BRITISH STANDARD

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Methods of analysis of

Wood preservatives and treated timber —

Part 6: Quantitative analysis of preservative solutions and treated timber containing pentachlorophenol, pentachlorophenyl laurate, γ-hexachlorocyclohexane and dieldrin

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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Committees responsible for this British Standard

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Association of Consulting Scientists
British Drier Manufacturers' Association
British Wood Preserving Association
Department of the Environment (Building Research Establishment, Princes Risborough Laboratory)
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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 Scope and field of application

This Part of BS 5666 describes procedures for the quantitative determination of pentachlorophenol (PCP), pentachlorophenyl laurate (PCPL), γ-hexachlorocyclohexane (γ-HCH) and dieldrin present in preservative solutions and treated wood. Both colorimetric and gas-liquid chromatographic (GLC) methods are given for the determination of PCP and PCPL but only a GLC method is given for the determination of γ-HCH and dieldrin as no suitable specific chemical methods are available for these chlorinated hydrocarbon insecticides.

The GLC method is suitable for the determination of the total amount of PCP, PCPL, γ-HCH and dieldrin within a single sample of wood. It may be used to study the distribution of these components through such a sample. Since the preservative solution, or the solution obtained by extraction of treated timber, is diluted to a suitable concentration for analysis, a wide range of concentrations may be assessed. In treated timber, at the lower end of the range, loadings down to 0.001 % (*m*/*m*) have been determined by this method. However, at very low loadings, where little or no dilution is required, it is necessary to consider the possibility of interference from what are normally minor constituents of the system (e.g. wood extractives or solvent impurities).

The colorimetric method is suitable for the determination of PCP or PCPL in solutions or extracts from treated timber containing γ-HCH, dieldrin, tributyltin oxide (TBTO), copper naphthenate, zinc naphthenate, *di*sodium octaborate and wood extractives.

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

CAUTION. Attention is drawn to the general safety precautions mentioned in clause **4** of Part 1 of this British Standard and also to the specific hazard warnings given in **2.2.7**, **2.2.12** and **2.3.2** of this Part.

2 Method I. Gas-liquid chromatographic method

2.1 Principle. PCP is extracted from treated wood with ethanol and preservative solutions containing PCP are diluted with ethanol before being methylated with diazomethane to produce pentachlorophenyl methyl ether. The resultant solutions are further diluted with hexane. γ-HCH and dieldrin are extracted from treated wood with ethanol and preservative solutions containing γ-HCH and dieldrin are diluted with ethanol before being further diluted with hexane.

Preservative solutions containing PCPL are saponified with morpholine and subsequently acidified to produce free PCP. For treated wood, the saponification is accomplished using morpholine as the extractant. The PCP is extracted into xylene and diluted with ethanol. Methylation and further dilution follow as for PCP.

The active components in all diluted solutions are determined using GLC with an electron-capture detector. Aldrin is used as an internal standard for PCP and γ-HCH, and 2,2-bis (4-chlorophenyl)-1,1-dichloroethylene (*p,p′*DDE) is similarly used for dieldrin.

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

Check the solvents for purity by passing samples through the chromatograph under the conditions of the determination. If a response is obtained on the chromatogram that is likely to cause significant errors in the determination on the test sample, redistil the solvent until a satisfactory response is obtained.

2.2.1 *Hexane*

2.2.2 *Ethanol*, complying with BS 507 or *industrial methylated spirits*, complying with BS 3591[1)].

2.2.3 *Morpholine*

2.2.4 *Xylene*

2.2.5 *Hydrochloric acid*, concentrated (ρ = 1.18 g/mL).

2.2.6 *Sodium hydroxide solution*, c(NaOH) = 0.1 mol/L approximately. Dissolve 4 g of sodium hydroxide in 1 L of water.

[1)] The ethanol used as a reagent in this determination may be replaced for this purpose by industrial methylated spirits, 95 % (*V*/*V*), complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by the Methylated Spirits Regulations 1983 (S.I. 1983, No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquors Duties Act 1979, Section 10, for purposes for which industrial methylated-spirits is an acceptable alternative to ethanol.

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2.2.7 *Diazomethane solution*[2]. Prepared as follows.

Dissolve 0.9 g of potassium hydroxide in 22 mL of ethanol (**2.2.2**) and add the solution slowly to a solution of 5 g of *N*-methyl-*N*-nitrosotoluene-4-sulphonamide in 70 mL of diethyl ether at 0 °C. Allow the resulting solution to stand for 5 min, transfer the flask containing the reaction mixture to a water bath and fit an efficient water-cooled condenser. Raise the temperature of the water bath to distil over the contents of the flask and collect the distillate in a flask cooled to below 30 °C. Continue the distillation until the reaction mixture appears colourless. The yellow distillate is an ethereal solution of diazomethane.

NOTE Diazomethane can also be prepared from *N*-methyl-*N′*-nitro-*N*-nitrosoguanidine and aqueous alkali. (See *Anal. Chem*., 1973, **45**, 2302.)

WARNING. Diazomethane is highly toxic and solutions may explode on contact with ground glass joints, sharp glass edges or in direct sunlight. It is imperative that all work involving diazomethane up to the final dilution is carried out behind a safety shield in an efficient fume cupboard. A diazomethane generator with smooth edges and joints throughout is commercially available and is recommended. It is essential that the preparation and use of diazomethane is carried out only by properly trained personnel.

2.2.8 *Standard aldrin solution*, 1 mg/L. Weigh 0.1000 g of aldrin into a 100 mL one-mark volumetric flask. Add approximately 50 mL of ethanol (**2.2.2**), shake well to dissolve and make up to the mark with ethanol. Dilute 5 mL of this solution to 100 mL in a one-mark volumetric flask with hexane (**2.2.1**). Shake well and similarly dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask.

