated timber

5.2.1 *Principle.* Chromium is extracted from timber samples and detected in solution by formation of a blue complex.

Table 1 — R_F values

Compound	Plate	Developing solvent (see **5.1.2.5** and **5.1.2.6**)	R_F	
			Unwashed plate	Washed plate
Tri-*n*-butyltin oxide	Cellulose	1	0.98	1.00
Copper naphthenate	Cellulose	1	0.89	0.80
Zinc naphthenate	Cellulose	1	0.75	0.30
2-phenylphenol	Silica gel	2	0.21	0.37
Pentachlorophenol	Silica gel	2	0.25	0.32
γ-hexachlorocyclohexane	Silica gel	2	0.50	0.69
Dieldrin	Silica gel	2	0.61	0.76
1-chloronaphthalene	Silica gel	2	0.68	0.85
Polychloronaphthalene	Silica gel	2	0.78	0.94
Pentachlorophenyl laurate	Silica gel	2	0.87	0.96

Table 2 — Detection limits

Compound	Detection limit	
	Normal procedure (unwashed plates)	High sensitivity procedure (washed plates)
	µg	µg
Tri-*n*-butyltin oxide	0.04	0.015
Copper naphthenate	0.07	0.03
Zinc naphthenate	0.05	0.01
2-phenylphenol	0.70	0.40
Pentachlorophenol	0.06	0.025
γ-hexachlorocyclohexane	1.0	0.40
Dieldrin	0.45	0.20
1-chloronaphthalene	0.2	0.2
Polychloronaphthalene	0.85	0.10
Pentachlorophenyl laurate	0.30	0.30

5.2.2 *Reagents.* All reagents shall be of recognized analytical reagent quality and water complying with the requirements of BS 3978 shall be used throughout.

5.2.2.1 *Concentrated sulphuric acid,* (ρ_{20} = 1.84 g/ml).

WARNING NOTE. Sulphuric acid (ρ_{20} = 1.84) is corrosive and causes burns. Care should be taken to avoid its contact with eyes and skin.

5.2.2.2 *Sulphuric acid solution,* 0.5M. Continuously add, with stirring and cooling, 28 ml of concentrated sulphuric acid (ρ_{20} = 1.84 g/ml) to 800 ml of water. Cool and dilute to 1 litre with water.

5.2.2.3 *Hydrogen peroxide solution,* 300 g/l (100 volumes).

5.2.2.4 *Sodium hydroxide solution,* 5M. Dissolve 40 g of sodium hydroxide pellets in 150 ml of water. Cool and dilute to 200 ml with water.

5.2.2.5 *Isopentyl acetate (isoamyl acetate).*

5.2.3 ***Apparatus***

5.2.3.1 *Hotplate,* thermostatically controlled.

5.2.3.2 *Stirrer,* of either the magnetic or mechanical type.

5.2.4 *Procedure.* Take 3 g of timber using a rasp, chisel or microtome. Place the shavings in a 250 ml beaker, add 50 ml of the sulphuric acid solution (**5.2.2.2**) and 5 ml of the hydrogen peroxide solution (**5.2.2.3**). Cover with a watch glass, boil for 20 min, cool and filter. Add 14 ml of the sodium hydroxide solution (**5.2.2.4**) and 6 ml of the hydrogen peroxide solution (**5.2.2.3**) to the filtrate and boil for about 15 min or until all effervescence ceases, whichever is the longer. Cool and transfer 6 ml of the mixture to a 25 ml beaker fitted with a stirrer (**5.2.3.2**). Add 15 drops of the concentrated sulphuric acid (**5.2.2.1**) and enough of the *iso*pentyl acetate (**5.2.2.5**) to form a layer 10 mm deep above the extract. Start the stirrer and add 4 ml of the hydrogen peroxide solution (**5.2.2.3**).

A blue colouration in the sample and subsequently in the *iso*pentyl acetate layer indicates the presence of chromium.

5.3 Specific test for arsenic in copper/chromium/arsenic (CCA) and fluorine/chromium/arsenic (FCA) preservative-treated timber

5.3.1 *Principle.* Arsenic is liberated as arsine from treated timber samples. The arsine is detected by its formation of a red complex with silver diethyldithiocarbamate.

5.3.2 *Reagents.* All reagents shall be of recognized analytical reagent quality and water complying with the requirements of BS 3978 shall be used throughout.

5.3.2.1 *Zinc,* 20 to 30 mesh or granulated, arsenic-free.

5.3.2.2 *Silver diethyldithiocarbamate solution,* 5 g/l. Dissolve 1.0 g of silver diethyldithiocarbamate in 200 ml of chloroform.

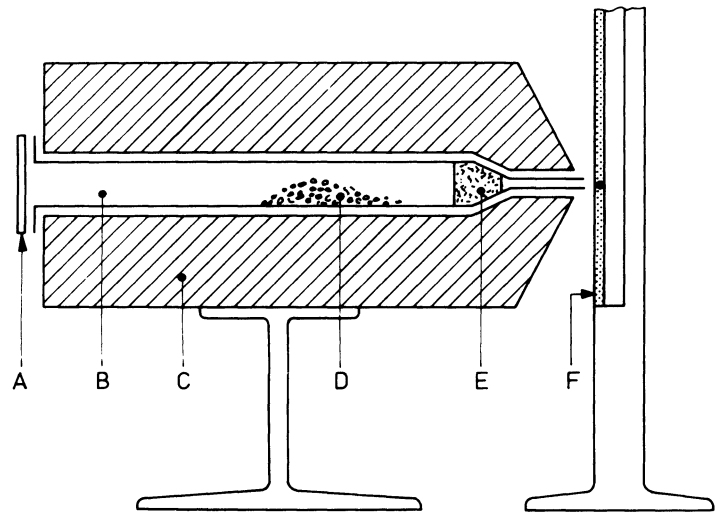
5.3.2.3 *Lead acetate solution,* 100 g/l. Dissolve 10 g of lead acetate in water and dilute to 100 ml with water.

