

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)
- Ministry of Defence

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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.

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, disodium 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 3591¹⁾.

2.2.3 Morpholine

2.2.4 Xylene

2.2.5 Hydrochloric acid, concentrated ($\rho = 1.18 \text{ g/mL}$).

2.2.6 Sodium hydroxide solution, $c(\text{NaOH}) = 0.1 \text{ mol/L}$ 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.

2.2.7 Diazomethane solution²⁾. 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 % pure³⁾, 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 μ m to 175 μ m) chromosorb W AW/DMCS carrying 5 % of QF 1 and fitted with a silanized glass-wool plug at its head⁵⁾;
- e) detector type, ⁶³Ni 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.