2.2.9 *Standard PCP solution*, 1 mg/L. Weigh 0.1000 g of PCP, 99 % pure[3], into a 100 mL one-mark volumetric flask. Add approximately 50 mL of ethanol (**2.2.2**), shake well to dissolve and make up to the mark with ethanol. Dilute 5 mL of this solution to 100 mL in a one-mark volumetric flask using the same solvent. Shake well and similarly dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask.

2.2.10 *Standard γ-HCH solution*, 0.1 mg/L. Weigh 0.1250 g of γ-HCH and transfer quantitatively to a 500 mL one-mark volumetric flask, washing-in with ethanol (**2.2.2**), shake well to dissolve and make up to the mark with ethanol. Dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask using hexane (**2.2.1**). Shake well and similarly dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask.

2.2.11 *Standard p,p′-DDE solution*, 0.25 mg/L. Weigh 0.1250 g of *p,p′*-DDE into a 100 mL one-mark volumetric flask. Add approximately 50 mL of ethanol (**2.2.2**), shake well to dissolve and make up to the mark with ethanol. Dilute 5 mL of this solution to 250 mL with hexane (**2.2.1**) in a 250 mL one-mark volumetric flask. Shake well and similarly dilute 5 mL of this solution to 500 mL in a one-mark volumetric flask.

2.2.12 *Standard HEOD solution* (for dieldrin analysis), 0.1 mg/L. Weigh 0.1250 g of 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-exo-1,4,-endo-5,8,-dimethanonaphthalene, (HEOD)[4] and transfer quantitatively to a 500 mL one-mark volumetric flask, washing in with ethanol (**2.2.2**), shake well to dissolve and make up to the mark with ethanol. Dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask with hexane (**2.2.1**). Shake well and similarly dilute 5 mL of this solution to 250 mL in a one-mark volumetric flask.

WARNING. All of the organochlorine compounds referred to in **2.2.8** to **2.2.12** are toxic and have to be handled with care to avoid ingestion or skin contact. p,p′-DDE is a suspected carcinogen.

2.3 Apparatus

2.3.1 *Volumetric glassware*, complying with the requirements for class A of BS 1583 or BS 1792, as appropriate.

2.3.2 *Electron-capture detector gas-liquid chromatograph* with the following characteristics:

a) column temperature 170 °C;

b) detector temperature 250 °C;

c) glass-lined injector zone, with silicone rubber septum and maintained at 190 °C to 200 °C;

d) glass column, approximately 1.5 m in length and 4 mm i.d., packed with 80-100 mesh (50 µm to 175 µm) chromosorb W AW/DMCS carrying 5 % of QF 1 and fitted with a silanized glass-wool plug at its head[5];

e) detector type, ^{63}Ni electron-capture detector, pulsed-mode or direct current type;

f) suitable recorder.

WARNING. The electron-capture detector contains radioactive material and it is essential that it is used in accordance with the manufacturer's instructions.

[2] For reference see A.I. Vogel *A textbook of practical organic chemistry*, p291, Longmans 1978.
[3] Technical PCP normally contains about 86 % pentachlorophenol.
[4] Technical dieldrin contains a minimum of 85 % HEOD.
[5] This plug is fitted to absorb unmethylated PCP.

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2.3.3 *Micro-pipette, syringe type*, suitable for accurately injecting 1 µL, and with a needle length so that on-column injection is avoided.

2.3.4 *Carrier gas*, nitrogen flowing at a rate of 75 mL/min, dried by passage through a molecular sieve before entering the column.

NOTE An argon/methane mixture is also suitable for use with a pulsed mode detector.

2.3.5 *Soxhlet apparatus* complying with BS 2071.

2.4 Procedure

2.4.1 *Preparation of the column.* Condition the column using nitrogen as the carrier gas for about two days at a temperature of approximately 225 °C with the detector disconnected.

2.4.2 *Instrument setting and operation.* Operate the instrument in accordance with the requirements stated in **2.3.2**.

NOTE Using these conditions the following retention times are typical of those obtained:

PCP methyl ether	1.9 min
γ-HCH	2.8 min
aldrin	3.5 min
p,p′-DDE	7.7 min
HEOD (dieldrin)	11.7 min

Changes in the oven temperature, gas flow rate and column dimensions may be made to vary the retention times according to the operator's requirements. However, it is important that complete resolution of the peaks is attained.

2.4.3 *Preparation of calibration solutions*

NOTE The calibration solutions prepared as described in this clause are stable for limited periods only and it is recommended that working standards are freshly prepared.

2.4.3.1 *Methylation of the PCP standard solution.* Pipette 10 mL of PCP standard solution (**2.2.9**) into a 50 mL conical flask and add diazomethane solution (**2.2.7**) until the yellow colour persists. Leave for at least 2 h and, if the yellow diazomethane is still present, gently warm the solution in the fume cupboard until colourless. Transfer the solution quantitatively to a 100 mL one-mark volumetric flask and make up to the mark with hexane (**2.2.1**).

2.4.3.2 *PCP calibration solutions.* Pipette 1 mL, 2 mL, and 3 mL of the methylated standard PCP solution (**2.4.3.1**) into 10 mL one-mark volumetric flasks, add 1 mL of standard aldrin solution (**2.2.8**) (see note 1) to each and make up to the mark with hexane (**2.2.1**). These calibration solutions contain 0.01 mg/L, 0.02 mg/L and 0.03 mg/L PCP respectively.

NOTE 1 It is recommended that the same pipette should always be used for the addition of the solution used as the internal standard.

NOTE 2 These concentrations and others used in the determinations of γ-HCH and dieldrin have been found to be suitable for a pulsedmode ^{63}Ni electron-capture detector. Instruments using direct current detectors may require the use of solutions about ten times more concentrated. It is important, however, that manufacturer's instructions regarding the maximum amount of organochlorine compounds to be injected are followed.