5.3.2.4 *Hydrochloric acid solution,* 5M. Continuously add, with stirring and cooling, 455 ml of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/ml) to 400 ml of water. Cool and dilute to 1 litre with water.

5.3.3 *Apparatus.* The apparatus required is shown in Figure 2.

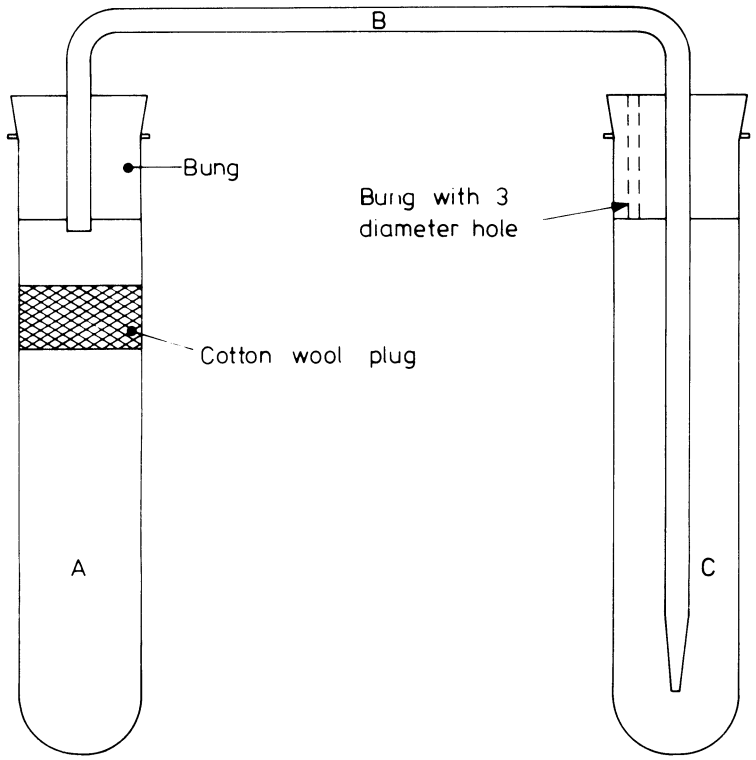
5.3.4 *Procedure.* Take 0.1 g to 0.2 g of the sample with a chisel, rasp, or microtome. Place the shavings in the boiling tube A (see Figure 2) together with 2 g of the zinc (**5.3.2.1**). Measure 5 ml of the silver diethyldithiocarbamate solution (**5.3.2.2**) into the boiling tube C. Select a piece of cotton wool that fits snugly into the boiling tube A and impregnate it with the lead acetate solution (**5.3.2.3**). Add 10 ml of the hydrochloric acid solution (**5.3.2.4**) to the boiling tube A, place the impregnated cotton wool in position (see Figure 2) and quickly secure the two bungs and the connecting tube B in the boiling tubes A and C. Effervescence should be visible in the boiling tube A and bubbles of gas should be seen issuing from the end of the connecting tube in the boiling tube C.

If arsenic is present, a red colouration will develop in the silver diethyldithiocarbamate solution within 5 min. The colour will intensify if the gas is allowed to continue bubbling through the solution.



A Seal (silicone membrane)
B Glass cartridge
C Heating block
D Sample
E Glass-wool
F Thin-layer chromatographic layer

Figure 1 — The Stahl oven



The dimension is in millimetres.

Figure 2 — Apparatus for qualitative arsenic determination

Appendix A Differentiation of heartwood and sapwood in *Pinus* species

A.1 Principle

The phenolic compounds in the heartwood of *Pinus* species are detected by the formation of a coloured complex with *o*-anisidine.

A.2 Reagents

Reagents shall be of recognized analytical quality and water complying with the requirements of BS 3978 shall be used throughout.

A.2.1 *o-Anisidine solution,* 5 g/l. Add 9 ml of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/ml) to 400 ml of water in a 500 ml measuring cylinder. Dilute to 500 ml with water. Dissolve 0.5 g of *o*-anisidine in 100 ml of this solution.

A.2.2 *Sodium nitrite solution,* 100 g/l. Dissolve 10 g of sodium nitrite in 100 ml of water.

A.3 Apparatus

Use the apparatus described in **4.3.1** or **4.3.2**.

A.4 Procedure

A.4.1 *General.* Proceed as described in **4.4.1**.

NOTE Problems with smearing do not occur.

CAUTION. It is advisable to wear protective goggles and work under a fume hood when carrying out spray tests.

A.4.2 *Colour test.* Mix equal volumes of the *o*-anisidine solution (**A.2.1**) and the sodium nitrite solution (**A.2.2**) and brush or spray the mixture on to the timber section.

Heartwood of most *Pinus* species is coloured red and sapwood is coloured pale yellow.

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Publications referred to

BS 507, *Ethanol.*
BS 3591, *Industrial methylated spirits.*
BS 3978, *Water for laboratory use.*

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