2.4.3.3 *γ-HCH calibration solutions.* Pipette 1 mL, 2 mL and 3 mL of standard γHCH solution (**2.2.10**) into 10 mL one-mark volumetric flasks, add 1 mL of standard aldrin solution (**2.2.8**) (see note 1 to **2.4.3.2**) to each and make up to the mark with hexane (**2.2.1**). These calibration solutions contain 0.01 mg/L, 0.02 mg/L and 0.03 mg/L γ-HCH respectively.

2.4.3.4 *HEOD calibration solutions* (for dieldrin analysis). Pipette 1 mL, 2 mL and 3 mL of standard HEOD solution (**2.2.12**) into 10 mL one-mark volumetric flasks, add to each 1 mL of standard *p,p*′-DDE solution (**2.2.11**) (see note 1 to **2.4.3.2**) and make up to the mark with hexane (**2.2.1**). These calibration solutions contain 0.01 mg/L, 0.02 mg/L and 0.03 mg/L HEOD respectively.

2.4.4 *Preparation of calibration graphs*

NOTE 1 It is important with this and all other determinations that the volumetric flasks are shaken immediately before the sample for injection is taken.

NOTE 2 It is important that the measurement of the test solution and the calibration solutions should be carried out with the same settings of the chromatograph. Treat the test solution and the calibration solutions in a single series of measurements.

2.4.4.1 *PCP or γ-HCH calibrations.* Inject 1 µL of one of the calibration solutions from **2.4.3.2** (PCP) or **2.4.3.3** (γ-HCH), as appropriate, into the chromatograph (**2.3.2**) and adjust the amplifier attenuation to obtain an aldrin peak height on the recorder chart of about 80 % full scale deflection and measure the peak height for the PCP or γ-HCH, as appropriate. Then inject, in succession, each of the remaining calibration solutions from either **2.4.3.2** or **2.4.3.3**, as appropriate, and measure the peak heights for PCP (or γ-HCH)[6]. Repeat the injections at least once.

[6] For well-resolved peaks with a relatively narrow base, such as are obtained for PCP, γ-HCH, and aldrin, it is appropriate to use the peak height ratio as a means of expressing the chromatograph's response.

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Calculate the peak height ratio values[7] by expressing the peak height obtained for PCP (or γ-HCH) in each solution as a percentage of the corresponding aldrin peak height. Average the peak height ratio values for each concentration of calibration solution and plot a graph of concentration against the respective average peak height ratios for PCP (or γ-HCH).

2.4.4.2 *HEOD calibration*. Inject 1 μL of one of the calibration solutions from **2.4.3.4** into the chromatograph (**2.3.2**) and optimize the amplifier attenuation to give the best measurable peaks for *p,p′*-DDE and HEOD and measure the peak areas[8] for *p,p′*-DDE and HEOD. Then inject, in succession, each of the remaining calibration solutions from **2.4.3.4**. Repeat the injections at least once and measure the peak areas for *p,p′*-DDE and HEOD in each.

Calculate the peak area ratio values[7] by expressing the peak areas obtained for HEOD in each solution as a percentage of the corresponding *p,p′*-DDE peak area. Average the peak area ratio values for each concentration of calibration solution and plot a graph of concentration against the respective average peak area ratios for HEOD.

2.4.5 *Analysis of preservative solutions*

NOTE Depending on the concentration of the constituent and the equipment used, changes may need to be made to the degree of dilution. It is also essential to ensure that the unknown sample does not contain significant amounts of the internal standard to be used or any other compound with a similar retention time.

2.4.5.1 *PCP determination*. Accurately weigh about 0.1 g of the preservative solution into a 50 mL one-mark volumetric flask and make up to the mark with ethanol (**2.2.2**). Pipette 1 mL of this solution into a 100 mL one-mark volumetric flask and make up to the mark with ethanol. Pipette 10 mL of this solution into a 50 mL conical flask and methylate as described in **2.4.3.1**, beginning at the addition of diazomethane solution, and make up to the mark with hexane (**2.2.1**). Pipette 2 mL of this diluted solution into a 10 mL one-mark volumetric flask, add 1 mL of standard aldrin solution (**2.2.8**) (see note 1 to **2.4.3.2**) and make up to the mark with hexane.

Inject 1 μL of this final solution into the chromatograph and measure the peak due to PCP as described in **2.4.4.1**. Determine the concentration of PCP, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

2.4.5.2 *PCPL determination*. Accurately weigh about 0.1 g of the preservative solution into a 50 mL round-bottomed flask. Add 10 mL of morpholine (**2.2.3**) with a few anti-bumping granules, fit a water-cooled condenser and reflux the solution for 30 min. Cool the solution and transfer quantitatively to a 100 mL separating funnel, washing in with two 20 mL portions of water.

Slowly add 20 mL of hydrochloric acid (**2.2.5**), gently agitate the solution and then allow it to stand for 5 min. Add 20 mL of xylene (**2.2.4**), shake the funnel and allow the layers to separate. Transfer as much as possible of the xylene layer by pipette into a 50 mL one-mark volumetric flask. Repeat the extraction and separation with two further 10 mL portions of xylene, transferring the xylene layers to the 50 mL flask and then discard the aqueous layer. Make up the solution to the mark with xylene.

Pipette 1 mL of this solution into a 100 mL volumetric flask and make up to the mark with ethanol (**2.2.2**). Pipette 10 mL of this solution into a 50 mL conical flask and methylate as described in **2.4.3.1**, beginning at the addition of diazomethane solution, and make up to the mark with hexane (**2.2.1**). Pipette 2 mL of this solution into a 10 mL one-mark volumetric flask, add 1 mL of standard aldrin solution (**2.2.8**) (see note 1 to **2.4.3.2**) and make up to the mark with hexane (**2.2.1**).

Inject 1 μL of this final solution into the chromatograph and measure the peak due to PCP as described in **2.4.4.1**. Determine the concentration of PCP, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

2.4.5.3 *γ-HCH determination*. Accurately weigh about 0.1 g of the preservative solution into a 50 mL one-mark volumetric flask and make up to the mark with ethanol (**2.2.2**). Pipette 1 mL of this solution into a 100 mL one-mark volumetric flask and make up to the mark with hexane (**2.2.1**). Pipette 2 mL of this solution into a 10 mL one-mark volumetric flask, add 1 mL of standard aldrin solution (**2.2.8**) (see note 1 to **2.4.3.2**) and make up to the mark with hexane.

Inject 1 μL of this final solution into the chromatograph and measure the peaks due to *γ*-HCH and aldrin and the peak height ratio for *γ*-HCH as described in **2.4.4.1**.

[7] For compounds with longer retention times, such as HEOD and *p,p′*-DDE the peaks will be broader and it is usually necessary to use peak area ratios. These can be calculated on the basis of the peak height multiplied by the peak width at half the peak height. If an integrator is being used peak areas will be obtained automatically for all compounds.

[8] For well-resolved peaks with a relatively narrow base, such as are obtained for PCP, *γ*-HCH, and aldrin, it is appropriate to use the peak height ratio as a means of expressing the chromatograph's response.

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Determine the concentration of γ-HCH, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

2.4.5.4 *Dieldrin determination.* Accurately weigh about 0.1 g of the preservative solution into a 50 mL one-mark volumetric flask and make up to the mark with ethanol (**2.2.2**). Pipette 1 mL of this solution into a 100 mL one-mark volumetric flask and make up to the mark with hexane (**2.2.1**). Pipette 2 mL of this solution into a 10 mL one-mark volumetric flask, add 1 mL of standard *p,p′*-DDE solution (**2.2.11**) (see note 1 to **2.4.3.2**) and make up to the mark with hexane.

Inject 1 μL of this final solution into the chromatograph and measure the peaks due to HEOD and *p,p′*-DDE and the peak area ratio value for HEOD as described in **2.4.4.2**. Determine the concentration of HEOD, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

2.4.6 *Analysis of treated timber*

2.4.6.1 *Preparation of wood sample for analysis.* Prepare the sample for analysis by converting the treated wood into a form suitable for extraction (i.e. shavings or sawdust) as described in Part 1 of this standard.

Divide the prepared sample into two portions of unequal mass (0.1 g and at least 0.5 g respectively). Determine the moisture content of the larger portion as described in **7.2** of Part 1:1978.

NOTE For the purposes of this clause, in line 2 of **7.2** of Part 1:1978, the word "extraction" should be read as "preparation".

2.4.6.2 *Ethanol extraction.* Accurately weigh about 0.1 g of the prepared wood sample and extract it with 40 mL of ethanol (**2.2.2**) in a soxhlet apparatus. Use at least 25 extraction cycles. Transfer the extract quantitatively to a 50 mL one-mark volumetric flask and make up to the mark with ethanol.

2.4.6.3 *PCP determination.* Pipette 10 mL of the solution from **2.4.6.2** into a 50 mL conical flask and methylate as described in **2.4.3.1**, beginning at the addition of diazomethane solution, and make up to 100 mL with hexane (**2.2.1**).

Dilute an aliquot portion of this solution with hexane so that the final solution gives a peak height ratio for PCP within the range of values given in the calibration graph (**2.4.4.1**) including 1 mL of standard aldrin solution (**2.2.8**) (see note 1 to **2.4.3.2**) per 10 mL of the final test solution.

Inject 1 μL of this final solution into the chromatograph and measure the peaks due to PCP and aldrin and the peak height ratio value as described in **2.4.4.1**. Determine the concentration of PCP, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

2.4.6.4 *PCPL determination.* Accurately weigh about 0.1 g of the wood sample and extract it with 40 mL of morpholine (**2.2.3**) in a soxhlet apparatus. Use at least 25 extraction cycles. Transfer the extract quantitatively to a 250 mL separating funnel, washing in with two 20 mL portions of water.

Slowly add 20 mL of concentrated hydrochloric acid (**2.2.5**), gently agitate the solution and then allow it to stand for 5 min. Add 20 mL xylene (**2.2.4**), shake and allow the layers to separate. Transfer as much as possible of the xylene layer into a 50 mL one-mark volumetric flask. Repeat the extraction and separation with two further 10 mL portions of xylene, transferring the xylene layers to the 50 mL flask and finally discard the aqueous layer. Make up the solution to the mark with xylene.

Pipette 10 mL of this solution into a 50 mL conical flask and methylate as described in **2.4.3.1** beginning at the addition of diazomethane solution. Dilute an aliquot portion of this solution with hexane so that the final solution gives a peak height ratio for PCP within the range of values given in the calibration graph (**2.4.4.1**) including 1 mL of standard aldrin solution (**2.2.8**) per 100 mL of the final solution.

Inject 1 μL of this final solution into the chromatograph and measure the peaks due to PCP and aldrin and the peak height ratio values as described in **2.4.4.1**. Determine the concentration of PCP, in mg/L, by reference to the calibration graph (**2.4.4.1**) (see note 2 to **2.4.4**).

2.4.6.5 *γ-HCH determination.* Dilute an aliquot portion of the ethanol extract from **2.4.6.2** with hexane (**2.2.1**) so that the final solution gives a peak height ratio for γ-HCH within the range of values given in the calibration graph (**2.4.4.1**) including 1 mL of standard aldrin solution (**2.2.8**) per 10 mL of the final solution.

Inject 1 μL of this final solution into the chromatograph and measure the peak heights due to γ-HCH and aldrin as described in **2.4.4.1**. Determine the concentration of γ-HCH, in mg/L, by reference to the calibration graph (see note 2 to **2.4.4**).

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2.4.6.6 *Dieldrin determination*. Dilute an aliquot portion of the ethanol extract from **2.4.6.2** with hexane (**2.2.8**) so that the final solution gives a peak height ratio for HEOD within the range of values given by the calibration graph (**2.4.4.2**) including 1 mL of standard *p,p′*-DDE solution per 10 mL of the final solution.

Inject 1 µL of this final solution into the chromatograph and measure the peak heights due to HEOD and *p,p′*-DDE as described in **2.4.4.2**. Determine the concentration of HEOD, in mg/L, by reference to the calibration graph.

2.4.7 ***Procedure for two or more of the compounds PCP, PCPL, γ-HCH and dieldrin present in preservative solutions or treated timber***

2.4.7.1 *Introduction*. The procedure for analysis of such solutions depends on the relative amounts of the individual compounds present. In preservative solutions the normal ratio of γ-HCH or dieldrin to PCP is between 1 : 1 and 1 : 10, but can exceed this range in timber, particularly if two or more treatments are applied. If PCPL is present in the solution for analysis, a series of absorption peaks will be obtained one of which will interfere with the determination of dieldrin and the others will increase the overall time for analysis considerably.

Use the modifications given in **2.4.7.2** to **2.4.7.5** to the procedures for individual compounds given in **2.4.5** and **2.4.6** to avoid overloading the detector of the chromatograph. These modifications are set out primarily with respect to **2.4.5** for preservative solutions; if testing wood samples, make the corresponding changes to the procedures given in **2.4.6**.

NOTE Other combinations of the preservatives may require different modifications to overcome the overloading of the detector.

2.4.7.2 *γ-HCH and dieldrin present, PCP and PCPL absent*. If the relative amounts of the two constituents are similar, prepare a solution in hexane as described in **2.4.5.3**, pipette 2 mL of this solution into a 10 mL one-mark volumetric flask and add 1 mL of standard aldrin solution (**2.2.8**) and 1 mL of the standard *p,p′*-DDE solution (**2.2.11**) and make up to the mark with hexane (**2.2.1**).

Inject 1 µL of this final solution into the chromatograph and measure the peaks due to γ-HCH, HEOD, aldrin and *p,p′*-DDE and determine the concentration of γ-HCH and HEOD as described in **2.4.5.3** and **2.4.5.4** respectively.

NOTE 1 It may be necessary, for some samples, to adjust the amplifier attenuation to allow for the different response of γ-HCH and HEOD. If so, the adjustment should be made only after all the aldrin has passed.

NOTE 2 If the ratio of the amounts of γ-HCH and dieldrin is very large, e.g. in extracts from treated timber, divide the initial hexane solution into two suitable aliquot portions and determine the γ-HCH and dieldrin separately using the procedures described in **2.4.5.3** and **2.4.5.4** respectively, taking care to avoid overloading the detector with the major component.

2.4.7.3 *PCP and γ-HCH and/or dieldrin present, PCPL absent*. Follow the procedure described in **2.4.5.1** to obtain the solution in hexane after methylation and pipette 2 mL of this solution into a 10 mL one-mark volumetric flask. If PCP and γ-HCH only are present, add 1 mL of standard aldrin solution (**2.2.8**). If PCP and dieldrin or PCP, γ-HCH and dieldrin are present, add 1 mL of standard aldrin solution and 1 mL of standard *p,p′*-DDE solution (**2.2.11**). Make up the solution to the mark with hexane (**2.2.1**).

Inject 1 µL of this final solution into the chromatograph and measure the peaks due to PCP, γ-HCH, HEOD, aldrin and *p,p′*-DDE as appropriate, as described in **2.4.5.1**, **2.4.5.3** and **2.4.5.4** respectively.

If the peaks due to γ-HCH and HEOD are too small, it will be necessary to prepare a test solution from which the majority of the PCP has been removed before analysing for γ-HCH and HEOD. Prepare a hexane solution as described in **2.4.5.3** or **2.4.5.4** and transfer a suitable aliquot (e.g. 25 mL) into a separating funnel and shake with one-fifth of its volume of sodium hydroxide solution (**2.2.6**). After separation discard the aqueous layer, which should contain the majority of the PCP contained in the original mixture.

Pipette 2 mL of the hexane layer into a 10 mL one-mark volumetric flask and add 1 mL of standard aldrin solution and/or standard *p,p′*-DDE solution, as appropriate, and make up to the mark with hexane. Determine the concentration of γ-HCH and/or HEOD as described in **2.4.5.3** and **2.4.5.4**.

NOTE Any PCP not extracted into the sodium hydroxide solution should be retained on the glass wool at the head of the column and the chromatogram should only give peaks for γ-HCH, HEOD and the respective internal standards added.

2.4.7.4 *PCPL and γ-HCH and/or dieldrin present, PCP absent*. Follow the procedure described in **2.4.5.2** to obtain a solution in xylene after saponification with morpholine and acidification. Transfer the xylene solution to a separating funnel and extract with one-fifth of its volume of sodium hydroxide solution (**2.2.6**). Run off the aqueous layer into a second separating funnel. Extract the remaining xylene layer with two 10 mL portions of sodium hydroxide solution and add the extracts to the second separating funnel. Retain the xylene layer for the determination of γ-HCH and/or dieldrin.

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Acidify the combined aqueous extracts with hydrochloric acid (**2.2.5**) and extract with 10 mL of xylene. Transfer the xylene extract to a 50 mL one-mark volumetric flask and repeat the extraction of the aqueous layer with two further portions of 5 mL of xylene adding the extracts to the flask. Dilute the combined xylene extracts to the mark with xylene then methylate and determine the PCP content as described in the last two paragraphs of **2.4.5.2**. The value obtained corresponds to the PCPL derived from the original mixture.

Dilute 1 mL of the xylene layer from the initial extraction to 100 mL with hexane (**2.2.1**) and determine the γ-HCH and/or dieldrin content as described in **2.4.5.3** and/or **2.4.5.4** after addition of the appropriate internal standards.

2.4.7.5 *PCP and PCPL present, γ-HCH and dieldrin present or absent*

NOTE The PCPL content is converted to PCP as described in **2.4.5.2** without changing the PCP already present and the total PCP is determined; a separate test portion is used for the determination of the initial PCP content and the initial PCPL content calculated from the difference between the two PCP contents after calculation (**2.5**).

Follow the procedure described in **2.4.5.2** and determine the total concentration of PCP, in mg/L. Calculate the PCP plus PCPL content of the sample as PCP (see **2.5**).

Accurately weigh a further portion of preservative solution, about 0.1 g, into a 50 mL one-mark volumetric flask and make up to the mark with xylene (**2.2.4**). Pipette a suitable aliquot portion into a separating funnel, dilute to about 20 mL and extract with 20 mL of sodium hydroxide solution (**2.2.6**) and run off the aqueous layer into a second separating funnel. Extract the remaining xylene layer with two further 10 mL portions of sodium hydroxide solution and add the aqueous extracts to the second separating funnel. Acidify the combined aqueous extracts with hydrochloric acid (**2.2.5**) and extract with xylene and methylate as described in paragraph 2 of **2.4.7.4** and determine the concentration of PCP, in mg/L, in the final solution. Calculate the PCP content of the sample (see **2.5**).

Calculate the PCP derived from the PCPL content of the sample as the difference between the two PCP contents and convert it to the PCPL content as described in **2.5.2**.

2.5 Calculations

NOTE The quantities determined according to the procedures in **2.4** are the masses of pure preservative compounds PCP, γ-HCH and HEOD and these are expressed as concentrations in the test sample using the formula in **2.5.1**.

If the concentrations are required in terms of technical grade material the values found should be converted using the factors given in **2.5.2**.

2.5.1 *Pure preservatives*

2.5.1.1 *Preservative solution*. The percentage by mass of preservative in the test sample is given by:

$$\frac{C \times V}{m \times 10\ 000}$$

where

- *C* is the concentration, in mg/L, of preservative in the final test solution as determined from the calibration graph;
- *V* is the volume, in mL, into which the mass of preservative taken for analysis was effectively diluted;
- *m* is the mass, in g, of preservative taken for analysis.

NOTE If the precise quantities and dilutions recommended in the procedures described in **2.4.5** have been used the calculations may be simplified as follows:

a) For PCP (see **2.4.5.1**) V = 250 000 mL; m = 0.1 g; formula simplifies to 250 × C.

b) For PCPL (see **2.4.5.2**) V = 250 000 mL; m = 0.1 g; formula simplifies to 250 × C (as PCP).

c) For γ-HCH (see **2.4.5.3**) V = 25 000 mL; m = 0.1 g; formula simplifies to 25 × C.

d) For dieldrin (see **2.4.5.4**) V = 25 000 mL; m = 0.1 g; formula simplifies to 25 × C (as HEOD).

2.5.1.2 *Treated timber*. The percentage by mass of preservative in the test sample is given by:

$$\frac{C \times V}{m \times 10\ 000}$$

where

- *C* is the concentration, in mg/L, of the preservative in the final test solution as determined from the calibration graph;
- *V* is the volume, in mL, into which the preservative extracted from the test sample was effectively diluted;
- *m* is the oven-dry mass of the test portion, in g, i.e. the mass of the test portion (see **2.4.6.2** or **2.4.6.4**) corrected for the moisture content (see **2.4.6.1**).

2.5.2 *Technical grade preservatives*. The factors for converting the concentrations of PCP and HEOD found to the corresponding concentrations in terms of technical grade material are as follows.

Technical PCP = 1.16 × PCP

Technical PCPL = 1.71 × PCP

Technical dieldrin = 1.18 × HEOD

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3 Method II. Colorimetric method

3.1 Principle. PCP in treated wood is extracted with acetic acid/methanol and preservative solutions containing PCP are diluted with acetic acid/methanol. The solution of PCP is transferred to an ion-exchange column and lower phenols and other constituents are removed by elution with acetic acid/methanol. PCP is eluted from the column with acetic acid and extracted into 1,1,1-trichloroethane.

PCPL in treated wood and preservative solutions containing PCPL are saponified with potassium hydroxide in ethanediol to liberate PCP which is extracted into 1,1,1-trichloroethane.

The test solution of PCP is evaporated to dryness and the residue taken up in methanol and reacted with 4-aminophenazone to produce a coloured complex which is extracted into 1,1,1-trichloroethane and determined photometrically at 585 nm.

3.2 Reagents. All reagents shall be of a recognized analytical reagent quality and water complying with BS 3978 shall be used throughout.

3.2.1 *Anion exchange resin*[9)]

3.2.2 *Acetic acid, glacial*

3.2.3 *Hydrochloric acid*, concentrated (ρ = 1.18 g/mL).

3.2.4 *Methanol*

3.2.5 *Standard PCP solution*. Weigh 0.100 g of PCP, (99 % pure) into a 100 mL one-mark volumetric flask, washing-in with 1,1,1-trichloroethane (**3.2.6**). Shake to dissolve and make up the solution to the mark with 1,1,1-trichloroethane.

3.2.6 *1,1,1-trichloroethane*, uninhibited.

3.2.7 *Acetic acid/methanol solution*. Dilute 100 mL of glacial acetic acid to 500 mL with methanol and mix.

3.2.8 *4-aminophenazone solution*. Dissolve 0.15 g of 4-aminophenazone in water, dilute to 50 mL with water and mix. This solution should be prepared freshly each day.

3.2.9 *Ammonium persulphate solution*. Dissolve 4 g of ammonium persulphate in water, dilute to 50 mL with water and mix. This solution should be prepared freshly each day and stored at 0 °C.

3.2.10 *Buffer solution*, pH value 7.0 to 7.5. Dissolve 0.13 g of *di*sodium tetraborate decahydrate ($Na_2 B_4O_7 . 10H_2O$) and 6.1 g of boric acid in water and dilute to 500 mL with water, mix and check the pH of the prepared solution; if necessary adjust the pH value by the addition of sodium hydroxide solution.

3.2.11 *Potassium hydroxide solution*, 2 mol/L in ethanediol. Dissolve 130 g of potassium hydroxide in sufficient ethanediol without heating then make up the volume to 1 L with additional ethanediol. If the solution is more than a week old, or becomes strongly coloured, discard it and prepare a fresh solution.

3.2.12 *Silver nitrate solution*. Dissolve 1.7 g of silver nitrate in 100 mL of a nitric acid solution prepared by diluting 100 mL of concentrated nitric acid solution (ρ_{20} = 1.42) to 220 mL with water.

3.2.13 *Sodium acetate solution*. Dissolve 100 g of sodium acetate trihydrate in water and dilute to 1 L with water.

3.3 Apparatus

3.3.1 *Chromatographic apparatus*. Semi-micro apparatus with ground glass joints, with an effective column length of 100 mm, a nominal bore of 10 mm and a grade 0 (BS 1752[10)]) porosity sintered glass retaining disc. The reservoir should have a nominal capacity of 50 mL and the Buchner receiving flask a nominal capacity of 150 mL.

3.3.2 *Soxhlet apparatus*, complying with BS 2071.

3.3.3 *Thermostatically-controlled water bath*, capable of operating at 20 ± 0.5 °C.

3.3.4 *Spectrophotometer or absorptiometer*, suitable for operation at a wavelength of 585 nm with suitable cells.

[9)] Dowex 2-8x, chloride form has been found suitable.
[10)] BS 1752 is in course of revision and the corresponding grade should be a pore size index P250.

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3.4 Procedure

3.4.1 *Preparation of chromatographic column*. Soak a quantity of resin (**3.2.1**) in water for approximately 24 h, then prepare a slurry of this resin in water and transfer sufficient to the column to give a bed 80 mm deep when settled. Elute with sodium acetate solution (**3.2.13**) ensuring that the level of the liquid in the column is kept at least 5 mm above the top of the bed. Adjust the elution rate to one drop per second and if suction is used to maintain this rate, take care to avoid compacting the bed. Check the eluate periodically for the presence of chloride, using silver nitrate solution (**3.2.12**). When no chloride is detected, elute, in succession, with 100 mL of water, 50 mL of glacial acetic acid (**3.2.2**) and 100 mL of methanol (**3.2.4**) and keep the column covered with methanol until used.

After each estimation regenerate the resin column by eluting, in succession, with 50 mL of glacial acetic acid and 50 mL of methanol.

NOTE Throughout all elution operations ensure that the level of liquid is kept at least 5 mm above the top of the bed.

3.4.2 ***Analysis of preservative solutions***

3.4.2.1 *Solutions containing PCP*. Prepare accurately a dilution of the preservative solution in acetic acid/methanol solution (**3.2.7**) which will give a concentration of PCP of approximately 50 µg/mL[11]. Dilute 1.00 mL of this solution with 25 mL of acetic acid/methanol solution.

Transfer this dilution to a freshly-prepared chromatographic column (**3.4.1**), and elute using 125 mL of acetic acid/methanol solution. Complete the elution with a further 125 mL of acetic acid/methanol solution and discard the eluate. Then elute with 50 mL of glacial acetic acid (**3.2.2**) and collect the eluate.

Transfer the eluate quantitatively to a 250 mL pear-shaped separating funnel and dilute to about 200 mL with water, mix and extract, in succession, with 5 mL, 4 mL and 3 mL of 1,1,1-trichloroethane (**3.2.6**), collecting the extracts in a 50 mL round-bottomed flask[12] held at 20 °C in a thermostatically-controlled water bath (**3.3.3**). Remove the solvent by passing a stream of air (free from oil and dust) at 20 °C through the flask and dissolve the residue in approximately 1 mL of methanol (**3.2.4**).

Add, in succession, with mixing, 10 mL of buffer solution (**3.2.10**), 0.5 mL of 4-aminophenazone solution (**3.2.8**) and 0.5 mL of ammonium persulphate solution (**3.2.9**). A strict time schedule shall be adhered to during the colour development and measurement (see note 1). Reagents should be brought to a temperature as close as possible to 20 °C before addition. Leave the flask to stand in the thermostatically-controlled bath for 3.5 min then pipette 5 mL of 1,1,1-trichloroethane (**3.2.6**) into the flask and shake for 1.5 min.

Transfer the contents of the flask to a 50 mL pear-shaped separating funnel, allow the phases to separate, then filter the 1,1,1-trichloroethane layer through a phase separation paper into a suitable cell and measure the absorption of the solution at 585 nm. Determine the mass of PCP, in µg, present by reference to the calibration graph (**3.4.4**).

NOTE 1 The coloured complex produced is both time and temperature sensitive.

NOTE 2 Throughout the separation and determination the temperature should be kept as close to 20 °C as possible.

3.4.2.2 *Solutions containing PCPL*. Transfer accurately a suitable mass of sample[13] to a 1 L round-bottomed flask. Add 100 mL of potassium hydroxide solution (**3.2.11**), 5 mL of water and a few anti-bumping granules. Boil under reflux for 30 min. Allow the flask and contents to cool to approximately 50 °C and then add 400 mL of water followed by 100 mL of hydrochloric acid (**3.2.3**). Arrange the flask and the condenser for normal distillation and distil at a steady rate until 400 mL of the distillate has been collected, then stop the distillation and allow the apparatus to cool.

Transfer the distillate to a 500 mL pear-shaped separating funnel and wash the condenser with 25 mL of 1,1,1-trichloroethane (**3.2.6**), collecting the washings in the separating funnel. Stopper and shake the separating funnel thoroughly, allow the two layers to separate and collect the 1,1,1-trichloroethane layer in a 100 mL round-bottomed flask, avoiding the transfer of any water. Extract the contents of the separating funnel with a further 25 mL portion of 1,1,1-trichloroethane (**3.2.6**) and transfer this also to the 100 mL round-bottomed flask.

[11] Commercial formulations containing 5 % (*m*/*m*) PCP have to be diluted 1 000 times to achieve this concentration.
[12] It may be necessary to remove water by use of a phase separation paper.
[13] For a 5 % solution of PCPL, take 1.4 g of the preservative solution.

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Place the flask in a thermostatically-controlled bath set at 20 °C and evaporate the solvent by passing a jet of air (free from oil and water) at 20 °C through the flask. Dissolve the residue left in the flask in acetic acid/methanol solution (**3.2.7**), transfer quantitatively to a 250 mL one-mark volumetric flask, make up to the mark with acetic acid/methanol solution and shake well to mix.

Transfer a 10 mL aliquot portion to a 100 mL one-mark volumetric flask and make up to the mark with acetic acid/methanol solution, shake well to mix and dilute 1.00 mL of this solution with 25 mL of acetic acid/methanol solution. Proceed as described in **3.4.2.1** beginning at the stage at which the dilution is transferred to the chromatographic column.

3.4.3 ***Analysis of treated wood***

3.4.3.1 *Preparation of wood sample for analysis.* Prepare the sample for analysis by converting the treated wood into a form suitable for extraction (i.e. shavings or sawdust) as described in Part 1 of this standard.

Divide the prepared sample into test portions including one of at least 0.5 g. Determine the moisture content of the 0.5 g test portion as described in **7.2** of Part 1:1978.

NOTE For the purposes of this clause in line 2 of **7.2** of Part 1:1978 the word "extraction" should be read as "preparation".

3.4.3.2 *Samples containing PCP.* Accurately weigh a suitable mass of the prepared wood sample and extract with 75 mL of acetic acid/methanol solution (**3.2.7**) in a soxhlet apparatus. Use at least 25 extraction cycles. Allow the extract to cool, with the reflux condenser in position and then transfer the extract to a freshly-prepared chromatographic column (**3.4.1**). Wash the soxhlet apparatus, condenser and flask with three 10 mL portions of acetic acid/methanol solution and transfer the washings to the column reservoir. Elute with 125 mL of acetic acid/methanol solution and discard the eluate. Then elute with 50 mL of glacial acetic acid (**3.2.2**) and collect the eluate.

Proceed as described in **3.4.2.1**, beginning at the transfer of the eluate to a separating funnel.

3.4.3.3 *Samples containing PCPL.* Accurately weigh a suitable mass of wood sample into a 1 L round-bottomed flask. Proceed as described in **3.4.2.2**.

3.4.4 *Preparation of calibration graph.* Pipette 10 mL of standard PCP solution (**3.2.5**) into a 100 mL one-mark volumetric flask and make up to the mark with 1,1,1-trichloroethane (**3.2.6**). Pipette 10 mL of this solution into a 100 mL volumetric flask and make up to the mark with 1,1,1-trichloroethane to give a solution containing 10 µg/mL PCP. Transfer by pipette 1 mL, 2 mL, 3 mL, 4 mL, 8 mL and 14 mL of this solution to a series of 50 mL round-bottomed flasks. The flasks will then contain 10 µg, 20 µg, 30 µg, 40 µg, 80 µg and 140 µg of PCP respectively. Place the flasks in a thermostatically-controlled bath (**3.3.3**) at 20 °C and when the flasks and their contents have reached temperature equilibrium, proceed as described in **3.4.2.1** beginning at the stage at which the solvent is removed by passing a stream of air through the flask. Adhere to a strict time schedule during the colour development and measurement. Plot the absorbances obtained against mass of PCP in micrograms.

3.5 Calculation

NOTE The quantities determined according to the procedures described in **3.4** are the masses of pure preservative and these are expressed as concentrations in the test sample using the formulae in **3.5.1**.
If the concentrations of technical grade material are required, the values found should be converted using the factors given in **3.5.2**.

3.5.1 ***Pure preservatives***

3.5.1.1 *Preservative solutions.* The percentage by mass of preservative in the test sample is given by:

$$\frac{m_1 \times V}{m_2 \times 10\,000}$$

where

- m_1 is the mass, in µg, of preservative in the final test solution as determined from the calibration graph;
- V is the volume, in mL, into which the mass of preservative taken for analysis was effectively diluted;
- m_2 is the mass, in µg, of preservative taken for analysis.

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3.5.1.2 *Treated timber*. The percentage by mass of preservative in the test sample is given by:

$$\frac{m_1 \times V}{m_2 \times 10\,000}$$

where

- m_1 is the mass, in μg, of preservative in the final test solution as determined from the calibration graph;
- V is the volume, in mL, into which the preservative extracted from the test sample was effectively diluted;
- m_2 is the oven-dry mass of the test portion, in g, i.e. the mass of the test portion (see **3.4.3.2** or **3.4.3.3**) corrected for the moisture content (see **3.4.3.1**).

3.5.2 *Technical grade preservatives*. The factors for converting the concentrations of PCP found to the corresponding concentrations in terms of technical grade material are as follows.

Technical PCP = 1.16 × PCP

Technical PCPL = 1.71 × PCP

4 Test report

The test report shall include the following information:

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

b) a reference to this British Standard and the method used, i.e. method I or II of BS 5666-6;

c) any deviation from the method described;

d) the results of the analysis;

e) any unusual features noted during the analysis.

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Publications referred to

BS 507, *Ethanol.*
BS 1583, *One-mark pipettes.*
BS 1752, *Laboratory sintered or fritted filters.*
BS 1792, *Specification for one-mark volumetric flasks.*
BS 3591, *Industrial methylated spirits.*
BS 3978, *Water for laboratory use.*
BS 5666, *Methods of analysis of wood preservatives and treated timber.*
BS 5666-1, *General considerations and sampling and preparation of materials for analysis.*
A I Vogel *A textbook of practical organic chemistry*. Longmans. 1978
Anal Chem. 1973, **45**.

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